The Impact of Modification Technologies on the Physicochemical, Rheological, and Thermal Characteristics of Selected Improved Non-Waxy Rice Flours.

By

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DEDICATION

This thesis is dedicated to Emeka David Ngadi,

who always thought it will be cool to call me "Dr. Anthoniii".

In the vastness of space and enormity of time, it is was a joy to coexist on this planet with you my brother.

ABSTRACT

Physical modification of rice flour plays a pivotal role in the adoption and utilization of non-waxy rice cultivars. Understanding and optimizing the impact of applying physical modification techniques holds the potential of enhancing food security in non-waxy rice producing communities through the development of acceptable novel rice-based products. This study investigated the impact of selected modification technologies on the physicochemical, rheological and thermal characteristics of non-waxy rice flours.

Structural, rheological, thermal, functional and physicochemical properties of flours from seven non-waxy rice cultivars were studied by different techniques namely X-ray diffractometry, rheometry and differential scanning calorimetry (DSC). Physicochemical properties varied for all the samples studied. Protein content ranged between 8.47 to 9.24%, while amylose content was between 25.90 and 30.85%. Relative crystallinity degree (%) of rice flours were significantly different, with the values ranging from 15.63 – 23.40%. Water holding capacity of rice flours ranged from 1.13 - 1.49 g/g. A strong negative correlation (0.965) was observed between the water holding capacity (WHC) of the rice flours and amylose contents. Oil holding capacity (OHC) of the tested flours ranged from 0.93 -1.27 g/g. A strong positive correlation (0.965) was observed between the OHC of the rice flours and their amylose contents. Glass transition temperature (T_g) values ranged between 45.43 – 72.57 °C. Onset of gelatinization (T_o), gelatinization peak,(T_p) and gelatinization conclusion, (T_c) temperatures values ranged from 56.70 - 81.27 °C, 62.91 – 87.57 °C and 72.47 – 89. 87 °C, respectively. Enthalpy of gelatinization, ΔH values was between 0.38 - 7.46 J/g.

Non-waxy rice flours were treated at different combinations of temperatures (85, 100 and 115 °C), times (1, 2 and 3 hr) and adjusted moisture content (25, 30 and 35 %). Processing temperature and moisture content during the heat-moisture treatment significantly ($P < 0.05$) influenced the physicochemical, thermal and rheological properties of the rice flours. Variation of the moisture contents and processing temperatures during heat-moisture treatment (HMT) led to a 3.75% reduction in the protein contents of the flour. Increase in processing temperature led to an increase in the WHC of the rice flours. Increasing processing temperature during the thermal treatment led to at least 20% increase in the WHC of the flours. OHC properties reduced from 1.28 – 1.20 g/g, when the moisture content was increased during HMT. Lightness (L*) of heat-moisture treated

SAH 177 flour was at least 4 - 6.25% less than the native flour. A 1.5°C decrease in gelatinization temperature (T_p) was observed for every percent increase in moisture content. Moisture content during HMT and ΔH were inversely associated. A 1 J/g reduction in the enthalpy of gelatinization for every percentage increase in the moisture content was observed during the HMT process. G', reduced from (63.86 Pa) to (5.84 Pa), as moisture contents were increased from 25 to 35% during HMT. G", values reduced from 17.21 to 4.97 Pa, as the processing temperature increased from 85 to 115 °C. Regression models developed for predicting the WHC, OHC, L*, b*, T_p and ΔH of the rice flours during HMT had a high R^2 of at least to the 80% significance.

Rheological, textural and color properties of rice puddings made from heat-moisture treated rice flours were assessed. G' and G" values of the pudding samples made from the heat-moisture treated flours were (0.44- 1121 Pa) and (0.18 - 79.50 Pa), respectively, while the G' and G" ranges of the puddings made from the control flours were (1.70 – 1034 Pa) and (0.15 - 101.50 Pa), respectively. Puddings exhibited thixotropic behavior with G' > G" at the studied frequencies (10 – 100 Hz). Hardness (0.04 - 0.16 N), gumminess (0.01 - 0.15 N) and chewiness (0.01 - 0.15 N) of puddings made from heat-moisture treated flours was higher than the hardness (0.14 - 0.26 N), gumminess (0.12 - 0.22 N) and chewiness (0.12 - 0.21 N) properties of the puddings made from the control rice flours. HMT significantly ($P < 0.05$) influenced the overall color (ΔE) of the rice puddings. Application of the heat-moisture treated rice flours increased the ΔE values of the puddings.

Physicochemical, rheological and thermal studies were carried out on microwave processed non-waxy rice flours. Variation of the moisture contents, processing time and power during microwave treatment significantly ($P < 0.05$) influenced the amylose content, viscosity, hardness, OHC and WHC of the non-waxy rice flours. OHC properties increased from 1.28 – 3.10 g/g, while WHC increased from 1.22 – 3.50 g/g during microwave treatment indicating moisture and time effects. However, interestingly the first 15 sec of microwave treatment saw at least a 23.58% reduction in the oil and water holding properties of the rice flour. The application of this microwaved rice flours with reduced oil and water holding capacities on quality properties of air fried chicken breast cuts were evaluated. Porosity ranges for the samples coated with untreated rice flour was 25.17 – 88.90%, while samples coated with microwaved rice flours ranged from 13.99 – 77.38%. The application of microwaved treated rice flours increased the coating pick up and cooking yield of the fried samples. Other properties of the fried samples such as texture, moisture content, oil content, and color were significantly ($P < 0.05$) influenced by the flour treatment.

Ultrasonication was used to investigate the effect of varying amplitudes over time at different moisture contents on non-waxy rice flour characteristics. Variation of the moisture contents, processing time and amplitude during ultrasound treatment significantly ($P < 0.05$) influenced the amylose content, swelling power, oil and water holding properties of the non-waxy rice flours. Amylose contents increased with ultrasonication time. The lowest swelling power was found in the untreated rice flour samples while the highest water holding properties were obtained in the flour samples treated under ultrasonication having around 24.59 and 58.20% water increase at amplitude levels of 20 and 30%, respectively, compared to the untreated sample. Other properties of the non-waxy rice flour such as protein contents, rheological and thermal were significantly ($P < 0.05$) influenced by ultrasonication treatment.

Finally, a comparative analysis was performed to evaluate the application of untreated, microwave and ultrasound treated non-waxy rice flours on the quality of air-fried chicken samples. The effective moisture diffusivity for the untreated coated, ultrasound coated, and microwave coated ranged between 5.68×10^{-5} to 12.14×10^{-5}, 4.90×10^{-5} to 8.01×10^{-5} and 5.36×10^{-5} to 6.34×10^{-5} m^2/s, respectively, and the R^2 values ranged between 0.89 and 0.98. Increased activation energy for moisture loss was observed in the samples coated with untreated rice flours, while the lowest activation energy for oil uptake was observed in samples coated with ultrasound treated rice flour.

RÉSUMÉ

La modification physique de la farine de riz joue un rôle essentiel dans l'adoption et l'utilisation de cultivars de riz non cireux. Les données obtenues grâce à l'application de techniques de modification physique ont le potentiel d'améliorer la sécurité alimentaire dans les communautés de producteurs de riz non cireux grâce au développement de nouveaux produits à base de riz acceptables. Cette étude a examiné l'impact de certaines technologies de modification sur les caractéristiques physicochimiques, rhéologiques et thermiques de certaines farines de riz non-cireuses améliorées.

Les propriétés structurelles, rhéologiques, thermiques, fonctionnelles et physicochimiques de farines provenant de sept cultivars de riz non-cireux du Nigeria du Sénégal ont été étudiées grâce à différentes techniques, à savoir diffractométrie à rayons X, rhéomètre, et calorimétrie différentielle à balayage (DSC). Les propriétés physicochimiques ont varié à travers chaque échantillon étudié. La teneur en protéines a varié entre 8.47 et 9.24%, tandis que la teneur en amylose a varié de 25.90 à 30.85%. Le degré de cristallinité relative (%) des farines de riz était considérablement différent avec des valeurs allant de 15.63 à 23.40. La capacité de rétention d'eau des farines de riz a varié de 1.13 à 1.49 g/g. Une forte corrélation négative (0.965) a aussi été observée entre la capacité de rétention d'eau des farines de riz et leur teneur en amylose. La capacité de rétention d'huile des farines étudiées a varié entre 0.93 et 1.27 g/g. Une forte corrélation positive (0.965) a été observée entre la capacité de rétention d'huile de farines de riz et leur teneur en amylose. Les valeurs de températures de transition vitreuse (T_g) ont varié entre 45.43 et 72.57 °C. Les valeurs de températures marquant le début de gélatinisation (T_o), le maximum de gélatinisation (T_p) et la conclusion de gélatinisation (T_c) ont varié entre 56.70 – 81.27 °C, 62.91 – 87.57 °C et 72.47 – 89.87 °C, respectivement. L'enthalpie de gélatinisation, les valeurs de ΔH, étaient 0.38 et 7.46 J/g.

Les farines de riz non-cireuses ont été traitées à différentes combinaisons de températures (85, 100, 115 °C), intervalles (1, 2 et 3 heures) et teneurs en humidité (25, 30 et 35%). La température de traitement et la teneur en humidité pendant le traitement thermique-humide a influencé considérablement ($P < 0.05$) les propriétés physicochimiques, thermiques et rhéologiques des farines de riz. Une augmentation de la température de traitement a mené à une augmentation de la

capacité de rétention d'eau des farines de riz. Augmenter la température de traitement lors du traitement thermique a mené à une augmentation d'environ 20% de la capacité de rétention d'eau des farines. Les propriétés de rétention d'huile ont diminué de 1.28 à 1.20 g/g, lorsque la teneur en humidité était augmentée pendant le traitement thermique-humide. La clarté (L*) de farine SAH 177 traitée par transformation thermique-humide était au moins 4 à 6.25% inférieure à celle de la farine native. Une baisse de 1.5 °C de la température de gélatinisation (T_p) a été observée pour chaque pourcentage d'augmentation de la teneur en humidité. La teneur en humidité pendant le traitement thermique-humide et ΔH étaient inversement corrélés. Une baisse de 1 J/g de l'enthalpie de gélatinisation pour chaque pourcentage d'augmentation dans la teneur en humidité a été observée lors du traitement thermique-humide. G' a diminué de 63.86 Pa à 5.84 Pa lorsque la teneur en humidité a augmenté de 25 à 35% lors du traitement thermique-humide. Les valeurs de G" ont diminué de 17.21 à 4.97 Pa lorsque la température de traitement a augmenté de 85 à 115 °C. Des modèles de régressions développés pour prédire la capacité de rétention d'eau et d'huile, L*, b*, T_p et ΔH des farines de riz lors du traitement thermique-humide avaient un haut R^2 avec une signifiance d'au moins 80%.

Les propriétés rhéologiques, texturales et de couleur ont été obtenues pour des riz au lait faits à partir de farines de riz traitées par transformation thermique-humide. Les valeurs de G' et G" des échantillons de riz au lait faits à partir de farines traitées par transformation thermique-humide étaient de (0.44 – 1121 Pa) et (0.18 – 79.50 Pa), respectivement, tandis que les intervalles de G' et G" des riz au lait fait à partir des farines de contrôle étaient de (1.70 – 1034 Pa) et (0.15 – 101.50 Pa), respectivement. Les riz au lait faits à partir de farines non-traitées et traitées par transformation thermique-humide ont présenté un comportement thixotropique avec G' > G" et aux fréquences étudiées (10 – 100 Hz). La dureté (0.04 – 0.16 N), la viscosité (0.01 – 0.15 N) et la mastication (0.01 – 0.15 N) des riz au lait faits à partir de farines traitées par transformation thermique-humide étaient supérieures à la dureté (0.14 – 0.26 N), la viscosité (0.12 – 0.22 N) et la mastication (0.12 – 0.21 N) des riz au lait faits à partir de farines de riz de contrôle. Le traitement thermique-humide a influencé considérablement ($P < 0.05$) la couleur globale (ΔE) des riz au lait. L'utilisation des farines de riz traitées par transformation thermique-humide a augmenté les valeurs de ΔE des riz au lait.

Des études physicochimiques, rhéologiques et thermiques ont été réalisées pour des farines de riz non-cireuses traitées par micro-ondes. Des variations de teneur en humidité, de durée de traitement et de puissance pendant le traitement par micro-ondes ont influencé considérablement ($P < 0.05$) la teneur en amylose, la viscosité, la dureté et la capacité de rétention d'eau et d'huile des farines de riz non-cireuses. La capacité de rétention d'huile a augmenté de 1.28 – 3.10 g/g, tandis que la capacité de rétention d'eau a augmenté de 1.22 – 3.50 g/g lors du traitement par micro-ondes ce qui indique l'effet de l'humidité et de la durée. Cependant, les quinze premières secondes de traitement par micro-ondes ont vu une baisse d'environ 23.48 % des propriétés de rétention d'eau et d'huile des farines de riz. L'utilisation de ces farines de riz traitées par micro-ondes avec des capacités réduites de rétention d'eau et d'huile sur des propriétés de qualité de morceaux de poitrine de poulet frit a été évaluée. Les intervalles de porosité des échantillons recouverts de farine de riz non-traitée étaient de 25.17 – 88.90%, tandis que ceux des échantillons recouverts de farine de riz traitée par micro-ondes ont varié de 13.99 – 77.38%. L'utilisation des farines de riz traitées par micro-ondes a augmenté l'absorption de la couche recouvrant la surface extérieure ainsi que le rendement de cuisson des échantillons frits. D'autres propriétés des échantillons frits telles que la texture, la teneur en humidité, la teneur en huile et la couleur ont été influencées considérablement ($P < 0.05$) par le traitement aux microondes de la farine.

Des ultrasons ont été utilisés pour étudier l'effet d'amplitudes évoluant avec le temps à différentes teneurs en humidité sur les caractéristiques de farines de riz non-cireuses. La variation de la teneur en humidité, du temps de traitement et de l'amplitude au cours du traitement par ultrasons a une influence significative ($P < 0.05$) sur la teneur en amylose, le pouvoir de gonflement et les propriétés de rétention d'eau et d'huile. Les teneurs en amylose ont augmenté avec le temps d'exposition aux ultrasons. Le pouvoir de gonflement minimal a été obtenu pour les échantillons de farines de riz non-traitées tandis que les propriétés de rétention d'eau maximales ont été obtenues pour les échantillons de farine traités par ultrasons, avec une augmentation d'eau de 24.59 à 58.20% pour des niveaux d'amplitudes de 20 à 30%, respectivement, comparé aux échantillons non-traités. D'autres propriétés de farines de riz non-cireuses telles que la teneur en protéines, les propriétés rhéologiques et thermiques ont été considérablement ($P < 0.05$) influencées par le traitement aux ultrasons.

Enfin, une analyse comparative a été réalisée afin d'évaluer l'utilisation de farines non-cireuses non-traitées, traitées par micro-ondes et traitées par ultrasons sur la qualité d'échantillons de poulet frit à l'air. La diffusivité hydrique efficace pour la couche recouvrant la surface extérieure non-traitée, traitée par ultrasons et traitée par micro-ondes a varié entre 5.68×10^{-5} et 12.14×10^{-5}, 4.90×10^{-5} et 8.01×10^{-5}, et 5.36×10^{-5} et 6.34×10^{-5} m^2/s, respectivement, et les valeurs R^2 ont varié entre 0.89 et 0.98. Une augmentation de l'énergie d'activation pour perte d'humidité a été observée dans les échantillons recouverts de farine de riz non traitée, tandis que l'énergie d'activation minimale pour prise d'huile a été observée chez les échantillons recouverts de farine de riz traitée par ultrasons.

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CONTRIBUTION OF AUTHORS

The contributions made by the different authors are as follows: **Mr. Chijioke A. Nwankpa** is the principal author of this study. He is the Ph.D. candidate who planned, designed and carried out the various stages of experimentation, data analysis, manuscript writing and revision for scientific publications. **Prof. Michael O. Ngadi** is the thesis supervisor, who guided the candidate in the process of planning, experimental design and execution of the various phases of the work which consist of all mentioned above. **Prof. Benjamin K. Simpson** and **Dr. Ebenezer Kwofie** helped in the development of the ideas and assisted in editing and reviewing some of the manuscripts in this study.

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NOMENCLATURE

a*	Redness
A_a	Amorphous peak
Ac	Crystalline peak
AM	Amylose
ANOVA	Analysis of Variance
AOAC	Association of Official Agricultural Chemists
AP	Amylopectin
b*	Degree of brownness
CUT	Come up time
db	Dry basis
D_{eff}	Effective diffusivity (m^2/s)
D_o	Effective diffusivity at a high liquid concentration (m^2/s)
DSC	Differential scanning calorimetry
E_a	Activation energy (kJ/mol)
Exp	Exponential sign
FC	Fat content (g/g, db)
g/g	Grams per gram
G'	Storage modulus
G''	Loss modulus
HMT	Heat-moisture treatment
k	Consistency index ($Pa \cdot s^n$)

K	rate constant (s^{-1})
kHz	Kilo Hertz
J/g	Joules per gram
L	Half-thickness of the sample (m)
L*	Degree of whiteness
LSD	Least significant difference
M	Instantaneous, initial and equilibrium moisture content (g/g, db)
M_e	Equilibrium moisture content (g/g, db)
M_0	Initial moisture content (g/g, db)
M_r	Moisture ratio ($M - M_e/M_0 - M_e$), dimensionless
MHz	Mega Hertz
mm/s	Millimeter per second
N	Newton
n	Flow behavior
OHC	Oil holding capacity
O_{eq}	Equilibrium fat content (g/g, db)
Pa	Pascal
R	Universal gas constant (8.314 $J.K^{-1}.mol^{-1}$)
rad/s	Radian per second
RMSE	Root Mean Square Error
SP	Swelling power
T	Absolute temperature (K)
tan δ	Loss factor

T_o	Onset of gelatinization
T_c	Conclusion of gelatinization
T_m	Melting temperature
T_g	Glass transition temperature
T_p	Peak of gelatinization
W	Watts
WAI	Water absorption index
WHC	Water holding capacity
W_i	Sample weight
W_r	Residue weight
W/cm	Watts per centimeter
w/w	Weight per weight (%)
X_c	Crystallinity index
ΔH	Enthalpy of gelatinization
ΔE	Overall color change
ɯ	Frequency
η*	Complex viscosity
σ	Shear stress (Pa)
γ	Shear rate (s^{-1})
µm	Micron meters
Ɛ	Porosity (%)
ρ_a	Apparent density (g/ml)
ρ_b	Bulk density (g/ml)

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1.0.General Introduction

1.1. Background

Rice is an important staple food for urban and rural consumers especially in West African countries like Ghana (Tomlins, 2005). There are mainly two species of rice: *Oryza sativa* L. (commonly referred to as Asian rice) and *Oryza glaberrima* Steud. (commonly referred to as African rice). A few decades ago, NERICA (New Rice for Africa) an inter-specific breed of *Oryza sativa* L. and *Oryza glaberrima* S. was released by the African Rice Center (Agnoun et al., 2012). The results of the NERICA project are rice cultivars with improved yields, grain head size, protein contents and increased tolerance to pest, drought and infertile soils (Agnoun et al., 2012; Fofana et al., 2011). The introduction of these varieties has seen an increase in rice production in West Africa, with Nigeria being the largest producer of rice in the region (Semon et al., 2004). Despite the high production of rice in countries like Nigeria, the region remains the second largest importer of rice in the world (Oyinbo et al., 2013). A combination of factors, such as the impact of poor post-harvest practices on rice quality, poor solubility of the rice flours in solvents, poor water binding potentials, reduced elasticity, instability of the flour in food systems under high shear, reduced low local rice production and lack of information on the functionality of flours from these local rice cultivars, can explain the local consumers' preference for foreign rice cultivars.

Rice flour is mainly composed of starch. Like all starches, rice starch is composed of polysaccharide chains of amylose and amylopectin molecules. Starch impacts the functional and rheological power of products. These influence results in control of moisture, texture, viscosity, consistency and shelf life during processing and in the final products. The textural and gelatinization properties of rice grains are characteristics of special importance (Chung et al., 2011). The amylose content of rice is another major factor that controls or explains the eating quality of the final products, such as cooked rice and rice noodles (Mestres et al., 1996; Thomas and Bhat, 2013). The impact of amylose-amylopectin ratio on the functional properties of rice has been widely investigated. Some studies have observed that the gelatinization temperature increased with higher amylose content (Jane et al., 1999; Liu et al., 2011). High amylose content and a higher proportion of long branch chains have been observed to cause an increase in the pasting temperature and setback viscosities of rice starch, and a decrease in the peak viscosity and shear thinning (Chung et al., 2011; Jane et al., 1999). Cooked non-waxy rice (high amylose rice) exhibits less granule disintegration and is harder and less sticky than cooked waxy rice. These properties make this type of rice of reduced interest to the food producers and domestic consumers. The natural arrangement and structural make up of starch in waxy and non-waxy rice are reflected in differences in functional properties such as water absorption, swelling, pasting and gelling behavior (Wang and Copeland, 2013). Rice flour has been utilized in several foods and industrial applications, such as agents for gelling, replacing fat, thickening and cosmetic dusting powder (Chung et al., 2011). The application of the functional properties of rice and its flour in food and non-food applications have been influenced by its crystalline structure, the amylose-amylopectin ratio and the fine structure of amylopectin (Chung et al., 2011; Lafayette, 2007). Industrial applications of rice flour and starch are dependent on their structural and functional properties (Jane et al., 1999; Lafayette, 2007).

Modified biomaterials provide food processors with an adaptable raw material that can meet the unique requirements of a variety of food systems (Kaur et al., 2011). Native flours or starches are typically unsuitable for most applications and therefore, need to be modified physically and/or chemically to improve their positive functionalities and/or to reduce their defects. For instance, the application of modified starch as a water-soluble gelling agent to ensure the stability of mayonnaise in the high-shear emulsion, especially for low-fat content of salad dressings and New Zealand-style mayonnaises, was reported by Depree and Savage (2001). In mayonnaise and salad dressings, much of the rheology of the product depends greatly on the gelling agent rather than the emulsion properties. In comparison to native starches, the application of modified starch as gelling ingredients in these products creates a strong enough gel which aids in stabilizing the food product under high shear without creating an unsatisfactory glutinous texture (Depree and Savage 2001).

A substantial amount of studies have been focussed on physical modification treatments such as heat-moisture treatment (HMT), ultrasound technologies, microwave heating, and high-pressure processing, (Gunaratne and Hoover, 2002; Lionetto et al., 2006b; Yu et al., 2013), and chemical modification, such as cross-linking and enzymatic modification (Kido and Kobayashi, 2013; Kurakake et al. 2009). The impact of modification technologies, such as heat-moisture treatment and ultrasound technologies, has been studied on several food materials such as beans, potato, and pea (Collado and Corke, 1999; Hoover and Manuel, 1996; Sankhon et al., 2014). These physical modification treatments have been shown to result in different changes in the crystallinity, swelling factors, water binding capacity, amylose content and pasting viscosities in certain biomaterials.

These changes have provided numerous desirable functional properties needed in various industries. For instance, starches and flours with increased swelling properties are more suitable for baking processes. Several studies have been reported on the effects of HMT (Hormdok and Noomhorm, 2007a), ultrasound (Manchun et al., 2012) and microwave treatments (Guardeño et al., 2011; Román et al., 2015) on rice starch, whereas relatively little work has been done on properties of waxy rice flour. Therefore, for industrial classification and control purposes, it is important to understand how these non-waxy rice flours responds to modification treatments, such as heat moisture, ultrasound and microwave processing.

In conclusion, rice quality and the development of rice-based products is a multi-faceted dimension defined by several elements, such as physical appearance, cooking and eating qualities, and nutritional value. Therefore, by studying the relationship between the food processing techniques utilized in this experiment and the functionality of the rice cultivars, this study can help increase the practical application and acceptability of rice originating from West African countries.

1.2 The hypothesis of this research

Studies have shown that under the same processing conditions, the same rice cultivars can exhibit different properties, such as the degree of gelatinization, viscosity, texture, consistency, and mouthfeel. The differences in the functional and thermal properties have been postulated to be a result of the chemical and structural makeup of the rice. This present study will seek to further clarify and understand the relationship between the chemical and physical makeup of the rice with its functional and thermal properties while also contributing to the process optimization and effective utilization of flours and starches from rice in food and non-food applications.

1.3. Overall objective

The overall objective of this study is to evaluate the impact of selected physical modification techniques on the quality of the flours of selected rice cultivars.

1.4. Specific objectives

The following specific objectives are proposed in this study:

1. Characterization of the differences in the structural, functional, thermal and rheological properties of the flour from different rice cultivars.

2. Evaluation of the impact of different heat moisture treatments (HMT) on the structural, functional, thermal and rheological properties of the flour from selected rice cultivars.
3. Accessing the use of ultrasound technology on the structural, functional, thermal and rheological properties of the rice flours.
4. Investigating the impact of microwave processing on the structural, functional, thermal and rheological properties of the rice flours.
5. Evaluation of the application of microwaved and ultrasound treated rice flours on moisture and fat contents of flour coated air-fried chicken cuts.

2.0. Literature review

2.1 Flours

Rice is principally composed of polysaccharide carbohydrates consisting of many glucose units joined together by glycosidic bonds. These starch molecules comprise of amylose and amylopectin as their major macromolecules and they vary in appearance, depending on their source. In the unmodified form, biomaterials such as starches and flours play limited roles in the food industry. In general, when heated, native flours form weak-structured, cohesive, rubbery pastes, and undesirable gels when the pastes are cooled (Adzahan, 2002). This is basically the reason why food processors generally prefer flours with better functional attributes than those provided by native starches. The attributes of starches and flours can be improved by various modifications.

Researchers have developed several physical and chemical methods to modify biomaterials such as starch and flour. The reason behind modification of starches and flours are generally to improve thier properties particularly for specific purposes such as to enhance the increase in water holding capacity, heat resistant behavior, reinforce its binding, minimized syneresis of starch and improved thickening (Adzahan, 2002; Miyazaki et al., 2006). However, before selecting modified biomaterials for a specific purpose, consideration, and studies about both the market and production, need to be carried out. Market-related properties of the modified materials include product attributes such as the structure, organoleptic perception and shelf stability (Sajilata and Singhal, 2004). Whereas, the production-related considerations include attributes like viscosity, resistance to shear, low pH and a high temperature of the modified biomaterials.

Today, modified food flours are being utilized in the food industry as food additives and the limits of modification, use, and labeling are clearly being controlled by the various government food regulation bodies (Sajilata and Singhal, 2004). For instance, enzymatic modification of starch produces low molecular weight molecules such as maltodextrin or dextrin (Miyazaki et al., 2006).

Chemical modification of biomaterials such as flours has been the mainstream research on improving functionalities of biomaterials in the last century. However, physical modifications are finding special interest in the food industry precisely because no chemical reagents are used, and as a result, the starch or flour product does not need to be labeled as a modified. These non-destructive modification treatments on rice flour and starches do not result in modification of the

D-glucopyranosyl units of the starch polymer molecules. Physical modification treatments generally produce changes only in the packing arrangements of starch polymer molecules within granules and the overall structures of starch granules. The physical modification involves pre-gelatinization, microwave, ultrasonication and heat moisture treatments (Miyazaki et al., 2006). Pre-gelatinized starches are pre-cooked starches that can be utilized as thickening agents in cold water. Heat-treatment processes, on the other hand, include heat–moisture and annealing treatments, both of which result in physical modification of starch without any gelatinization, a breakdown in granular integrity, or loss of birefringence. These structural changes have been observed to impact the properties of the starch, the attributes of its pastes and gels, and even its digestibility.

2.2 Heat Moisture Treatment

Heat-moisture treatment (HMT) is a hydrothermal process which involves the exposure of biomaterials such as rice flour or starch to higher temperatures, typically above the gelatinization temperature at very defined moisture contents (<35% moisture w/w) for certain time periods (15 min to 24 h) (Collado and Corke, 1999). Heat moisture treatment studies have been conducted on the flours of rice, locust beans, sweet potatoes, beans, peas, lentils and maize (Collado and Corke, 1999; Hoover and Manuel, 1996; Hoover, 2010; Sankhon et al., 2014). Few studies have been carried out on the impact of heat-moisture treatment on non-waxy and waxy rice starches (Anderson et al., 2002; Punchaarnon and Uttapap, 2013). The type of equipment used in the heat-moisture treatment of starches and flours can also impact the degree of changes in the physical and chemical properties (Hoover and Manuel, 1996). For instance, waxy rice starches heat-treated in the microwave or conventional ovens at the melting temperature (T_m) revealed minor but significant increases in digestibility of the treated starches compared to non-treated starches at all incubation times (Hoover and Manuel, 1996). Duration of heat-moisture treated rice starches has also revealed contrasting changes in the physicochemical properties of rice starch (Hoover and Manuel, 1996).

Regardless of the flour or starch origin, heat moisture treatment impacted the gelatinization temperatures, crystallinity, swelling volume, and solubility. All these changes consequently lead to changes in the functionality of the modified biomaterial. However, depending on the cultivar origin and treatment conditions, differences have been reported to occur with HMT (Zavareze and Dias, 2011). Relatively few studies have been done to investigate the effect of different times, moisture content and temperature on the functional and structural properties of rice flours. HMT flour-based products have good potential to be used as unmodified thickeners in processed foods as a result of their temperature and shear stability (Hoover, 2010). The use of HMT products has been evaluated in several food applications such as noodles, doughs and baked goods such as bread and cakes. Currently, studies have subsequently focused on extending HMT processes to flours (Amadou et al., 2014; Chung et al., 2012; Satmalee and Matsuki, 2011; Seo and Kim, 2011; Sun et al., 2014). HMT flours and starches have exhibited good potential for applications as thermoplastic materials, resins (Khamthong and Lumdubwong, 2012) and films (Zavareze et al., 2012).

2.2.1. Impact of HMT on structural arrangements in granules

The temperature and moisture treatment conditions of HMT encourage increased mobility of starch chains and helical structures which result in structural changes to both crystalline and amorphous regions of starch granules (Gunaratne and Hoover, 2002; Hoover and Manuel, 1997). With regards to the crystalline portion, HMT breaks down the least stable structures and alters the arrangement of higher-ordered helical structures, crystallites within granules, or both (Vermeylen et al., 2006). Examples of induced changes caused by HMT on the crystalline regions of rice granules include partial or complete conversion of the B-type crystalline packing organization to the A polymorphic form (Jiranuntakul et al., 2011; Lee et al., 2012; Tattiyakul et al., 2012; Vermeylen et al., 2006), disruption of V-type, single-chain AM structures within rice flour and starch granules (Varatharajan et al., 2011), disruption of stacked lamellae (Vermeylen et al. 2006; Varatharajan et al., 2011), and reorientation of disrupted starch chains and/or helical structures (Kim and Huber, 2013; Sankhon et al., 2014; Varatharajan et al., 2011; Vermeylen et al., 2006). In addition, HMT also induces changes in the amorphous region of rice granules. Major alterations include breakdown of helical structures residing within the granule amorphous regions (Gunaratne and Hoover, 2002); formation of new ordered structures and/or crystallites arising from AM-AM, AM-AP, or AP-AP linkage (Kim and Huber, 2013;Varatharajan et al., 2011) and formation and/or strengthening of AM-lipid complexes (Miyoshi, 2002).

A major impact of HMT is its ability to change the native crystalline packing organization within starch granules. Rice starches that exhibit B helical packing arrangement may be transformed to an A or a combined form polymorphic arrangement via HMT (Gunaratne and Hoover, 2002). Starches with an A-type packing arrangement are not transformed by HMT and therefore retain their native polymorphic arrangement (Hoover, 2010). B→A transformation is encouraged by high temperatures and moisture contents during HMT (Varatharajan et al., 2011; Vermeylen et al., 2006), with the temperature being appropriate to evaporate water molecules (stabilizing the B-type unit cell) and to permit lateral and/or vertical registration of double helices to form the more rigid A-type polymorph (Gunaratne and Hoover, 2002; Vermeylen et al., 2006). The changes in crystallinity in all these studies have been observed as a function of temperature treatments (Lee et al., 2012; Pinto et al., 2012; Varatharajan et al., 2011) and moisture content (Andrade et al., 2014; Lee et al., 2012; Pinto et al., 2012; Zavareze et al., 2010).

2.2.2. Impact of HMT on granule morphology

Several studies have noted a lack of visible differences between native and HMT rice granules. However, treatment temperatures greater than 110°C (Hoover 2010) in addition with moisture contents of 20 to 35% (Anderson et al., 2002; Kim and Huber 2013; Lee et al., 2012) have been reported to induce noteworthy morphological alterations to granules of rice. Observed morphological changes in rice granules include little changes to granule size (Vasanthan et al. 1995), surface cracking (Lee et al., 2012), hollowed granule centers (Kim and Huber, 2013; Lee et al., 2012; Varatharajan et al. 2011), reduced birefringence at the centers of the granules and/or peripheries (Chung et al., 2009; Lee et al., 2012; Varatharajan et al., 2011; Vermeylen et al., 2006), surface indentations or internal granules collapse (Lee et al., 2012; Varatharajan et al., 2011), and partial clustering of granules (Jiranuntakul et al., 2011; Sankhon et al., 2014; Sun et al., 2014). With the aid of high-resolution atomic force microscopy (AFM) imaging, Jiranuntakul et al., (2013) reported increased smoothness in the surfaces of starch granules (corn, rice, and waxy rice) following HMT at 100°C for 16 h at a moisture content of 25%. The observed physical changes were suggested to be as a result of partial melting and restructuring of starch chains at the granule surface which thereby created a physical barrier that impeded water migration into granules.

2.2.3. Impact of HMT on gelatinization properties

Compared to their native starch counterparts, HMT rice starches generally exhibit an upward transition in gelatinization temperature [onset temperature of gelatinization (T_o), the peak temperature of gelatinization (T_p), conclusion temperature of gelatinization (T_c)] and increased gelatinization temperature ranges (Anderson et al., 2002). These alterations have been attributed to increased associations among starch polymer chains (AM-AP and AP-AP) and starch-lipid complexes within granule amorphous regions which have led to decreasing starch chain mobility within amorphous regions and a consequent increase in crystallite melting temperatures (Hoover 2010, Zavareze and Dias 2011). In the same way, at constant temperature conditions (100°C, 16 h), Varatharajan et al. (2011) noted increased gelatinization temperatures for potato and waxy potato starches subjected to HMT at 80–100°C (27% moisture) but reported no decrease in starch transition temperatures (T_o, T_p, and/or T_c) even at higher treatment temperatures ranges of 120–130°C. The work of these authors proposes that high treatment temperatures contribute to higher starch chain mobility, consequently resulting in the breakdown of hydrogen bonds between crystalized starch chains [these alterations simultaneously occurred with decreases in gelatinization enthalpy (ΔH)]. Several studies have reported a decrease in ΔH of gelatinization (Anderson et al., 2002; Shin et al., 2005; Hoover, 2010; Vasanthan et al., 1995; Vermeylen et al., 2006), with the degree of decrease often reportedly increased by an increasing treatment moisture content (Lee et al., 2012; Kim and Huber, 2013; Sun et al., 2014) or temperature (Varatharajan et al., 2011). On the other hand, some authors have reported that ΔH either remains unaltered (Hoover and Manuel, 1996) or increases (Sankhon et al., 2012; Tattiyakul et al., 2012) as a result of HMT. Low treatment moisture contents (≤ 20%), have been observed to reduce ΔH with subsequent ΔH increase when the moisture content was increased to moisture content ranges of 25 and 30% (Anderson et al., 2002). The increase in the ΔH can be attributed to the increase in starch chain mobility (a result of the high higher treatment temperature and moisture content) which consequently leads to the formation of highly arranged molecular structures.

2.2.4. Impact of HMT on the swelling power and solubility

In comparison to their respective native properties, several studies reveal a decrease in the swelling power (SP) of starches following HMT (Kim and Huber 2013; Pinto et al., 2012; Tattiyakul et al., 2012; Sankhon et al., 2014; Sun et al., 2014). Reduction in the swelling power of rice starches is generally associated with an increase in the temperature or moisture content (Senanayake et al., 2013). Hoover (2010), proposed that swelling properties of starch are influenced by factors such as the extent of disruption of starch crystallites, increased starch crystallinity, the formation of AM-lipid complexes, structural relinking between AM and AP chains, and polymorphism (B→A) changes. On the other hand, the influence of HMT on starch solubility is varied, with some studies reporting an increase (Sankhon et al., 2014) and others a decrease (Pinto et al., 2012; Sun et al., 2014b).

2.2.5. Impact of HMT on pasting properties

When compared to their native flours and starches, HMT rice flours and starches have been reported to exhibit an increase in the pasting temperature or time and a decrease in peak, trough and breakdown viscosities (Anderson et al., 2002; Puncha-arnon and Uttapap, 2013). Literature report varies on the impact of HMT on setback and final viscosities (Anderson et al., 2002; Puncha-arnon and Uttapap, 2013). Reports regarding the retrogradation stability of starch pastes or gels vary for HMT starches (Anderson et al., 2002; Puncha-arnon and Uttapap, 2013) with retrogradation stability being dependent on the botanical source and HMT conditions. Despite HMT starches having improved heat and shear stabilities when compared to their respective native starches, the improved stability of these modified starches comes at the cost of reduced swelling, which could involve higher quantities of starch to achieve needed viscosity development for desired food applications (Watcharatewinkul et al., 2010).

2.3. Ultrasound treatment.

Ultrasound modification is a non-destructive method of processing that utilizes sound waves exceeding 16–18 kHz (the threshold of human hearing). During ultrasound treatment, waves of ultrasound travel through the dispersion (usually liquid) at a rate selected by the nature of the wave and the medium through which it is passing. Ultrasound treatment of biomaterials such as flours and starches are generally carried out in aqueous systems. Water is an ideal medium for producing cavitation bubbles due to its low vapor pressure and viscosity. The passage of ultrasound waves through the dispersion causes micrometer-size cavities (bubbles) that consequently propagate until they reach a size at which the bubbles breakdown. The collapse of the cavities subsequently creates shock waves, small regions of high-pressure gradients and local heating; a resultant effect of intense thermal energy accompanied by high cooling rates (Soria and Villamiel, 2010; Suslick and Flannigan, 2009). Therefore, the nature of the ultrasonication medium such as its vapor pressure, surface tension, the nature and concentrations of dissolved gasses and the type of ultrasound input are important parameters that can influence ultrasound treatments (Soria and Villamiel, 2010). High-intensity ultrasound and low-frequency ultrasound (16–100 kHz) often referred to as power ultrasound can be utilized in food processing (Chandrapala et al., 2012; Chemat et al., 2011; Soria and Villamiel, 2010). The interest in determining what effects such intensities of ultrasounds can have on food ingredients have led a few researchers to examine the effects of ultrasonic treatments on both granular and pasted biomaterials. Varying frequencies, powers (W/cm^2 values), amplitudes, temperatures, atmospheres, treatment times, and starch concentrations can have effects on the ultrasound treatment. One of these studies reported a temperature increase in the system during ultrasound treatment (Yu et al., 2013). These temperature increase led to partial gelatinization of the modified starches. There are few ultrasound studies on rice granules (Sujka and Jamroz, 2013) as compared to starches from other plant materials such as wheat (Lionettom et al., 2006). A thorough study of ultrasound treatment on rice granules could provide a clear picture of the impact on its starches and flours.

2.3.1. Impact of ultrasound on granule morphology

Depending on the treatment conditions and nature of the material, when starch or flour granules in suspension (either in water or water-alcohol solutions) are treated with ultrasound, several changes occur on the morphology of starches (Huang et al., 2007; Luo et al., 2008; Sujka and Jamroz, 2013). Ultrasound treatment has been characterized by the formation of new structures on rice granule surface variously referred to as pits, pores, notches, grooves, fissures, cracks, depressions, and dents (Sujka and Jamroz, 2013). Although, Wang and Wang, (2004) and Zuo et al. (2009) reported that no surface or other granule damage occurred with the starches following ultrasonication. Herceg et al. (2010) suggested that cavitational forces during ultrasound treatment disrupt granules, consequently increasing the water permeability of the granules.

2.3.2. Impact of ultrasound on structural arrangements in granules

Depending on the experimental conditions and type of biomaterial, ultrasound treatments impacts the crystallinity of biomaterials to several extents (Luo et al., 2008; Zheng et al., 2013; Zhu et al., 2012). Although the distortion of the crystal structure has been observed following ultrasound treatment, Huang et al. (2007) observed no significant change in the A-type crystal pattern of maize starch granules, and Luo et al. (2008) found no significant change in the X-ray diffraction patterns of maize starches as a result of ultrasound treatment. While the starch polymorph is generally not affected by ultrasound treatment, the crystallinity percentage of the ultrasound treated granules either increases or decreases, depending on the conditions of ultrasound treatment (Huang et al., 2007; Zheng et al., 2013). For instance, ultrasound treatment (starch concentration 30% and 500 W) up to 3 min increased the crystallinity degree of starch, and further treatment up to 15 min decreased it (Huang et al., 2007). The initial increase in crystallinity could be related to the breakdown of amorphous regions, and further ultrasound treatment may encourage the erosion of the crystals in the granules (Huang et al., 2007). It can then be inferred that different packing of the crystalline and amorphous regions in the starch granules gives rise to their different susceptibility to the ultrasound treatment.

Herceg et al. (2010) proposed that granule damage occurred in the amorphous regions, while Zhu et al. (2012) proposed that amorphous regions containing amylose molecules are more susceptible to ultrasound treatment than were those containing amylopectin. Sujka and Jamroz (2013) proposed that increases in swelling power and solubility following ultrasound treatment of corn, rice, wheat, and potato starch granules were as a result of the damage to crystalline regions of the treated starches.

2.3.3. Impact of ultrasound on swelling power and solubility

Following ultrasound treatment of starches, increases in swelling power (SP) and solubility properties of rice starches have been reported (Sujka and Jamroz, 2013). The increase in SP following ultrasound treatment has been related to the morphological changes that increase the permeability of granules (Huang et al., 2007; Sujka and Jamroz, 2013), breakdown of intermolecular hydrogen bonds (Luo et al., 2008; Zuo et al., 2009), and breakdown of crystallites (Jambrak et al., 2010; Sujka and Jamroz, 2013). The degree of the changes in the swelling and solubilization properties of granules following ultrasound treatment seems not only dependent on the conditions of ultrasound treatment but at the same time on the nature and composition of the starch or flour sample. For instance, ultrasonication with dual-frequency (25 kHz and 80 kHz) has been observed to increase the solubility of sweet potato starch more than that of a single-frequency (25 kHz or 80 kHz) (Zheng et al., 2013). Swelling power of starches from maize and rice has also been reported to increase with increasing ultrasound power and intensity (Jambrak et al. 2010; Sujka and Jamroz, 2013). The degree of increase in swelling and solubility after ultrasound treatment followed the order of high amylose (50% amylose content) > normal > waxy starches

(Luo et al., 2008), proposing that the amylose portion in the granules is more easily disrupted by ultrasound treatment.

2.3.4. Impact of ultrasound on gelatinization properties

The gelatinization properties of ultrasound treated starches can be determined by differential scanning calorimetry (DSC), and enthalpy change/gelatinization enthalpy (ΔH) and the gelatinization temperatures including onset (T_o), peak (T_p), and conclusion (T_c) temperatures defined. Peak temperature is suggested to be an index of crystallite quality related to double helix length, while the enthalpy change reflects the loss of molecular order in the granule (Sang et al., 2008). Several studies have varying reports on the effects of ultrasound treatment on gelatinization properties on the same type of material. For instance, ultrasound treatment has been reported to increase the gelatinization temperatures of maize starch (Huang et al., 2007) and have little impact on the gelatinization properties of the same type of maize starch (Jambrak et al., 2010).

2.3.5. Impact of ultrasound on rheology

The flow behavior of flour dispersions as affected by ultrasound can be mathematically modeled by the power law equation. The application of ultrasound on maize starch particularly was reported to reduce the consistency coefficient in an ultrasound power dependent manner and the flow index was reported to be higher than 1 (Jambrak et al., 2010). The observations of this study suggest that starch chains became shorter with increasing ultrasound intensity (Czechowska-Biskup et al., 2005; Jambrak et al., 2010).

Rapid ViscoAnalyser (RVA) studies have also reported different impacts of ultrasound treatments on the pasting properties of several starches (Chan et al., 2010; Zuo et al., 2009). Several studies employing varying types of starches from maize, sweet potato, rice have shown that ultrasound reduced the viscosity during pasting (Herceg et al., 2010; Luo et al., 2008; Zheng et al., 2013; Zuo et al., 2009). Reduction in the pasting viscosity of starches following ultrasound treatment can be attributed to the broken-down granules with less swelling capacity, the damages on granules encouraging the intake of water for hydration, and the disrupted starch chains. Depending on the parameters of ultrasound treatment, the disruption of starch chains on the molecular level may not occur, but still, a reduced viscosity can be achieved through the physical breakdown of the starch granules (Zuo et al., 2009). In contrast, a few variations can be noted on the impact of ultrasound treatment on the peak viscosity of starches. For instance, Chan et al., 2010 noted that ultrasound treatment increased (e.g., potato) and had little impact (e.g., mung bean) on the peak viscosity of starch paste. The variations in these results may be associated with the difference in the ultrasound treatment conditions, which results in the varying altered structure of the amorphous and crystalline parts of the granules, which have different susceptibility to ultrasound treatment. The impact of ultrasound treatment seems dependent on the type of starch as well as the ultrasound conditions. For instance, under the same experimental conditions, pasting behaviors of starches with high amylose contents have been noted to be much more affected by ultrasound than that of waxy starches, therefore concluding that amylose in the granules is more easily broken down (Luo et al., 2008). Other types of rheological properties such as dynamic oscillation and small deformation rheology of starch as affected by ultrasound treatment remain to be studied.

2.3.6. Impact of ultrasound on retrogradation

Starch retrogradation occurs when gelatinized starch paste in the flour begins to cool. Retrogradation characteristics include an increase in crystallinity, gel firmness, turbidity and the formation of a B-type polymorph (Hoover, 2010). Short-term retrogradation is attributed to the reassociation of amylose molecules, while long-term retrogradation is associated with the recrystallization of amylopectin (Hoover, 2010). Ultrasound treatment, i.e. before the gelatinization, was reported to decrease the enthalpy of gelatinization and increase the onset melting temperature of retrograded rice starch; increased power input had more impact (for instance, 1000 W vs 100 W) (Yu et al., 2013). Luo et al. (2008) also reported that ultrasound treatment reduces the syneresis of the gel of waxy maize starch while also increasing that of high amylose maize starch (amylose content 50%) during the freeze-thaw cycle. A concluding fact from these studies is that the type and composition of biomaterial influence the outcome of ultrasound treatment even though the observed results from these varying studies is hard to compare due to the variation in the ultrasound setting, experimental method, and type of starch.

2.4. Microwave processing

Microwaves are oscillating electromagnetic waves in the frequency range of 300–300,000 MHz. These pastes are formed as a result of polar molecules absorbing microwave energy and orienting themselves with respect to the electric field. The instantaneous change in their orientation produces heat by friction resulting from the molecules rotating, rubbing against, and bumping into each other as they try to orient themselves with the field creates thermal energy. (Sumnu, 2001), resulting in bulk heating throughout the sample and a faster heating rate compared with the conventional heating.

Microwave treatment is generally influenced by factors such as wattage and frequency of the microwave source, the moisture content of the sample, and duration of the treatment. With respect to moisture content, Hagiwara et al. (1986) observed a loss of crystallinity and an increased susceptibility to amylases when potato starch with moisture contents of 5 to 25% was subjected to microwave (2,450 MHz). Generally, the results of microwave heating on biomaterials such as flours and starches reveal an increase in peak, trough/hot-paste, and final viscosities for nonwaxy starches and decreases in the same for waxy starches, increase and reduction in swelling power and solubility, increase in onset of gelatinization and peak of gelatinization parameters, decrease in the enthalpy of gelatinization, changes in X-ray diffraction patterns from the B type to the A+B type and few or no change in the degree of crystallinity.

The results of a few microwave studies seem to indicate that waxy starches are more influenced by the microwave treatment than nonwaxy starches. Szepes et al. (2005) reported that when corn and potato starches were microwaved (450W for 15 min) without added moisture, the starches' moisture contents dropped to near zero, the crystallinity content of the corn starch reduced from 85 to 30% and the starch's polymorph type changed from a B type to an A type. Szepes et al. (2007) microwaved the same samples at a higher wattage (900W) for the same time and observed decreased crystallinity of the potato starch and that the swelling power increased irreversibly. Microwave impact on corn starch was observed to be insignificant. On the other hand, Lee et al. (2007) study on the impact of microwaves on maize, waxy maize, and amylomaize starches whose moisture contents had been adjusted to 10–35%, revealed that maize and amylomaize starches were more susceptible to microwave heating than waxy maize starches. Loss of crystallinity increased pasting temperatures, reduced peak viscosities, and pastes with reduced breakdown properties were observed following microwave treatment.

CONNECTING TEXT

Rice flour quality is very important in defining rice flour attributes such as texture, heat and mass transfer, nutritional composition and thermal properties. It was noted that information on these characteristics of non-waxy rice flour is scarce. In chapter 3, the physical, chemical, thermal and rheological characteristics of flours from some selected non-waxy rice cultivars were evaluated.

3.0.Physicochemical, thermal and rheological properties of flour from different non-waxy rice cultivars.

3.1. Abstract

The physiochemical, thermal and rheological properties of flours from seven cultivars were evaluated. The amylose and protein contents of flours from different cultivars differed significantly. The results showed that the physicochemical properties of the rice flours were related to their chemical composition. The flours of all the rice cultivars displayed A-type crystallinity patterns. The water and oil holding capacity of the rice flours were correlated to the amylose and protein. Swelling power and water absorption index were dependent on temperature. Thermal properties, the onset temperature (T_o), peak temperature (T_p), conclusion temperature (T_c), enthalpy of gelatinization (ΔH_G) and glass transition temperature (T_g) were related to the structural properties of the rice. The rheological properties of all the rice flour dispersions (4, 6 and 8% concentration) exhibited non-Newtonian shear thinning behavior. Dynamic responses of the studied samples showed that storage (G') and loss (G") moduli both increased with increasing frequency (ω). The magnitudes of both G' and G" also increased with increase in dispersion concentration. Dynamic rheological responses exhibited a gel-like viscoelastic property with G' higher than G".

Keywords. Rheology. Rice Starch. Dynamic Properties. Rice Flour.

3.2. Introduction

Rice is a major staple food consumed by more than half of the world's population (Kong et al. 2015). It can be utilized as whole grains or processed into rice related materials such as rice starch or flour. The utilization of flours derived from rice has attracted considerable attention in the production of so many commercially processed products such as breakfast cereals, candies, non-dairy ice creams, noodles, and soups. Processing of these rice-related products is related to the physical, chemical and rheological properties of the rice flour. Therefore, documentation on these properties is essential in the production of rice-related products with favorable qualities.

Physicochemical and gelatinization properties of rice are dependent on different factors such as the cultivar type, genetic background, climatic and soil conditions of the grain during its agronomic practices (Wani et al., 2013). Functional properties such as solubility, swelling, water, and oil absorption capacity provide vital insights into the several potential applications of rice flour for industrial and domestic uses (Hoover, 2010). In addition, gelatinization properties such as the onset (T_0), peak (T_p) and conclusion temperatures (T_c) provide information on the thermal requirements of the rice flour. The physicochemical, gelatinization and rheological properties of a certain flour are influenced by the ratio of amylose and amylopectin in the matrix of the flour. During thermal processing of rice flours, the starches in the rice flour undergo gelatinization i.e. starch granules swell and form gels. Amylose has been suggested to restrict the swelling of starch granules (Varavinit et al., 2003). This means that waxy cultivars swell to a greater extent than non-waxy rice cultivars due to their low amylose content. Varavinit et al. (2003) reported a positive correlation between amylose and the onset, peak and conclusion temperature of gelatinization of rice flours from different rice cultivars from Thailand. Nakamura and Ohtsubo, (2010) reported a negative correlation between the amylose of rice flour and oil uptake. Lii et al., (1996) observed a positive correlation between the storage modulus (G') and the amylose content of rice. The correlation was related to resistance during thermal degradation caused by amylose on the granule structure. Such correlations between amylose and the physicochemical, gelatinization and rheological properties could serve as an index of predicting the amylose content of rice flour.

Flour is an essential material utilized in the food industry due to its gelling and thickening capabilities. The dispersions formed from rice are typically biphasic systems made up of a continuous phase i.e. the aqueous solution of the leached-out amylose as a result of granule swelling, and a distributed phase composed of the swollen granules. With regards to understanding the rheological behavior of these rice flour or starch dispersions, studies have focused on the rheological variation in different rice starch blends (Samutsri and Suphantharika, 2012), effect of concentration and temperature on rice flour rheology (Yoo, 2006) and general rheological characterization of modified rice starches (Lin et al., 2011) or un-modified starches from newly released rice cultivars (Falade and Christopher, 2015; Wani et al., 2013). Conclusively, these studies all agree that rheological properties of rice flour or starch dispersions are a function of processing temperature, the concentration of the rice flour or starch in the dispersion and type of rice cultivar. The studies showed that information on the rheological behavior of rice flours and starches are essential for controlling process parameters, modeling flow systems, predicting desirable textures of foods and expanding the utilization of rice (Chun and Yoo, 2004).

