Optimization of a two-stage drying process for brewers' spent grains

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Presented to the Department of Bioresource Engineering
In partial fulfillment of the requirements
For the Degree of Master of Science

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January 9, 2019

ABSTRACT

This thesis examines a two-stage drying method to dehydrate brewers' spent grains as part of exploring a waste valorization process. Drying the grains may be beneficial from the brewer's perspective in that it (i) reduces waste disposal costs, especially when coupled with a densification method such as pelletizing, and (ii) produces a co-product that can be sold as animal feed, or to other industries, for its physical and chemical properties. Drying parameters were evaluated using a response surface methodology having a face centered central composite design. The two stages included dewatering initially to mechanically remove free water, followed by a thermal drying technique in order to reduce the moisture level enough for safe storage or further processing. The efficiency of the two-stage drying method was examined and the quality of the feed was assessed following each drying test.

The optimal condition to reduce the maximum amount of water through lab-scale mechanical pressing was found, and the predictive model was statistically significant (p <0.0001) having a strong correlation with experimental results (R^2=0.98). As a maximum, water content was reduced by 35%, which resulted from a pressure of 1.19 MPa and holding time of 2 minutes and 44 seconds. Subsequently optimization of the thermal drying stage was studied. Seven response variables related to quality and efficiency were measured as a function of drying time and microwave power density (W g^{-1}). Only the drying time response was adequately modeled (p<0.0006) with a high coefficient of determination (R^2=0.99) to predict the most efficient drying time. Predictive models were insignificant (p >0.05) for color (i.e., L^*, a^* and b^*), crude protein, acid detergent protein (ADP as % CP) and acid detergent fiber (ADF). Finally, air temperature and microwave density were optimized to yield increased grain lightness (L^*), minimize drying time, minimize the percentage of bound protein to fiber (ADP) and minimize acid detergent fiber (ADF). The latter two parameters are chemical indicators of grain overheating. The most desirable drying parameter setting was an air temperature of 64 ºC and microwave assisted with a power density setting of 2 W g^{-1} in order to achieve the most efficient drying time with the best product quality.

RÉSUMÉ

Cette étude porte sur le développement d'une méthode de séchage en deux étapes pour sécher les drêches de brasserie comme procédé de valorisation des déchets. Du point de vue du brasseur, le séchage des drêches est bénéfique dans la mesure où elle permet de réduire les coûts d'élimination des déchets, en particulier lorsqu'elle est associée à une méthode de densification telle que la granulation, et qu'elle permet de faire un coproduit pouvant être utilisé comme aliment pour animaux, ou par d'autres industries, pour ses propriétés physiques et chimiques. Des techniques de séchage ont été évaluées en utilisant une méthodologie de surface de réponse ayant une conception composite centrale centrée sur la face. Les deux étapes comprenaient une étape initiale de déshydratation partielle par compression mécanique suivie d'un séchage thermique microonde pour réduire la teneur en eau et permettre un stockage sécuritaire. L'efficacité de la méthode de séchage en deux étapes a été évaluée et la qualité du produit traité a été suivie tout au long des essais.

La condition optimale pour extraire la quantité maximale d'eau par compression mécanique a été optimisée et les résultats ont permis de développer un modèle prédictif. Le modèle était statistiquement significatif ($p < 0,0001$) et présentait une forte corrélation avec les résultats expérimentaux ($R2 = 0,98$). C'est ainsi qu'il a été possible de réduire de près de 35% la teneur en eau des drêches en maintenant une pression de 1,19 MPa pour une période de 2 minutes et 44 secondes. Par la suite, l'optimisation de la phase de séchage thermique a été étudiée. Sept variables liées à la qualité et à l'efficacité du procédé ont été mesurées en fonction du temps de séchage et de la densité de puissance des micro-ondes ($W \cdot g^{-1}$). Seule la réponse au temps de séchage a été modélisée de manière adéquate ($p < 0,0006$) avec un coefficient de détermination élevé ($R2 = 0,99$). Le modèle mathématique permet de prédire le temps de séchage en fonction des conditions utilisées. Les corrélations entre les conditions de séchage et la couleur, la protéine brute, la protéine détergente acide (ADP en% CP) et des fibres insolubles dans les détergents acides (ADF), n'étaient pas significatives. Les résultats ont permis d'identifier les conditions optimales de température de l'air et de la densité de puissance micro-ondes pour maximiser la pâleur de la couleur des drêches séchées (L *), pour minimiser le temps de séchage, pour minimiser le pourcentage de protéines liées aux fibres (ADP) et pour minimiser les fibres insoluble au détergent acide (ADF). Les deux derniers paramètres sont des indicateurs chimiques de la surchauffe du grain. Le paramètre de séchage le plus souhaitable pour minimiser le temps de séchage et pour optimiser la qualité du

produit séché était une température de l'air de 64 ºC et une densité de puissance micro-ondes de 2 $W\cdot g^{-1}$.

ACKNOWLEDGEMENTS

I would like to sincerely thank Dr. Mark Lefsrud for providing the platform to explore and challenge scientific research in the areas of biomass and waste valorization. His support throughout the process has been invaluable and I genuinely enjoyed his insightful collaboration. I would like to thank Yvan Gariepy for his dedication and support in the lab. It was a joy to work with him, and he helped to keep the analytical work in the lab moving forward. He is an incredible asset to the department. I would like to thank the Bioresource Engineering department and Biomass Laboratory for fostering a creative environment. Finally, I would like to thank my beloved family and husband. Thank you James for your unwavering support.

FORMAT OF THESIS

The current thesis explores a two-stage drying process in efforts to valorize brewers' spent grains, which is a byproduct from the brewing industry. Chapter 3 will discuss the initial dewatering stage to predict water loss from 2 independent variables: press pressure (MPa) and holding time (mins). Chapter 4 describes the methods employed to determine the most efficient thermal drying conditions in order to predict the best drying temperature to reach the targeted moisture content of 15% w.b. Finally, Chapter 5 will conclude the study by maximizing quality parameters under the most efficient drying conditions. The drying time and quality characteristics are predicted under defined hot air microwave-assisted dryer parameters.

CONTRIBUTION OF AUTHORS

For this thesis the contribution of authors are as followed: (1) Christine Crowe – identified the research topic, designed and optimized the two-stage drying process for BSG, managed and conducted all experiments, performed all data collection, determined testing and organized shipment of samples which required 3rd party lab testing, processed and interpreted data, and explanation of results; (2) Mark Lefsrud – supervised research, provided guidance, knowledge and reviewed thesis; (3) Yvan Gariepy – supervised laboratory research described in Chapters 3, 4 and 5, provided guidance for experimental design and testing, and reviewed the research proposal.

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Chapter 1: Introduction

1.1 Brewers' spent grains and the importance of waste valorization

Brewers' spent grains (BSG) are a by-product from the fermentation or beer making process, and more specifically is the solid mass of grains separated from the wort during the lautering stage (Briggs et al., 2004). BSG grains include residues from malted barley or non-malt fermentable sugars, more often referred to as adjuncts, such as wheat, rice or maize (Mussatto et al., 2006) and are 85% of the total by-products created from the beer making process (Mussatto, 2014). Per 100 liters of beer, 20 kilograms of waste as spent grains is produced (Buffington, 2014). BSGs may be considered underutilized when composted or landfilled due to their rich protein content ranging from 18-31% dry matter (Santos et al., 2003, Johnson et al., 2010, Machado et al., 2015, Canedo et al., 2016), and should have more value for human and animal consumption. Crude protein was increased even further 2 to 4 times with the use of micoorganisms and solid-state fermentation (Canedo et al., 2016). In terms of BSG directly from the brewery, the protein content in these grains may vary slightly between breweries due to brewery mashing techniques and their malt source; for example, lager malt is known to have higher protein than ale malts (Robertson et al., 2010; Ivanova et al., 2017).

1.2 Problem statements and the proposed solution

As previously stated, brewers' spent grains (BSG) are the insoluble grain residues remaining from the wort separation in the brewing process. However, roughly 39 million tonnes per year of these grains are generated globally and have a high moisture content (70-85% w.b.) causing rapid spoilage (Lynch et al., 2016; Mussatto et al., 2014). Within 48 hours of production, BSG can transform into a toxic waste (Machado et al., 2015). Therefore this residue needs to be disposed of rapidly as a low value food waste.

These BSG are considered a high protein and fiber-containing residue, which is why most often BSG are used as animal feed (Lynch et al., 2016; Mussatto et al., 2004). Moreover, stricter regulations regarding food waste disposal have been implemented to prevent landfill disposal (CEC, 2017) and more novel valorization techniques have been employed to make better use of the physical, chemical and nutritional composition of BSG (Mussatto, 2014). Innovative, economically viable techniques should be developed further to improve the shelf-life of BSG,

without compromising composition and flavor (Ikram et al., 2017). Therefore the current study aims to optimize the parameters for a laboratory-scale 2-stage drying process in efforts to facilitate BSG food waste valorization techniques geared towards the animal feed market.

1.2.1 Research objective

The primary research objective is to find the optimal drying conditions to dehydrate brewers' spent grains in order to use this biomass as animal feed. The study will examine an initial dewatering stage followed by a thermal drying technique in order to improve the shelf life of the BSG. This study will include 3 stages:

1. To optimize mechanical dewatering of spent grain originating at 70-80% moisture (w.b.). At the dewatering stage, the goal is to reduce the final moisture content to 25-50-% (w.b.). This will be done by establishing the relationship between the quantity of water removed as a function of the applied load and holding time.
2. Following dewatering, compare the predictive drying models of spent grain in a heated air convection microwave assisted dryer. The final moisture content of 15% was chosen in order to be able to further process (e.g. pelletize) and reduce the water activity for safe storage.
3. Assess quality characteristics as a result of thermal drying treatments. Drying efficiency will be optimized while maximizing quality of the grain biomass residue. The intention is to retain protein quality expressed as ADP (acid detergent insoluble protein or bound protein), and determine if the drying treatments result in increased bound protein, which is a common quality indicator of heat damaged grain feed.

