Nutritional and functional properties of popped little millet (*Panicum sumatrense*)

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ABSTRACT

Food industries are focusing energies towards the development of functional foods and food ingredients. Several ancient grains are being used as a source of functional nutrients. Millets are minor cereals which have high nutritional value, are non-glutinous and are easily digestible. In spite of this, their consumption is limited. This could be attributed to their non-availability in ready-to-eat and ready-to-use foods. Processing of millets to incorporate them in ready-to-eat foods can increase their nutritional value, availability and economic value. Thermal processing can improve the bioavailability of certain vitamins and minerals and can also help in lowering the water activity thus, preventing the growth of microorganisms. Thermally processed foods also have better organoleptic properties. One interesting method of thermal processing is popping. Popping enhances the carbohydrate and protein digestibility by inactivating some of the enzymes and enzyme inhibitors. Popping also improves the color, appearance, aroma and taste of the processed food commodity. In the present study, the popping quality of little millet (*Panicum sumatrense*) and the effects of popping on the nutrient composition and the functional properties of the millet were studied. The popping quality of little millet was optimized with respect to the temperature of the particulate medium and the moisture content of the millet, both of which were found to determine the yield of popping. The optimized conditions for popping little millet were obtained at 16% grain moisture and particulate medium temperature of 260°C. The total protein, crude fat and total ash content of the popped millet was almost equal to that of the native millet. Popping increased the non-resistant starch content of little millet. The availability of total phenolics increased from 225 mg GAE/100g sample (db) in native millet to 661.462 mg GAE/100g sample (db) in popped millet. Popped millet flour (PMF) had a higher oil absorption capacity at room temperature as well as at 140°C and also exhibited higher swelling power and solubility. While the cold paste viscosity of the native millet flour (NMF) was 5.359 X 10^{-3} Pa s, that of PMF varied from 1.5 to 7.5 Pa s. NMF had a hot paste viscosity (HPV) of 0.1908 Pa s whereas the HPV of PMF varied from 1.9 to 7.5 Pa s. From the results obtained in the present

study, it was deduced that PMF would form pastes of uniform viscosity which would be more stable to heat during cooking and would have a greater shelf-life. It was also confirmed that popped millet flour had the advantage over native millet flour with improved nutrient availability and better functional properties.

Propriétés fonctionnelles et nutritionnelles du petit mil soufflé (*Panicum sumatrense*)

RÉSUMÉ

L'industrie alimentaire vise le développement d'aliments et d'ingrédients fonctionnels. De nombreux grains anciens sont utilisés comme source d'éléments nutritifs fonctionnels. Le millet est une petite graminée qui possède une excellente valeur nutritive, sans gluten et facilement digestible. Cependant cette céréale est peu consommée. Cela s'explique en partie par la non-disponibilité de produits prêts à manger issus du millet. La transformation du millet afin de l'incorporer dans une variété d'aliments peut améliorer sa valeur nutritive, sa disponibilité et sa valeur économique. Les traitements thermiques peuvent améliorer la biodisponibilité de certaines vitamines et minéraux et peuvent aider également a diminué l'activité de l'eau en prévention de la multiplication des microorganismes. Les aliments transformés par procédés thermiques ont souvent de meilleures propriétés organoleptiques. Un intéressant procédé de transformation thermique est l'expansion à sec qui entraîne l'éclatement du grain. L'expansion thermique à sec améliore la digestibilité des hydrates de carbone et des protéines en inactivant les enzymes et les inhibiteurs d'enzymes. L'expansion thermique peut aussi améliorer la couleur, l'apparence, l'arôme et le goût des céréales soufflées. Dans la présente étude, la qualité de petit mil (*Panicum sumatrense*) a été étudiée afin de déterminer les effets du traitement thermique sur la composition nutritionnelle et les propriétés fonctionnelles du millet soufflé. La qualité du millet soufflé a été optimisée en tenant compte du traitement thermique, soit la température des particules solides et la teneur en eau des grains, qui affectent tous deux le rendement. La protéine brute, la matière grasse brute, et la fraction totale de cendres n'ont pas été affectés par le traitement. L'expansion thermique a cependant augmenté l'amidon non-résistant du petit mil. La disponibilité des composants phénoliques a augmenté de 225 mg GAE/100g (base sèche) des échantillons témoins à 661.462 mg GAE /100g (base sèche) pour les grains soufflés. La farine de millet soufflé avait une capacité d'absorption d'huile plus élevée autant à la température de la pièce qu'à 140°C, avec également un pouvoir de gonflement et une solubilité plus élevés. La viscosité à basse température de la pâte de farine de millet témoin était de 5.359×10^{-3} Pa s par

rapport à une valeur variant de 1.5 à 7.5 Pa s pour la farine de millet soufflé. La viscosité à haute température de la pâte de farine de millet témoin était de 0.1908 Pa s par rapport à une valeur variant de 1.9 à 7.5 Pa s pour la farine de millet soufflé. Ainsi, de par les résultats obtenus, la farine de millet soufflé permet la formation d'une pâte de viscosité uniforme qui serait stable face à un procédé thermique, lui assurant une meilleure conservation. Les meilleures conditions pour l'expansion thermique du petit mil sont établies à une teneur en eau des grains de 16%, et une température des particules chauffantes de 260°C. La recherche a confirmé que la farine de millet soufflé est supérieure à la farine témoin (millet non-soufflé) avec une amélioration de la disponibilité des éléments nutritifs et de meilleures propriétés fonctionnelles.

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Chapter 1

Introduction

1.1 MILLETS

Millet is a generic term applied to a heterogeneous group of small seeded cereal crops which are known for their small coarse grains (Weber and Fuller, 2006). Millets are forage grasses which belong to the family Poaceae (Hunt and Jones, 2006; Weber and Fuller, 2006). Millets have a short growing season and are capable of tolerating environmental stress (Dogget, 1986). Much of millets' success in surviving through the ages has been their ability to grow well in hot, arid and drought prone areas (Rachie, 1975). Millets are minor cereals that form food for a large segment of the population, especially those with low socio-economic status. The millet grain is a small, round, ivory or light brown colored (dehusked) seed with a diameter of 1-2 mm (Web Ref. #1). There are about 6,000 varieties of millets grown around the world, the most important ones being *Eleucine corocana, Panicum miliaceum, Panicum sumatrense, Setaria italica, Pasplaum scrobiculatum* and *Echinochloa colona* (Hunt and Jones, 2006). Certain other varieties of millets like *Echinochloa crus-galli, E. stagnina, Pennisetum glaucum, Paspalidium flavidum, Pennisetum alopecuroides, Setaria glauca, Setaria verticillata, Urochloa spp.* and *Panicum spp*. also find importance occasionally, especially in cases of famine (Hunt and Jones, 2006). Recovered from archaeological sites throughout the world, millets are considered to be one of the oldest grains cultivated by man (Hunt and Jones, 2006).

Millets can be classified as Major Millets and Minor Millets. Sorghum and Pearl Millet come under the category of Major Millets and the Minor Millets comprise Foxtail Millet, Finger Millet, Barnyard Millet, Little Millet, Kodo Millet and Proso Millet (Fig. 1; Web Ref. #1).

The diversity represented by millets as a group varies from plants that hardly grow more than 1m in height and mature in less than 75 days to plants that grow beyond 4m in height and take more than 150 days to mature fully (Balasubramanian and Viswanathan, 2009).

Millets have an alkaline pH and are the only grains that keep their alkaline properties even after being cooked. As another plus, millet is a gluten free grain and thus, is ideal for people with wheat/gluten allergies or intolerance (Baltensperger and Cai, 2004).

Fig 1.1 (a) Sorghum

Fig 1.1 (b) Pearl Millet

Fig 1.1 (c) Finger Millet

Fig 1.1 (d) Little Millet

Fig 1.1 (e) Foxtail Millet

Fig 1.1 (f) Kodo Millet

Fig 1.1 (g) Proso Millet

Fig 1.1 (h) Barnyard Millet

Fig 1.1 Major Millets – (a) & (b); Minor Millets – (c) to (h)

***Source*:** Web Ref. #1

Millets can be called as store-houses of nutrition as by any nutritional criterion millets are more advanced than rice and wheat (Web Ref. #1). Depending on the species, the proximate composition varies (Bavec and Bavec, 2006). The fiber content of millets is way higher than that of wheat and rice, with Barnyard millet having fifty times the fiber content of rice (Web Ref. #1).

Millets are rich in vitamin B and also in minerals like Potassium, Phosphorous, Iron, Copper, Magnesium, Manganese and Zinc (Web Ref. #1). Millets have a higher oil content of 4.2% of which 50% is polyunsaturated. Millets are also a rich source of non-nutritional components like phenols, tannins, phytates and flavonoids (Pradeep and Guha, 2010). These compounds serve as antioxidants and millets could also be used as a source of extremely beneficial phytochemicals in the pharmaceutical and food industry (Pradeep and Guha, 2010).

Millet can be milled into flour and can be used in bread making. Millet flour can also be used to make traditional products like *ambali* (stiff porridge), *mudde* (dumplings) and *roti* (unleavened thin flat bread) (Dharmaraj and Malleshi, 2011; Krishnan et al., 2011). Some of the millets when presoaked in water can be cooked as rice. In Japan, millet is puffed and then made into sweetened cookies called *Awaokoshi* (Fig. 1.2, Web Ref. #2). In Nepal and Eastern India, millet is brewed into an alcoholic drink called *Tongba* (Fig. 1.3, Web Ref. #3).

Fig. 1.2 Awaokoshi: Sweetened cookies made from puffed millet

***Source*:** Web Ref. #2

Fig. 1.3 Tongba – Alcoholic drink made from brewed millet

***Source*:** Web Ref. #3

Processing of millets not only improves their digestibility and taste but can also increase their nutritional and economic value (Ushakumari et al., 2004). Processing the not-so-popular millets into nutritious ready-to-eat food products would increase their consumption by the masses thus improving their market prospects. Processed millets can be incorporated in ready-to-eat and ready-to-use food products. One interesting method of thermal processing is popping. Popping enhances the carbohydrate and protein digestibility by inactivating some of the enzymes and enzyme inhibitors (Muralikrishna et al., 1986; Subramanian et al., 1986). Popping can also improve the color, appearance, aroma and taste of the processed food commodity (Muralikrishna et al., 1986).

Unlike for major millets, the minor millets have not been exploited to their full potential. Little millet is one such minor millet which is rich in nutrients but has been long ignored. Little millet (*Panicum sumatrense*) is grown widely in India, Pakistan, Sri Lanka and Western Myanmar (Web Ref. #4). The plant is 30-90 cm in height (Figs. 1.4 and 1.5) and has an oblong panicle that is 14 - 40 cm in length (Hulse et al., 1980).

Fig. 1.4 Little millet plant

Source: Web Ref. #5

Fig. 1.5 Little millet seeds

Source: Web Ref. #6

The objectives of the present study were:

1. To optimize the particulate medium popping conditions of little millet (*Panicum sumatrense*) and,

2. To study the effects of popping on the nutrient availability and the functional properties of little millet.

Chapter 2

Literature review

2.1 Types of millets

Millet is a collective term for highly variable small-seeded grains that are grown around the world (Table 2.1, McDonough et al., 2000; Ushakumari et al., 2004). They are typically annual cereal grasses that are well adapted to grow in regions of hot environmental conditions, low moisture and low soil fertility (Rachie, 1975). Though classified as cereals as they are grown for their edible starchy seeds, millets are also important for forage production (Baltensperger and Cai, 2004). Millets are a group of diverse crops which can withstand drought as well as water logging conditions and this is why they can be found from tropical areas with average rainfall more than 1200 mm per year to the steppes where the average precipitation is less than 300 mm (Baltensperger and Cai, 2004). Millets have a short cultivation cycle and can be harvested only 45-65 days after planting (Web Ref. #1). Millets are considered as crops of food security as they are usually used in emergency cases following crop failure (Ramprasad and Gowda, 2004).

2.1.1 Pearl Millet

Pearl millet (*Pennisetum glaucum*) is also known as spiked millet, bajra and bulrush millet (Bavec and Bavec, 2006). It is certainly the most important and widely grown millet with more than 15,000 lines in the World Germplasm Collection in India (McDonough et al., 2000). Pearl millet originated in tropical western Africa, where the greatest number of both wild and cultivated genotypes occurs (FAO, 1995). Depending on the genotype, the plant may grow from 0.5 to 3.5 - 4 m and the seeds can be white, light yellow, grey, brown, purple or slate blue in color (Figs 2.1-2.2) (Bavec and Bavec, 2006; FAO, 1995; Hulse et al., 1980). The 1000 seed-weight lies between 2.5 to 14 g with an average weight of 8 g (Bavec and Bavec, 2006; FAO, 1995). Thousand seed-weight (TSW) is the weight of 1000 seeds and gives a measure of the seed size. TSW varies from crop to crop and can vary between varieties of the same crop. Due to this variation, the number of seeds in a pound is also variable (Agriculture and Rural Development, Alberta, Canada, Web Ref. #7).

Fig 2.1 Pearl millet cobs in field

Source: Web Ref. #8

Fig 2.2 Pearl millet seeds

Source: Web Ref. #9

2.1.2 Sorghum

Sorghum (*Sorghum bicolor*) is also known as great millet, jowar, dura, guinea corn and mtama (Bavec and Bavec, 2006). Although a perennial grass, it is treated as an annual and can be harvested many times. The kernels of sorghum are usually spherical and can be white to yellow to brown to deep purple-brown in color (Figs. 2.3-2.4) (FAO, 1995). The 1000 seed-weight lies between 25-30 g (FAO, 1995).

Fig 2.3 Sorghum crop in field

Source: Web Ref. #10

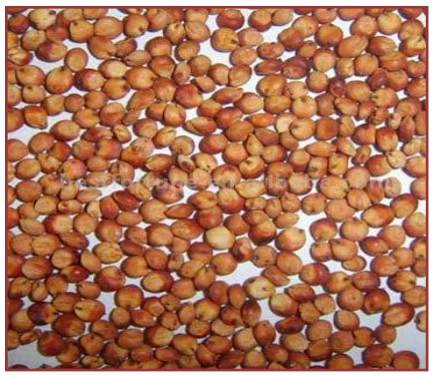

Fig 2.4 Sorghum seeds

***Source*:** Web Ref. #11

Table 2.1 Scientific and common names of millets (McDonough et al., 2000)

Scientific names	Common names	Locations
Sorghum bicolor	Sorghum, great millet, guinea corn, kafir corn, dura, mtama, jowar, cholam, milo, kaoling, milo-maize	Northeast quadrant of Africa (Ethiopia-Sudan border)
Pennisetum glaucum, P. americanum, P. typhoides	Pearl millet, bajra, cattail millet, bulrush, spiked millet	India, Africa
Panicum milaceum	Proso millet, broomcorn millet, hot millet, samai, panivarigu, common millet	China, Russian Federation, United States
Eleusine coracana	Finger millet, ragi, bird's foot millet, African millet, tamba	India, Africa, China
Setaria italica	Foxtail millet, navane, Italian millet, German millet, kangni	China, Near East, Europe
Degitaria exilis, D. iburura	Fonio, acha, pene, fundi, hungry rice, iburu	West and North Africa
Panicum sumatrense, P. psilopdium	Little Millet, sama	India, Nepal, Burma
Eragrostis tef, E. abyssinica	Teff	East Africa, Ethiopia
Paspalum scorbiculatum, P. commersoni	Kodo millet, varagu	Southern Asia
Echinochloa crusgalli, E. utilis, E. frumentacea, E. colona	Japanese barnyard, Japanese millet, barnyard millet, sawa	Asia

2.1.3 Finger Millet

Finger millet (*Eleusine coracana*) represents a staple food for a large segment of the population (FAO, 1995). It has high nutritional value and is a good source of calcium, phytochemicals and dietary fiber (Bavec and Bavec, 2006; Krishnan et al., 2011). As the grains can be stored for long duration without damage from pests and insects, it plays a major role during natural calamities (FAO, 1995). The height of plants reaches from 0.4 to 1 m and the length of the spikes is between 3 and 13 cm (Figs. 2.5-2.6) (Bavec and Bavec, 2006; FAO, 1995). The grains are smaller than those of pearl millet and vary in color from white to orange-red to deep brown to almost black (FAO, 1995).

Fig 2.5 Finger millet plant in field

Source: Web Ref. #12

Fig 2.6 Finger millet seeds

***Source*:** Web Ref. #13

2.1.4 Foxtail Millet

Foxtail millet (*Setaria italica*) is also known as Italian, Hungarian, German and Siberian millet (Bavec and Bavec, 2006). Though China is the leading producer of foxtail millet, it is also grown widely in India and is the most important millet in Japan (Bavec and Bavec, 2006; FAO, 1995; Rachie, 1975). The plant height varies between 1-1.5 m. The inflorescence is tight, has short side branches and varies from 7.5-25 cm in length and 1.2-5 cm in diameter (Figs 2.7-2.8) (Bavec and Bavec, 2006; FAO, 1995). The panicle resembles the tail of a fox. The color of the grain varies from yellow to orange to red to brown and black (Bavec and Bavec, 2006; FAO 1995). The weight of 1000 seeds is about 2 g (FAO, 1995).

Fig 2.7 Foxtail millet plant

Source: Web Ref. #14

Fig 2.8 Foxtail millet seeds

Source: Web Ref. #15

2.1.5 Proso Millet

Proso millet (*Panicum miliaceum*), often referred to as common millet, broom-corn millet, hog millet, Russian millet and brown corn millet, is an ancient millet and is known for maturing quickly (Bavec and Bavec, 2006; FAO, 1995). Proso millet is of great importance as it can be used for food and fodder and also for industrial uses (Bavec and Bavec, 2006). The seeds are used for making porridge and flour while the straw is used as animal feed (Bavec and Bavec, 2006). The plant grows to a height of 0.3-1 m (Figs 2.9-2.10) with an average growing period of 80 days (Bavec and Bavec, 2006; FAO, 1995). The starch content of the seeds is high, making it fit for industrial applications (Bavec and Bavec, 2006) like in making gels. The seeds are enclosed in hulls which are difficult to remove, thus, contributing to the high percentage of indigestible fiber (FAO, 1995). The 1000 seed-weight varies between 4.7-7.2 g with an average weight of about 5 g (FAO, 1995).

Fig 2.9 Proso millet plant

***Source*:** Web Ref. #16

Fig 2.10 Proso millet seeds

***Source*:** Web Ref. #17

2.1.6 Little Millet

Little millet (*Panicum sumatrense*), also known as small millet, is grown widely in India, Pakistan, Sri Lanka and Western Myanmar. Unlike other millets, little millet has not received much attention from plant breeders. The plant grows to a height of 30-90 cm and has oblong panicle that is almost 14-40 cm in length (Figs. 2.11-2.12) (FAO, 1995; Hulse et al., 1980). Like other millets, little millet can withstand drought and water logging conditions (Rachie, 1975). The seeds are yellow in color and are usually smaller than those of proso millet (Bavec and Bavec, 2006 and, FAO, 1995). The 1000 seed weight varies between 2.3 - 2.09g (Ninganagoudar et al., 2012)

Fig 2.11 Little millet plants

Source: Web Ref. #4

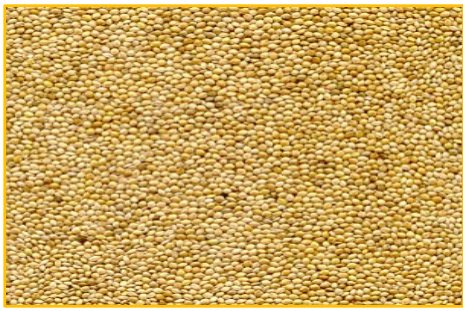

Fig 2.12 Little millet seeds

***Source*:** Web Ref. #5

Based on the inflorescence morphology, *Panicum sumatrense* is divided into subspecies *psilopodium* and *sumatrense* and the subspecies *sumatrense* is further classified into races Nana and Robusta. The race Robusta is further classified into subraces called Compacta and Laxa and the race Nana is divided into subraces called Erecta and Laxa (Fig 2.13), Web Ref. #4).

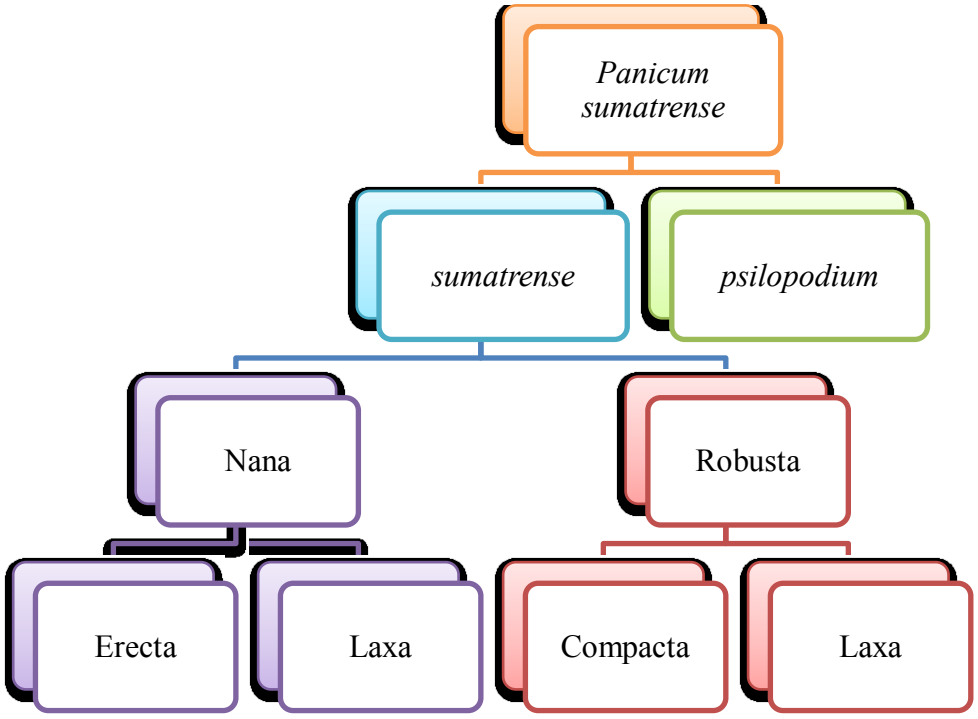

Fig. 2.13 Flowchart showing the classification of *Panicum suamtrense* (Web Ref. #4)

2.1.7 Barnyard Millet

Barnyard millet has two subspecies – Barnyard grass (*Echinochloa crus-gali*) and Japanese Barnyard millet (*Echinochloa colona*) (Bavec and Bavec, 2006; FAO, 1995). It is the fastest growing of all millets and can produce a crop in six weeks (FAO, 1995). The plant height varies from 0.5 to 1 m (Figs. 2.14-2.15) (Bavec and Bavec, 2006). It is grown in India, China and Japan and is usually used to substitute rice in case of failure of rice crops (FAO, 1995). The TSW varies from 1.7- 4.17 g (Singh et al., 2010)

Fig. 2.14 Barnyard millet plant

Source: Web Ref. #1

Fig. 2.15 Barnyard millet seeds

***Source*:** Web Ref. #18

2.1.8 Kodo Millet

Kodo millet (*Paspalum scrobiculatum*) is grown primarily in India (FAO, 1995). It is an annual tufted grass and grows to a height of 0.9 m (Fig. 2.16) (Bavec and Bavec, 2006). The seeds are prone to fungus infestation due to which some of the varieties are poisonous for animals and humans (FAO, 1995). The grains can be light red to dark grey in color (Fig. 2.17) (Bavec and Bavec, 2006).

Fig. 2.16 Kodo millet plant

Source: Web Ref. #19

Fig.2.17 Kodo millet seeds

Source: Web Ref. #20

2.2 STRUCTURE OF GRAINS

The kernels of millets vary considerably from each other in terms of their color, size, shape and anatomy (McDonough et al., 2000; FAO, 1995). The three main components of a kernel are – pericarp, embryo or germ, and endosperm (Hulse et al., 1980; FAO, 1995). The millet seeds are of two types – utricles and caryopses (McDonough et al., 2000; FAO, 1995). In the caryopses type, the pericarp is completely fused with the endosperm as in the case of pearl millet and sorghum (FAO, 1995). Finger, foxtail and proso millets are of the utricle type in which the pericarp forms a sac and is loosely attached to the endosperm only at one point (McDonough et al., 2000). Table 2.2 presents the comparison between the structural features of different millets. Figure 2.18 presents a diagrammatic representation of a sorghum caryopsis.