Food product dispersions typically undergo temperature variations during processing, distribution, and storage. Several rheological studies have been carried out on the impact of temperature and concentration on rice starches or flours (Ahmed et al., 2008; Dorglamud et al., 2013; Lin et al., 2011; Zhong et al., 2009).

In general, most rheological studies have been carried out on rice or rice based products of waxy origin (Dorglamud et al., 2013; Lin et al., 2011; Yoo, 2006). The abundance and low cost of non-glutinous rice cultivars in Sub-Saharan countries like Nigeria, Ghana, Togo can proffer an alternative option to the application of glutinous rice cultivars. There has been no attempt made to study the rheological properties of flours from non-waxy/glutinous rice cultivars and the influence of concentration, processing time and temperature. It is reasonable to think that the development of rice-related base food products can be encouraged by a comprehensive study of the structural impact of rice type on the rheological properties of rice flour dispersions. Thus, such information could prove vital in developing and improving the rheological properties, distribution and storage stability of flour-based systems.

In this study, the physicochemical, gelatinization and rheological properties of non-waxy rice cultivars of FARO 60, FARO 55, FARO 57, FARO 44, SAHEL 328, SAH 177 and SAH329 were studied. This study provides a vital reference for non-glutinous rice applications (or application development).

3.3. Materials and methods

3.3.1. Raw materials

Seven rice cultivars, four from Nigeria and three from Senegal were used in this study. FARO 60, FARO 55, FARO 57, FARO 44 was supplied and confirmed by the Breeding Unit of Rice Research Program, National Cereal Research Institute (NCRI), Badeggi, Nigeria while SAHEL 328, SAH 177 and SAH329 were purchased from a local market in Dakar, Senegal.

3.3.2. Sample preparation

Prior to all the test, the raw rice grains were milled using a coffee grinder (SUMEET Multi Grind, India) and the flour was later passed through a 250µm sieve before further use.

3.3.3. Protein analysis

Crude protein content was determined by combustion using a LECO Nitrogen Analyzer (CNS 2000, St. Joseph, MI, USA). 100mg of flour sample was weighed into a porcelain sample holder (boat) for introduction into the combustion chamber (1300°C) of an automated sample loader. Nitrogen was produced because of the combustion process. The amount of protein present in the samples was estimated by multiplying the nitrogen content by a factor of 6.25.

3.3.4. Amylose determination

3.3.4.1. Isolation of starch

Isolation of starch from rice flour was carried out using a slightly modified version of alkaline deproteination (Lii et al., 1996). 100g of sieved rice flour were mixed with 300 ml of 0.5M NaOH. The mixture was stirred for 4h and left to stand for 24h at 22°C. The supernatant was decanted, and the solid residue washed several times with 500ml of distilled water until the pH of the filtrate was between 6.0 and 6.5. The isolated starch was oven-dried at 40°C for 48h and then ground with a pestle and mortar to pass through a 250 micron (number 60) mesh screen.

3.3.4.2.Amylose contents analysis

Amylose content analysis was carried out using the method of Hoover and Ratnayake (2001). 20 mg of the isolated starch sample was vortexed with 8 ml of 90% Dimethylsulfoxide (DMSO) then incubated in a shaking water bath at 85 ºC for 15 min. The mixture was cooled at room temperature (22°C) for 45 min and diluted with distilled water to 25 ml. Aliquot of 1ml from the diluted solution was added to 40 ml of distilled water then mixed vigorously with 5 ml of iodine solution (0.0025 M Iodide/0.0065 M KI mixture). The volume was adjusted to 50 ml with distilled water, mixed vigorously and allowed to develop color for 15 min. Absorbance was measured at 600 nm against a reagent blank. The percentage of amylose was estimated from an equation obtained from the standard curve prepared with amylose/rice starch as the standard material.

3.3.5. Degree of crystallization

The crystalline structure of the flours (moisture content 8.0-8.2%) was analyzed by using a Bruker D8 Discovery X-ray diffractometer (D8, Bruker Inc, WI, USA) at 40 kV and 20mA Cu-Kα radiation. Diffractograms were obtained from 10° to 90° (2θ) at a scan rate of 2°/min. Multi-peak fitting was used to estimate the integrated area of crystalline peaks (Ac) and amorphous peak (Aa). The crystallinity index [X_c (%)] was calculated as follows:

$$X_c(\%) = 100 \times \left(\frac{A_c}{A_c + A_a} \right) \tag{1}$$

3.3.6. Flour holding properties

The water holding capacity (WHC) was estimated by dispersing 1.000 ± 0.005 g of flour with distilled water (10 ml) and keeping the resulting mixture at ambient temperature for 24 hours. The supernatant was decanted. WHC was expressed as grams of water retained per gram of solid.

Oil holding capacity (OHC) was determined by slightly modifying the method of De la Hera et al. (2013). Briefly, rice flour (1.0 ± 0.2 mg) was mixed with 1.0 ml of vegetable oil. The resulting mixture was stirred for 1 min with a wire rod to disperse the sample in the oil. After a period of 30 min in the vortex mixer, the mixture was centrifuged at 3000 × g and 4°C for 10 min. The supernatant was removed, and the holding vessels inverted for 25 min to drain the oil. After this, the resulting residue was referred to as W_r. The oil absorption capacity was expressed as grams of oil bound per gram of the sample on a dry basis. For the determination of all the hydration properties, three replicates were made for each sample.

$$OHC\left(\frac{g}{g}\right) = \frac{W_r}{W_i} \quad (2)$$

where W_r is the residue weight and W_i is the sample weight (g).

3.3.7. Gel hydration properties

Swelling power and Water absorption index (WAI) of different rice flour fractions were estimated following slight modification of the method of De la Hera et al. (2013). Briefly, the flours 2.00 ± 0.1 mg) was mixed in 1.0 ml of distilled water in an Eppendorf tube and cooked at 90°C for 10 minutes in a water bath. The cooked paste was cooled in an ice water bath for 10 minutes and then centrifuged at 3000 × g at 4°C for 10 min. The supernatant was decanted into an evaporating dish and the dry solids were recovered by evaporating the supernatant with a Thermo savant freeze dryer (model ModulyoD) till constant weight was achieved. Three replicates were carried out for each sample. Residues (W_r) and dried supernatants (W_s) were weighed and Water Absorption Index (WAI) and Swelling Power (SP) were estimated as follows:

$$WAI\left(\frac{g}{g}\right) = \frac{W_r}{W_i} \quad (3)$$

$$SP\left(\frac{g}{g}\right) = \frac{W_r}{W_i - W_s} \quad (4)$$

3.3.8. Differential scanning calorimetry (DSC)

The gelatinization properties of the flour were analyzed at least in triplicate with a Differential Scanning Calorimeter (DSC Q100, TA Instruments, Wilmington, DE USA). Flour samples (5.0mg) was placed in individual pans. Considering the moisture content of each sample, an appropriate volume of distilled water was added to the pan to achieve a water/flour ratio of 2.5: 1. The pans were hermetically sealed and equilibrated at room temperature for at least 2 hrs before the scan. The sealed samples were heated from 20 to 100°C at a heating rate of 10°C/min. A sealed empty pan was used as a reference. From the DSC thermograms, thermal parameters such as the onset temperature (T_o), peak temperature (T_p), conclusion temperature (T_c), enthalpy of gelatinization (ΔH_G) and glass transition temperature (T_g) were calculated.

3.3.9. Dynamic shear properties

3.3.9.1. Preparation of dispersion

Rice flour dispersions (4, 6 and 8% w/w) were prepared by mixing rice flour with distilled water. The mixtures were stirred for 30 min at ambient temperature and then heated at 95°C in a water bath for 30 min with mild agitation carried out by a magnetic stirrer. After heating, the freshly prepared pastes were held at room temperature (22°C) for 10mins. The resulting paste was later loaded onto to the rheometer to estimate the rheological properties.

3.3.9.2. Dynamic rheological measurement

Dynamic shear measurements, storage modulus (G'), loss modulus (G"), complex viscosity (η*) and tan δ = (G")/ (G') of the different dispersions of the rice flours were carried out with a rheometer (AR 1000, TA Instruments, New Castle, DE) using a parallel plate system (4 cm diameter) at gap of 500 µm at 25°C. Dynamic shear data obtained were from frequency sweeps over the range of 0.63- 62.8 rad/s at 2% strain. The rheological measurements were performed in triplicate.

To describe the rheological data of the rice flour, the data were fitted to the power law (Equation 5) model.

$$\sigma = K\gamma^{n} \tag{5}$$

where σ is the shear stress (Pa), γ is the shear rate (s^{-1}), K is the consistency index ($Pa \cdot s^{n}$) and *n* is the flow behavior (dimensionless unit).

The storage (G') and loss (G") modulus were modeled as a power function of oscillatory frequency (ω). Equations 6 and 7 is commonly used utilized in describing the viscoelastic behavior of food and dispersions (Rao 2007).

$$G' = k' \cdot \omega^{n'} \tag{6}$$

$$G'' = k'' \cdot \omega^{n''} \tag{7}$$

Where k', k" are consistency coefficient in power-law model of viscoelastic properties [Pa $s^{n'}$, Pa $s^{n''}$] and n' and n" represent the behavior index in power-law model of viscoelastic properties.

3.3.10. Statistical analysis

All the measurements were conducted at least in duplicate. Analysis of variance and least significant difference tests were carried out with JMP software (Version 14; SAS, NC, USA). Turkey's test was applied to detect variations in the means, and $P < 0.05$ was considered to be statistically significant.

3.5.Results and discussion

3.5.1. Chemical composition

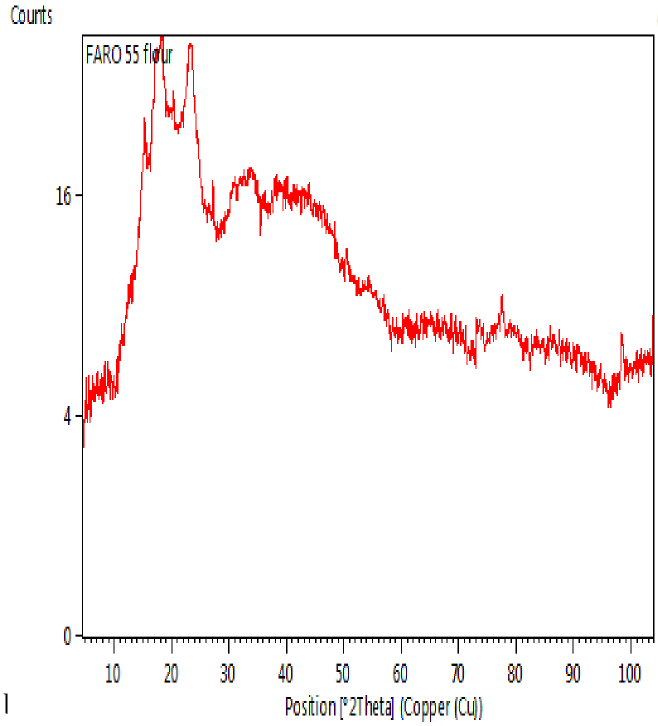
The protein content of the rice flours is listed in Table 3.1. Protein is an important criterion used to evaluate the processing and nutritious quality of rice flour. It was observed that the protein content varied from 8.47% for FARO 44 to 9.24% for SAH 329. The protein contents of these cultivars are comparably higher to protein ranges reported for *Indica* rice flours (6.93- 8.33%), aromatic and non-aromatic Indian rice (6.87 - 8.33%) and waxy- Korean rice (6.70 -8.10%) (Kang et al., 2011; Verma and Srivastav 2017; Yu et al., 2012). The variation of protein contents in the different rice cultivars studied might be related to factors such as water supply, soil nitrogen availability and growing conditions.

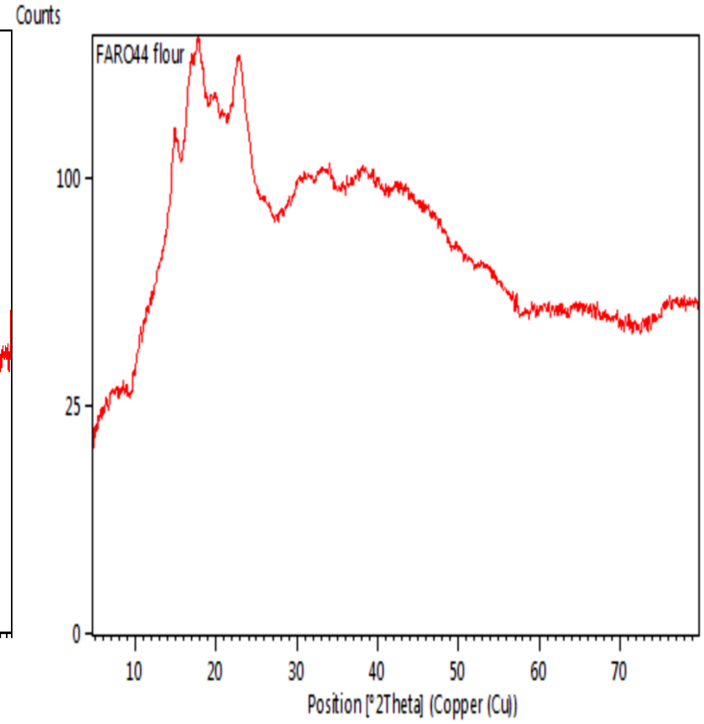
Amylose content of FARO 55, 44, 57, 60 and SAH 177, 328 and 329 were 25.90, 28.30, 25.01, 27.75, 29.95, 31.48 and 30.85%. All cultivars showed high amylose contents (>25%). Rice cultivars have been classified based on their amylose content into waxy (0-2%), very low (3- 9%), intermediate (20-25%) and high (>25%) (Cruz and Khush, 2000). Thus, all the cultivars used in this study are classified as high non-waxy/glutinous rice types. The variations in amylose content of the cultivars are attributed to factors such as genotype, cultural practices, soil type during cultivation, storage and climatic conditions (Falade and Christopher, 2015). The amylose fraction of starch molecules has linear arrangements of α-1,4-linked glucose units. This fraction impacts the digestion, thermal and rheological responses of polymers (Falade et al., 2014). For instance, starches with low amylose content have been reported to digest more easily compared to starches with high amylose content (Riley et al., 2004).

3.5.2. Crystallinity properties of rice flours

The X-ray diffractograms of the different studied rice flours are presented in Fig. 3.1. The X-ray diffraction parameters and crystallinity degrees were estimated from the ratio of the diffraction peak area and total diffraction area as seen in Table 3.2. As shown in Fig.3.1, rice flour samples displayed clear A-type diffraction patterns with major reflections at 2θ around 15° and 28.3°, and unclear doublet reflections at 17° and 18°. These patterns are typical for A-type diffraction patterns seen in other X-ray diffraction studies of rice (Singh et al., 2007; Yu et al., 2010). In addition, the absence of peaks at 5° is a clear indication that these flours aren't B-type diffraction patterns, while the intensity of the peaks at 15° is related to the amylose content; non-waxy rice cultivars show higher reflection at 15° compared to waxy-rice cultivars (Yu et al., 2010). Relative crystallinity degree (%) of rice flours were significantly different. The relative crystallinity (%) is an index of the amylopectin degree (Yu et al., 2012). The order of relative crystallinity degree of the studied rice flours was found to be FARO 60 > SAH 177 > SAH 328 > FARO 44 >FARO 57> FARO 55 > SAH 329. These crystallinity data may be influenced by protein and/or lipid interaction with the amylopectin fraction of the rice granules (Yu et al., 2012).

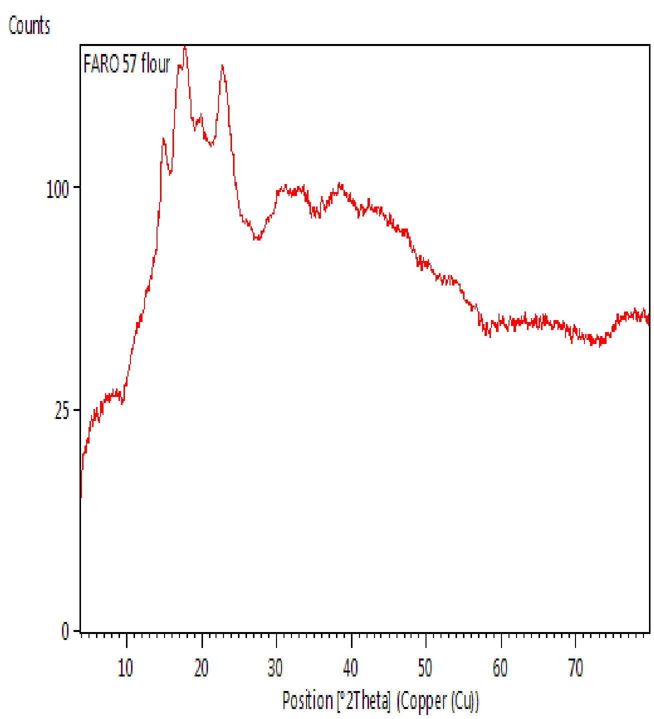
Non-waxy flours are less crystalline than regular type waxy rice flour (Vandeputte et al., 2003). Amylopectin is the major component responsible for crystallinity in flour and therefore the ratio of amylose to amylopectin in the flours is responsible for the difference in the relative crystallinity of the rice cultivar (Kasemsuwan et al., 1995). X-ray diffraction pattern and the relative crystallinity degree is also reported to depend on the type of cultivar, the origin of rice starch and environmental conditions (Huang et al., 2008).

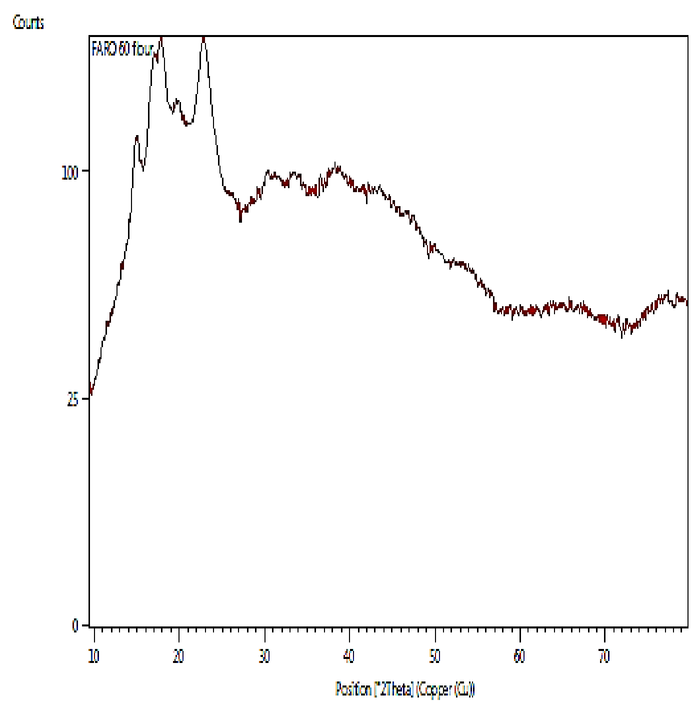

Counts
FARO 55 flour
16
4
0
10 20 30 40 50 60 70 80 90 100
Position [°2Theta] (Copper (Cu))


Counts
FARO44 flour
100
25
0
10 20 30 40 50 60 70
Position [°2Theta] (Copper (Cu))

(a)

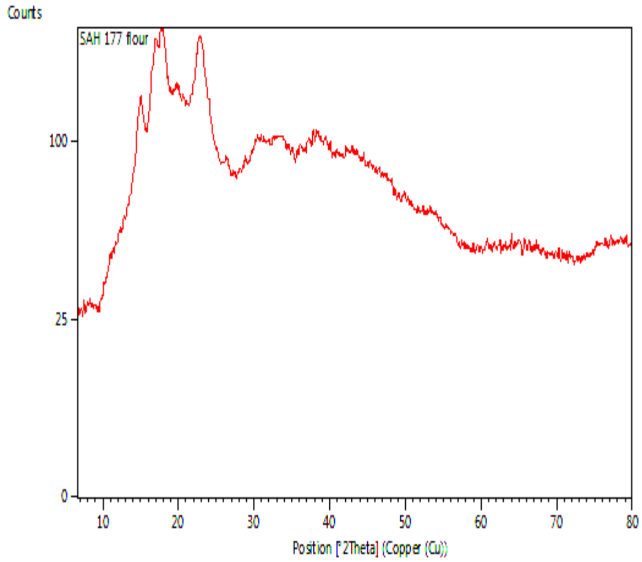
(b)


Counts
FARO 57 flour
100
25
0
10 20 30 40 50 60 70
Position [°2Theta] (Copper (Cu))

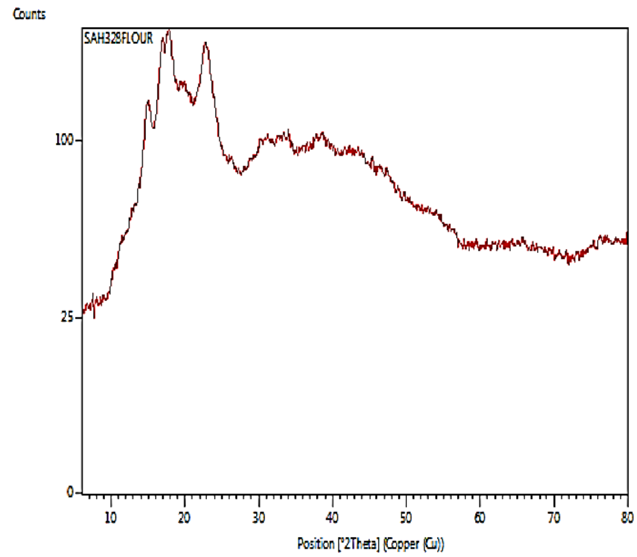

Counts
FARO 60 flour
100
25
0
10 20 30 40 50 60 70
Position [°2Theta] (Copper (Cu))

(c)

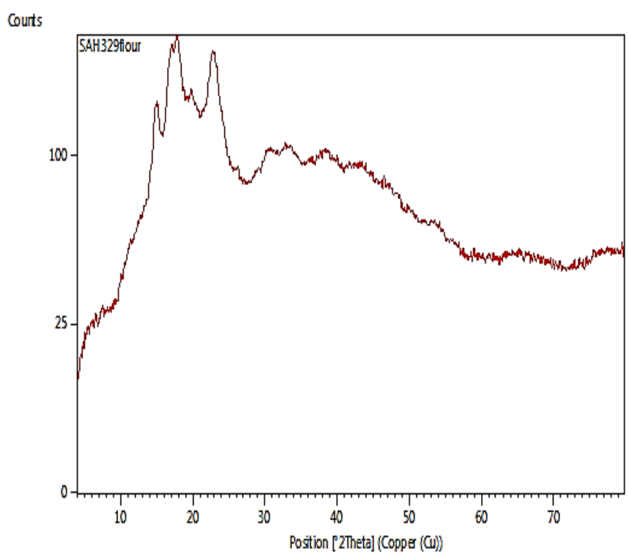
(d)



(e)



(f)



(g)

Fig 3.1. X-ray diffractograms of (a) FARO 55, (b) FARO 44, (c) FARO 57, (d) FARO 60, (e) SAH 177, (f) SAH 328 and (g) SAH 329 rice flours.

Table 3.1. Protein content, crystallinity, oil and water holding capacities of rice flours.

Rice cultivars	Protein content (%)	Relative Crystallinity (%)	Oil holding capacity(g/g)	Water holding capacity (g/g)
FARO 55	8.72 ± 0.15cd	16.27 ± 0.12*cd*	1.00 ± 0.10*a*	1.43 ± 0.04*a*
FARO 44	8.47 ± 0.25d	17.88 ± 1.01*bc*	1.00 ± 0.15*a*	1.49 ± 0.04*a*
FARO 57	9.14 ± 0.11*ab*	17.60 ± 1.13*bc*	0.93 ± 0.25*a*	1.44 ± 0.01*a*
FARO 60	9.00 ± 0.16*bc*	23.40 ± 0.00*a*	1.07 ± 0.20*a*	1.33 ± 0.05*b*
SAH 177	8.67 ± 0.12*bcd*	21.97 ± 0.29*a*	1.10 ± 0.17*a*	1.23 ± 0.02*c*
SAH 328	9.44 ± 0.09*a*	18.77 ± 0.12*b*	1.17 ± 0.29*a*	1.13 ± 0.0*c*
SAH 329	9.24 ± 0.06*abc*	15.63 ± 0.55*d*	1.27 ± 0.15*a*	1.22 ± 0.01*d*

Means within each column with same letters are not significantly different ($P<0.05$). Data are means ± SD.

3.5.3. Water Holding Capacity (WHC) and Oil Holding Capacity (OHC) of rice flours

The water and oil holding capacities of the flours are presented in Table 3.1. Water holding capacity of rice flours ranged from 1.13 - 1.49 g/g. FARO 44 reported the highest WHC (1.49 g/g). Increase in WHC increases amylose leaching, solubility and a loss in the crystalline framework of starch (Ahmed et al., 2015). The results reported in this study are of interest as the water holding capacity of rice flours noted in this current study agrees with the WHC range (1.35-1.97 g/g) for basmati rice flours reported by Ahmed et al. (2015). The observations with WHC is likely due to the presence of major components like amylose, protein, and fiber, which are responsible for increased water holding capacity (Kinsella, 1979). A negative correlation (-0.6357) was observed in this study between the protein content of the studied rice flours and their WHC. Rice cultivars are composed of different amounts of proteins. Since protein in rice may have different hydrophobicity, the amount of water held by these constituents may differ, and therefore impact the WHC of the rice. A strong negative correlation (0.965) was also observed between the WHC of the rice flours and amylose contents. The hydrophobicity of the amylose molecule affects the amount of water held by rice flour granules (Xu et al., 2015). A linear predictive model was established in this study to describe the impact of amylose content (Ac) and protein content (Pc) on water holding capacity (Equations 8 and 9).

$$WHC = 2.582 - 0.045\ Ac\ (\mathbf{R^2 = 0.960, RMSE = 0.028}) \quad (8)$$

$$WHC = 4.825 - 0.393\ Pc\ (\mathbf{R^2 = 0.935, RMSE = 0.043}) \quad (9)$$

Oil holding capacity of the tested flours ranged from 0.93 -1.27 g/g as seen in Table 3.1. The OHC of the flours in this study were comparably lower than the OHC (1.66 – 2.05 g/g) reported for basmati rice by Ahmed et al. (2015). SAH 329 reported the highest OHC (1.27g/g), while FARO 57 had the lowest OHC (0.93 g/g). A strong positive correlation (0.9204) was reported between the OHC of the rice flours and their respective protein contents. Menon, Majumdar, and Ravi (2015) attributed the increase in oil holding capacity to the presence of hydrophobic proteins in the flour. These hydrophobic proteins possess superior lipid binding properties. Kinsella (1979), indicated that oil absorption is a physical entrapment process which could be dependent on the non-polar side chains of the proteins as well as the conformational features of the proteins. Proteins have the ability to bind oil, therefore, making these flours useful in food systems that require optimum oil absorption. In addition, the high content of starch (amylose and amylopectin) in rice makes it an important factor in determining the oil uptake of rice flours. A strong positive correlation (0.965) was also reported between the OHC of the rice flours and their amylose contents. Amylose molecules possess a hydrophobic helical interior that binds to other hydrophobic molecules such as lipids (Xu et al., 2015). Based on the linear relationship observed between amylose content (Ac) and protein content (Pc) on OHC, a linear predictive model was established in this study (Equations 10 and 11).

$$OHC = -1.152 + 0.082\ Ac\ (\mathbf{R^2 = 0.965, RMSE = 0.042}) \quad (10)$$

$$OHC = -3.396 + 0.516\ Pc\ (\mathbf{R^2 = 0.965, RMSE = 0.041}) \quad (11)$$

The OHC information reported in this suggestion makes this rice flours applicable in the production of foods such as sausages that require flavor enhancement or retention. Our investigations suggested that the synergistic effect of amylose and proteins play an important role in the water holding capacity and oil holding capacities of rice flours.

3.5.4. Swelling power and water absorption index of rice flours

The swelling power (SP) and water absorption index (WAI) of rice samples are presented in Table 3.2. Swelling power of FARO 55, FARO 44, FARO 57, FARO 60, SAH 177, SAH 328 and SAH 329 flours increased from 6.23 to 9.09, 5.88 to 8.88, 6.89 to 9.89, 6.10 to 9.06, 5.52 to 8.96, 6.25 to 9.21 and 6.56 to 9.06 g/g rice flour, respectively. The swelling power of FARO 57 was higher than the other flours at the same temperature. The differences in SP of the studied rice cultivars can be attributed to the amylose and amylopectin content (Tester and Morrison, 1990) and the compositions of other constituents such as proteins and lipids in the rice flour matrix (Yu et al., 2012). However, this study could not find any significant relationship between the amylose content of the rice cultivars and its SP. Proteins packed tightly with rice starch amyloplast inhibit water permeation in rice flour granules and encourage lower swelling power.

WAI also increased with increase in temperature. The water absorption index of cereal grains like rice is an estimate of the volume held by the gelatinized starch after swelling in excess water (Abebe et al., 2015). This gel hydration property can be influenced by the particle size of the granule, level of starch damage and the interactions between starch molecules and non-starch components like proteins and cell wall materials.

Table 3.2. Swelling power and water absorption index properties changes with temperature

Cultivars	Swelling power (g/g)			Water absorption index (g/g)		
	70°C	80°C	90°C	70°C	80°C	90°C
FARO 55	6.23 ± 0.01*c*	7.59 ± 0.01*d*	9.09 ± 0.00*c*	6.13 ± 0.01*c*	7.61 ± 0.05*c*	9.12 ± 0.00*c*
FARO 44	5.88 ± 0.01*d*	7.39 ± 0.01*f*	8.88 + 0.01*d*	5.93 ± 0.00*e*	7.42 ± 0.01*d*	8.92 ± 0.01*e*
FARO 57	6.89 ± 0.01*b*	8.39 ± 0.01*b*	9.89 ± 0.01*a*	6.89 ± 0.01*a*	8.41 ± 0.01*a*	9.92 ± 0.00*a*
FARO 60	6.10 ± 0.00*c*	7.56 ± 0.05*d*	9.06 ± 0.01*c*	6.13 ± 0.01*c*	7.62 ± 0.05*c*	9.12 ±0.01*c*
SAH 177	5.52 ± 0.05*e*	7.46 ± 0.00*e*	8.96 ± 0.01*d*	5.99 ± 0.00*d*	7.49 ± 0.01*d*	9.01 ± 0.02*d*
SAH 328	6.25 ± 0.04*c*	7.72 ± 0.00*c*	9.21 ± 0.00*b*	6.27 ± 0.01*b*	7.76 ± 0.01*b*	9.26 ± 0.00*b*
SAH 329	6.56 ± 0.12*a*	8.48 ± 0.01*a*	9.06 ± 0.01*c*	6.11 ± 0.01*c*	7.68 ± 0.08*bc*	9.26 ± 0.01*c*

Means within each column with same letters are not significantly different ($P<0.05$). Data are means ± SD.

3.5.5. Thermal properties of rice flours

The results of the DSC analysis of rice flours from different rice cultivars are summarized in Table 3.3. Glass transition temperature, T_g for the rice cultivars ranged between 45.43 – 72.57 °C. These values were consistent with the findings of Thuc et al. (2010) and Biliaderis et al. (1986) who reported glass transition temperatures ranges of 40.38 - 47.70 °C and 61.90 – 81.60 °C, respectively for rice. The glass transition temperature indicates the temperature required to cause rearrangement of the molecular structure of the starch during thermal processing. SAH 329 reported the highest onset of gelatinization, T_o (81.27 °C) while FARO 44 showed the lowest T_o (56.70 °C). Gelatinization peak, (T_p) and gelatinization conclusion, (T_c) temperatures of the flours from the different cultivars ranged between 62.91 – 87.57 °C and 72.47 – 89. 87 °C, respectively. SAH 329 and SAH 328 flour showed the highest T_p values 87.57 °C and 79.55, respectively. These observations are likely due to high amylose contents reported by SAH 329 (31.48%) and SAH 328 (30.85%). High levels of crystallinity confer structural stability and make the granule more resistant to gelatinization (Singh et al., 2006). T_o, T_p and T_c reflect the breakdown in the double helical and crystalline arrangements during gelatinization. These results were higher than the findings of Singh et al. (2006) who reported T_o (61.10 -75.76 °C), T_p (66.91 – 79.21) T_c (71.93 - 84.59) for low amylose (< 16%) containing rice starches. SAH 177 reported the highest enthalpy of gelatinization, ΔH_G values (7.46 J/g), while FARO 60 showed the least (0.38 J/g). ΔH_G typically reflects the loss of molecular (double-helical) arrangement in the starches of the studied cultivar (Cooke and Gidley 1992). High enthalpy of gelatinization has been reported to result from a high degree of crystallinity. The variation in the studied thermal properties T_o, T_p, T_c and ΔH_G of the studied flours are likely due to the differences in amounts of longer chains of amylopectin (Singh et al., 2006). These long chains of amylopectin require higher temperatures to break them down completely. A quadratic predictive model was established in this study to describe the relationship between the amylose content (Ac) and the T_p (Equation 12).

$$T_p = 37.279 + 1.369\ A_C + 0.594\ Ac^2 (\mathbf{R^2 = 0.900, RMSE= 1.806}) \tag{12}$$

Table 3.3. Thermal properties of rice flours.

Rice cultivars	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_G (J/g)	T_g (°C)
FARO 55	72.57 ± 3.13a	76.78 ± 0.98g	82.00 ± 0.04b	4.06 ±0.08a	70.89 ± 3.08a
FARO 44	56.70 ± 3.14b	62.91 ± 4.39b	72.47 ± 2.55c	3.81 ± 1.77b	72.57 ± 3.18b
FARO 57	75.84 ± 1.88c	78.41 ± 0.77a	84.36 ±2.18a	1.69 ± 0.01c	65.59 ± 0.01c
FARO 60	74.25 ± 0.06d	74.50 ± 0.21c	76.93 ± 0.04g	0.38 ± 0.02d	68.57 ± 0.10d
SAH 177	75.63 ± 0.02e	79.46 ± 0.05d	83.61 ± 0.02d	7.46 ± 0.03g	45.43 ± 4.84e
SAH 328	76.96 ± 1.22g	79.55 ± 0.88e	83.67 ± 0.01e	6.80 ± 0.06e	47.30 ± 1.15f
SAH 329	81.27 ± 0.01f	87.57 ± 0.03f	89.87 ± 0.01f	2.45 ± 0.01f	66.03 ± 0.03g

Means within each column with same letters are not significantly different ($P<0.05$). Data are means ± SD.

3.5.6. Rheological properties

3.5.6.1. Flow behavior

Rheological spectrum (complex viscosity, η*) of rice flours (4, 6 and 8% w/w) at 95°C is shown in Fig 3.2. As seen in Fig. 3.2. it is evident that irrespective of the concentration, all the dispersions showed a time-dependent reduction of complex viscosity η* with increasing frequency. This reduction is typical of shear thinning/pseudoplastic behavior. When the slopes of η* (8% flour-water dispersion) against frequency angular frequency was calculated, the following slopes for FARO 44, FARO 55, FARO 57, FARO 60, SAH 177, SAH 328 and SAH 329 were -0.863, -0.793, -0.836, -0.837, -0.855, -0.911, -0.886, respectively. These slopes are steeper than the -0.76 value reported by Morris (1990), which was used to explain the "weak gel" behavior of polysaccharide gels generated by overlapping and entangled flexible random coil chains (Ye et al., 2016). Moreover, the nature of the curves (Fig.3.2) indicates that for all concentrations, the complex viscosity is influenced to a higher degree at low frequency than at high frequency. According to Bertuzzi et al. (2007), at a low shear rate, in this case, angular frequency, the time required for structure relaxation is higher than the rate of angular frequency change. As angular frequency increases, swollen starch granules in the rice flour disintegrate, molecular conformations break down and molecules take an extended arrangement. The internal framework of the polymer re-orientates to support angular frequency decreasing their complex viscosity. Thus, the mobility of starch granules in the flour is increased and the relaxation structure is reduced. Finally, the attained structure of the rice flour dispersions stays in equilibrium with the imposed angular frequency and complex viscosity becomes almost constant (Liu et al., 2015; Singh et al., 2006).

Fig 3.2. shows the effect of angular frequency on complex viscosity on the rice flour dispersions. The results of this study show that all the cultivars exhibited a similar behaviour. The change in complex viscosity reflects the impact of individual starch molecules of the rice flours in the dispersion (Liu et al., 2015). As angular frequency increases, the interaction of starch molecules in the flour become weaker and a lesser proportion of the water molecules become immobilized, thus resulting in a further fall of complex viscosity.

The power law model of $G' = K' \omega^{n'}$ (Equation 6) was used to evaluate the flow properties of the rice flour gels under dynamic shear. The relaxation exponent, n and consistency index, K are shown in Table 3.4. The relaxation exponents, n' (storage modulus, G') and n'' (loss modulus, G'') showed non-linear changes with an increase in concentration for all cultivars. The relaxation exponent values (n' and n'') in most cases decreased as concentration increased, thus suggesting concentration had no impact on n' and n'' values. This observation, therefore, reinforces the stand that the material exhibits shear thinning/pseudoplastic behavior. The study revealed that the relaxation exponents of the dispersions for FARO 60 and FARO 44 increased at 6% concentration and then decreased at 8% concentration. On the other-hand FARO 57, FARO 55, SAH177, SAH 328 and SAH 177 dispersions showed a decrease in the relaxation exponents with increasing concentration. The results of this study were consistent with the finding of Ye et al. (2016) which reported n' (0.05 -0.42) and n'' (0.26- 0.43) values for brown rice flours from Indica rice. The relaxation exponents (n' and n'') serve as a reference point for analyzing the transition behavior near gel point of a polymeric dispersion. Prasad et al. (2013) reported that relaxation exponents closer to 1 indicate softer gels, while exponents closer to 0, suggest a stiffer gel. In general, the relaxation exponents of the cultivar dispersions at all concentrations indicate the formation of poorly elastic gels. Relaxation exponents are greatly dependent on the molecular and structural properties of the polymer which in turn affect the development of viscoelastic suspensions (Liu et al., 2015). The impact of the molecular and structural framework of the flours on these rheological parameters can be studied in the future.

The consistency indices, k' and k'', signify the viscous behavior of a fluid. The higher the value of k' and k'', the higher the viscosity of the material (Kaur and Das 2014). This study observed that the consistency indices k' (storage modulus, G') and k'' (loss modulus, G'') were concentration dependent with the values of k' and k'' at 8% concentration higher than the values of k' and k'' at 6% and 4%, respectively (Table 3.3). The k' and k'' values in this study were consistent with the k' (0.10 – 5.28 Pa $s^{n'}$) and k'' (0.36 -3.07 Pa $s^{n''}$) reported by Chun and Yoo (2004) for Korean rice flour dispersions. Similar viscoelastic trends have been reported for basmati rice flour (Prasad et al., 2013) and barley flour (Kaur and Das 2014). The volume distribution of the flour particles might have influenced the consistency index of both the storage and loss modulus.

Table 3.4. Rheological responses of rice flours based on power law model.

	n'			n''			k' (Pa s)$^{n'}$			k''(Pa s)$^{n''}$		
Cultivar	4%	6%	8%	4%	6%	8%	4%	6%	8%	4%	6%	8%
FARO 60	0.03	0.04	0.03	0.03	0.04	0.03	0.14	0.70	2.79	0.14	0.69	2.79
FARO 57	0.08	0.03	0.03	0.08	0.03	0.03	0.14	1.60	2.19	0.14	1.60	2.19
FARO 44	0.17	0.39	0.03	0.17	0.39	0.03	0.10	0.27	2.33	0.10	0.27	2.33
FARO 55	0.28	0.06	0.04	0.28	0.06	0.04	0.16	0.30	2.06	0.16	0.30	2.06
SAH 177	0.35	0.04	0.03	0.35	0.04	0.03	0.25	0.98	3.00	0.25	0.98	3.00
SAH 328	0.09	0.03	0.01	0.09	0.03	0.01	0.13	0.48	2.94	0.13	0.50	2.94
SAH 329	0.11	0.10	0.02	0.11	0.10	0.02	0.13	0.45	2.78	0.13	0.45	2.77

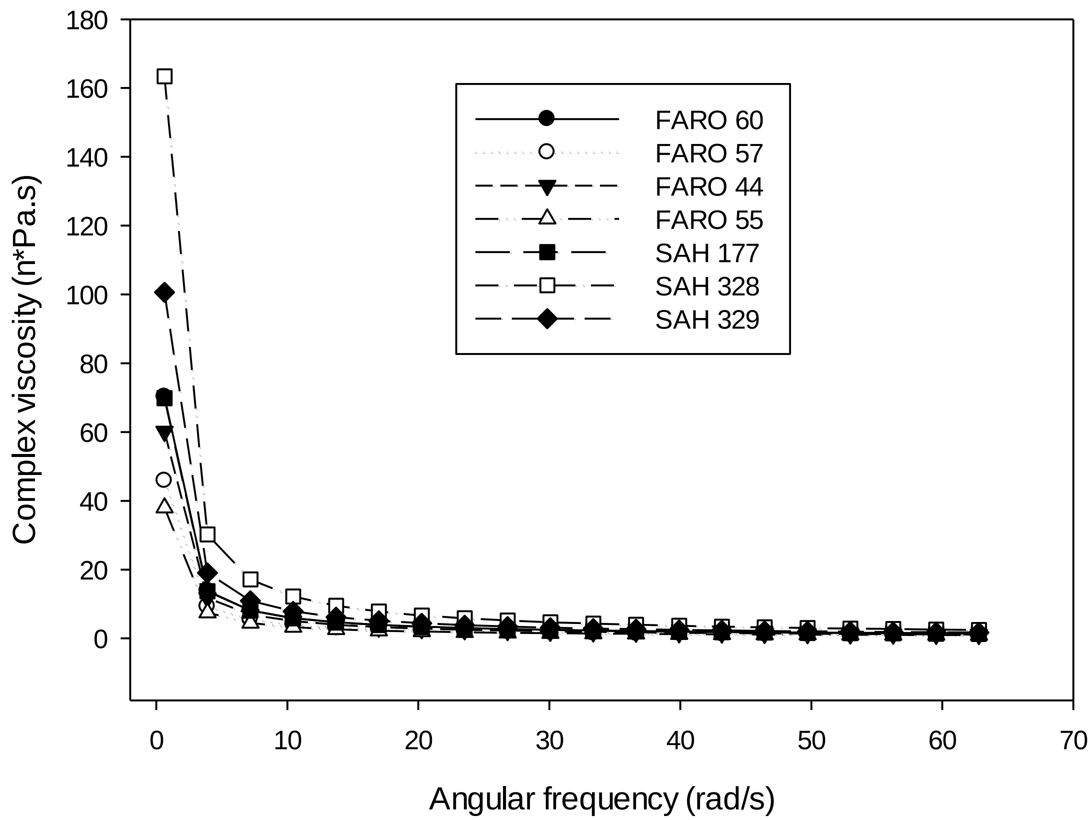


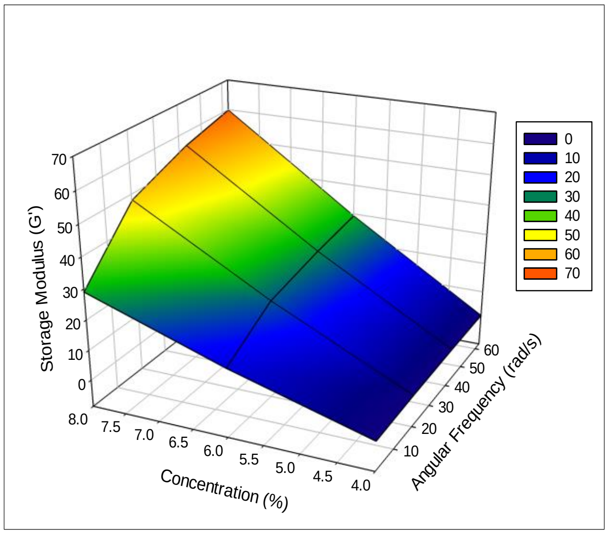
Fig 3.2. Complex viscosities of rice flours as a function of angular frequency.

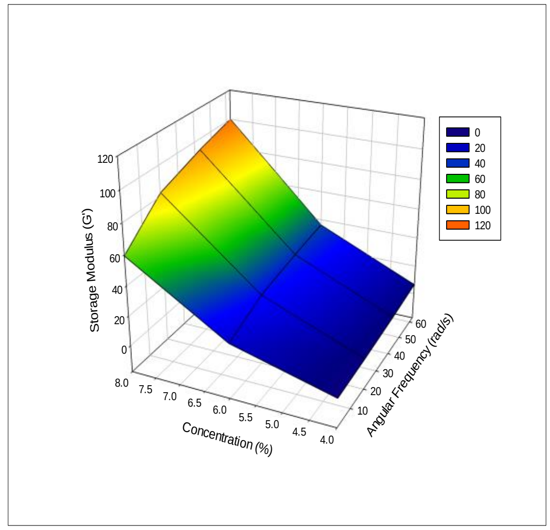
3.5.6.2. Storage modulus **G'** *and loss modulus* **G''**

The interactive effects of concentration and angular frequency on the storage modulus, G' of SAH 328, SAH 329, FARO 57 and FARO 60 are shown in Fig. 3.3. The results of the study show a wide variation in the magnitude of G' values of studied cultivar dispersions. The maximum G' observed in this study for SAH 328, SAH 329, FARO 57 and FARO 60 was 151.7, 126.7, 59.84 and 100.1, respectively. FARO 57 displayed the lowest maximum G' (59.84), while SAH 328 showed the highest maximum G' (151.7). The lower G' values of FARO 57 may be attributed to the strong swelling power (9.89 g/g) of its flour granules. This correlation was consistent with the findings of Sun and Yoo (2011) who correlated low G' of Korean rice cultivar dispersions with its maximum swelling power. A similar trend was observed in the magnitude of loss modulus, G" values (Fig 3.4). Maximum G" values of all the cultivar dispersions reported in this study were in the range of 21.55 - 28.63. The maximum G" values of the dispersions of the cultivars from Senegal were higher than that of the cultivars from Nigeria. Kong et al. (2015) correlated higher G' and G" values of rice flour and starch dispersions with higher amylose content. The protein content of rice flour has also been related to G' of rice flour. SAH 328 had the highest protein content and G' values in comparison to the other studied cultivars. The result of this study supports Kong et al. (2015) observation that an increase in protein significantly increased the G' values of rice flour. The changes in the elastic and viscous component may be due to the differences in their granular properties (such as rigidity and integrity) caused by the protein and amylose components of the flours (Sun and Yoo, 2011).

The dispersions in this study all exhibited quantitative differences in the two moduli. It is noteworthy to state that in comparison to the cultivars from Nigeria, the response surface methodology plots for G' and G" showed a plateau for the cultivars from Senegal at 4% to 6% concentration for G' and G". The plateau reflects the variation in G' and G" values (less than 5%) of the cultivars from Senegal in relation to concentration change was insignificant at these concentrations. However, beyond 6% a greater increase in the G' and G" was observed. The qualitative trend of the impact of concentration on G' and G" values was the same for the cultivar dispersions from Nigeria, however, the magnitude of G' and G" in relation to concentration was not as prominent in comparison to the cultivar dispersions from Senegal.

The frequency dependence of G' and G" in rice flour dispersions illustrated in Fig.3.3 and 3.4 show that G' was consistently more than 2 times higher than G" in the frequency range studied and both moduli showed little frequency dependency at all concentrations, thus exhibiting a weak like gel-like characteristic.





(a)

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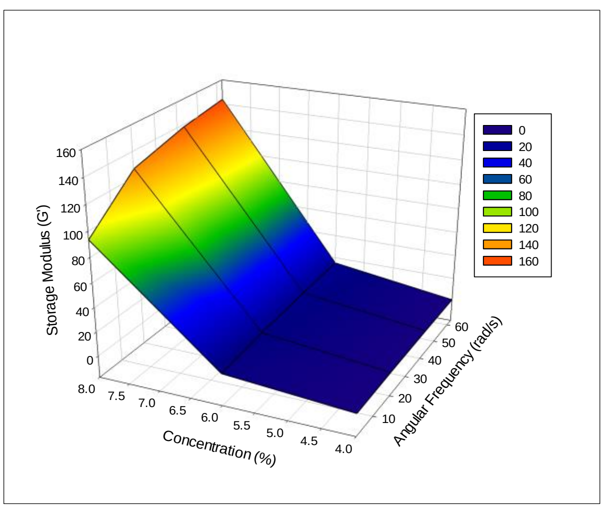
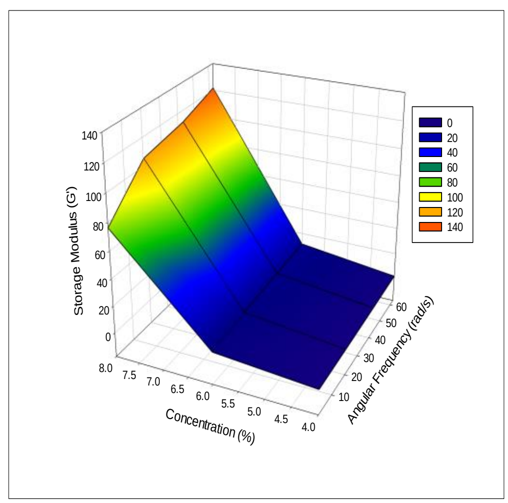
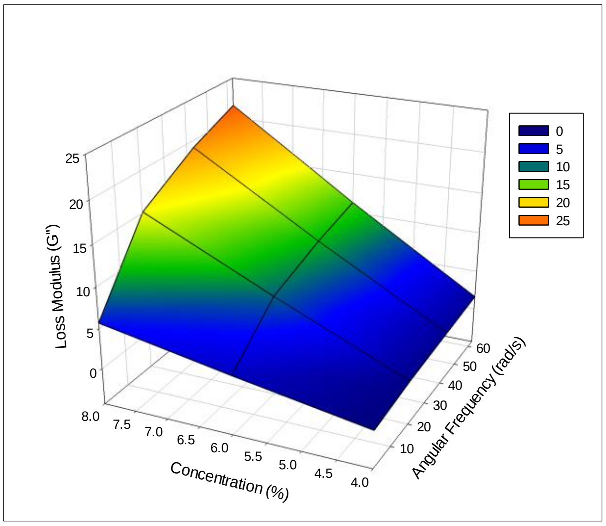




(c)

(d)

Fig 3.3. Impact of concentration and angular frequency on the G' of (a) FARO57, (b) FARO 60, (c) SAH 328 and (d) SAH 329 rice flour dispersions.



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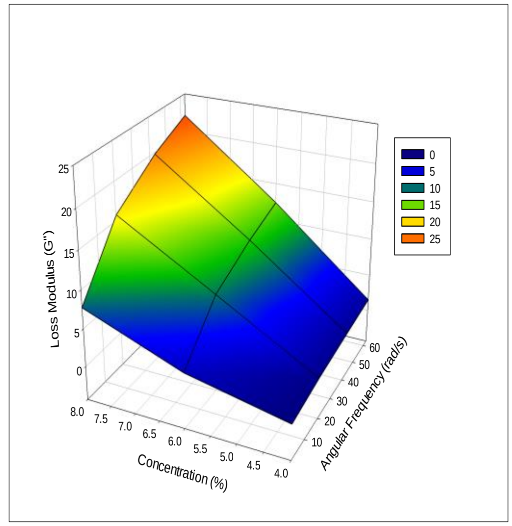

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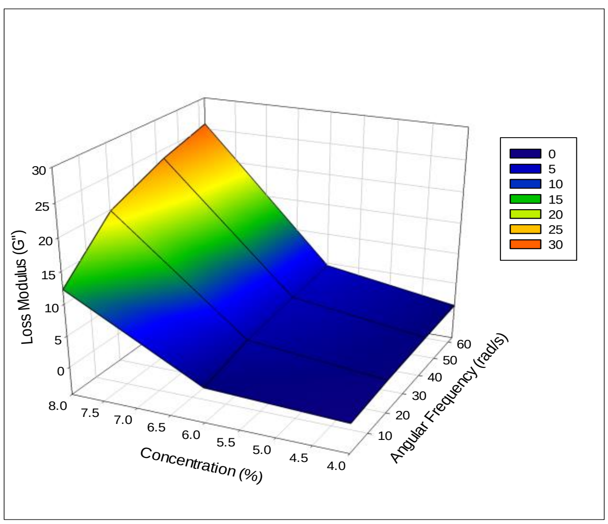

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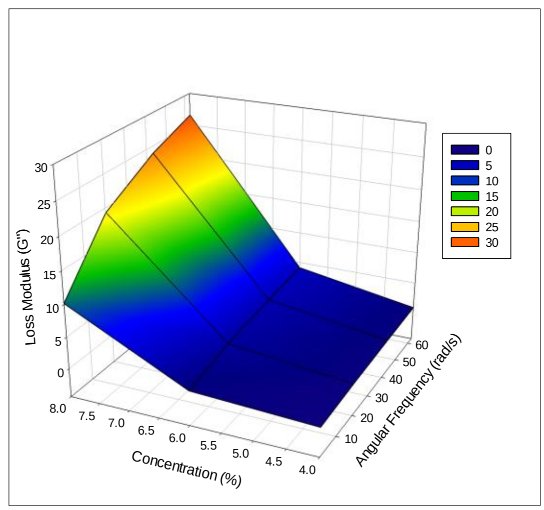


d

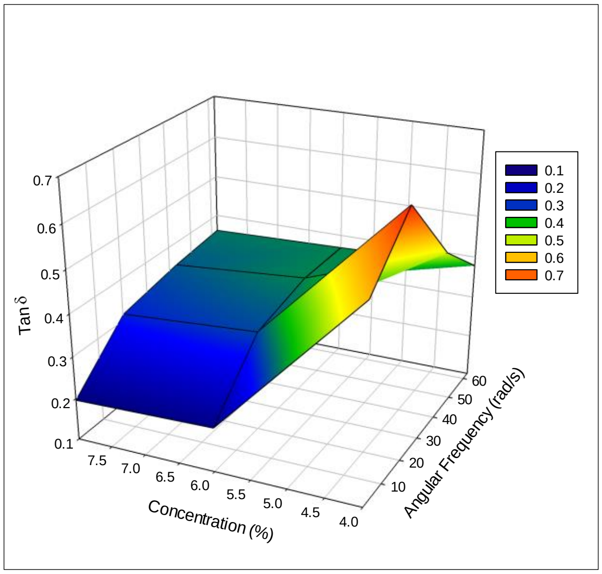
Fig 3.4. Impact of concentration and angular frequency on the G" of (a) FARO57, (b) FARO 60, (c) SAH 328 and (d) SAH 329 rice flour dispersions.