Results from assessing the lab-scale 2-stage drying process will be used to determine an optimal drying method to diversify the utilization of brewers' spent grain in efforts to create a value-added by-product from the brewing process.

1.2.2 Hypothesis

For food waste such as brewers' spent grains, efficient processing conditions should be maximized in order to be able to yield a value added product in efforts to valorize waste. The experiment will highlight the 2-stage drying process related to mechanical water loss, drying efficiency and product quality as a result of drying treatments. The hypotheses are:

- Increasing press pressure increases water loss

- Increasing press holding time increases water loss
- Drying efficiency improves as temperature increases
- Drying efficiency improves as power density increases
- Protein digestibility will decrease as temperature increases
- The grains will darken linearly as temperature increases

Chapter 2: Review of BSG valorization and current state

2.1 Introduction to brewers' spent grains

The protein originating from the barley grain used in brewing is distributed in the different parts of the grain itself, and at varying concentrations among these structural parts (Figure 1). More specifically, the germ and aleurone layers contain the highest concentration of protein, however the starchy endosperm is the largest part of the grain and therefore includes roughly 70-80% of the total protein (Serna-Saldivar, 2010). In terms of material processing, having an understanding where the protein exists within the different size fractions of BSG by-products is important to consider in order to retain the size fraction that yields the most protein in order to generate a protein rich co-product. Various studies have shown that protein content relates to grain fraction size, where the fine fraction (<250um) contains the majority of protein (Jay et al., 2008; Celus et al., 2006).

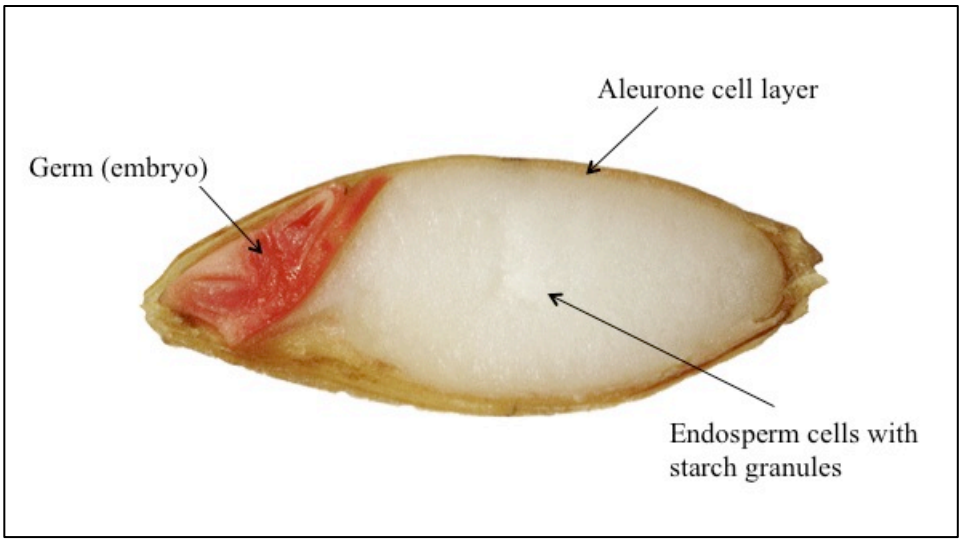


Figure 1. Barley grain anatomical structure pregermination (reprinted with permission from MAGB secretariat, see Appendix B).

2.1.1 Brewers' spent grain as animal feed

Spent grains are considered a nutritional animal feed, specifically due to their fiber and protein contents, however the protein composition and whether it is degradable, undegradable or a soluble protein source are important classification values of the protein (Sniffen et al., 1992). The soluble protein fractions of the grain, albumins and globulins, contain enzymes,

nucleoproteins and glycoproteins, where these proteins account for the best amino acid balance, including a large quantity of lysine, and are easily digested (Serna-Saldivar, 2010). BSG is known for having a higher bypass or undegradable protein content (Thomas et al., 2010, Serna-Saldivar, 2010), which is suitable for a diet of high-producing, early-lactation dairy cows or rapidly growing starter beef cattle (Westendorf et al., 2002; Omafra, 2012). Additionally, in terms of nutrition, BSG is known to have high mineral and vitamin contents (Huige, 1994, Pomeranz and Dikeman, 1976, Mariani, 1953, as cited in Mussatto, 2006). Beyond BSG protein quantity and quality, Westendorf et al (2002) references the high fiber and fat contents found in BSG resulting in this grain to be a significant source of energy.

Ikram et al (2017) created a comprehensive review on potential techniques to extract proteins and phenolic compounds from BSG to increase the number of applications to extend beyond animal feed to consume the antioxidant phenolic compounds. Antioxidants such as ferulic acid and *p*-coumaric acid, are known to be present in the lignin of BSG (Ikram et al 2017; Bartolome and Gomez-Cordoves 1999; Mussatto et al., 2006; Jay et al 2008). Ivanova et al (2017) examined different malt blends and conditions in order to maximize the total phenolic compound value and antioxidant capacity of the BSG by-product. With this type of analysis on the brewing process, breweries can then target certain malt blends and conditions to yield food co-products. Therefore valorization efforts impact industries beyond the animal feed industry, with potential for human nutrition as well. Vieira et al (2016) emphasized that the availability of BSG residues at no to low cost throughout the year, while having a high nutritional value, makes it a promising raw material to exploit for both human nutrition and biotechnology processes.

2.1.2 Waste management and handling issues with brewers' spent grains

The problem with BSG is that it has a short shelf life and degradation can begin almost immediately once discarded due to the rapid onset of microflora (Robertson et al., 2010) and spoils within 48 hours (Machado et al., 2015) to 7-10 days (El-Shafey et al., 2004). Degradation can accelerate in warmer temperatures due to its high moisture content (75-80%) and high fermentable sugar content (Johnson et al., 2010). These are favorable conditions for increased microbial activity leading to spoilage, and ultimately an unwieldy waste product to dispose of that produces foul odors and butyric acid. In a study from Mussatto et al (2014), it was recommended to reduce the moisture content of BSG to less than 100 g kg^{-1}, which can be costly due to the initial high

moisture content. Therefore, efficiency in the drying process is a crucial piece to the valorization process of spent grains.

BSG originates from the mash tun where brewing adjuncts, such as ground malt and cereals, are mashed or infused in order for enzymes to convert starch and dextrins to soluble sugars, cause the partial breakdown of proteins, degrade nucleic acids and other substances, to ultimately extract carbohydrates prior to adding hops to the wort (Briggs et al., 2004). High temperatures of the grains exiting the mash tun, high moisture, proteins, and sugars foster a favorable environment for microbial growth. In addition, the physical properties of BSG such as particle size, volume weight, specific density, porosity and water-holding capacity may play an integral role in the growth of microflora and ultimate spoilage of BSG as well (Wang et al., 2001, Mussatto et al., 2014).

Due to the nature of spent grains, handling and stabilizing are time sensitive. For example, Robertson et al (2010) found that at the point of production, the BSG is within acceptable limits for food use with respect to its microbiology (i.e., micro-organisms, yeasts and molds were below or within limits). However, with the presence of aerobic thermophilic bacteria as a result of temperatures used in the mashing process, bacterial growth can quickly begin from this point due to the spent grains exiting at a high temperature and commonly stored in areas with reduced airflow. This leads to anaerobic conditions and the quick proliferation of anaerobes post-production. Robertson et al (2010) showed a rapid onset of such anaerobic conditions where bacteria thrive and reduced the value of these grains due to deterioration, which ultimately drives a costly disposal of saturated grains. Therefore, a drop-in dewatering and drying system would be beneficial to quickly process the exiting grains.

2.1.3 Argument for a waste valorization solution

In general, breweries focus their efforts on the science of beer production and limited attention is paid towards the burden of discovering the most efficient waste removal methods. In the US, 2.4 million tons or 500,000 dry tons of BSG is produced annually, assuming 200 million barrels of beer are produced in a year, and based on a 5:1 ratio of final beer product to spent grains produced (Buffington, 2014). Therefore, brewery waste management is a current and growing issue. Furthermore, the number of breweries is increasing, as current trends indicate from the Brewers Association, and could escalate into a more serious waste management concern (Brewers Association, 2016). According to the Brewers Association (2016), the number of US breweries rose by 158% from 2012–2017, with microbreweries rendered most successful. As a result, more

waste is inevitably generated, especially as the number of breweries per capita increases in urban areas (Brewers Association, 2018), and not every microbrewery will have a farm nearby or enough capital to invest in an anaerobic digester, or some other form of onsite waste-to-energy conversion. Capital costs for waste conversion would include the reactors themselves, highly trained personnel, laboratory testing or equipment, permits, etc. Regarding waste transportation, the cost alone is an average US$16 per tonne of wet BSG transported a distance of 5 miles (~8 km) (Mussatto et al., 2014). One previous example of savings from dewatering food waste was in New York City with New York Wa$teMatch. This organization was able to save roughly $17,000 in one year from dewatering food waste (Orsat and Raghavan, 2007).

If breweries are presented with a cost-effective and sociably responsible solution, they can then be empowered to sensibly manage BSG by-product that has a market value for its chemical and physical properties rather than landfilling or composting. For example, Mussatto et al (2006) describes a number of ways BSG may be reused. Specifically as animal feed, dairy cattle are the primary source for BSG and have been known to increase milk production, milk total solids and milk fat without affecting the animal's health or fertility. More protein is required during lactation in dairy cows and therefore protein requirements increase with milk production (Serna-Saldivar, 2010). In addition, the brewing process naturally heats the grains and enhances their nutritional value due to starch gelatinization and protein denaturation, which improves palatability, digestibility and feed efficiency in ruminants (Serna-Saldivar, 2010). Furthermore, this feed can be enriched to include all amino acids for ruminants by adding low cost nitrogen sources such as urea (Mussatto et al., 2006).