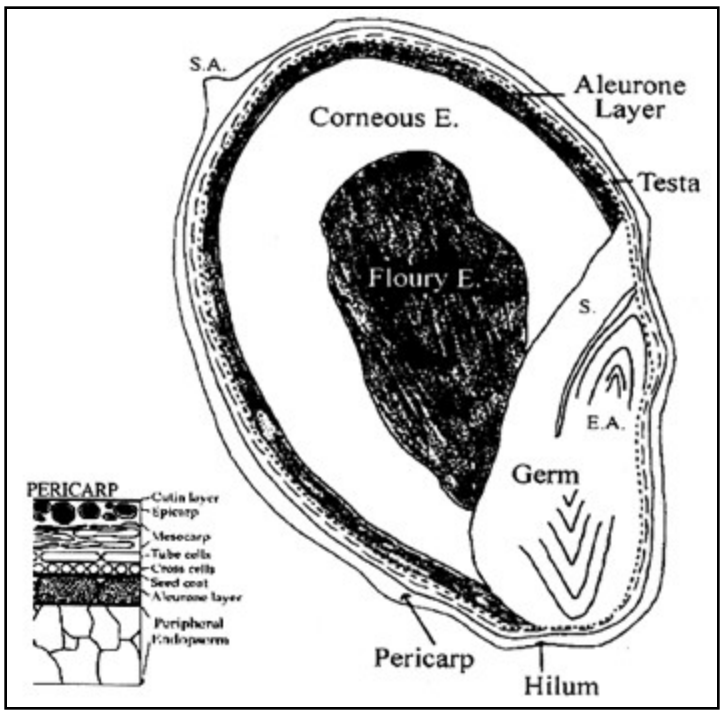

Fig. 2.18 Diagram of a sorghum caryopsis

Source: Web Ref. #21

2.2.1 Pericarp

The original ovary wall forms the outermost layer of the seed and is called the pericarp which is surrounded by a waxy cuticle and is composed of three sublayers called the epicarp (outermost), mesocarp (middle layer) and endocarp (innermost) (Hulse et al., 1980; FAO, 1995). The epicarp is composed of epidermis, which is formed of two to three layers of elongated, thick and rectangular cells, and the hypodermis, which is formed of slightly smaller cells as compared to the epidermis (Hulse et al., 1980; FAO, 1995).

Pearl millet has a thick epicarp of one to two layers (McDonough et al., 2000). The thickest mesocarp is found in sorghum but the thickness of the mesocarp varies from grain to grain and varies due to genetic factors (FAO, 1995). The pericarp of pearl, proso and foxtail millets contains some antinutritional factors which are due to the presence of certain pigments present in the pericarp (McDonough et al., 2000).

The testa or seed-coat is present just below the endocarp (FAO, 1995). Its thickness varies throughout the kernel- thin near the embryo and thick near the crown (Hulse et al., 1980; FAO, 1995). The testa may be pigmented due to genetic factors (Hulse etal., 1995). Pearl millet has a lightly pigmented testa, whereas red finger millet has a thick pigmented testa (McDonough et al., 2000; FAO, 1995). The grains that have a thick mesocarp are traditionally decorticated by hand-pounding (McDonough et al., 2000).

2.2.2 Endosperm

The major storage tissue, the endosperm, is the largest component of the grain (McDonough et al., 2000; FAO, 1995). It is composed of an aleurone layer and the peripheral, corneous and floury endosperm areas (FAO, 1995; Hulse et al., 1980; McDonough et al., 2000). The aleurone is a single layer of cells that surrounds the endosperm (McDonough et al., 2000). The cells of the aleurone are rich in minerals, oil, proteins, enzymes and B-complex vitamins (FAO, 1995; Hulse et al, 1980). The cells of the peripheral endosperm are densely packed and are long and rectangular in shape (FAO, 1995). The protein content of the peripheral endosperm is higher than the rest of the endosperm (Hulse et al., 1980; McDonough et al., 2000). The millet protein is rich in enzymes like phosphatases, protease, 3-galactosidase and 3-glucosidase (FAO, 1995). The starch granules present in the peripheral endosperm are buried in the protein matrix, thus hindering its availability for enzyme digestion (FAO, 1995; Hulse et al., 1980). The starch granules in the floury area are spherical whereas those in the corneous endosperm are polyhedral (McDonough et al., 2000; FAO, 1995).

The texture of the millet kernel is determined by the proportions of floury and corneous endosperm- the more the floury endosperm, the softer the millet kernel (FAO, 1995; Hulse et al., 1980). Proso millet has an intermediate texture whereas finger millet has a hard endosperm. The texture of pearl millet and sorghum varies from very soft (all floury) to very hard (all corneous) endosperm (FAO, 1995; Hulse et al., 1980).

Table 2.2 Structural comparisons of some millets (McDonough et al., 2000)

	Sorghum	Pearl Millet	Finger Millet	Proso Millet	Foxtail Millet
Seed type	Caryopsis	Caryopsis	Utricle	Utricle	Utricle
Pericarp	Attached	Attached	Unattached	Unattached	Unattached
Seed coat:	1 layer	1 layer	5 layers	1 layer	1 layer
Pigmented	Sometimes	Sometimes	Yes	No	
Thickness (µm)	0.4	0.4	10.8-24.2	0.2-0.4	
Aleurone	1 layer	1 layer	1 layer	1 layer	1 layer
Cell size (µm)		16-30 X 5-15	18 X 7.6	12 X 6	
Starch granules:					
Diameter (µm)	20-30	10-12	3-21	2-10	10
Peripheral (µm)		6.4	8-16.5	3.9	
Corneous(µm)		6.4	3-19	4.1	
Floury(µm)		7.6	11-21	4.1	
Type	Simple	Simple	Simple/ compound	Simple	
Protein bodies					
Size (µm)	0.3-3	0.6-0.7	2.0	0.5-1.7	
Location (µm)	All areas	All areas	Peripheral/ corneous	Peripheral	
Germ					
Size (µm)		1420 X 620	980 X 270	1100 X 310	
Endosperm to germ ratio	8.4:1	4.5:1	11:1	12:1	12:1

2.2.3 Germ

The germ consists of the embryonic axis, scutellum, a plumule and a primary root (Hulse et al., 1980). The germ is relatively smaller than the endosperm in finger and proso millets but in pearl millet and fonio, the germ is large (FAO, 1995; McDonough et al., 2000). The scutellum acts as a storage tissue and is rich in enzymes, lipids, proteins and minerals (FAO, 1995). The oil present in the germ of sorghum is a good source of polyunsaturated fatty acids (FAO, 1995).

2.3 NUTRIENT COMPOSITION OF MILLETS

Millets are store-houses of nutrition as they are way-ahead of wheat and rice in terms of their nutrient content (Web Ref. #1). The proximate composition of millets varies from species to species and is dependent on the environmental as well as the genetic factors (McDonough et al., 2000). The protein content of millets is almost equal to that of wheat and rice (Web Ref. #1). Based on their protein content, millets can be grouped into high protein millets and low protein millets. Pearl millet, foxtail millet, barnyard millet and proso millet have high protein content of 14.5%, 11.7%, 11.8% and13.4%, respectively (McDonough et al., 2000). Finger millet has the lowest protein content of 8.0% (McDonough et al., 2000). The protein content of the grains depends upon the level of nitrogen in the soil, the growing conditions and the total grain yield (FAO, 1995; Hulse et al., 1980; McDonough et al., 2000). Millets are rich in fiber, ash content, Vitamin B and also in certain minerals like magnesium, manganese, iron, copper, phosphorous and zinc (Web Ref. #1). Table 2.3 presents the proximate composition of the different millets.

Recent studies have shown that the fat content of minor millets and pearl millet is higher than that of wheat, rice and sorghum (Geervani and Eggum, 1989). The crude fat content of common millet and foxtail millet is higher than that of other millets and this is why they have a tendency of becoming rancid following milling (Ravindran, 1991). This can be avoided by storing them in airtight containers (Ravindran, 1991). The crude fiber content of millets is higher than that of wheat and rice (Geervani and Eggum, 1989; Ravindran, 1981). Millets vary in their carbohydrate content with finger millet being the richest source of carbohydrates (Ravindran, 1991). The fiber content of finger millet is higher than its carbohydrate content and this is what makes it a right

choice for diabetic consumers. The high levels of fiber help in decreasing the digestibility and thus, making it ideal for diabetics (Ravindran, 1991).

Table 2.3 Proximate analysis of cereals and millets per 100g edible portion (McDonough et al., 2000; FAO, 1995)

Cereal type	Protein (%)	Fat (%)	Crude fiber (%)	Ash (%)	Starch (%)	Ca (mg)
Rice (brown)	7.9	2.7	1.0	1.3	76.0	33
Wheat	11.6	2.0	2.0	1.6	71.0	30
Maize	9.2	4.6	2.8	1.2	73.0	26
Sorghum	10.4	3.1	2.0	1.6	70.7	25
Pearl millet	14.5	5.1	2.0	2.0	71.6	42
Finger millet	8.0	1.5	3.0	3.0	59.0	350
Foxtail millet	11.7	3.9	7.0	3.0	55.1	31
Proso millet	13.4	9.7	6.3	4.2	57.1	8
Little millet	9.7	5.2	7.6	5.4	60.9	17
Barnyard millet	11.8	4.9	14.3	4.9	60.3	22
Kodo millet	10.4	3.7	9.7	3.6	72.0	35

Though millets are rich in their mineral content, it varies greatly among the different varieties. The mineral content is affected by the environmental conditions at the time of growing and harvesting the crops (FAO, 1995). Usually the calcium content of cereals is in the range of 0.01-0.05% but finger millet has the highest calcium content of 0.24% (Ravindran, 1981).

Lysine, an essential amino acid, is limited in all millets (Geervani and Eggum, 1989; McDonough et al., 2000). Pearl millet and finger millet have the most lysine (McDonough et al., 2000). Proso millet has the poorest essential amino acid composition but when germinated it shows an increase in tryptophan, lysine and other free amino acids (Geervani and Eggum, 1989). The threonine and methionine content of minor millets is higher than that of other cereal grains (Geervani and Eggum, 1989). Barnyard millet has high levels of glutamic acid with concentrations varying from 16.37 ± 0.7 - 32.43 ± 0.5 mg/g, followed by leucine and alanine (Kim et al., 2011). Recent studies on barnyard millet show that storing the grains for a long time has a negative effect on the germination rate (Kim et al., 2011).

Dehusking of millets can lead to reduced levels of fiber and tannin (Geervani and Eggum, 1989). During dehusking, the aleurone and germ layers are removed which also cause a decrease in the level of vitamin B (McDonough et al., 2000).

The water binding capacity of the starch isolated from foxtail and proso millets is higher than that of wheat starch (McDonough et al., 2000). As compared to wheat, millet starches have higher amylograph viscosity (McDonough et al., 2000). The low pasting values and glutinous characteristics of starch are due to the low amylase content (Kim et al., 2011). Pasting properties play an important role in deciding the application of flours and starches. Finger millet starch has low solubility in water (McDonough et al., 2000).

The fat content as well as the level of unsaturated fatty acids in pearl millet is higher than that of other cereals. Also, it has high enzymatic-hydrolytic activity and does not have naturally occurring fatty acids. All these factors are responsible for the rapid development of off-flavors and off-odors following the milling of pearl millet (McDonough et al., 2000).

Processing of millets also affects their nutritional values. Studies on pearl millet show that soaking and sprouting have a negative effect on the value of lipids, carbohydrates and the tannin content of the grains (Obizoba and Atii, 1994).

Singh et al. (2004) reported the changes in functional properties of foxtail millet after popping, flaking, extrusion and roller drying. They reported that the degree of starch gelatinization was highest in the case of roller dried millet followed by popped and flaked and the least degree of gelatinization was found in extruded millet. It was also reported that the oil and water absorption properties were also influenced by the method of processing (Singh et al., 2004). While the popped, flaked, extruded and roller dried millet showed a 1.5 fold increase in the hot and cold water absorption capacity, the decorticated millet showed an increase of 9.2 times. The decorticated millet flour had the highest swelling power but the lowest solubility index whereas extruded millet flour had the lowest swelling power and the highest solubility index (Singh et al., 2004).

Pradeep and Guha (2010) reported that the different methods of processing, namely, germination, steaming and roasting, have a significant effect on the nutraceutical and antioxidant properties of little millet (*Panicum sumatrense*). The Total Phenolic Content of little millet increased from 453.3 mg GAE/100g (native) to 521.0 mg GAE/100g in the roasted millet, the order being roasted > steamed > native.

Dharmaraj and Malleshi (2011) reported that hydrothermal processing of finger millet decreases the overall extractability of proteins by 50% and also leads to slight thermal degradation of starch. It causes no change in the nutrient composition of the millet but changes their profile. The hydrothermally processed finger millet increased its carbohydrate and protein digestibility by 12% as compared to its native form (Dharmaraj and Malleshi, 2011).

2.4 POSTHARVEST TECHNOLOGY

2.4.1 Storage

The primary aim of storage is to maintain the quality of the grain for future use (FAO, 1995). Grains are stored either to be used as food for a long period or to use as seeds for the next harvest season. To prevent losses due to microbiological activity during storage, it is important to control the moisture of the grains and the storage temperature. In order to limit the movement of insects, the grains are packed very tightly or at times with sand so as to fill the intergranular spaces

(FAO, 1995). Millets are less susceptible to damage caused by insects and pests (McDonough et al., 2000). One reason for this could be that millets are usually grown in dry areas of the world where the relative humidity is low thus, making it unfavorable for insect growth (McDonough et al., 2000). Millets can be stored in silos, small on-farm granaries, jute bags and even clay pots (FAO, 1995).

2.4.2 Methods of processing

It is unusual to eat cereals as raw, uncooked whole grains and this is why cereals are processed. Processing not only improves the organoleptic properties of the millets but also increases their digestibility. Different processing methods have different effects on the composition and the nutritive quality of food.

2.4.2.1 Milling

The primary objective of milling is to remove the hull (FAO, 1995). This is usually done by pounding followed by sieving at various stages to sift fine particles, coarse particles and bran (McDonough et al., 2000). The grains are either moistened with water or soaked overnight in preparation for milling (FAO, 1995). When pounding soft grains, the endosperm breaks into small particles and is separated by sieving and screening while in hard grains, the endosperm remains intact and is removed by winnowing (FAO, 1995). Hand pounding is labor intensive, inefficient and time consuming (McDonough et al., 2000). Traditional methods of grinding make use of two round grinding stones that rotate horizontally against each other or with a mortar and pestle (McDonough et al., 2000). Modern technology involves the use of abrasive disks in mechanical dehullers (emery boards) or attrition type dehullers (FAO, 1995; Hulse et al., 1980, McDonough et al., 2000). To ensure greater shelf-life of the flour, the milling process should be able to remove most of the germ (FAO, 1995). Wet milling process involves overnight soaking of the grains followed by grinding into a batter (McDonough et al., 2000; FAO, 1995).

2.4.2.2 Steaming

A method of thermal processing, steaming, involves preconditioning or steeping of grains and then heating them under steam at high pressure in an autoclave. Complete gelatinization of kernel starch ensures that the grain has been steamed properly (Shobana and Malleshi, 2007). Steaming of millets has been reported at pressures between 0-5 kg/cm^2 (Pradeep and Guha, 2010; Shobana and Malleshi, 2007). Decorticated steamed grains can be consumed as such or can be flaked (Subramanian et al., 1986). Steaming leads to changes in the texture of millet endosperm, the changes being similar to those observed in parboiled rice (Shobana and Malleshi, 2007). Finger millet has a floury endosperm and this makes decortication difficult. Steaming followed by low temperature drying causes changes in the grain endosperm making it corneous (hard). The millet can then be decorticated in a mill. The decorticated millet can then be cooked as rice (Shobana and Malleshi, 2007).

2.4.2.3 Popping

Corn is unambiguously the oldest cereal that was popped and used as a snack food. It was after the principle behind the popping of corn was unfolded and the structure of various other grains studied that other grains were also considered for popping. It is not usual for all cereals to pop (Johnson, 2000). To pop well, a few conditions must be fulfilled. Corn pops exceptionally well as it has a hard pericarp which ruptures at a combination of high internal pressure and very high temperature of about 177°C (Johnson, 2000).

Millets like sorghum, finger millet and foxtail millet are also known to pop. As studied in corn, popping occurs when the kernel is heated to high temperatures (Johnson, 2000). When the temperature of the kernel rises, the moisture inside the kernel vaporizes to form steam and this steam fills the voids between the starch granules within the kernel (Lara and Ruales, 2002). This steam creates a pressure build-up inside the grain. This increase in temperature and pressure melts the starch granules and ruptures the pericarp leading to an explosive expansion (Hulse et al, 1980). The starchy endosperm expands as a flower. The starch granules of the endosperm form a soap bubble structure (Hulse et al., 1980; Lara and Ruales, 2002). As the water vaporizes, the starch granules solidify to yield a spongy matrix (Lara and Ruales, 2002).

The moisture content of the grain before and after popping plays a significant role in determining the flavor, tenderness, popped volume and the shape of the popped kernel (Johnson, 2000). Hoseney et al. (1983) stated that too little or too high of a moisture content resulted in a lower degree of expansion of popcorn kernels. The optimum moisture content at which popcorn kernels expand best is around 13% - 17%.

Mangala et al. (1999) reported that popping not only enhances the color, aroma, flavor and appearance of the raw product but also improves the carbohydrate and protein digestibility. Popping inactivates some of the enzymes and enzyme inhibitors thus, improving the nutritional quality. Muralikrishna et al. (1986) studied the effects of popping on the physicochemical properties of starch of pearl millet, foxtail millet and finger millet, and found out that the starch granules of popped millets lost their birefringence characteristics, typical of native starch. Birefringence is the phenomenon observed when starch granules are viewed under a microscope using polarized light. This polarized light gets refracted by the crystalline regions of the starch granules and gives a characteristic 'Maltese Cross' pattern. When starch granules are heated in a starch-water system, the starch granules lose their crystalline structure which leads to the disappearance of the crosses and helps in determining the gelatinization temperature. Muralikrishna et al. (1986) also confirmed that starch from popped millets exhibited higher solubility and swelling power which could be due to the spongy starch matrix formed due to partial gelatinization of starch on popping.

Studies conducted on foxtail millet by Singh et al. (2004) show that the cold oil absorption capacity of the popped millet was higher than that of flaked, extruded and roller-dried millet. One reason for this could be the inter-granular spaces created by popping in the endosperm (Okpala and Mamah, 2001). These inter-granular spaces are capable of retaining oil thus, accounting for the higher oil absorption capacity.

2.5 MEDIUM FOR POPPING

The medium used for popping must be capable of reaching high temperature and of effectively transferring heat to the food material. The conventional methods make use of hot air, hot oil or hot sand as mediums for popping. These mediums are usually heated to temperatures ranging

between 200-250°C. Low temperatures do not support popping whereas too high a temperature can impart a burnt flavor to the food. The current technological development makes use of microwave energy to pop grains. Corn pops exceptionally well in a microwave oven as the hull (pericarp) of a popcorn kernel is very hard which allows for gradual heating of the grain and thus leads to a high pressure build-up inside the grain (Mohamed at al., 1993)

Conventional methods of popping make use of hot air, though it has been shown to be an inefficient heat transfer medium (Sibley and Raghavan, 1985). Using air as a medium can increase the popping time as air reaches saturation level before all the sensible heat has been utilized because air not only transfers its heat to the sample but also helps in removing the moisture which is released when the sample is heated. As both of these processes occur simultaneously, by the time air heats up the sample, it becomes saturated with the moisture released from the sample thus, increasing the time of popping. Heat transfer by conduction using a particulate medium serves as an effective heat transfer method as the heat capacity of solids is much higher than that of air. Popping using 'particulate medium' involves immersion of the grains in a bed of hot granular medium, such as salt or sand, for a very short contact time with continuous mixing. A plus point of using particulate medium is that it causes uniform heating of the grains in a very short time (Sibley and Raghavan, 1985).

Sand is the most common particulate medium used because of its easy availability and low cost (Sotocinal et al., 1997). Other particulate materials that are used are salt, zeolite, silica and molecular sieves. Salt has been used as it has better heat transfer characteristics and is also non-toxic in nature. The factors that affect popping in a particulate medium are initial temperature, initial grain moisture, particulate medium to grain mass ratio and mixing (Sotocinal et al., 1997).

Grains are mixed directly with the hot particulate medium and the processed grains are separated from the medium by screening (Sotocinal et al., 1997). A perforated metal sheet or a wire mesh is usually used for this purpose. The grains are cooled to room temperature before storage.

2.6 FOOD VALUES OF MILLETS

Millets are consumed directly as food in Africa, India, China and southern Russia (McDonough et al., 2000). Sorghum, pearl millet and finger millet are the most common millets used in India. Pearl millet flour is used for making *rotis* or *chapattis* (unleavened flat pancakes) (McDonough et al., 2000). Millets are used for making porridges and can also be cooked as rice (Shobana and Malleshi, 2007). Millet finds a greater use as food in Africa where it is usually consumed as flatbreads (fermented or unfermented), thick and thin porridges (fermented or unfermented), alcoholic beverages, snacks, and steamed or boiled cooked products (Baltensperger and Cai, 2004; McDonough et al., 2000). Composite flour made from millets is used for making noodles, bread, cookies, etc. (McDonough et al., 2000). Malt and alcoholic beverages are prepared from pearl millet whereas sorghum is used for making opaque sour beers. Pearl millet can be used to make lager beer by replacing 25% of barley malt and the beer thus produced has organoleptic and analytical characteristics similar to standard beer. In Nigeria, pearl millet has substituted sorghum in making *ndaleyi* and *kunun gyada* (a weaning food) (McDonough et al., 2000). Pearl millet is also used to make a type of gruel called *bulummardam* which is prepared by blending it with baobab flour (Baltensperger and Cai, 2004). Millet is also dry-milled into flour and is mixed with wheat flour for making bread and biscuits (Baltensperger and Cai, 2004).

20-35% proso millet flour mixed with wheat flour is used for making breads (McDonough et al., 2000). Japanese barnyard millet is good for people with allergic diseases like atopic dermatitis (Kim et al., 2011). In Korea, barnyard millet is generally used as animal forage, cereal crop and as a constituent of soup (Kim et al., 2011). Puffed or popped finger millet is used as a snack food in India (Singh et al., 2004). In Inner Mongolia, it is a common practice for people to add fried millets to milk tea or butter tea as millets help in enhancing the flavor and texture of the tea (Baltensperger and Cai, 2004; Web Ref. #22; Web Ref. #23).

Finger millet seed coat is a good source of fiber but as it imparts a dark color and chewy texture to the food it is usually separated from the grains. Krishnan et al. (2011) studied the quality characteristics of biscuits made from composite flour of finger millet seed coat matter and wheat aiming at incorporating the vitamin and phyto-nutrient rich seed coat into edible food products.

Millets have been used traditionally to prepare weaning foods. One such weaning food that is prepared by combining pearl millet with amaranth, green gram and jaggery has been found to have low cooked paste viscosity and high energy density when mixed with green gram flour (FAO, 1995). The weaning food thus prepared has been reported to have nutritional value that is comparable to commercially available weaning foods such as Cerelac (prepared by roller drying wheat or maize and skim milk powder fortified with minerals and vitamins) which is a good source of macro and micronutrients and energy (Adeyemi et al., 1989; McDonough et al., 2000). Germinated sorghum flour is also used as a weaning food as it reduces the viscosity of the food product. Sorghum and millet based weaning foods which are prepared using malting and extrusion techniques have been promoted as high energy and high protein foods (FAO, 1995).

As millets are not only rich in proteins, carbohydrates, minerals, fats and fiber but also have balanced amino acid content, the consumption of millets on a daily basis could help prevent and reduce certain human diseases (Baltensperger and Cai, 2004).

2.7 FUTURE OF MILLETS

Millets are capable of growing in adverse climatic conditions and are early maturing crops. They are a nutritious food for a large segment of the population, especially in Asia and Africa (FAO, 1995). Millets are an excellent source of fibers and proteins and are currently being used directly as food and also in the formulation of functional foods (McDonough et al., 2000). More research on millets will ensure a greater market for millet based products.

Chapter 3

Materials and Methods

3. MATERIALS AND METHODS

3.1 Materials

Little Millet (var. Sukshema) was procured from University of Agricultural Sciences, Dharwad, Karnataka, India. The millet was cleaned of dirt and other particles and stored in aerated sacs. Pierce BCA Protein Assay Kit (Rockford, IL, USA) was used for determining the protein content and Megazyme Resistant Starch Kit (Megazyme, Ireland) was used for analyzing the starch content of the flour samples. All chemicals used were of analytical grade (Fisher Scientific, Ottawa, Canada, and Sigma-Aldrich, Canada).

3.2 Equipment and apparatus

The particulate medium used for popping, table salt (Brand, Sifto), was heated using a Gas Chromatograph Oven (HP 5890A). A rheometer (Model - Advanced Rheometer AR 2000, TA Instruments) was used to measure the viscosity and the pasting characteristics of the flour samples.

3.3 Design of experiment

A Central Composite Design (CCD) was used to determine the optimal popping conditions of the millet. The reason behind choosing the CCD lies on the fact that the CCD is effective in revealing the effects and interactions between different factors on a particular response (Box and Wilson, 1951). The CCD was performed using Design Expert Software (Ver. 6, Stat-Ease Inc., Minneapolis, MN). Temperature of the popping medium (°C) and the initial moisture content of the millet (%) were taken as the independent factors at three levels each. Preliminary experiments were conducted to get a range of values for the temperature and the moisture content. Thirteen combinations of three different temperatures (220°C, 240°C and 260°C) and three different moisture contents (14%, 16% and 18%) including five central points were studied. All experiments were conducted in duplicates.