3.5.6.3. Loss factor (tan δ)

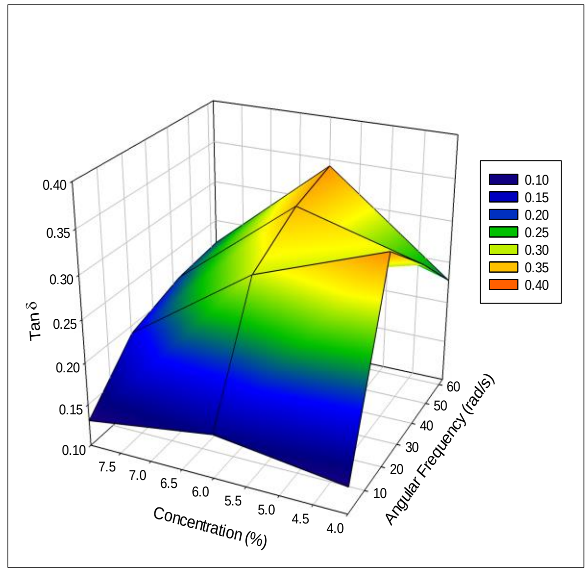
Fig 3.5. presents the loss factor, tan δ = (G" / G'). It is a sensitive sign of cross-linking in a polymeric material. This dimensionless measurement compares the amount of energy lost (loss modulus) during a test cycle to the amount of energy stored (storage modulus) during the test time (Ye et al., 2016). Except for SAH 328 (maximum tan δ value of 1.42) which reported a maximum tan δ, the other cultivar dispersions exhibited tan δ values within the range of 0.11 – 0.83 (Fig 3.5.), indicating that the samples were more elastic than viscous. The result of this study shows that the values of tan δ for cultivar SAH 328 dispersion were significantly higher compared to other cultivars. SAH 328 cultivar had a higher protein content (9.44%) than the other cultivars, indicating that the dispersion gets more viscous with an increase in protein content. This finding concurs with results from Pedersen et al. (2004), who also reported increasing tan δ with increasing protein in dispersion made from wheat.

As seen in Fig 3.5, the tan δ values of SAH 328, SAH 329 and FARO 57 decrease with increasing concentration. On the other hand, tan δ values of FARO 60 increased with increasing concentration (4 to 6%) and then decreased beyond 6% concentration. The decrease in tan δ indicates network formation and the loss of non-homogeneity in the dispersions (Rao 2007). The reduction observed in the values of tan δ with increasing concentration indicates an improved network being formed. This improvement is a result of stronger interactions between the molecules with increasing flour to water concentration. The initial resistance in molecular interaction as evident in the increase of tan δ values of FARO 60 with increasing concentration (i.e. 4 to 6% concentration) can be attributed to the presence of amylose in the material, which is distinctive for the cultivar. Singh et al. (2003) reported the ability of amylose molecules to form lipid complexes, which retards their leaching and consequently the swelling capacity and improved network.

As presented in Fig. 3.5, for all the cultivar dispersions, tan δ exhibited less dependency to the angular frequency at a low angular frequency range (10 - 30 rad/s); while it had higher dependency at a higher frequency range (above 30 rad/s). This behavior as well demonstrated the weak gel properties of the dispersions. The implications of the tan δ values could be beneficial in the utilization of these flours in dough production. Values of tan $\delta < 1$ reflect a solid-like characteristic, whereas tan $\delta > 1$ imply an increased capability to flow (Guadarrama-Lezama et al., 2016).



(a)



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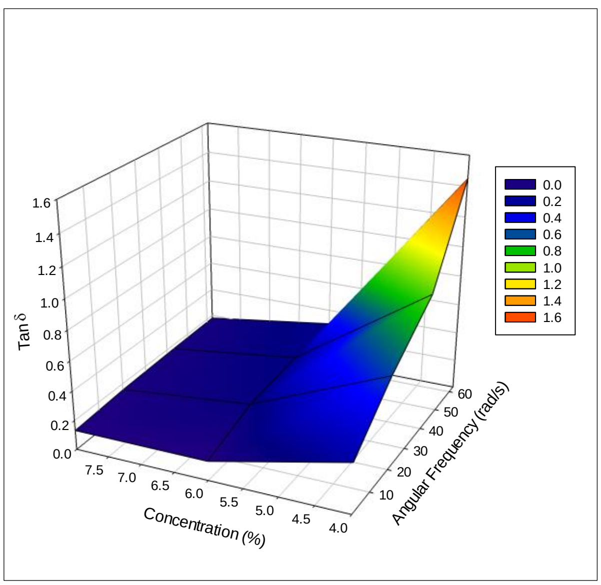

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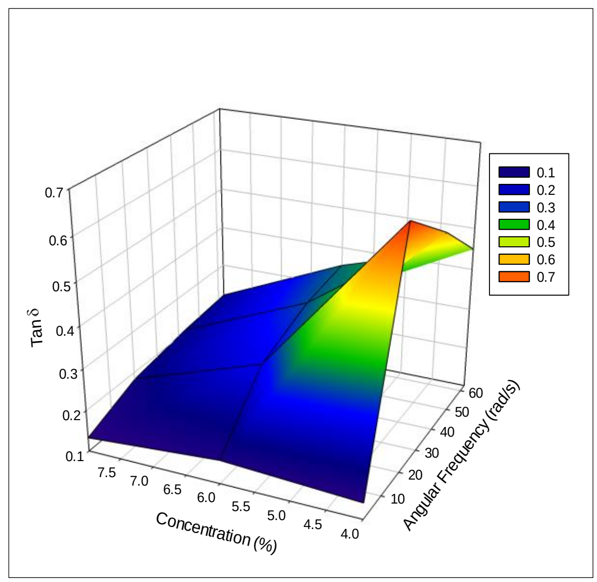


(d)

Fig 3.5. Impact of concentration and angular frequency on the Tan δ of (a) FARO57, (b) FARO 60, (c) SAH 328 and (d) SAH 329 rice flour dispersions.

3.6.Conclusion

Physicochemical, thermal and rheological characteristics of the rice clearly varied with cultivar. Generally, the rice cultivars showed high amylose contents (>25%). The X-ray diffraction patterns of the rice cultivar flours were A-type, and the degree of crystallinity was related to the amount and distribution of amylose and amylopectin in the rice. Oil and water holding capacities of the studied cultivar samples were related to the protein and amylose contents. The swelling power and WAI were presumably related to temperature and amylose content. Thermal properties of the rice flours showed higher gelatinization peaks (T_p) but low glass transition temperatures (T_g); providing an insight into the flour's behavior under specific thermal processing conditions expected in industrial processes. The predictive models designed in this study (equations 8, 9, 10, 11 and 12) were suitable for describing the experimental observations for the functional (water and oil holding capacity) and gelatinization properties of flour from the non-waxy rice cultivars with the R^2 values, which is a measure of the goodness of fit, higher than 0.900. The suitability of equations 8, 9, 10, 11 and 12 for predictive purposes was confirmed using the root mean square of error (RMSE), which were found to be generally low (< 1.806). These relatively low RMSE values indicate good fit and suitability of a model for practical purpose. Furthermore, all the rice flour dispersions at different concentrations (4-8%) displayed a non-Newtonian shear-thinning behavior. G' was greater than the G" at all the values of ω utilized in this study. The rice flours from this study all formed weak viscoelastic gels. The obtained data from this study provides a choice of rice flour with varying amylose content for the development of unique gluten-free products with desirable functional and rheological attributes.

CONNECTING TEXT

In chapter 3, the physical, chemical, structural, thermal and rheological properties of flours from seven non-waxy rice cultivars from Nigeria and Senegal were obtained. SAH 329 and SAH 177 were selected based on the significant high amylose contents reported in the previous chapter and since there is lack of literature on rice cultivars from this part of the world. In chapter 4, heat-moisture treatments were used to study the physical, chemical, thermal and rheological properties of SAH 329 and SAH 177 rice flours under different moisture, temperature and time regimes.

4. Impact of heat-moisture treatments on the physicochemical, functional, thermal and rheological properties of high amylose rice flour

4.1. Abstract

High amylose rice flours SAH 177 and SAH 329 were heat-moisture treated at different temperatures (85, 100 and 115 °C) for 1, 2 and 3 hr at a moisture content of 25, 30 and 35%. The effects of heat-moisture treatment (HMT) on the protein content, amylose content, water and oil absorption capacities, color, thermal and rheological properties were studied. Varying the moisture content and processing temperature during heat-moisture treatment led to significant changes in these selected studied properties. Increasing the moisture content during heat-moisture treatment produced significant reductions in the protein contents, amylose contents and oil absorption capacities of the rice flours. Data obtained in this study showed that increasing the processing temperature during heat-moisture treatment resulted in an increase in the water absorption properties of the flours. Our data showed a 1.5°C reduction in gelatinization temperature for every percent increase in moisture content. The rheological analysis provided information on the yield stress of the heat-moisture treated flours. The results showed that the yield stress of the heat-moisture treated rice flours was significantly lower than native rice flours. Overall, these results suggest that adjusting heat-moisture treatment parameters could help in the production of flours with properties unique for particular functionalities.

Keywords. Rice. Response surface methodology. Heat moisture treatment. Rice flour.

4.2.Introduction

Rice is a principal staple food crop consumed by nearly half of the world's seven billion people (IRRI, 2017). Commercial rice flour is produced either by dry or wet milling of broken rice grains. Currently, rice flours have been utilized in the production of novel and traditional food products such as infant foods, puffed grains, and gluten-free products. However, rice flour, in its native form, has limited application in the industry due to negative functionalities of the flour such as poor resistance to shear force and low elastic gel-forming properties (Qin et al., 2016). Changes in these aspects would enhance the versatility of rice flours in food products such as noodles, puddings, fried batter food, and breakfast cereal foods. Therefore, it is important to look at modifying processes that can enhance the application of rice flours.

Heat-moisture treatment (HMT) is a physical modification method applied to starches. In principle, HMT involves treatment of starch granules at higher temperatures (typically above gelatinization temperature) at controlled moisture content (<35%) for a specified period (15mins – 16h). In comparison to chemical modification treatment, it is natural and safe (Sun et al., 2013). Much attention has been paid to the influence of hydrothermal technologies on the physicochemical, thermal, rheological and functional properties of starch and flour (Puncha-arnon and Uttapap, 2013). Several studies have illustrated the significant influences HMT has on the viscosity, gelatinization parameters, granule swelling, water absorption property and oil absorption capacity of cereals such as rice, maize and millet (Bourekoua et al., 2016; Bucsella et al., 2017; Cetiner et al., 2017; Fathi et al., 2016; Liu et al., 2016; Qin et al., 2016; Shang et al., 2016; Sun et al., 2013).

Although, considerable studies have been carried out to evaluate the impact of heat-moisture treatments on various properties of rice (Chen et al., 2017; Fathi et al., 2016; Hormdok and Noomhorm, 2007; Jiranuntakul et al., 2011; Liu et al., 2016; Shang et al., 2016; Silva et al., 2017; Zavareze et al., 2010), additional work is still needed on this topic. In most part of the past investigations in the literature, heat-moisture treatment is applied to waxy rice flours rather than on non-waxy rice. Flour varies greatly in form and functionality among and within botanical species. Waxy rice flour differs from non-waxy rice mainly in having low or no amylose (<5%) in the starch matrix but mainly amylopectin molecules. This difference in amylose and amylopectin content and distribution impacts many of the functionalities of flour (Puncha-arnon and Uttapap, 2013). Therefore, this study aims at providing vital information on the impact of heat-moisture treatment on non-waxy rice flours. Secondly, in a good part of the literature, heat-moisture treatment of rice flour is carried out in glass containers (Bian and Chung, 2016; Chung et al., 2014; Chung et al., 2012; Majzoobi et al., 2015; Puncha-arnon and Uttapap, 2013) expected to be poor heat and mass transfer materials. It is therefore important to evaluate this hydrothermal treatment process for rice flour in thermally stable aluminum sealed containers. These variations between the existing literature and this present investigation are to broaden the understanding of HMT while elucidating the combined impact of process conditions and optimizing the process input using response surface methodology.

The aim of this study was to evaluate the impact of heat-moisture treatments on the physicochemical, thermal, functional and rheological properties of rice flour using response surface methodology, and to provide some information on the functional applications of rice flour.

4.3.Materials and Methods

4.3.1. Materials

Two cultivars of rice, SAHEL 329 and SAHEL 177, grown and purchased in Senegal were used in this study.

4.3.2. Methods

4.3.2.1. Experimental design for the heat-moisture treatments

JMP statistical software (SAS Campus Drive, NC, USA) was utilized for generating the experimental model for this study. Moisture (M), Temperature (T) and Time (S) were selected as the three independent factors in this heat moisture study. Quality characteristics of the rice; physicochemical, structural, thermal, functional and dynamic properties were chosen as the dependent factors. The temperatures in this study were 85, 100 and 115 °C, the moisture contents were 25, 30 and 35% and the time duration were 1, 2 and 3 hours. The experimental model created sixteen temperature-moisture-time combinations with two points in the middle (Table 4.1).

Table 4.1. Experimental model for heat moisture treatment

Run	Time (hr)	Temperature (°C)	Moisture content (%)
1	1	115	25
2	3	85	35
3	2	100	30
4	1	115	35
5	1	85	25
6	2	115	30
7	1	100	30
8	2	85	30
9	2	100	35
10	2	100	30
11	2	100	25
12	3	115	35
13	3	85	25
14	3	115	25
15	1	85	35
16	3	100	30

4.3.3. Milling

Rice grains were milled using a coffee grinder (SUMEET Multi Grind, India), screened through a 250μm sieve, packed and stored in several airtight plastic bags at ambient temperature for further use.

4.3.4. Heat-moisture treatment

Rice flour (150 g each) were adjusted to moisture contents of 25, 30 and 35% (the MC of native flours was predetermined) by dispersing in an appropriate amount of distilled water. Prior to the heat-moisture treatment, the rice flour samples were held in sealed plastic bags and kept for 24 h at 4 °C to prevent the possible moisture losses. Heat treatments of the samples in screw-capped airtight aluminum containers were carried out for 1, 2 and 3 hours, after achieving the target temperatures in a convection oven.

4.3.5. Physicochemical studies

4.3.5.1. Chemical analysis

The protein values (N content x 6.25) of the flour samples were estimated by combustion using a LECO Nitrogen Analyzer (CNS 2000, Operation Manual, Leco, St. Joseph, MI, USA). Flour samples were weighed into a porcelain sample holder (boat) for introduction into the combustion chamber (1300°C) of an automated sample loader. Nitrogen was produced as a result of this combustion process. The amount of protein present in the samples was estimated by multiplying the nitrogen content by a factor of 6.25. Amylose content of flour samples was analyzed using the iodine-binding colorimetric method of Juliano (1971). The absorbance of blue color from the binding was measured with a spectrophotometer at 620nm. Standard curves prepared from potato amylose were used to estimate the amylose content of the flours.

4.3.6. Flour hydration properties

The water holding capacity (WHC) was estimated by dispersing 1.000 ± 0.005 g of flour in distilled water (10 ml) and keeping the resulting mixture at ambient temperature for 24 hours. The supernatant was decanted. WHC was expressed as grams of water retained per gram of solid.

Oil holding capacity (OHC) was determined by the method of de la Hera et al. (2013). Rice flour (100.0 ± 0.2 mg) was mixed with 1.0 ml of vegetable oil. The resulting mixture was stirred for 1 min with a wire rod to disperse the sample in the oil. After a period of 30 min in the vortex mixer, the mixture was centrifuged at 3000 × g and 4°C for 10 min. The supernatant was removed, and the holding vessels inverted for 25 min to drain the oil. After this, the resulting residue was referred to as W_r. The oil absorption capacity was expressed as grams of oil bound per gram of the sample on a dry basis. For the determination of all properties, three replicates were made for each sample.

4.3.7. Color

Colorimetric measurement of flour samples was determined using a colorimeter (CR-10, Konica Minolta Sensing Inc, Osaka, Japan). The CIE values were recorded as L* (lightness), a* (redness) and b* (yellowness).

4.3.8. Thermal analysis

Thermal properties such as peak gelatinization temperature (T_p) and enthalpy of gelatinization (ΔH) were examined using a differential scanning calorimeter (DSC Q2000; TA instrument, New Castle, PA, USA). Samples (2.5 mg, dry basis, db) were weighed into an aluminum pan and mixed with distilled water to obtain a sample -water suspension of 2:1. The samples were hermetically sealed and allowed to reach equilibrium overnight at room temperature before heating in the DSC. The instrument was calibrated with indium and an empty pan as a reference. The sample pans were heated at a rate of 5 ˚C /min from 25 to 125 ˚C.

4.3.9. Dynamic shear properties

4.3.9.1. Preparation of dispersion

Rice flour dispersion (8% w/w) was prepared by mixing rice flour with distilled water. The mixtures were stirred for 30 min at ambient temperature and then heated at 95°C in a water bath for 30 min with mild agitation carried out by a magnetic stirrer. After heating, the cooked rice flour dispersions were immediately introduced to the rheometer to estimate the steady and dynamic rheological properties.

4.3.9.2. Dynamic rheological measurement

Dynamic shear properties such as storage modulus (G'), loss modulus (G") and tan δ = (G"/ G'), of the different dispersions of rice flours were determined with a rheometer (AR 1000, TA Instruments, New Castle, DE) using a parallel plate system (4cm diameter) at gap of 500µm at 25°C. Dynamic shear data obtained were from frequency sweeps over the range of 0.63 - 62.8 rad. $^{s-1}$ at 2% strain. The rheological measurements were performed in triplicate.

4.3.10. Statistical analysis

All measurements were performed in duplicate. Analysis of variance (ANOVA) and the least significant difference was carried out using the using JMP software (Version 14; SAS, NC, USA). Turkey’s test was applied to detect differences in the means and $P < 0.05$ was statistically significant.

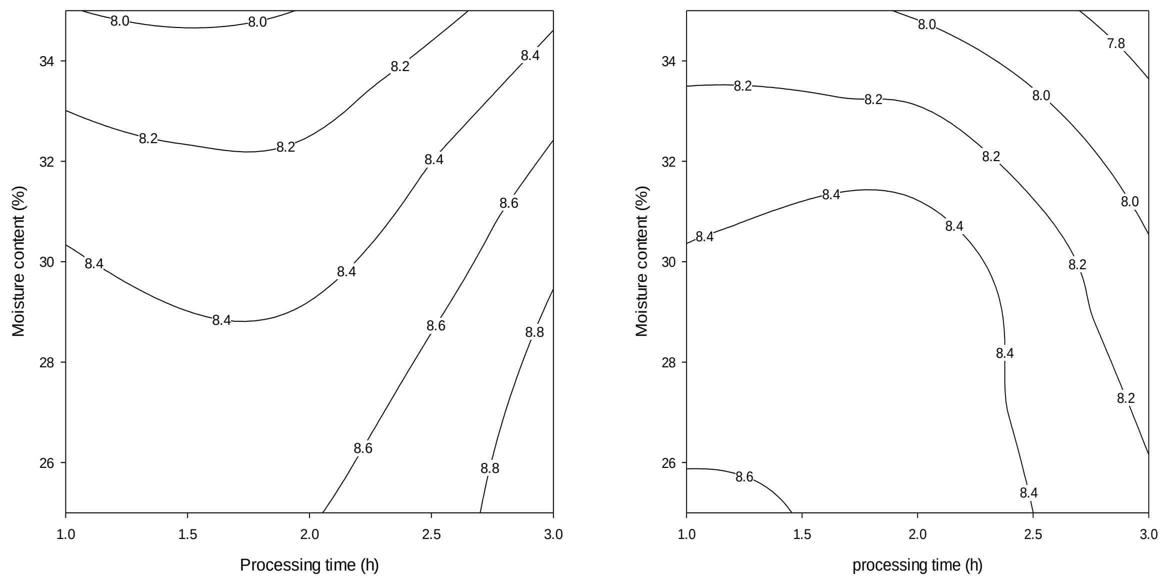
4.4.Results and discussions

4.4.1. Impact of HMT treatment on Protein and Amylose contents

4.4.1.2. Protein content

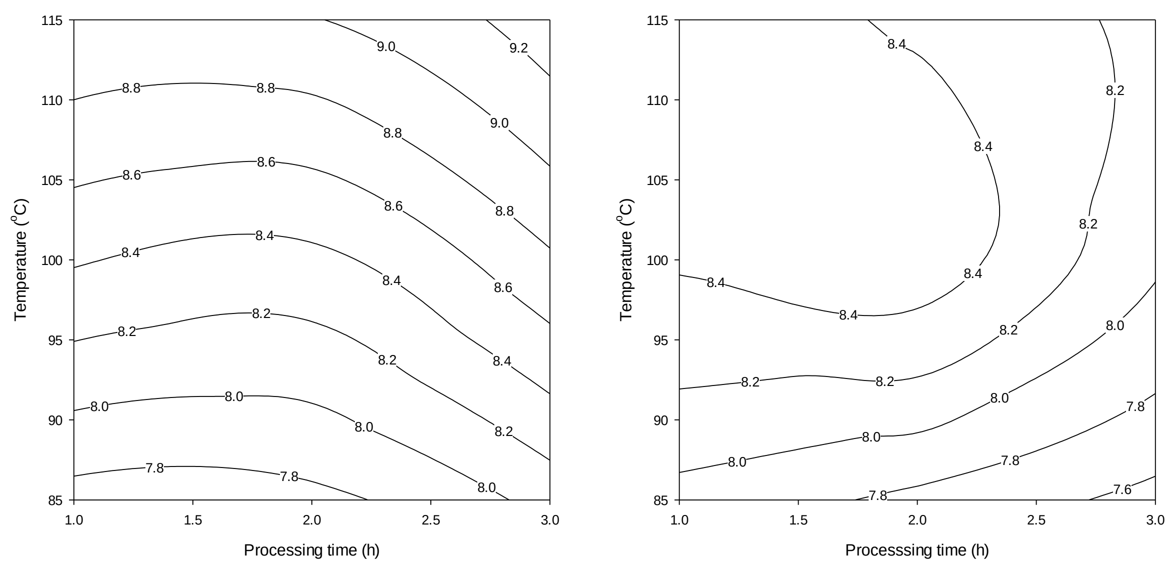
Protein plays a functional role in cooked rice flour texture because they form a complex with starch that impairs starch granule swelling. This swelling impact both viscosity intensity and the degree of starch gelatinization in the flour matrix. The impacts of moisture content and temperature during HMT on the protein content of treated rice flours are shown in Fig 4.1 and 4.2, respectively. The result indicates a general decline in protein content as the moisture content was increased during HMT (Fig 4.1). A total decline of up to 0.8% was observed depending on the treatment time. For instance, when the rice samples are subjected to a 2-hour heat treatment at 100 ^{o}C, a 0.2 ± 0.03% reduction in the protein content is observed for every 5% increase in moisture. The observed decline in protein as moisture increases could be due to changes in solubility during heating, which may have caused the proteins to leach out into the water. Chen et al. (2016) observed a similar decline when *F. thumbergii* flours were treated from 4.35 to 0.39%.

Unlike moisture, there seems to be a direct relationship between process temperature and protein content. It was clear from these plots (Fig 4.2) that at a specific moisture content, the impact of temperature was dominant compared to time. For instance, at 30% moisture content, a 13% increase in protein is seen as temperature increases from 85 to 115 ^{o}C when samples are processed for an hour. However, at the same moisture content, only a 2.4% increase in protein could be achieved as processing time increased from 1-3 h at 100^{o}C. Denaturation and changes in solubility of protein due to increases in temperature lead to the unfolding of the primary structure of protein molecules which in turn could lead to increase in protein size and quantity (Ortolan and Steel, 2017).



(a) (b)

Fig 4.1. Effect of moisture content on the protein content (%) of HMT treated rice flour (a) SAH 329 (b) SAH 177

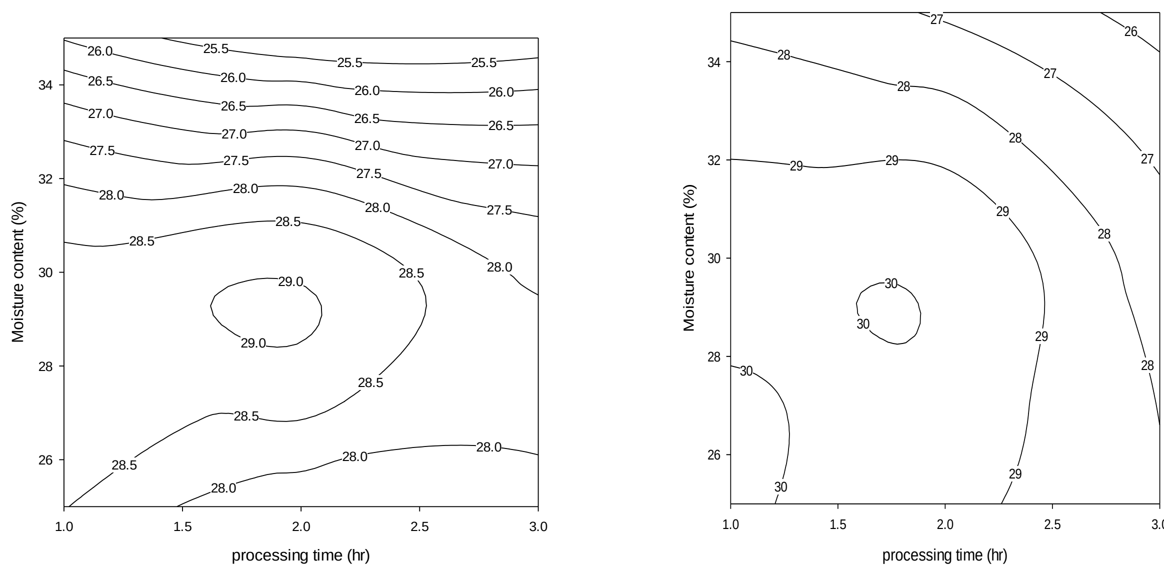


(a) (b)

Fig 4.2. Effect of temperature on the protein content (%) of HMT treated rice flour (a) SAH 329 (b) SAH 177

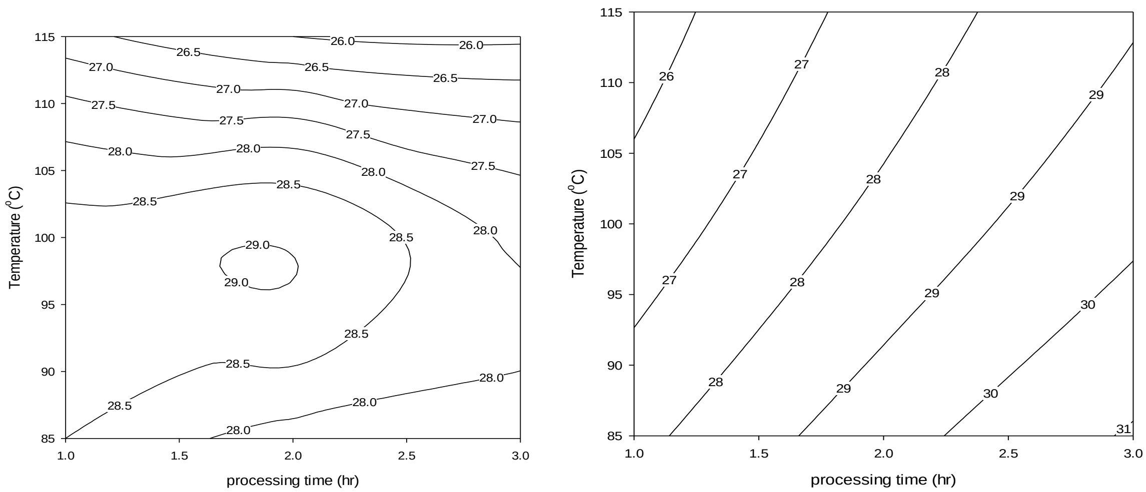
4.4.1.3.Amylose content

Flour gelatinization is dependent on its amylose content. The interaction of amylose with other constituents in rice flour can dictate the functional, thermal and rheological behavior of the flour (Tukomane and Varavinit, 2008). Fig 4.3 and 4.4 provide an insight into the influence of moisture content and temperature on the amylose contents of HMT treated rice flours. The results reveal a slow decline in amylose content as the moisture content increases in both cultivars for any given processing time (Fig 4.3). Flour granules are quite resistant to breakdown by both water and hydrolytic enzymes due to the formation of hydrogen bonds within the starch molecules and with other neighboring entities. However, these inter- and intra-hydrogen bonds can become weak as the moisture content of the suspension is raised. When an aqueous suspension of flour is heated, the hydrogen bonds weaken in the amylose molecule, water is absorbed, and the starch granules swell. These changes in the amylose molecules lead to changes in their quantity, structure, and interaction with other molecules in the rice flour (Sun et al. 2013).



(a) (b)

4.3. Effect of moisture on amylose content (%) of HMT treated rice flour (a) SAH 329 (b) SAH 177



(a) (b)

Fig 4.4. Effect of temperature on amylose content (%) of HMT treated rice flour (a) SAH 329 (b) SAH 177

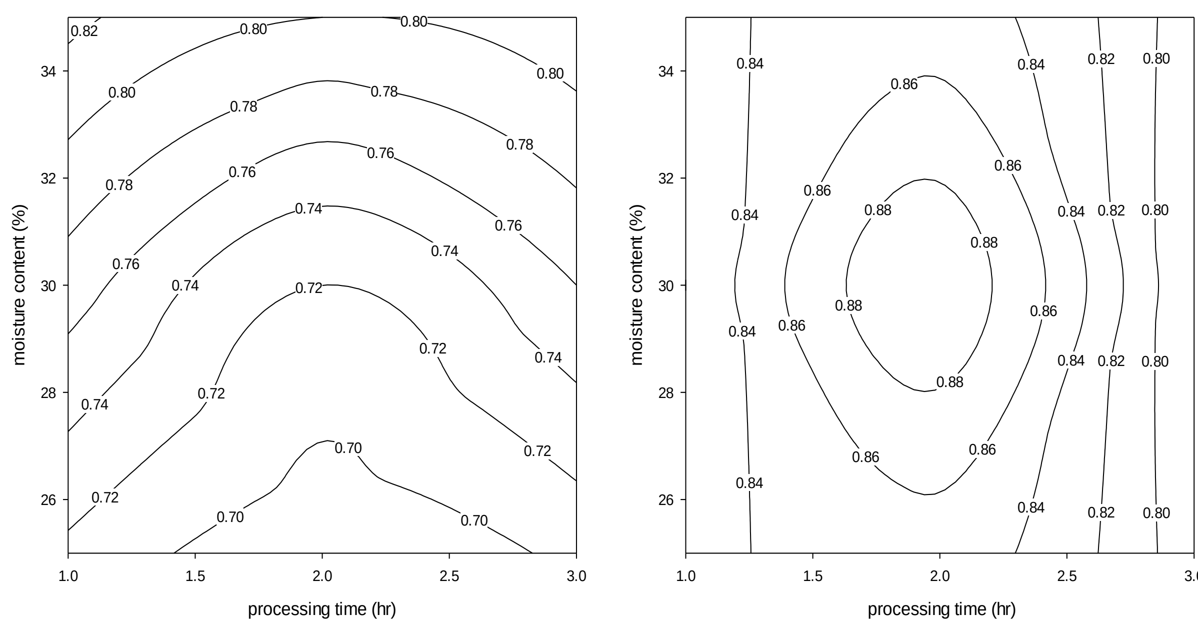
According to Fig 4.4, increasing the processing temperature results in a decrease in the amylose composition. For instance, at 30% moisture content, at least $1 \pm 0.05\%$ decrease in amylose content is seen as temperature increases from 95 to 115 ^oC when samples are processed for an hour. Bao et al. (2001) and Wu et al. (2002) reported a reduction in amylose content during thermal processing. This reduction in amylose content was attributed to the molecular disorganization of both amylose chains and long chains of amylopectin caused by an increase in heating temperature during thermal processing (Chung and Liu, 2010).

The alterations brought about by HMT on the amylose content of the rice flours influence interactions between amylose-amylose, amylose-amylopectin, amylose-lipids and/or amylose-protein molecules. The interactions suppress the mobility of the starch chains in the amorphous regions of the flour's matrix. Consequently, these suppressions depending on the degree, could influence the thermal properties (Qin et al., 2016; Sharma et al., 2015), water binding properties (Majzoobi et al., 2016), oil binding properties (Sharma et al., 2015; Tabara et al., 2015) and rheological properties (Arns et al., 2015; Sharma et al., 2015; Sun et al., 2013) of biological materials. These changes proffer several advantages in the food industry. For instance, HMT has been shown to increase thermal stability, resistance to shear and stability in processed foods (Yoshino et al., 1994).

4.4.2. Impact of HMT treatment on flour hydration properties

4.4.2.1. Water holding capacity

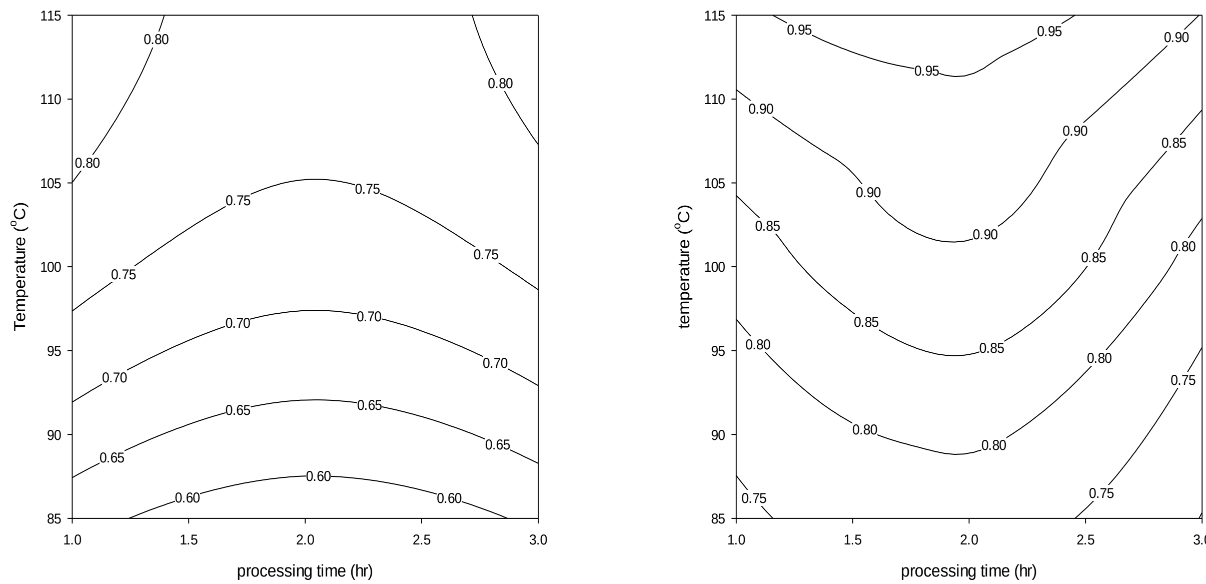
The water holding capacity of rice flour is an important functional property needed in food formulations, most especially food applications requiring dough handling (Iwe et al., 2016). The influence of heat-moisture treatment on the water holding capacity (WHC) of the rice flours is shown in Fig 4.5. The result indicates that the water holding capacity of the SAH 329 rice flour increased with increasing moisture content, while the water holding capacity of SAH 177 showed resistance to changes when moisture content was increased. Protein molecules possess both hydrophilic and hydrophobic nature and therefore can interact with water (Iwe et al., 2016). Changes in the conformational characteristics of proteins during heat-moisture treatment may have influenced the degree of interaction of protein molecules in the rice flour with water.



(a) (b)

Fig 4.5. Effect of moisture on the water holding capacity (g/g) of HMT treated rice flour (a) SAH 329 (b) SAH 177

The result also revealed that increasing processing temperature during heat-moisture treatment led to an increase in the WHC of the selected rice flour as seen in Fig 4.6. The observed variations in the water holding capacities of both flours can be attributed to the influence of temperature during heat-moisture treatment on amylose solubility, loss of starch crystalline structure and protein concentrations (Samuel, 2005).



(a) (b)

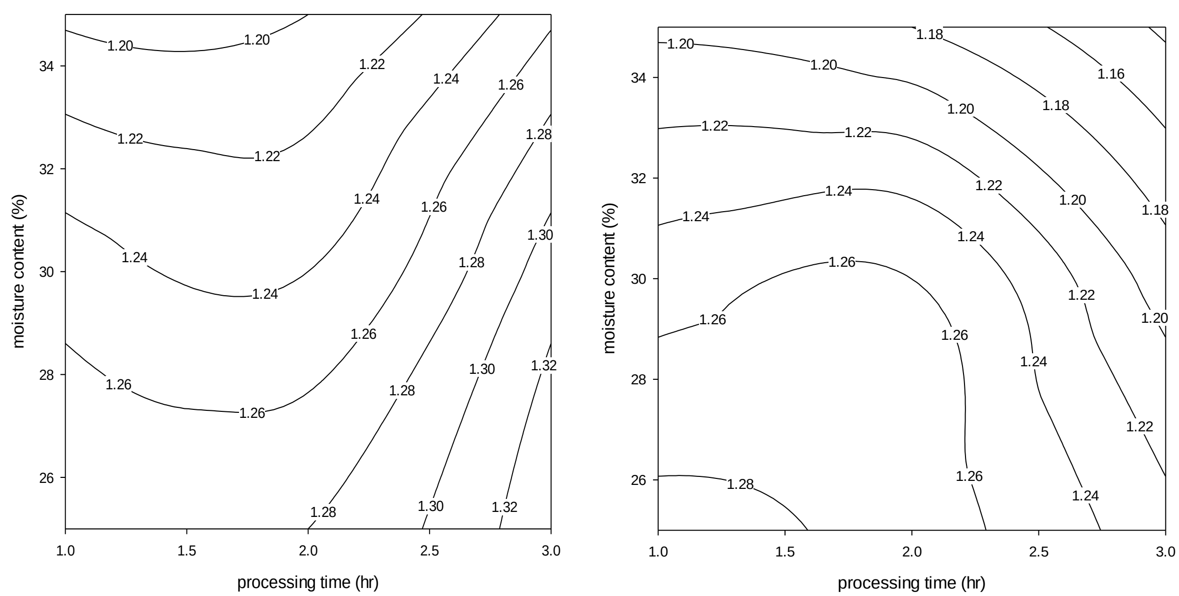
Fig 4.6. Effect of temperature on the water holding capacity of HMT treated rice flour (a) SAH 329 (b) SAH 177

Overall, the results of the heat-moisture treatment suggest an increase in WHC for both flours when the processing temperature and the sample's moisture content was increased. High WHC of flours suggests that the flours can be used in the formulation of foods such as sausage, dough, processed cheese and bakery products (Iwe et al., 2016; Samuel, 2005). Therefore, the rice flours with the highest water holding capacity are best suited for baking applications.

4.4.2.2.Oil holding capacity

Oil holding capacity (OHC) is also another important functional property that defines the mouthfeel and retention of the flavor of flour-based materials (Iwe et al., 2016). The influence of moisture content during heat-moisture treatment on oil absorption capacity (OHC) of both rice flours is presented in Fig 4.7. Fig 4.8 shows the influence of temperature during heat-moisture treatment on the oil absorption capacity of the treated rice flours. On the other hand, for any given processing time, an increase in the processing temperature led to an increase in the oil absorption capacity of the rice flours.

The results of this study suggests that moisture content and temperature during heat-moisture treatment may have led to conformational changes in the protein molecules which in turn may be responsible for the observed oil absorption properties of the treated flours. Menon et al. (2015) related the increase in oil holding capacity to the presence of hydrophobic proteins in the flour. These hydrophobic proteins possess superior lipid binding properties. Oil holding is a physical entrapment process which could be dependent on the non-polar side chains of the proteins as well as the conformational features of the proteins (Chandra, 2013).



(a) (b)

Fig 4.7. Effect of moisture on the oil holding capacity (g/g) of HMT treated rice flour (a) SAH 329 (b) SAH 177

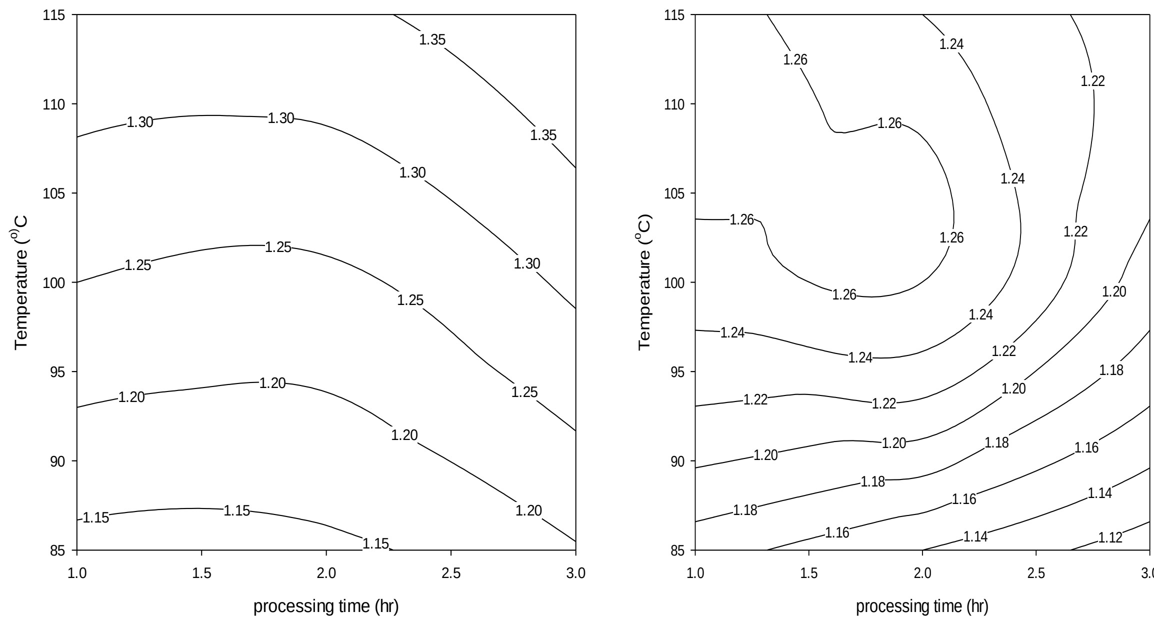
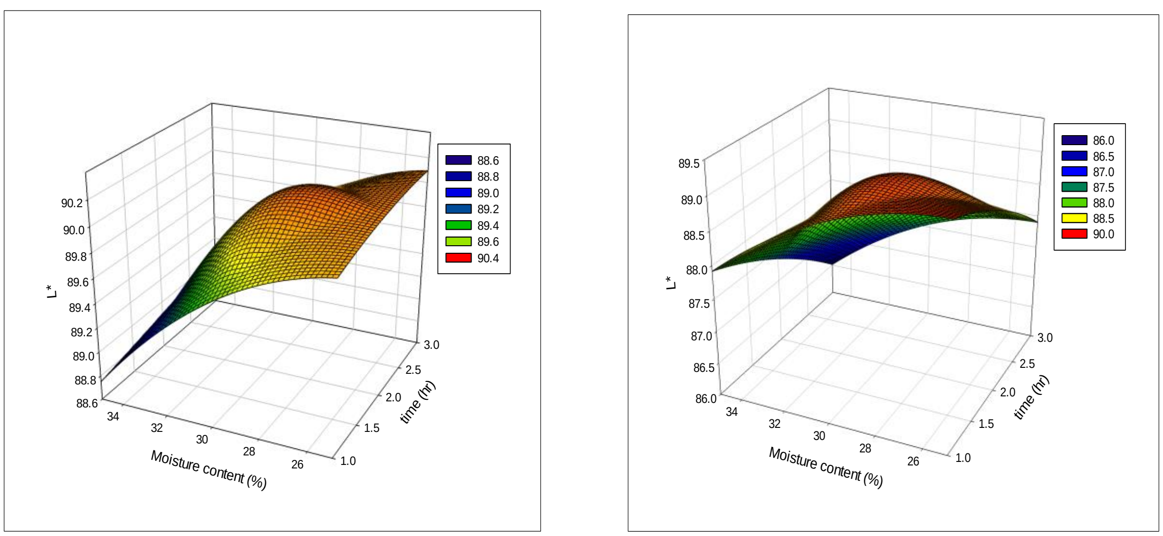


Fig 4.8. Effect of temperature on the oil absorption capacity(g/g) of HMT treated rice flour (a) SAH 329 (b) SAH 177

4.4.3. Color

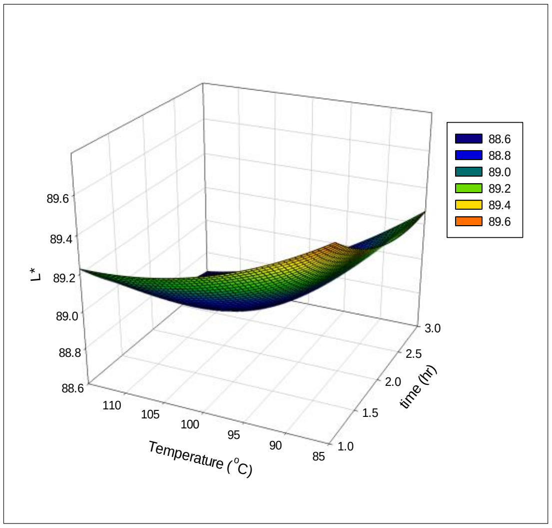
Color influences consumer acceptability of rice flour. Heat-moisture treatment performed on flours of both cultivars reduced the value of L* (degree of whiteness) with increasing moisture content and temperature (Fig 4.9 and 4.10). On the other hand, the value of b* (degree of yellowness) increased with increasing moisture content and temperature (Fig 4.11 and 4.12). Several researchers have observed similar heat-moisture treatment effects on color in various botanical sources (Hoover, 2010; Slam et al., 2002; Tabara et al., 2015). The surface plots suggest that the optimal moisture and processing temperature to achieve the highest L* and lowest b* is estimated to be 30% and 105 ^{o}C, respectively for 2 hrs. This observation implies that the processing conditions during heat-moisture treatment could be adjusted to achieve specific whiteness of rice (L* value) which may be useful for quality control of the product and thermal process. Overall, this study revealed that the L* of heat-moisture treated SAH 177 flour was at least 4% less than the native flour, whereas the L* of SAH 329 was 6.25% less than its native flour. In general, the reduction in the value of L* and increase in the value of b* during heat-moisture treatment is associated with the increased migration of bran pigments and nonenzymatic browning during heat-moisture treatment obtained at the varying processing temperature and moisture content (Chung and Liu, 2010).

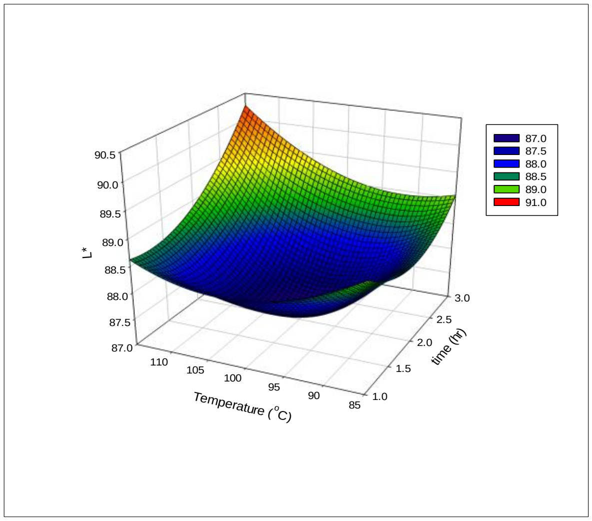
Statistical analysis of the overall color changes of the treated rice flours was significantly influenced by the protein content of the treated SAH 329 (R^2= 0.86, P ≤ 0.05) and SAH 177 (R^2= 0.80, P ≤ 0.05) rice flours. According to Arns et al. (2014), color changes observed in rice materials during the hydrothermal processing, are due to structural alterations occurring in glycosides and peptides bonding, known as Maillard reaction, which produces a dark pigment referred to as melanoidins. The formation of melanoidins involves carbonyl linking of reducing sugars and the amino group of amino acids (especially lysine), proteins or peptides. Increasing moisture content and processing temperature during heat-moisture treatment intensifies the formation of these color forming pigments.