From Mussatto et al (2006), other animals can benefit from consuming BSG such as poultry, pigs and fish. Additional utilization methods described include human nutrition, energy production, charcoal production, a constituent in brick making, paper manufacturing, as an adsorbent for removing VOCs, biotechnological processes, and as a substrate for enzyme production and the cultivation of microorganisms (Mussatto et al., 2006). Buffington (2014) and Machado et al (2015) highlight current research where BSG is used to produce a number of high value products which include food grade chemicals as a result of its composition, energy through microbial fermentation, and the building blocks for bio-plastics (i.e., lactic acid).

2.2 Current state of brewers' spent grains

Although BSG has a number of potential uses, it is most notably used for animal feed due to its high protein and fiber content, or unfortunately landfilled due to challenges with material handling. This is where dewatering and drying of BSG to reduce transportation costs, and find a higher value for multiple end-users, becomes a worthy solution to investigate. Although chemical methods to increase the storage time of BSG exist by using benzoic and formic acids, in addition to potassium sorbate in pressed BSG (Kuntzel and Sonnenberg, 1997, as cited in Mussatto et al., 2006), preserving the natural state of the BSG is vital in order to market the product to multiple end-users, and water removal is imperative for efficient transport. According to OMAFRA, wet spent grains as a feed are currently valued at $45 per tonne, and $172 per tonne dried (OMAFRA, 2016). These feed prices may vary up to 20% based on corn and soybean meal prices (Table 1). Buffington (2014) reported a similar fetch price of $40 per wet ton, and the high moisture and reduced sugar content, limits the viability for reuse of this biowaste from large producing centralized breweries and decentralized locations. Solutions must be sought out in order to improve waste management techniques while simultaneously producing a profitable by-product, and at a price below landfill tipping fees.

Table 1. Feed value comparison based upon variable corn and soybean meal prices according to Ontario Ministry of Agriculture, Food and Rural Affairs (OMAFRA, 2016).

	Price per Tonne								
Corn Grain	$120			$150			$180		
Soybean Meal - 48%	$300	$320	$340	$300	$320	$340	$300	$320	$340
	Value per Tonne								
Bakery Waste	$133	$134	$135	$163	$164	$165	$193	$194	$195
Brewers Grain - Wet	$45	$47	$49	$48	$50	$52	$51	$53	$56
Brewers Grain - Dried	$172	$180	$188	$184	$193	$201	$197	$205	$213
Corn Distillers - Wet	$68	$71	$74	$73	$76	$79	$79	$82	$85
Corn Distillers - Dried	$208	$217	$226	$224	$234	$243	$241	$250	$260
Corn Gluten Feed	$86	$90	$93	$93	$97	$101	$101	$105	$108
Corn Gluten Meal - 60%	$364	$390	$417	$356	$382	$408	$348	$374	$400
Corn Hominy	$141	$141	$142	$172	$173	$174	$204	$205	$206

In North America, common disposal routes from breweries to farmers is challenging and are subjected to food and safety compliance as animal feed. This challenge most likely stems from

when BSG spoil; they become less palatable and can have adverse health effects on animals (Westendorf et al., 2002). Westendorf et al (2002) highlighted a number of points regarding the issues with using unregulated BSG as animal feed. Such issues include the need to use the grains quickly where small dairy feedlots may not be able to keep feed rates ahead of spoilage, especially during summer months, resulting in spoiled, less palatable grains. In addition, farmers may not be aware of the grain condition they obtained. For example smaller, microbreweries do not brew everyday and therefore the grains could have already spoiled or the grains could be contaminated with mycotoxins. Finally, only a limited amount of the wet grains can be fed to ruminants with respect to dry matter intake without detrimental effects on performance.

In 2013 the FDA originally proposed a rule to regulate spent grains with the, "Current Good Manufacturing Practice and Hazard Analysis Risk-Based Preventive Controls for Food for Animals" Docket No.:FDA-2011-N-0922, and finally became effective in November 2015. This rule would have required additional controls for brewers' spent grains in order to prevent foodborne illnesses rather than retroactively reacting to such incidences in animals and humans. However, the FDA decided not to regulate the by-product as animal feed since there are no illnesses known with BSG waste and there is no physical or chemical contamination to this product. The FDA states however, breweries still need to follow the CGMP's proposed rule to prevent contamination when holding or distributing the waste, at least until the first major incident occurs and then we should expect more stringent regulation. Under the current rule, further processing such as drying and pelletizing, would then require compliance with the preventive controls for animal food rule. Therefore if a drying and pelletizing process grows to commercialization, it should develop as fully compliant with the FDA in order to gain consumer confidence.

Some breweries may wish to continue donating or privately selling their grains in return for goods (e.g., beef) as long as the FDA does not regulate the by-product as an animal feed. However, if presented with a profitable solution, breweries have the option to sell the co-product in order to absorb some value from the large amount of waste generated. Likewise, breweries will have an alternative solution in the event these grains are regulated.

2.3 Current drying solutions reported for BSG

The value of dried BSG increases nearly 4 times from the original as wet grain (Table 1). However, drying food waste can be an energy intensive process where innovative and sustainable design is crucial to explore in order to make additional processing steps a worthy investment. This

is where the intervention of hybrid technologies and general process enhancement becomes interesting. Benali and Kudra (2010) discussed this promising concept beyond optimization and process engineering as "process intensification" (PI) which works to identify opportunities to boost heat transfer in drying, for example, and integrate proven technologies into existing plant processes that would upgrade its economic impact. Benali and Kudra (2010) specifically targeted dewatering and drying technologies in food processing where drying time was reduced by 35-45% through power ultrasound and radio frequency heating to bolster diffusion rates and mass transfer coefficients. This global concept to improve industrial processes as the PI Action Plan was introduced over a decade ago with a 20% energy reduction goal in industrial processes by 2050. Specific sectors were targeted and success was to be achieved through reducing process plant sizes, equipment, energy consumption, material usage and waste generation (Benali and Kudra 2010). Therefore brewers' spent grain becomes a relevant candidate to research the application of process intensification or optimization, to valorize a food waste by-product to form a nutritional value-added product.

The complexity of the drying process to maintain a certain nutritional value in food items, while reducing costs and drying sufficiently enough to maintain its safe storage is a carefully orchestrated process that has been tackled by many researchers (Woods et al., 1995). Regarding BSG, numerous drying solutions have been reported in literature, and more recently novel techniques have been explored to produce high value products as a result of proven drying processes. From previous experimental research, a final moisture content of 12-15% was obtained by using a dewatering technique comprised of an integrated filter press with membranes and thermal vacuum drying, where the thermal energy is from hot water originating in the brewing process as opposed to costly, energy intensive techniques like fluidized bed or rotary drum drying requiring an external source of artificial heat (Machado et al., 2015). This technique was successful at removing 59% of the water mechanically and bringing BSG from 75% to 15% using recessive membrane plates. Results from this study were mainly dependent on the final cake thickness and drying time, but ultimately resulted in a less energy intense method to remove moisture while preserving proteins.

Johnson et al (2010) summarize a variety of preservation methods, which can affect the nutritional quality of BSG. This study highlights how freezing can affect the arabinose content

which is known to manage glucose and insulin peaks when consumed with sucrose. According to Smith (2007), freeze-drying produces the best quality after rehydration. Johnson et al (2010) reiterate how superheated steam (SHS) drying has distinct benefits of preserving nutrients, although removes the aroma and flavor, resulting in a less palatable product for reuse with humans (Mussatto et al., 2006). While various drying methods have been tried to yield products with specific chemical and physical properties, a scalable, efficient and simple method is generally most sought after.

2.4 Two-stage drying process evaluation

This current BSG valorization research will examine a two-stage drying method to stabilize brewers' spent grain in efforts to increase its market value as an easily managed by-product such as animal feed. The initial stage begins with dewatering to mechanically remove free water followed by a thermal drying technique in order to bring the grains to a moisture level that can be further processed, or easily stored, while having quality protein available. While in Canada there is no storage moisture content standard for BSG, the Canadian Grain Commission advises to store BSG grains at 11 % wt where grains could be stored up to 3 months depending on the relative humidity and ambient storage temperature. The presumed advantages to a two-stage processing method is greater drying efficiency and reduced water activity or microbial growth, in order to increase shelf-life and reduce the disposal costs that are associated with BSG. Pelletizing the feed as an auxiliary step to a 2-stage drying process ultimately reduces transportation costs and improves material handling, and this is why the current study targeted 15% moisture (w.b.) for subsequent densification processing.

2.4.1 Mechanical dewatering

The initial stage of the experiment examined mechanical dewatering to remove free moisture from pressing the grains. Common versions of the technology exist in the industry and include screw press, belt press, screens and centrifuges. Orsat and Raghavan (2008) describe advanced or intensified hybrids such as electro-osmotic belt filters, ultrasonic and vibrations, electro-acoustic and vapor pressure dewatering which can potentially increase yields and improve quality of both the liquid and solid fractions. Orsat and Raghavan (2008) encourage this concept of 'combined field separation' as a promising advanced dewatering technology to improve

efficiency with a single piece of equipment. Then combined with a chemical or biological waste conditioning strategy, filtration can be further improved.

Specific to BSG, Machadeo et al (2016) were successful in dropping the moisture content of BSG from 75% to 15% by employing a hybrid dewatering technology using an integrated membrane filter press with thermal vacuum drying. El-Shafey et al (2004) discovered similar results where a 20-30% moisture level was achieved utilizing a similar hybrid technology. This study found that the cake thickness between the membrane plates was the most important factor in determining drying efficiency. However the current study will examine pressure and time parameters using a laboratory scale press. Optimizing parameters for a press was selected since a press is generally a common and cost-effective piece of equipment, which is readily available.

2.4.2 Hot air convection microwave assisted drying

Microwave-assisted hot air drying has been proven to reduce drying times and increase moisture diffusion in broccoli stalk slices, carrot slices and wheat distiller's grain with solubles (Salim et al., 2016; Hu et al., 2016; Mosqueda and Tabil, 2011). Increasing the initial microwave power density results in a high moisture transfer driving force that should result in shorter drying times (Hu et al., 2016). The initial microwave power density helps to shorten the drying time, however in an example with carrots, temperature greatly influences the drying time (Hu et al., 2016). The effective moisture diffusion coefficient (D_{eff}) increased with inlet air temperature at the same power density, while D_{eff} only increased slightly with a constant temperature and increasing power density (Hu et al., 2016). In this study the D_{eff} was further improved using drying pretreatment solutions such as a 20% sugar solution (Hu et al., 2016). In Salim et al (2016) moisture diffusivity (D_{eff}) under microwave assisted hot air drying increased by double in comparison to hot air alone. As the product temperature increases, (D_{eff}) increases and as reported in both carrots and broccoli, the increase in temperature helps to improve D_{eff} (Salim et al. 2016; Hu et al., 2016).