Table 3.1 Central Composite Design for optimizing the popping conditions of little millet

Run	Temperature of popping medium (°C)	Initial moisture content of millet (%)
A	260	14
B	240	14
C	220	14
D	220	16
E	240	16
F	240	16
G	240	16
H	240	16
I	240	16
J	260	16
K	260	18
L	240	18
M	220	18

3.4 Popping of little millet

The two most important factors that govern the popping of a grain are the temperature of the popping medium and the initial moisture content of the grain. The temperature of the medium should be high enough to vaporize the moisture present inside the grain and to cook the starch so that it gelatinizes partially. The moisture content of the grain also plays a crucial role because too little a moisture would not create the sufficient pressure inside the grain required to burst open and too high a moisture would damage the grain and burst the seed coat preventing the necessary pressure build-up. Thus, a precise balance of temperature and moisture is required to get that perfectly popped grain.

Thus, in order to adequately pop, the grain kernels need to be at a specific level of moisture. For this purpose, the millet grains were tempered with a definite calculated volume of water. To ensure that the added volume of water brought the millet to the desired moisture level, 5g of the millet was kept in a hot air over overnight at 105°C and the moisture content was calculated by AACC Method 44-15.02 (described later). It was observed that the calculated volumes of water were just sufficient to bring the millet to the desired three levels of moisture.

Samples of 300g of little millet, initially at 9.2% moisture level, were thus tempered with a calculated volume of water of 16.74 ml, 24.285 ml and 32.194 ml to bring the three different samples to the desired moisture content of 14%, 16% and 18%, respectively. The millet was soaked in closed glass containers for a period of 18 hours with intermittent mixing to ensure uniform absorption of water. The particulate medium for popping, 500g of table salt (Brand, Sifto), was heated to the desired temperature in a Gas Chromatograph oven (HP 5890A). As the ratio of the grain mass to the particulate medium is an important factor in determining the yield, only 20g of millet was popped at a time. Thus, the ratio of the particulate medium to the grain mass was 25:1.

Little millet was mixed with the hot salt using a rubber spatula and popping was observed in less than two minutes (Fig. 3.1). The popped grain was manually sifted from the unpopped grain and salt using a metallic strainer. The percentage of the popped and the unpopped millet was calculated. The popped grain was stored in air-tight containers.

Fig. 3.1 Popping of little millet

The yield of popped millet was calculated using the following equation:

$$\textbf{\textit{Yield (\%)}} = \frac{Weight\ of\ popped\ \ millet\ \ (g)}{Weight\ of\ popped\ \ millet\ \ (g) + Weight\ of\ unpopped\ \ millet\ \ (g)} \times 100 \qquad (3.1)$$

A coffee grinder was used to pulverize the popped millet into flour. The particle size of the flour was measured by Soiltest Mechanical Shaker (CL-305A, IL, US) using Fischer Brand standard sieves. The particle size distribution of the flour varied as:

Particles larger than 500µm: – 7.748%

Particles between 500µm - 250µm – 65.879%

Particles between 250µm - 150µm – 19.497%

Particles between 150 µm – 100 µm – 6.875%

The flour samples were used to study the effect of popping on the proximate components, namely protein, fat, resistant starch, moisture content and total ash content. Total phenolics and other functional properties namely swelling power and solubility, oil absorption, viscosity and the pasting characteristics of millet flour were also studied and detailed later on. Native millet flour (with husk) was used as control.

3.5 Proximate Analysis

3.5.1 Moisture Content:

The moisture content was determined using the AACC protocol (AACC Method 44 – 15.02, 1999). 3-5 g of flour samples were weighed and placed overnight in a hot air oven maintained at 105°C. The weight of the samples was recorded after 24 hours of drying and the difference in the initial and final weight was calculated. The percentage moisture content was calculated using equation 3.2:

$$\% \textit{ Moisture} = \frac{\text{Diffe rence in weight (g)}}{\text{Initial weight (g)}} \times 100 \qquad (3.2)$$

3.5.2 Protein:

The standard method for the estimation of proteins in cereal based compounds is the Kjeldahl method (AACC, 1986). Though highly reliable, this method is labor-intensive as it involves separate steps for protein digestion and quantification by titration which leads to limited samples being analyzed at a time. Another drawback associated with the Kjeldahl method is that it can lead to overestimation of proteins in samples which may contain a large proportion of non-protein nitrogen. A number of rapid methods are available for the quantification of proteins. These include colorimetric methods like the Lowry assay, the Bradford assay and the Bicinchoninic Acid (BCA) assay.

The International Union of Pure and Applied Chemistry (IUPAC) name for BCA is 2,2'-diquinonyl-4,4'-dicarboxylic acid. BCA assay is highly sensitive in quantifying insoluble proteins. The BCA assay has leverage over other methods as it has decreased sensitivity to interferences, exhibits color stability, needs just one working reagent and is time efficient. The assay involves two steps – first, the reduction of Cu^{2++} to Cu^{+} by the protein, and second, the complex formation between Cu^{+} and BCA to form a purple chromophore which is freely soluble in aqueous solution. The purple chromophore is formed by the chelation of one Cu^{+} and two BCA molecules (Fig 3.2).

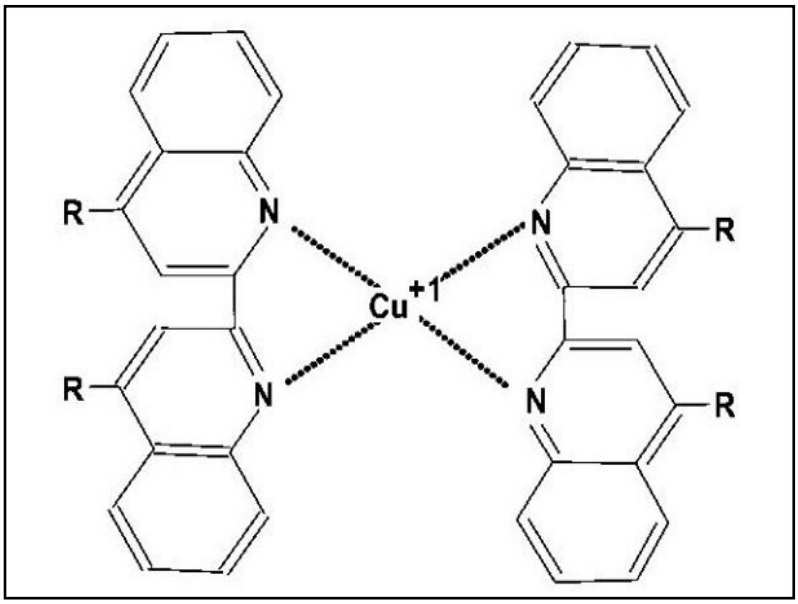

Fig. 3.2 Complex formation between 2,2'-bicinchoninic acid and Cu+

Source: Owusu-Apenten, 2002

The total protein content of all millet flour samples was determined by the Bicinchoninic Acid assay as described by Chan and Wasserman (1993).

3.5.2.1 Preparation of BCA Working Reagent (WR):

The working reagent was prepared by mixing 50 parts of BCA reagent A with one part of reagent B to give a green colored solution. Addition of one reagent to the other creates turbidity which disappears quickly once the reagents are mixed.

3.5.2.2 Preparation of Bovine Serum Albumin standards:

A standard curve (0-500 µg/ml) was prepared using Bovine Serum Albumin at 2mg/ml in 0.9% saline and 0.05% sodium azide. The samples containing different concentrations of BSA were brought to 1 ml using BCA working reagent and were incubated for 30 minutes at 37°C. A dark

purple colored solution was formed. To stop the reaction, the samples were cooled in an ice water bath for 5 minutes. 0.4 ml of the sample was diluted with 1.6 ml of BCA reaction buffer A and mixed thoroughly. The absorbance was read at 562 nm.

3.5.2.3 Test procedure:

10 mg of the sample was weighed in a 2 ml microcentrifuge tube and mixed with 1 ml of BCA working reagent followed by incubation at 37°C for 30 minutes in a water bath. The samples were mixed intermittently on a vortex mixer every 10 minutes. A dark purple colored solution was formed. To stop the reaction, the samples were cooled in an ice water bath for 5 minutes followed by centrifuge at 3300 rpm for 10 minutes. 0.4 ml of the supernatant was taken in another centrifuge tube and was diluted with 1.6 ml of BCA reaction buffer A. The solution was mixed well with a vortex mixer and its absorbance was read at 562 nm.

3.5.3 Resistant Starch (RS) :

As the name suggests, resistant starch is tolerant to hydrolysis by enzymes present in the small intestine. It enters the large intestine where it is fermented. Resistant starch forms one of the components of Total Dietary Fiber. Popping is believed to increase the digestibility of starch by increasing its availability for enzymatic digestion.

Resistant starch content of the samples was analyzed by using Megazyme Resistant Starch Assay Procedure which is based on AOAC Method 2000.02 and AACC Method 32-40. The method involves the solubilisation of non-resistant starch in the presence of pancreatic α-amylase and amyloglucosidase (AMG) over a period of 16 hours at 37°C. These two enzymes solubilize the non-resistant starch and hydrolyze it to D-glucose. To terminate the reaction, an equal volume of ethanol is added and the contents are centrifuged. The RS, recovered as a pellet, is washed twice with aqueous IMS (Industrial Methylated Spirit) and centrifuged. The supernatant is decanted and the RS pellet is dissolved in 2M KOH by vigorous stirring in an ice-water bath. Acetate buffer is added to neutralize this solution. AMG is then used to hydrolyze the RS to D-glucose

which is then measured using GOPOD (glucose oxidase/peroxidase) reagent. The supernatants from all the three washings are mixed and non-resistant starch, which is already present in the hydrolyzed form, is tested for D-glucose with GOPOD reagent.

Reagents provided in the kit:

Bottle 1 – Amyloglucosidase [12 ml, 3300 U/ml on soluble starch] at pH 4.5 and 40°C.

Bottle 2 – Pancreatic α-amylase (Pancreatin, 10 g, 3 Ceralpha Units/mg). Can remain stable for more than 3 years at 4°C.

Bottle 3 – GOPOD Reagent Buffer. Potassium phosphate buffer (1M, pH 7.4), p-hydroxybenzoic acid (0.22M) and sodium azide (0.4% w/v). This buffer can remain stable for more than 3 years at 4°C.

Bottle 4 – GOPOD Reagent Enzymes. Glucose oxidase (> 12,000 U) plus peroxidase (> 650U) and 4-aminoantipyrine (80 mg). Freeze dried powder. Can remain stable for more than 5 years at -20°C.

Bottle 5 – D-Glucose standard solution (5 ml, 1.0 mg/ml) in 0.2% w/v benzoic acid. This solution can remain stable for more than 5 years at room temperature.

Bottle 6 – Resistant Starch control. Resistant starch content shown on the label. Can remain stable for more than 5 years at room temperature.

3.5.3.1 Preparation of reagents (Not supplied in the kit)

i) Sodium maleate buffer (100 mM, pH 6) plus 5 mM calcium chloride dehydrate and sodium azide (0.02% w/v)

11.6 g of maleic acid was dissolved in 800 ml of distilled water and the pH was adjusted to 6.0 with 4 M (40 g/250 ml) sodium hydroxide solution. 0.37 g of calcium chloride dihydrate

($CaCl_2.2H_2O$) and 0.2 g of sodium azide were added to the buffer and dissolved well. The volume was adjusted to 1L. The buffer can remain stable for 12 months at 4°C.

ii) Sodium acetate buffer (1.2 M, pH 3.8)

69.6 ml of acetic acid was added to 800 ml of distilled water and the pH adjusted to 3.8 using 4 M sodium hydroxide solution. The volume was made to 1L with distilled water. The buffer can remain stable for 12 months at room temperature.

iii) Sodium acetate buffer (100 mM, pH 4.5)

5.8 ml of acetic acid was added to 900 ml of distilled water and the pH adjusted to 4.5 using 4 M sodium hydroxide solution. The volume was made to 1L with distilled water. This buffer can remain stable for 2 months at 4°C.

iv) Potassium hydroxide solution (2M)

28.05 g of KOH was added to 225 ml distilled water and dissolved by stirring. The volume was made to 250 ml. The solution can remain stable for 2 years at room temperature.

v) Aqueous ethanol or IMS (approx. 50% v/v)

500 ml of industrial methylated spirit (IMS; denatured ethanol; ~95% v/v ethanol plus 5% methanol) was added to 500 ml of distilled water and mixed well. The solution can remain stable for 2 years at room temperature.

3.5.3.2 Preparation of reagents solutions/suspensions (Supplied in the kit)

i) Dilute AMG: The AMG solution (12 ml, 3300 U/ml) as provided in the kit was very viscous. 2 ml of this solution was diluted to 22 ml with 0.1 M sodium maleate buffer (pH 6). The solution was divided into aliquots of 5 ml and stored frozen. This solution can remain stable to repeated freeze-thaw cycles, and for five years at -20°C.

ii) This dilute AMG was used to prepare a fresh solution of pancreatic α-amylase. 1 g of pancreatic α-amylase was mixed with 100 ml of sodium maleate buffer (100 mM, pH 6) and

mixed for 5 minutes. To this, 1 ml of dilute AMG (300 U/ml) was added and mixed well. This solution was centrifuged at 3300 rpm for 10 minutes and the supernatant was collected. This supernatant was used fresh.

iii) GOPOD reagent buffer (bottle 3) was diluted with 1L of distilled water and used right away.

iv) GOPOD Reagent Enzymes (bottle 4) were dissolved in 20 ml of diluted GOPOD reagent buffer (solution iii) and mixed with the rest of solution iii. The reagent formed by mixing GOPOD Reagent Buffer and GOPOD Reagent Enzymes is called **GOPOD Reagent** (Glucose Determination Reagent). As this reagent was light-sensitive, the storage bottle was covered with a thick aluminium foil. GOPOD Reagent remains stable for 3 months at 2-5°C or for 1 year at -20°C.

***Note*: (i)** Freshly prepared GOPOD Reagent is light pink or light yellow in color but develops a darker color over a period of 2-3 months even at 4°C. If this reagent is stored frozen then it should be so divided into aliquots so that they can be used for only one freeze-thaw cycle.

(ii) The buffers and the reagents were used within the stability period ensured by Megazyme.

3.5.3.3 Test Procedure

3.5.3.3.1 Hydrolysis and solubilisation of non-resistant starch

100 mg of flour was weighed in a 15 ml centrifuge tube. The tubes were capped and tapped gently so that the flour fell to the bottom. 4 ml of freshly prepared pancreatic α-amylase containing dilute AMG (3 U/ml) was added to each tube. The contents were mixed properly using a vortex mixer and were then fixed in a shaking incubator maintained at 37°C for exactly 16 hrs. The shaking speed was set at 200 strokes per minute. The tubes were removed from the incubator and the contents were treated with 4 ml of ethanol (99% v/v) with vigorous stirring on a vortex mixer.

The tubes were then centrifuged at 3300 rpm for 10 minutes. The supernatants were decanted carefully in separate 50 ml centrifuge tubes and the pellets were re-suspended in 2 ml of 50 %

IMS. The contents were mixed vigorously on a vortex mixer. Another 6 ml of 50 % IMS was added to the tubes, mixed and the tubes were centrifuged at 3300 rpm for 10 minutes. The supernatants were decanted and the suspension and centrifugation step was repeated once more. The supernatants were decanted and the tubes were inverted on paper towel to drain any excess liquid.

Note: While decanting the supernatant, it was observed that the flour pellet did not settle completely at the bottom and had a tendency of flowing out along with the last 1-2 ml of the supernatant. To overcome this, the tubes were centrifuged again for another 10 minutes.

3.5.3.3.2 Measurement of Resistant Starch:

A magnetic stirrer bar was added to each tube and they were placed in an ice water bath over a magnetic stirrer. 2 ml of 2 M KOH was added to each tube to re-suspend the pellets. This was done to dissolve the resistant starch. It was important to keep the contents stirring vigorously while adding KOH as otherwise lumps of starch would form which are difficult to dissolve. The stirring was carried out for 20 minutes. Mixing was not carried out on a vortex mixer as it would cause emulsification of the starch.

8 ml of 1.2 M of sodium acetate buffer (pH 3.8) was added to each tube while stirring on the magnetic stirrer. This was followed by addition of 0.1 ml of AMG (3300 U/ml; solution 1). The contents were mixed well on the magnetic stirrer and the tubes were then placed in a water bath maintained at 50°C. The tubes were incubated for 30 minutes with intermittent mixing on a vortex mixer every 10 minutes.

The tubes, without any dilution, were centrifuged at 3300 rpm for 10 minutes. 0.1 ml of this undiluted supernatant was transferred to another tube and mixed with 3 ml of GOPOD reagent. A reagent blank was prepared by mixing 0.1 ml of 100 mM sodium acetate buffer (pH 4.5) and 3 ml of GOPOD reagent. D-glucose standard solution was prepared by mixing 0.1 ml of D-glucose (provided in the kit) with 3 ml of GOPOD reagent. GOPOD reagent was added to the samples, reagent blank and D-glucose standard solution at the same time and all the tubes containing the samples, reagent blank and D-glucose solutions were incubated at 50°C for 20 minutes. A light

pink colored solution was formed. The tubes were brought to room temperature and absorbance was read at 510 nm against reagent blank. The RS content was determined as follows using equation 3.3 :

Resistant Starch (g/100g sample), for samples containing less than 10% RS:

$$RS = \Delta E \times F \times (10.3/0.1) \times (1/1000) \times (100/W) \times (162/180)$$

$$RS = \Delta E \times (F/W) \times 9.27 \quad (3.3)$$

Where,

ΔE = absorbance read against reagent blank

F = 100 (µg of D-glucose) / GOPOD absorbance for this 100 µg of D-glucose

100/0.1 = volume correction (0.1 ml taken from 100 ml)

1/1000 = conversion from µg to mg

W = weight of sample (db)

100/W = factor to present RS as a percentage of sample weight

162/180 = factor to convert from free D-glucose to anhydro D-glucose as occurs in starch

10.3/0.1 = volume correction (0.1 ml taken from 10.3 ml) for samples containing less than 10% RS where the incubation samples are used as such and the final volume is nearly 10.3 ml.

3.5.3.3.3 Measurement of Non-Resistant (Solubilised) Starch:

The supernatant solutions that were collected after the centrifugation of the initial washing with 99% ethanol and after two subsequent washings with 50% IMS were combined and the volume was made up to 100 ml in a volumetric flask using 100 mM sodium acetate buffer (pH 4.5). The contents were mixed well. 0.1 ml of this solution was mixed with 10 µL of dilute AMG solution (300 U/ml) and incubated for 20 minutes at 50°C. Reagent blank and D-glucose standard

solution was prepared as earlier. 3 ml of GOPOD reagent was added to the samples, reagent blank and D-glucose standard solution and the tubes were incubated at 50°C for another 20 minutes. A dark pink colored solution was formed. The tubes were brought to room temperature and absorbance was measured at 510 nm. The Non-Resistant Starch content was determined as follows using equation 3.4 :

Non-Resistant (Solubilised) Starch (g/100g sample):

$$= \Delta E \times F \text{ X } (100/0.1) \times (1/1000) \times (100/W) \times (162/180)$$

$$= \Delta E \times (F/W) \times 90 \qquad (3.4)$$

Where,

ΔE = absorbance read against reagent blank

F = 100 (µg of D-glucose) / GOPOD absorbance for this 100 µg of D-glucose

100/0.1 = volume correction (0.1 ml taken from 100 ml)

1/1000 = conversion from µg to mg

W = weight of sample (db)

100/W = factor to present RS as a percentage of sample weight

162/180 = factor to convert from free D-glucose to anhydro D-glucose as occurs in starch

10.3/0.1 = volume correction (0.1 ml taken from 10.3 ml) for samples containing less than 10% RS where the incubation samples are used as such and the final volume is nearly 10.3 ml.

The Total Starch Content was calculated as the sum of resistant starch and non-resistant starch using equation 3.5:

Total Starch = Resistant Starch + Non-Resistant Starch (3.5)

3.5.4 Total Ash:

The ash content was analyzed using the standard AACC protocol (AACC Method 08 – 01.01, 1999). 2 g of samples were weighed in crucibles and kept in hot air oven overnight at 105°C. The weight of the dried samples was taken and the samples were kept in a muffle furnace at 500°C for 16 hours. The crucibles were cooled to room temperature in a desiccator and the weight of ash was taken. The percentage ash was calculated using equation 3.6:

$$\% \, Ash = \frac{Weight \; of \; ash \; (g)}{Weight \; of \; sample \; ,dry \; basis \; (g)} \times 100 \qquad (3.6)$$

3.5.5 Crude Fat:

Crude fat in food samples is determined as the change in weight recorded after exhaustively extracting the food sample with a non-polar solvent. The conventional AACC method (AACC Method 30 - 25.01, 1999) involves the use of the Soxhlet apparatus which has three parts – the reactor, where the food sample is added, a condenser and the lower chamber (usually a flat bottomed or round bottomed flask) that contains the reservoir for the organic solvent.

About 3 g of sample was weighed in a thimble and its mouth was plugged using a cotton plug to prevent the sample from overflowing during extraction. The thimble was placed in the upper chamber i.e., the reactor. Petroleum ether was used as the solvent for extraction. About 175 ml of the solvent was taken in a flat bottomed flask and a magnetic bead was added to it to ensure uniform mixing while heating. The reactor was fitted at the mouth of the flask and the condenser was fitted at the mouth of the reactor. The set-up was placed on a magnetic hot plate. As the solvent reached its boiling point, it started vaporizing. The vapors went to the condenser through the side arm and started condensing at the walls of the reactor thus, filling it. The solvent percolated through the sample and reached the flat bottomed flask extracting oil from the sample. This cycle was run twelve times till all the fat present in the sample was extracted. The set-up was removed from the hot plate and allowed to cool.

The flat-bottomed flask was fixed at the mouth of the rotavapor fixed with a condenser. The solvent was allowed to evaporate and condense. The solvent was collected in a separate flask fitted with the condenser while oil remained in the flat-bottomed flak. The weight of oil was taken and the percentage fat in the sample was calculated per gram sample dry basis.

$$\textbf{\textit{Crude fat (\%)}} = \frac{Weight\ of\ oil\ (g)}{Weight\ of\ sample\ ,dry\ basis\ (g)} \times 100 \qquad (3.7)$$

3.6 Functional Properties

3.6.1 Total Phenolic Content

The Total Phenolic Content (TPC) was determined by the Folin-Ciocalteau's Assay as modified by Singh et al. (2011). Gallic acid standard solution was prepared at a concentration of 1mg/ml and a calibration curve was plotted for the same (R^2 = 0.980). Methanol was used as the extraction solvent.

3.6.1.1 Preparation of extract:

1g of millet flour was taken in a 15 ml centrifuge tube and suspended in 10 ml of methanol. The contents were mixed vigorously for 5 minutes using a vortex mixer. The tubes were then centrifuged at 300 rpm for 15 minutes. As popped millet flour forms two layers when mixed with any solvent, it was filtered using Whatman No. 1 filter paper. The flour remained on the filter paper and the clear extract was collected.

3.6.1.2 Test Procedure:

10 ml of the extract was added to 40 ml distilled water and was mixed using a vortex mixer. 1 ml of this solution was taken in a 15 ml centrifuge tube and to it 7.5 ml of double distilled water was

added followed by 0.5 ml of Folin-Ciocalteu's reagent (Sigma-Aldrich, Canada) and 1 ml of 7.5% sodium carbonate solution. The contents were mixed using a vortex mixer. The tubes were incubated in dark for 30 minutes. A dark blue color developed and the absorbance was read at 765 nm. The result was expressed as mg of gallic acid equivalent (GAE) / 100g of sample (db).

3.6.2 Swelling Power and Solubility:

Swelling power and solubility were determined according to the procedure of Schoch (1964) as modified by Unnikrishnan and Bhattacharya (1981). About 500 mg of sample was cooked with 10 ml of distilled water for 30 minutes in a water bath maintained at different temperatures, namely 30°C, 50°C and 98°C. The cooked slurry was transferred to 15 ml centrifuge tubes. The weight of the slurry was made equivalent to 15 g by adding distilled water. The tubes were centrifuged at 3300 rpm for 15 minutes. Supernatant was collected and the residue was weighed for the determination of swelling power. 10 ml of the supernatant was taken in a petri dish and kept on a hot plate to evaporate. The dishes were dried at 105°C for 3 hrs in a hot air oven, cooled and weighed to determine the solubility. The swelling power and solubility were calculated using the following equations:

$$\textbf{\textit{Swelling Power}} = \frac{\text{Weight of wet residue (g)}}{\text{Weight of sample (db)(g)} - \text{Weight of dry residue (g)}} \tag{3.8}$$

$$\textbf{\textit{Solubility}} = \frac{\text{Weight of dry residue (g)}}{\text{Weight of sample (db)(g)}} \times 1.5 \times 100 \tag{3.9}$$

Where,

1.5 = correction factor. 10 ml of supernatant was taken from an equivalent weight of 15g of flour slurry.