(a) (b)

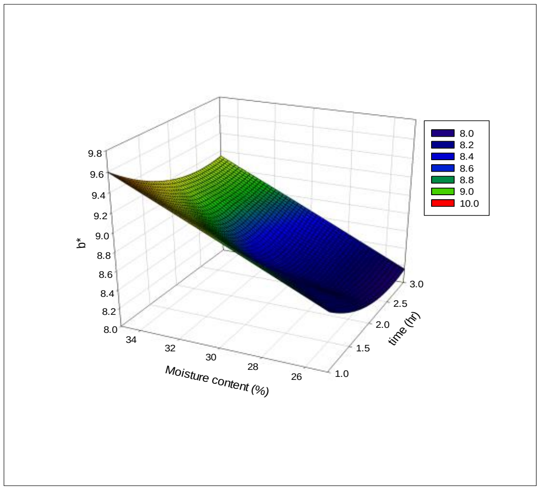
Fig 4.9. Effect of moisture on L* value of HMT treated rice flour (a) SAH 329 (b) SAH 177

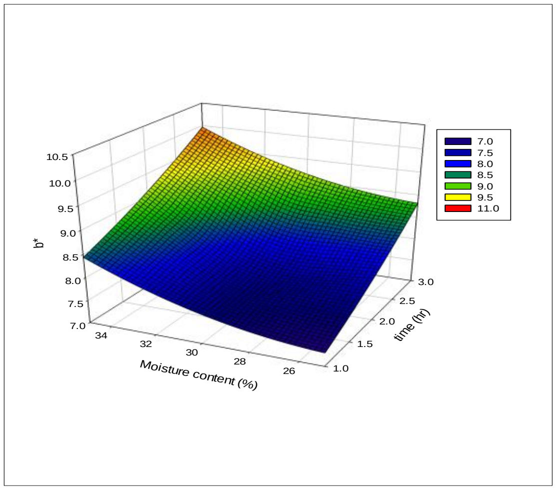




(a) (b)

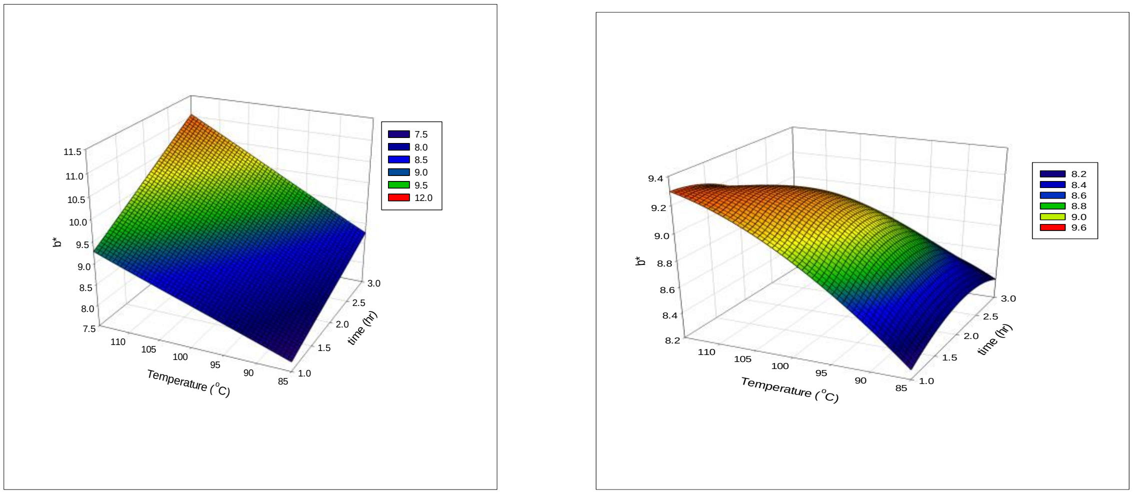
Fig 4.10. Effect of temperature on L* value of HMT treated rice flour (a) SAH 329 (b) SAH 177





(a) (b)

Fig 4.11. Effect of moisture on b* value of HMT treated rice flour (a) SAH 329 (b) SAH 177

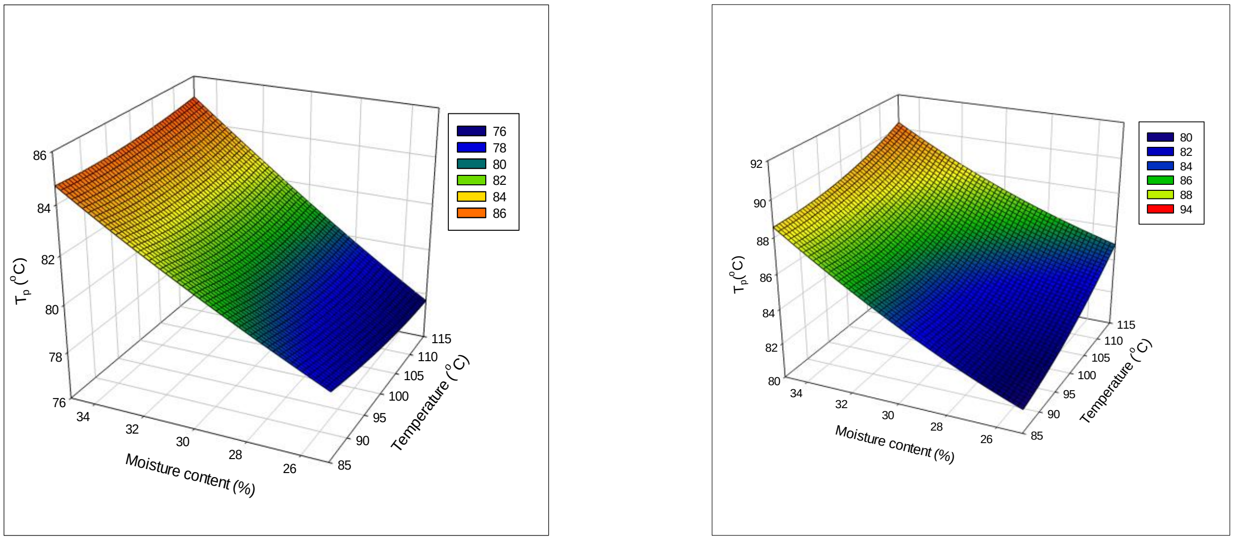


(a) (b)

Fig 4.12. Effect of temperature on b* value of HMT treated rice flour (a) SAH 329 (b) SAH 177

4.4.4. Thermal properties

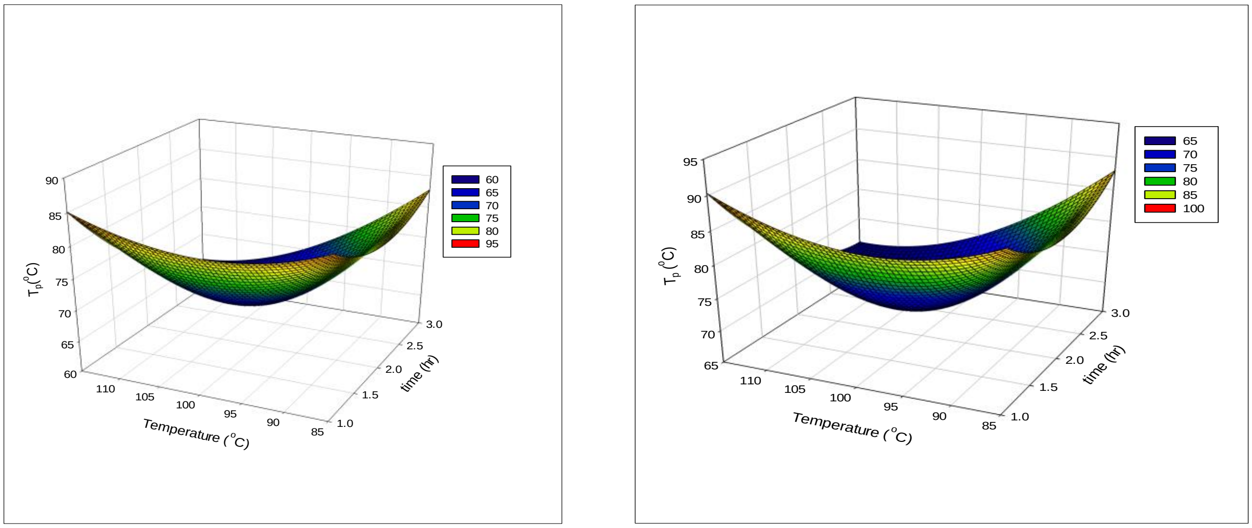
The peak of gelatinization (T_p) is the temperature required to cause a maximum conversion of the crystalline structure in the starch granules of the flour to an amorphous structure. The moisture content and processing temperature at any given processing temperature during heat-moisture treatment significantly ($P \leq 0.05$) influenced the peak gelatinization temperature (T_p) of all the treated rice flours for both cultivars as shown in Fig 4.13. However, moisture content during heat-moisture treatment seems to have a more pronounced effect as far as peak gelatinization temperature is concerned. Increasing moisture generally increases the peak gelatinization temperature. The data seems to suggest that a 1.5°C decrease in gelatinization temperature could be achieved for every percent increase in moisture within the experimental range. According to Adebowale et al. (2009), an increase in peak gelatinization temperature is related to the hydration and swelling of the amorphous regions in the starch granules of the flour during heat-moisture treatment. When the amorphous region swells, it imparts stress on the crystalline networks and polymer chains are broken down from the surface of starch crystallites. After heat-moisture treatment, various interactions such as amylose–amylose and amylose–lipid interactions reduce the mobility of the amorphous section.



(a) (b)

Fig 4.13. Effect of moisture on T_p value of HMT treated rice flour (a) SAH 329 (b) SAH 177

The effect of process temperature on the peak gelatinization temperature is shown in Fig 4.14. The result indicates marginal effect was observed outside the 100 ± 5 °C. For instance, processing at 85 or 115 °C will both result in less than 5°C variation in peak temperature, compared to a 10 - 12 °C variation at 100°C. The minimal effect observed in this temperature ranges can be related to the breakdown of crystalline regions and double helices which in turn restricts the hydration and swelling of the amorphous regions of starch granules. Further evaluation on all the treated rice flours revealed that T_p was significantly influenced by the amylose content of the treated SAH 329 (R^2= 0.80, p≤ 0.05) and SAH 177 (R^2= 0.82, p≤ 0.05) rice flours. The optimal treatment conditions to achieve the lowest gelatinization temperature for all the treated rice flours is found to be 30% moisture content, 100 °C processing temperature, and 2 hrs treatment time. This information suggests that the processing parameters could be manipulated to attain specific thermal properties and maximize the energy requirements required in rice flour processing.

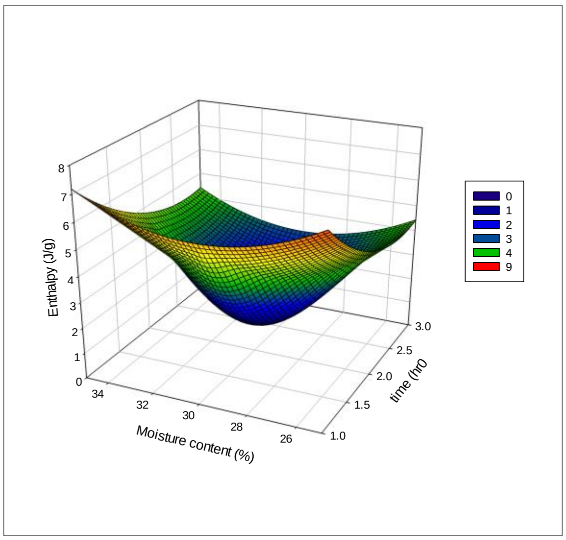


(a) (b)

Fig 4.14. Effect of processing temperature on T_p value of HMT treated rice flour (a) SAH 329 (b) SAH 177

Enthalpy of gelatinization (ΔH) is the quantity of energy required to change the crystalline structure in the starch granules of the flour to an amorphous structure (Zavareze and Dias, 2011). This property reflects the degree of crystallinity of the starch arrangement in the flour granules. The 3D plots shown in Fig 4.15 and 4.16 reveal moisture content and enthalpy of gelatinization were inversely related for all the treated rice flours. The results seem to infer a 1 J/g reduction in the enthalpy of gelatinization for every percentage increase in the moisture content. The optimal moisture content and time to obtain the lowest for ΔH was found to be 30.5% and 2.1 hr for SAH 177 and 30.5% 2.2 hr for SAH 329. Other studies have also reported a reduction in ΔH promoted by HMT in cassava (Gunaratne and Hoover, 2002), potato (Collado and Corke, 1999), yam (Adebowale et al., 2009) and millet (Sharma et al., 2015). It is possible from this study to infer that during heat-moisture treatment, moisture content may have contributed to the disruption of the double helices present in the crystalline and non-crystalline regions of the flour granules. Hormdok and Noomhorm, (2007) suggested that the reduction in ΔH due to hydrothermal treatment may be due to the partial gelatinization of amylose and amylopectin molecules that are less stable during thermal processing.

From Fig 4.15 and 4.16, a decrease in ΔH was observed as processing temperature and moisture content increased. The results demonstrated that the treatments may have caused similar alterations in the internal granular arrangement. Reduction in the gelatinization enthalpy as a result of temperature variations also indicates an incomplete gelatinization of some fewer stable macromolecules (such as amylose, amylopectin and protein) in the rice flours. Chung et al. (2010) suggested that an increase in temperature may also increase the mobility of the double helix, forming a crystalline structure. This may lead to the breakdown of the hydrogen bonds attached to adjacent double helices, which is the reason for the reduction in the enthalpy value of the heat-moisture treated flours compared with native flour.



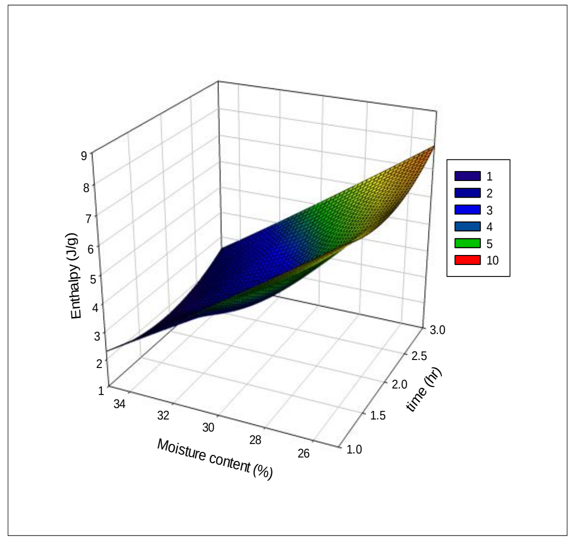


Fig 4.15. Effect of moisture content on ΔH value of HMT treated rice flour (a) SAH 329 (b) SAH 177

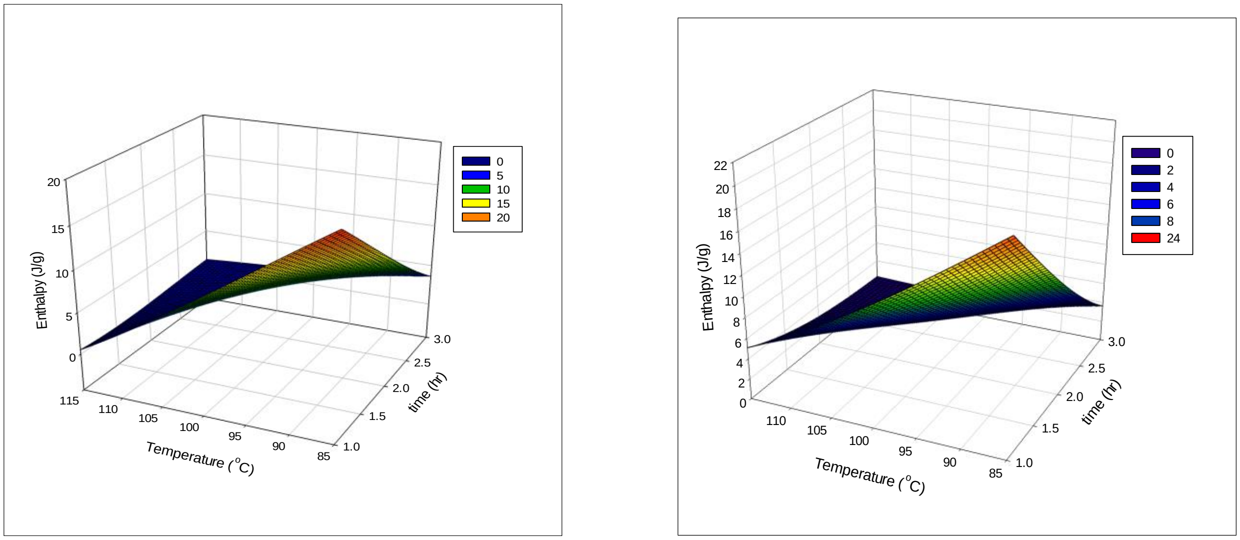


Fig 4.16. Effect of processing temperature on ΔH value of HMT treated rice flour (a) SAH 329 (b) SAH 177

4.4.5. Dynamic rheological properties

Changes in G' (storage modulus) and G" (loss modulus) as a function of variable stress and moisture content during HMT are presented in Fig 4.17. The corresponding values of G', G", loss factor, yield stress are presented in Table 4.2. Within the linear viscoelastic range (LVE), rice flour paste revealed the G' > G", therefore indicating gel-like characteristics. Moisture content and processing temperature during HMT significantly ($P \leq 0.05$) influenced the structural strength (G') of all the studied rice flour paste. The influence of moisture content on the storage modulus during heat-moisture treatment followed the order, native SAH 329 (63.20 Pa) > HMT-25% (33.95 Pa) > HMT-30% (5.96 Pa) > HMT-35% (5.70 Pa). The same trend of G' reduction was observed for SAH 177. An increase in processing temperature was shown to lead to an increase in G' values of both cultivar flours. The lower G' values observed in this study may be attributed to the deformability of starch granules in the flour matrix or reduced elasticity of the continuous phase of the rice flour paste. Moisture content and temperature during heat-moisture treatment also significantly influenced ($P \leq 0.05$) the loss factor (G"/ G') of all the studied rice flours. Maximum loss factor observed because of moisture influence on SAH 329 and SAH 177 was 0.44 and 0.94 respectively. While maximum loss factor values for SAH 329 and SAH 177 was 1.30 and 0.80

respectively when temperature influence was studied. The G' and G'' curves for native and HMT-25% samples increased sharply after LVE range, and therefore had high spreadability. The steady increase of G' curve irrespective of the influence (moisture content and temperature) suggest that these flour paste had resilient characteristics. The G'' curve of the SAH 329 and SAH 177 showed an increase as moisture content and temperature content was increased. This observation indicates good network formation in the paste. The yield stress of the heat-moisture treated flour samples was significantly lower than native rice flours for both cultivars and they further decreased significantly ($P \leq 0.05$) as the moisture content and processing temperature increased (Table 4.2). Moisture studies on SAH 329 and SAH 177 samples revealed that the lowest yield stress was 3.16 and 3.35 Pa, respectively. While temperature studies revealed that the lowest yield stress for SAH 329 and SAH 177 rice flour samples was 0.80 and 0.63 Pa, respectively. The curves of G' and G'' for both SAH 329 and SAH 177 samples rose steadily, representing a non-homogenous deformation and elasticity. Hard-paste is important in applications that require more force for fluidity and lead to non-homogenous mixing. The paste structure of HMT-25%, HMT-30 and HMT-35% did not change to liquid phase with a sharp fracture edge, suggesting a less balanced ratio of the elastic and viscous portion.

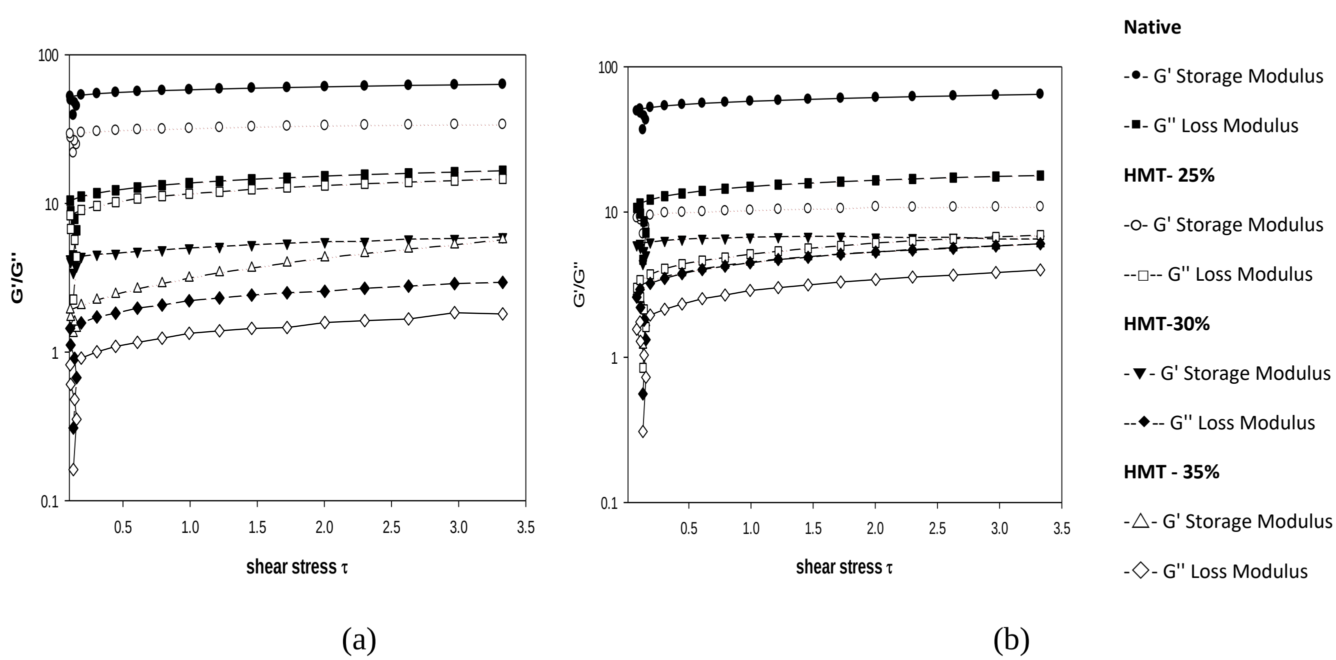


Fig 4.17. Changes in G' (storage modulus) and G'' (loss modulus) as a function of variable stress and moisture content for native and HMT flours for (a) SAH 329 and (b) SAH 177

Table 4.2. Rheological properties of native and heat-moisture treated SAH 329 and SAH 177 rice flour

Treatment	G' (Pa)		G" (Pa)		Loss factor (G'/G")		Yield stress (Pa)	
	SAH 329	SAH 177	SAH 329	SAH 177	SAH 329	SAH 177	SAH 329	SAH 177
Native	$63.20 \pm 0.02_{a}$	$64.51 \pm 0.03_{d}$	$16.58 \pm 0.01_{a}$	$17.84 \pm 0.02_{c}$	$0.26 \pm 0.01_{a}$	$0.28 \pm 0.01_{d}$	$22.58 \pm 0.01_{a}$	$22.81 \pm 0.02_{b}$
HMT-25%	$33.95 \pm 0.01_{b}$	$10.84 \pm 0.01_{c}$	$14.61 \pm 0.04_{b}$	$6.92 \pm 0.01_{d}$	$0.43 \pm 0.02_{bc}$	$0.77 \pm 0.02_{c}$	$18.43 \pm 0.02_{c}$	$11.04 \pm 0.02_{a}$
HMT-30%	$5.96 \pm 0.03_{c}$	$6.51 \pm 0.003_{b}$	$2.94 \pm 0.01_{c}$	$6.04 \pm 0.00_{b}$	$0.44 \pm 0.02_{b}$	$0.92 \pm 0.02_{d}$	$15.13 \pm 0.01_{b}$	$8.91 \pm 0.01_{c}$
HMT-35%	$5.70 \pm 0.01_{d}$	$6.04 \pm 0.001_{a}$	$1.80 \pm 0.03_{d}$	$3.99 \pm 0.001_{a}$	$0.32 \pm 0.01_{c}$	$0.94 \pm 0.02_{d}$	$3.16 \pm 0.24_{d}$	$3.35 \pm 0.02_{d}$
HMT-85ºC	$10.83 \pm 0.01_{g}$	$6.24 \pm 0.01_{h}$	$14.11 \pm 0.02_{g}$	$9.56 \pm 0.02_{g}$	$1.30 \pm 0.02_{g}$	$0.80 \pm 0.01_{i}$	$8.42 \pm 0.001_{g}$	$4.06 \pm 0.01_{g}$
HMT-100ºC	$8.65 \pm 0.02_{h}$	$12.5 \pm 0.01_{g}$	$9.75 \pm 0.02_{i}$	$6.75 \pm 0.02_{h}$	$0.95 \pm 0.01_{h}$	$0.75 \pm 0.02_{h}$	$6.75 \pm 0.01_{h}$	$3.50 \pm 0.01_{h}$
HMT-115ºC	$6.819 \pm 0.01_{i}$	$14.99 \pm 0.02_{j}$	$5.47 \pm 0.02_{h}$	$4.46 \pm 0.02_{i}$	$0.80 \pm 0.001_{i}$	$0.63 \pm 0.01_{g}$	$5.73 \pm 0.02_{i}$	$0.10 \pm 0.01_{i}$

HMT heat-moisture treatment, G' Storage modulus, G" loss modulus, Yield stress (measured at constant frequency 62.8 rad/s), mean values (n=3) with the same superscript letters within a column and treatment are not significantly different

4.4.6. Predicting the heat-moisture treatment effect

Linear and multiple regressions were carried out on the impact of heat-moisture treatment parameters to enable the prediction of the thermal processing influences (Table 4.3). The models derived can be used for predicting the physicochemical, functional and thermal properties. Temperature, moisture content and time were utilized as the parameters for evaluation. It is important to note that this predictive model is applicable for non-waxy rice flours with amylose content with ranges of 29.95 - 30.85 %. Regression analysis in this study was carried out using stepwise backward selection due to many variable interactions. The predictive model with the best fit to the variation was chosen over other models explaining rhat residual variations were also included until no other predictive model was significant. The regression models in this study provide predictive models that are statistically significant. Akaike Information Criterion (AIC) was utilized to prevent overfitting the regression model with predictive models which are not capable of explaining the changes in the model. In addition, adjusted R-square (R^2) which provides insight into the potential of a regression model with different predictive models was selected over R-square, since R-square increases each time a variable is added or subtracted to a model which could be misleading probably due to luck in the data. Higher adjusted R-square and lower AIC were chosen as an improvement. Temperature, time and moisture content were the factors that significantly provide insight into the changes observed during heat-moisture treatment. All the heat-moisture treatment predictive models had 34 observations. For protein content estimation, temperature, time and moisture Eq. (2) had a high adjusted R^2 and the least AIC which provides better insight into the variation in data set than temperature and moisture content Eq.(1). The model fit (Eq. (1)) can be improved by excluding the moisture content of the rice flour materials during heat-moisture treatment (Eq. (2)). This was the lowest significance observed in the predictive models derived in this study. The low significance suggests that time, temperature and moisture content may not clearly explain the variations in amylose content during heat-moisture treatment. The predictive model fit for Eqs. (5), (7), (9), (11), (13) and (16) had high adjusted R^2 and the least AIC thus explains the variation in data sets in determining the WAC, OAC, L*, b*, T_p and ΔH properties. The results of these predictive models suggest that time, temperature and moisture content contributed to the studied responses.

Table 4.3. Regression models of Heat-moisture treatment impact.

Response	Predictive model	Model number	Number of observations	Adjusted R^2	AIC
Protein content	7.95 + 0.02 x A - 0.06 x C +(B -2) x [(C -30)x 0.03]	(1)	34	0.7843	10.48
	7.95 + 0.023 x A -0.06 x C	(2)	34	0.7249	10.02
Amylose content	40.84 - 0.12 x A -0.69 x B - 0.01 x C	(3)	34	0.6001	68.90
	39.05 - 0.12 x A	(4)	34	0.5201	63.64
WAC	0.13 + 0.006 x A + (B -2)[(A - 100) x 0.002]+(B -2)x[(C - 30) x 0.011]	(5)	34	0.7801	37.82
	0.13 + 0.01 x A +(B -2) x [(C -30) x 0.011]	(6)	34	0.7336	39.33
OAC	1.17 + 0.004 x A - 0.001 x C + (B - 2) x [(C -30) x 0.005]	(7)	34	0.7827	8.80
	1.17 - 0.001 x C + 0.004 x C	(8)	34	0.7201	9.26
L*	92.35 - 0.001 x C + [(A - 100) (C -30) x 0.01] + [(B - 2) x (A - 100) x -0.03] + (B -2) x (A-100) x [(C -300 x 0.005)]	(9)	34	0.7674	31.46
	92.35 - 0.09 + (A - 100) x [(C - 30) x -0.001] + (B -2) x [(A - 100) x -0.03]	(10)	34	0.6264	33.71
b*	3.50 + 0.03 x A + 0.07 x C + (B- 2) x [(A -100) x 0.02] + (A - 100) x [(C -30) x 0.01]	(11)	34	0.7101	38.50
	3.50 + 0.03 x A + 0.07 x C + (A - 100) x [(C -30) x 0.01]	(12)	34	0.6001	38.55
T_p	99.07 -4.01 x B - 0.28 x A + 0.36 x C + (B - 2) x [(A - 99) x - 0.30]	(13)	34	0.7001	103.06
	109.89 - 4.01 x B - 0.26 x A - (B - 2) x [(A-99) x -0.30]	(14)	34	0.6048	104.86
∆H	7.40 (B - 2) + [(A- 101) x (C -30) x -0.01)]- 2.02 x A + (A - 2.09) x [(C -29.56) x .33] + A - 101.4 x 9 [(C - 29.55) x 0.01]	(15)	34	0.8616	43.27
	8.40 - 2.40 x B + (B -2.09) x [(C -29.55) x 0.27]	(16)	34	0.7532	51.3

A: heating temperature, B: heating time, C: moisture content

4.5. Conclusion

The impact of heat-moisture treatment on the physicochemical, thermal and rheological properties of non-waxy flours from SAH 329 and SAH 177 rice cultivars was evaluated. Through this study, it was possible to verify that depending on the temperature, time and moisture content, various responses could be obtained for the studied properties of the heat-moisture treated flours. Increasing the moisture content during heat-moisture treatment revealed reductions in the protein contents, amylose contents, oil absorption capacities of the flours while increasing the processing temperature during heat-moisture modification led to an increase in the water absorption properties of the flours. Our studies also revealed a decrease in enthalpy of gelatinization as processing temperature increased during heat-moisture treatment. Reduction in the gelatinization enthalpy suggests incomplete gelatinization in the rice flours during heat-moisture treatment. Overall, desirable modified rice flour could be achieved by fine-tuning the heat-moisture process to enable the production of flours with unique functional properties.

CONNECTING TEXT

In chapter 4, the impact of heat-moisture treatment on SAH 329 and SAH 177 was evaluated. Heat-moisture treatment significantly influenced the amylose composition of these rice flours. It is therefore imperative to evaluate how this impact will influence the utilization of heat-moisture treated flours in food applications. In chapter 5, heat-moisture treated rice flours were used to study the influence of the modification treatments on rice pudding.

5. Rheological and textural properties of rice puddings made from heat-moisture treated flours.

5.1. Abstract

The aim of this work was to study the impact of heat-moisture treated rice flours on the properties of puddings. Flours from SAH 177 and SAH 329 rice cultivars were adjusted to three different moisture levels (25, 30 and 35%) and heated at 100 and 115°C for 1 and 2 hrs. The puddings made from the control and heat-moisture treated rice flours were analyzed for their rheological, textural and color properties at day 0 to day 14. All the puddings displayed fragile gel-like structures with G' > G" and with tan $\delta < 1$ at all studied frequencies (0.1 – 100 Hz). Textural studies revealed that heat-moisture treated flours produced less firm textured puddings than the control. The combined effect of the chemical composition of the flours and heat-moisture treatment resulted in varying rheological, textural and color properties that could be utilized in producing puddings with different characteristics.

Keywords. Rice flour, Rice pudding, Rheology, Texture, Functional food.

5.2. Introduction

Rice is one of the most consumed cereal for over half of the world's population (Lantin, 1999). It can be consumed as a cooked whole grain or processed into flours which can be utilized for several domestic and industrial purposes. A good example of an application of rice flour is in the production of rice-based pudding. Rice pudding is a milk-based flour paste with a semisolid food texture (Lim et al., 2006). Rice pudding can be described as a suspension of deformable particles (swollen granules of rice grains or flour) dispersed in a continuous medium composed of milk proteins (Alamprese et al., 2011). The formulation of this dessert is generally milk, rice (grain or flour), sugar and eggs. Some consumers do not like the rough texture of rice grains in a rice pudding, thus encouraging the utilization of flour in the production of the desert. The flour plays a major role in the suspension as it imparts body and mouthfeel to the product. In addition, the utilization of modified flours in rice-based puddings may remove the unwelcoming rough texture.

Rice flour has previously been utilized as substitution agent in several food commodities. Wheat flour in food products such as pasta, noodles, cake, and bread was replaced by rice flour (Jeong et al., 2016; Phongthai et al., 2017; Wang et al., 2016a; Wang et al., 2016b). Non-waxy flour has been reported to produce noodles with a firmer texture, reduced cooking loss and greater elastic properties (Jeong et al., 2016). In addition, Ha et al. (2007) observed that cakes, cookies, and bread made from non-waxy rice flour exhibited similar rheological properties to that made from waxy rice flours. However, there is no study on the utilization of non-waxy flour in the formulation of rice pudding.

Recently, the application of different milk substitutes such as rice drink, skimmed milk, soy drink, and water has been examined in the formulation of pudding. Alamprese et al. (2011) studied the pasting, rheological and textural properties of commercial pudding powders dissolved in rice drink, light soy drink, partially skimmed milk and water. Nunes et al. (2003) carried out investigations on the rheological and textural properties of vegetable-based milk puddings. Both studies agree that the various milk substitutes resulted in different rheological and textural attributes of the final products. Recently, Thaiudom and Pracham, (2016) used different waxy jasmine rice flours with different rice protein contents in producing pudding. The impact of mixed stabilizers at different concentrations on the textural and rheological properties of the puddings was also investigated.

Presently, the food industry is evolving towards developing novel products by using modified food ingredients that confer several functional benefits. Heat-moisture treated (HMT) flours are flours that have been physically modified under high temperature (84 -120°C), restricted moisture content (less than 35%) and time (15 min to 16 hours) (Khamthong et al., 2012). The major molecular components (amylose and amylopectin) in the flours are structurally altered, resulting in improved binding with solvents such as water. These alterations lead to a decrease of the flour hardness and thermal properties but contribute to an increase in their swelling power. With heat-moisture treatment, flours are modified with heat and moisture, leading to the production of pre-gelatinized flours. These pre-gelatinized flours are both hydrophilic and hydrophobic in nature, thus resulting in a strong hydrophobic interaction between amylose and amylopectin in the starch framework and greater hydrophilic bonding with solvents such as water (Ortega-Ojeda et al., 2005). These interactions could improve in the resistance and gel-forming nature (Puncha-arnon et al., 2013) of native rice flours, therefore increasing their potential applications in the food and non-food industries (for instance, in the production of paper coating agents and cosmetic products).

The aim of this paper is to evaluate the influence of heat-moisture treatment of non-waxy rice flours on their color, pudding rheology, and texture.

5.3.Materials and methods

5.3.1. Materials

Fresh eggs, sugar and rice milk used in this study were purchased from a local supermarket in Montreal, Canada. Two rice cultivars, SAHEL 177 and 329 cultivars, were purchased from a local market in Dakar, Senegal.

5.3.2. Production of flour

Rice grains were milled using a coffee grinder (SUMEET Multi Grind, India), passed through a 250µm screen (Number 60 sieve), packed and stored in airtight plastic bags for further use.

5.3.3. Preparation of heat-moisture treated rice flour

Flour samples (150 g) were adjusted to 25, 30 and 35% moisture levels and placed in screw-capped airtight aluminum containers and equilibrated at room temperature for 12 hours. Samples were heated at 100 and 115°C for 1 and 2 hours. After heat-moisture treatment (HMT), the flour samples were kept at 4°C in a refrigerator for further analysis. The flour was then analyzed for its chemical composition. The crude protein content of flour samples was analyzed using the VELP Nitrogen Analyzer (NDA 701, Usmate, Italy). About 100 mg of rice flour samples was introduced into the Analyzer's combustion chamber (1300°C). The amount of protein was calculated by multiplying the nitrogen content produced by the combustion process by a factor of 6.25. Amylose content of flour samples was analyzed using the iodine-binding colorimetric method of Juliano (1971). The absorbance of blue color from the binding was measured with a spectrophotometer at 620 nm. Standard curves prepared from potato amylose were used to estimate the amylose content of the flours.

5.3.4. Rice pudding preparation

For pudding preparations, slight modifications were made to the methods of Alamprese et al., (2011). Formulation for the pudding includes 76 g rice milk, 3 g sugar, 12 g egg, and 9 g flour. All the ingredients were mixed and heated to 85°C for 20 min. The puddings were cooled to room temperature (25°C) and then transferred into straight-sided glass jars with 3 cm diameter and 8 cm height. The samples were later stored in a refrigerator (4.5 ± 0.01°C) prior to further analysis. The coding of the flour samples used in this study was in the format of: Sample name - HMT processing time (hrs) – HMT processing temperature (^{o}C) - Moisture content (%) during HMT. The flours used to make the pudding in this study were: SAH 177 control, SAH 329 control, SAHEL 177 1-115-25, SAHEL 177 1-115-35, SAHEL 177 2-100-30, SAHEL 177 2-100-35, SAH 329 1-115-25, SAHEL 329 1-115-35, SAHEL 329 2-100-30 and SAHEL 329 2-100-35.

5.3.5. Rheological properties

The rheological behavior of rice puddings was studied using a rheometer (AR 2000 rheometer, TA Instruments, New Castle, USA). The rheological properties of the puddings were obtained using a 4 cm parallel plate at gap 500 µm. Dynamic properties of the puddings were obtained from frequency sweeps range of 0.1 Hz – 100 Hz at 0.1% constant strain. Storage modulus (G′), loss modulus (G″) and loss tangent (tan δ) of puddings were measured from 0 to 14 days with a temperature platform of 25°C.

5.3.6. Textural properties

Textural properties (hardness, gumminess, and chewiness) of rice puddings were evaluated using a texture analyzer (TA. HD. Plus, Stable Micro System, Godalming, UK). Textural studies were carried out at ambient conditions (25°C) with a 25 mm diameter probe, 60% strain of penetration, 5s of waiting time, and 2 mm^{-1} of test speed. The textural parameters of the rice pudding samples were evaluated at different days from day 0 to day 14.

5.3.7. Color properties

The color properties of the rice puddings were evaluated using a Konica Minolta spectrophotometer (model CM-3500d, Osaka, Japan), following the method of Mba et al. (2015). The instrument was calibrated before analysis with a black plate followed by a white ceramic plate. The color values were expressed as L*(brightness), a* (redness) and b* (yellowness). The average of five scans for each pudding sample was used to determine the L* a* b* properties. Furthermore, the total color changes (ΔE) were evaluated using Equation 1.

$$\text{Total color change } (\Delta E) = \left[(L^* - L_{hmt})^2 + (a^* - a_{hmt})^2 + (b^* - b_{hmt})^2\right]^{0.5} \qquad (13)$$

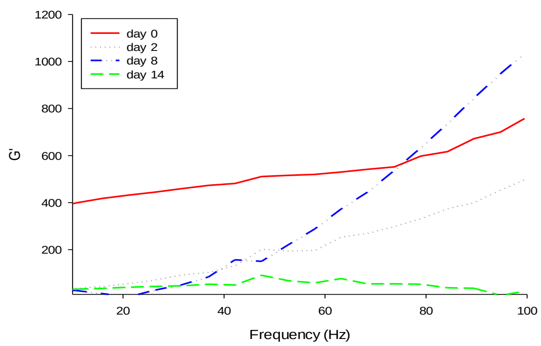
Where L*, a* and b* values are L a b values of control samples; L_{hmt} a_{hmt} and b_{hmt} values are L a b values of heat modified samples.

Color analysis of the rice pudding samples was conducted from day 0 to day 14.

5.4. Results and discussions

5.4.1. Rheological properties of rice puddings

All controls and heat moisture-treated rice puddings exhibited soft gel structure with G' > G" (Figs. 5.1 and 5.2) and with tan $\delta < 1$ (Fig. 5.3) at all studied frequencies. This observation was consistent with the study of Alamprese and Mariotti (2011) and Thaiudom and Pracham (2016). G' and G" of SAH 177 were higher than those of SAH 329 on the first day of storage (Figs. 5.1 and 5.2). The tan δ of SAH 329 was greater than that of SAH 177 on day 14 (Fig. 5.3).



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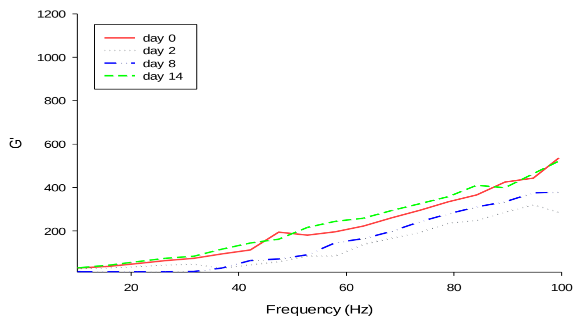

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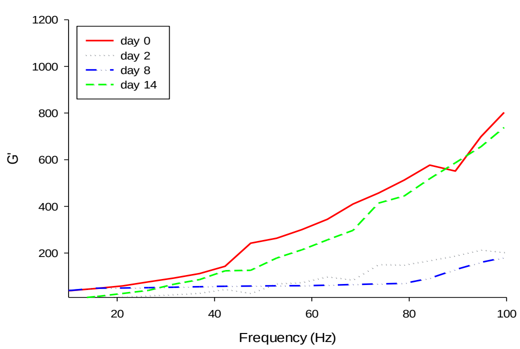

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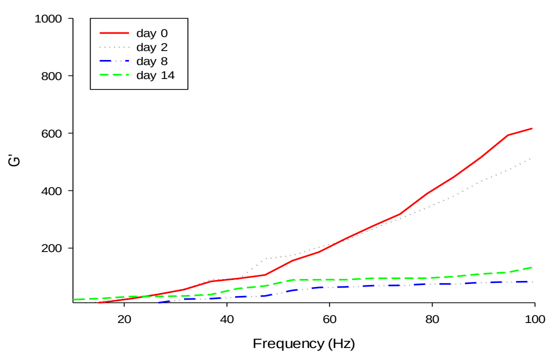

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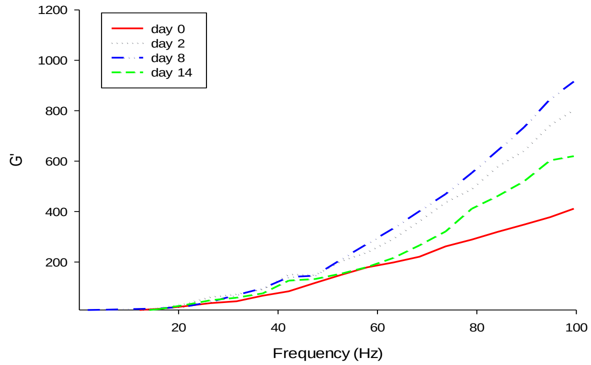

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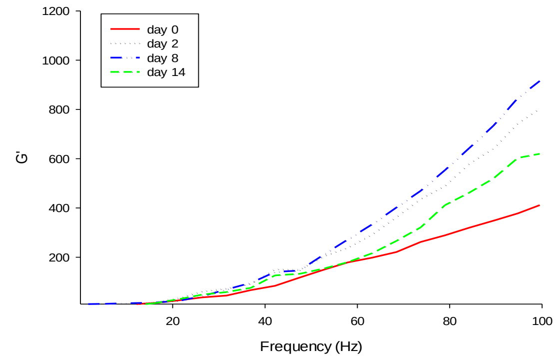

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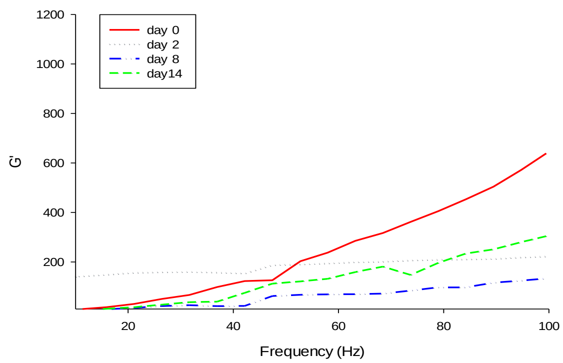

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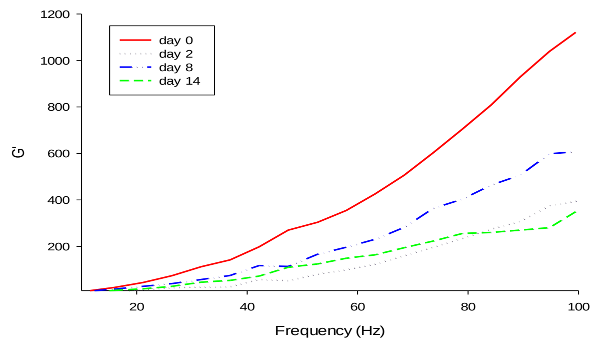

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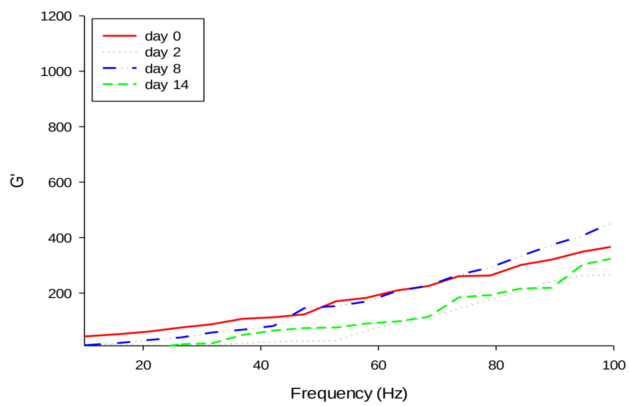
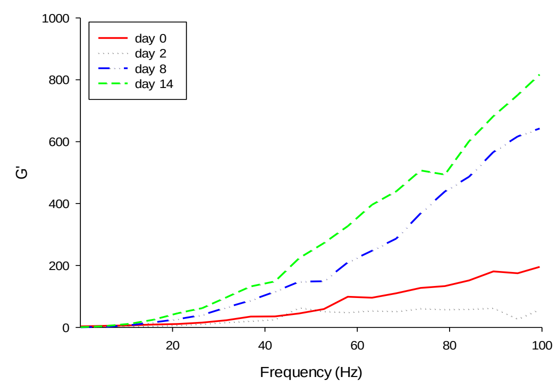


(i)



(j)

Fig. 5.1. G' of puddings made from (a) SAH 177 (b) SAH 329 (c) SAH 177 1-115-25 (d)SAH 329 1-115-25 (e) SAH 177 1-115-35 (f) SAH 329 1-115-35 (g) SAH 177 2-100-30 (h) SAH 329 2-100-30 (i) SAH 177 2-100-35 (j) SAH 329 2-100-35 at day 0, 2, 8 and 14.

Looking at the impact of heat-moisture treated rice flours on rheological properties of the puddings (Figs 5.1 and 5.2), the G' and G" ranges of the pudding samples made from the heat-moisture treated flours were (0.44- 1121) and (0.18 - 79.50), respectively, were comparable to the G' and G" ranges of the control puddings (1.70 - 1034) and (0.15 - 101.50), respectively. Typically, heat-moisture treatment leads to partial gelatinization of the starch molecules (Heidi and Delcour, 1998). The various flow behavior observed in this study may be because of the strong hydrophobic and hydrophilic interactions between partially gelatinized starches in the flour and other macromolecules in the pudding system. Gelatinization typically leads to re-orientation of starch molecules which subsequently leads to addition or losses in the polar and non-polar regions of starch molecules. Changes in the polar and non-polar arrangement of gelatinized starch molecules may influence the interaction and formation of complexes between starch molecules and other macromolecules such as lipids and proteins, thus leading altering the flow behavior of puddings made from the modified flour. This is remarkable due to the impact heat-moisture treatment had on the chemical composition of the rice flours, more specifically its amylose content (Table 5.1).

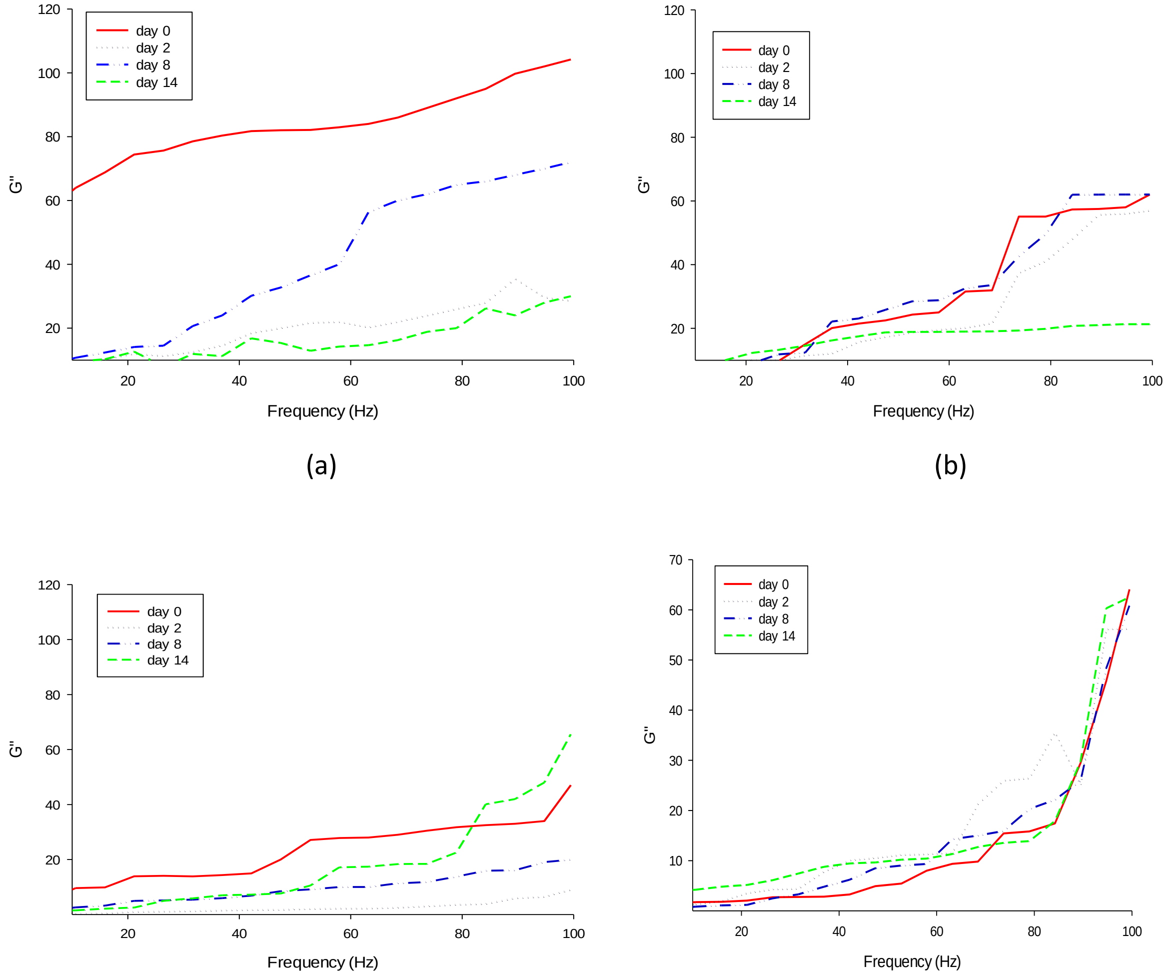
Table 5.1. Chemical composition of rice flour samples used in producing the rice puddings

Sample	Protein (%)	Amylose (%)	Moisture (%)
SAH 177	8.67 ± 0.12a	29.95 ± 0.05j	15.00 ± 0.04a
SAH 177 1-115-25	8.49 ± 0.02b	23.45 ± 0.06i	15.00 ± 0.00a
SAH 177 1-115-35	7.78 ± 0.12c	22.99 ± 0.05h	15.00 ± 0.03a
SAH 177 2-100-30	8.43 ± 0.10d	24.05 ± 0.12g	15.00 ± 0.02a
SAH 177 2-100-35	7.98 ± 0.26e	23.30 ± 0.06f	15.00 ± 0.00a
SAH 329	9.24 ± 0.06f	30.85 ± 0.10e	15.00 ± 0.02a
SAH 329 1-115-25	8.79 ± 0.08g	26.75 ± 0.05d	14.70 ± 0.03b
SAH 329 1-115-35	7.96 ± 0.12h	24.75 ± 0.09c	15.00 ± 0.00a
SAH 329 2-100-30	8.43 ± 0.18d	28.95 ± 0.02b	15.00 ± 0.00a
SAH 329 2-100-35	8.01 ± 0.13i	25.09 ± 0.01a	15.00 ± 0.02a

Means within each column with same letters are not significantly different ($P<0.05$). Data are means ± SD

The presence of partially gelatinized starch complexes hinders water penetration into the rice and its swelling. Furthermore, the longer chain amylopectin molecules have also been confirmed to play a major role in the rheological behavior of starch-based samples (Philpot et al., 2006).

Amylopectin molecules have been shown to restrict swelling of amylose molecules, thus resulting in a reduction of leached out amylose molecules (Philpot et al., 2006; Wang et al., 2015). This finding suggests a decrease in network formation and loss of non-homogeneity in the rice puddings with storage time.