More specifically, wheat distiller's grain with solubles (WDGS) dried under microwave assisted hot air convection reduced the drying time significantly and interestingly consumed less energy (Mosqueda and Tabil, 2011). Drying time was reduced by about 50% in WDGS, and this reduction in time as a result of microwave assisted drying is similar to other food products under microwave assisted drying (Salim et al., 2016; Mosqueda and Tabil, 2011). In WDGS the quality of the product decreased linearly with temperature (Mosqueda and Tabil, 2011). Quality in this

study was determined by color (L = lightness) and lysine content, where lysine increased with L. Drying air temperature produced a stronger correlation with loss in lysine and increased WDGS darkening more than microwave (Mosqueda and Tabil, 2011). Lysine is a valuable amino acid in feed that is easily susceptible to damage through thermal processing due to the Maillard reaction which binds amino acid groups with the carbonyl compounds of reducing sugars (Mosqueda et al., 2013; Aryee et al., 2017). Therefore, knowledge regarding how temperature and microwave power density affect the final product quality is a worthy area of investigation in order to optimize drying conditions.

2.5 Determining product quality

The quality of the feed will be examined to ensure the grains are not burned from excess heating through determining the acid detergent insoluble protein (ADIP or ADP) content and level of color change. Preserving protein quality is essential, including specific amino acids of interest for animal feed such as lysine, cysteine and tryptophan (Serna-Saldivar, 2010). According to Lynch et al (2006), oven drying is the most suitable method for spent grain preservation; however, it must be conducted at temperatures <60 °C due to unpleasant flavors generated at higher temperatures. Regardless of initial temperatures the exit temperatures of grains in convection ovens must be monitored to reduce this risk of burning grains (Lynch et al., 2006; Mussatto et al., 2006). However, the current study examines temperatures above 60°C since the goal of the study is to determine the optimal, and most efficient drying parameters, to minimize undesirable consequences to grain protein digestibility for ruminants as a result of overheating. In addition, the current study examines whether or not color alterations are observed at higher temperatures, indicating the grains have been significantly burned or toasted. This change in color would presumably correlate with loss in protein digestibility.

2.5.1 Protein assessment and The Cornell Net Carbohydrate and Protein System

The Cornell Net Carbohydrate and Protein System (CNCPS) is a method used to quantify protein quality in feedstuff through a submodel that predicts degradation rates in the rumen, the passage of undegraded feed in the lower gut, and the amount of metabolizable energy (ME) and protein that is available to the animal (Sniffen et al., 1992). From this model, the portion of protein in the feed that is represented as the acid detergent insoluble nitrogen (ADIN), is considered unavailable to the animal since it cannot digest this portion of protein because it has become

irreversibly bound to fiber. Despite the fact that CNCPS methodology is used as an industry standard, Mackacek and Kononoff (2009) argued that ADIN might not be the best indicator for unavailable protein since 58% of ADIN in their study was found to be digestible.

Sniffen et al (1992) states, the ADIN fraction of bound true protein represents protein associated with lignin, tannin-protein complexes, and products from a Maillard reaction that are highly resistant to microbial and mammalian enzymes. Therefore, this fraction is used to measure the severity of heat damage to a feed that may result from high moisture (>80%) due to biological self-heating, or through grain processing conditions. This is measured by the amount of nitrogen (N) associated with the acid detergent fiber (ADF) residue. Crude protein is determined by multiplying nitrogen (N) x 6.25. Some portion of nitrogen is found as non-protein nitrogen and is therefore referred to as crude rather than true protein (Sniffen et al., 1992).

On laboratory feed reports, the acid detergent insoluble nitrogen fraction (ADIN) may be reported as acid detergent insoluble protein (ADIP), acid detergent insoluble crude protein (ADICP), acid detergent protein (ADP) and acid detergent fiber nitrogen (ADF-N). According to the SGS Agri-Food Laboratories (Guelph, Ontario, Canada) laboratory standards, often this portion of protein is subtracted from the feed's total crude protein to estimate available crude protein if bound protein is detected. In this case, bound protein, or overheating in feed, can be surmised if values higher than 12% as a percentage of crude protein are found, since all forages contain a certain percentage of ADP. Schroeder et al (1996) proved that the digestibility of undegradable protein in heat-treated plant proteins did not decrease significantly when ADIN values were below 12-15%, but digestibility progressively decreased when ADIN was above this range. This early study helped draft the industry benchmark where ADIN values above 12% suggest that substantial heat damage has occurred. Therefore in terms of quantifying available protein, this portion of protein is subtracted from crude protein once above 12%. This value is then reported as an adjusted crude protein value. In many reported publications, ADP is monitored and used as an indication to grain overheating as temperature and power density increases (Mosqueda et al., 2011; Mosqueda et al., 2013; Heim and Krebs, 2018; Mustafa, 2001).

Acid detergent fiber (ADF) was monitored which measures cellulose and lignin in the cell wall of the silage. This value is important since it indicates heat damage. As ADF increases, the digestibility of the forage decreases (Sniffen et al., 1992). Previous research had reported that the amount of ADF residue correlates with the amount of insoluble N in feed, suggesting as ADF

increased, the concentrations of ADIN also increased (Van Soest, 1994). However, Mackacek and Kononoff (2009) found a poor correlation (R^2 =0.19) between ADF and ADIN, despite ADIN as a percentage of N rose as ADF increased. Therefore, in the current study ADP was selected to minimize during the drying process and ADF was observed.

2.5.2 Color assessment

Color assessment is a helpful quality parameter to correlate many quality indicators such as nutritional properties (e.g., protein), taste and aroma (Shingare and Thorat, 2013; Salim et al. 2016; Hu et al., 2016; Mosqueda and Tabil, 2011; Mosqueda et al., 2013). The CIE *L*a*b** color coordinate method uses a colorimeter to detect changes along an axis in lightness (*L**), green to redness (*a**) and blue to yellowness (*b**) (Robertson, 1977). As seen in other food products, the change in color (ΔE), or individual color parameters (e.g., *L*, *a*, *b*), is a rapid method to quantify a physical change to the product as a result of a chemical transformation over different experimental treatments (Shingare and Thorat, 2013; Salim et al., 2016; Hu et al., 2016; Mosqueda and Tabil, 2011). Five color systems exist, however *L*, *a*, *b* is one of the most common systems used in food drying (Shingare and Thorat, 2013).

Connecting statement to Chapter 3

In chapter 3 the initial stage of the two-stage drying process was explored. The initial stage was designed to maximize water loss mechanically. Therefore an experimental dewatering examination to maximize water loss as a function of press pressure (MPa) and static holding time (mins) of the lab press was carried out. This chapter describes the statistical design of the experiment employed using holding time and press pressure as predictor variables to define optimal parameter conditions.

Chapter 3: Mechanical dewatering

3.1 Introduction

The first part of the study examined the mechanical dewatering process. Mechanical dewatering to remove free moisture from high moisture organics is a critical pre-drying step to manage drying costs. Mechanical dewatering does not induce a phase change during the water removal process and therefore can reduce costs before introducing a conventional drying method to bring the grains to a safe storage moisture level (Orsat et al., 1996). The efficiency of the initial dewatering phase was determined as a function of holding time and pressure.

A response surface methodology was used to predict the optimal pressure, and length of time to hold this static pressure, in order to remove the greatest amount of water. The amount of water removed from the dewatering stage was determined, followed by a comparison in efficiency for thermal and microwave assisted drying discussed in later chapters.

3.2 Materials and methods

3.2.1 Material characterization

Raw brewers' spent grain was collected from a microbrewery in Orford, Quebec from the same batch, on the same day and stored below 0 °C. The sample was assessed for initial moisture content, crude protein content, acid detergent fiber and acid detergent insoluble protein. All samples post drying treatment were analyzed for crude protein, acid detergent fiber and acid detergent insoluble protein. A complete feed quality assessment was performed on the sample dried with hot air at 60 °C. This analysis reported the protein, fiber, fat, starch, minerals and energy value of the brewers' spent grains used in the current study.

As-received moisture content

Moisture content of the spent grains was determined by drying 15g sample over 3 hours at 105 ºC (Shreve et al., 2006) using an oven with forced hot air. The samples were measured in triplicates. Moisture content was calculated on wet basis:

$$M.C.(wb,\%) = \frac{(wet\ mass - dry\ mass)}{wet\ mass}\ x\ 100 \quad \textit{Equation 1}$$

3.2.2 Dewatering equipment

The experimental setup for dewatering the grains comprised of an Instron Universal Testing Machine equipped with a compression cell, which was used to compress the spent grain samples (Figure 2). The compression cell had a diameter of 22 mm and a length of 59 mm. An electronic balance measured the amount of water expelled from the samples.

3.2.3 Procedure

A 16 gram spent grain sample size was used to completely fill the cell prior to pressing the material. A steady load of 0.3, 0.9 or 1.5 MPa with a holding time of 0, 2.5 or 5 minutes was controlled by the testing machine's software. Each combination of load treatment levels (i.e., 0.3, 0.9, and 1.5 MPa) was tested in order to measure the amount of water removed from the sample governed by holding time and pressure.