3.6.3 Oil Absorption:

The oil absorption capacity of the flour was determined by the method of Lin and Humbert (1974). 8g of oil was taken in a pre-weighed 15 ml centrifuge tube and 1g flour was added to it. The contents were mixed vigorously for 5 minutes using a vortex mixer. The tubes were incubated for 30 minutes at 30°C in a water bath and then centrifuged at 3300 rpm for 25 minutes. The supernatant oil was decanted through a pre-weighed filter paper as popped millet flour when mixed with any solvent but water, forms two layers – the heavier particles along with the husk settle at the bottom and the lighter particles form a layer at the surface of the solvent. The weight of the residues with oil absorbed was recorded.

To determine the oil absorption capacity of the sample at 140°C, 5 g of popped millet was taken in a metallic tea-strainer and dipped in oil maintained at 140°C for 15 ± 3 seconds (Singh et al., 2004). The surface oil was blotted off with a paper towel and the sample was transferred to a thimble. The oil content of the samples was determined using the Soxhlet apparatus. Petroleum ether was used as the extraction solvent.

3.6.4 Viscosity:

The viscosity of the flour was measured at room temperature and also after cooking. A 20% w/v slurry (Singh et al., 2004 and, Li and Yeh, 2001) was prepared by mixing 2.5 g of flour with 12.5 ml distilled water and was left at 30°C for 30 minutes with intermittent stirring. The cold paste viscosity was measured at a shear rate of 42.5 s^{-1} using a controlled stress rheometer (AR 2000 Rheometer, TA Instruments) operated with a parallel plate geometry of 40 mm diameter and a gap of 1 mm. To simulate the cooking condition in the kitchen, 2.5 g of flour was suspended in 12.5 ml of distilled water maintained at 100°C (Brandtzaeg et al., 1981). The paste was allowed to cool down to room temperature and the cooked paste viscosity was determined.

3.6.5 Pasting Characteristics:

The pasting properties were measured according to the method of Ibanez et al. (2007). A controlled stress rheometer (AR 2000 Rheometer, TA Instruments) was used at a constant shear rate of 200 s^{-1}. The rheometer was operated with a parallel plate geometry of 40 mm diameter and a gap of 1 mm. An 8% w/v slurry was prepared by suspending 140mg of flour with 1.75 ml of distilled water. The slurry was mixed well using a vortex mixer and allowed to stand for fifteen minutes at room temperature. The geometry was adjusted to zero gap and the slurry was injected between the two plates using a micropipette. A solvent trap was used to reduce the water loss during measurements.

The slurry was heated from 45°C to 95°C in 3 min 45 s, held at 95°C for 2 min 30 s, cooled from 95°C to 50°C in 3 min 45 s and then held at 50°C for 2 min 30 s. As described by Ibanez et al. (2007), heating and cooling the slurry over such a wide range of temperature helped in determining the initial gelatinization temperature (temperature of the initial viscosity increase, °C), peak viscosity (maximum viscosity recorded during heating and cooling cycles, Pa s), hot paste viscosity (minimum viscosity after peak, Pa s), cold viscosity (viscosity of the paste at the end of the test), breakdown viscosity (the difference between the peak and the hot paste viscosity, indicating the breakdown in paste viscosity during the 95°C holding period, Pa s) and setback viscosity (the difference between the final and hot paste viscosity, indicating starch retrogradation during cooling, Pa s).

Note : To ensure uniform distribution of sample in the slurry, the 8% slurry was prepared in 1.75ml of water as this was the exact volume that would go between the two plates.

Chapter 4

Results and Discussion

4. Results and Discussion

4.1 Popping Yield

Popping conditions were optimized with respect to the temperature of the particulate popping medium and the moisture content of the millet. The objective was to find the right combination of temperature and moisture to obtain the maximum popped yield. Temperature and moisture were selected as the two independent factors as both of them play a significant role in governing the yield of popped millet. Moisture content plays an important role in popping as the right amount of moisture is necessary to build up enough pressure inside the grain so that it bursts open. When the moisture content is low, sufficient steam is not generated in the endosperm which is required for complete expansion whereas very high moisture content can lead to cracks in the outer seed coat due to swelling which then prevent pressure build-up. Likewise, the temperature of the particulate medium should be high enough to change the moisture present inside the grain into superheated steam – too low temperatures do not generate sufficient heat inside the grain to convert the moisture into superheated steam and too high temperature can impart a burnt flavor to the grain or, at times, burn the grain.

Preliminary experiments conducted on little millet showed that moisture levels below 14% and above 18% resulted in unpopped or poorly popped millet. It was also observed that at temperatures below 220°C the percentage of popped grains was very low. In the present study, the gas chromatograph oven that was used to heat up the particulate medium limited the highest temperature to 260°C. Moreover, in a study by Malleshi and Desikachar (1981) it has been reported that the yield of puffed ragi (finger millet) decreased at temperatures higher than 270°C, confirming the choice for the range of moisture and temperature tested presented in Table 4.1.

Table 4.1 Design of experiment

Factor	Level
Moisture content (%)	14 16 18
Temperature (°C)	220 240 260

It was observed that both the temperature of the particulate medium and the moisture content of the millet influenced the yield, as the percentage of popped millet varied considerably amongst the different combinations of popped samples.

The highest yield (78.44%) was obtained for the millet that had been tempered to 16% moisture content and popped at 260°C while the lowest yield (30.91%) was observed for the millet tempered to 14% moisture content and popped at 220°C. It is clear from Fig.4.1 that the yield of the popped millet increased with an increase in both, the temperature and the moisture, to a certain extent.

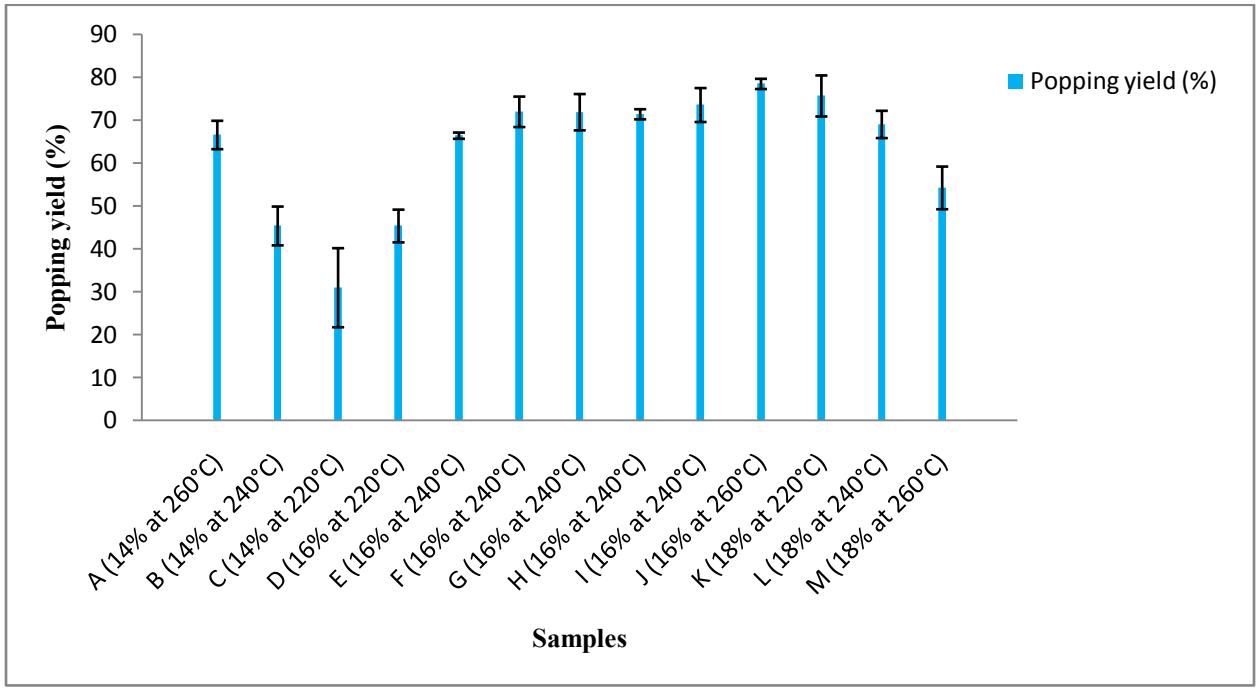

Fig. 4.1 Graphical representation of the yield of popped millet at different combinations of temperature and moisture content.

This was confirmed by statistical analysis (Table 4.2) which showed that moisture, '*A*', ($p<0.0007$) and temperature, '*B*', ($p<0.0001$) significantly influenced the popping yield. The quadratic term for moisture (A^2, $p<0.0048$) had a significant effect while that of temperature (B^2, $p<0.0781$) did not. It was also observed that the interaction between the factors ($A\times B$, $p<0.1147$) did not influence popping significantly.

Table 4.2 ANOVA results presenting the effect of different combinations of temperature and moisture on the yield of popping.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	2420.48	5	484.10	31.24	0.0001
A	523.60	1	523.60	33.79	0.0007
B	1355.92	1	1355.92	87.51	<0.0001
A^2	254.88	1	254.88	16.45	0.0048
B^2	65.88	1	65.88	4.25	0.0781
AB	50.25	1	50.25	3.24	0.1147
Residual	108.46	7	15.49		
Lack of Fit	79.07	3	26.36	3.59	0.1246
R^2	0.9571				

The predicted model for the yield can be described in terms of coded factors by the following equation (4.1):

$$\textbf{\textit{Popping \%}} = 69.79 + 9.34 * A + 15.03 * B - 9.61 * A^2 - 4.88 * B^2 - 3.54 * A \times B \quad (4.1)$$

The response surface graph (Fig. 4.2) for the yield of popping clearly showed that the percentage of popped millet was influenced significantly by the temperature of the medium and the moisture content of the millet and varied from 30.911% to78.441% under the present experimental conditions. It was clear from the graph that popping yield attained a maximum value when the moisture content was between 16% and 17% and started decreasing when the moisture increased to 18%. Also, for the present range, an increase in temperature increased the yield of popping. In a similar study, Delost-Lewis et al. (1992) reported that the puffing yield of proso millet increased significantly, from 7% to 72% for different varieties of proso millet (*Panicum*

miliaceum), as the moisture content was raised from 12% to 18%. In another study conducted on the popping quality of grain amaranth (*Amaranthus caudatus*), Lara and Ruales (2002) used a hot air popcorn popper to pop grain amaranth at three different temperatures namely 200°C, 220°C and 240°C and, at three different moisture levels namely 12%, 14% and 16%. They confirmed that the popping quality of amaranth was governed by grain moisture but in their case the effect of temperature was not significant.

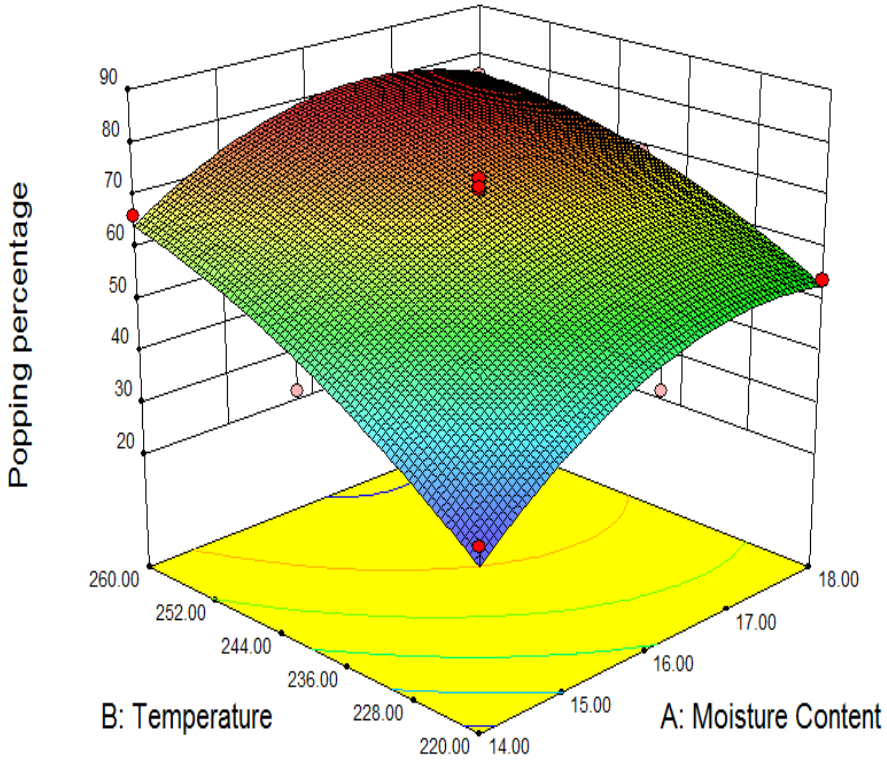

Fig. 4.2 Response Surface Plot presenting the effect of temperature and moisture content on the yield of popping (%).

Hoseney et al. (1983) used an aluminium popcorn popper with oil and popped the grain at five different temperatures namely 182.2°C, 176.7°C, 173.9°C, 171.1°C and 165.6°C. The range of moisture content varied as 9.2%, 9.9%, 11.1%, 14.4% (control), 17.5% and 19.7%. It was observed that the number of popped kernels decreased at temperatures below 176.7°C. Though the effect of moisture was less significant, popping was best observed at moisture level of 13-

17%. In a similar study, Gokmen (2004) studied the popping of corn using different media namely microwave, hot plate, hot air popped and hot plate with salt and oil. Kernels at moisture levels ranging from 8% to 20% were popped. Microwave was used at power level 1200W. The temperature of popping for the conventional methods was not mentioned in the study. It was observed that the highest number of popped kernels was obtained at a moisture content of 14%. It was then established that for popping corn, an optimum moisture content of 13-17% was required. Corn moisture below and above this range resulted in less popped kernels. All these results were in accord with the present study that grain moisture content influences popping and with an increase in moisture level, for the range tested, the number of popped grains increases.

The popping quality of a grain also depends on grain variety and the type of thermal medium used for popping (Gokmen, 2004). The kind of medium used determines the temperature of popping as different mediums have different thermal conductivity. In a study by Malleshi and Desikachar (1981) it was reported that the puffing yield of ragi (*Eleucine coracana*) varied from 64% to 97% among 14 different varieties, where the optimum conditions for puffing were fixed at 19% moisture, 4 hrs of tempering and puffing in sand medium at 270°C. As mentioned earlier, Delost-Lewis et al. (1992) proved that the puffing yield of proso millet varied from 7% to 72% among the different varieties at different levels of moisture. Choudhury et al. (2010) investigated the popping quality of purple and yellow varieties of foxtail millet (*Setaria italica*) and concluded that the best yields that were obtained for the yellow and purple varieties were 30% and 26.3%, respectively at 230°C.

Thus, the results obtained from the present study showed that the optimum conditions of popping for little millet were 16% moisture content and a temperature of 260°C.

4.2 Proximate Analysis

4.2.1 Moisture Content

The moisture content of the millet was measured by AACC Method 44 – 15.02 (1999). Thermal processing is known to decrease the water activity of food samples, thus, protecting them against microbial activity. In the present study it was observed that while the moisture content of the

procured native millet was 10.0%, that of popped millet varied considerably from 1% to 6%. Such low moisture levels can be attributed to the high temperatures at which the millet was popped as during popping the moisture inside the grain escaped as steam. Fig. 4.3 compares the moisture content of the popped millet samples with that of native millet. A similar study by Sailaja (1992) proved that the moisture content of sorghum reduced following popping.

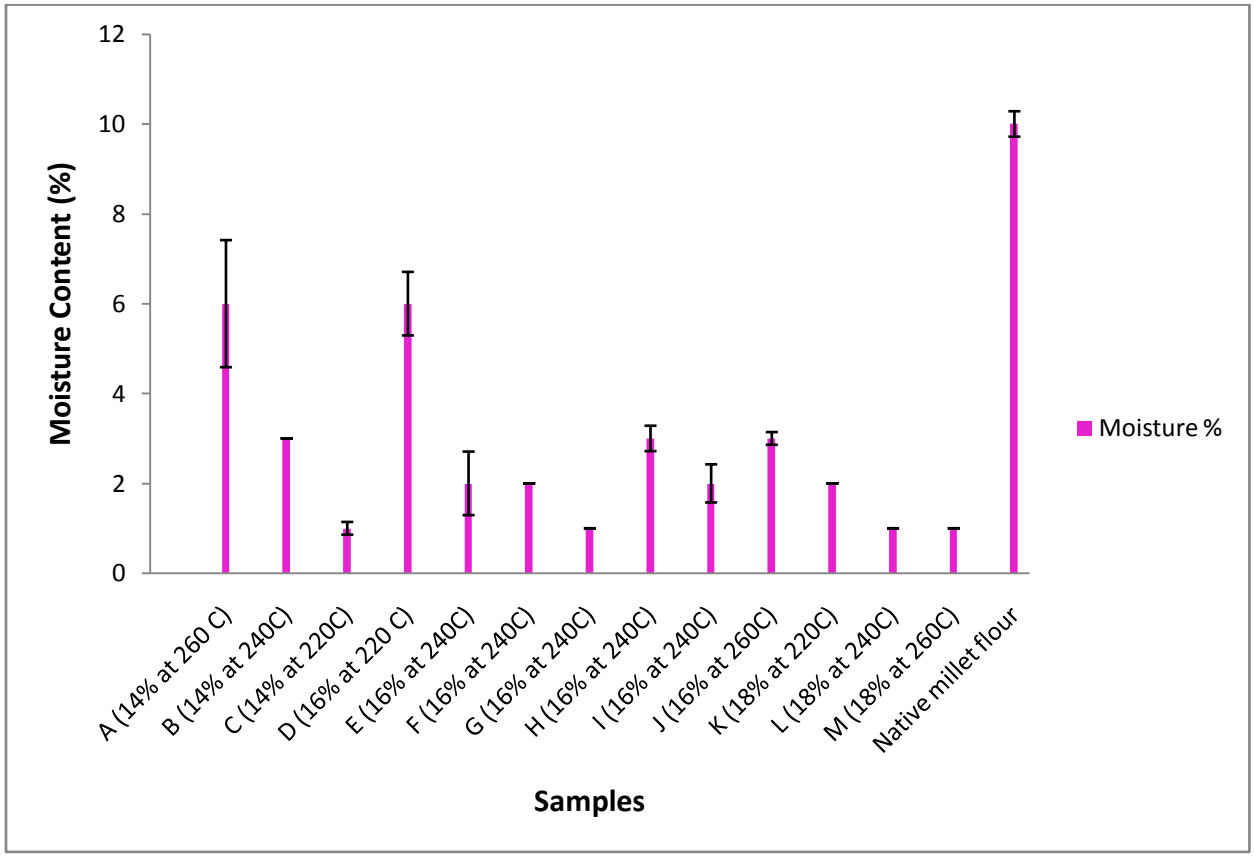

Fig. 4.3 Graphical representation of the comparison of moisture content of the popped millet samples with that of native millet.

4.2.2 Protein

The protein content of popped and native little millet was estimated by the Bicinchoninic Acid Assay. It was observed that the protein content of popped millet varied from 10.02% to 11.41%

and that of native millet was 11.69% (Fig. 4.4). This showed that popping did not have a significant effect on the protein content of the millet thus, establishing popped millet to be as good a source of protein as native millet. Studies conducted by Malleshi and Desikachar (1981) on puffing quality of ragi and by Lara and Ruales (2002) on the popping quality of amaranth grain also confirmed the same.

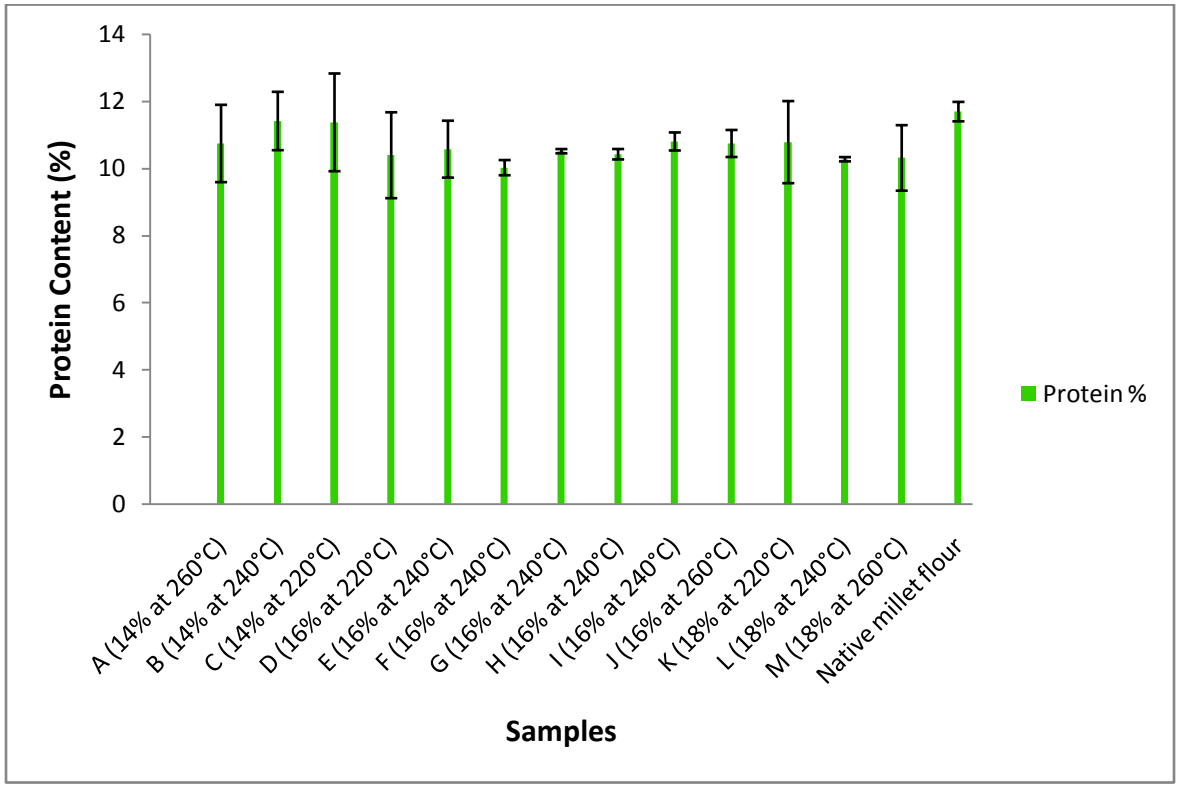

Fig. 4.4 Graphical representation of the comparison of the total protein content of popped millet samples with that of native millet.

The statistical analysis, presented in Table 4.3, showed that moisture content, '*A*' ($p < 0.0093$), was the only factor that significantly influenced the protein content of popped millet unlike temperature, *'B'* ($p< 0.7721$). Also, the interaction between moisture and temperature, ($A \times B$, $p< 0.0663$) did not have a significant effect on the protein content.

Table 4.3 ANOVA results presenting the effect of popping conditions on the Total Protein content.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	1.42	4	0.35	5.29	0.0221
A	0.78	1	0.78	11.61	0.0093
B	6.017E-003	1	6.017E-003	0.090	0.7721
A^2	0.33	1	0.33	4.95	0.0567
AB	0.30	1	0.30	4.52	0.0663
Residual	0.54	8	0.067		
Lack of Fit	0.21	4	0.053	0.65	0.6580
R^2	0.7257				

The predicted model for the protein content can be described in terms of coded factors by the following equation (4.2):

$$\textbf{\% Protein} = 10.49 - 0.36 * A + 0.032 * B + 0.32 * A^2 + 0.28 * A \times B \quad (4.2)$$

The response surface graph for the protein content (%) of popped millet presented in Fig. 4.5 showed that if the effects of factors were considered individually then an increase in moisture decreased the availability of protein while an increase in temperature increased it. Though the statistical analysis showed that the effect of the interaction between moisture and temperature was insignificant ($p< 0.0663$), the graph showed that the effect could not be negated completely. It was clear from the graph that low temperature and low moisture favored the availability of protein whereas high moisture and low temperature resulted in a decrease. Also, at low moisture and high temperature the protein availability decreased slightly below the maximum value. The graph also showed that at 260°C, the protein availability decreased when the moisture varied from 14% to 16.5% followed by an increase at moisture levels above that. The total protein

content was calculated per 100g of sample (db) which included the weight of the popped millet (endosperm) plus the weight of the husk (the husk did not separate completely from some of the grains). As the ratio of husk to endosperm varied in the different samples of the popped millet, a slight difference was observed in the total protein content among the popped samples which could be due to the difference in the weight of endosperm (most of the protein bodies are concentrated in the endosperm) taken for the calculation.