(a) (b)

(c) (d)

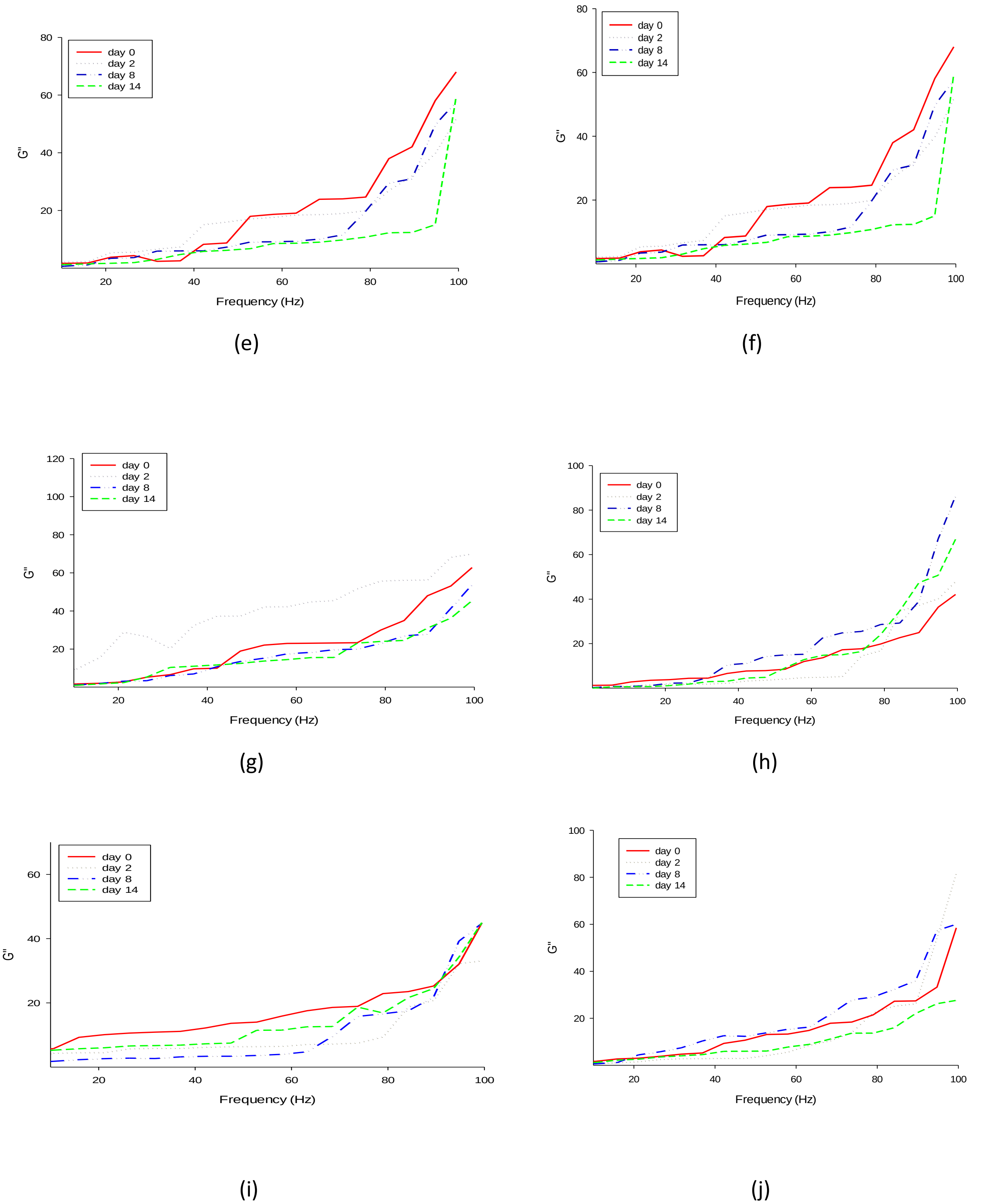
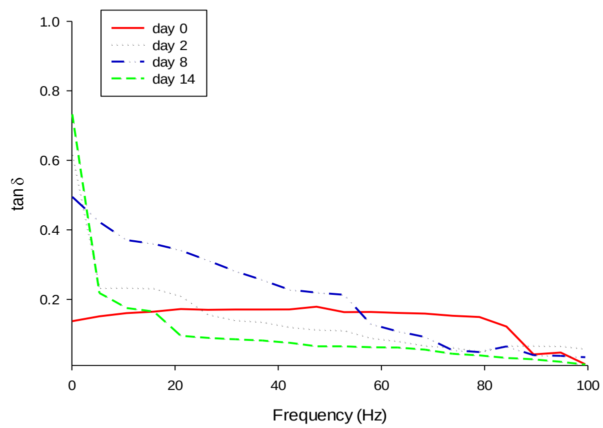
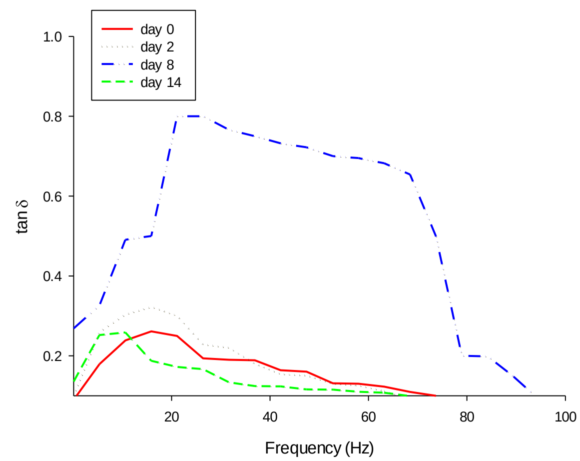


Fig 5.2. G'' of puddings made from (a) SAH 177 (b) SAH 329 (c) SAH 177 1-115-25 (d) SAH 329 1-115-25 (e) SAH 177 1-115-35 (f) SAH 329 1-115-35 (g) SAH 177 2-100-30 (h) SAH 329 2-100-30 (i) SAH 177 2-100-35 (j) SAH 329 2-100-35 at day 0, 2, 8 and 14.


day 0
day 2
day 8
day 14
tan δ
Frequency (Hz)

(a)


day 0
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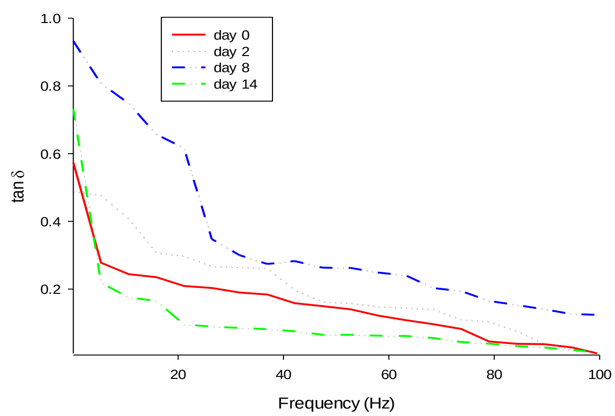
Frequency (Hz)

(b)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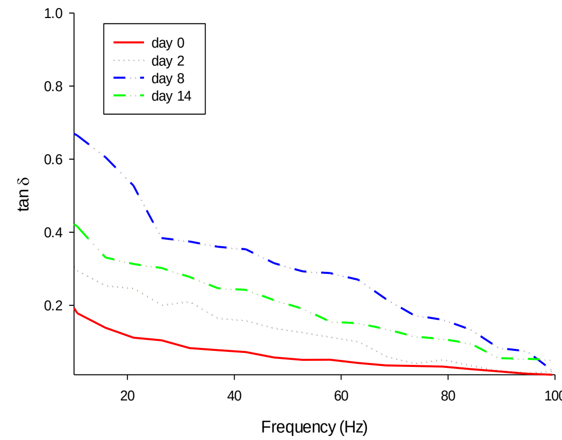
tan δ
Frequency (Hz)

(c)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Frequency (Hz)

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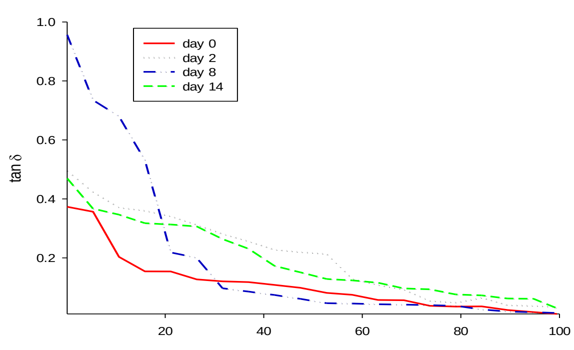
day 0
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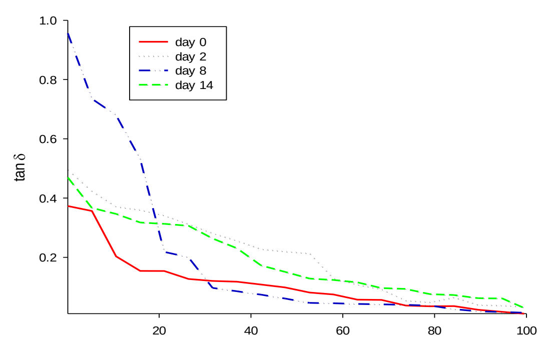
Frequency (Hz)

(e)


day 0
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tan δ
Frequency (Hz)

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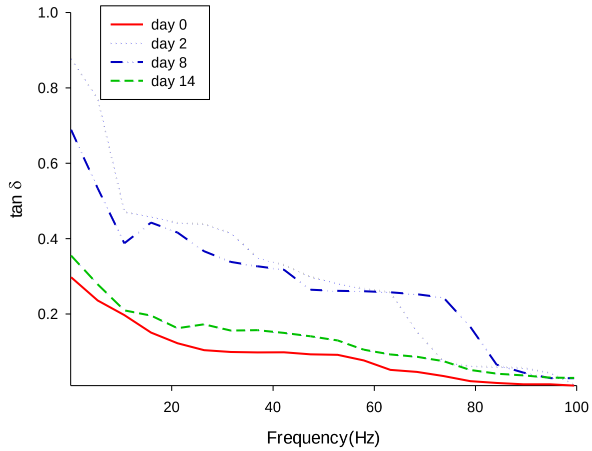

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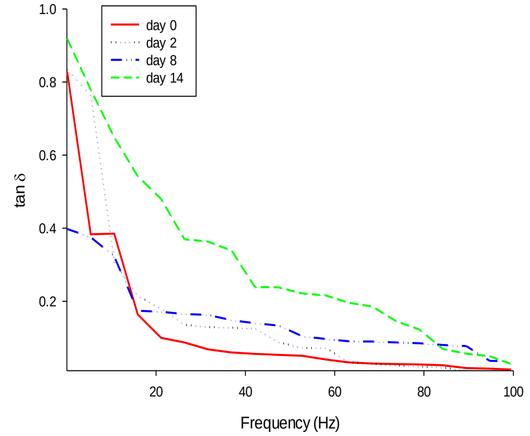

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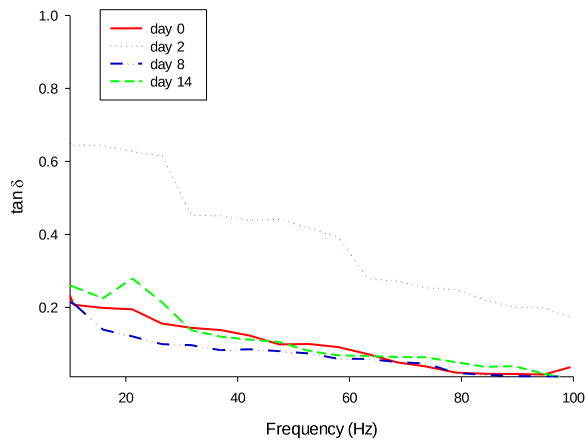

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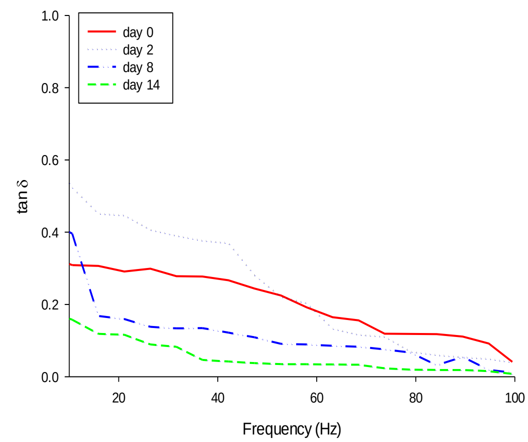


(j)

Fig 5.3. tan δ of puddings made from (a) SAH 177 (b) SAH 329 (c) SAH 177 1-115-25 (d)SAH 329 1-115-25 (e) SAH 177 1-115-35 (f) SAH 329 1-115-35 (g) SAH 177 2-100-30 (h) SAH 329 2-100-30 (i) SAH 177 2-100-35 (j) SAH 329 2-100-35 at day 0, 2, 8 and 14.

5.4.2. Textural properties of rice puddings.

The textural properties of SAH 177, SAH 329, and puddings made from heat moisture-treated rice flours composed of different amylose contents are presented in Fig. 5.4. Based on the results in this study, the hardness, gumminess, and chewiness of puddings made from of the control samples (SAH 177 and SAH 329) were higher than those of pudding made from the flours of the heat-moisture treated flours for all the whole period of storage (Fig.5.4). The results of the hardness (0.04 - 0.26 N), gumminess (0.01 - 0.21 N) and chewiness (0.01 - 0.21 N) properties in this study were comparatively lower than the results of the hardness (0.98 - 9.81 N), gumminess (0.49 - 5.39) and chewiness (0.49 - 5.39) reported by Thaiudom and Pracham, (2016). The low values observed in this investigation can be attributed to the absence of caseins in the rice milk utilized in this study. Through hydrophilic interactions, amylose molecules can entrap casein micelles in mammalian milk (Semo et al., 2007), thus resulting in tighter, stronger and higher texture profiles found in the study of Thaiudom and Pracham, (2016). Generally, the hardness, gumminess, and chewiness values of control puddings (SAH 177 and SAH 329) and the values of the heat-moisture treated rice puddings decreased when storage time increased (day 2) but showed little or no variation after the 2nd day of storage. The texture of SAH 177 pudding during the first day of storage was much harder than that of the SAH 329 rice pudding, probably because of more amylose leaching from the granules of SAH 177. This observation was consistent with the results of the rheological properties mentioned in the previous part of the study. Leached out amylose molecules can create networks with other micronutrients such as lipids and protein when the starches cool down (Alamprese et al., 2011; Wang et al., 2015). These interactions result in increased hardness in the rice pudding. In addition, the higher degree of amylose leaching from the control pudding samples (SAH 177 and SAH 329) added to the observed higher hardness, gumminess and chewiness of the control puddings when the storage time increased, resulting in quicker re-aligning of amylose and amylopectin molecules in the control pudding samples compared to those in the heat-moisture treated moisture puddings (Fig. 5.4).

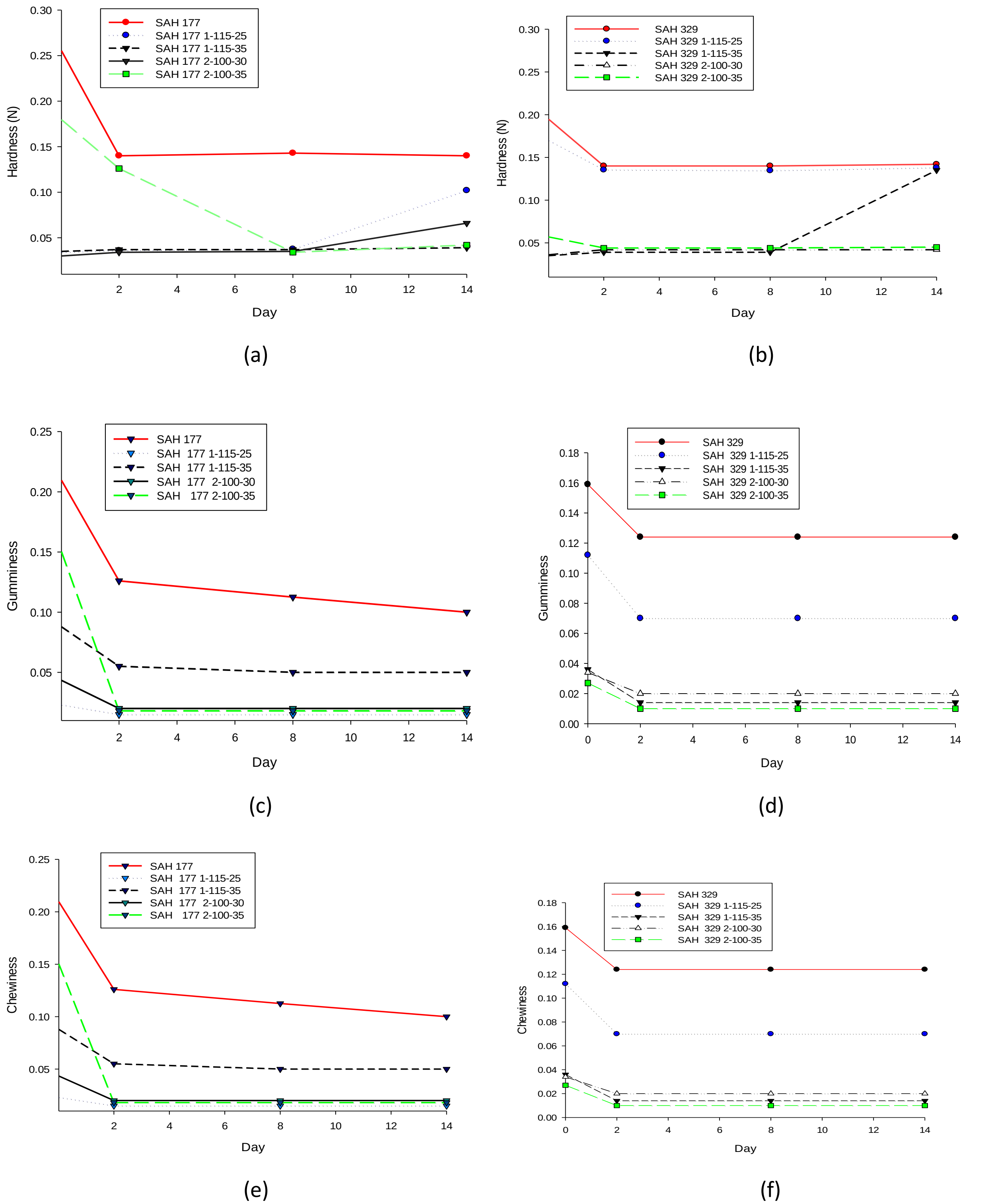


Fig. 5.4. Hardness (a, b), gumminess (c, d) and chewiness (e, f) of puddings made from SAH 177 (control and heat-moisture treated samples) and SAH 329 (control and heat-moisture treated samples).

Regarding the influence of heat-moisture treated rice flours on textural properties of the puddings, all the textural properties of the heat-moisture treated flours were significantly different from the control samples (Fig 5.4). Heat modification of rice flours resulted in a reduction of the hardness, gumminess and chewiness properties of the puddings. Under the same preparation conditions, control samples exhibited much higher pudding hardness, gumminess, and chewiness than the heat-moisture treated flour samples. It was suggested that pre-gelatinized starch granules of the heat-moisture flour samples with higher moisture content swelled less, and thus formed weaker pudding systems. The low gumminess and chewiness properties exhibited by the heat-moisture treated puddings suggest greater compatibility of the dispersion to that of the control pudding. Moreover, lipid complexes formed during heat-moisture treatment could retard the swelling of rice granules (Biliaderis and Tonogai, 1991). Lipid complexes result in incomplete hydration and incomplete gelatinization of the pre-gelatinized starch molecules which consequently leads to a reduction in the gelling zone and reduced gumminess and chewiness.

5.4.3. The color of rice puddings

Color is an important attribute of processed food products because, in addition to texture and flow behavior, it determines to a great extent the consumer's preference. The physicochemical properties (protein composition, moisture, and reducing sugars) of the pudding are determinants that could impact its color characteristics. Fig. 5.5 presents the color parameters (L*, b* and ΔE) of the pudding variation from day 0 to day 14 of storage. Overall, the results of this investigation show that the lightness (L*) values of all the studied rice puddings displayed a steady increase throughout the entire study time. On the other hand, the control puddings (SAH 177 and SAH 329) exhibited higher b* (Yellowness) values than the other modified puddings. This was expected as there are more reducing sugars (amylose) and proteins in the control puddings than in the heat-moisture treated puddings (Table 5.1). The b* value is generally an indication of the degree of browning reactions in a given system. Generally, the b* values of all the studied rice puddings showed a steady decrease as storage time increased. The increase in lightness positively correlated with the decrease in the yellowness (b*) of the pudding with storage time. These variations indicate the degree of browning. Browning reactions in the food system have been shown to be triggered by Maillard reactions between proteins and reducing sugars and are influenced by the distribution and availability of water (i.e. moisture) in the food system, in this case, the pudding.

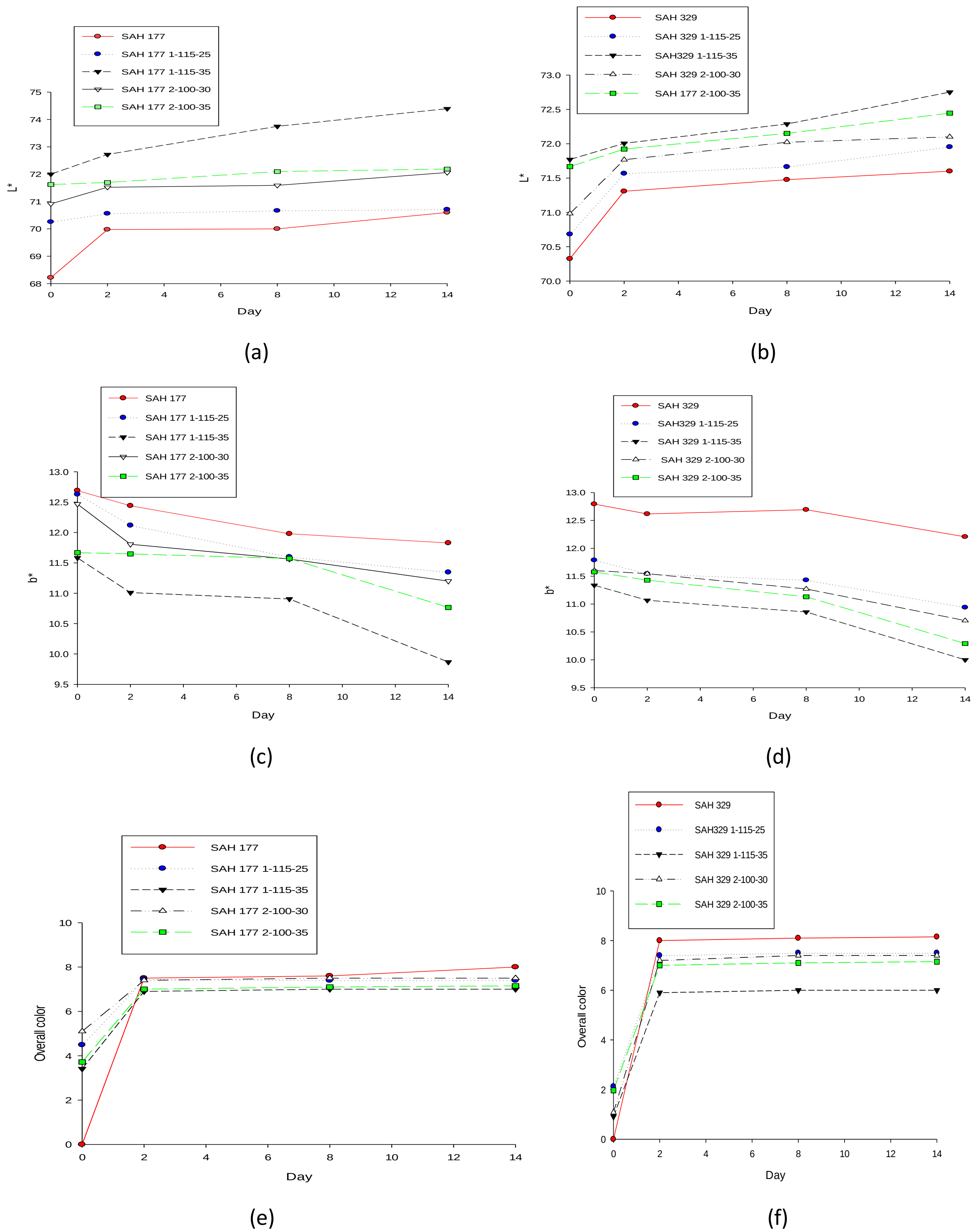


Fig. 5.5. Lightness (a, b), browning index (c, d) and overall color (e, f) of puddings made from SAH 177 (control and heat-moisture treated samples) and SAH 329 (control and heat-moisture treated sample).

Rice puddings are naturally mellow yellowish in color due to the presence of amino acids, polysaccharides, and moisture in the eggs, rice, and milk used in the product preparation. Among the studied heat moisture treated puddings, SAH 177 1-115-25 and SAH 329 1-115-25 showed the least L* value of 70.25 ± 0.50 and 70.68 ± 1.50 respectively. On the other hand, the maximum L* values among the heat-moisture treated puddings was observed for puddings SAH 177 1-115-35 (74.39 ± 1.50) and SAH 329 1-115-35 (72.55 ± 0.25). Likewise, the clearer heat-moisture treated puddings exhibited the lowest b* (yellowness) values (9.9 ± 1.5). The b* values of the heat-moisture treated puddings were within the ranges of 9.9 – 12.53 and showed a steady decrease as the total protein content in the puddings decreased, reaching a minimum of 9.9 ± 1.5 in SAH 329 1-115-35 pudding.

The overall color (ΔE) variations in the puddings are depicted in Fig. 5.5 (e and f). The results of the study confirmed that heat-moisture modification treatment significantly ($p<0.05$) influenced the color attributes of the rice puddings. The control samples showed the maximum changes while SAH 177 1-115-35 and SAH 329 1-115-35 showed the least variations. Regarding the heat-moisture treated puddings, SAH 177 1-115-25 and SAH 329 1-115-25 showed the maximum color change, thus leading to the assumption that heat moisture treated rice flours significantly influenced the color attributes of the rice pudding.

5.5. Conclusion

The different heat-moisture treated rice flours had a lesser impact on the rheological behavior of the puddings, in comparison to the puddings made from the untreated rice flour samples. The G' and G" values of the pudding samples made from the heat-moisture treated flours were significantly different from the G' and G" data of the puddings made from the untreated rice flour samples. In addition, textural studies of the heat-moisture treated puddings revealed less firm textural properties than the control puddings. The variations observed in the textural parameters of all the studied pudding samples were correlated to the degree of amylose leaching in the flour samples and degree of pre-gelatinization prior to the application of the flour in the pudding preparation.

CONNECTING TEXT

In chapters 4 and 5, the influence of heat-moisture treatment on the properties of flours and flour-based products from SAH 329 and SAH 177 was evaluated. In chapter 6, the impact of another physical modification, microwave processing, was studied. Rheological, physicochemical, thermal and functional data showing the influence of varying the moisture content, processing time and power during microwave on non-waxy rice flours from SAH 329 and SAH 177 were obtained.

6. Influence of microwave power, processing time and moisture content on the physicochemical, rheological and thermal responses of high amylose rice flours.

6.1. Abstract

The objective of this work was to understand the physicochemical, rheological and thermal changes induced in high amylose rice flour samples during microwave heating. SAH 329 and SAH 177 rice flour were dispersed in distilled water, to obtain dispersions having 20, 30 and 40% moisture content. The samples were heated in a microwave oven at two power levels (550 and 850 W/g) for 15, 25, 35 and 45 secs. The results of the time-temperature profiles indicated that the heating rate was approximately 1.35 ^{o}C/s for SAH 329 and SAH 177. Microwave treatment led to observable changes in the amylose and protein composition of both rice flours. The amylose content increased with microwave processing time, while protein content decreased during the same conditions. Beyond 15 secs of microwave treatment, the oil and water holding properties of both flours showed an increasing trend. Thermal analysis revealed an increase in the gelatinization properties of the flours with increasing processing time. Rheological data revealed that the (G'), loss modulus (G''), complex viscosity (η*) and hardness values of flour gels were enhanced with an increase in microwave processing time. The microwaved treated flour samples indicated solid-like gel characteristics with G' > G'' at all studied frequencies (0.01 – 10 Hz). Clearly, microwave processing time had a greater influence than microwave power on the studied parameters in this study.

Keywords. Rice Flour, Microwave, Amylose, Rheology, Starch

6.2.Introduction

Rice is an important staple food for most of the world's population (Liu et al., 2013). Rice flour, due to its low allergenicity is highly utilized in gluten-free product applications (Jeon et al. 2011). Non-waxy rice is difficult to handle; it has a hard texture, poorly stable under high shear system, produces inelastic paste and requires a lot of energy during processing (Ong and Blanshard, 1995). The non-optimal functional characteristics of non-waxy rice contributed to its flour being one of the most underutilized rice types in the world. With the increasing popularity of ready-to-eat (RTE) meals, waxy rice is often selected for meal preparations over and above the non-waxy rice. The selection of waxy rice types is often because of its high oil absorption properties, soft and gentle texture (Bao and Bergman, 2018). To enhance the inclusion of non-waxy rice in industrial and domestic applications their techno-functional properties can be improved using modification techniques such as microwave heating.

Conventional heating is caused by heat transfer through the thermal movement of atoms, molecules, and free radicals which create heat at the contact surface and the heat disperses inwards to the center of the material being heated (Jiang et al., 2011). Microwaves are electromagnetic waves in the frequency range of 300 – 300,000 MHz (Sumnu, 2001). During microwave processing, polar molecules absorb microwave energy and reorient themselves in relation to the absorbed electric field. This rapid alteration in the molecular organization of the microwaved material caused by molecular friction leads to uniform heating throughout the entire material and a much faster heating efficiency compared with conventional heating (Fan et al., 2012). Microwave technology is increasingly playing an important role in thermal processing at homes and in the food industry because of the rapid heating rate and ease of practice.

Khraisheh et al. (2004) evaluated the quality and structural changes in starchy foods during microwave and convective thermal processing. This study reported less destruction of vitamin C but an increased rehydration property in the microwave-processed sample. Lewandowicz et al. (2000) reported that microwave heating of cereal starches resulted in a change in the gelatinization range and a reduction in the solubility and crystallinity. Modification of waxy flour by microwave heating has also been studied by Uthumporn et al. (2016). From this study, the researchers observed that microwave processing increased the amylose concentration and gelatinization temperature of the modified flour. The conclusion from these studies indicated that the microwave treated biomaterials depicted greater functionality than non-microwave treated samples.

Despite many research focused on microwave processing of starches from potatoes (Hódsági et al., 2012; Lewandowicz et al., 1997), non-waxy rice (Anderson and Guraya, 2006; Fan et al., 2012), corn flours (Román et al. 2015; Uthumporn et al., 2016) and non-waxy rice flours (Smith et al. 2018; Uthumporn et al., 2016), there are still limited research works on the impact of the infuence of moisture content, processng time and power variation during microwave processing on the rheological, thermal, functional and physicochemical properties of non-waxy rice flours.

The moisture content, microwave power and microwave processing time are major factors influencing the behavior of biomaterials during microwave processing. It was reported that microwave processing of tuber starches at a restricted moisture content (< 35%) led to an increase in starch pasting temperature, a decrease in solubility and alterations in the crystalline arrangement (Lewandowicz et al., 1997). Anderson and Guraya (2006) evaluated the influence of microwave treatment on waxy and non-waxy rice starches at 20% moisture content. They reported significant alterations in viscosity characteristics after microwave radiation treatment. Sirisoontaralak and Noomhorm, (2006) observed that the peak viscosity of non-waxy rice starch decreased as microwave power applied to the biomaterial increased. Xie et al. (2013) also reported an increase in the magnitudes of G' and G" as processing time increased during the microwave treatment of potato starch.

The present study was therefore carried out to understand the heating influence of microwave processing on non-waxy rice flour and investigate the impact of the microwave processing factors such as moisture content, processing time and microwave power on the physical characteristics, chemical properties, thermal and rheological properties of the rice flour.

6.3.Material and methods

6.3.1. Raw material

Two rice cultivars namely, SAH 177 and SAH 329 were purchased from a local market in Dakar, Senegal.

6.3.2. Sample preparation

Prior to the entire test, the selected raw rice grains were milled using a versatile grains and seeds grinder (SUMEET Multi Grind, India). The milled flour was sieved using a 250µm sieve to ensure particle uniformity.

6.3.3. Microwave treatment of samples

Dispersions of non-waxy rice flours (SAH 177 and SAH 329) in distilled water were prepared to a final solids concentration of 20, 30 and 40 % on a dry weight basis.

The non-waxy rice flour dispersions were homogenized using a magnetic stirrer for 10 minutes at a low speed. The flour dispersions were then heated in a Panasonic NN-SN651W (Panasonic Corporation, Osaka, Japan) microwave oven (2450 MHz, power 1200 W) for 15, 25, 35 and 45 secs in Pyrex glass cylinders open at both ends (50 x 40 mm) at power levels of 550 and 850W. The cylinders were covered with Saranwrap ® to control moisture loss during the thermal process. The weight of the heated samples was 70 ± 0.2 g. Care was taken to place the sample at the same place in the microwave during each treatment.

Following the different treatments, the samples were cooled to room temperature (25°C) and then immediately freeze-dried. The freeze-dried samples were then milled using a coffee grinder, sieved through a 60-mesh sieve sieve and stored in tightly sealed glass vials. Freeze-dried samples were later used for the different analyses that followed.

Preliminary studies helped in choosing the parameters used in this experiment. In addition, thermal analysis was carried out to estimate the maximum/melting temperature (T_m). This preliminary studies revealed that following 4 s of heating the flour was ungelatinized, while after 50 s, the resultant sample was a hard gel due to excessive moisture loss. The temperature of the samples of before and after the microwave process was monitored using a thermometer. To prevent gelatinization at high moisture content, the T_m of the microwave process was evaluated at 65^{o}C for samples with 40% moisture. For flour samples with 20 and 30% moisture content, T_m was kept at 98^{o}C.

6.3.4. Chemical properties

6.3.4.1. Amylose content of rice flour.

Isolation of starch from the rice flours was carried out using the alkaline deproteination procedure described by (Lii et al., 1996) with minor modification. Amylose content was determined using a commercial amylose/amylopectin assay kit from Megazyme (Ireland) according to the procedure described by (Gunaratne and Hoover, 2002). Twenty milligrams of the isolated starch from each sample was vortexed with 8 mL of 90% Dimethylsulfoxide (DMSO) followed by heating in a shaking water bath at 85ºC for 15 min. The mixture was kept at room temperature (25°C) for 45 min. The cold mixture was further diluted with and made up to 25 mL with distilled water. Aliquot of 1 mL from the diluted solution was added to 40 mL distilled water and 5 mL iodine solution (0.0025 M Iodide/0.0065 M KI mixture) and then mixed vigorously. The volume was adjusted to 50 mL with distilled water, mixed again and allowed to stand for 15 min for the color to develop. Absorbance was measured at 600 nm against the reagent blank.

6.3.4.2. Protein content

Crude protein content was determined by AOAC combustion method 968.06 (AOAC, 2005) using a LECO Nitrogen Analyzer (CNS 2000, St. Joseph, MI, USA). One hundred milligrams of each flour sample was weighed into a porcelain sample boat and placed in the ports of the automated sample loader. The samples were combusted in the thermal reactor (combustion chamber) at 1300°C. The signals from the nitrogen gas produced from the sample combustion process were recorded by the thermal conductivity detector of the instrument. The amount of protein present in the samples was calculated by multiplying the nitrogen content by a factor of 6.25.

6.3.5. Oil and water holding properties

The water holding capacity (WHC) was determined by dispersing 1.0 ± 0.01 g of flour into 10 mL distilled water (De la Hera et al., 2013). The final mixture was left at ambient temperature for 24 hours, after which the supernatant was decanted. The WHC was calculated and expressed as grams of water retained per gram of solid.

Oil holding capacity (OHC) was determined by the method of de la Hera et al (2013). Briefly, 100.0 ± 0.2 mg rice flour was mixed with 1.0 mL vegetable oil. The resulting mixture was stirred for 1 min to thoroughly disperse the sample in the oil. A vortex mixer was used to work on the sample for 30 min., then, the mixture was centrifuged at 3000 × g for 10 min at 4°C. The oily supernatant was removed, and the holding tubes inverted for 25 min to drain surface adhering oil. After this, the resulting residue was weighed, and the weight referred to as W_r. The oil holding capacity was expressed as grams of oil bound per gram of the sample on a dry basis.

6.3.6. Thermal studies

Samples (2.5 mg) were weighed into an aluminum pan and mixed with distilled water to obtain a sample-water suspension of 2:1. The samples were hermetically sealed and allowed to reach equilibrium overnight at room temperature before heating in the DSC. Thermal properties such as peak gelatinization temperature (T_p) and enthalpy of gelatinization (ΔH) were examined using a differential scanning calorimeter (DSC Q2000; TA instrument, New Castle, PA, USA). The sample pans were heated at a rate of 5°C /min from 25 to 125°C.

6.3.7. Rheological properties

6.3.7.1.Preparation of dispersion

Rice flour dispersion (8% w/w) was prepared by mixing rice flour with distilled water. The mixtures were stirred for 30 min. at ambient temperature and then heated at 95°C in a water bath for 30 min with mild agitation. After heating, the cooked rice flour dispersions were immediately introduced to the rheometer to estimate the steady and dynamic rheological characteristics of the treated samples.

6.3.7.2. Steady flow rheology

Steady flow analysis (shear stress, shear rate, apparent viscosity) was done over a shear rate range from 0.1 to 100 s^{-1} at 25°C in triplicates. Flow curves of the microwave-treated dispersions at 25°C were modeled using Rheology Advantage software, (TA Instruments, New Castle, DE, USA). The Power Law model, (equation (6)) was used to evaluate the pseudoplastic characteristics of the test materials.

$$\sigma = K\gamma^n \tag{6}$$

where σ is the shear stress (Pa), γ is the shear rate (s^{-1}), K is the consistency index ($Pa \cdot s^n$) and *n* is the flow behavior (dimensionless unit, n=2 for Newtonian fluids and less than 1 for pseudoplastic materials).

6.3.7.3.Dynamic rheology

Dynamic shear properties such as storage modulus (G'), loss modulus (G") and complex viscosity (η*) of the different dispersions of the rice flours were also analyzed with a rheometer (AR 1000, TA Instruments, New Castle, DE) using a parallel plate system (4cm diameter) at gap of 500μm at 25°C. Dynamic shear data were obtained from frequency sweeps over the range of 0.01 – 10 Hz at 2% strain. The rheological measurements were carried out in triplicates.

6.3.8. Textural properties

Flour gels made from the above rheological studies were analyzed using a texture analyzer (TA. HD. Plus, Stable Micro System, Godalming, UK). Textural property (hardness) was determined with a 25-mm diameter cylinder plate 60% strain of penetration, 5s of waiting time, and 2 mm^{-1} of test speed.

6.3.9. Statistical analysis

The data in this study are averages of triplicate observations. The results were analyzed using Analysis of Variance (ANOVA) and expressed along with the standard error of the mean value. The averages were compared by Fisher's least significant difference (LSD) test, and differences at $P < 0.05$ were considered significant. The data were subjected to statistical analysis using JMP Statistical Software version 17 (Version 14; SAS, NC, USA).

6.4. Results

6.4.1. Time-temperature profile during microwave processing

As shown in Figs. 6.1 and 6.2, irrespective of the power levels, the temperature of the non-waxy rice flours of both cultivars increased with increasing time and exposure to microwave energy. The increasing rate of temperature rise in the microwave processed samples can be attributed to the increased energy conversion efficiency in rice flours as a result of changes in crystallinity arrangement that accompanied the phase change. However, for the 20 – 30 % flours, when T_m (maximum temperature/melting temperature) was 98^{o}C, no clear plateau was observed after the initial temperature increase. At microwave power levels of 550 and 850 W, the temperature increased rapidly from the 25th second in all the studied moisture concentration for SAH 329 and 177. A plateau was observed after the initial temperature increase, followed by a drop in the rate of temperature rise.

The correlation coefficient of the temperature curves is presented in Table 6.1. The results showed that the correlation coefficient range was 0.3811 - 0.7921 for SAH 329 and 0.3721 – 0.7181 for SAH 177.

The low correlation coefficient observed in this study suggests a poor linear relationship between microwave processing time and the temperature changes in the flour matrix. This result is in agreement with literature reports. For instance, Zhu and Guo, (2017) reported that varying moisture content in flour samples and their various components (proteins, lipids, and starch) resulted in different dielectric properties in the flour granule which in turn influences the amount of energy absorbed by the material. Starches, proteins, and lipids are exceptionally complex biomolecules which possesss a combination of neutral, polar and charged side chains. These structural arrangements not only stabilize the molecule arrangement but also contribute to the electrical and dielectric properties of the individual molecules (Lu and Abbott, 2004; Ndife and Bayindirli, 1998.; Zhu and Guo, 2017). Increase in microwave processing time could lead to structural modifications of the molecules by altering the hydrophilic/hydrophobic properties of the molecules via addition or loss of ionic or polar charges or hydrophobic groups to of from the granule. These modifications may also lead to changes in the response to microwave radiation through alterations in dielectric properties or loss of the chemical groups or loss of functional characteristics such as viscosity and of phase transition temperatures (Miller et al., 1991). The results of this study also showed a decrease in the correlation coefficient at power levels of 550W, as the moisture content of SAH 177 and SAH 329 was increased. The temperature curve of the samples in this study was higher than that of the conventional slow process (0.05 ^{o}C/s) heating rate reported by Fan et al. (2013). The difference could be attributed to the higher heating rate used in the present work which was approximately 1.35 ^{o}C/s for SAH 329 and SAH 177 rice flour samples.

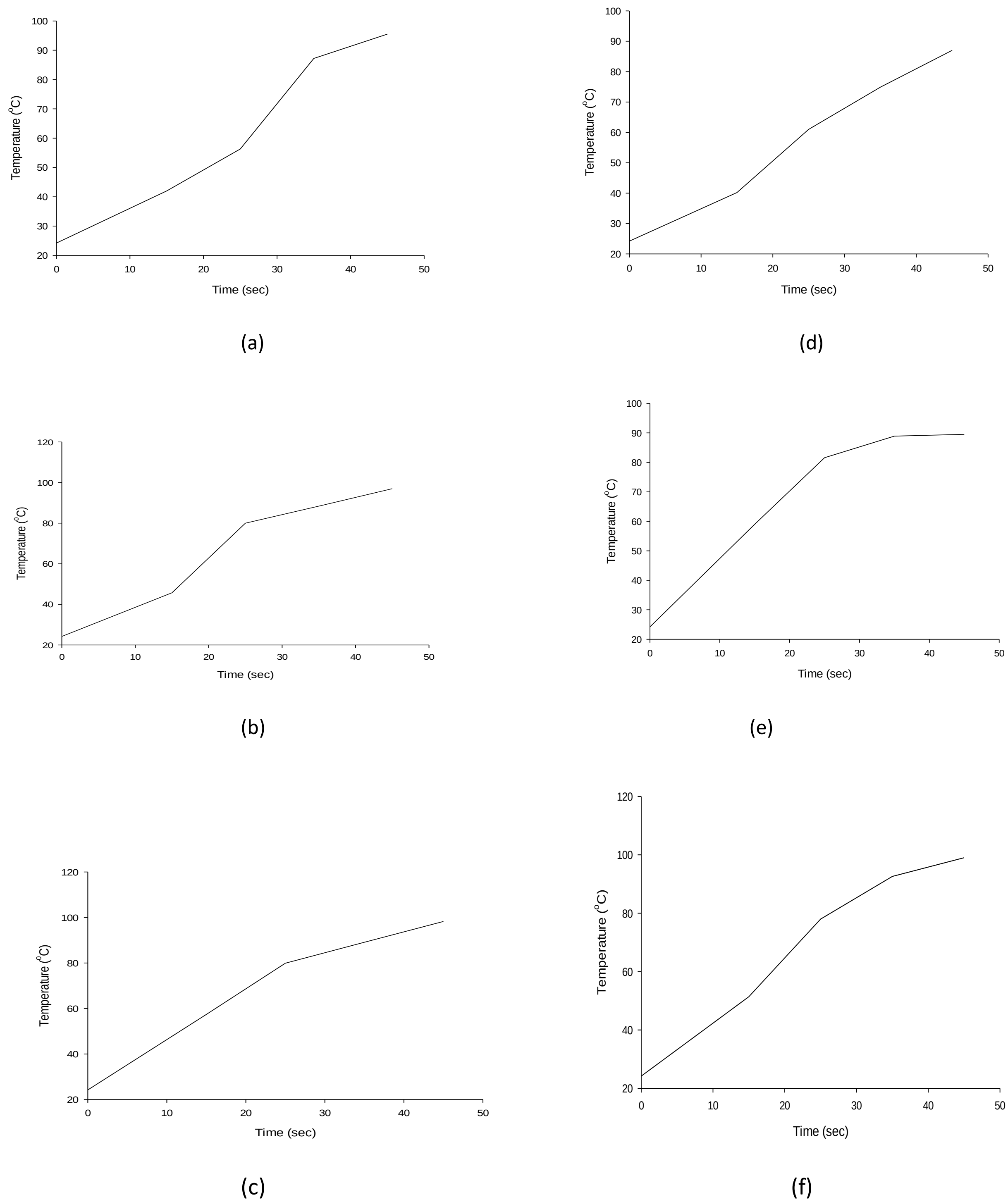
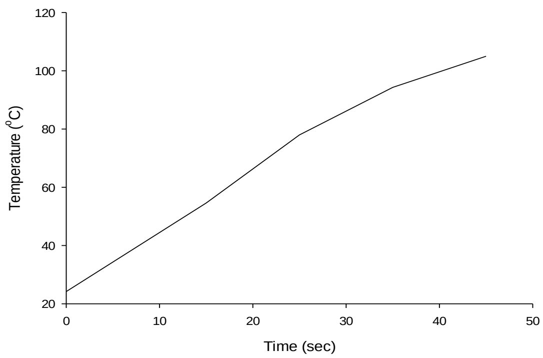
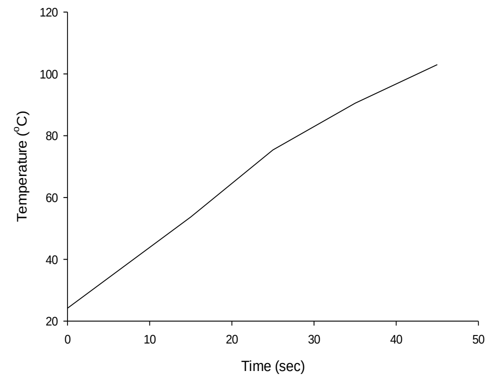


Fig 6.1. Time-temperature profiles at different solid concentrations for SAH 329 rice flour: (a) 20% (b) 30% and (c) 40% and SAH 177: (d) 20% (e) 30% and (f) 40% at power level of 550W.



(a)



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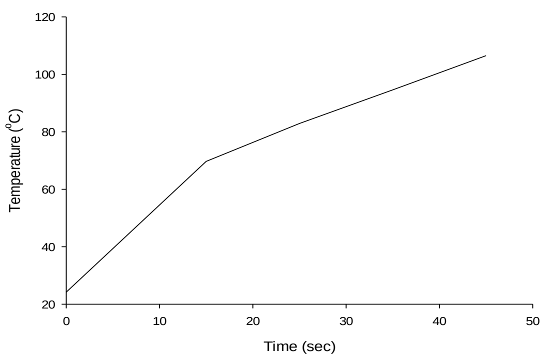
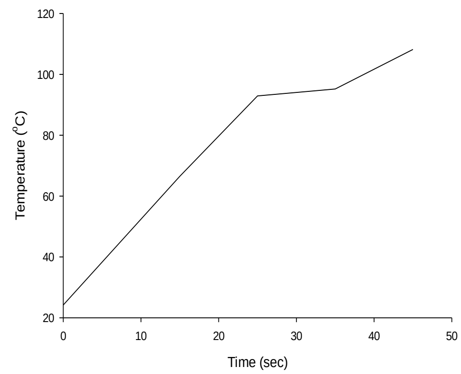
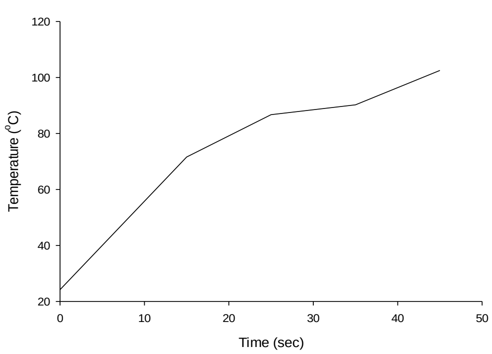


(b)



(e)



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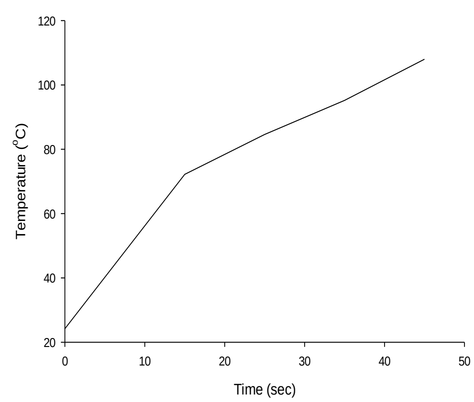


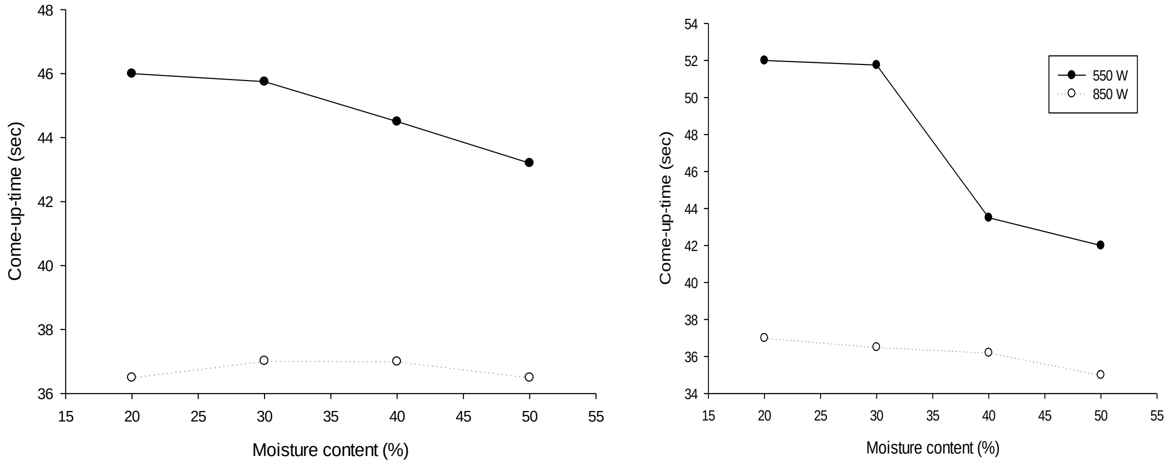
(f)

Fig. 6.2. Time-temperature profiles at different solid concentrations for SAH 177 rice flour: (a) 20% (b) 30% and (c) 40% and SAH 177: (d) 20% (e) 30% and (f) 40% at power level of 850W.

Table 6.1. The correlation coefficient of the temperature-time curves of the rice flours at different moisture contents (20, 30 and 40%) and power levels (550 and 850 W)

	550 W			850 W		
	20%	30%	40%	20%	30%	40%
SAH 329	0.7921	0.6859	0.5779	0.7271	0.5317	0.3811
SAH 177	0.6951	0.3721	0.6834	0.7181	0.5433	0.5095

The come-up-time (CUT) represents the processing time needed for the flour sample to heat to its T_m during microwave treatment (Anderson and Guraya, 2006). The CUT for the microwave treated samples is shown in Fig 6.3. The result showed that the CUT was different in the two power levels monitored. A faster rate of reduction in CUT was noted in the flour samples treated with 550 W power. At all moisture concentrations tested, CUT was shorter at 850 W than at 550 W. This implied that the rate of heat transfer was higher in 850 W than at 550 W. Non-waxy rice flours are 15-40 % amylose comprising α (1→4) glucosidic linkages in addition to α (1→6) linkages involved in the branched portion of the molecule. This spatial configuration of the starch molecules in these rice flours confers a more bulky structure to flour, which leads to slower hydrolysis. This means that non-waxy rice flours are less susceptible to breakdown than waxy rice flours. A negative linear correlation was observed between moisture content and come-up time for both cultivars (Fig 6.3). Statistical analysis also showed that the power of microwave processing had a significant influence ($P < 0.05$) on the come-up-time.



(a) (b)

Fig 6.3. Come-up time of (a) SAH 329 and (b) SAH 177 with different moisture contents at 550 and 850 W. (*Note*: Come-up-time = time to heat to T_m of rice flour.)

6.4.2. Chemical composition studies

6.4.2.1. Amylose content studies

The amylose composition of SAH 329 and SAH 177 flours as a function of microwave power (550 and 850 W), processing time (sec) and moisture content are presented in Fig 6.4. The results show that the microwave time and moisture content had a significant influence ($P < 0.05$) on the amylose content of both rice flours. Highest amounts of amylose content were detected on microwave treated samples at 40% moisture contents. This result suggests that the increase in dispersion concentration provided an increasing medium for a rise in the weakening of the amylopectin molecules thus increasing the amylose content of the flours. The extent of amylose increase rose markedly with increasing moisture content. At all power levels, there was a minimal rise in amylose content at 20% moisture content. This tends to suggest that moisture content of 20% would be optimal to strengthen the starch linkages in amorphous sections and reduce the extent of amylose increase/amylopectin breakdown.