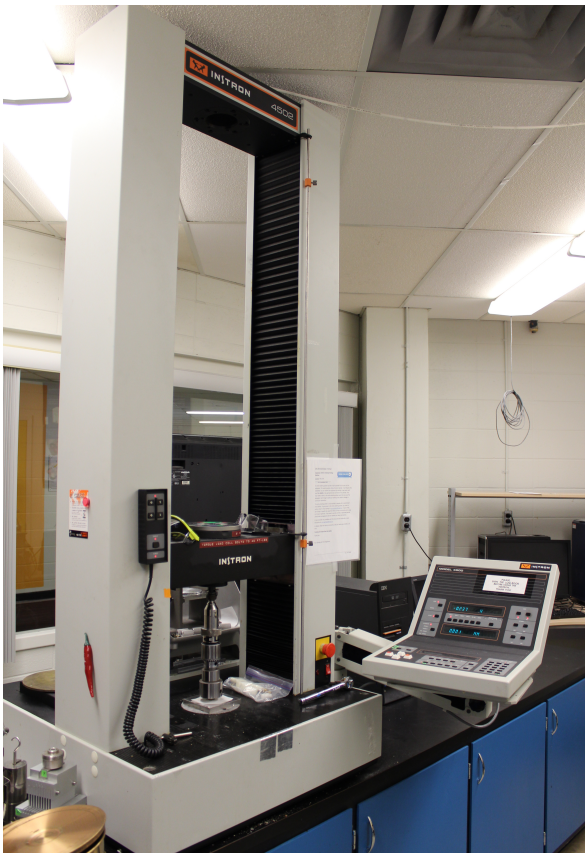


Figure 2. Instron Universal testing machine used to compress grains.

3.2.4 Statistical design

The experiment was designed to assess the effects of pressure and holding time on dewatering the grains in order to find the optimal conditions, with respect to these treatments, to yield the maximum amount of water removal. Therefore a central composite design (CCD) having uniform precision with a centered composite face, response surface analysis was chosen. The experiment included two factor combinations at three levels (Table 2), with the central point parameters of 0.9MPa and 2.5 minute holding time repeated 5 times. The advantage to a central composite design is the reduction in experimental units (13) in comparison to a full factorial design with triplicates (27) (Panneton et al., 1999). The trials were performed at room temperature and the statistical analysis was performed with JMP® Pro 13.2.0 (SAS Institute Inc., Cary, NC, USA).

Table 2. Description of the experimental design.

Factors	Levels	Description
Pressure	1	0.2 MPa
	2	0.3 MPa
	3	0.4 MPa
Holding Time	1	0 min
	2	2.5 min
	3	5 min

3.3 Results

3.3.1 Response surface analysis and model fitness

A central composite design (CCD) response surface methodology (RSM) was carried out to find the optimal parameter settings to maximize water loss for a brewers' spent grain dewatering application. Therefore the variance between modeled and observed values used to optimize a water loss response function as a result of holding time and pressure was determined. The model's significant probability value (p = <0.0001) from the analysis of variance (ANOVA) demonstrated in Table 3 that the model itself can accurately predict the holding time and pressure variables.

Table 3. Model analysis of variance (ANOVA) results.

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	650.15	130.03	61.96
Error	7	14.69	2.10	**Prob > F**
C. Total	12	664.84		<.0001

Figure 3 depicts the leverage plot for linear effect in a simple regression analysis, which illustrates that the modeled values for the water loss response from the predictor variables (i.e., holding time and pressure) significantly fit the model (p = <0.0001) and that the data fits the regression model well (R^2 = 0.98), where 98% of the response variability can be explained by the model.

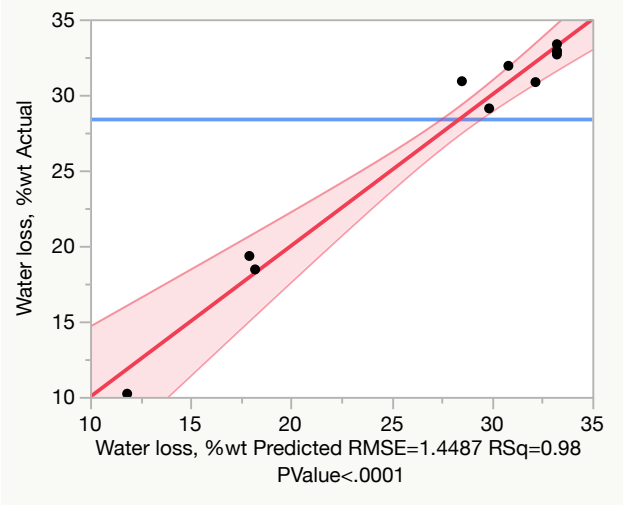


Figure 3. Actual versus predicted plot for water loss.

However, the linear quadratic model was inadequate and did not fit the observed data well for predicting water loss since the model was highly significant according to the lack of fit test (p = 0.0012) (Table 4). The variance from the lack of fit (MS = 4.77) was significantly higher than variance from pure error (MS = 0.09), which suggests a significant bias in the results.

Table 4. Lack of fit report.

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	3	14.32	4.77	52.09
Pure Error	4	0.37	0.09	**Prob > F**
Total Error	7	14.69		0.0012
				Max RSq
				0.9994

Therefore the model effects were studied further in order to improve predicted water loss as a function of holding time and pressure. There were no highly insignificant effects ($p > 0.10$) to remove and reduce the model (Table 5) and therefore the residuals in the model were examined using the residual by predicted plot (Figure 4) to assess whether the residuals reflected independent, homoscedastic and normally distributed variance.

Table 5. Effect test summary report.

Source	PValue
Pressure, MPa (0.3,1.5)	0.0000
Pressure, MPa*Pressure, MPa	0.0001
Holding Time, min*Holding Time, min	0.0127
Holding Time, min (0,5)	0.0174
Pressure, MPa*Holding Time, min	0.1041

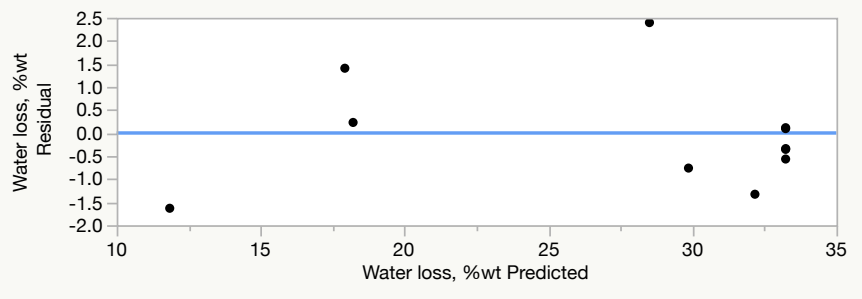


Figure 4. Residuals by predicted plot for water loss.

After removing two outliers from the residual plot, the lack of fit test was insignificant ($p = 0.1186$), which improved the model's adequacy to predict the observations (Table 6).

Table 6. Lack of fitness report with outlier residuals removed.

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	1	0.36	0.36	3.93
Pure Error	4	0.37	0.09	**Prob > F**
Total Error	5	0.73		0.1186
				Max RSq
				0.9994

Prior to removing outliers from the model analysis, the effects of model parameters and their interactions was determined with an ANOVA analysis (Table 7). All linear and quadratic effects were statistically significant ($p < 0.05$). However, the interaction between pressure and holding time was insignificant ($p = 0.1041$). In addition, pressure ($p = <0.0001$), as a predictor variable, was more statistically significant than holding time ($p = 0.0174$) according to the ANOVA analysis (Table 7). However once the outliers were removed, all model main effects and their interactions were significant ($p = <0.001$) (Table 8).

Table 7. Water loss ANOVA analysis for model effects and their interactions.

Source	DF	Sum of Squares	F Ratio	Prob > F
Pressure, MPa (0.3,1.5)	1	351.75	167.60	<.0001
Holding Time, min (0,5)	1	20.13	9.59	0.0174
Pressure, MPa*Holding Time, min	1	7.32	3.49	0.1041
Pressure, MPa*Pressure, MPa	1	161.76	77.08	<.0001
Holding Time, min*Holding Time, min	1	23.20	11.05	0.0127

Table 8. Water loss ANOVA analysis for model effects and their interactions with residual outliers removed.

Source	DF	Sum of Squares	F Ratio	Prob > F
Pressure, MPa (0.3,1.5)	1	161.45	1111.01	<.0001
Holding Time, min (0,5)	1	8.93	61.42	0.0005
Pressure, MPa*Holding Time, min	1	9.05	62.25	0.0005
Pressure, MPa*Pressure, MPa	1	83.01	571.24	<.0001
Holding Time, min*Holding Time, min	1	36.99	254.58	<.0001

Finally, through employing the model, including outliers, the predicted value at the optimal conditions of 1.19 MPa and 3 minutes holding time resulted in 35.27 % water loss (Figure 5).

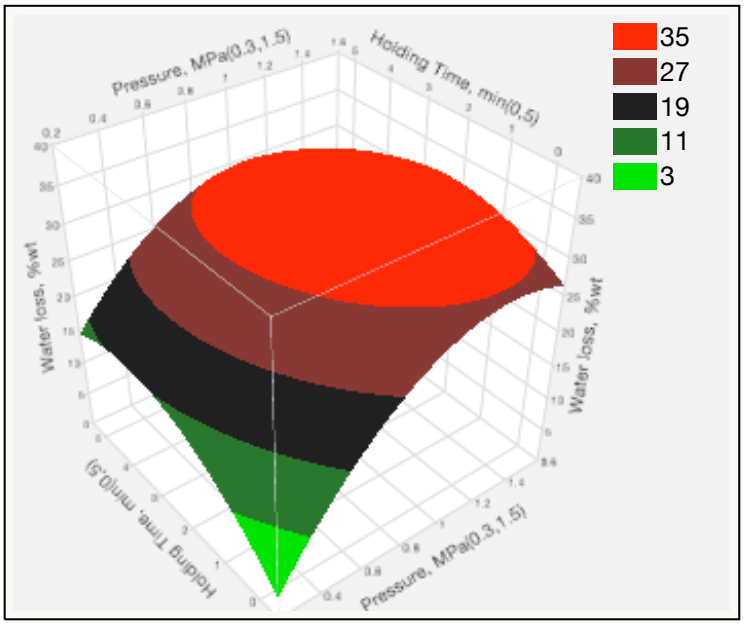


Figure 5. Water loss surface plot illustrating optimal parameter settings in order to maximize water loss resulting in 35%.