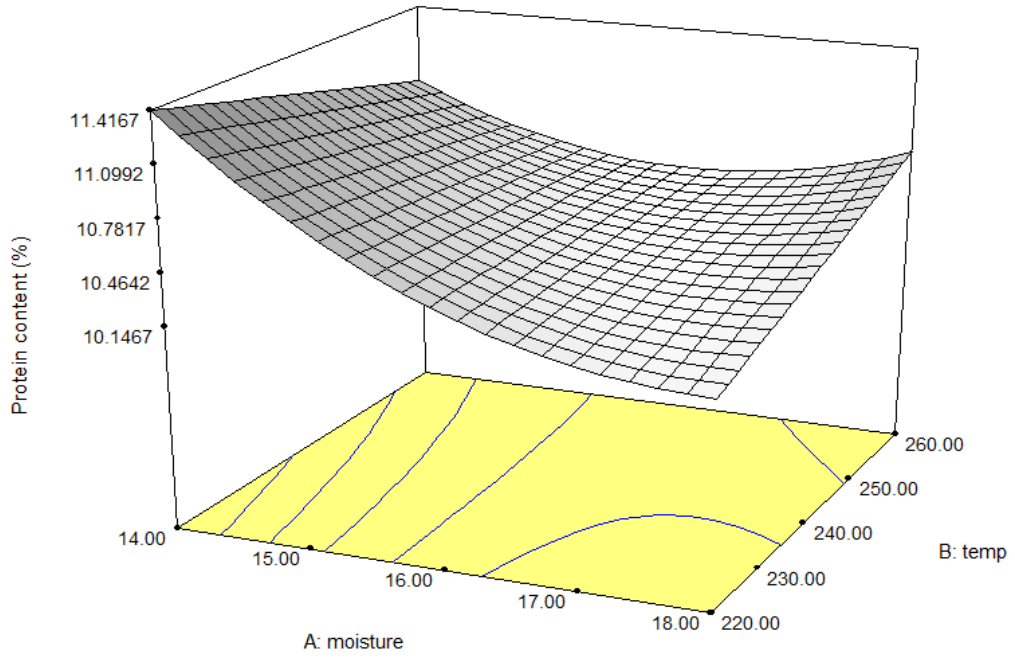

Fig. 4.5 Response Surface Plot showing the effect of temperature and moisture content on the yield of the total protein content (%).

Certain methods of processing like dehulling, flaking and extrusion require the removal of the seed coat which results in the loss of a significant amount of endosperm from the outer walls of the seed. Due to the loss of endosperm, some protein is also lost (Chibber et al., 1978). Singh et al. (2004) showed that popped foxtail millet had higher protein content than dehulled, flaked, roller dried and extruded foxtail millet. Thus, it could be concluded that popped millet stood out as a better source of protein than millet processed by any other method.

Contrary to the results produced in the present study, popped sorghum was reported to have higher protein content than its native form (Sailaja, 1992). A similar study Delost-Lewis et al. (1992) showed that the protein content of puffed millet was higher as compared to its native form. However, Gamel et al. (2004) found out that popping of amaranth grain resulted in a decrease in its protein content.

Thus, the results obtained in the present study showed that popped millet was as good a source of protein as native millet as processing the millet by popping did not negatively affect its protein content.

4.2.3 Resistant Starch (RS)

In a millet kernel, the starch granules present in the peripheral endosperm are buried in the protein matrix and thus, remain protected against enzymatic digestion (FAO, 1995; Hulse et al., 1980). As starch in native form is unavailable for enzymatic digestion, the resistant starch content is high. Following popping, the endosperm expands forming a soap bubble structure. This increases the availability of starch for enzymatic digestion. Fig 4.6 shows the structure of a raw maize endosperm and the alignment of starch granules within the protein matrix.

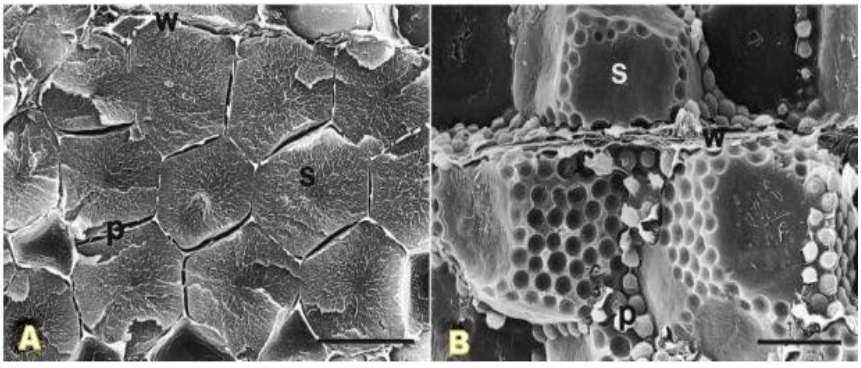

Fig. 4.6 (A) - Fractured polygonal starch granules (s) in the vitreous endosperm of raw maize. 'p' denotes protein bodies that are embedded in thin layers protein matrix between 's'. 'w' denotes the cell wall.

(B) – Starch granules (s) surrounded by spherical protein bodies (p) in raw maize (Parker et al., 1999).

Fig 4.7 shows the structure of the endosperm of popped maize and sorghum. It is clear from the figure that starch granules form bubble like structure on popping, thus, increasing the surface area and the availability of starch for enzymatic digestion.

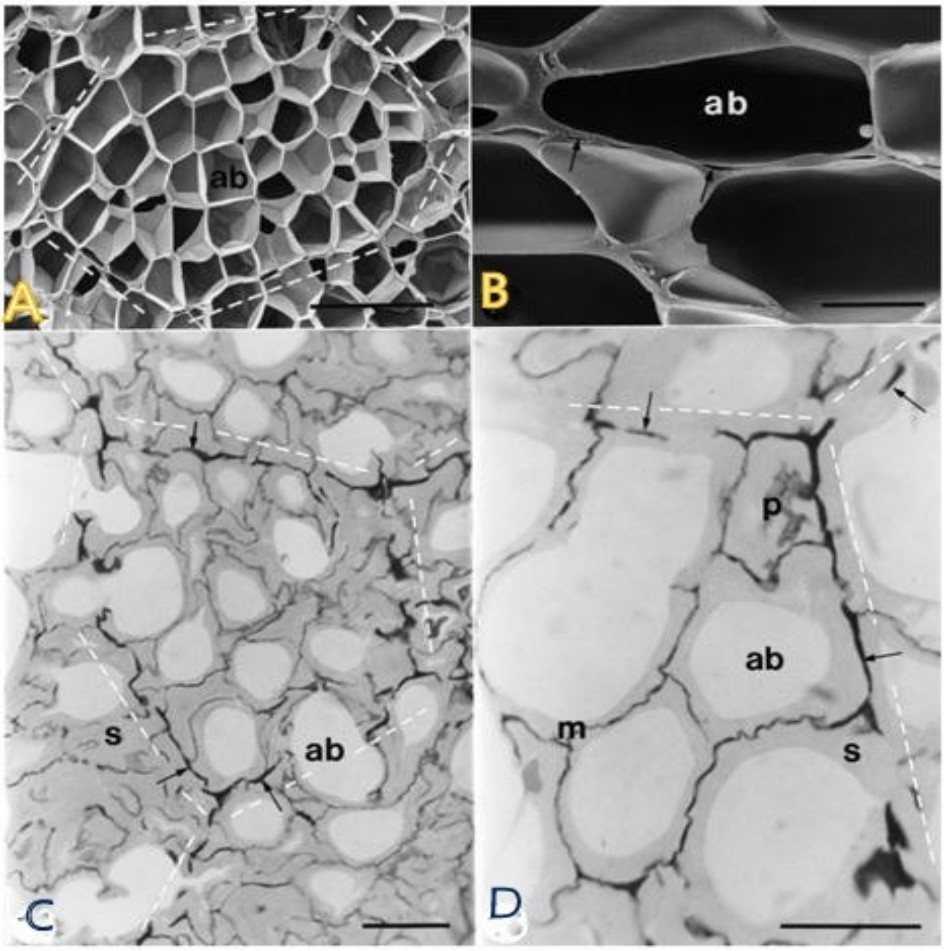

Fig. 4.7 (A) – Cut surface of popcorn showing the starch granules expanded to form a foam-like structure. 'ab' indicates each air bubble formed from one expanded starch granule.

(B) – Starch foam of popped sorghum. Arrows indicate remnants of cell wall in a thin starch film.

(C) –Section of popcorn stained with toluidine blue. Arrows denote the dark staining fragments of cell walls. 'ab' indicates each air bubble in the starch foam (s).

(D) –A higher magnification of the toluidine stained section of popcorn showing the protein bodies (p) and the protein matrix (m) in the starch foam (s). Arrows indicate cell wall fragments and 'ab' indicates air bubbles (Parker et al., 1999).

To determine the resistant starch (RS), non-resistant starch (NRS) and total starch content of the popped millet, the Megazyme Resistant Starch Assay Kit was used. It was observed that while the RS content of native millet was 16.85% that of popped millet varied from 1.82% to 5.57% stating that popping had a significant effect on the RS content of the millet. Similarly, the Non-Resistant Starch content of native millet was observed to be 25.53% while that of popped millet increased considerably, varying from 59. 99 % to 69.30%. The total starch content was calculated as the sum of the RS and the NRS. A decrease in RS followed by simultaneous increase in Non-Resistant Starch (NRS) content meant that RS got converted to NRS, thus, increasing the digestibility of the starch.

Though a large difference was observed between the RS content of native and popped millet, the statistical analysis presented in Table 4.4 showed that neither the temperature of the popping medium nor the moisture content of the millet or the interaction between the two factors influenced the RS content significantly.

Table 4.4 ANOVA results presenting the effect of popping conditions on the Resistant Starch content.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	5.36	2	2.68	2.67	0.1178
A	3.76	1	3.76	3.75	0.0817
B	1.60	1	1.60	1.59	0.2357
Residual	10.04	10	1.00		
Lack of Fit	8.04	6	1.34	2.68	0.1795
R^2	0.3480				

The predicted model for the RS content can be described in terms of coded factors by the following equation (4.3):

Resistant Starch Content (%) *=3.48 – 0.79 * A – 0.53 * B* (4.3)

Though the model did not hold good for either of the factors, a trend could still be observed from the graph (Fig. 4.8) which showed that the RS content decreased with an increase in both moisture and temperature. A decrease in RS content at high temperatures could be attributed to the partial gelatinization that the starch granules underwent during popping.

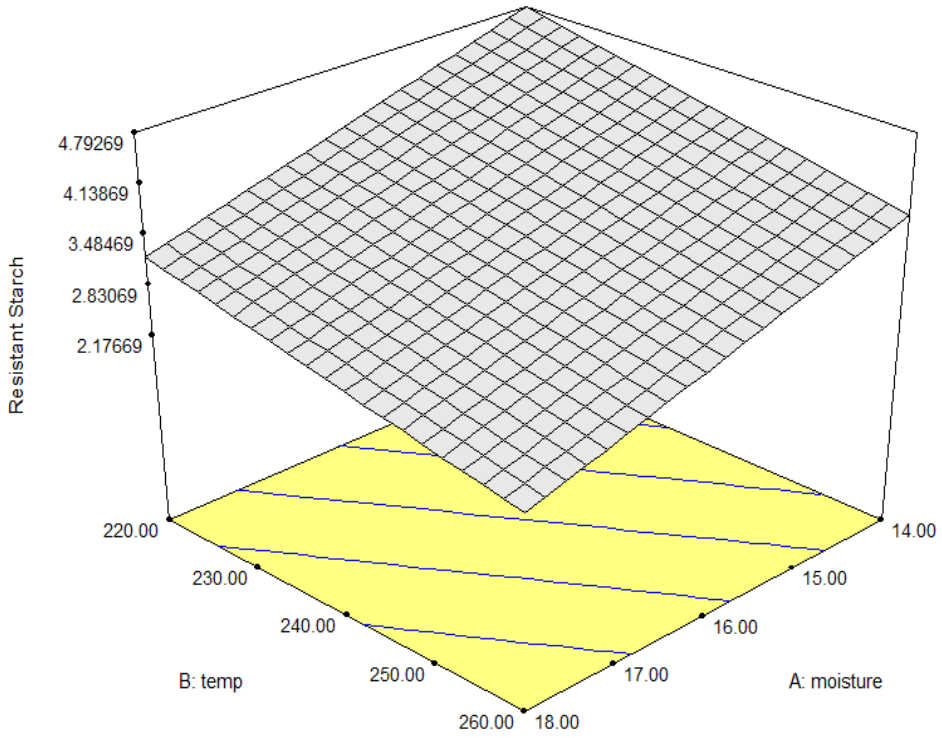

Fig. 4.8 Response Surface Plot presenting the effect of temperature and moisture content on the Resistant Starch content (%).

In a study on two species of grain amaranth, Gamel et al. (2005) indicated that popping significantly affected the resistant starch content of the grains by decreasing it considerably. Parchure and Kulkarni (1997) found out that heat treatments like roasting, extrusion and frying reduced the RS content of amaranth and rice grains. Contrary to this, Lara and Ruales (2002)

reported that popping of amaranth grain did not significantly alter the resistant and non-resistant starch contents.

As for the NRS content, statistical analysis (Table 4.5) showed that temperature, '*B'*, was the only factor that influenced it significantly ($p<0.0226$) while moisture content and the interaction between the factors did not have any effect on the NRS.

Table 4.5 ANOVA results presenting the effect of popping conditions on the Non-Resistant Starch content.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	4240.04	1	40.04	7.03	0.0226
B	40.04	1	40.04	7.03	0.0226
Residual	62.68	11	5.7		
Lack of Fit	51.92	7	7.42	2.76	0.1718
R^2	0.3898				

The predicted model for the NRS content can be described in terms of coded factors by the following equation (4.4):

Non Resistant Starch Content (%) *= 65.23 + 2.58 * B* (4.4)

The response surface plot in Fig. 4.9 showed that the NRS increased with an increase in temperature under the present experimental conditions. This explained that if the only factor taken into consideration was temperature, then the NRS would have increased with an increase

in temperature as a higher temperature would have resulted in fully expanded kernels providing greater accessibility to enzymes for starch degradation.

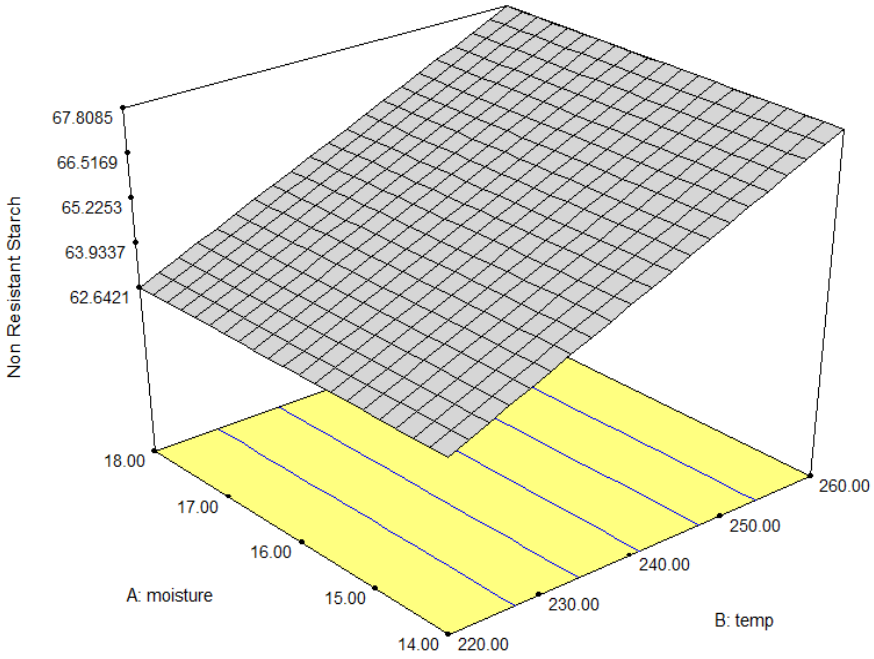

Fig. 4.9 Response Surface Plot presenting the effect of temperature and moisture content on the Non-Resistant Starch content (%).

A study by Saravanabavan et al. (2011) on sorghum showed that popping decreased the RS content from 3.4±0.4 % to 2.0±0.3% in popped sorghum, from 4.3±0.7% to 3.1±0.5% in *Malandi* variety and from 3.9±0.6% to 2.9±0.3% in Red sorghum variety, and caused a simultaneous increase in the NRS content thus, increasing the starch digestibility.

The total starch content of popped little millet, which was calculated as the sum of the RS and NRS, was affected significantly by the moisture content of the millet, '*A*', ($p<0.0500$) and temperature of the popping medium, '*B*', ($p<0.0141$) but not as much by the interaction between the two factors ($A \times B$, $p<0.3568$), as shown in Table 4.6.

Table 4.6 ANOVA results presenting the effect of popping conditions on the Total Starch content.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	42.79	4	10.70	4.07	0.0433
A	13.97	1	13.97	5.32	0.0500
B	25.63	1	25.63	9.76	0.0141
B^2	0.68	1	0.68	0.26	0.6244
AB	2.51	1	2.51	0.96	0.3568
Residual	21.01	8	2.63		
Lack of Fit	17.06	4	4.27	4.33	0.0924
R^2	0.6707				

The predicted model for the Total Starch content can be described in terms of coded factors by the following equation (4.5):

$$\textit{Total Starch Content (\%)} = 68.92 - 1.53 * A + 2.07 * B - 0.46 * B^2 + 0.79 * A \times B \qquad (4.5)$$

It could be interpreted from Fig. 4.10 that the total starch content increased with an increase in temperature and decreased with an increase in moisture. Highest percentage of starch was obtained at the highest temperature and lowest moisture content whereas the lowest total starch was obtained at the lowest temperature and highest moisture content. This made it very clear that the percentage of total starch obtained was directly proportional to temperature and inversely proportional to the moisture content during processing.

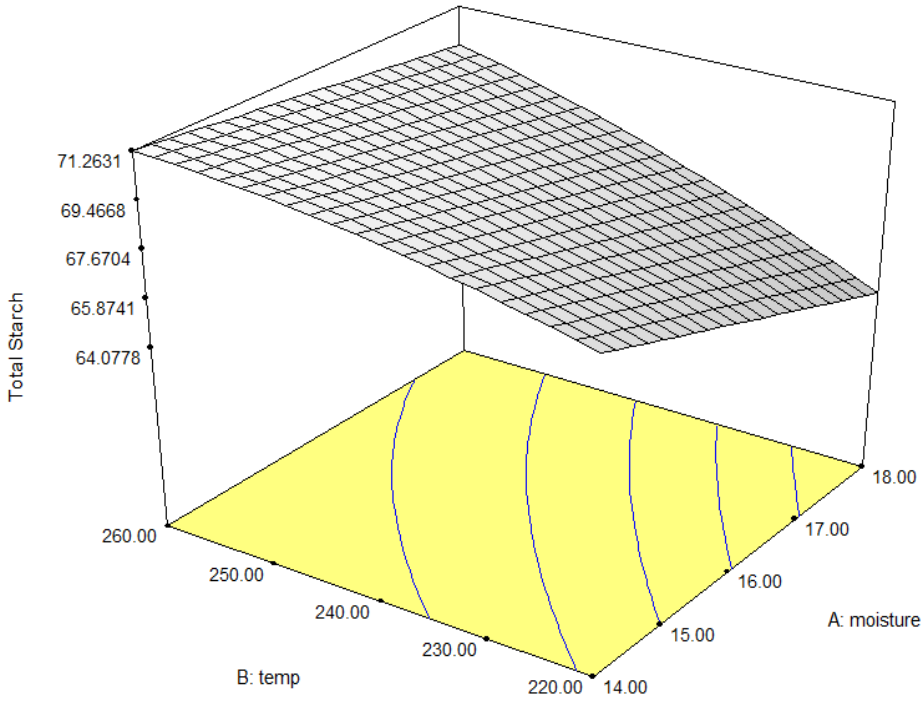

Fig. 4.10 Response Surface Plot presenting the effect of temperature and moisture content on the Total Starch content (%).

An increase in the total starch content could be explained based on its availability. In unprocessed millet kernels, the starch granules remain protected against attack by enzymes due to the envelope created by the protein matrix. While during popping, the endosperm bursts open to form a spongy matrix, at which point, the starch granules become more susceptible to attack by enzymes, thus, increasing the total (available) starch content detected by the enzyme kit. The total starch content of popped little millet was almost similar to that of popped foxtail millet of 68% that was reported by Singh et al. (2004). Saravanabavan et al. (2013) reported a total starch content of 74.3 % to 78.3 % in popped sorghum.

4.2.4 Total Ash

The measure of the total amount of minerals present in a food sample gives its total ash content. Ash can also be defined as the residue formed after the removal of water and organic matter from

the food sample as a result of heating at very high temperatures. In the present study, the ash content was determined by AACC Method 30 - 25.01 (1999) which involved overnight drying of 24 hrs of the sample followed by burning the sample to ash in a muffle furnace at 500°C for 16 hrs.

The ash content of native millet was determined to be 4.508g/100g sample (db) whereas that of popped millet was in the range 3.468- 5.349 g/100g sample (db) (Fig. 4.11). This meant that the ash content of the native millet and the popped millet was almost equal with variations higher and lower likely affected by the presence or absence of the bran in the popped millet. Native millet was ground to flour with the hull intact (all bran), while the popped millet that was ground to flour also only had remnants of pericarp attached to it (partial bran).

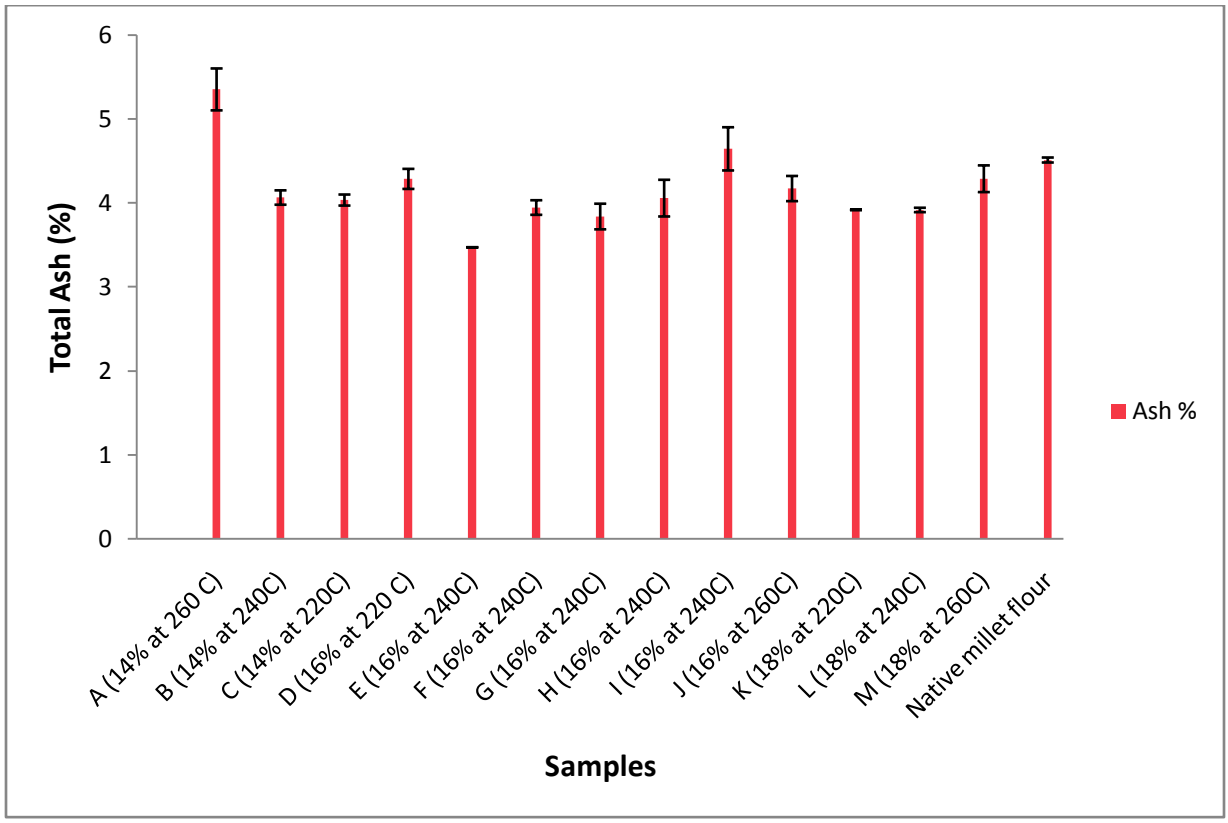

Fig. 4.11 Graphical representation of the comparison of total ash content of the popped millet samples with that of native millet.

Statistical analysis presented in Table 4.7 showed that the model was insignificant ($p<0.4031$) for either of the two factors, i.e., moisture content and the temperature of the popping medium. This meant that the factors that were considered for the present study could not explain the variation.