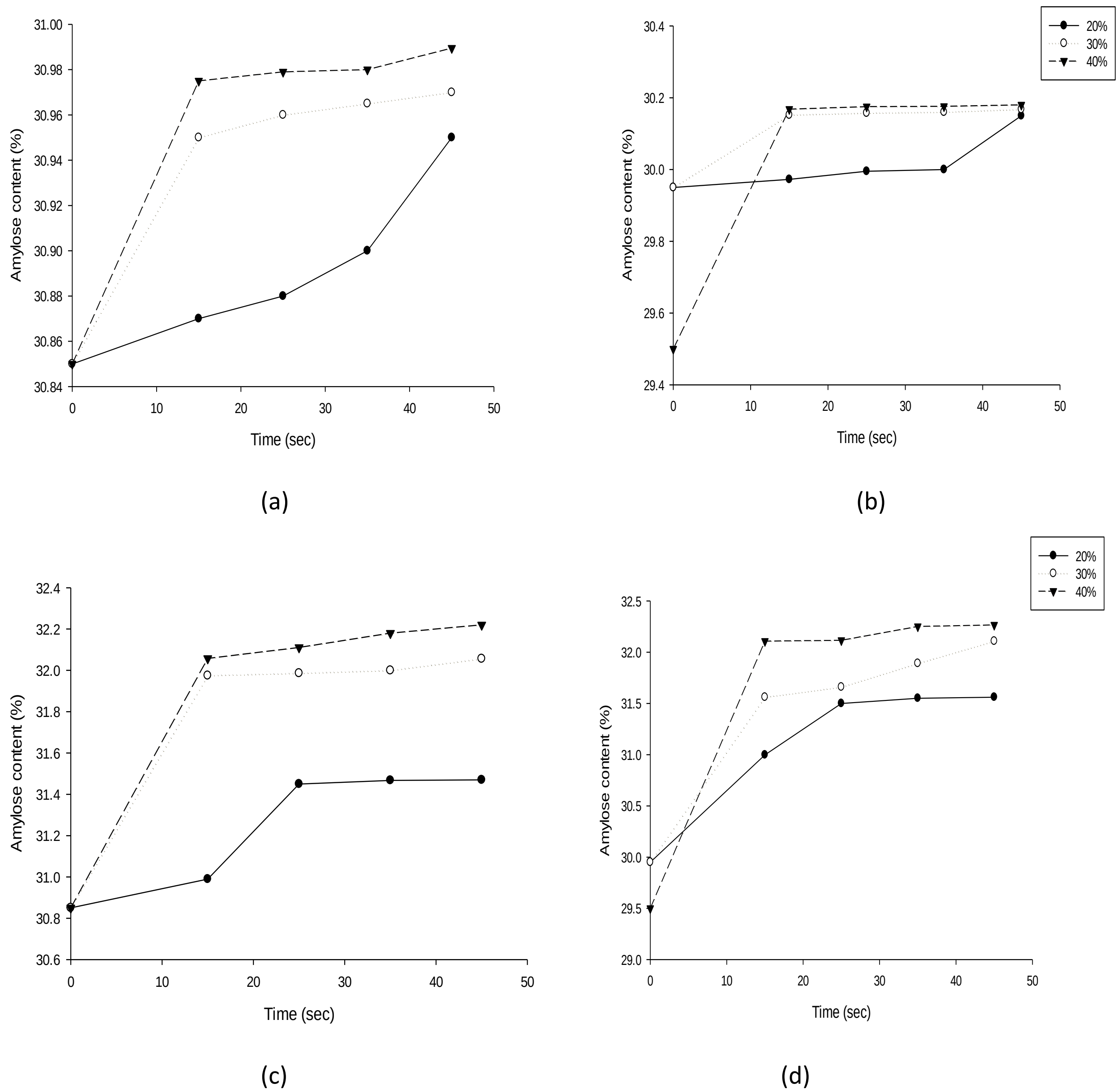


Fig 6.4. Amylose content variation of (a) SAH 329 and (b) SAH 177 flours as a function of microwave power (550 W) and (c) SAH 329 and (d) SAH 177 flours as a function of microwave power (850 W)

Microwave processing time also had a significant influence on the degree of amylose increase (Fig 6.4). Increase in amylose content was observed with prolonged microwave treatment time. This change could be related to the breakdown in the branched chain amylopectin molecules in the starch molecules of the rice flour.

Also, the presence of amylose in the treated samples increased as the microwave power level was increased (Fig 6.4). This result showed that microwave power had a significant influence on the change in starch structure in the process of microwave treatment. Similar findings of microwave treatment on amylose content were reported by Uthumporn et al. (2016) and Zhang et al. (2009).

6.4.2.2.Protein content

The protein composition of SAH 329 and SAH 177 flours as a function of microwave power (550 and 850 W), processing time (sec) and moisture content are illustrated in Fig 6.5. A rapid reduction in the protein content was observed in the first 15 secs of treatment. There was a 33.25% decrease in the protein content of treated samples compared to the untreated samples. Thermal processing induces changes in the non-covalent hydrophobic interactions and intermolecular disulfide crosslinks that denature proteins and contribute to their insolubilization. Smith et al. (2018) also observed a minor change in the protein content of rice after microwave treatment and attributed this to the scission of disulfide bonds between cysteine residues present in rice protein. Nevertheless, the level of microwave power and the variation in moisture content also play a major role in the breakdown of the associative noncovalent interactions and peptide bonds maintaining the protein associative structure, rather than disruption of covalent linkages (Smith et al. 2018).

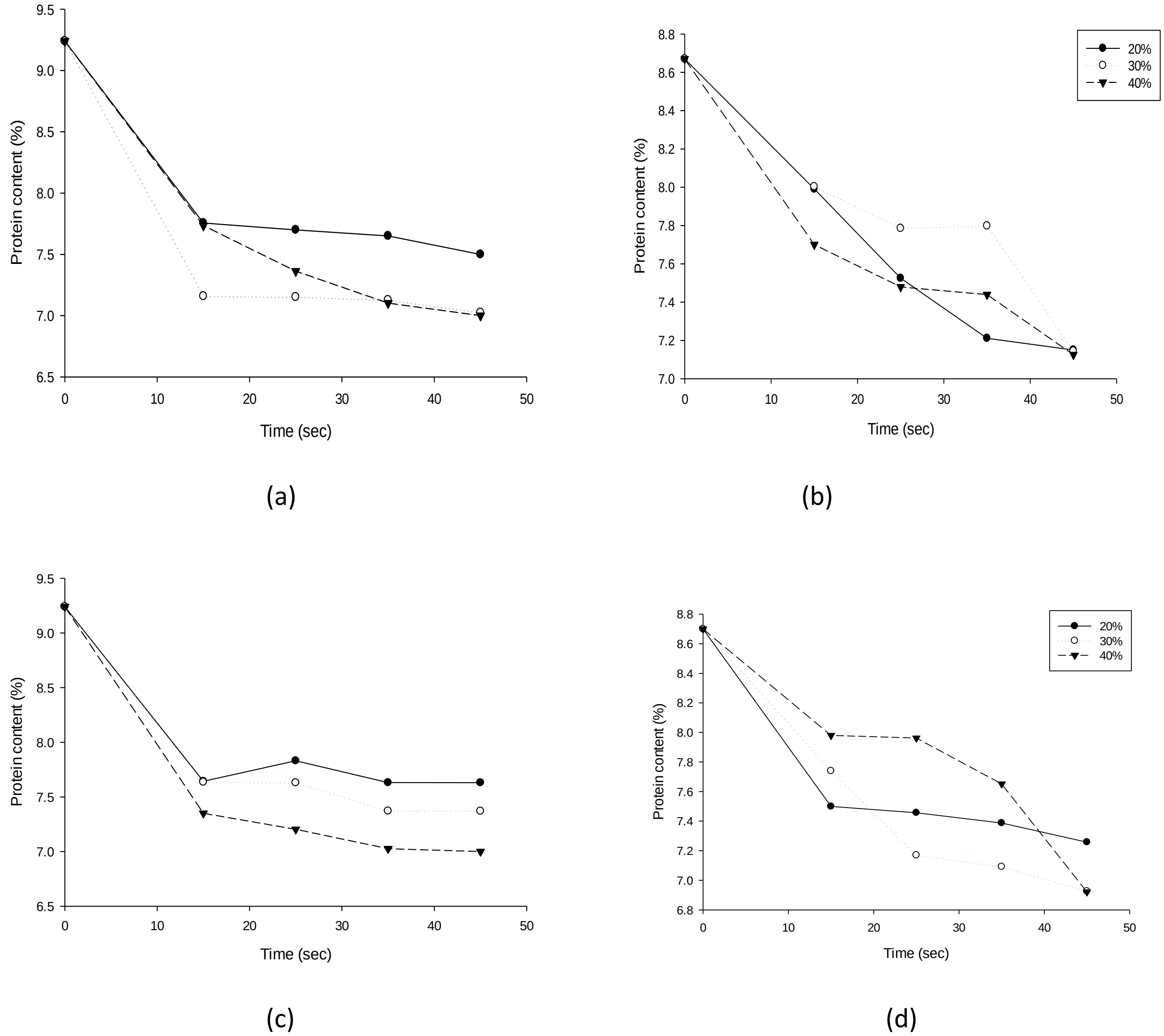


Fig 6.5. Protein content of (a) SAH 329 and (b) SAH 177 flours as a function of microwave power (550 W); (c) SAH 329 and (d) SAH 177 flours as a function of microwave power (850 W)

6.4.3. Oil and water holding capacities

Oil and water holding capacity properties play key roles in flour utilization and other applications. The oil and water holding capacity properties of SAH 329 and SAH 177 samples are presented in Figs. 6.6 and 6.7, respectively.

The trend showed that water and oil holding capacity typically increased with increasing heating time (Figs. 6.6 and 6.7). Within the first 15 secs of heating, a slight reduction in both the oil and water holding parameters was recorded. Thereafter, an increase in both properties was observed with increasing treatment time. Greater oil and water holding capacities were noted in the 40% flour samples than in the 20 or 30% flour samples processed at 550W. The higher rates of increase in the oil and water holding properties of the 40% flour gels are likely due to the greater influence of moisture content on processing treatments and the structural arrangements of the various chemical components (such as protein, lipids, carbohydrates) in the food matrix. During flour processing, microwave treatment causes alterations to the structure of proteins in the flours which in turn influences the charges on ionizable groups which alters the conformation of proteins by either exposing or concealing water (Ahmed et al., 2016; Zhao et al., 2012) and oil (Chandra, 2013) binding sites. The amounts of bound oil and water in the flour matrix depend on the amino acid composition, especially the number of polar groups exposed for oil holding, water binding, the conformation of proteins, surface hydrophobicity and processing conditions (Ahmed et al., 2016).

Previous research have also related starch gelatinization with the oil (Ratnayake and Jackson, 2008) and water (Babu et al., 2015; Hoover et al., 1996) holding properties of the flours. The binding of lipid or water is somewhat reliant on the molecular accessibility or solubility of starch polymers, especially amylose. Disruption of the crystalline structure of starch fractions during microwave processing could impact the interaction of hydrogen bonding between starch and other molecules such as water and oil. If starch in the flour matrix is completely gelatinized (amylose-solubilized) during microwave radiation, and the chain-length of saturated fatty acids in the lipids or hydrogen bonding in water content increases, the amount of starch-lipid or starch-water binding also increases. Since starch-lipid or starch-water binding is not a surface process, amylose molecules are not readily available for binding with lipid and/or water if the flour samples are not gelatinized (Ratnayake and Jackson, 2009). This could explain the low oil and water-holding properties of the control samples. Therefore, as the starch granule swells during gelatinization, more amylose is molecularly accessible for binding with lipid and water.

The binding between flour and water and/or oil has practical implications in the formulation of several flour-based food products. For instance, several surfactants (mono- and diglycerides) and lipids are widely applied to limit staling in bread. During breadmaking, starch granules in the flour become swollen in the presence of amylose-complexing lipid components (Jackson, 2003). Since, during baking, starch-lipid associations occur while the flour granule is swelling (50–60°C), but prior to gelatinization, lipids can associate with amylose prior to its solubilization.

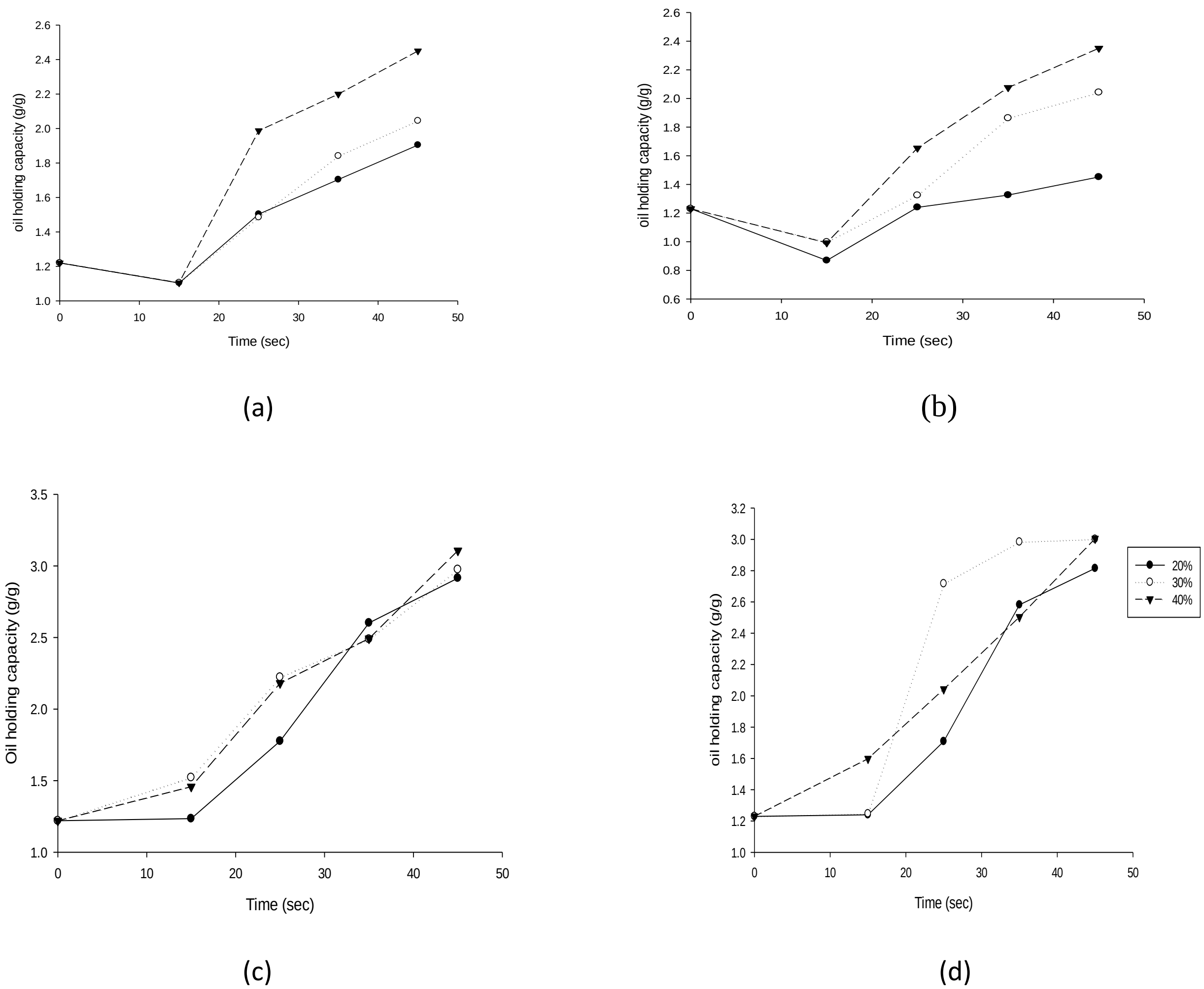


Fig 6.6. Oil holding capacity variation of (a) SAH 329 and (b) SAH 177 during microwave processing of 550 W; (c) SAH 329 and (d) SAH 177 flours as a function of microwave power (850 W)

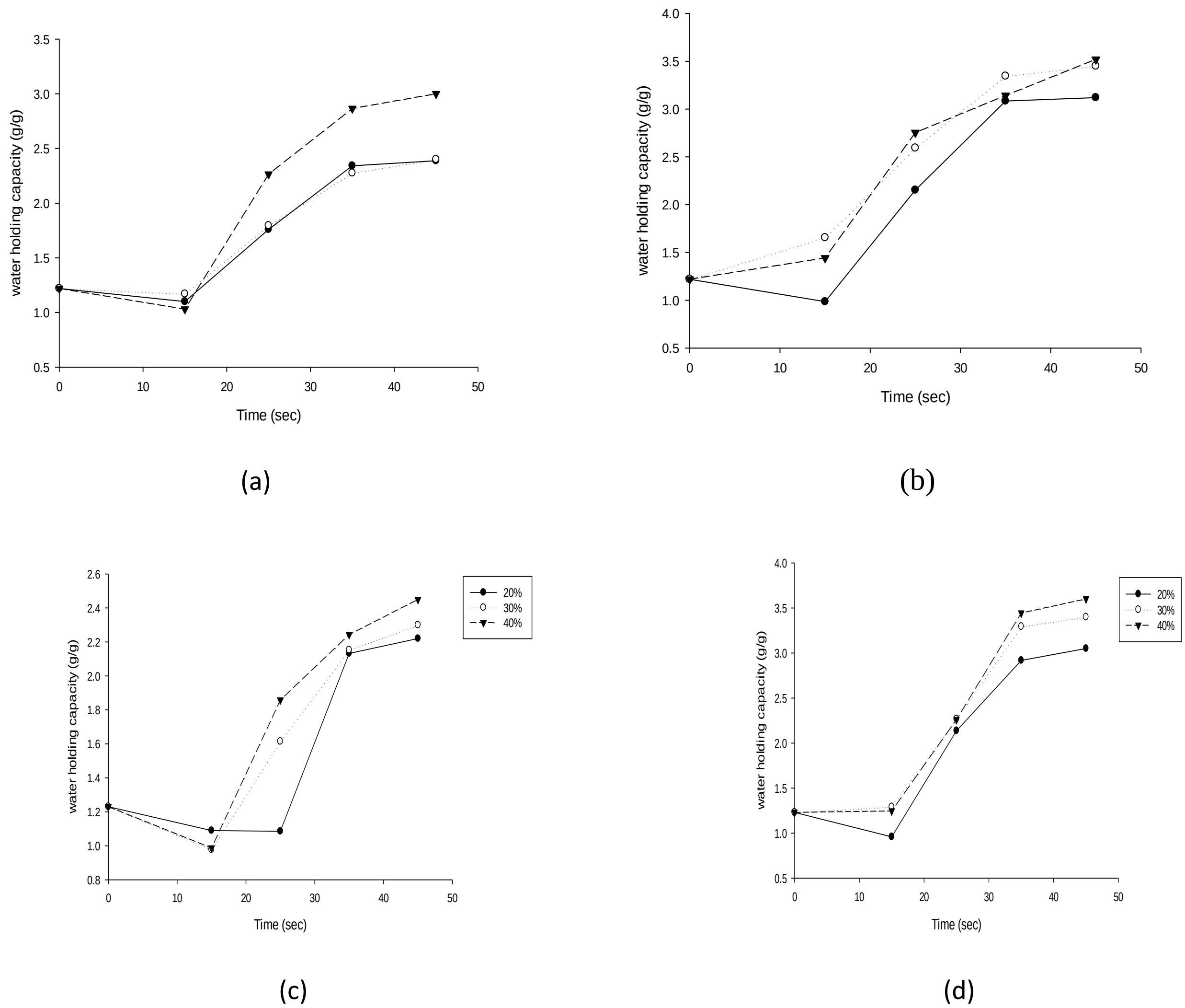


Fig 6.7. Changes in the water holding capacity of (a) SAH 329 and (b) SAH 177 during microwave processing of 550 W; (c) SAH 329 and (d) SAH 177 flours as a function of microwave power (850 W)

6.4.4. Thermal analysis

Gelatinization is a phenomenon which involves the flour granules swelling due to water absorption in amorphous regions, amylose leaching, loss of crystallinity arrangement, leaching of amylopectin from the flour granule and finally breakdown of the flour matrix. The result of the thermal properties of the control and microwave heated SAH 329 rice flour samples are summarized in Table 6.2.

Table 6.2. Thermal properties of control and microwave treated SAH 329 rice flour

Power level			550 W				850 W			
Cultivar Type	MC %	Time(sec)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)
SAH 329	20	0	81.27^{a}	87.57^{a}	89.87^{d}	2.45^{d}	81.27^{a}	87.57^{c}	83.61^{c}	2.45^{d}
		15	71.12^{b}	70.18^{d}	74.21^{c}	1.63^{b}	84.43^{c}	89.22^{b}	89.71^{a}	12.96^{b}
		25	73.59^{c}	75.74^{c}	78.22^{a}	8.92^{a}	85.64^{b}	90.06^{a}	92.11^{d}	18.26^{c}
		35	76.44^{d}	79.24^{b}	83.04^{e}	10.05^{c}	86.23^{d}	90.96^{b}	93.01^{b}	28.98^{e}
		45	80.22^{e}	82.29^{e}	84.21^{b}	12.72^{e}	88.63^{e}	91.76^{e}	94.11^{e}	30.16^{a}
	30	0	81.27^{a}	87.57^{a}	89.87^{c}	2.45^{d}	81.27^{a}	87.57^{c}	83.61^{c}	2.45^{d}
		15	72.43^{e}	75.76^{a}	80.01^{b}	1.96^{a}	85.66^{b}	90.02^{a}	90.18^{e}	24.43^{b}
		25	77.23^{b}	79.26^{c}	83.51^{a}	17.55^{e}	86.92^{d}	91.16^{c}	93.88^{b}	29.42^{c}
		35	78.63^{c}	82.16^{e}	84.97^{d}	22.26^{b}	87.64^{c}	92.98^{e}	94.05^{c}	33.42^{d}
		45	82.23^{d}	83.47^{b}	86.69^{c}	25.24^{c}	89.14^{e}	93.06^{d}	95.25^{d}	36.46^{e}
	40	0	81.27^{a}	87.57^{a}	89.87^{c}	2.45^{d}	81.27^{c}	87.57^{c}	83.61^{c}	2.45^{d}
		15	73.72^{e}	76.98^{c}	82.14^{c}	2.16^{a}	92.89^{b}	93.46^{a}	93.72^{a}	40.72^{c}
		25	82.24^{c}	92.46^{b}	92.82^{b}	28.45^{c}	93.21^{e}	94.06^{e}	94.71^{b}	42.45^{a}
		35	86.42^{a}	93.06^{d}	93.64^{a}	39.47^{e}	93.93^{a}	94.16^{c}	95.21^{a}	50.26^{b}
		45	89.64^{b}	94.14^{d}	94.91^{d}	40.16^{b}	94.03^{d}	94.26^{d}	95.61^{d}	62.49^{e}

Values expressed as mean ± standard deviations of three independent experiments (n=3). Distinct letters represent statistical differences within rows for the mean values at ($P<0.05$).

The data show that the thermal properties were significantly influenced by the processing time, moisture content and microwave power. From Table 6.2 it can be seen that at any given moisture content and power level, the microwave heated flours at 45 secs have significantly higher T_o, T_p, $T_{c,}$ and ΔH in both cultivars. Uthumporn et al. (2016) also observed that microwave processing increased the gelatinization properties of waxy rice flour. Interestingly, at 550 W, there was a reduction in the T_o T_p and T_c for both cultivars during the first 15 secs of microwave radiation, after which a steady increase was observed with increasing microwave processing time. This result indicates the intrinsic stability and heterogeneity in size and arrangement of the crystalline regions, contributing to a low degree of thermal properties obtained by the DSC measurements. On the other hand, at 850 W, an increasing trend for T_o T_p and T_c were observed. The result of this study is in agreement with previous research by Uthumporn et al. (2016) which reported a similar trend when corn and rice flours were exposed to microwave radiation over time. It is a well-known fact that gelatinization involves the uncoiling and melting of double helices composed of external chains of amylopectin and partial amylose in the flour granules. The melting of the double helix fraction during gelatinization is aided by hydration of amorphous segments, which in turn causes stress on crystalline regions, thereby stripping polymer chains from the surface of starch crystallites in the flour matrix (Uthumporn et al., 2016). Molecular reorganization in the granule leads to a more stable structure which in turn increases the T_o, T_p, T_c properties of the flour samples (Uthumporn et al., 2016). Thus, the increase in these properties caused by microwave treatment may be linked to better hydration of the amorphous regions.

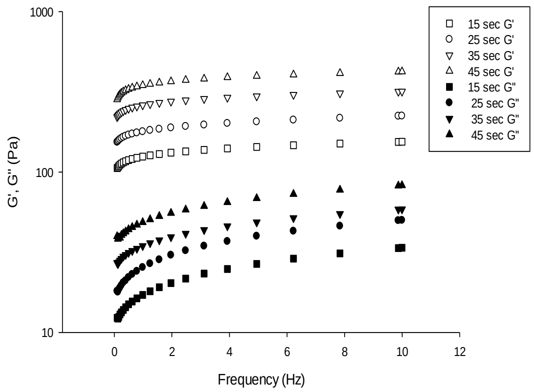
The enthalpy of gelatinization (ΔH) was more influenced in the microwave treatment samples than untreated samples (control) (Table 6.2). For example, at 550 W, a decrease in the ΔH values of the rice flours was observed during the first 15 secs of heating. The decrease in the ΔH values suggests that some of the double helices available in the crystalline and amorphous regions of the granule may have been disrupted under the conditions that prevailed during the microwave treatment. This observation agrees with previous studies by Lewandowicz et al. (2000) and Uthumporn et al. (2016). They reported that enthalpy change of gelatinization decreased substantially in microwave treated corn and rice flours. It is important to note the enthalpy of gelatinization is correlated to the thermal breakdown of the double-helical arrangement of the starch structure in the rice flour rather than that of the crystalline organization of the double helices (Lee et al., 2012).

In addition, for all treated flours, the values of T_o T_p, T_c and ΔH were higher than those of the untreated rice flour samples. These changes conclude that amorphous regions and crystalline orders within the flour matrix were altered by microwave treatment.

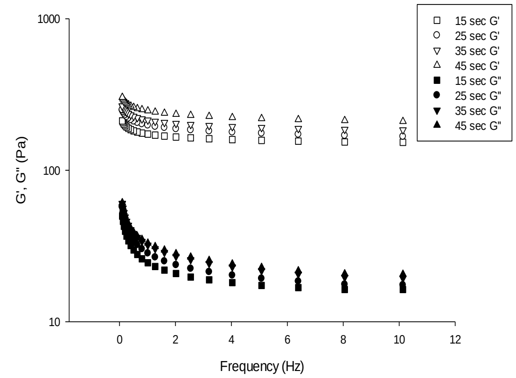
6.4.5. Dynamic rheological properties of microwave-treated rice flour.

Fig. 6.8 shows typical changes in dynamic mechanical spectra (G' and G") as a function of frequency for 20, 30 and 40 % SAH 329 dispersion at 550 and 850 W. For SAH 329, G' exceeded G" at all frequencies (0.1 – 10 Hz) indicating the domination of gel elastic flow behavior in the microwave treated samples. This result is in agreement with the findings of Wani et al. (2013) who reported that the G' values in waxy rice starches increased due to the effective concentration of the polymers in their respective microphases. The viscoelastic characteristics of SAH 329 were modified as processing time and power level changed at all the moisture concentration levels tested (20, 30 and 40%). For example, SAH 329 at all concentrations (20, 30 and 40%) and power (550 and 850 W) showed a gel-like behavior (G' > G") at all studied frequencies (0.1 – 10 Hz).

The mechanical firmness of microwave-treated thin flour dispersions turned into gel depends upon applied processing time and the moisture content (Wani et al., 2013). The degree of structure deformation increased with increasing processing time. For instance, at 20% moisture content (at 550 and 850 W), the magnitudes of G' and G" increased as microwave treatment time progressed from 15 to 45 sec. This could be related to the disruption of the well-organized structure of flour granules by microwave radiation. This is typically a series of changes that occur progressively until the granules are completely broken down. The strongest gel was formed when the sample was processed at 45 secs at both microwave power (550 and 850 W). However, at 30 and 40% moisture content (at 550 and 850 W), the progression of microwave treatment time had no significant influence on the G' and G" values of the flour. The difference in mechanical strength under microwave power of these modified flour samples at different processing time could be attributed to several factors such as the level of amylopectin crystallinity, amylose-lipid complex formation, water-flour interaction, microwave process and chain length (Uthumporn et al. (2016). The increase in G' and G" values may be related to the degree of gelatinization and the development of the network associated to those chains leached from the flour granules during the microwave heating. Generally, the overall alterations in SAH 329 gel network can be associated with the mechanism of phase interaction and thermodynamic combability between the various polysaccharides in the flour matrix.



(a)



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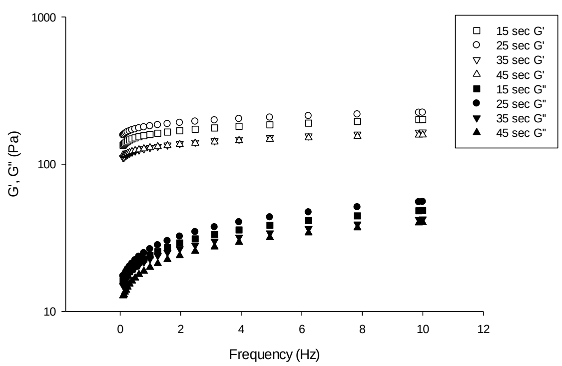

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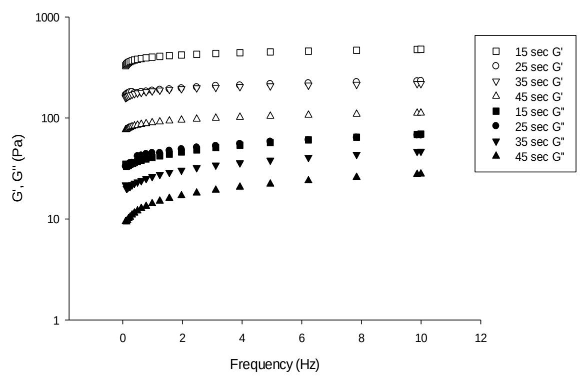

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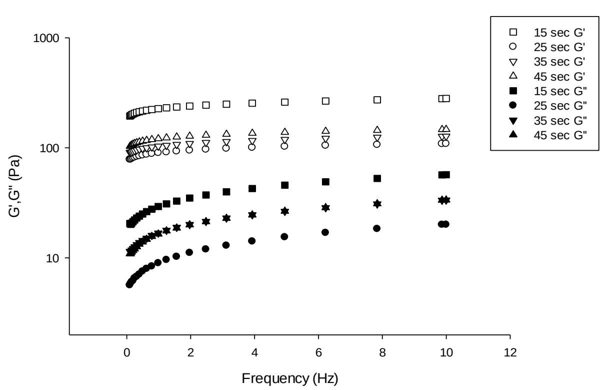

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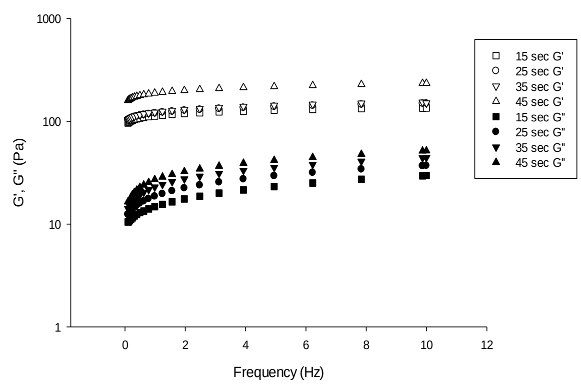


(f)

Fig 6.8. Rheological properties of SAH 329: dynamic mechanical spectra (open symbols, storage modulus, G'; closed symbols, loss modulus, G'') measured during microwave treatment at power of 550 W: (a) 20%, (b) 30% and (c) 40%; 850 W, (d) 20%, (e) 30% and (f) 40%

6.4.6. Steady shear flow behavior of microwave-treated rice flour

The steady shear flow behavior of SAH 329 flour at different moisture concentrations (microwave treated and untreated) are shown as apparent viscosity determined at a shear rate of 10 s^{-1}, and the flow behavior index (n) and consistency index (k) data are included in Table 6.3 The shear stress (τ) versus shear rate ($\dot{y}$) data at 25°C were well fitted with Power law model (Eq. (6)) with high determination coefficients ($R^2 = 0.92 – 0.99$) as shown in Table 6.3 The flow behavior index (n) reported in this study suggest the extent of shear thinning behavior or pseudoplasticity as it strays from 1, was observed in the rage of 0.03 – 0.33. Since lower values of n suggest more pronounced pseudoplasticity, it can be assumed that microwave treatment induced higher pseudoplasticity. The shear thinning behavior of microwave-treated rice flours could be due to the alteration in the macromolecular arrangement of the mixtures at variable shear rates and applied microwave power-time effect. The magnitudes of apparent viscosity at a specific shear rate of 10 s^{-1} increased with increasing time of microwave heating from 0 to 45 sec. The consistency index and flow behavior also displayed a similar relationship with microwave heating time. The increase in these rheological parameters is typically credited to the rupturing of the starch granules and gradual gelatinization of the granules as a result of the vibrational motion of the polar molecules during microwave radiation (Bilbao-Sáinz et al., 2007). These results are in good agreement with those seen in non-waxy rice flours (Chun and Yoo, 2004; Yoo, 2006b)

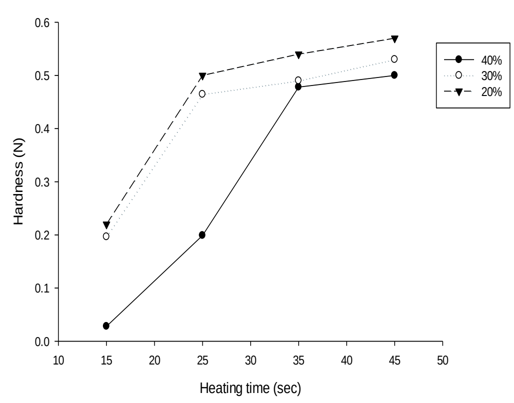
The n value for the rice flour samples increased with increasing microwave processing time. This implied that the pseudoplastic behavior of flour dispersions was weakened by the continuous application of microwave treatment. In addition, all microwave treated rice flours showed increasing n values compared to the untreated samples and these values were not dependent on the moisture content.

Table 6.3. Apparent viscosities (at 10 s^{-1}), consistency index ($Pa.s^n$), flow behavior index for SAH 329 flour dispersions with different moisture concentration at 850 W (with and without processing)

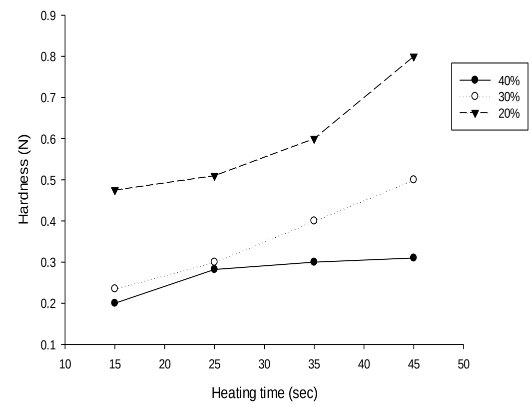
Cultivar type	MC %	Time(sec)	ŋ (Pa.s) at 10 s^{-1}	K (Pa. s^n)	n (-)	R^2
SAH 329	20	0	0.14^a	2.77^a	0.03^e	0.99
		15	1.67^b	7.65^b	0.23^a	0.99
		25	2.15^c	10.58^c	0.26^b	0.99
		35	2.20^d	11.47^d	0.32^d	0.99
		45	2.22^e	14.01^e	0.33^c	0.99
	30	0	0.14^a	2.77^a	0.03^e	0.99
		15	0.82^b	3.50^c	0.10^c	0.99
		25	0.93^e	6.36^b	0.23^a	0.99
		35	1.24^d	10.31^d	0.29^b	0.98
		45	1.26^c	12.31^d	0.32^d	0.97
	40	0	0.14^a	2.77^a	0.03^e	0.99
		15	1.15^c	5.77^b	0.15^a	0.99
		25	1.35^e	6.30^c	0.27^b	0.98
		35	1.58^d	11.55^e	0.29^c	0.99
		45	2.01^b	12.01^d	0.33^d	0.92

6.4.7. Textural Properties

The hardness values of the gels from SAH 329 rice flour increased with increasing heating time and moisture content (Fig 6.9). The increase in the hardness values of 20% moisture content was higher than that observed in the 30 or 40% flour samples processed at both 550 and 850W. The observed increase in the hardness of the 20% flour gel may be attributed to the higher presence of solid matter in the gel formulation and possibly the increased closeness of the polymers. These factors increased the degree of complex formation between the amylose and amylopectin fractions in the flour granules. Palav and Seetharaman, (2007) also observed that the hardness of starch gels increases upon microwave heating. Flour matrix is predominantly made of starch fractions. Eliasson and Bohlin (1982) reported that starch gels possess a continuous phase of polysaccharides in water and that the leached amylose component was responsible for the reduction in the modulus of elasticity and a further increase in gel strength. Therefore, it is possible that the formation of gel texture is likely due to the increased concentration of the gel and dry matter caused by loss of moisture in the flour matrix and possibly due to the leaching out of amylose from the flour granules during microwave processing.



(a)



(b)

Fig 6.9. Hardness of SAH 329 flour gels as a function of heating time, moisture content and power level (a- 550 W, b- 850 W).

6.5. Conclusion

The results of this study showed that microwave processing had a significant influence on the physicochemical, rheological and thermal attributes of rice flour. The variations observed in the properties studied reflect the differences in the moisture content, microwave power and heating time. The differences in the properties studied also provide an insight into the different heat and mass transfer behavior due to the different moisture contents and microwave power. The significant changes observed in the amylose content suggest that starch in flour granules during microwave processing lost its structural organization because of the vibrational motion of the polar molecules and rapid increase in temperature of the flour dispersions. The study strongly showed that the changes in the structure and composition of polymers caused by microwave processing time led to increasing amylose content, oil holding capacity, water holding capacity, gel hardness as well as decreasing come up time of processing.

CONNECTING TEXT

In chapter 6, the influence of microwave processing on flours from SAH 329 and SAH 177 rice cultivars was studied. Microwave processing was shown to cause changes in the oil and water absorption properties of these rice flours. Oil and water absorption properties are essential properties when selecting flours as breading materials for frying processes. In chapter 7, the application of microwaved flours as a coating material for frying of chicken breast cuts was evaluated. The study provided information on the application of modified rice flours on the quality of coated chicken fried products.

7. Textural, color and moisture changes of fried lean meat portions coated with microwaved treated rice flour

7.1 Abstract

The influence of non-waxy rice flours (untreated and microwaved) on quality properties (texture, moisture content, oil content, color, coating pick up, cooking yield and porosity) of air fried chicken breast cuts were evaluated. Chicken breast samples, 3 cm in length, 1.5 cm in thickness and 3 cm in width, cut from chicken breast portions, were coated with untreated and microwaved treated non-waxy rice flour (SAH 329) from Senegal. Samples were air fried at 170, 180 and 190°C for 4, 8, 12 and 16 min. Shear force, overall color and oil contents of chicken breast cut increased, whereas moisture content and porosity decreased with increasing air frying time. Application of microwaved modified coatings increased the coating pick up and cooking yield of the samples significantly. The highest porosity and oil retention properties were obtained when control samples were applied as a coating. Microwaved flours were shown to produce a product with reduced oil content, increased coating pickup, and cooking yield.

Keywords Frying. Chicken Nuggets. Rice. Microwave. Modification. Flour.

7.2. Introduction

The market for coated products is gaining more interest in expanding and improving the application of coating materials to food materials such as meats, vegetables and dairy products such as cheese. It has been estimated that over 1.2 billion lb of food products are coated in North America, most of which was poultry, followed by seafood (e.g., shrimps) and vegetable products such as onion rings. One of the most astounding commercial applications of coating materials with relation to poultry products has been the chicken nugget which was introduced to the tables of North American consumers by the fast food industries in early 1970 (Barbut, 2013). Typically, the product was made from a whole breast muscle strip which was battered, breaded and fried. Now, nuggets can be made from an array of various meats (poultry, fish, and pork), and cuts (ground meat, whole meat, and portions of dark and white meat).

Generally, nuggets have been produced commercially and domestically by deep fat frying. Deep fat frying is a process of cooking and drying through immersive contact with hot oil, which eventually leads to instantaneous heat and mass transfer (Voong et al., 2018). However, the ever-changing consumer need for fried food products with reduced fat contents and increased retention of oil-soluble vitamins, has led to food processors coming up with a novel means of frying know as air frying. This new process of frying involves spraying oil on the surface of the food and rather than immersing the food product in the hot oil, the hot air is utilized as the medium for heat and mass transfer. During both frying techniques, an array of physical and chemical changes arises such as protein denaturation, starch gelatinization and crust development (Ghaitaranpour et al., 2018). The browning reactions taking place between protein fractions and reducing sugars, the degree of absorption of frying oil, the temperature of heating, the density of fried products, frying technique and duration are all factors responsible for color development during the frying process.

Flour coating plays a major role in the acceptability and quality of fried food products. The quality of flour coating is based on the uniformity and thickness of the coating and its adhesion to the product, along with its influence on the overall color, appearance, taste, and crunchiness of the fried product (Román et al., 2018). The use of hydrocolloids such as rice and wheat flour as coating agents is generally due to the ability of the flours to absorb and hold water, thereby acting as regulators of viscosity. Flours also add to the coating potentials by enhancing adhesion to the food material and stability to freeze-thawing, eventually improving the textural properties, preserving freshness and limiting oil absorption during frying (Loewe, 2011).

Today's industry has seen a surge in the application of pregelatinized hydrocolloids in food coating formulations primarily due to the increasing coating pick up of these coating agents. It is thus important that the application of a flour modified via thermal modification treatment such as microwave be studied. Other than starch pregelatinization, microwave processes also lead to starch disintegration, protein denaturation, Maillard reactions and enzyme (in)activation, whose degree is related to the severity of the modification process (Román et al., 2015; Uthumporn et al., 2016). The intensity of microwave treatment, which is linked to moisture content, microwave power and duration of the process, changes flour properties by altering their hydration potential as well as its rheological and thermal characteristics (Uthumporn et al., 2016). Hence, microwave treated flour with a higher degree of pregelatinization possesses a better thickening potential in cold conditions than native flours, along with improved water absorption and retention potentials, yielding comparable functionality to that of pregelatinized starches (Román et al., 2015). An added benefit of microwave flours is that both processes are sustainable physical treatments that permit the change of flour functionality while still sustaining a clean non-toxic label (Eckhoff & Watson, 2009). A further case can be made that there is a reduced environmental influence and cost in producing pregelatinized flours as compared to that of starches (Román et al., 2018). Furthermore, knowing that the kind of flour employed strongly influences the development of color in thermally processed foods, such as bread, it is expected that the type of flour used in the development of fried coated products would also impact its final color. Furthermore, it is essential to mention that, in this study, the modified flour are utilized as coating agents at different temperatures, during which processing Maillard and caramelization phenomenon are due to happen, which can, in turn, further modify the color profile of the fried product.

Despite the fact that pregelatinized flours pose both industrial and economic benefits, the influence of microwave treated flours on fried meat products during frying has never been assessed. Therefore, the main objective of this study was to evaluate the influence of microwave treated flours on the quality of prepared fried meat products and conduct a comparative study on the quality changes during air frying.

7.3. Materials and methods

7.3.1. Materials

Fresh skinless, deboned chicken breast fillets (200 ± 8g) and vegetable oil (Canola Harvest, Richardson International, QC, Canada) purchased from a local supermarket were used in this study. Native rice cultivar (SAH 329) from Senegal was purchased from a local market in Dakar, Senegal. The raw rice grains were milled using a coffee grinder (SUMEET Multi Grind, India) and the flour was later filtered through a 250μm sieve before further use.

7.3.2. Rice flour modification

SAH 329 rice flour was dispersed in distilled water to attain a final solid concentration of 40% on a dry weight basis. The non-waxy rice flour dispersion was homogenized by using a magnetic stirrer for 10 minutes at a low speed. Microwave treatment of the flour dispersion was carried out with a Panasonic NN-SN651W (Panasonic Corporation, Osaka, Japan) microwave oven (2450 MHz, power 1200 W) for 15 secs in Pyrex cylinders open at both ends (50 x 40 mm) at a power level of 850W. The cylinders were covered with Saranwrap ® to control moisture loss during the thermal process. Emphasis was made to place the sample at the same place in the microwave for each treatment.

7.3.3. Frying process

The dimensions of the chicken breast cuts samples were about 3cm (length) x 3 cm (width) x 1.5 cm (thickness) (± 0.2 cm). The individual chicken cuts were pre-dusted with un-modified and modified rice flour. Coated chicken cuts were fried by air frying. The air frying studies were done for 0, 4, 8, 12 and 16 mins at three temperatures (170, 180 and 190°C). No emphasis was made to rotate the air fried samples during air frying as hot air enveloped all parts of the product's surface. The internal temperature of the chicken cuts was measured using a digital K-type thermocouple. At the end of both frying processes and after reaching the ambient temperature, the samples were blotted with a paper tissue to remove surface oil and packaged.

7.3.4. Coating pick-up

The amount of coating pick-up of the sample prior to frying was determined by the weight of the coated sample divided by the weight of the sample before coating multiplied by 100.

7.3.5. Texture

The shear force was determined using the razor blade method previously described by (Barbut, 2013). Briefly, a 9mm craft knife is applied to shear the surface of the product while positioned at 90° to the chicken cut direction; each cut was analyzed at 4 different points. The blade was mounted on a texture analyzer (Model TA.XT2, Stable Micro Systems, Texture Technologies Coro., Scarsdale, NY) and moved down 15 mm at 1.5mm/s.

7.3.6. Moisture and fat content

Moisture content was determined as the mass of moisture in the sample to the overall mass of the product sample on a wet mass basis (wb). Fried samples were taken at different sampling intervals and heated in an oven (Isotemp 700, Fisher Scientific, Pittsburgh, PA) at 105°C for 24 hours and the difference in mass before and after was used for the analysis. For the fat analysis, fried samples were freeze-dried and ground in a coffee grinder (SUMEET Multi Grind, India). 3 g of the grounded samples were weighed into a thimble for fat extraction in a Soxhlet solvent extractor (SER, Velp Scientifica, Usmate, Italy) using petroleum ether. Fat content was noted as the ratio of the mass of extracted fat and dry matter of the sample.

7.3.7. Cooking yield

Cooking loss was determined as the percentage loss before and after cooking. For coated samples, the pre-cooked weight used included the meat in addition to the coating agent.

7.3.8. Color analysis

The color profile of the coated fried chicken cuts was determined using a Konica Minolta spectrophotometer (model CM-3500d, Osaka, Japan) using the L*, a* and b* color scale. Total color change (ΔE) was evaluated from the following equation in which white color was used as a reference point, which was denoted by L_0, a_0, and b_0.

$$\textit{Total color change } (\Delta E) = \left[(L^* - L_0)^2 + (a^* - a_0)^2 + (b^* - b_0)^2\right]^{0.5} \quad (1)$$

7.3.9. Porosity

Porosity (Ɛ) was estimated following the methods described by Adedeji and Ngadi, (2011). Porosity was defined as the ratio of bulk density, ρ_b, to apparent density, ρ_a, subtracted from one. That is to say the ratio of the void volume present in the sample after frying to the overall volume.

$$\varepsilon = (1 - (\rho_b/\rho_a)) \times 100 \quad (2)$$

To estimate apparent density (ρ_a), the apparent volume of the samples was first estimated. Fried lean meat portions were kept for 20 minutes at room temperature to cool down and their apparent volumes were evaluated (volume of the sample including closed pored but excluding the open pores). The apparent density of the weighed lean meat samples was analyzed in a helium pycnometer (Model 1305 Multivolume, Micometrics Instrument Corporation, Norcoss, GA) following the set methodology (Adedeji and Ngadi, 2011). The study was carried out in triplicate. Apparent density was estimated as the ratio of the mass of the sample divided by the apparent volume.

After the apparent density was measured, the same set of samples used for apparent density were weighed and then immersed in melted paraffin wax in order to fill up the surface openings of the samples. The sample was then cooled at room temperature. The difference between the weights of the samples before and after immersing the samples in paraffin wax was taken as the mass of paraffin wax. The volume of the paraffin coating layer was estimated from the mass and density of paraffin. The variation between the volume of paraffin wax and the volume of the sample dipped in paraffin wax was defined as the bulk volume of the sample. The bulk density (ρ_b) was estimated as the ratio of the mass of the sample divided by the bulk volume.

7.3.10. Statistical analysis

All experiments were carried out in triplicate. Obtained data were analyzed by one-way analysis of variance (ANOVA), and significant differences among mean were analyzed by Duncan's multiple range test at $P < 0.05$, using JMP software (Version 14; SAS, NC, USA).

7.4. Results and discussion

7.4.1. Coating pick up

The coating composition plays a big role in the amount of coating picked-up by the lean meat portions. The percentage pick up of the microwaved rice flours was observed to be significantly higher (89.70 %) than those of the control sample (49.97 %). This increased coating pick up is probably linked to the increased water binding potential of the pregelatinized flour.

7.4.2. Shear force

Figure 7.1 shows the influence that the different flour types had on shear force of air fried lean meat portions at different frying temperatures. Shear force was observed to increase linearly with frying time. The linear increase in the shear force can be attributed to the closing of the small spaces between the muscle fibers and the compactness of the meat surface caused by continuous heating. Air frying with control flour (at 180^{o}C) after 8 mins resulted in little or no changes in the shear force values. There was no significant difference in the rate of increase in the shear forces of the control and microwaved air-fried portions. Application of microwaved rice flours was observed to improve the texture development of fried lean meat products due to the improvement of the film forming characteristics of the coating agents. After 16 min of air-frying (at all the studied temperature), the control coating samples was shown to be crisper, with the highest shear force value. Because of its linear molecular organization, this high amylose coating provided a crisper and more brittle layer to the air fried product (Altunakar et al. 2004). During air frying, swelling of flour granules releases the amylose regions and creates a film barrier that does not permit oil penetration and moisture loss from the lean meat portions. The production of the film barrier and gelatinization play a key role in the development of crispiness and texture of the finished fried product. In addition, the increased force to penetrate the air fried product was probably due to the moisture loss of the product.

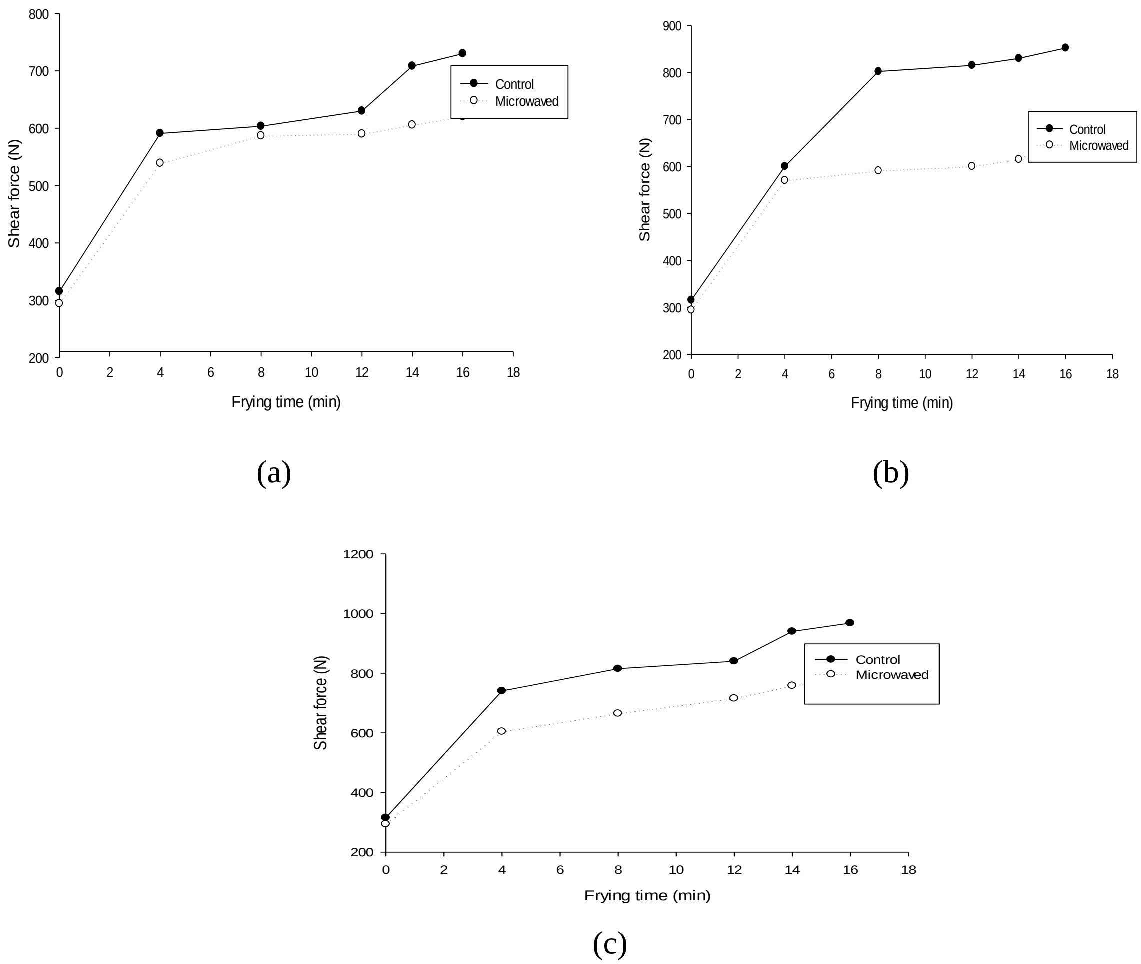
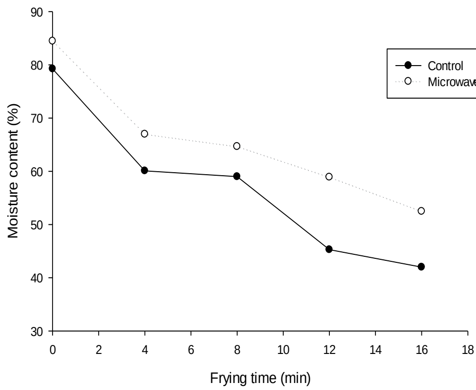


Fig 7.1. Effects of flour types (control and microwaved) on the shear force of air-fried lean meat portions during frying at (a) 170^{o} C (b) 180^{o} C and (c) 190^{o} C

7.4.3. Water and oil contents of the coated fried products.

Figure 7.2. reports on the influence the different flour coatings had on the moisture content of the air-fried lean meat portion. Rice protein has the ability to create a viscoelastic framework after hydration with can subsequently reduce the moisture content. Proteins have been reported as the major component responsible for the viscoelastic properties of flours; although the starch association with proteins in the flour matrix have displayed an important function as well on the viscoelasticity of flours (Dixit and Bhattacharya, 2015; Ibáñez et al., 2007). During air frying, processing at a higher temperature such as 190 °C may have resulted in a greater starch swelling and protein denaturation in the control coated samples, which subsequently breaks down the structure of the film produced by the flour and subsequently reduced moisture content values. Mean separation showed that the moisture content was found to decrease with frying time for both coated samples. Generally, for all the coated samples, the temperatures during frying in this study influenced the moisture content of the fried product. As temperature increased the rate of moisture loss increased. Analysis of variance of the data points revealed that the type of flour used had a significant influence on the moisture content values ($P < 0.05$). At all frying temperature, the microwaved coated fried lean meat portions displayed the highest moisture content due to it's higher water absorbing potential. The incorporation of microwaved rice flour coating during air-frying, therefore, can aid much more efficient moisture retention in fried products.