The resulting model equation that was used to predict the water loss response:

$$\%\,Water\ loss = 33.07 + 7.05 * ((\text{"}Pressure, MPa\text{"}) - 0.9) / 0.6) + 1.92 * (((\text{"}Holding\ Time, min\text{"}) - 2.5) / 2.5) + (((\text{"}Pressure, MPa\text{"}) - 0.9) / 0.6) * (((\text{"}Holding\ Time, min\text{"}) - 2.5) / 2.5) * -2.27) + ((((\text{"}Pressure, MPa\text{"}) - 0.9) / 0.6) * ((((\text{"}Pressure, MPa\text{"}) - 0.9) / 0.6) * -7.09) + (((\text{"}Holding\ Time, min\text{"}) - 2.5) / 2.5) * (((\text{"}Holding\ Time, min\text{"}) - 2.5) / 2.5) * -4.41)$$

Equation 2

3.4 Discussion

The initial mechanical dewatering stage of spent grains offers a cost-effective method to remove free moisture in order to optimize the drying conditions as described in previous research (Orsat et al., 1996). Therefore the parameters of the dewatering process were measured to maximize water loss and ultimately improve the efficiency for the entire two-stage drying process. Two factors were assessed: pressure and holding time. Holding time was examined in the lab as a method to quantify the effect of processing time on water loss. This analysis was used to mimic screw speed, which is a common parameter to optimize using a dewatering screw presses technique. For example, Pérez-Gálvez et al. (2009) found both pressure and screw speed to have a statistically significant ($p < 0.05$) effect on water loss in seafood. Although, this study reports greater water loss with lower screw speeds, higher screw speeds resulted in lower organic load in the press liquor, which was considered a positive liquor quality.

The water loss predictive model designed in the current study was statistically significant, however a lack-of-fit for the experimental versus predicted data was found. Therefore to improve and investigate the causes for the model lack-of-fit, the model was dissected to examine model main effects, interactions and data point outliers. From the original model, the interaction between pressure and holding time was removed since it was insignificant ($p > 0.05$), however removing the interaction from the model did not alter the critical values substantially for process optimization (Table 9). Without the interaction, the critical value for pressure was 1.19 MPa and the holding time increased slightly to 3:29 minutes, resulting in 35.49% water loss under these conditions. The interaction between pressure and holding time was removed from the model, in addition to the main effect pressure, holding time was insignificant ($p = 0.5535$). Therefore independently, pressure has a significant effect on water loss, but not holding time. Consequently the model should be refined to increase the number of intervals for holding time, or reduce the distance between holding time intervals, to determine whether or not holding time should be included in the model if these results are considered to design a dewatering system. A press with vacuum drying may be considered since water loss efficiency has been shown to increase to 59 % water removal using a filter press and membrane planes with vacuum drying (Machado et al., 2016). Other variables, such as temperature, sample size bed thickness, particle size or grain type could strengthen the model to find optimal processing conditions, and across different types of grains.

3.5 Conclusion

Despite the model's inadequacy to fit experimental data, a solution with a maximum was found (Table 9). The predicted value at the optimal conditions of 1.19 MPa and 3 minutes holding time resulted in 35.27 % water loss, reducing the grains from 70 %MC w.b. to 60 %MC w.b. (Figure 5). However, the critical value for pressure is most relevant since the holding time as a variable is insignificant and the interaction between pressure and holding time was insignificant. After removing the outliers from the original model, the optimal parameters only changed slightly (Table 10) where holding time was reduced from 3 minutes to 2:44. Therefore, it can be estimated that 35 % water loss can be achieved with 1.19 MPa pressure and 3 minute holding time. Holding time can be further optimized and adequately modeled by shortening the time intervals to determine whether or not time can have a significant affect, however 3 minutes was concluded under the current experimental conditions.

Table 9. Water loss solution maximum report. Results are from the response surface analysis.

Variable	Critical Value
Pressure, MPa (0.3,1.5)	1.19
Holding Time, min (0,5)	3.01

Table 10. Water loss solution maximum report. Results are from response surface analysis with outliers removed.

Variable	Critical Value
Pressure, MPa (0.3,1.5)	1.19
Holding Time, min (0,5)	2.73

Connection statement to Chapter 4

In Chapter 4 the second stage of the drying process is examined. The second stage will assess drying efficiency as a function of drying temperature and microwave power density. This chapter describes the statistical design of the experiment employed where processing conditions are optimized to predict the most efficient drying parameters.

Chapter 4: Convective versus convective microwave assisted drying efficiency

4.1 Introduction

Drying efficiency as a function of temperature and microwave power was examined during forced hot air convection drying and forced hot air convection microwave assisted drying. The air temperature settings to perform the experiments were set at 60 ºC, 75 ºC and 90 ºC. The microwave power density was set to 0, 1 or 2 W g^{-1}. The purpose of performing these drying tests was to examine the drying kinetics of the grains under said conditions in order to create predictive models for each drying treatment. Then subsequently model the optimal drying conditions in order to preserve digestible protein levels in the sample. Protein under thermal processing is susceptible to heat damage, reducing protein solubility (Guerrieri & Cavaletto 2018), justifying the need to monitor grain thermal conditioning.

4.2 Materials and methods

4.2.1 Material characterization

Brewer's spent grains as described in Section 3.2.1 were used for thermal drying treatments. Moisture content was determined prior following the same protocol described in 3.2.1 and quantified using Equation 1.

4.2.2 Hot air convection and microwave drying equipment

Drying test trials were performed using an automated microwave-assisted laboratory dryer comprised of a 2450 MHz microwave generator, air blower connected to the bottom of the microwave chamber and a fiber optic temperature sensor to automatically record the sample temperature (Figure 6). The setup is equipped with a data logger weighing system. The microwave generator included three manual tuning screws for adjusting and minimizing the reflected power. The dimension of the microwave cavity of the dryer setup is roughly 47 x 47 x 27cm.

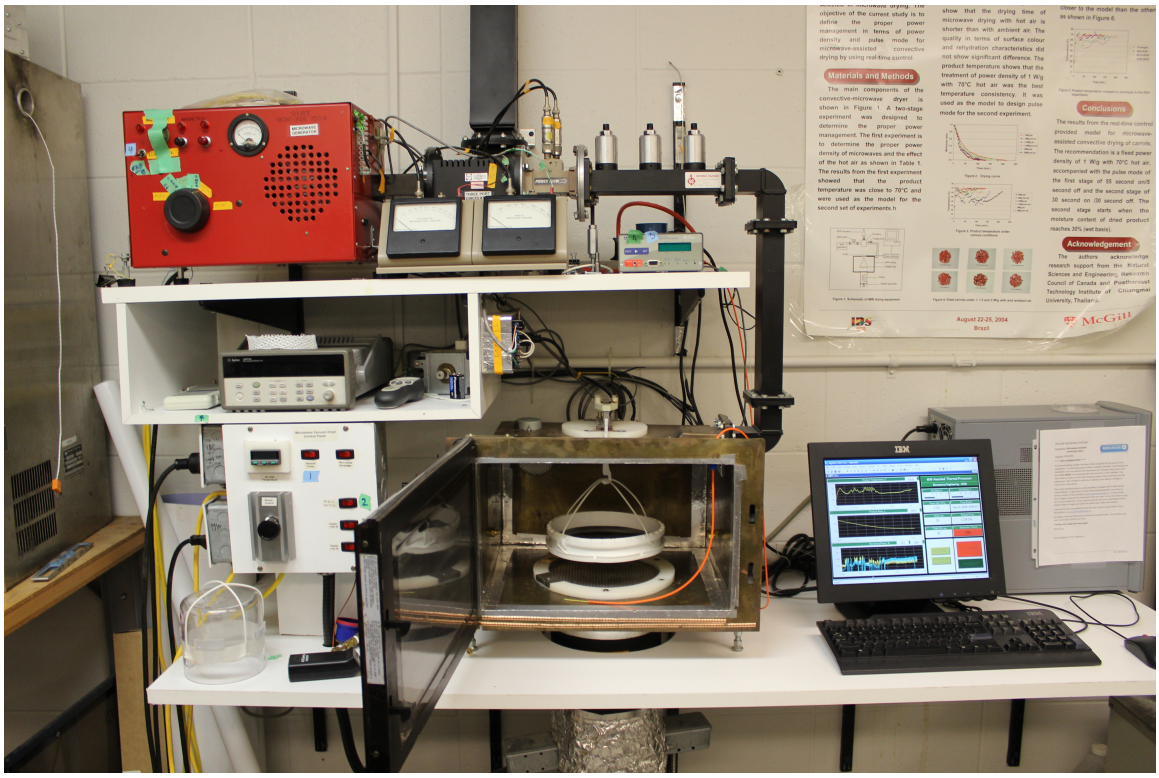


Figure 6. Experimental dryer setup.

4.2.3 Experimental drying procedure

In each trial 150-100 grams of sample was evenly spread on the sample tray and hung inside the dryer chamber (Figure 7). Sample size varied due to a limited supply of sample after multiple trials. The sample was placed above the center where the hot air entered the chamber. Air supplied by the blower was heated to temperatures 60 ºC, 75 ºC and 90 ºC depending on the trial treatment. The air was supplied at an estimated velocity of 1.4 m s^{-1}. The microwave power was supplied from 0 to 200 W depending on the targeted power density. The power density for each hot air drying test included one hot air drying treatment without microwave assistance and two drying test trials with microwave assistance. The microwave power was manually adjusted to maintain an accurate microwave power to weight ratio as moisture loss increased. For each temperature setting, one microwave-assisted trial was carried out with a power density of 1 W g^{-1}, which varied throughout the experiment between 0.8-1.4 W g^{-1}, while the second trial was carried out with 2 W g^{-1}, which was considered between1.5 – 2.4 W g^{-1}.

Figure 7. Dryer sample tray with brewers' spent grain sample.

The temperature of the samples was monitored and automatically recorded via a fiber optic temperature sensor, and sample mass of the sample was recorded through a data acquisition system (Hewlett-Packard, USA). Samples for all treatments were dried until the sample reached 15% w.b. All drying treatments were performed in duplicates to ensure ≤15% deviation.