Table 4.7 ANOVA results presenting the effect of popping conditions on the Total Ash content.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	1.54	5	0.31	2.32	0.1513
A	0.29	1	0.29	2.22	0.1800
B	0.12	1	0.12	0.87	0.3813
A^2	0.018	1	0.018	0.14	0.7214
B^2	0.28	1	0.28	2.13	0.1876
AB	0.71	1	0.71	5.38	0.0535
Residual	0.93	7	0.13		
Lack of Fit	0.20	3	0.067	0.37	0.7809
R^2	0.6238				

Seed coat forms a major portion of the ash content. Removal of seed coat leads to a decrease in the ash content of the sample. Serna-Saldivar et al. (1994) showed that the ash content of whole sorghum was 1.76 % while that of decorticated sorghum was 1.36% and the ash content of whole pearl millet was 1.77% while that of decorticated pearl millet was 1.44%. A study by Singh et al. (2004) on foxtail millet showed that the ash content of decorticated millet (1.7%) was less than that of popped millet (2.9%). The higher ash content of popped millet could be attributed to the partial presence of bran remaining in it. The ash content also varies with the different processing methods; some methods like cooking by boiling may lead to a greater reduction in the ash

content due the diffusion of certain components in water, while other methods like popping, pressure cooking and microwaving may not affect it to the same extent (Saleh and Tarek, 2006). Dharmraj and Malleshi (2011) studied the hydrothermal processing of finger millet and reported that the ash content of native finger millet (2.00%) varied considerably from that of hydrothermally treated (1.60%) and decorticated finger millet (1.00%).

This showed that the results obtained in the present study were in agreement with the results obtained in reported similar studies.

4.2.5 Crude Fat

The crude fat content gives a measure of the total lipids present in the food sample. In this study, the crude fat was determined by AACC method 30 - 25.01 (1999), using petroleum ether as the extraction solvent. It was observed that the fat content of popped millet was almost similar to that of native millet with certain combinations showing a slightly higher percentage of fat. In a millet kernel, the lipids are concentrated in the endosperm and the germ. As mentioned earlier, the endosperm bursts open during popping, thus, making the bound fat globules more easily accessible to the solvent. Fig. 4.12 shows the comparison of the fat content of native millet with popped millet. Native millet flour had a fat content of 5.5% while that of popped millet flour ranged between 5.5% - 6.3%. The results obtained here were in accordance with other studies conducted on cereals in that there was very little difference in the fat content of the treated and untreated samples.

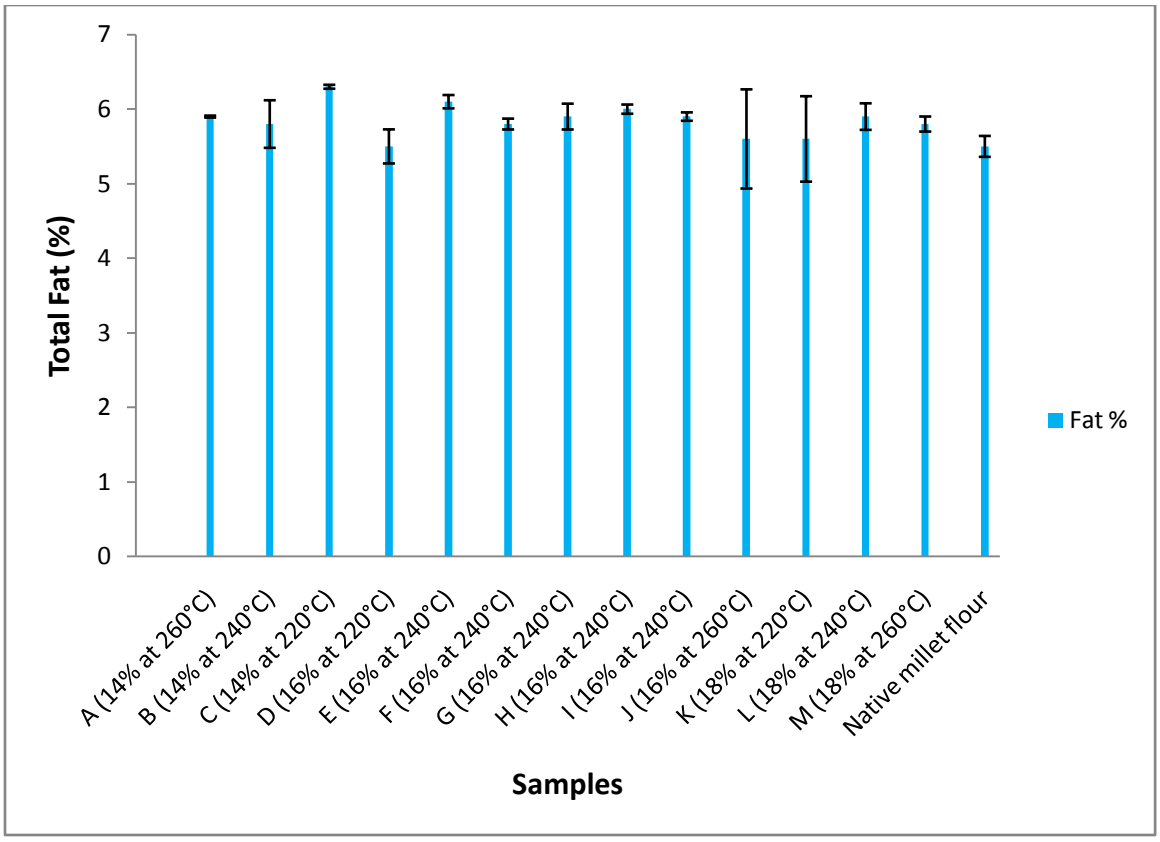

Fig. 4.12 Graphical representation of the comparison of total fat content of the popped millet samples with that of native millet.

Statistical analysis presented in Table 4.8 showed that the model was insignificant in terms of moisture and temperature with $p < 0.3490$ stating that the intensity of the popping process did not have a significant effect on the fat content of little millet.

Table 4.8 ANOVA results showing the effect of popping conditions on the Crude Fat content.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	0.27	5	0.054	1.34	0.3490
A	0.082	1	0.082	2.03	0.1976
B	1.667E-003	1	1.667E-003	0.041	0.8447
A^2	0.041	1	0.041	1.03	0.3447
B^2	0.087	1	0.087	2.16	0.1850
AB	0.090	1	0.090	2.23	0.1788
Residual	0.28	7	0.040		
Lack of Fit	0.23	3	0.077	5.90	0.0596
R^2	0.4891				

As the popped millet was analyzed for total fat content along with the seed coat, there was very little difference observed in the fat content of the native millet and the popped millet. Dharmaraj and Malleshi (2011) conducted a study on the hydrothermal processing of finger millet and found out that the total fat content of finger millet decreased from 1.5% in the native form to 0.77% in the decorticated form. This decrease in the fat content was attributed to the loss of seed coat which is known to contain about 30-40% of the nutrients in finger millet. In another study on the functional properties of foxtail millet, Singh et al. (2004) also showed that the fat content of popped millet was higher than that of decorticated millet. They also showed that other methods of thermal processing like extrusion and roller drying resulted in a decrease in the total fat content of foxtail millet.

4.3 Functional Properties

4.3.1 Total Phenolic Content

Polyphenols are heat sensitive compounds that come under the category of naturally occurring antioxidants. Consumption of food components rich or fortified with polyphenols has been reported to prevent cardiovascular diseases. Polyphenols also impart anti-carcinogenic, antiviral, anti-glycemic and antioxidative properties to food.

The Total Phenolic Content (TPC) was determined by the Folin-Ciocalteau's Assay using gallic acid as the standard and methanol as the extraction solvent, and was expressed as mg gallic acid equivalent (GAE)/ 100g sample, dry basis. It was observed that there was a significant increase in the total phenolic availability of little millet after popping. The total phenolic availability of native millet was 225 mg GAE/100g sample (db) while that of popped millet ranged from 346.996 – 661.462 mg GAE/100g sample (db) (Fig. 4.13).

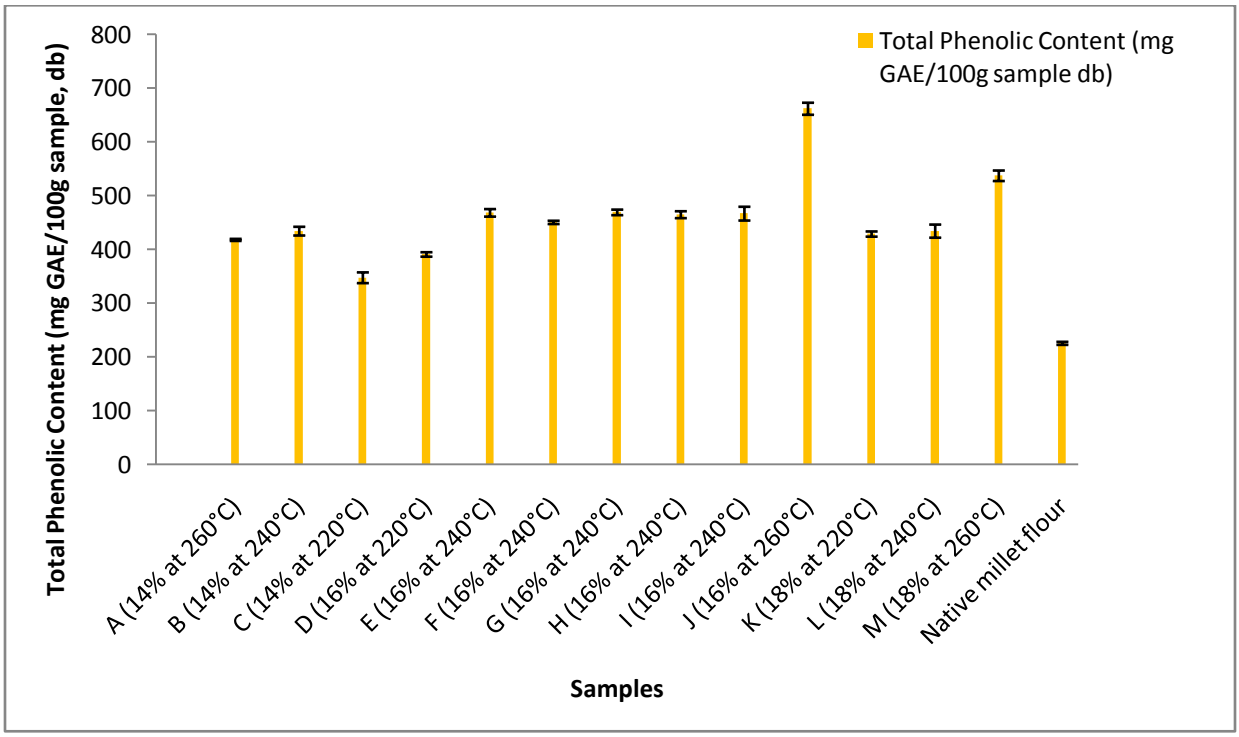

Fig. 4.13 Graphical representation of the comparison of the total phenolic content of popped millet samples with that of native millet.

An increase in the total phenolic availability could be explained on the basis that polyphenols in a millet kernel are concentrated in the seed coat (Chethan and Malleshi, 2007). Popping caused the endosperm to burst open, thus, separating the seed coat. The seed coat bound phenolics thus became easily accessible to the solvent during the analytical extraction process. Moreover, popping was performed at high temperatures namely 220°C, 240°C and 260°C. High temperatures could have weakened the phenol-polysaccharide and phenol-protein linkages and could have softened the tissues, thus leading to an easier migration of the phenolics into the extraction solvent (Chethan and Malleshi, 2007).

Statistical analysis presented in Table 4.9 showed that temperature of the popping medium and moisture content of the millet influenced the total phenolic availability significantly ($p<0.0460$) but the 'lack of fit' was also significant ($p<0.0003$). To make the 'lack of fit' insignificant, the outliers (14% at 260°C , 16% at 220°C and 16% at 240°C (sample H)) were removed. The new analysis, with the outliers removed, showed that moisture, 'A', ($p<0.2572$) did not affect the TPC significantly whereas temperature, 'B', ($p<0.0002$), its quadratic term, '$B^{2'}$', ($p<0.0022$) and the interaction between temperature and moisture, 'AB', ($p<0.0063$) had a significant effect. It was also observed that the quadratic term for moisture, A^2, ($p<0.0305$) influenced the TPC significantly.

Table 4.9 ANOVA results showing the effect of popping conditions on the Total Phenolic content.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	602224.38	5	12044.88	60.07	0.0007
A	349.48	1	349.48	1.74	0.2572
B	30690.41	1	30690.41	153.06	0.0002
A^2	2156.83	1	2156.83	10.76	0.0305
B^2	9730.07	1	9730.07	48.53	0.0022
AB	5540.05	1	5540.05	27.63	0.0063
Residual	802.05	4	200.51		
Lack of Fit	567.91	1	567.91	7.28	0.0739
R^2	0.9869				

The predicted model for the Total Phenolic Content can be described in terms of coded factors by the following equation (4.6):

$$\textit{Total Phenolic Content (mg GAE/100g sample, db)} = 465.33 - 10.49 * A + 113.24 * B - 36.83 * A^2 + 72.40 * B^2 - 56.40 * A \times B \quad (4.6)$$

The response surface plot presented in Fig. 4.14 showed that the TPC increased with an increase in temperature. The TPC content increased as the moisture content increased from 14% to 16.5% and decreased with a further increase in moisture. The interaction between the two factors also affected the total phenolic availability. It can be interpreted from the graph that a combination of low temperature and low moisture favored the total phenolic availability whereas that of high temperature and high moisture did not. A low value of TPC was obtained at high temperature and high moisture combination.

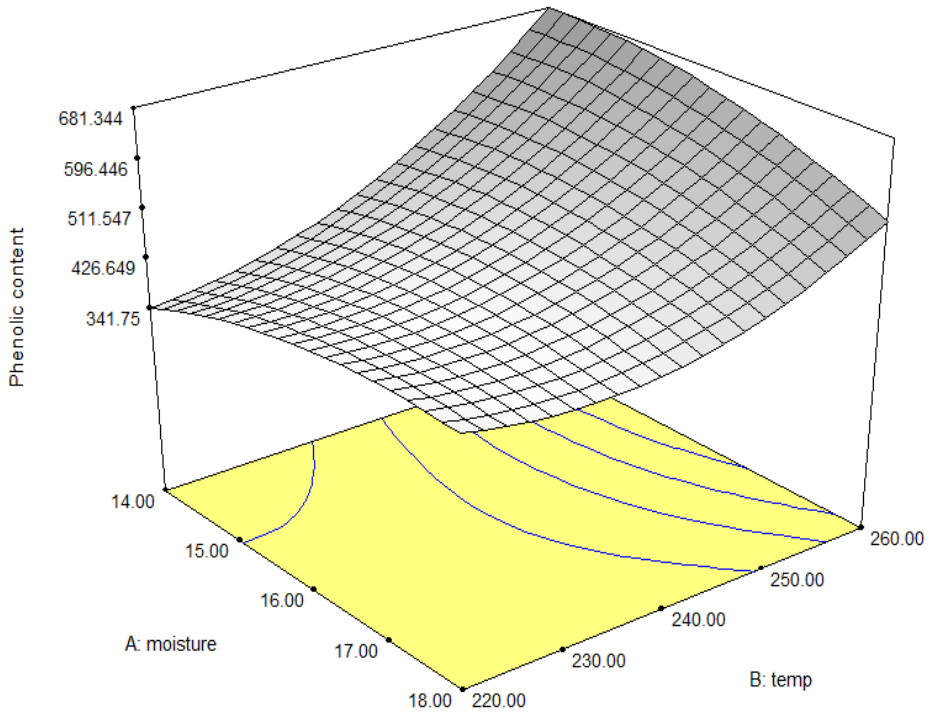

Fig. 4.14 Response Surface Plot presenting the effect of temperature and moisture content on the Total Phenolic Content (mg GAE/100g sample, db).

In a reported study on little millet, Pradeep and Guha (2007) reported that the TPC of little millet increased significantly from 429.9 mg GAE/100g sample (db) to 453.3–521.0 mg GAE/100g sample (db) after processing(germination, steaming and roasting). Gallegos-Infante et al. (2010) found out that thermal processing of barley grains by roasting and cooking increased the TPC. In a study on dry beans, Boateng et al. (2008) reported that toasting significantly increased the TPC from 6.121 mg GAE/g sample (db) to 6.737 mg GAE/g sample (db). All these studies showed that the TPC is increased by processing which supported the results obtained in the present study.

4.3.2 Swelling Power and Solubility

The swelling power (SP) test measures the uptake of water by flour or starch samples during the process of gelatinization of starch. Starch granules swell as a result of the interaction among the starch chains in the crystalline and amorphous domains, the degree of this interaction being governed by amylose to amylopectin ratio, the degree and length of branching and confirmation (Hoover, 2001;Ratnayake et al. 2002; Nemtanu and Brasoveanu, 2010). Swelling power gives an indication of the extent of associative forces between the starch granules (Moorthy and Ramanujan, 1986). The two factors that affect the changes in starch granules in an aqueous medium are temperature and water availability from the system. When heated in excess of water the starch molecules lose their crystalline structure. The exposed hydroxyl groups in amylose and amylopectin get linked to the water molecules by hydrogen bonding which leads to an increase in granule swelling and solubility (Singh et al., 2003; Nemtanu and Brasoveanu, 2010).

The SP test requires a very little quantity of sample, is easy to perform and can be carried on a large number of samples at the same time. Determination of swelling power and solubility of flour and starches finds importance commercially, especially in food industries where quality based products depend on the swelling ability of starch granules like in making of noodles, pasta and the formation of dough for bread making.

Native millet flour and popped millet flour were analyzed for their swelling power and solubility. Both swelling power and solubility were determined at three different temperatures, namely 30°C, 50°C and 100°C. Both, SP and solubility increased with an increase in temperature. An increase in the SP of native millet flour could be attributed to the relaxation of the crystalline structure due to which amylose and amylopectin easily form hydrogen bonds with water molecules (Nemtanu and Brasoveanu, 2010). It was observed that the SP of the millet increased considerably after popping. A higher SP of popped millet flour could be attributed to the high porosity of the spongy matrix formed due to partial gelatinization of the starch during popping (Dharmaraj et al., 2012).

The millet flour, whether in native form or in popped form, exhibited a strange behavior as presented in Fig 4.15 – the SP of the flour decreased when measured at 50°C and increased again at 100°C. This unusual behavior was also reported by Li and Yeh (2001) who worked on 10

kinds of starches from cereals, tubers, roots and peas, and found out that SP increased with an increase in temperature except for potato, waxy corn and tapioca starch where a drop in SP was observed at temperatures higher than 80°C. Another study that showed a similar trend in SP was conducted by Singh et al. (2000) on different varieties of rice flour and starch. They observed that the SP of Japonica rice flour increased slowly to reach its maximum value at 70°C, decreased till 80°C and again increased between 80°C to 90°C.

It was observed in Fig. 4.15 and 4.16 that the swelling power decreased when the temperature increased from 30°C to 50°C followed by an increase when the temperature was raised from 50°C to 100°C for both the native millet flour and the popped millet flour. The SP of native millet decreased from 2.42 at 30°C to 2.36 at 50°C and again increased to 6.79 at 100°C. Contrary to this, the popped millet flour exhibited a SP that was thrice that of native millet flour at 30°C but did not vary much with a further increase in temperature. This could be explained on the basis of the porosity of the popped millet flour. As the starch matrix of the popped millet flour was already porous, there was no room for it to take up more water than what it absorbed. It was also noted that SP followed the same pattern for all the samples except samples F (16%, 240°C), K (18%, 220°C) and M (18%, 260°C) for which the SP did not decrease at 50°C.

Figs. 4.15 and 4.16 show the pattern of swelling power of the popped millet flour (PMF) samples in comparison with native millet flour (NMF).

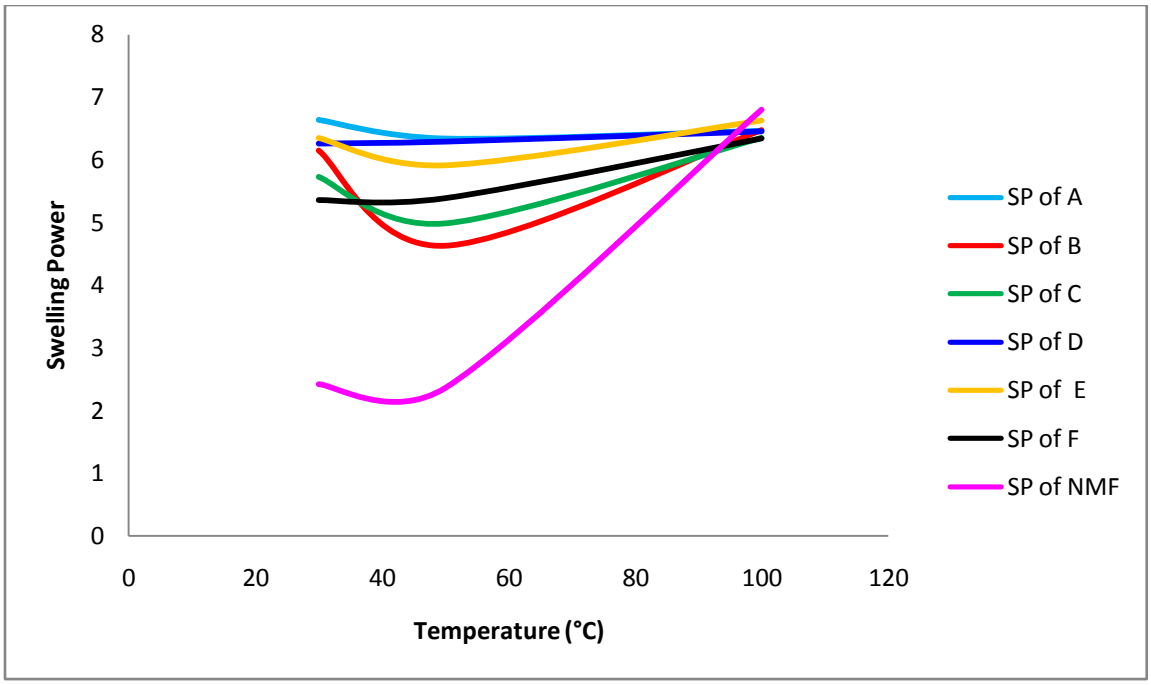

Fig. 4.15

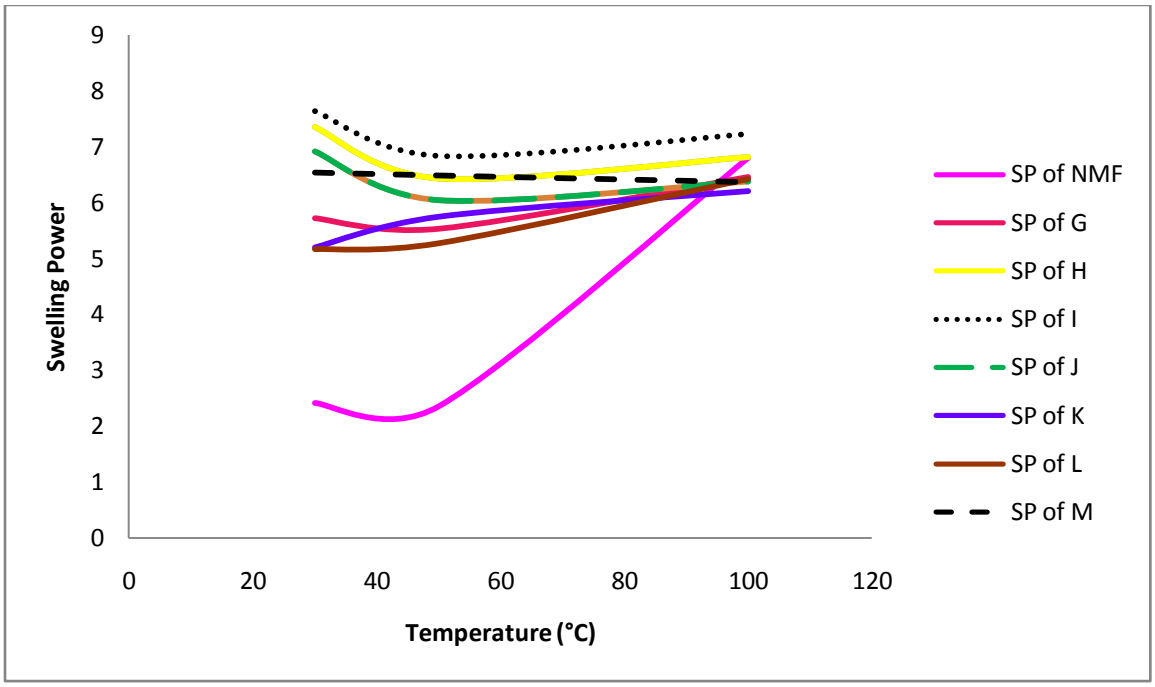

Fig. 4.16

Fig. 4.15-4.16 Swelling Power of popped millet flours in comparison with native millet flour.

SP and solubility go hand in hand in the understanding of the flour behavior. When starch granules swell due to uptake of water, the starch solubility increases. In the present study, the solubility of native and popped millet flour increased with an increase in temperature, the highest solubility being observed at 100°C. This suggested that at higher temperatures it was easier for water to penetrate into the starch granules (Li and Yeh, 2001).

In the present study it was observed that while the solubility of native millet flour increased from 3.55% at 30°C to 6.01% at 50°C and reached a value of 11.01% at 100°C, that of popped millet flour was almost thrice of native millet flour at 30°C and varied amongst millet popped under different levels of moisture and temperature. The popped millet flour samples and the native millet flour exhibited almost similar solubility at 100°C.