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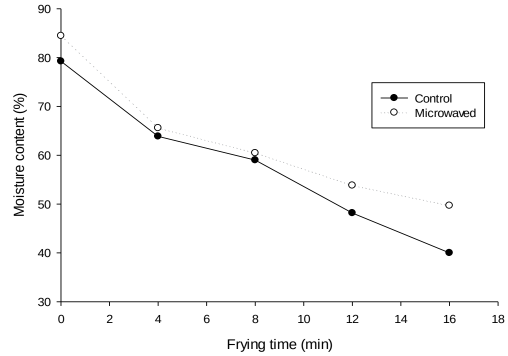

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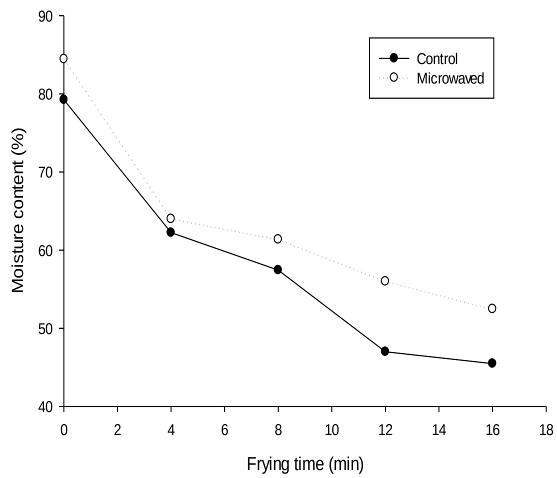
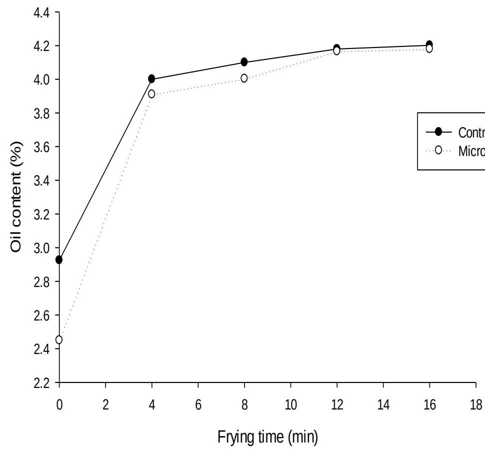


(c)

Fig. 7.2. Effects of flour typed (Control and Microwaved) on moisture contents of air-fried lean meat portions during frying at (a) 170^{o} C (b) 180^{o} C and (c) 190^{o} C

The influence of the application of the different flours used as coating agents on the oil content of air-fried lean meat portions is shown in Figure 7.3. The data of this study revealed that oil uptake increased with increasing frying time. Statistical evaluation of the oil uptake plots showed that the coefficient of determination was at least 0.92.



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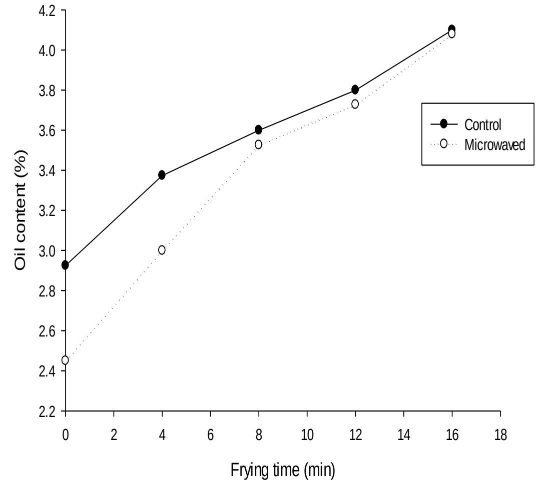

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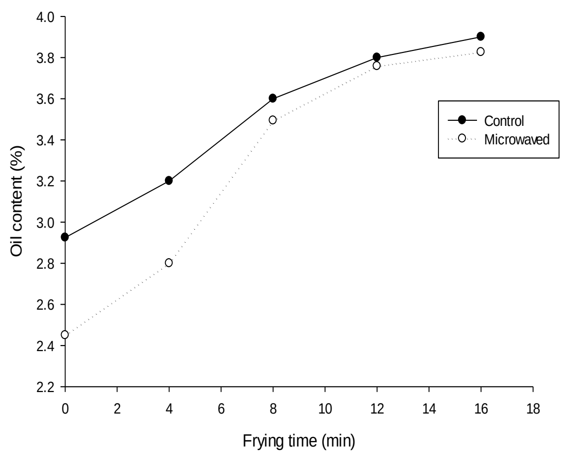


(c)

Fig. 7.3. Effects of flour type (Control and Microwaved) on oil contents of air-fried lean meat portions during frying at (a) 170^{o} C (b) 180^{o} C and (c) 190^{o} C

Duncan's multiple range test carried out on the data of this study revealed that the application of microwaved rice flour reduced the oil content of air-fried lean meat portions significantly. Air frying was shown to significantly increase the oil contents of the coated products ($P < 0.05$). The increased coating pick up, which relates to a more efficient coverage on the surface of the lean meat portions may be responsible for the controlled in and our movement of oil into the product when microwaved gelatinized flours were applied as a coating agent. Microwaved flour was observed to have the lowest oil contents. It is clear that there was a negative correlation between the moisture content and oil contents of the samples (r = 0.72). Gelatinization of the starch in the rice flours during air frying play a vital role in reducing fat contents (Kawas and Moreira, 2001). This is because the starch gelatinization tends to create a barrier that limits oil absorption in the product. When flour coated products are air fried at temperatures above 90 °C, elastic networks are produced. Proteins, starch, and lipids are involved in the formation of these elastic networks. The degree of gelatinization can limit and/or control the functionality of the created elastic film network when it comes to oil permeability into the fried product.

The decrease in the moisture content and the increase in the oil content could be attributed to the break down of the coating materials and meat structure during air frying. Denaturation in meat protein during air frying leads to an increase in moisture loss and hardening of the product's texture. The reason for the high moisture content and low oil content of the microwaved coated lean meat portions can also be related to its high coating pick up. The increased coating pick up leads to an increased coating structure and retards moisture migration from the lean meat portions. Therefore, making the microwaved coated lean meat portions more juicy in comparison to that coated with the untreated rice flour.

7.4.4. Cooking yield

Figure 7.4 shows the effect that flour types have on the cooking yield. The cooking yield provides a picture on the extent of adhesion of the coating agent during frying. The role of improved adhesion of coating agents provides an economic advantage to food processors. The results of the cooking yield in this study revealed that the microwaved rice flour was the most effective ingredient at all tempeatures tested. The microwaved flour provided a better barrier against mass transfer from the food matrix.

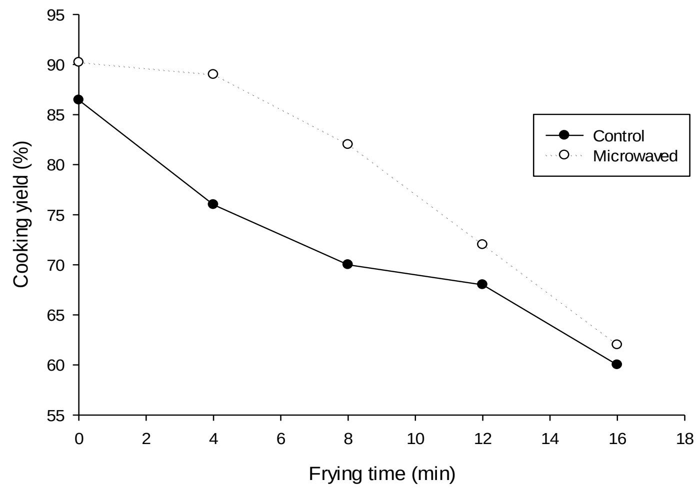
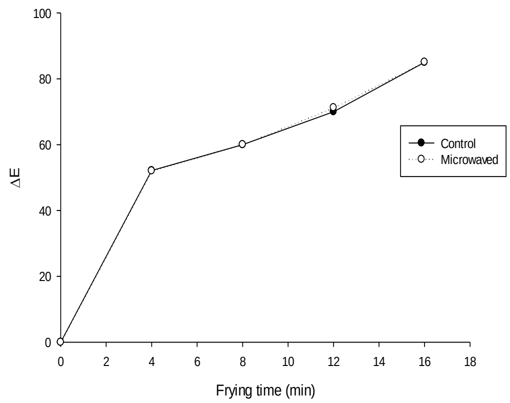


Fig. 7.4. Effects of flour type (Control and Microwaved) on cooking yield of air-fried lean meat portions during frying at 190^{o} C.

7.4.5. Total color

Flour type was observed to have no significant impact on color (ΔE) development of air-fried lean meat portions as can been seen in Figure 7.5 Interestingly, the color development of the coated lean meat portions (control and microwaved coated) occurred quickly (within the first 4-5 min) and then remained fairly stable until the lean meat cuts were fully cooked inside. At all studied temperatures, as frying time increased ΔE value increased. Figure 7.5 shows the increase in ΔE at all the studied frying temperatures. The temperature of frying had a significant influence on the extent of color developments of the air-fried lean meat portions. The coefficient of determination values were at least 0.90. The amounts of proteins and reducing sugars in the coating mix could be responsible for the Maillard reactions on the crust during the air frying. The results of this study revealed that no significant diffrence in color development as a function of flour type.



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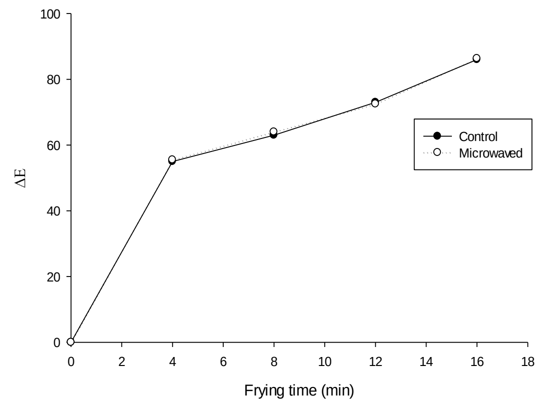

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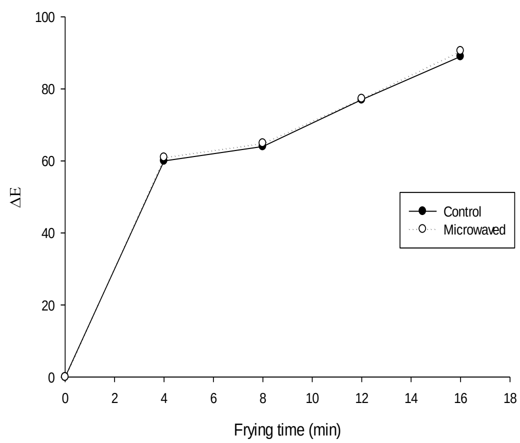
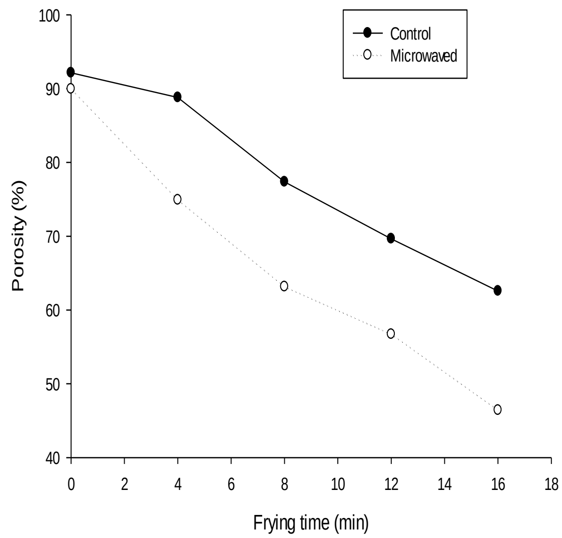


(c)

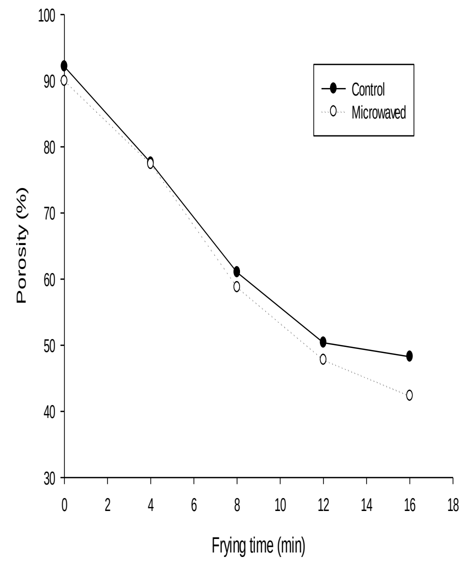
Fig. 7.5. Effects of flour type (Control and Microwaved) on the color of air-fried lean meat portions during frying at (a) 170^{o} C (b) 180^{o} C and (c) 190^{o} C

7.4.6. Porosity

Our investigations showed that at all the studied frying temperatures, porosity increased with frying time (Fig 7.6). Visual observations of fried lean meat portions coated with the native and microwaved flours revealed shrinkages and swelling of the product pore's structure. Adedeji and Ngadi, (2011) have suggested that the increase in porosity is positively related to oil uptake during frying. The type of coating used in the study significantly influenced the porosity (P<0.05) of the fried lean meat cuts. The fact that porosity values of the microwaved coated fried lean meat cuts were different from that of the control coating suggests that there is a difference in oil-uptake mechanisms for coatings due to the film forming properties of different flour types. This authenticates the importance of the coating type in controlling porosity and oil retention during air frying. The results of this work also showed that frying temperature also significantly influenced the porosity values. The interaction effect of temperature level and frying time on porosity at a confidence interval of 90% was significant. Mean evaluation of the data in this study revealed that there is significant variation (P<0.05) between porosities at 170°C, compared to the values at 180 and 190°C. Duncan's new multiple range test (MRT) at 4-16 min was carried out to compare the impact of frying temperature on porosity. Control coated samples displayed significantly (P < 0.05) greater porosity than microwave coated rice flour products at 170, 180 and 190 °C. Porosity values for air-fried lean meat cuts coated with untreated rice flours ranged from 25.17 – 88.90%, while that of air-fried lean meat cuts coated with microwaved rice flours ranged from 13.99 – 77.38%. The ranges suggest that the native and microwaved flours were made up of several constituents such as water and air pockets. During air-frying, minimal water was lost during frying, thus producing less pores on its way out of the meat product. The results of this study suggest that more pore spaces were observed in the control coated product in comparison to the microwave coated fried products. Some of these created pores in both untreated and microwave coated fried products were thereby satiated with fat during thermal processing. The high porosity recorded in the control coated fried products was associated greatly to water that was removed prior to analysis. This observation was substantiated by the reduced moisture content observed in this study. It is a fact that the more frying time, the less free water available for removal in the fried product, therefore the reducing pattern in porosity (Adedeji and Ngadi, 2011). The pronounced higher porosity observed for control coated samples could be associated also with the degree of film produced because of flour gelatinization during air-frying. The created film produced during thermal frying can influence moisture retention and loss during air-frying, subsequently controlling moisture loss during air frying and pore development. The results of this study are in agreement with those of other authors (Altunakar et al., 2004; Ghaitaranpour et al., 2018).



(a)



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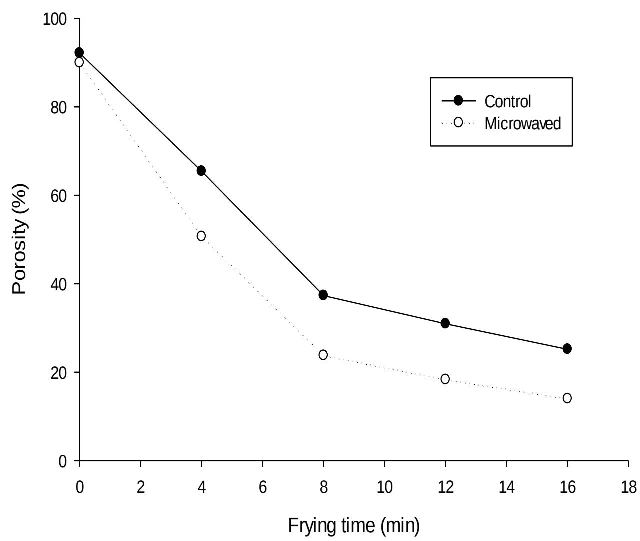


(C)

Fig. 7.6. Effects of flour type (Control and Microwaved) on the porosity of air-fried lean meat portions during frying at (a) 170^o C (b) 180^o C and (c) 190^o C

7.5. Conclusion

Application of coating treatments on the oil uptake, moisture loss and some physicochemical properties of lean meat portions during air frying was examined. The results of this study indicate that modification of non-waxy rice flour could be of interest to food manufacturers in producing coated chicken products. Coating pick up was found to be influenced by the water binding potentials of the coating flour type. Samples coated with microwaved non-waxy flours displayed a higher coating pick up, in comparison to samples coated with untreated rice flour. Shear force, oil content, and color of the lean meat cuts increased during air frying. A correlation was made between the moisture and oil content of lean meat portions.

In conclusion, coating lean meat portions with microwaved flour led to improved quality of the lean meat cuts in terms of reducing oil absorption, increasing cooking yield and coating pick up. In conclusion, the application of microwaved rice flours can be recommended as a coating agent for air-fried foods.

CONNECTING TEXT

In chapters 7, the impact of microwave-treated flours on the textural, porosity, oil and coating pick up of chicken breast cuts coated with microwave flours were studied. The results showed that this physical modification process improved the quality of the coated fried meat products. In chapter 8, the influence of ultrasound processing on the physicochemical, rheological, physicochemical and thermal properties of SAH 329 and SAH 177 was studied. Data obtained will be used in evaluating the potential usage of ultrasound treated flours for food applications.

8. Ultrasound processing: Influence on the physicochemical, rheological and thermal properties of high amylose rice flours.

8.1. Abstract

Ultrasonication was applied to two rice flours, namely SAH 329 and SAH 177 from Senegal at various treatment times (60, 360, 1080 and 1800 sec), moisture contents (15 and 25%) and ultrasonication amplitudes (20 and 30%). The amylose content, protein contents, water, and oil-holding properties, swelling power, thermal and rheological properties of the ultrasound treated rice flours were examined. Differential scanning calorimetry was used to examine the onset, peak and enthalpy properties of the rice flours. The results of this study revealed that ultrasound processing of the rice flours increased the amylose content of the rice flours. The data of differential scanning calorimetry analysis revealed that increasing ultrasound processing time led to a decrease in the onset, peak and enthalpy properties of the rice flours. The G′ (storage modulus) and G″ (loss modulus) properties increased stepwise jointly with the increasing ultrasound treatment time. Ultrasonication significantly ($P<0.05$) increased swelling power and water holding properties, whereas protein contents and oil holding properties declined with ultrasonication. The variations of all these properties during ultrasound treatment were linked to the moisture contents of the flour dispersion and cavitation impact created by ultrasound-induced bubbles on the flour granules, which were clearly observed in the rice flours.

Keywords. Ultrasound. Flour. Rheology. Starch. Modification.

8.2.Introduction

Rice flour properties have been understudied for many years. Native rice flour rarely provides the desired functionality and unique physicochemical properties required by food processors to produce food products. Development of new food products usually requires modification of these native rice flours via chemical, enzymatic, genetic and physical processes. Of recent, physical modifications are gaining increased attention in food industries due to the reduced dependency on chemical agents (Carmona-García et al., 2016). Ultrasonic processing provides an effective technique of flour modification, as the process displays an array of advantages in terms of product quality, reduced use of chemicals and less processing time, and therefore considered as eco-friendly processing. Ultrasonic waves are produced by sound energy above the frequency audible for humans (18–20 kHz). In general, sonication treatment can be applied to the native rice flours suspended in solution or on the gelatinized flour (Meng et al., 2014; Yue Zuo et al., 2009). Ultrasonication treatment typically disorganizes the amorphous region of starch granules in the flour matrix. This disruption has been observed to have little or no impact on the shape and size of the amorphous regions in the flour matrix, but their surface becomes permeable and therefore improves the physicochemical characteristics such as the swelling power, water holding capacity, oil holding capacity and rheological properties (Sujka and Jamroz, 2013). To the food processor, ultrasonic treatment has been seen as a beneficial technology as the treatment has offered several advantages such as increased product volume, enhanced product quality, reduced processing time and maintenance costs (Patist and Bates, 2007).

The strength and efficiency of the ultrasound process have been shown to depend on various variables, such as sonication amplitude, power, time and temperature during treatment, and the properties of the flour dispersion, namely moisture and or flour concentration and biological origin of the flour (Kaur and Gill, 2019). During ultrasonication, the reduction of pressure produces bubbles of gasses in the cavities which collide with the flour granules before the matrix collapses as the pressure increases (Awad et al., 2012). The phenomenon is referred to as cavitation. The swift break down of the bubbles created during ultrasonication leads to the production of shear forces in the medium. The shear forces produced by the process disorganizes the polymer chains in the flour matrix such as starch and protein. In addition, the formation of radicals as a result of solvent molecules dissociations during ultrasonication can lead to polymer degradation (Setyawati et al., 2016; Sujka and Jamroz, 2013). Consequently, these structural alterations on the polymers further influence the physicochemical properties of flour.

In developing countries like Nigeria and Senegal, non-waxy (non-glutinous) rice is a major staple. It is predicted that the world demand for this type of rice will increase by 40% by 2030 (Khush 2005). Despite the expected surge in worldwide demand for this rice type, the flour produced from non-waxy rice has a lower industrial potential when compared to the flour of waxy-rice types in the food processing industry. The industrial applicability of the non-waxy rice types has been related to the physicochemical properties of the starch properties in the flour's matrix (Wani et al. 2012). As flour is a key element utilized in several foods and industrial applications, therefore, there is a need to evaluate and summarize the influence of ultrasonication on the physicochemical, functional, chemical and rheological properties of flours from a non-waxy origin.

8.3.Materials and methods

8.3.1. Materials

SAH 329 and SAH 177 rice grains were procured from a local market in Dakar, Senegal. The grains were stored at room temperature until further use.

8.3.2. Flour production

Flours from the different non-waxy rice grains were produced by milling the grains with a coffee grinder (SUMEET Multi Grind, India) and the produced flour was filtered through a 250µm sieve before further use.

8.3.3. Ultrasound treatment

The flour samples were dispersed in distilled water to attain final solids concentrations of 15 and 25% (dry weight basis). The dispersions for ultrasound treatment were homogenized using a magnetic stirrer for 10 minutes at a low speed. The flour dispersions were then sonicated using a Sonics sonicator (VCX 500, Newtown, CT, USA) for 60, 360, 1080 and 1800 secs in a flat-bottomed conical flask at an ultrasound power of 500 W and amplitude levels of 20 and 30%. During treatment, the vessels containing the flour samples were held in an ice bath to control any rise in temperature by ultrasonication. Following sonication, the flours were centrifuged (3000 x g) and dried in a freeze dryer.

8.3.4. Amylose content

Isolation of starch from the non-waxy rice flours was carried out using a slightly modified version of alkaline deproteination procedure described by (Lii et al.,1996). Amylose content was estimated using a commercial amylose/amylopectin assay kit from Megazyme (Ireland) according to the methodology described by (Gunaratne and Hoover, 2002).

8.3.5. Protein content

Crude protein content was analyzed by combustion using a LECO Nitrogen Analyzer (CNS 2000, St. Joseph, MI, USA). 100mg of flour sample was placed into a porcelain sample holder (boat) for introduction into the combustion chamber (1300°C) of an automated sample loader. The amount of protein present in the samples was evaluated by multiplying the nitrogen content produced as a result of combustion by a factor of 6.25.

8.3.6. Color properties

A colorimeter (CR-10, Konica Minolta Sensing Inc, Osaka, Japan) was used in estimating and comparing the color changes in the samples under different treatment conditions. The color scale used was L*, a*, and b* CIE color values. The overall color change (ΔE*) were measured from L*, a*, and b* values and then compared to those with the untreated sample.

Total color change (ΔE) = $[(L^* - L_{ultr})^2 + (a^* - a_{ult})^2 + (b^* - b_{ult})^2]^{0.5}$ *(1)*

8.3.7. Oil and water holding properties

Oil holding capacity (OHC) was determined by the method of de la Hera et al. (2013). Rice flour (100.0 ± 0.2 mg) was mixed with 1.0 ml of vegetable oil. The resulting mixture was homogenized for 1 min with a wire rod to dissolve the sample in the oil. After 30 min in the vortex mixer, the mixture was centrifuged (3000 × g) at 4°C for 10 min. The supernatant was decanted, and the holding vessels held upside down for 25 min to trench the oil. After this, the resulting residue was referred to as W_r. The oil holding/absorption capacity was expressed in grams by dividing the resulting residue weight by the initial weight of the sample.

The water holding capacity (WHC) was measured by dispersing 1.000 ± 0.005 g of flour with distilled water (10ml) and allowing the resulting mixture to rest for 24 hours at ambient temperature. The supernatant was decanted. WHC was stated as grams of water held per gram of solid.

8.3.8. Swelling power

Swelling power of the rice flours was analyzed following the method of De la Hera et al. (2013). Briefly, the flours (2.00 ± 0.1 mg) were mixed in 1.0 ml of distilled water in an Eppendorf tube and cooked at 90˚C for 10 minutes in a water bath. The cooked paste was cooled in an ice water bath for 10 minutes and then centrifuged at 3000 × g at 4˚C for 10 min. The supernatant was decanted into an evaporating dish and the dry solids were recovered by evaporating the supernatant with a Thermo savant freeze dryer (model ModulyoD) till constant weight is achieved. Three replicates were carried out for each sample. Residues (W_r) and dried supernatants (W_s) were weighed and Swelling Power (SP) was estimated as follows:

$$SP\left(\frac{g}{g}\right) = \frac{W_r}{W_i - W_s} \tag{2}$$

8.3.9. Thermal studies

Onset of gelatinization properties (T_o), gelatinization temperature (T_p) and enthalpy of gelatinization (ΔH) were examined using a differential scanning calorimeter (DSC Q2000; TA instrument, New Castle, PA, USA). 2.5 mg of the sample was weighed into an aluminum pan and thoroughly mixed with distilled water to obtain a sample -water suspension of 2:1. The samples were sealed and allowed to attain equilibrium before heating in the DSC. The sample pans were heated at a rate of 5 ˚C /min from 25 to 125 ˚C.

8.3.10. Rheological properties

For the preparation of flour dispersion (8% w/w), rice flour was mixed with distilled water. The mixtures were thoroughly stirred for 30 min at ambient temperature and then heated at 95°C in a water bath for 30 min with mild agitation carried out by a magnetic stirrer. Immediately after heating, the cooked rice flour paste was immediately transferred to the rheometer to estimate the steady and dynamic rheological characteristics of the treated samples.

Dynamic shear properties such as storage modulus (G') and loss modulus of the prepared ultrasound treated rice flour dispersions was carried out also with a rheometer (AR 1000, TA Instruments, New Castle, DE) using a parallel plate system (4cm diameter) at gap of 500µm at 25°C. Dynamic shear data obtained were from frequency sweeps over the range of 0.01 – 10 Hz at 2% strain.

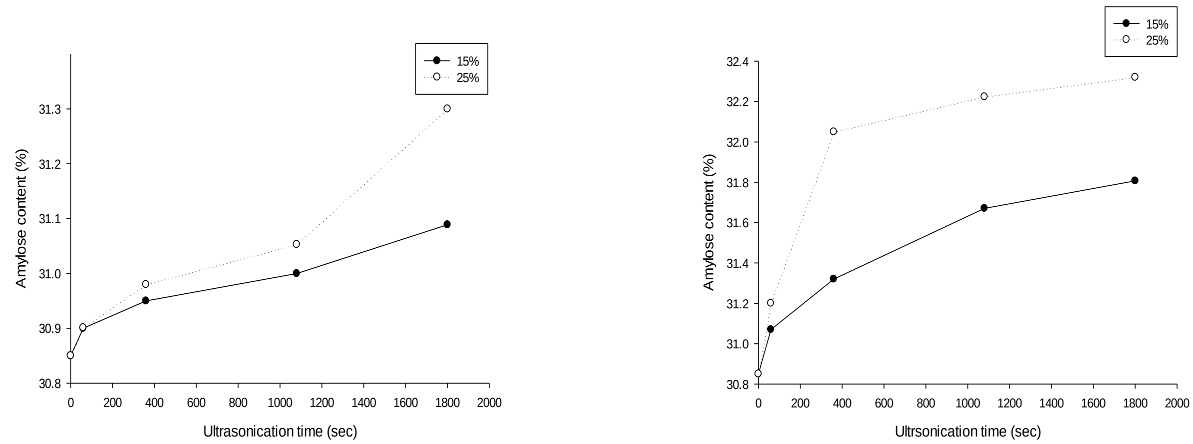
8.3.11. Statistical analysis

The data reported in all the tables are averages of triplicate observations. The results were evaluated using Analysis of Variance (ANOVA) and expressed along with the standard error of the mean value. The averages were compared by Fisher's least significant difference (LSD) test, and differences at $P < 0.05$ were considered significant. The data were subjected to statistical analysis using JMP Statistical Software version 17 (Version 14; SAS, NC, USA).

8.4. Results and discussion

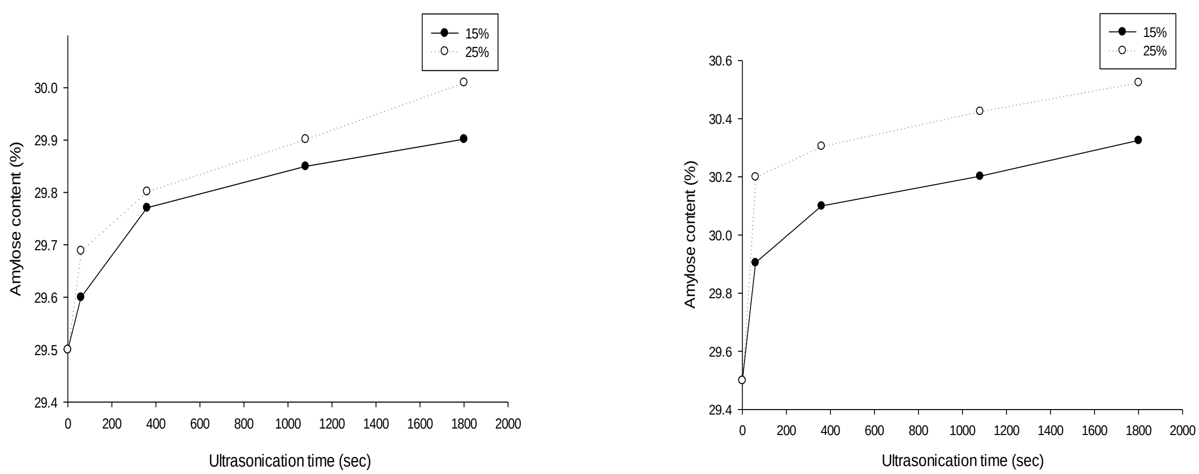
8.4.1. Amylose content

The amylose content of SAH 329 and SAH 177 flours as impacted by the amplitude levels (20 and 30%), ultrasonication time (60, 360, 1080 secs and 1800 secs) and moisture contents (15 and 25%) are shown in Fig 8.1 and 8.2 For both flour types, the highest amylose content was observed for flours with 25% moisture content. This data could be attributed to the increased effect the moisture activity had on the phosphate groups of the amylopectin molecules. The degree of bonding within the crystalline regions may have been weakened by phosphate groups during ultrasonication at increased moisture content. The process time during ultrasonication also significantly ($P< 0.05$) increased amylose content of both rice cultivar flours. Statistical analysis also suggested that the amplitude during ultrasonication had a significant ($P < 0.05$) impact on the degree of amylose increase of both flours during ultrasonication. These changes in amylose content could signal the physical and chemical crippling of the starch granules in both flour matrices. It is noteworthy to say that the amorphous regions in the starch granules are specifically broken down by cavitation during ultrasonication, probably due to its poor structural integrity (Sujka and Jamroz, 2013). Similar observations on the depolymerization impact of ultrasound treatment have been reported by Sujka and Jamroz, (2013) for potato, rice, wheat, and corn starches. This disruption subsequently results in the release of amylose into the aqueous medium, thereby increasing the amount of available amylose content in the flour.



(a) (b)

Fig 8.1 Amylose content variation of SAH 329 rice flours as a function of amplitude powers of 20 % (a) and 30% (b).



(c) (d)

Fig 8.2. Amylose content variation of SAH 177 rice flours as a function of amplitude powers of 20 % (a) and 30% (b).

8.4.2. Protein content

Table 8.1 shows the impact of ultrasonication on the protein contents of the studied rice flours. Previous studies have evaluated the influence of ultrasonication on the digestibility and structure of food proteins (Chandrapala et al., 2012; Jiang et al., 2014; Majid et al., 2015). However, to our knowledge, no one has yet to evaluate the quantitative impact ultrasonication has on the protein content of rice flours. Comparison of the protein contents at any given amplitude or moisture content (Table 8.1) revealed that the protein content of the ultrasonicated samples was higher. Processing time significantly ($P < 0.05$) reduced the protein contents of the studied flours. This result indicates that ultrasonication induced a certain degree of molecular unfolding on the proteins in the rice flour, and subsequently lead to the decrease in the number of hydrophobic interactions that are originally present inside the protein molecules. Ultrasonication induces cavitation that disrupts non-covalent hydrophobic interactions and intermolecular disulfides linkages in the polypeptides of the protein molecules. The functional native arrangement and quantity of proteins are determined by the subtle balance between many noncovalent interactions; this equilibrium can be easily disrupted by sonication, leading to protein denaturation and aggregation (Jiang et al., 2014).

8.4.3. Color properties

Control of the overall color after and during food processing is a key factor that defines consumer acceptability of food products. Impact of ultrasonication on overall color (ΔE) of the rice flours is presented in Table 8.1. It was observed that ultrasonication significantly ($P < 0.05$) influenced the color. With the extension of ultrasonication time, the overall color values gradually increased. The rate of increase was significantly ($P < 0.05$) influenced by the variation of moisture contents and amplitudes of ultrasonication. No study has evaluated the influence of ultrasonication on the overall color of rice flours. However, it is well known that ultrasonication produces cavitation which in turn could damage the pigments structures in the rice flour. Lieve et al. (2006) suggested that the overall color of rice flour is related to the concentration of pigment in the flour. Also, ultrasonication has been shown in this study to influence the protein content of the rice flours. The darkening effect of processed rice flours has been related to Maillard reactions between reducing sugars from starch and the amino groups in proteins fractions of rice flour (Lee et al., 2015). The suggested absence of this functional group as a result of ultrasonication may have influenced the number of amino groups available for browning in the rice flour.

8.4.4. Water and Oil holding capacity

The data illustrating the influence of ultrasonication on the water and oil capacities of SAH 329 and SAH 177 rice flours are presented in Table 8.1. The observations of this study showed that there was a significant ($P < 0.05$) change in both properties with increasing processing time. As shown in table 8.1, the water holding capacities were increased 1.02 – 1.65% while the oil holding capacities were reduced by around 8.50 – 48.20% after ultrasonication treatments. A similar influence of ultrasonication on the water and oil holding properties on plum seed isolate have been reported by (Xue et al., 2018). Higher water capacities and reduced oil capacities values were reported in the 15% (moisture content) flour samples sonicated at 30% amplitude. In fact, the results of this study suggested that the extent of ultrasonication on the structure and functional groups may depend on the amplitude during ultrasonication, ultrasonication processing time and flour to water ratio. The changes in the water and oil holding capacities could be related to the greater impact of moisture content on ultrasonication and the molecular organizations of the protein component in the flour material. Ultrasonication leads to denaturation of the protein molecules and subsequently alters the hydrophobic and/or hydrophilic groups in the protein structures (Jiang et al., 2014; Xue et al., 2018). In addition, ultrasonication may have caused changes in the physical geometry of pores and channels on the surface of flour granules permitting water molecules to infiltrate more easily into the large volume of the granule, thereafter increasing the water holding capacity of the granules in the flours.

Table 8.1. Protein, overall color, oil and water holding properties of ultrasound treated SAH 329 and SAH 177 rice flours

Amplitude level			20%				30%			
Cultivar Type	MC %	Time(sec)	Protein (%)	ΔE	OHC (g/g)	WHC (g/g)	Protein (%)	ΔE	OHC (g/g)	WHC (g/g)
SAH 329	15	0	9.24a	0.00a	1.27e	1.22a	9.24e	0.00c	1.27a	1.22e
		60	8.09b	3.19b	1.21a	1.25b	7.81b	3.67a	1.18b	1.29a
		360	7.50c	3.73c	1.17b	1.32c	7.17a	3.96b	1.15c	1.42b
		1080	7.48d	4.37d	1.11c	1.45d	6.23c	4.58d	1.07d	1.53c
		1800	7.40e	4.72e	1.06d	1.52e	5.52d	5.40e	1.02e	1.64d
	25	0	9.24a	0.00e	1.27a	1.22b	9.24e	0.00a	1.27e	1.22a
		60	7.68e	3.30a	1.18d	1.28a	7.54a	3.82b	1.11a	1.34b
		360	7.60b	4.50c	1.10b	1.42c	7.09b	4.72c	1.02b	1.58c
		1080	7.38d	4.95d	1.05c	1.56d	5.69c	5.14d	0.86c	1.72d
		1800	7.04c	5.85b	0.89e	1.63e	5.12d	6.10e	0.79d	1.93e
SAH 177	15	0	8.67c	0.00a	1.10a	1.23e	8.67c	0.00e	1.10a	1.23b
		60	7.57a	3.20b	1.05b	1.26a	7.23b	3.80b	1.02b	1.36a
		360	7.52b	3.67c	0.97c	1.37c	7.11a	4.11c	0.90c	1.62c
		1080	7.27e	4.60d	0.92e	1.49d	6.97e	5.10d	0.82d	1.75d
		1800	6.98d	4.70e	0.85d	1.57b	6.52d	7.00a	0.75e	1.90e
	25	0	8.67b	0.00e	1.10a	1.23a	8.67c	0.00a	1.10d	1.23a
		60	7.63a	4.18a	0.92e	1.32b	7.31a	4.58b	0.89b	1.42e
		360	7.58c	4.57c	0.82b	1.48c	7.09b	4.84c	0.72a	1.76b
		1080	7.42d	5.03b	0.77c	1.56d	6.51d	5.22d	0.65c	1.92c
		1800	7.13e	5.84d	0.69d	1.64e	6.38e	7.12e	0.58a	2.04d

Values expressed as mean ± standard deviations of three independent experiments (n=3).

Distinct letters represent statistical differences within rows for the mean values at ($P<0.05$).

8.4.5. Swelling power

The swelling power (SP) of both SAH 329 and SAH 177 was evaluated to obtain information on the structural differences and molecular arrangement of the flour granules. From the results in Table 8.2 one can observe an increase in the SP with increasing ultrasound amplitudes and moisture contents. For instance, at 30% amplitude, values for SP for ultrasonically treated SAH 329 rice flour suspensions (15% moisture content) 60 secs (9.87 g of hydrated molecules/g flour dry matter), 360 secs (9.99 g of hydrated molecules/g flour dry matter), 1080 secs (10.31 g of hydrated molecules/g flour dry matter) and 1800 (10.52 g of hydrated molecules/g flour dry matter) are higher than untreated (9.06 g of hydrated molecules/g flour dry matter) and for 20% amplitude treated rice flour suspensions (60 sec – 9.19 g of hydrated molecules/g flour dry matter, 360 sec – 9.52 g of hydrated molecules/g flour dry matter, 1080 sec – 9.82 g of hydrated molecules/g flour dry matter and 1800 – 9.96 g of hydrated molecules/g flour dry matter). The increase in SP for both studied rice flours, when applying ultrasound is also higher for samples at higher moisture content (25%) than at 15% moisture content. Ultrasonication significantly ($P < 0.05$) increased SP of both rice flours at the applied moisture contents and ultrasonication amplitudes. This variation in SP could be linked to related to water uptake and retention properties affected by ultrasonication. The SP is directly linked to the increase with the water absorption properties of rice flour samples. The molecular organization, which is a factor of the amount, structure and organization of amylose and amylopectin, allows an assessment of the kind of organization and interactions happening in the interior of the rice flour matrix. Therefore, it is fair to suggest that the increased capacity for water movement in both rice flour matrix is as a result of ultrasound disruption of the flour granule, subsequently leading to increased water uptake and retention (Kaur and Gill, 2019). Cavitation forces play a major role in breaking down flour granule structure. During ultrasonication processing, the collapse of cavitation bubbles results in high-pressure gradients and increased local velocities of liquid layers in their vicinity which leads to shear forces that can disrupt the chemical interactions and organization of the flour's chemical constituents such as starch and protein molecules. The crystalline molecular structure of corn starch is broken, and the water molecules are bonded to the free hydroxyl groups of amylose and amylopectin by hydrogen bonds, which could cause an increase in swelling power.

Table 8.2. Swelling power of native and ultrasonicated SAH 329 and SAH 177 rice flours

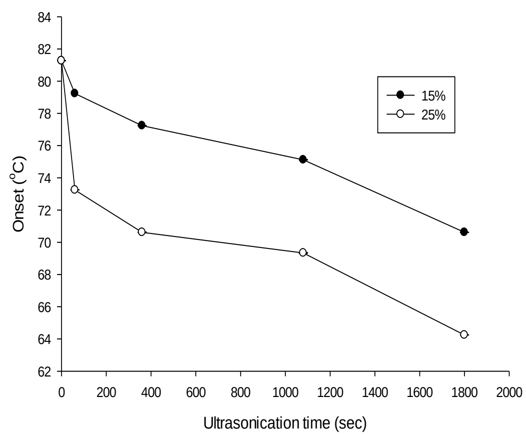
Amplitude level			20%	30%
Cultivar Type	MC %	Time(sec)	Swelling power (g/g)	Swelling power (g/g)
SAH 329	15	0	9.06a	9.06e
		60	9.19b	9.87b
		360	9.52c	9.99a
		1080	9.82d	10.31c
		1800	9.42e	10.52d
	25	0	9.06a	9.06e
		60	9.88e	10.04a
		360	9.99b	10.29b
		1080	10.13d	10.69c
		1800	10.24c	11.32d
SAH 177	15	0	8.96c	8.96c
		60	8.58a	9.23b
		360	8.62b	9.51a
		1080	9.22e	10.17e
		1800	9.98d	10.52d
	25	0	8.96b	8.96c
		60	9.13a	10.31a
		360	9.68c	10.29b
		1080	10.42d	10.51d
		1800	11.13e	11.38e

Values expressed as mean ± standard deviations of three independent experiments (n=3).

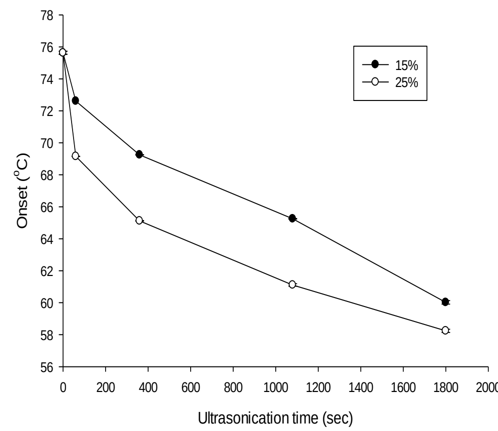
Distinct letters represent statistical differences within rows for the mean values at ($P < 0.05$).

8.4.6. Thermal properties

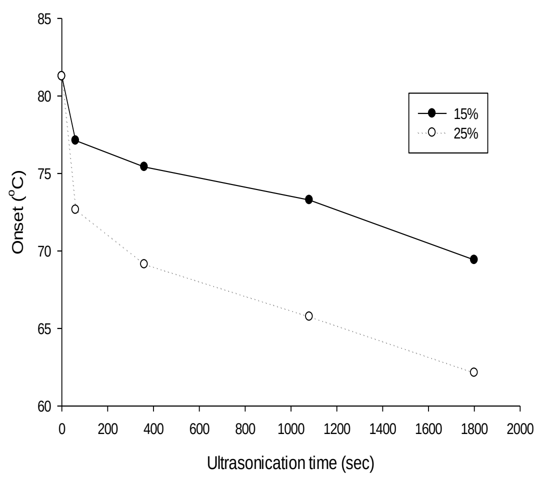
As shown in Figs. 8.3 and 8.4, the onset and peak temperatures decreased with moisture content when ultrasonication time was increased from 0 to 1800 sec. The onset and peak values of both rice flours were much higher when the moisture content was 15% and much lower when the moisture content was 25%. The onset and peak temperature values which represented ultrasound treated flours at 15% did not overlay that ultrasonically treated at 25% moisture content. This observation suggests that under the selected amplitudes, moisture content had a significant influence on the onset and peak temperatures of both rice flours. When ultrasonication was applied at 25% moisture content, the onset and peak temperature values were greatly reduced when compared with the application of ultrasound at 15% moisture content. The results of this study conclude that ultrasonication treatment at 25% moisture content was stronger in causing a significant impact on the crystal structure of the starch in the rice flour. Similar kind of observations on the impact of ultrasound on the crystalline organization of cereal products was earlier reported by Kaur and Gill (2019). Jambrak et al. (2009) suggested that ultrasound damages the starch granules in the flour matrix by collapsing the cavitation bubbles created by high-pressure gradients and increased local velocities of liquid layers in the treatment area. These reactions and changes, in turn, produce shear forces that break the polymers linkages and subsequently damages the starch granules. During ultrasound treatment, it is suggested that water molecules easily move into the rice flours, and some portion of the starch in the rice flours easily gelatinizes on the surface of the rice flour. This partially gelatinized flour easily reacts with water, invariably leading to a reduction of the onset and peak temperature values when during the ultrasonication process.



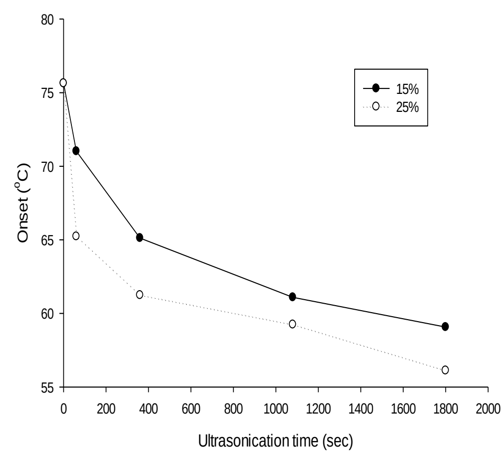
(a)



(b)



(C)



(d)

Fig 8.3. Change in the onset of gelatinization of (a) SAH 329 and (b) SAH 177 during ultrasonication amplitude of 20%; (c) SAH 329 and (d) SAH 177 flours as a function of ultrasonication amplitude (30%).

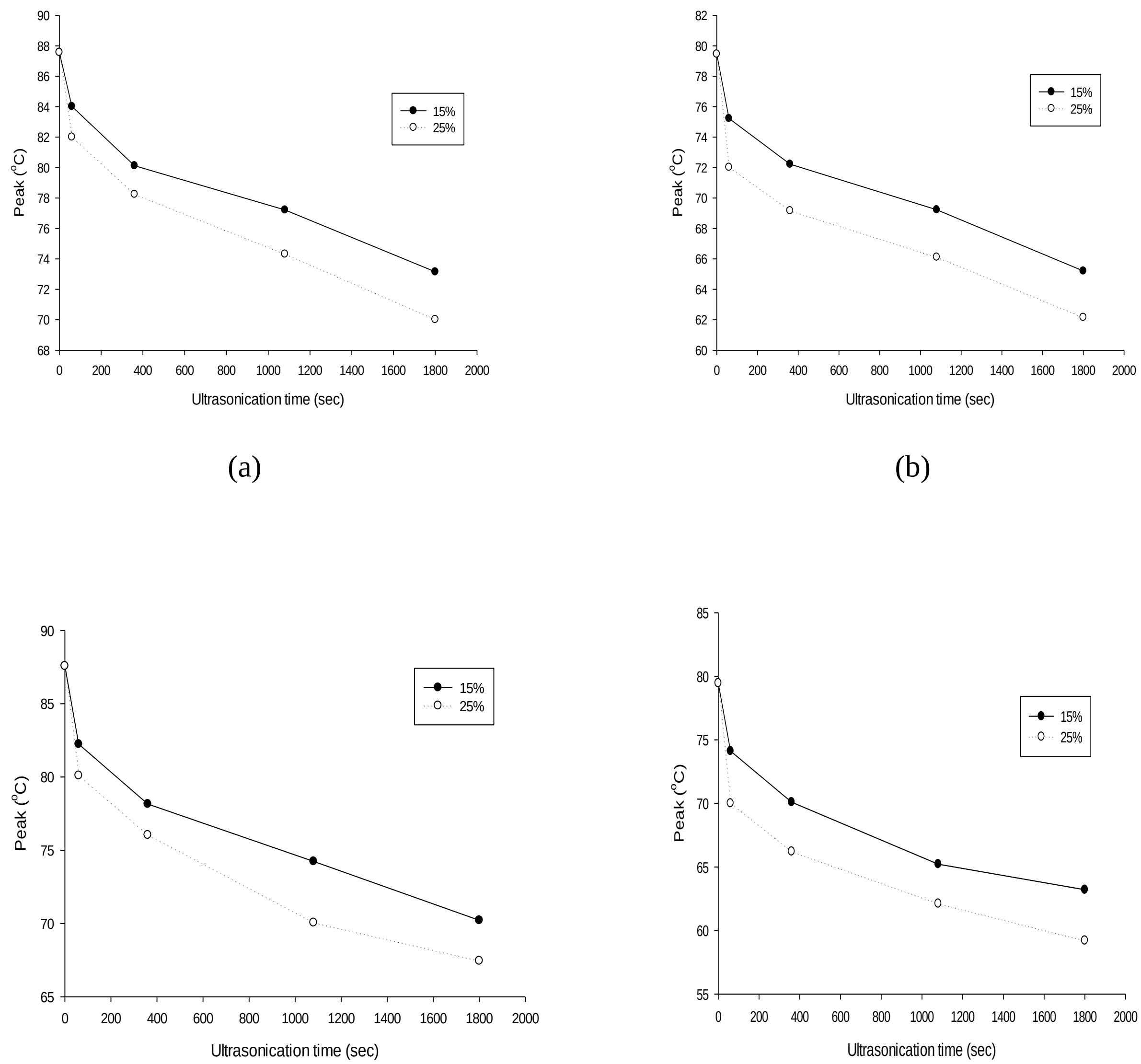


Fig 8.4. Change in the peak of gelatinization of (a) SAH 329 and (b) SAH 177 during ultrasonication amplitude of 20 %; (c) SAH 329 and (d) SAH 177 flours as a function of ultrasonication amplitude (30 %).

As seen in Fig 8.5, the thermal enthalpy values of both rice flours decreased with ultrasonication time throughout the studied processing times. Enthalpy of gelatinization is an index of phase transition of the granules in the rice flour from an organized state to a disordered one during heating in excess water (Błaszczak et al., 2007). It involves melting of organized fragments in the flour matrix, both on the crystallite and on the level of double-helical order. Ultrasound processing of rice flours alters the crystalline region in starch matrices prior to reversible hydration of the amorphous phase, which leads in the disruption of the granular organization (Chan et al., 2010). The enthalpy of gelatinization values of both rice flours was much higher when the moisture content was 15% and much lower when the moisture content was 25%. The greatest decrease in enthalpy for ultrasonically treated flours at 25 % moisture content suggest that these flours require the least energy for gelatinization as compared to the ultrasonically treated samples at 15% moisture content. The results of this investigation indicate that moisture content during ultrasonication had a significant impact on the starch organization and structure in the rice flours; the higher moisture content during ultrasonication easily broke down the flour structure because of the increased permeation of water molecules into the flour matrix. The movement of water molecules in and out of the flour granules leads to amylose molecule runoff from the interior of flour, which in turn leads to leads to differences among the bonding forces of the double helix forming the amylopectin crystallography, which also results in differ alignments of the hydrogen bonds within the starch molecules in the rice flours (Sandhu and Singh, 2007). The loss of double-helical order in the starch molecule of both flours is suggested to be the reason for the enthalpy variation observed in this study. The results of this investigations are similar to ultrasonication studies which lead to alterations on the starch granule structure, and subsequently variations on the physicochemical characteristics of rice (Yu et al., 2013) and corn starches (Jambrak et al., 2009; Kaur and Gill, 2019).