4.3.4 Mathematical modeling of drying curves

The experimental drying data collected is applied to calculate the moisture ratio. Sample weight is recorded on 30-second intervals for all experimental conditions. The resulting moisture ratio of the grains over time is used to model the drying kinetics of the grains. The moisture ratio (MR) was calculated using the following equation:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad \textit{Equation 3}$$

where MR is the moisture ratio, M_t is the instantaneous moisture content (g water/ g dry matter), M_0 is the initial moisture content and M*e* the equilibrium moisture content. The equation was simplified for use in the current study:

$$MR = \frac{M_t}{M_0} \quad \textit{Equation 4}$$

The drying rate (g water/ minute) of the spent grains as a function of drying treatments was calculated using the formula:

$$\frac{dM}{dt} = \frac{M_{t+dt} - M_t}{dt} \quad \textit{Equation 5}$$

where t is the drying time (min), M_t and M_{t+dt} are the moisture content at t and t + dt (g water/ g dry matter).

In order to predict the drying time to a final moisture content 15% w.b., the resulting moisture ratio was analyzed using different non-linear regression models in Curve Expert Professional (Hymans Development, ver. 2.6.5). A suitable drying model was selected based on a correlation coefficient (R^2) equal to or greater than 0.99, demonstrating a high correlation with the experimental data.

4.4 Results

4.4.1 Drying curves

Under thermal drying conditions, the moisture ratio at all temperatures with and without microwave was plotted over time. Figure 8 illustrates a trend where increased temperature with microwave reduced the time to reach the targeted final moisture content. During the drying constant rate, a sharp drop in moisture content using microwave assisted drying was observed and resulted in a reduced time for the falling rate period in comparison to convection forced hot air only samples (Figure 8).

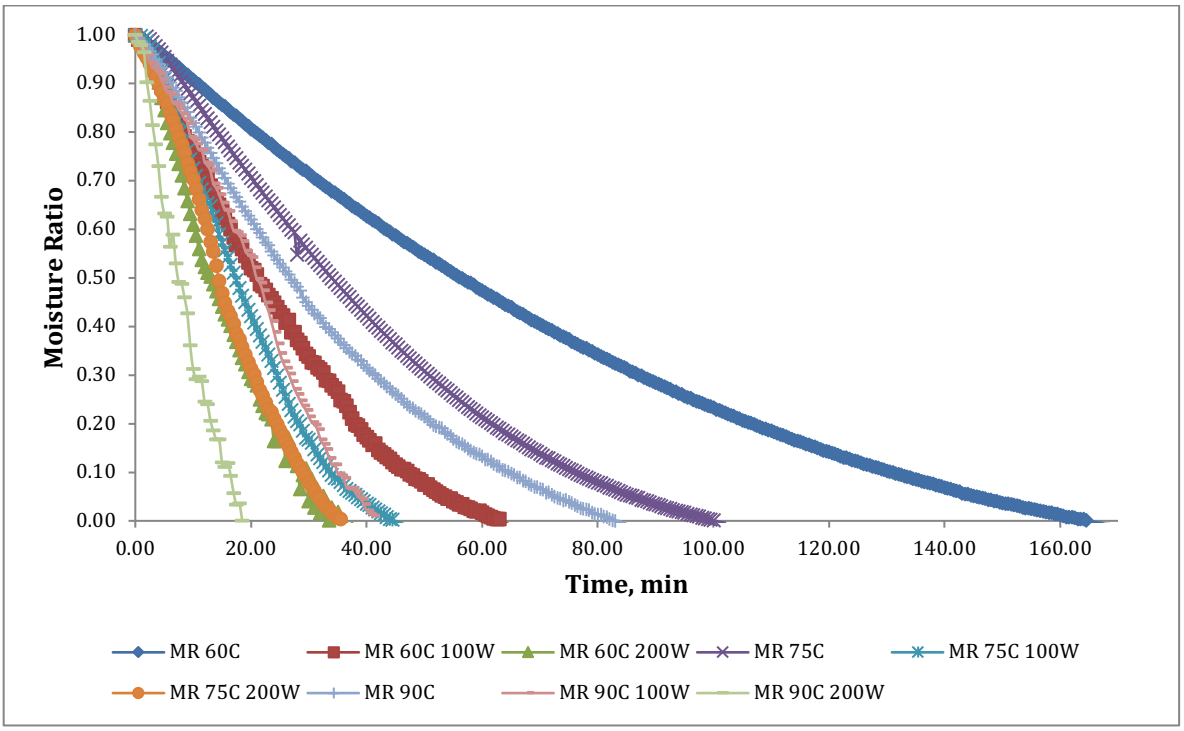
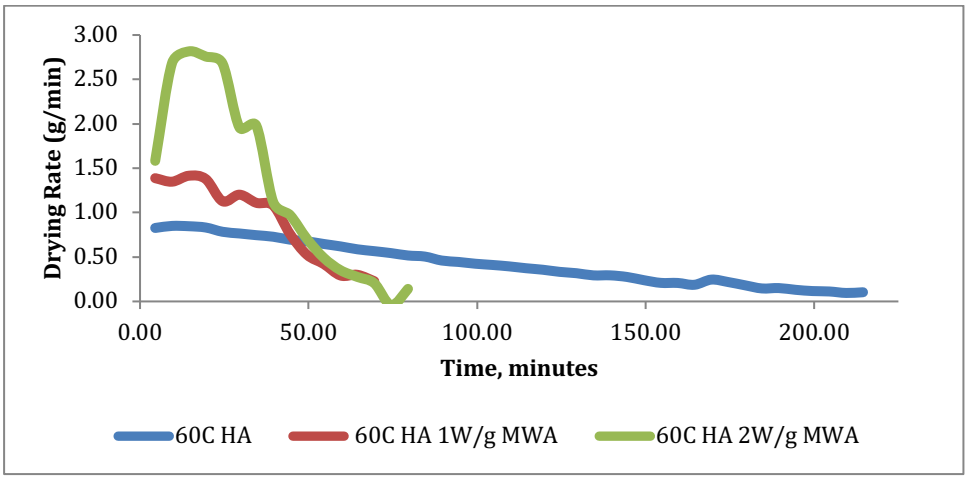


Figure 8. Moisture ratio hot air convection versus microwave-assisted convection.

The drying rate (g water/ minute) for forced hot air convection and forced hot air microwave assisted are illustrated in Figure 9. The drying rate for forced hot air convection is a longer and steadier process when compared to microwave assisted due to a characteristic falling rate. Water loss during the constant drying rate is nearly tripled with a power density of 2 (W g^{-1}) and reduces or eliminates the falling drying rate.

a.



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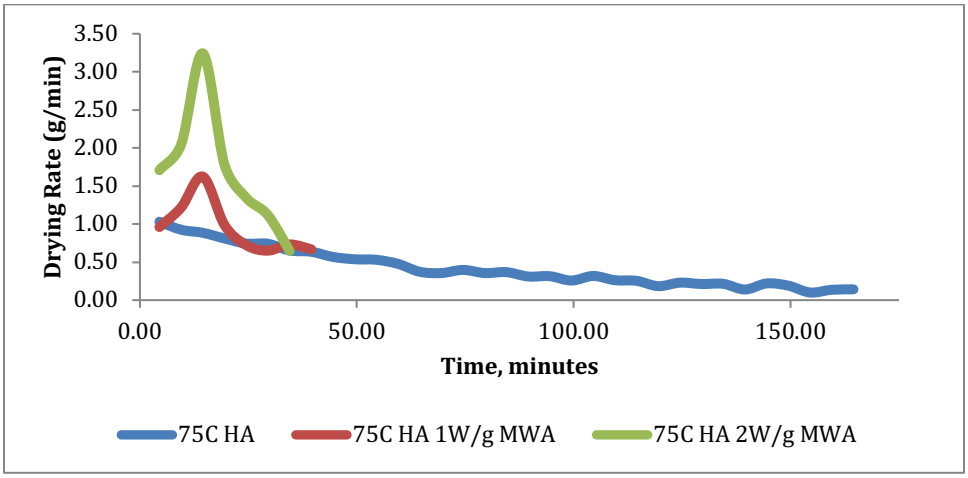

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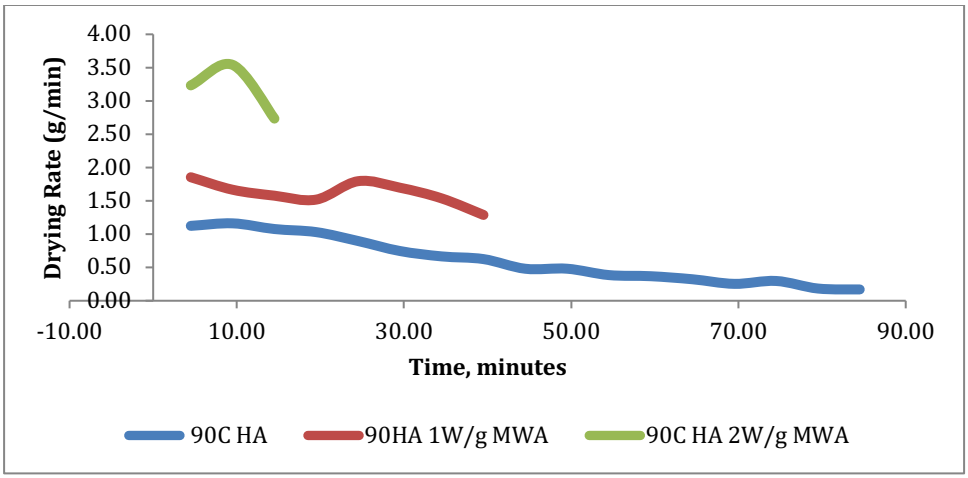


Figure 9. Drying rates for hot air convection and microwave-assisted convection. Presented are the drying rates at different temperatures (60, 75, 90°C) and power densities (0, 1, 2 W g^{-1}). The drying rate nearly tripled with a 2 W g^{-1} power density and reduced the drying time substantially.