The popped millet flour samples did not follow the same pattern however for solubility. While the solubility of certain samples (A, E, H, J, K, L and M) increased with an increase in temperature, that of samples C, D, G and I followed a pattern similar to that of SP, i.e., decreasing slightly at 50°C and increasing again at 100°C. The solubility of sample D decreased with an increase in temperature. Sample B (14% moisture content, 240°C) followed a very peculiar trend. The solubility for this sample reached a maximum value of 20.64% at 50°C and then decreased to 9.92% at 100°C, and this behavior remains unexplained.

Figs. 4.17 and 4.18 show the solubility pattern of the popped millet flour samples in comparison with native millet flour.

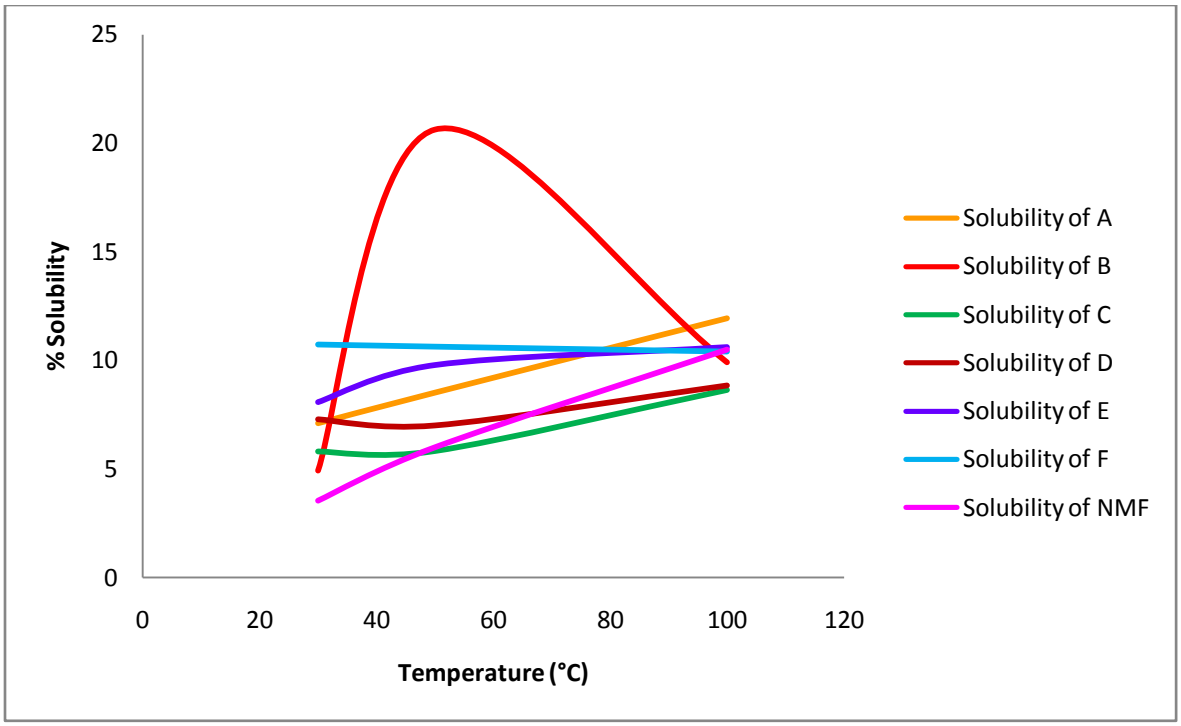

Fig. 4.17

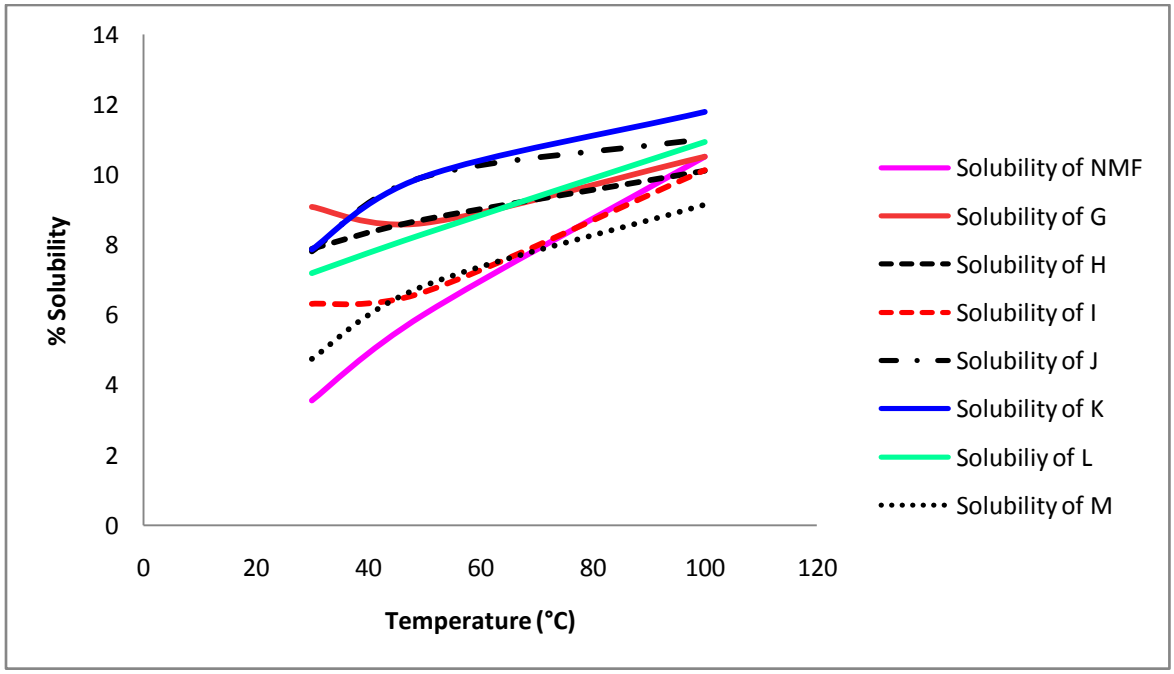

Fig. 4.18

Fig. 4.17 – 4.18 % Solubility of popped millet flours in comparison with native millet flour.

Statistical analysis (Tables 4.10 - 4.12) showed that the model was insignificant in terms of both, moisture and temperature for SP at 30°C, 50°C and 100°C. As far as solubility is considered, it was observed that the statistical model was insignificant in terms of both temperature and moisture for solubility at 30°C and 50°C (Tables 4.13 – 4.14).

Table 4.10 ANOVA results presenting the effect of popping conditions on SP of popped millet flour in water at 30°C.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	3.05	5	0.61	0.97	0.4935
A	0.43	1	0.43	0.69	0.4346
B	7.848E-003	1	7.848E-003	0.013	0.9140
A^2	1.35	1	1.35	2.15	0.1856
B^2	0.15	1	0.15	0.24	0.6414
AB	1.26	1	1.26	2.01	0.1992
Residual	4.39	7	0.63		
Lack of Fit	0.44	3	0.15	0.15	0.9245
R^2	0.4103				

Table 4.11 ANOVA results presenting the effect of popping conditions on SP of popped millet flour in water at 50°C.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	2.60	5	0.52	1.48	0.3080
A	0.40	1	0.40	1.14	0.3209
B	0.56	1	0.56	1.60	0.2462
A^2	1.31	1	1.31	3.71	0.0955
B^2	0.78	1	0.78	2.22	0.1803
AB	0.094	1	0.094	0.27	0.6221
Residual	2.47	7	0.35		
Lack of Fit	1.00	3	0.33	0.91	0.5131
R^2	0.5133				

Table 4.12 ANOVA results presenting the effect of popping conditions on SP of popped millet flour in water at 100°C.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	0.30	5	0.060	0.82	0.5718
A	0.014	1	0.014	0.20	0.6715
B	6.337E-003	1	6.337E-003	0.086	0.7777
A^2	0.071	1	0.071	0.97	0.3577
B^2	0.10	1	0.10	1.40	0.2752
AB	6.250E-004	1	6.250E-004	8.494E-003	0.9291
Residual	0.52	7	0.074		
Lack of Fit	0.037	3	0.012	0.10	0.9534
R^2	0.3695				

Table 4.13 ANOVA results presenting the effect of popping conditions on solubility of popped millet flour in water at 30°C.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	17.41	5	3.48	1.68	0.2570
A	0.63	1	0.63	0.30	0.5987
B	4.06	1	4.06	1.96	0.2045
A^2	8.78	1	8.78	4.23	0.0786
B^2	0.25	1	0.25	0.12	0.7380
AB	0.84	1	0.84	0.40	0.5456
Residual	14.52	7	2.07		
Lack of Fit	3.85	3	1.28	0.48	0.7129
R^2	0.5454				

Table 4.14 ANOVA results presenting the effect of popping conditions on solubility of popped millet flour in water at 50°C.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	0.30	5	0.060	0.82	0.5718
A	0.014	1	0.014	0.20	0.6715
B	6.337E-003	1	6.337E-003	0.086	0.7777
A^2	0.071	1	0.071	0.97	0.3577
B^2	0.10	1	0.10	1.40	0.2752
AB	6.250E-004	1	6.250E-004	8.494E-003	0.9291
Residual	0.52	7	0.074		
Lack of Fit	0.037	3	0.012	0.10	0.9534
R^2	0.3695				

It was observed that the model was significant for solubility at 100°C but the 'lack of fit' was also significant. To make the 'lack of fit' insignificant, an outlier (Sample D – 16% at 220°C) was removed. The new analysis (Table 4.15), with the outlier removed, showed that the effect of moisture content, 'A', was barely significant ($p< 0.0512$) while the quadratic term for moisture ('A^2', $p< 0.4271$), temperature ('B', $p< 0.4466$) and the quadratic term for temperature ('B^2', $p< 0.6617$) did not have any significant effect on the solubility of flour at 100°C. The interaction between the two factors, 'AB', was the only factor that influenced the solubility at 100°C significantly with $p< 0.0002$.

Table 4.15 ANOVA results presenting the effect of popping conditions on solubility of popped millet flour in water at 100°C (Sample D removed).

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	8.06	5	1.61	15.8	0.0021
A	0.60	1	0.60	5.90	0.0512
B	0.068	1	0.068	0.66	0.4466
A^2	0.074	1	0.074	0.73	0.4271
B^2	0.022	1	0.022	0.21	0.6617
AB	7.27	1	7.27	71.24	0.0002
Residual	0.61	6	0.10		
Lack of Fit	0.41	2	0.21	4.14	0.1060
R^2	0.9294				

The predicted model for solubility of popped millet flour in water at 100°C can be described in terms of coded factors by the following equation (4.7):

$$\textit{\% Solubility (100°C)} = 10.42 + 0.32 * A + 0.12 * B - 0.19 * A^2 + 0.10 * B^2 - 1.35 * A \times B \quad (4.7)$$

Fig. 4.19 presents the response surface plot for the effect of temperature and moisture content on solubility of popped millet flour in water at 100°C. It is clear from the graph that value of solubility was lowest at 14% and 220°C and was highest at 18% and 220°C. Also, the solubility of the popped millet flour increased with an increase in temperature at 14% moisture content and decreased with an increase in temperature at 18% moisture content.

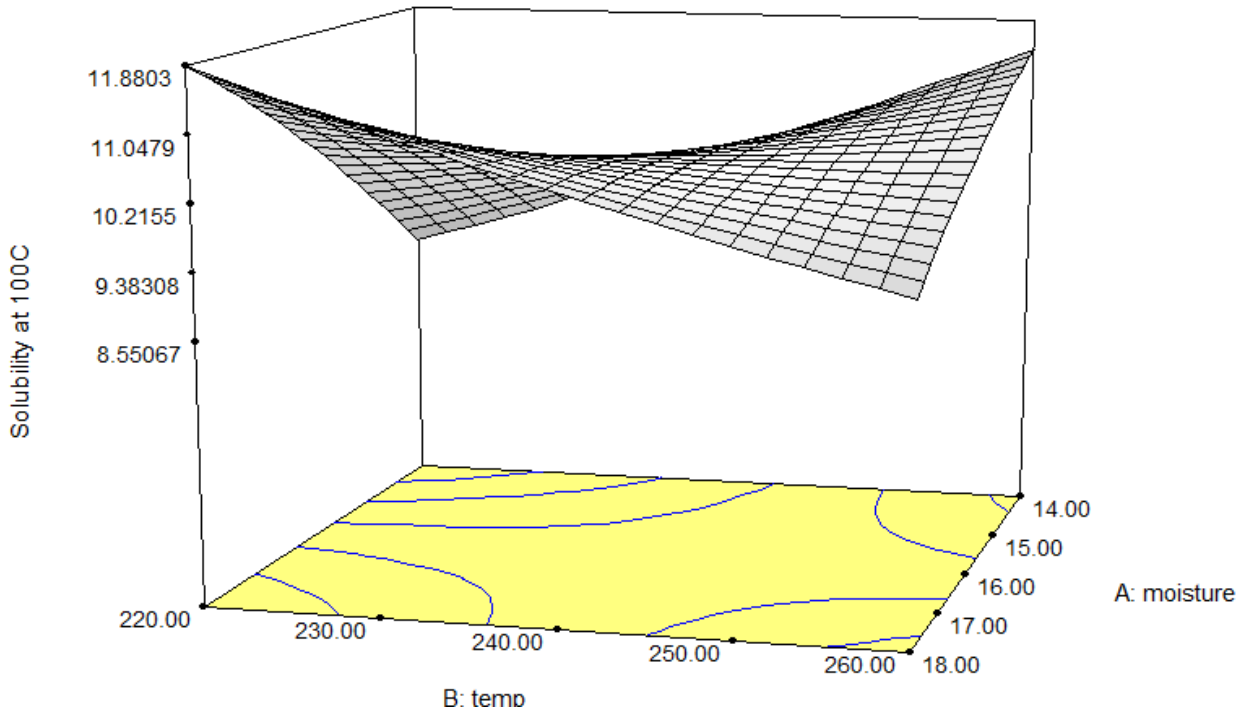

Fig. 4.19 Response Surface Plot presenting the effect of temperature and moisture content on solubility of popped millet flour in water a 100°C.

An increase in SP and solubility of flours and starches had been reported in several other studies. Ikegwu et al. (2010) conducted studies on *Brachystegia eurycoma* flour and starch and showed that both swelling power and solubility were temperature dependent and increased with an increase in temperature. They also observed that the SP of *Brachystegia eurycoma* starch (10.05%) was higher than that of its flour (5.95%). This was due to the presence of lipids and proteins in the flour which formed inclusion complexes with amylose and restricted the swelling.

An increase in SP as a result of popping of grain amaranth has also been reported by Gamel et al. (2005) where the SP of amaranth increased from 107.4% in the native form to 117.0% in the popped form.

In a study on millet starches, Muralikrishna et al. (1986) found out that the SP and solubility of popped pearl millet, finger millet and foxtail was higher than their native counterparts. Murugesan and Bhattacharya (1989) also showed that the SP and solubility of popped rice flour increased with an increase in temperature. Holm et al. (1988) studied the properties of starch in processed wheat and reported out that both, SP and solubility of popped wheat flour were higher than that of the native wheat flour - the SP increased from 3.1 in the native wheat to 7.5 - 10 in the popped wheat whereas the solubility depicted an increase from 1.5% in the native wheat to 3.5 - 40.3% in the popped wheat.

4.3.3 Oil Absorption

Oil absorption capacity gives a measure of the oil absorbed by the sample (here as flour) and is expressed as a percentage. It is important to know the oil absorption capacity of flour as it gives an indication of the amount of oil that the flour takes up during food processes like frying.

In the present study, the oil absorption capacity (OAC) was measured according to the method of Lin and Humbert (1974) and was determined at room temperature (30°C) and at 140°C. It was observed that the oil absorption capacity of popped millet flour was higher than that of native millet flour, both at room temperature and at 140°C, and that the OAC of the flour was higher at room temperature (Fig. 4.20). Higher OAC of popped millet flour could again be attributed to its porous nature which allowed oil to percolate through it more easily unlike for the native millet flour. As far as temperature is considered, higher oil absorption at 30°C could be due to a longer duration of contact (30 minutes of standing period and 25 minutes of centrifugation) between oil and the flour unlike at 140°C where the sample was brought into contact with oil only for 15±3 seconds. This was done to simulate frying conditions where the duration of contact is very small. Lower OAC of native millet flour might be due to the unavailability of the lipophilic proteins from the structure which are responsible for binding lipids. Popping might have caused some

changes in the protein confirmation resulting in the exposure of certain non-polar residues leading to higher binding of lipids (Narayana and Rao, 1982).

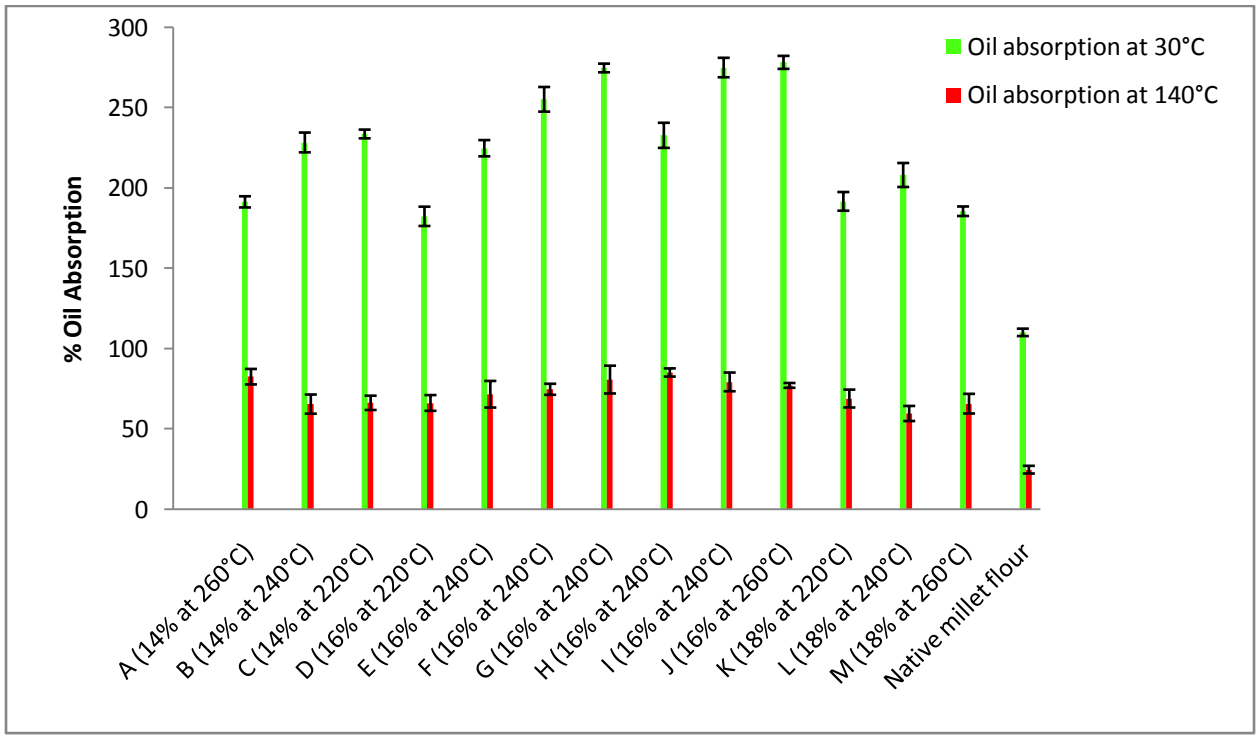

Fig. 4.20 Comparison of OAC of popped millet flour with native millet flour at 30°C and 140°C.

Statistical analysis (Tables 4.16 and 4.17) showed that the model was insignificant for either of the two factors i.e., temperature and moisture with $p<0.2409$ for oil absorption at 30°C and $p<0.1267$ for oil absorption at 140°C, which meant that the OAC was neither affected by the temperature at which the millet was popped nor by the moisture content of the millet. This phenomenon was similar to that of swelling power, which depicted the amount of water absorbed and was not affected by the temperature or the moisture content.

Table 4.16 ANOVA results presenting the effect of popping conditions on oil absorption capacity at room temperature of popped millet flour.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	8149.67	5	1629.93	1.75	0.2409
A	771.48	1	771.48	0.83	0.3925
B	594.41	1	594.41	0.64	0.4501
A^2	2840.89	1	2840.89	3.06	0.1239
B^2	1106.62	1	1106.62	1.19	0.3113
AB	585.88	1	585.88	0.63	0.4533
Residual	6504.98	7	929.28		
Lack of Fit	4341.40	3	1447.13	2.68	0.1828
R^2	0.5561				

Table 4.17 ANOVA results presenting the effect of popping conditions on oil absorption capacity at 140°C of popped millet flour.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	487.08	5	97.42	2.56	0.1267
A	66.45	1	66.45	1.74	0.2283
B	154.15	1	154.15	4.04	0.0843
A^2	200.55	1	200.55	5.26	0.0555
B^2	0.92	1	0.92	0.024	0.8808
AB	42.93	1	42.93	1.13	0.3238
Residual	266.85	7	38.12		
Lack of Fit	155.15	3	51.72	1.85	0.2783
R^2	0.6461				

Fig. 4.20 shows that although popped millet flour absorbed more oil than native millet flour, there was variability among the different popped millet samples and even among the central points of the statistical analysis. Though the analysis showed that the model was insignificant for both temperature and moisture, it can be interpreted from Fig. 4.20, that the oil absorption capacity was affected by the moisture content – it increased with an increase in moisture from 14% to 16% and decreased when the moisture content was increased from 16% to 18%, which is in accordance with the study by Rock-Dudley (1993) where he stated that when the initial moisture content was low the oil absorption was higher.

Singh et al. (2004) conducted a study on foxtail millet and reported that both, the hot and cold OAC of popped millet were higher than that of decorticated millet. In a study on the functional properties of sorghum-peanut composite flours, Singh and Singh (1991) stated that the OAC of the roasted and boiled flour samples was higher than that of the native ones, thus showing that thermal processing increased the OAC. An increase in oil absorption due to thermal processing had also been reported by Narayana and Rao (1982), where they showed that the OAC of thermally treated winged bean flour (2.2 g/g flour) was higher than that of the native flour (1.4 g/g flour).According to Hutton and Campbell (1981) the ability of food to absorb oil and water might help in improving the sensory properties like mouth-feel and flavor retention. This meant, in our case, that popped millet flour may have a higher degree of flavor retention and mouth-feel.

4.3.4 Viscosity

It is important to know the viscosity of a food sample and its ingredients as it helps in the formulation of different kind of food items like weaning foods, gruel, soups and porridge by indicating their flow properties at different temperatures. The viscosity of a food also plays a role in its textural properties and mouth-feel. The viscosity of the popped and native millet flour samples was measured as cooked paste viscosity and viscosity at room temperature (cold paste viscosity).

Both, the cold paste viscosity and cooked paste viscosity showed higher values for popped millet flour (PMF) than native millet flour (NMF). The cold paste viscosity of NMF was 5.395×10^{-3} Pa s while that of PMF was in the range 1.48 – 7.46 Pa s. NMF had a cooked paste viscosity of

0.191 Pa s whereas that of PMF varied between 1.98 – 7.54 Pa s. Also, the cooked paste viscosity of NMF was higher than its cold paste viscosity. A reason for this increase in viscosity, due to the addition of hot water and due to popping, could be the inactivation of α-amylase as the enzyme is known to have a liquefying action on starch (Web Ref. #24).

Statistical analysis presented in Table 4.18 showed that while both temperature ($p< 0.0102$) and moisture ($p< 0.0426$) significantly affected the cold paste viscosity, the interaction between the two factors ($p< 0.8055$) did not. Also, as seen in Fig. 21, there was a point of maxima reached by both temperature and moisture. The quadratic term for moisture had a significant effect on the viscosity with $p< 0.0427$ while that of temperature ($p< 0.0757$) did not.

Table 4.18 ANOVA results presenting the effect of popping conditions on the cold paste viscosity of popped millet flour.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	30.56	5	6.11	7.03	0.0118
A	5.32	1	5.32	6.12	0.0426
B	10.56	1	10.56	12.14	0.0102
A^2	5.32	1	5.32	6.11	0.0427
B^2	3.77	1	3.77	4.34	0.0757
AB	0.057	1	0.057	0.065	0.8055
Residual	6.09	7	0.87		
Lack of Fit	2.13	3	0.71	0.72	0.5906
R^2	0.8339				

The predicted model for the viscosity at room temperature (RT) can be described in terms of coded factors by the following equation (4.8):

$$\textbf{\textit{Viscosity at RT (Pa s)}} = 6.09 + 0.94 * A + 1.33 * B - 1.39 * A^2 - 1.17 * B^2 + 0.12 * A \times B \quad (4.8)$$

The response surface plot (Fig. 4.21) shows the effect of temperature, moisture and their interaction on the cold paste viscosity. The cold paste viscosity increased as the moisture increased from 14% to 16%, reached a maximum and then decreased with a further increase in moisture. It exhibited a similar trend for temperature. The cold paste viscosity increased with an increase in temperature, reached a point of maxima at around 250°C and then decreased with a further increase in temperature. The interaction between the factors did not have any effect on the cold paste viscosity.