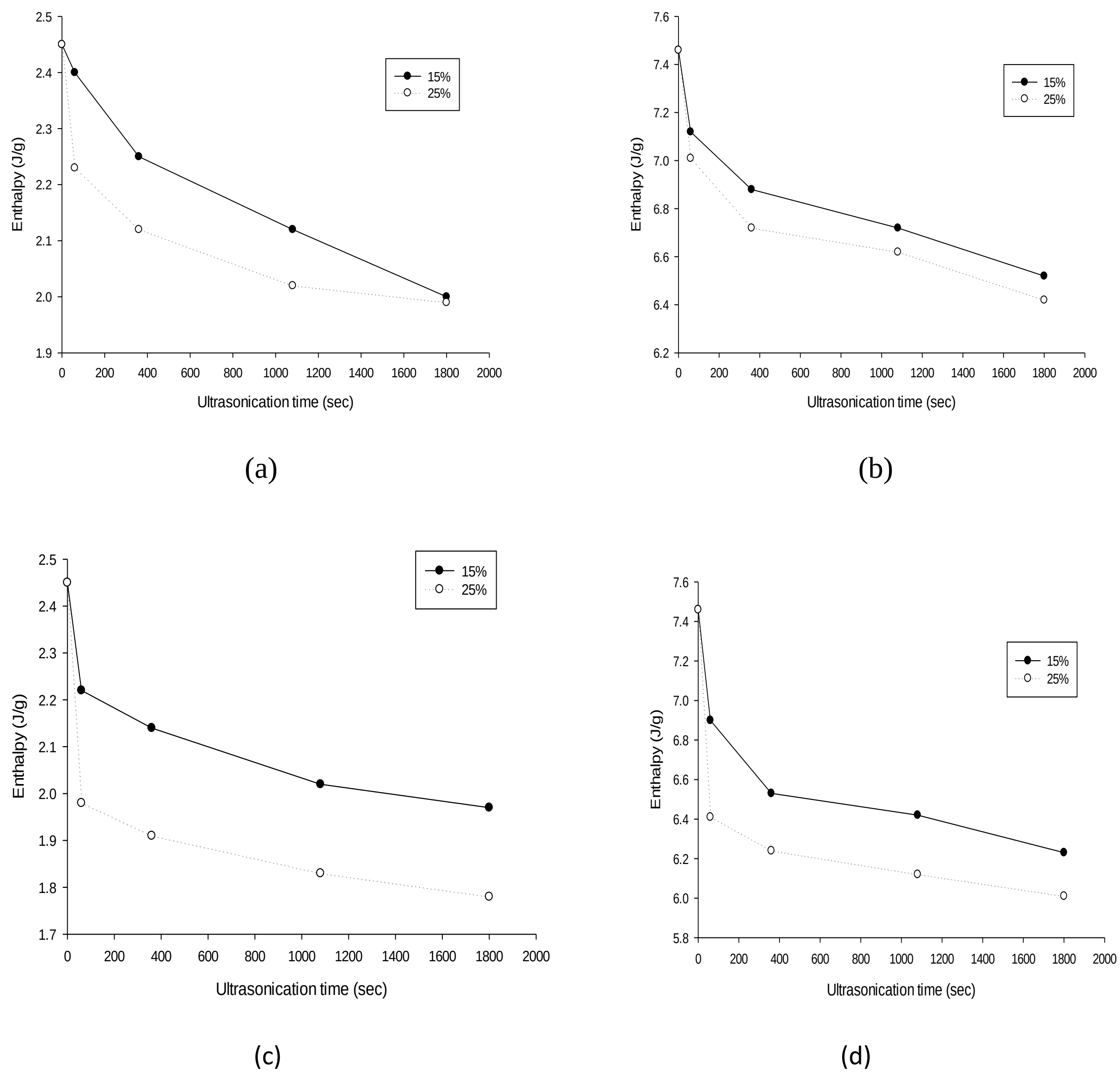


Fig 8.5. Change in the enthalpy of (a) SAH 329 and (b) SAH 177 during ultrasonication amplitude of 20%; (c) SAH 329 and (d) SAH 177 flours as a function of ultrasonication amplitude (30%).

8.4.7. Rheological properties

Fig 8.6. Illustrates the variations recorded in the rheological properties after SAH 329 rice flour was subjected to ultrasonication. From the rheological data of the rice flour, it was concluded that the highest values of G′ (storage modulus) and G″ (loss modulus) were observed at 1800 secs of ultrasonication. The increased values of G′ over G″ indicate the dominance of solid/elastic behavior of the flour. During ultrasonication at 15% moisture content (20% amplitude), G′ and G″

values of SAH 329 rice flour dispersions ranged from 112.00 – 534.10 Pa and 12.32 – 90.32 Pa respectively. When the moisture content was increased to 25% moisture content at the same amplitude (20% amplitude), the observed G′ and G″ values of the treated flours was 90.79 – 509.40 Pa and 9.38 – 74. 57Pa. Increasing the amplitude to 30%, the G′ and G″ values of SAH 329 rice flour dispersions (15% moisture content) was 9.37 – 74.57 Pa and 92.17 – 779.00 Pa; while at an increased moisture content of 25% at the same amplitude, the observed G′ and G″ data was 145.00 – 457.60 Pa and 8.21- 62.50 Pa. Ultrasound processing has been shown to improve the degradation of the flour granules by cavitation impact and therefore the flour materials become more permeable to water. The reported values of G′ and G″ at 1800 sec (at both studied amplitudes) suggest there is an increased breakdown of the flour structure and organization with increasing ultrasound processing time and that the crystalline portion of the flour matrix becomes deteriorated, subsequently leading the molecules in the flour matrix to entrap more water molecules which leads to an increased viscosity of the flour material. Kaur and Gill, (2019) reported a decrease in the G′ and G″ values of rice starch at ultrasonication treatment of 1800 sec. The differences between our results and this can be related to the type of treated material and the use of an ice bath in our study to control the temperature variations that occur during ultrasonication. Increased temperature rise during ultrasonication leads to an increase in the translational energy absorbed by the treated material. This energy absorption reduces the retained moisture and therefore leads to a reduced flour viscosity. Processing of rice flour dispersions by ultrasound treatment breaks down the crystalline framework and chemical interactions in the rice flour; leading to a loose and disrupted flour structure. Our findings conclude that at our selected moisture contents or amplitudes utilized in this work, the flour dispersions displayed shear thinning characteristics, as G′ > G″, a typical property displayed by pseudoplastic fluids i.e. flours displaying a greater elastic characteristic over its viscous potentials. In conclusion, the ultrasound treated flours in this study can be utilized in the food industry where high apparent viscous ingredients are needed.

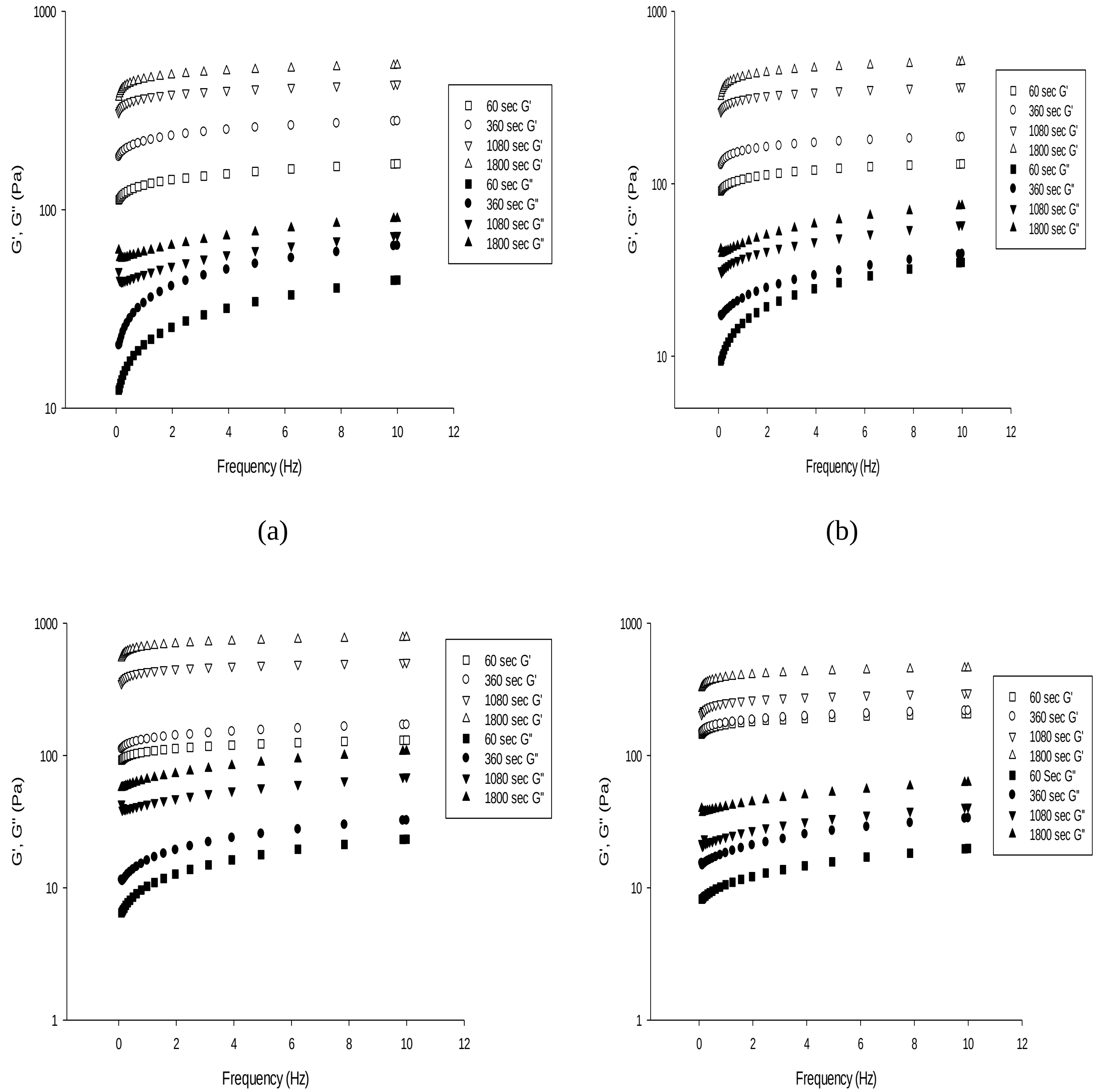


Fig 8.6. Dynamic rheological properties of SAH 329: dynamic mechanical spectra (open symbols, storage modulus, G'; closed symbols, loss modulus, G") measured during ultrasound treatment at 20% amplitude: (a) 15% and (b) 25% moisture contents; 30% amplitude, (c) 15% and (d) 25% and moisture contents.

8.5. Conclusion

Ultrasonication is a physical modification process that suggests a new means of modification and enhancement of the physicochemical, functional and rheological composition properties of food products. This treatment can be successfully used in the food manufacturing industry for various flour linked food applications to produce desirable and unique food products with improved product yield, reduced time of processing, eco-friendly and reduced energy usage. Ultrasound treated flours can be incorporated into food products as a functional ingredient. Thus, the process of ultrasonication provides an eco-friendly and chemical free means of creating and improving functional and shelf-life stable flour-based food products. In this investigation, ultrasonication increased the amylose contents and swelling power of booth selected rice flours. Further investigation of the impact of the moisture content during ultrasonication, revealed that the concentration of water activity of the medium influenced the degree of change of both parameters. Protein contents revealed that ultrasonication may have caused a certain degree of molecular unfolding on the proteins in the rice flour. When ultrasonication was applied to both rice flours, the water and oil holding properties displayed an increase and decrease respectively. These changes were linked to the protein contents, the degree of crystallinity and changes in the flour structure and organization. Thermal analysis revealed that ultrasonication reduced the onset temperature, peak temperatures and enthalpy of gelatinization properties of both flour samples. In addition, an increase in the moisture contents of the flour dispersions during ultrasonication led to a greater reduction of the studied thermal properties. Rheological analysis showed that selected moisture contents or amplitudes utilized in this work, the flour dispersions exhibited shear thinning characteristics, as $G' > G''$, a typical property displayed by pseudoplastic fluids. Overall, the results of this study showed that ultrasound treatment can be used to modify rice flours that can be applied in the production of several food products.

CONNECTING TEXT

In the previous chapter, physicochemical, thermal and rheological data were established for ultrasound treated non-waxy SAH 177 and 329 rice flour. It was established in previous chapters that ultrasound and microwave treatment could effectively change the physicochemical properties of our selected rice flours. Noteworthily, ultrasound application significantly influenced the water and oil holding properties of SAH 329 rice flour. In chapter 9, chicken breast cuts were coated with untreated, ultrasound and microwaved treated non-waxy SAH 329 rice flour. Frying kinetic data for these various coated products were evaluated to examine the influence the coating types on the mass transfer rate for moisture loss and oil uptake.

9. Frying kinetics of lean meat coated with ultrasound processed rice flour

9.1. Abstract

The aim of this work was to investigate the influence of ultrasound processed non-waxy SAH 329 rice flour coatings on mass transfer during air frying of chicken breast cuts. The influence of untreated, ultrasound and microwaved rice flour coatings on mass transfer in the product was determined. The results showed an interaction effect of these processing conditions on mass transfer. The untreated (without modification) and the modified coated samples were air fried at temperatures of 170, 180 and 190 °C for 4, 8, 12 and 16 min. First order kinetic model was utilized in examining the mass transfer rate for moisture loss and oil uptake. Results of this investigation showed that the first-order kinetic model fitted the moisture and fat transfer data well. The effective moisture diffusivity for the untreated coated, ultrasound coated, and microwave coated ranged between 5.68×10^{-5} to 12.14×10^{-5}, 4.90×10^{-5} to 8.01×10^{-5} and 5.36×10^{-5} to 6.34×10^{-5} m^2/s, respectively, and the R^2 values were between 0.89 and 0.98. Fat transfer rate constant for all the studied coated fried products was between 3.60×10^{-3} and 43.90×10^{-3} $s^{-1}$with R^2 from 0.64 to 0.97. The highest activation energy for moisture loss was observed in the samples coated with untreated SAH 329 rice flours, while the lowest activation energy for oil uptake was found in samples coated with ultrasound treated SAH329 rice flour. The results from this study proved that modification of local non-waxy rice flours can be used to optimize moisture loss and oil uptake during air frying of coated chicken breast cuts.

Keywords. Air frying. Coating. Mass transfer. Chicken nuggets. Modification. Ultrasound.

9.2.Introduction

In today's market, several approaches have been suggested for providing today's consumers with fried products that possess unique and desirable characteristics such as reduced oil contents, smooth mouthfeel, distinct flavor, color, and palatability. Air fried foods seem to provide such benefits. Today's consumers are concerned about health implications related to consuming food products with increased fat contents (Ngadi et al., 2006). Therefore, there is an increased interest by food processors to adopt techniques of reducing fat absorption during frying via the application of novel processes such as air-frying. The aim of this study is to understand the mechanism of oil uptake and moisture loss during air frying of chicken cuts with various coating materials.

Several studies have suggested various approaches to decreasing fat absorption during frying. These include modification of coating materials via physical (heat-moisture treatment, microwave processing, ultrasound treatments) and chemical modification processes, frying methods (air and deep-fat frying) and optimization of frying conditions. Application of pregelatinized coating to chicken cuts has been reported to reduce oil absorption during frying (Altunakar et al., 2004). Oladejo et al., (2017) concluded that ultrasound pretreatment significantly lowered oil uptake in fried sweet potato. Mohammadalinejhad and Dehghannya, (2018) also reported a 23.18% reduction in oil uptake in fried potato strips when ultrasound was applied to it prior to deep-fat frying. Karizaki et al. (2013) investigated the influence of ultrasound-assisted osmotic dehydration (UAOD) pretreatment on the quality of deep fat fried potato and reported that fried potato slabs pretreated by UAOD had a 12.5% reduction in oil uptake compared to control. These ultrasonication studies suggested that the alterations in the functionality of protein and starch contents of the materials during the modification studies were responsible for the reduced fat uptake. Application of pregelatinized starches as coatings have also been shown to provide a fried product with low oil content, increased moisture content, coating pick up and product volume (Altunakar et al., 2004). Modification of flours or starches used as coating agents has also been reported to reduce fat absorption (Altunakar et al., 2004). The properties of these fried foods are important in optimizing and limiting fat absorptions. Therefore, the application of coating materials is an essential approach to reducing the oil content of fried products (Xue and Ngadi, 2007). In addition, the application of modified coatings has been suggested to produce improved film forming barriers and subsequent reduction in fat uptake during frying (Altunakar et al., 2004). The literature on the kinetics of fat uptake in modified coated fried meat products is scarce in literature, although the dynamics of various physical modification methods such as microwave and ultrasound processing influence on other non-meat products have been widely studied. In this study, we evaluated the effect of microwaved and ultrasound treated flour coatings on the kinetics of moisture loss and fat retention during air frying. The findings of this research work will be beneficial in optimizing non-waxy rice flour applications for both domestic and industrial processing of chicken cuts for reduced fat retention.

9.3. Materials and methods

9.3.1. Modelling

Air frying is a dehydration process involving simultaneous moisture loss and fat uptake. Both phenomena are connected and have a linear association (Dobraszczyk et al., 2006; Yıldız et al., 2006). Moisture loss and oil retention during air frying are typically referred to as diffusion controlled (Adedeji et al. 2009) as expressed:

$$\frac{\partial}{\partial L}\left(D_{eff}\ \frac{\partial M}{\partial L}\right) = \frac{\partial (M)}{\partial t} \tag{1}$$

Assuming an initial uniform moisture content, insignificant external resistance to mass transfer, limited shrinkage, and that mass transfer was carried out from both sides of the chicken cut portions, a solution of the above partial differential equation was expressed by (Crank, 1975) as shown as:

$$M_r = \frac{M-M_e}{M_o-M_e} = \frac{8}{\pi}\sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left(-(2n+1)^2\, \pi^2\, \frac{D_{eff}t}{4L^2}\right) \tag{2}$$

Assuming M_e (equilibrium moisture content) is very limited, Equation (2) can be made simple as presented as:

$$M_r = \frac{M}{M_o} = \frac{8}{\pi} \exp\left(-\frac{\pi^2 D_{eff}t}{4L^2}\right) \frac{8}{\pi} \exp(-kt) \tag{3}$$

Modeling fat uptake during air frying required the application of a first order kinetic model postulated by Krokida et al. (2000).

$$F_c = C_o\,[1 - \exp(-kt)] \tag{4}$$

The changes of apparent diffusion coefficient (D_{eff}) and equilibrium oil contents (O_{eq}) in the flour coated samples with temperature was evaluated using an Arrhenius type equation.

$$D_{eff} = D_o \exp\left(\frac{E_a}{RT}\right) \tag{5}$$

$$O_{eff} = \mathrm{k} \, . \exp\left(\frac{E_a}{RT}\right) \tag{6}$$

9.3.2. Rice flour modification

9.3.2.1.Ultrasound flour treatment

SAH 329 rice flour were dispersed in distilled water to attain final solid concentration of 25% (dry weight basis). The dispersion for ultrasound treatment was homogenized using a magnetic stirrer for 10 minutes at a low speed. The flour dispersion was sonicated using a Sonics sonicator (VCX 500, Newtown, CT, USA) for 30 minutes in a flat-bottomed conical flask at an ultrasound power of 500 W and amplitude level of 30%. During ultrasound processing, the vessel containing the flour samples were held in an ice bath to control any rise in temperature by ultrasonication. Following sonication, the flours were centrifuged (3000 x g) and dried in a freeze dryer.

9.3.2.2.Microwave flour treatment

SAH 329 rice flour was dispersed in distilled water to attain a final solid concentration of 30% (db). The suspension was mixed thoroughly with a magnetic stirrer for 10 minutes at a low speed. Microwave treatment of the flour dispersion was carried out with a Panasonic NN-SN651W (Panasonic Corporation, Osaka, Japan) microwave oven (2450 MHz, power 1200 W) for 15 secs in Pyrex cylinders open at both ends (50 x 40 mm) at a power level of 550W. The containing vessels were covered with Saranwrap ® to limit moisture loss during the heating process. After microwave processing, flours were dried in a freeze dryer.

9.3.3. Frying process

The dimensions of the chicken breast cuts samples were about 3cm (length) x 3 cm (width) x 1.5 cm (thickness) (± 0.2 cm). The uniformity of the thickness of samples was checked by using a kulis vernier. The individual chicken cuts were coated with unmodified and modified (ultrasound and microwaved) SAH 329 rice flour. Coated chicken portions were fried by air frying. The air frying studies were also carried out at five intervals between 0 and 16 mins at three temperatures (170, 180 and 190°C). The internal temperature of the chicken cuts was measured using a digital K-type thermocouple. At the end of air frying and after the samples were cooled down to ambient temperatures, the samples were blotted with a paper tissue to remove surface oil and packaged.

9.3.4. Analysis of fried samples

Moisture content analyses of the whole fried sample were reported as the mass of moisture in the sample to the overall mass of the product sample on a wet mass basis (wb). The drying process of the fried samples was taken at various sampling intervals and heated in an oven (Isotemp 700, Fisher Scientific, Pittsburgh, PA) at 105°C for 24 hours and the difference in mass before and after was used for the analysis. For the fat content determination, fried samples were freeze-dried and ground in a coffee grinder (SUMEET Multi Grind, India). The ground samples (3g) were weighed into a thimble for fat extraction in a Soxhlet solvent extractor (SER, Velp Scientifica, Usmate, Italy) using petroleum ether. Oil content was noted as the ratio of the mass of extracted oil and dry matter of the sample.

9.3.5. Statistical Analysis

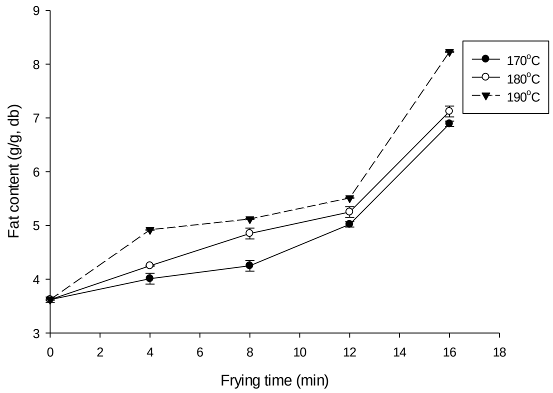
The experiments were carried out in triplicate. Recorded data were analyzed by one-way analysis of variance (ANOVA), and significant differences among mean were analyzed using Duncan's multiple range test at $P < 0.05$, using JMP software (Version 14; SAS, NC, USA).

9.4. Results and discussion

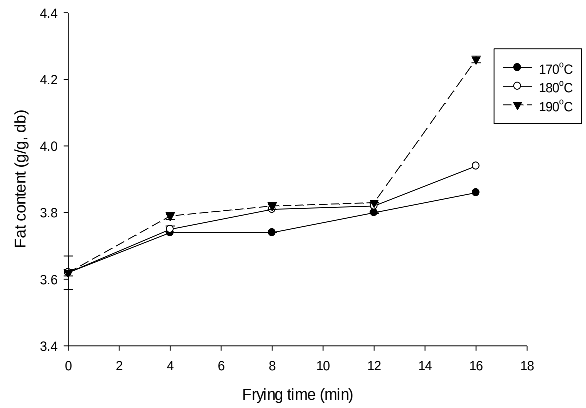
9.4.1. Fat retention and moisture loss

Figure 9.1 shows the oil absorption profile for the untreated and modified coated chicken cuts at different frying temperatures. The oil content for the untreated coated products fried at 170, 180 and 180^{o}C ranged from 4.01 – 6.89 g/g (db), 4.25 – 7.12 g/g (db) and 4.92 – 8.23 g/g (db), respectively; while the oil content for the ultrasound coated products air fried at 170, 180 and 180^{o}C was 3.74 – 3.86 g/g (db), 3.75 – 3.94 g/g (db), 3.79 – 4.26 g/g (db), respectively. Also, oil content ranges for the microwaved coated products air fried at 170, 180 and 180^{o}C was 3.90 – 4.17 g/g (db), 3.01 – 4.08 g/g (db) and 2.79 – 3.80 g/g (db), respectively. Air frying with microwaved and ultrasound flour coatings produced chicken breast cuts with the least fat uptake at 170,180 and 190 ^{o}C, which shows that microwaved and ultrasound coated breast cuts within this level possess the capability of reducing fat absorption (Fig 9.1). The physical modification (ultrasound and microwave) done on the rice flour prior to frying made the modified flours gain water thereby making the structure to be rigid with little damage on the structure. This promotes the formation of crust (hardening) during air frying as the temperature increased. The crust retards the movement of oil into the product during and after frying (cooling), hence, the reduced oil uptake. Similar results have been reported by (Dehghannya et al., 2015).

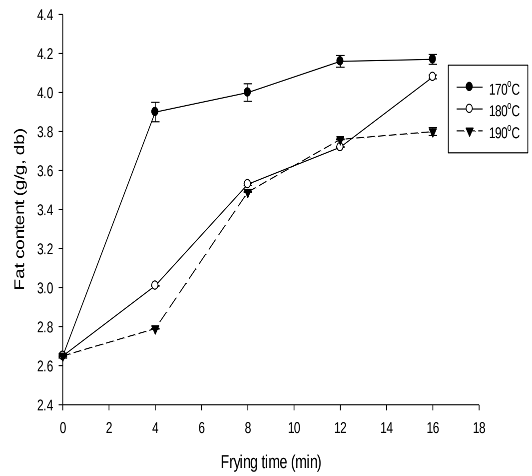
There was a general increase in the oil content of the untreated (Fig. 9.1.a), ultrasound (Fig. 9.1.b) and microwaved coated fried products (Fig. 9.1.c) during air frying. These observations illustrate the absorption property of the coating into the core part. ANOVA shows that the coating applied (untreated or modified treated) had an influence on the oil contents of the chicken breast cuts. Further statistical analysis revealed that the fat transfer was significantly ($P < 0.05$) influenced by the interaction effect between the frying temperature and frying time for the different coatings of the chicken breast cuts. For both untreated and modified coated products, there was a swift increase in the fat content of the coated fried products during the first 4 min of air frying. Adedeji et al. (2009) observed a similar increase in the oil absorptions for coated chicken fried nuggets during the initial stages of frying. During air frying, intense boiling was observed on the surface of both the uncoated and modified coated products in the first 12 min of frying, thus revealing moisture loss pattern from within the food products into the air and hot air penetration into the product. The oil saturation process of both untreated and modified coated fried products at increased frying times and temperatures could be associated with the surface effect of the coatings and also the structural changes brought about by gelatinization of the coating's starch components, protein denaturation, pore changes resulting to variations in porosity of the crust, all of which results to development of a compact crust surface which regulates mass transfer (Adedeji et al. 2009). Further statistical analysis also revealed that frying temperature did not significantly affect oil uptake ($P < 0.05$) which is consistent with results reported by Troncoso and Pedreschi, (2009) in vacuum frying of potato slices, but contrary to those reported Dehghan et al. (2011) in deep fat frying of shrimp nuggets. While several studies have evaluated the influence of frying temperature on oil absorption, there is no complete agreement. For example, some studies have surprisingly shown that frying temperature did not significantly influence oil uptake during conventional frying (Miranda and Aguilera, 2006).



(a)



(b)



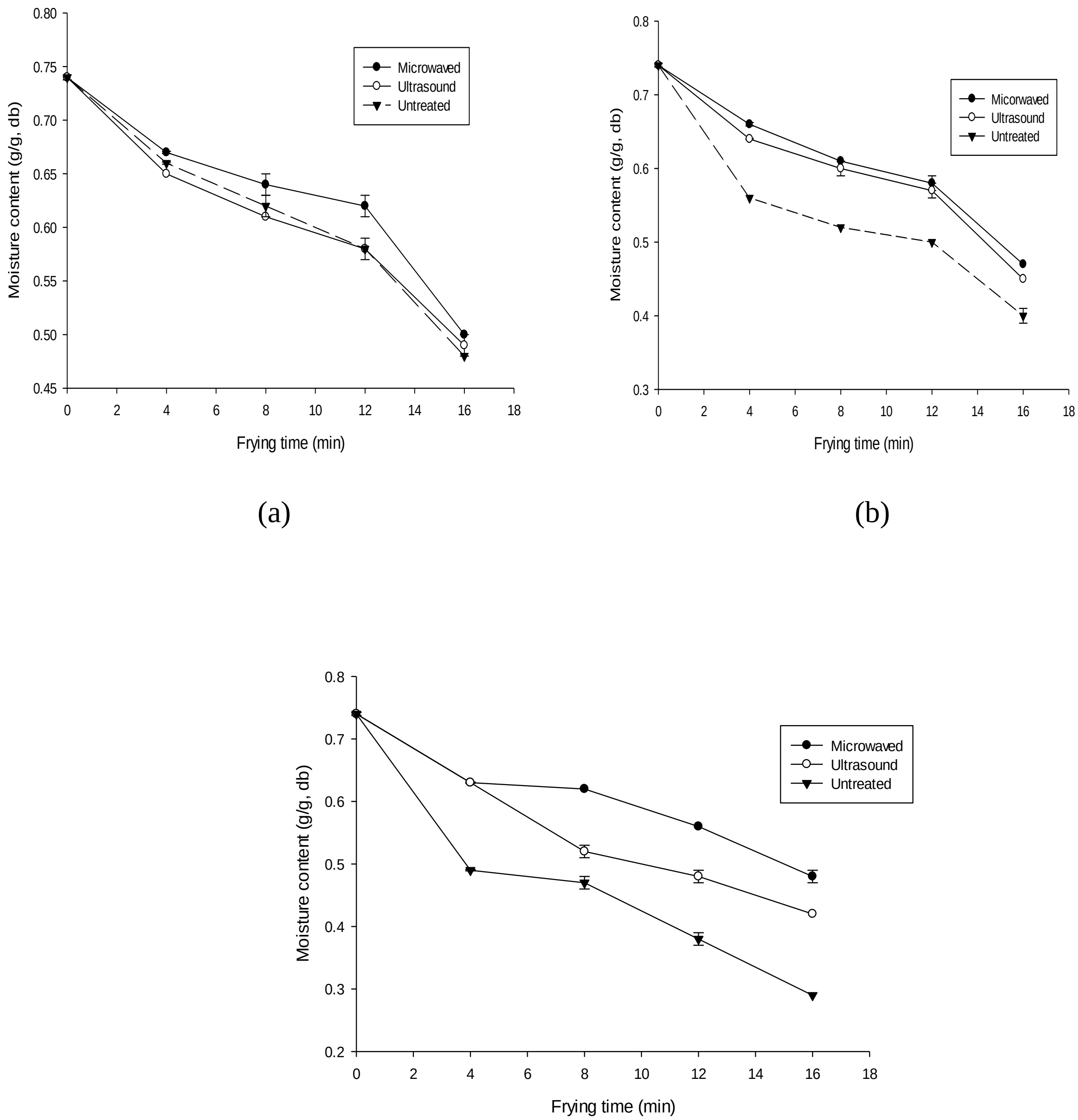
(c)

Fig. 9.1. Fat content as a function of frying time and temperature of chicken cuts coated with (a) unmodified (b) ultrasound and (c) microwaved treated SAH 329 rice flour.

The effects of different coating materials on the moisture content of fried chicken cuts at different temperature of 170, 180 and 190 °C are shown in Fig 9.2 a-c, respectively. Overall, there was a significant effect (P < 0.05) of coating applied on the moisture content for the chicken cuts during air frying. The moisture loss pattern in our investigation followed the general characteristic trends unique to most fried products (Hindra and Baik, 2006; Ngadi et al., 2006). The moisture content for the untreated coated products fried at 170, 180 and 180°C ranged from 0.48 – 0.66 g/g (db), 0.40 – 0.56 g/g (db) and 0.29 – 0.49 g/g (db), respectively; while the moisture content for the ultrasound coated products air fried at 170, 180 and 180°C was 0.50 – 0.67 g/g (db), 0.47 – 0.66

g/g (db), 0.42 – 0.63 g/g (db), respectively. In addition, moisture content ranges for the microwaved coated products air fried at 170, 180 and 180°C was 0.49 – 0.65 g/g (db), 0.45 – 0.64 g/g (db) and 0.48 – 0.63 g/g (db), respectively. The highest moisture contents were obtained in fried samples coated with ultrasound and microwaved treated SAH 329 rice flours. The physical modification treatments, namely microwave and ultrasound processing, might have weakened the structures in the granules of the rice flour coating subsequently improving movement of water into the internal structure of the product (Patist and Bates, 2007). Physical modification processes such as microwave treatment have been observed to improve the water absorption properties of the treated food products (Uthumporn et al., 2016).

As it is shown in Fig 9.2 a-c, the moisture content of both untreated and ultrasound coated products significantly decreases with frying time with an initial rapid decline in the first 4 minutes of frying, which is probably due to evaporation of surface water in all the products. The reduction in the initial moisture contents of all the coated products prior to frying favored reduced retention of moisture in the samples after air frying. Many researchers have observed similar findings in various fried food products (Adedeji et al., 2009; Krokida et al., 2000; Román et al., 2018). ANOVA showed that air frying temperature also significantly ($P < 0.05$) influenced the moisture loss of untreated, microwaved and ultrasound coated chicken cuts. The increased air frying temperature resulted in partial evaporation of water from the products, which moves away from the samples through the surrounding hot air in the air fryer. As it is shown, the highest moisture contents for all the coated products were found in the samples air fried at 170°C, while the lowest moisture contents were found at 190°C. This might be due to temperature influence on the film forming properties of the protein and the water binding capacity of coating material that control moisture loss during air frying. Seeing as oil uptake in fried products is typically a surface process and that oil can only be absorbed where water is evaporated, moisture loss and oil uptake are generally correlated (Adedeji et al., 2009; Dehghan et al., 2012; Durán et al., 2007). Increased moisture content in the final fried product generally means a product with a reduced final fat content (Adedeji et al., 2009)



(c)

Fig. 9.2. Effect of different coatings (untreated, ultrasound and microwaved) on the moisture content of fried chicken cuts at (a) 170°C (b) 180 °C (c) 190°C.

9.4.2. Modeling diffusion during air frying

Modeling of food processes is a multifaceted task due to the lack of data concerning mechanisms, the difficulty for conducting research and obtaining various reliable information, and the uncertainties concerning food properties. Modeling encompasses the construction and application of tools capable of predicting a product characteristic or a process with good and satisfactory precision. A vital aspect of frying is the removal of moisture and uptake of oil from the food during the process. Thus, it is important to develop models to predict and control moisture loss and oil uptake. In other words, modeling moisture loss and oil uptake as a function of time contributes to the study of the effect of process conditions on the rate of frying. The moisture contents data of the various coated fried chicken products were fitted to the exponential model (Eq. (3)). Effective moisture diffusivity was estimated from the rate constant, k, as shown below:

$$D_{eff} = \frac{4kL^2}{\pi^2} \tag{6}$$

The model parameters of moisture diffusion into the products are shown in Table 9.1. Table 9.1 shows the effective moisture diffusion coefficient (D_{eff}) of chicken breast cuts as influenced by different frying temperatures and coating treatments. The effective moisture diffusivity of the chicken breast cuts ranged between 4.90×10^{-5} and 12.14×10^{-5} m^2/s with R^2 between 0.89 and 0.98. The variations observed in the moisture diffusivities of the various coated products could be because of the modification treatments, which may have influenced the water absorption capacities of SAH 329 rice flour. The values in our investigation were higher than values reported by Adedeji et al. (2009) and Ngadi et al. (2006) for chicken nuggets, but were within the range of 2.05×10^{-8} and 5.71×10^{-8} m^2/s estimated by Dehghan et al. (2011) for shrimp nuggets fried between 150 and 190°C. The data of this study revealed that frying temperature had a positive influence on the effective moisture diffusivity of the samples coated with the untreated and ultrasound treated SAH 329 flour. As shown in our findings D_{eff} increased with increasing hot air temperature for the samples coated with the untreated and ultrasound treated SAH 329 rice flours. Typically, by increasing temperature during air frying, the temperature of the water vapor increases and subsequently improves the loss of moisture and increases this coefficient (Mohammadalinejhad and Dehghannya, 2018).

Mohammadalinejhad and Dehghannya, (2018) reported that D_{eff} is influenced by factors such as sample moisture content, process temperature, food structure, porosity, and pore distribution. Also, our findings revealed that the reaction rate constants for moisture loss in all samples coated with untreated SAH 329 rice flours were higher than that of microwaved and ultrasound coated products at all frying temperatures.

Table 9.1. Model parameters for moisture transfer in air fried chicken breast cut coated with untreated and ultrasound coated SAH 329 flours

Coating type	T(°C)	*k x 10 $^{-3}$(s^{-1})*	D_{eff} (m^2/s)	R^2	RSME
Untreated	170	24.90	5.68 x 10^{-5}	0.95	0.04
	180	33.60	7.67 x 10^{-5}	0.92	0.07
	190	53.20	12.14 x 10^{-5}	0.95	0.09
Ultrasound	170	21.50	4.90 x 10^{-5}	0.89	0.06
	180	25.90	5.91 x 10^{-5}	0.95	0.05
	190	35.10	8.01 x 10^{-5}	0.98	0.03
Microwaved	170	23.50	5.36 x 10^{-5}	0.96	0.04
	180	27.80	6.34 x 10^{-5}	0.93	0.06
	190	24.60	5.61 x 10^{-5}	0.95	0.04

Oil absorption is a surface phenomenon. Data on fat absorption in the coated products are shown in Table 9.2. The constant rate (k) was derived by fitting oil content data with Eq. (4). As seen in Table 9.2, rate constants ranged between 3.60 × 10^{-3} and 43.90 × $10^{-3}s^{-1}$ with coefficient factors of 0.80 – 0.95. The rate constants in this study were influenced by increasing temperature and the type of coating material used. For the untreated and ultrasound coated products, the rate constants increased with increasing temperature. The modification done on the coating materials reduced the rate constants of fat absorptions. The pre-treatment, namely microwave and ultrasound treatment, carried out on SAH 329 rice flour apparently altered the structure of the coating material subsequently leading to the large reduction in rate constants. The results in this study are contrary to the observations of Moyano and Pedreschi, (2006) which observed a large increase in fat absorption rate constant from 0.007 s^{-1} for control, to 0.218 s^{-1} for pre-blanched and dried fried potato samples. The fat absorption rate constants may be influenced by process conditions such as hot air temperature, type of product, pretreatment of the coating material, frying conditions and product thickness (Dehghan et al., 2011; Troncoso and Pedreschi, 2009). Dehghan et al. (2011) reported rate constants in the ranges of 3.50 × 10^{-3} and 7.80 × $10^{-3}s^{-1}$ for shrimp nuggets, Adedeji et al. (2009) reported a range of 0.04 – 49.96 s^{-1}, Troncoso and Pedreschi, (2009) presented a range of 3.70 × 10^{-3} and 7.80 × $10^{-3}s^{-1}$ for potato strips, Durán et al. (2007) observed rate constant in the range of 0.18–2.00 s^{-1} for pre-treated potato chips fried between 120 and 180 °C. Several studies have suggested the importance of the microstructure development during the frying process since it is an important factor in understanding oil transport mechanism and porous crust formation (Moreira et al. 1997; Ouchon et al. 2003). Generally, the O_{eq} values (an indication of the maximum quantity of oil absorbed) of the untreated and ultrasound coated chicken cuts increased considerably as the frying temperature increased for the three pretreatments in this study (Table 9.2). This observation disagrees with findings that increased frying temperatures lead to lower oil retention (Dehghan Nasiri et al., 2011; Moyano and Pedreschi, 2006). Thus, our studies suggest that oil absorption is a dynamic and complex mechanism influenced by numerous factors, such as product composition, surface-active agents, etc.

Table 9.2. Parameters of oil retention model of chicken breast cuts.

Coating type	T(°C)	$k \times 10^{-3}(s^{-1})$	O_{eq} *(g/g, db)*	R^2	RSME
Untreated	170	37.80	8.63×10^{-5}	0.90	0.09
	180	39.10	8.92×10^{-5}	0.95	0.06
	190	43.90	1.00×10^{-4}	0.89	0.11
Ultrasound	170	3.60	8.21×10^{-6}	0.91	0.01
	180	4.70	1.07×10^{-5}	0.93	0.01
	190	8.40	1.92×10^{-5}	0.80	0.03
Microwaved	170	24.30	5.55×10^{-5}	0.64	0.13
	180	26.90	6.14×10^{-5}	0.97	0.03
	190	25.50	5.81×10^{-5}	0.90	0.06

Arrhenius expression (Eq.5 and 6) was used to express the relevance of temperature on the effective diffusivity coefficient for the oil and moisture contents of the fried chicken cuts (Table 9.2). The natural logarithm of the effective diffusivity of oil and moisture during air frying was plotted against the reciprocal of the absolute temperature. The slope was multiplied by the universal gas constant (8.314 $J.K^{-1}.mol^{-1}$) to obtain the respective activation energies. Activation energy is an index of the threshold energy required to transform a reactant to the product (Oyedeji et al., 2016). Therefore, the activation energy in this work is the minimum energy required to cause moisture loss or oil uptake during air frying. The activation energy for the effective moisture diffusivity during air frying ranged between 4.08 and 64.38 kJ/mol, with the untreated coated fried products having the greatest value. The highest activation energy for moisture loss was found in untreated coated samples while the lowest activation energy for moisture loss was observed in the samples coated with microwaved treated rice flours. This result suggests that the air fried products coated with the untreated rice flours had the greatest sensitivity and dependency on temperature variation during air frying. The low activation energy for moisture diffusivity in the microwaved and ultrasound coated products could be related to weakening influence of the physical modification treatments on the flour granules, hence resulting in increased movement of moisture from the product to the surrounding hot air in the air fryer during frying. Oladejo et al. (2017) reported a range of 25.39 – 39.99 kJ/mol for moisture diffusion during deep fat frying of ultrasound pretreated sweet potato. The activation energy for the oil diffusivity process during air frying ranged between -4.21 and -72.08 kJ/mol (Table 9.3). This suggests the formation of crust on the chicken breast cut surface during air frying, thereby delaying oil absorption through the coating surface. The negative activation energies obtained in the coated air fried products also showed the decreasing trend in equilibrium oil content in the product as the temperature was increased. Adedeji et al. (2009) also reported negative activation energy ranges between -13.45 and - 27.58 kJ/mol for oil uptake of deep fat fried shrimp nuggets.

Untreated and ultrasound flour coated chicken cuts displayed R^2 values greater 0.81. These high values show a good prediction for these coated products (Table 9.3). The data for the microwaved coated products were poorly fitted as shown by very low R^2 values. This result suggests a more compact crust in these products, thus retarding moisture and oil diffusion through the coating materials. Also, other important structural events that take place during frying such as shrinkage and porosity increase make water transport inside the food sample more difficult (Troncoso and Pedreschi, 2009).

Table 9.3. Computed activation energy and R^2 values for the different coated chicken breast cuts.

Coating type	MC		FC	
	E_a (kj/mol)	R^2	E_a (kj/mol)	R^2
Untreated	64.38	0.81	-12.71	0.90
Ultrasound	41.74	0.98	-72.08	0.95
Microwaved	4.08	0.07	-4.21	0.23

9.5.Conclusion

The interaction effect of coating type, frying temperature and time significantly influenced moisture loss during air frying. Typically, our results showed that the moisture content of all the coated food products decreased with increasing frying time. Application of ultrasound and microwaved SAH 329 rice flours as coating materials significantly increased the moisture contents of the fried products during frying. Modification of the coating material and frying temperature influenced oil uptake for the air fried chicken breast cuts. Air frying with microwaved and ultrasound flour coatings produced chicken breast cuts with the least fat uptake at 170,180 and 190 ^{o}C, which shows that microwaved and ultrasound coated breast cuts posses the ability to reduce fat absorption. The application of ultrasound and microwaved flours significantly reduced the oil uptake of fried chicken breast cuts compared to the untreated sample at all the frying temperatures. Application of kinetics models provided an ideal and varied fit for moisture diffusion and oil transfer, respectively, for the fried products. A poor fit was produced for fat transfer and moisture loss in the microwaved coated products. The Arrhenius plot revealed that temperature during air frying influenced mass diffusion in the samples. The results from this work showed that application of microwaved and ultrasound treated non-waxy rice flours on chicken breast cuts prior to frying is a good technique, which can be used to optimize moisture loss and oil uptake during air frying.

10. General Summary

10.1. General Conclusion

Non-waxy rice flours have little usage in today's food industry mainly because of poor functional, rheological, thermal and physicochemical properties of their native flour. The industrial and domestic concern about the utilization of these flours in food processes and applications necessitated the development of food modification techniques that can improve functionalities. Modification of rice flours can help provide food processors with an adaptable raw material that can meet the unique requirements of a variety of food systems. It is also not very clear how structural changes made by modification processes such as microwave, ultrasound and heat-moisture treatments can relate to enhanced food product quality. Detailed studies of the impact of physical modification technologies on the quality of rice flours and rice-based products made from these modified flours are lacking.

In this study, the structural, rheological, thermal, functional and physicochemical properties of flours from seven non-waxy rice cultivars from Nigeria and Senegal were studied by different techniques namely X-ray diffractometry, rheometry, differential scanning calorimetry (DSC) and Association of Official Agricultural Chemists (AOAC) methods. The rice flours all exhibited A-type X-ray diffraction patterns. The degree of crystallinity of the rice flours was linked to the amount and distribution of amylose and amylopectin in the rice. Rheological evaluation of the rice flour dispersions (4-8%) produced from the rice cultivars displayed a non-Newtonian shear-thinning behavior. Thermal studies were effective in characterizing the thermal properties of the rice flours. Our studies revealed that the rice flours showed higher gelatinization peaks (T_p) but low glass transition temperatures (T_g). Further investigation revealed that the oil and water holding properties of the rice flour samples of the studied cultivar samples were influenced by the amounts of protein and amylose in the rice samples. The swelling power and WAI were presumably related to temperature and amylose content. Models designed in this study showed that experimental observations such as the gelatinization, water, and oil holding capacity properties could be predicted for non-waxy rice cultivars.

Heat moisture treatment via a convection oven was used to study the effect of the thermal modification on the flour characteristics of non-waxy rice flours. The study showed that depending on the temperature, time and moisture content during heat-moisture treatment, an array of responses could be found for heat-moisture treated flours. Protein contents, amylose contents, oil and water absorption properties, and thermal properties were evaluated for the heat-moisture treated rice flours. Increasing the moisture content during heat-moisture treatment led to a significant reduction in the protein contents, amylose contents, oil absorption capacities of the rice flours. Water absorption properties increased with increasing processing temperature during heat-moisture treatment. A decrease in enthalpy of gelatinization was observed as processing temperature increased during heat-moisture treatment.

The application of heat-moisture treated rice flours in food systems such as rice puddings was evaluated. Rheological, textural and color properties of pudding made from heat-moisture modified rice flour were evaluated. The heat-moisture treated rice flours had a reduced impact on the rheological behavior of the puddings when compared to that made from the untreated rice flour samples. The textural evaluation showed that the puddings made from the heat-moisture treated flours were less firm in comparison to puddings made from the untreated rice flours. Color analysis showed that the puddings made from heat-moisture treated flours had increased lightness when compared to puddings made from untreated rice flour samples.

Data obtained from the application of microwave processing showed the process had a significant influence on the physicochemical, rheological and thermal attributes of the selected non-waxy rice flours. The microwave process significantly influenced the amylose contents of the treated flours. These structural changes led to an increase in the amylose content, oil holding capacity, water holding capacity and gel hardness of the non-waxy rice flours. The application of the microwaved flours as coating materials for lean meat products was further evaluated. Oil uptake, moisture loss and some physicochemical properties of the coated lean meat portions during air frying were examined. Products coated with microwaved non-waxy flours showed a higher coating pick up, in comparison to samples coated with untreated non-waxy rice flour. Coating lean meat portions with microwaved non-waxy flour produced fried products with reduced oil absorption, increased cooking yield and coating pick up.

The impact of ultrasonication on the physicochemical, thermal, and rheological properties of selected non-waxy rice flours was evaluated. Physicochemical studies showed that the stepwise application of ultrasonication significantly increased the amylose contents and swelling power of non-waxy rice flours. A further look showed that the concentration of water during ultrasonication influenced the degree of change of both parameters. The water and oil holding properties were significantly influenced by the processing conditions during ultrasonication. These changes were related to the impact of ultrasonication on the protein contents and degree of crystallinity in the flour. Ultrasonication significantly reduced the onset temperature, peak temperatures and enthalpy of gelatinization properties of the treated flour samples. Ultrasound treated flour dispersions typically exhibited shear thinning characteristics, as $G' > G''$, a typical property displayed by pseudoplastic fluids.

A comparative study was done to investigate the influence of applying untreated, microwave and ultrasound treated non-waxy rice flours as coating agents during air frying. Application of ultrasound and microwaved SAH 329 rice flours as coating materials significantly increased the moisture contents of the fried samples. Physical modification of the rice flour coatings and frying temperature influenced oil uptake for the air fried chicken breast cuts. Air frying studies revealed that microwaved and ultrasound flour coatings produced chicken breast cuts with the least fat uptake at 170,180 and 190 ^{o}C.

All the modification techniques were effective in altering the physicochemical, rheological and thermal properties of the non-waxy rice flours. Further application of these modified non-waxy rice flours in the manufacture of food products such as coated lean meats and puddings, revealed that the modified flours imparted improved qualities to the final product.

10.2. Claims of contribution to knowledge

This study made the following original contributions to knowledge:

1. Predictive models were established in this study to predict the gelatinization, water and oil-holding properties of unmodified flours from non-waxy rice cultivars. The predictive models in this work displayed a good fit, with the R^2 values higher than 0.90. The suitability of the equations in this study for predictive purposes was confirmed using the root mean square of error (RMSE). The low RMSE values (< 1.806) in our model suggest a good fit and suitability for practical purposes.

2. Rheological investigation of the unmodified rice flour dispersions at different concentrations (4-8%) displayed a non-Newtonian shear-thinning behavior. The G' was greater than the G" at all the values of ɯ utilized in this study. The obtained data from this study provides a choice of rice flour with varying amylose content for the development of unique gluten-free products with desirable rheological attributes.

3. Heat moisture treatment was performed on non-waxy rice flours at different levels of moisture content, processing time and temperatures. This research work provided an insight into the influence of moisture content and temperature during heat moisture treatment on the amylose contents on non-waxy rice flours. The higher the moisture content of the flour during HMT, the lower the amylose content of the non-waxy rice flour. Our study showed that when non-waxy rice flour samples were subjected to a 2-hour heat treatment at 100 ^{o}C, a 0.5 ± 0.05% decrease in the amylose content is observed for every 1% increase in moisture. Increasing the processing temperature during HMT also led to decreased amylose compositions in the non-waxy flours. This study showed around 1 ± 0.05% (for every 1hr) decrease in amylose content as temperature increased from 95 to 115 ^{o}C.

4. This study established the possibilities and limitations of utilizing heat-moisture treated non-waxy rice flours in the production of rice puddings. The G' and G" values of the pudding samples made from the heat-moisture treated flours were (0.44- 1121) and (0.18 - 79.50), respectively. Rheological data acquired showed that the heat moisture-treated rice puddings displayed soft gel structures, with G' > G" and with tan $\delta < 1$ at all studied frequencies. The textural properties of the puddings made from the heat-moisture treated flours was namely: hardness (0.04 - 0.16 N), gumminess (0.01 - 0.15 N) and chewiness (0.01 - 0.15 N). Further analysis of textural data obtained in this study showed that the hardness, gumminess, and chewiness of puddings made from the untreated flour samples were higher than those of puddings made from the flours of the heat-moisture treated flours for all the whole period of storage

5. Pore characteristics of air-fried meat products coated with untreated and microwaved rice flours were obtained using Helium pycnometry. Porosity range for the meat cuts coated with untreated rice flour was 25.17 – 88.90%, and meat cuts coated with microwaved rice flours ranged from 13.99 – 77.38%. It was established that the treatment carried out on the rice flours significantly influenced pore development in the fried product. The inclusion of microwaved rice flour led to reduced porosity. This study showed that porosity of the coated products obtained with pycnometer reduced with frying time.

6. A comparative study was carried out to evaluate the influence of applying untreated and modified (microwaved and ultrasound treated) non-waxy rice flours as coating materials during air frying. The effective moisture diffusivity for the untreated coated, ultrasound coated, and microwave coated products ranged between 5.68×10^{-5} to 12.14×10^{-5}, 4.90×10^{-5} to 8.01×10^{-5} and 5.36×10^{-5} to 6.34×10^{-5} m^2/s, respectively. The highest activation energy for moisture loss was evident in the samples coated with untreated (64.38 kJ/mol) rice flour, while the lowest activation energy for oil uptake was found in samples coated with ultrasound (-72.08 kJ/mol) treated rice flour. Our study showed that the treatment done on the rice flours significantly influenced the observed kinetic parameters during frying.

10.3. Recommendations for future work

There is need for further investigation into how the different modification processes namely ultrasound, microwave and heat-moisture treatment influence the structural organization of the protein and starch molecules in non-waxy rice flours.

Industrial microwave and ultrasound systems with higher powers should be explored in characterizing non-waxy rice flours to capture data that might exist at a larger application scale and thus highlight any possible limitations of this process. In addition, the cost of making such modifications on a large scale needs to be explored.

It is further recommended that consumer testing studies of these fried products made with these modified rice flours be carried out.

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