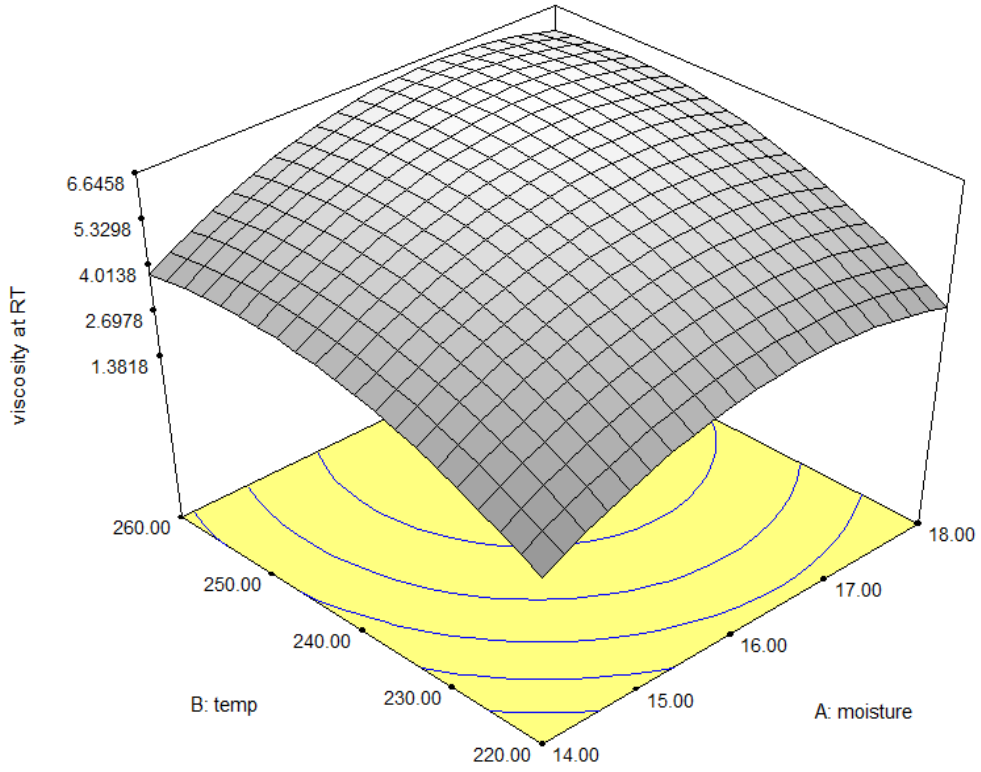

Fig. 4.21 Response Surface Plot presenting the effect of temperature and moisture content on viscosity at room temperature (30°C).

The statistical analysis presented in Table 4.19 for cooked paste viscosity showed that only the moisture content (‘*A*’, $p< 0.0216$) and its quadratic term (A^2, $p< 0.0688$) had a significant effect

on it. Neither the temperature nor the interaction between the factors had any effect on the cooked paste viscosity.

Table 4.19 ANOVA results presenting the effect of popping conditions on the cooked paste viscosity of popped millet flour.

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	18.48	2	9.24	5.77	0.0215
A	11.83	1	11.83	7.39	0.0216
A^2	6.65	1	6.65	4.16	0.0688
Residual	16.00	10	1.60		
Lack of Fit	12.50	6	2.08	2.38	0.2103
R^2	0.5360				

The predicted model for the cooked viscosity can be described in terms of coded factors by the following equation (4.8):

$$\textit{Cooked Paste Viscosity (Pa s)} = 5.07 + 1.40 * A - 1.43 * A^2 \quad (4.9)$$

The response surface plot (Fig. 4.22) for the effect of popping conditions on cooked paste viscosity showed that it increased with an increase in moisture, reached a maximum value around 16.5% moisture content and then started decreasing. The temperature of the popping medium did not have any effect on the cooked paste viscosity. Though there was not much difference observed in the cold paste and cooked paste viscosity of the PMF, NMF showed a remarkable difference between its cold paste and cooked paste viscosity. Brandtzaeg et al. (1981) reported that the viscosity of malted and unmalted ragi flour increased after toasting. As mentioned earlier, an increase in viscosity after a thermal treatment could be due to the inactivation of α-amylase, an enzyme that has a liquefying effect on starch.

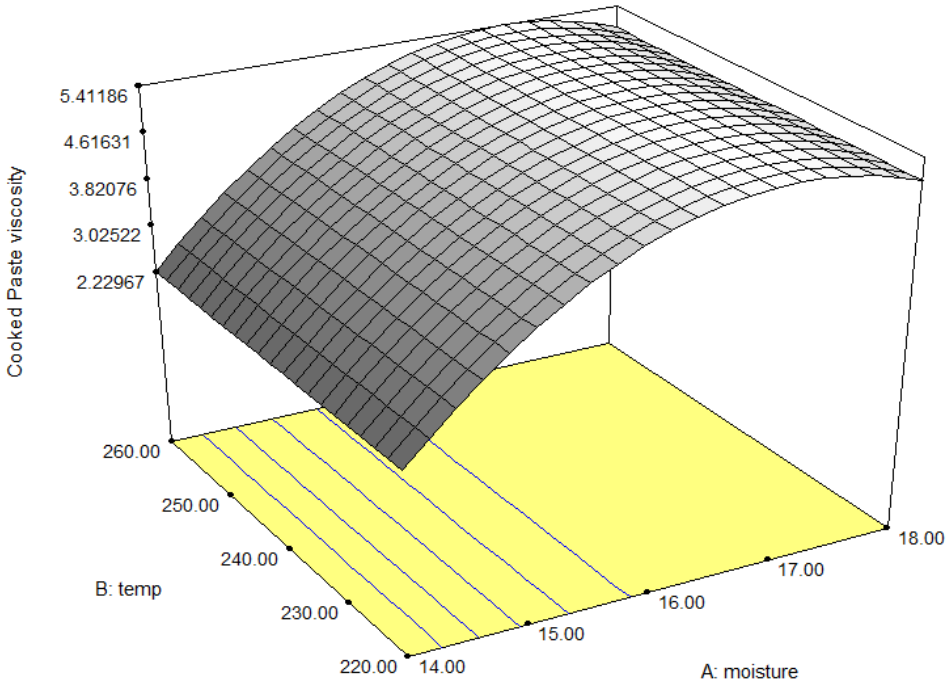

Fig. 4.22 Response Surface Plot presenting the effect of temperature and moisture content on the cooked paste viscosity.

As stated by Olukku and Rha (1978), the viscosity of flour is related to the behavior of its starch content. Viscosity changes due to the swelling pattern of the starch contained in the sample. The starch granules can absorb water only up to a particular limit and once they are saturated with water they leave the starch matrix and move freely in the solution, thus causing a decrease in the viscosity of the starch-water system. The millet was popped using different combinations of moisture and temperature and therefore, its content of partially gelatinized starch also varied and hence, the difference in viscosity.

4.3.5 Pasting Characteristics

It is important to classify the viscosity pattern of starch so that it could be categorized for end product recommendation. Quite a lot of changes occur upon heating starch in a water system which include increased viscosity, higher swelling and solubility and loss of birefringence

(Ikegwuet al., 2010). Changes in the viscosity of starch in suspension occur due to the swelling and solubilisation of starch granules upon heating. When cooled, the amylose molecules start re-associating and form an opaque gel or precipitate. This process is called retrogradation or setback (Ibanez et al., 2007).

The pasting characteristics of popped, whole and decorticated little millet flour were determined using a stress controlled rheometer (AR 2000 Rheometer, TA Instruments). It was observed that the pasting pattern of little millet flour, be it the whole (native), decorticated or popped form, was very different from other flours like rice flour and wheat flour. While other flour samples have been reported to show sharp peaks and troughs in their pasting pattern, this was not the case with little millet flour. Figs. 4.23 and 4.24 show the pasting pattern of whole, popped and decorticated little millet flour.

It is clear from Fig. 4.23 that popped millet flour (PMF) followed a pasting pattern similar to that of the native millet flour (NMF) with a slight increase in viscosity values. From Fig. 4.24, it can be inferred that the pasting pattern of decorticated millet flour (DMF) was different from that of PMF and NMF and also, it exhibited higher viscosities as compared to the other two. High viscosity values of DMF could be due to the relative absence of husk in the flour.

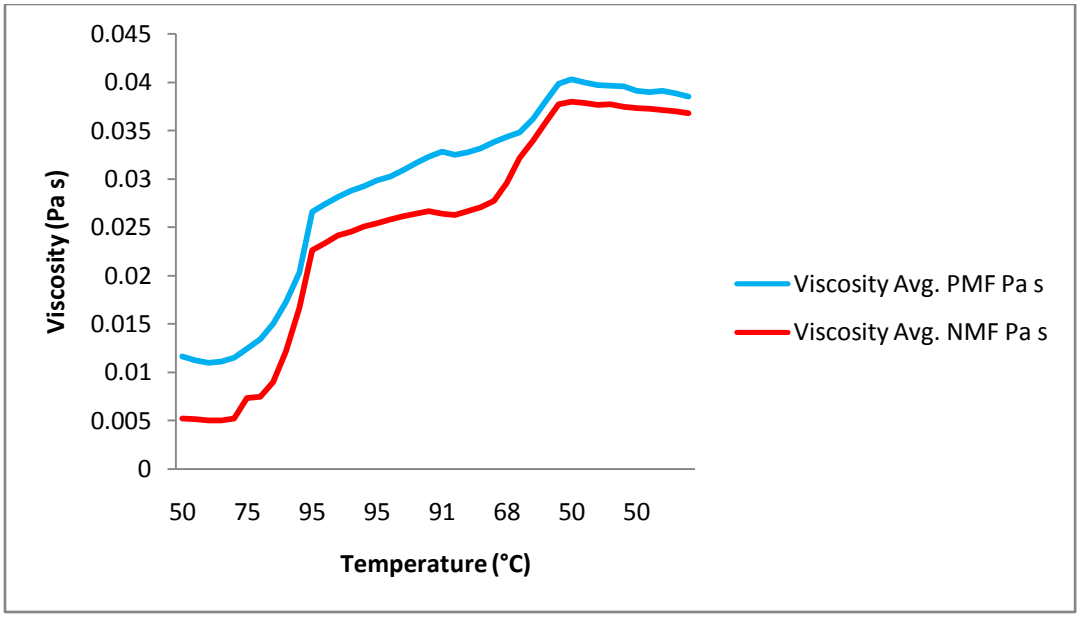

Fig. 4.23 Comparison of the pasting behavior of native and popped little millet flour.

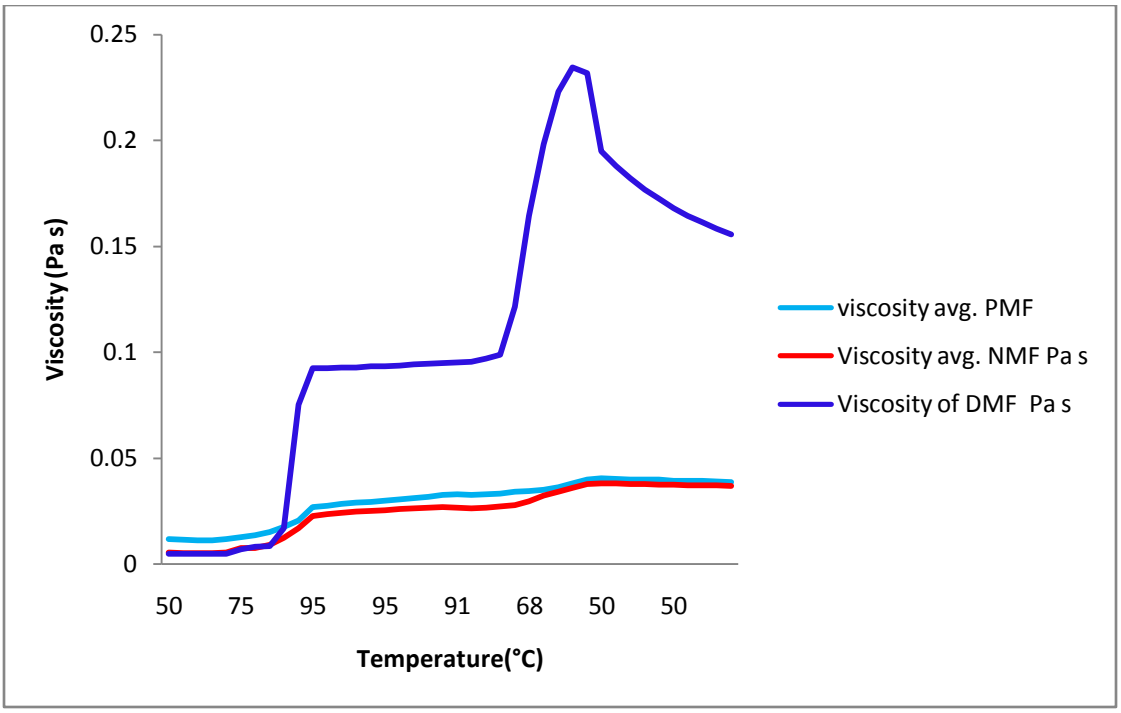

Fig 4.24 Comparison of the pasting behavior of whole, decorticated and popped little millet flour.

The parameters determined from the pasting pattern of flour/starch are the initial gelatinization temperature, peak viscosity, hot paste viscosity, cold paste viscosity, breakdown viscosity and setback viscosity. All these parameters are significant as they help in defining the type of starch in the flour which has further industrial applications. Table 4.20 shows the values obtained for the three samples. Both NMF and PMF had an initial gelatinization temperature of 75°C whereas DMF started gelatinizing at 70°C. The gelatinization temperature for all the three flour samples was similar to that of other cereal flours. In a study on finger millet flour, Malleshi and Shobana (2007) reported that the gelatinization temperature of native finger millet flour was 76°C. Ibanez et al. (2007) confirmed that the initial gelatinization temperature of rice flour and starch were in the range of 63-65°C. In another study conducted on wheat flour, Brenan et al. (2008) stated that the gelatinization temperature of wheat flour was around 68°C.

The water binding capacity of starch is indicated by the peak viscosity (Ikegwu et al., 2010). As there were no sharp peaks and troughs observed for any of the three samples, the highest viscosity reached after the onset of gelatinization and before the holding period was considered

as the peak viscosity namely 0.0226 Pa s for NMF, 0.0266 Pa s for PMF and 0.0925 Pa s for DMF which was almost 4 times the values of the other two. High peak viscosity values are required for high gel strength and elasticity. The low peak viscosity values of NMF and PMF showed that the flour would have less elasticity when kneaded into dough as compared to the DMF which had high peak viscosity values. As reported by Ibanez et al. (2007), the peak viscosity value for rice flour were in the range 0.0430-0.482 Pa s and that for rice starch was in the range 0.0477-0.594 Pa s. These values suggested that rice flour and starch had a higher water binding capacity than little millet flour.

As pasting properties are usually measured using a Rapid Visco Analyzer, not much literature was available to compare the results. This was because a Rapid Visco Analyzer gives the viscosity values in Rapid Visco Units (RVU) and the viscosity measured in the present study was expressed in Pa s.

Table 4.20 Pasting characteristics of native, popped and decorticated little millet flour.

Sample	Initial gelatinization temperature (°C)	Peak viscosity (Pa s)	Hot paste viscosity (Pa s)	Cold paste viscosity (Pa s)	Breakdown viscosity (Pa s)	Setback viscosity (Pa s)
Native millet flour	75	0.0226	0.0263	0.0368	-0.0037	0.0105
Popped millet flour	75	0.0266	0.0331	0.0385	-0.0065	0.0054
Decorticated millet flour	70	0.0925	0.0989	0.1557	-0.0064	0.0568

The hot paste or trough viscosity is the minimum viscosity value obtained in the constant temperature phase and determines the ability of the paste to withstand breakdown during cooling. As mentioned earlier, there were no clear peaks or troughs obtained for any of the three samples

so the last viscosity value just before the second rise was taken as the hot paste viscosity. The hot paste viscosity of NMF and PMF were 0.0263 and 0.0331 Pa s while that of DMF was 0.0989 Pa s.

When aqueous suspensions of starch granules are heated, the granules start swelling due to which they become susceptible to heat and shear. This leads to the fragmentation of starch and a reduction in viscosity which indicates the breakdown of starch. Breakdown is not desirable as it results in uneven viscosity and would affect the cohesive nature of the starch paste (Moorthy, 2004).The breakdown viscosity is calculated as the difference between the peak viscosity and the hot paste viscosity. It is an important factor affecting the properties of starch in food. As shown in Table 4.20, NMF, PMF and DMF had negligible breakdown viscosity. Due to this, they have a higher tendency to withstand heat and shear stress during cooking. Moreover, they would form pastes of uniform viscosity in food formulations.

The cold paste viscosity is the value obtained at the end of the test and gives the change in viscosity after holding the cooked starch. The cold paste viscosity was higher than the hot paste viscosity in all the three cases being 0.0368 Pa s for NMF, 0.0385 Pa s for PMF and 0.1557 Pa s for DMF. This could be attributed to the retrogradation of starch during cooling which results in formation of a gel like structure. When gels are cooled down they do not dissociate, instead solidify, thus increasing the viscosity of the paste. It is important to know the final viscosity of starch as it indicates the quality of starch and determines its stability to form gels and pastes.

Setback viscosity is another important factor which is attributed to the retrogradation of starch during cooling and is calculated as the difference between the cold paste viscosity and hot paste viscosity. From Table 4.20 it is clear that PMF had the lowest setback viscosity of 0.0054 Pa s while the setback viscosity of NMF 0.0105 Pa s was almost double of PMF. DMF had the highest value of setback viscosity of 0.0568 Pa s which was almost 5 times that of NMF and 10 times that of PMF. It was observed that DMF had a higher tendency of forming a gel like structure as compared to NMF and PMF. The PMF would lose a lot of water and would become dry by the end of the experiment but did not form anything close to a gel. This meant that there was less retrogradation in PMF. Supporting this result was the study by Ikegwu et al. (2010) where they stated that a lower setback value is indicative of lower retrogradation in food products. This was again supported by the fact that retrogradation reduces the digestibility of

starch whereas popping increases starch digestibility (Mangala et al., 1999). As popped millet had an improved digestibility, its retrogradation value was lower. This indicated that PMF could be used for formulating food products with better shelf life.

Chapter 5

Summary and Conclusion

5. Summary and conclusion

Millets are minor cereals that are usually consumed by people with a low socio-economic status in many parts of the world, in particular in Africa and India. Millets are wonder crops as they can grow in hot, dry and drought prone areas and have a short harvesting period of around 45-65 days. Studies show that millets are highly nutritious cereals with protein levels as high as those of rice and wheat. The fiber content of millets is way higher than that of wheat and rice. Millets are also rich in vitamin B and certain minerals like potassium, zinc, magnesium, manganese, iron, phosphorous and copper. Certain non-nutritional compounds like phenols, tannins and flavonoids, that have antioxidant properties, are also found in millets. Millets are non-glutinous cereals and are perfect for people with wheat/gluten allergies.

In spite of all these beneficial properties, the consumption of millets is low. This is partly because of their non-popularity among high income populations and partly because of their unavailability in ready to eat forms. As millets lack gluten, it is almost impossible to use them for bread making without the addition of any hydrocolloids, the addition of which would increase the cost of the product and make it difficult to market. Developing countries are focusing energies on using millets by making them readily available to the masses. The present study aimed at making the millets available to people in ready-to-eat form with some enhanced nutritional, functional and physico-chemical properties. Little millet (*Panicum sumatrense*, var. Sukshema) was selected for this purpose as this millet has been mostly neglected by research groups addressing millets from around the world.

In the present study, little millet was popped like popcorn to make it available in a ready-to-eat snack format. The study aimed at optimizing the popping conditions and to study the effects of popping on the proximate and functional properties.

The millet was popped using salt as the hot particulate medium. The popping conditions were optimized with respect to the temperature of the particulate medium and the moisture content of the millet. The experiment was designed for two factors at three levels each: temperature (220°C, 240°C and 260°C) and moisture (14%, 16% and 18%) and a total of thirteen combinations were obtained using a Central Composite Design with five central points. A gas chromatograph oven HP 5890A) was used to heat the salt to the desired temperatures. Prior to

popping, the grains were tempered for a period of 18 hours, with a calculated amount of water, to bring them to the desired level of moisture. The salt was heated to the desired temperature and the tempered grains were mixed with it using a spatula. Popping was observed in less than two minutes. The popped grains were mechanically sifted from the unpopped grains and salt using a sieve. The weight of the popped and unpopped grains was recorded and the yield of popped millet was calculated as a percentage. It was observed that the yield of popped millet was governed by both, the temperature and the moisture content. The highest popping yield was obtained at a moisture content of 16% and temperature of 260°C while lowest was obtained at 14% moisture and 220°C.

For further analysis, the popped millet was pulverized to flour using a coffee grinder. Native millet flour was used as the control. The popped millet flour (PMF) and native millet flour (NMF) were analyzed for moisture content, total protein (BCA Assay Kit), resistant starch (Megazyme Resistant Starch Assay Kit), crude fat, total ash, total phenolic content, oil absorption, swelling power and solubility, viscosity and pasting characteristics.

The moisture content of the millet decreased after popping. This was beneficial as low moisture prevents growth of microbes and increases the shelf-life of the product. There was a negligible difference observed between the total protein content of PMF and NMF. The total protein content of NMF was 11.69% while that of popped millet flour varied from 10.02% to 11.41%. This showed that popping did not affect the protein content of the grain thus, keeping the flour as proteinaceous as the one obtained from native millet. Similar results were obtained for total ash and crude fat content. The total ash content of PMF varied from 3.47% - 5.35% while that of NMF was 4.508% which showed that popped millet contained almost the same amount of minerals as native millet. The crude fat content of native millet flour was 5.5% while that of PMF ranged between 5.5 – 6.3%.

Millet starch, in its native form, was mostly unavailable for enzymatic digestion (trapped within the matrix) and thus, its resistant starch (RS) content was high (16.85%). Popping resulted in the expansion of the endosperm to form a spongy starch matrix and thus increased the availability of starch for enzymatic digestion. The RS content of little millet decreased considerably after popping and varied from 1.82% - 5.57%. This increased the Non-Resistant starch content and the

Total Starch content of the popped millet and also resulted in an increase in starch digestibility by making it readily available for enzymatic digestion.

Millets are known to contain polyphenols that are naturally occurring antioxidants and have nutraceutical properties. The availability of total phenolics increased from 225 mg GAE/100g sample (db) in NMF to 661.46 mg GAE/100g sample (db) in PMF thus establishing popped millet to be a better source of polyphenols.

The swelling power (SP) and solubility are the two properties of flour that have a great industrial importance especially in the formulation of pasta, noodles and during bread making. The swelling power and solubility of the flour samples were measured at three different temperatures namely 30°C, 50°C and 100°C. It was observed that the swelling power and solubility of PMF were way higher than that of NMF at 30°C and 50°C but had almost similar values at 100°C. The higher SP and solubility of PMF could be attributed to the porous nature of its starch matrix. A strange behavior was observed in the swelling pattern of both PMF and NMF – the swelling power of the flour gave a lower value at 50°C than what it gave at 30°C and then again increased when measured at 100°C. The solubility of NMF increased steadily with an increase in temperature but that was not the case with PMF. The solubility for popped millet samples followed different patterns – it increased steadily for certain samples, decreased for one and followed the same pattern as SP for certain other samples. As PMF exhibited a higher SP and solubility than NMF, it could be a better option for the formulation of foods like pasta and noodles. As millet flour is non-glutinous, mixing it with certain hydrocolloids could serve the purpose for certain bakery applications.

PMF absorbed more oil than NMF both at room temperature and at 140°C thus confirming that PMF may not be most suitable for making food items that require frying or need to be mixed with oil for making dough or certain other formulations. The cold paste and hot paste viscosity of PMF were higher than that of NMF which meant that the slurry formed using PMF would be thicker. Usually, food formulations require thin and free flowing gruels which are less viscous for ease of distribution. PMF would be inappropriate for such food formulations but could be used in making other ready-to-make food products that are to be prepared as a thick paste.

The pasting properties showed that PMF and NMF exhibited a gelatinization temperature similar to common cereals. The pasting properties of PMF were compared with NMF and Decorticated Millet Flour (DMF). According to the results obtained it can be concluded that food products prepared from PMF would form pastes of uniform viscosity which would be more stable to heat and shear stress during cooking and would have a higher shelf life.

The present study successfully optimized the popping conditions for little millet and also established the popped millet to be a good source of nutrients. It also confirmed that popped millet flour has certain advantages over native millet flour with improved functional and nutritional properties. Popped millet can be consumed directly as a snack, while flour made from popped millet can be incorporated in various formulations for ready-to-eat and ready-to-make food products. The study highlights that the not-so-popular little millet can be used as a nutritious food component with improved industrial applications thus broadening the market for little millet and can also be used to provide economical, healthy and nutritious choices for the low income populations where little millet is traditionally grown.

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