4.4.2 Drying model

The resulting moisture ratio (MR) drying curves (Figure 8) for all dryer parameter scenarios were fitted to a non-linear regression model using Curve Expert Professional® (Hymans Development, USA, ver. 2.6.5) in order to predict the length of time to dry samples to 15% MC (w.b.). The rational function supported all model forms and is written as followed:

$$MR = \frac{a+b*T}{1+c*T+d*T^2} \quad \textit{Equation 6}$$

where MR refers to the moisture ratio; T is the drying time (minutes); and a, b, c and d are the model coefficients in the regression model. The coefficient of determination (R^2) values from the prediction model were all $p > 0.990$ for the drying parameter coefficients, indicating that the experimental data fit the regression model well (Table 11).

Table 11. Drying time non-linear regression model coefficients.

Air Temperature (°C)	Microwave Power Density (W g^{-1})	Model Coefficients					
		a	b	c	d	RMSE	R^2
60ºC	0	1.00	-0.01	0.00	0.00	0.00	0.9999
	1	0.99	-0.02	0.00	0.00	0.01	0.9991
	2	1.02	-0.03	-0.00	0.00	0.02	0.9976
75ºC	0	1.03	-0.01	0.00	0.00	0.00	0.9998
	1	1.00	-0.02	-0.01	0.00	0.00	0.9994
	2	1.04	-0.03	-0.00	0.00	0.01	0.9989
90ºC	0	1.01	-0.01	0.01	0.00	0.02	0.9935
	1	0.98	-0.02	-0.01	0.00	0.01	0.9984
	2	1.03	-0.05	0.02	0.00	0.02	0.9944

The regression model selected as a result of the experimental data, predicted the drying times in Figure 10 to reach 15% MC (w.b.). Drying time to reach 15% moisture (w.b.) was targeted for further processing applications such as densification (i.e. pelletizing), however <11% moisture (w.b.) is recommended for safe grain storage as per the Canadian Grain Commission (CGC, 2009).

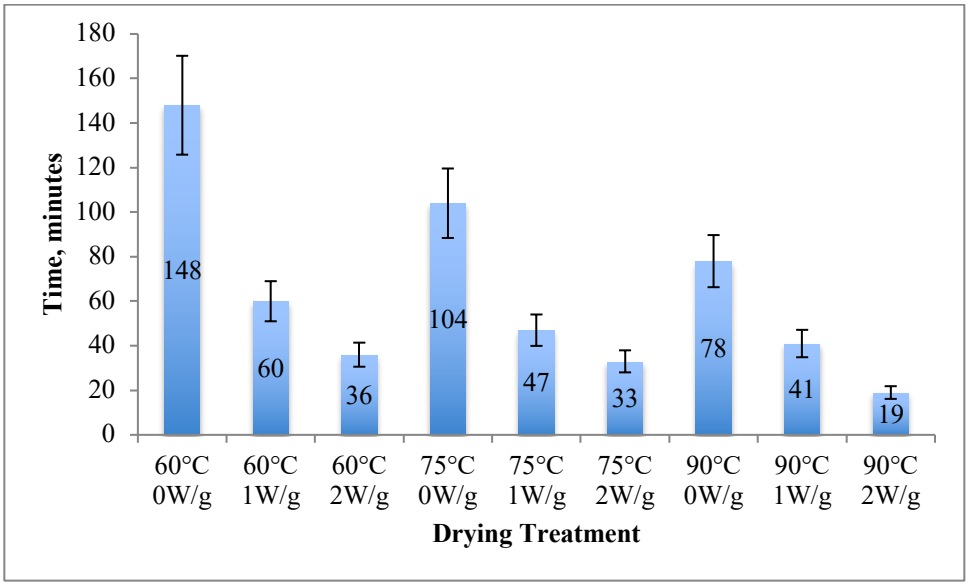


Figure 10. Predicted drying times resulting from the regression model to reach 15% MC w.b.

4.5 Discussion

Drying curves

As expected, increases in microwave power density and temperature improved drying efficiency (Figure 8). Similar to the current study, the increase in inlet temperature and microwave power density is known to enhance moisture diffusion rates in other food products such as carrot slices (Hu et al., 2016). The drying rate (g/min) increased nearly 3 fold during the constant rate with a 2 W g^{-1} power density setting (Figure 9) and the falling rate was eliminated or reduced significantly compared to no microwave assistance. A 60 °C inlet temperature lacking microwave assistance resulted in the longest drying time to reach 15% MC (w.b.) with 148 ± 24 minutes. This drying curve demonstrates a typical falling rate, which exceeds the constant rate and a tailing drying curve. With an inlet temperature of 90 ºC and microwave power density setting 2 W g^{-1}, moisture loss to reach 15% MC (w.b.) was most rapid at 19 ± 8 minutes with no falling rate.

Microwave-assisted convection drying reduced the drying time by 40-60% in all cases for a final moisture of 15% (w.b.). Salim et al (2016) reported similar results where drying time was reduced by 55% with microwave-assisted hot air (MWAHA) compared to hot air alone for drying broccoli stalk slices. With regards to hot air convection only tests, the air temperature alone reduced the drying time by 30% with 75 ºC and 47% with 90 ºC respectively, when compared to the drying time for 60 ºC hot air convection. The greatest margin in loss for drying time was 59% and observed for 60 ºC hot air convection with a power density setting of 1 W g^{-1} compared to no microwave assistance at 60 °C. As anticipated, 90 ºC hot air convection with a power density setting of 2 W g^{-1} reported the most rapid drying time compared to the other temperature and microwave assisted dryer settings.

Drying model

The drying curves fit the rational model best ($R^2 > 0.99$) which is a nonlinear regression model from Curve Expert Professional® (Hymans Development, USA, ver. 2.6.5). This model proved to be an adequate model for predicting drying times due to the high coefficient of correlation ($R^2 > 0.99$) and low root mean square error (RMSE < 0.10) for all drying treatments (Table 11). The drying rate constant *b* in this model was highest and particularly for 90 °C + 2 W^{-1} at -0.05/min. Model constant *b* was similar for each temperature however it increased as

microwave power density increased, which demonstrates that increasing power density improved the drying rate.

In general the most prominent theoretical drying model used in drying applications is Fick's (*k*) second law of diffusion. However semi-theoretical thin layer drying models such as the Lewis model and Page's model are easier to use due to temperature, relative humidity, air velocity and moisture content range for which they were developed (Mohapatra and Rao, 2015). The current study does not compare the rational model to standard semi-theoretical thin layer drying models, however comparison of models could determine whether standard models can be applied to the BSG drying treatments used in the present study. Although as previously reported with dried distiller's grain with solubles (DDGS), Page's model described the drying behavior best (Mosqueda and Tabil, 2011).

4.6 Conclusion

Experimental data was collected for drying temperatures at 60 °C, 75 °C and 90 °C with microwave assisted power densities 0 Wg^{-1}, 1 Wg^{-1} and 2 Wg^{-1}. Moisture ratio curves were generated for identifying an empirical drying model to predict drying times as a function of drying treatments. The rational model could adequately model drying times since experimental data fit the modeled data well (R^2 = >0.99). Increasing air temperature and microwave power density improved drying efficiency. For all air temperatures, the addition of 2 Wg^{-1} microwave improved drying efficiency by 68-76%. Temperature alone improved drying efficiency by 47% from 60 °C to 90 °C. The most efficient drying method resulted in a drying time of 19 minutes to reach 15% MC (w.b.) and was with treatment 90°C + 2 Wg^{-1}.

Connecting statement to Chapter 5

In Chapter 5 the grain quality is maximized under the most efficient drying conditions. Quality parameters assessed for the grains include soluble protein content, lightness (L^*) and acid detergent fiber. The statistical design of the experiment is described to predict optimal results from a new temperature and microwave power density setting, which considers quality. In addition, a complete feed analysis is provided to compare the current grains analyzed against brewers' spent grains previously reported.

Chapter 5: Product quality

5.1 Introduction: color and protein

A color analysis was used to determine product quality and analyze whether the material darkens as heat is applied, which would suggest material burning or browning under thermal treatments. In addition to color, ADP was analyzed to determine if the protein availability is reduced as a result of the thermal treatments since elevated values in ADP signify heat damage to the grain (Sniffen et al., 1992). It was hypothesized that as temperature increases, browning (darkness) increases, and therefore protein availability decreases. However the objective was to maintain product quality while minimize drying time, and therefore increasing the power density with microwave assisted drying would decrease the drying time while reducing the negative effect of reducing protein availability by means of overheating the grains. Therefore a central composite design response surface analysis was employed to find the optimal conditions to reduce drying time without compromising protein availability.

5.2 Materials and methods

5.2.1 Color tests

Color tests were performed in order to detect any change in color as a function of thermal drying treatments to the spent grains. Furthermore, any correlation between color and protein alteration as a result of thermal treatments was investigated. If a correlation is proven, then color testing could be used as a low-cost, rapid and non-invasive test method.

Color characteristics were evaluated using the CIE L*a*b* color coordinate method (Robertson, 1977). The CIE L*a*b* was designed by the "Commission Internationale de l'éclairagé" (CIE) and describes all colors visible to the human eye and it was created to serve as a reference independent model. Each color coordinate value represents a specific color and functions as followed:

L* = lightness, 0 = black and 100 = white

a* = green to red, negative values = green and positive values = red

b* = blue to yellow, negative = blue and positive = yellow

The measurements were performed using a chroma meter (CR-300X, Minolta Camera Co. Ltd., Japan) with a pulsed xenon arc lamp inside the mixing chamber to provide diffused, uniform

lighting over the 8 mm diameter specimen measurement area (Figure 11). The instrument was calibrated prior to use with the CR-A43 plate.

*Figure 11. Grain color determination. The CIE color method was used to determine L*a*b* color coordinates.*

Two subsamples were extracted from each sample and triplicates were performed on each subsample. A total of 54 measurements were considered in the evaluation. The total color change (ΔE) between L^*, a^* and b^* was calculated from averaged color values and using the sample dried at 60 °C as the baseline reference for comparison. The Hunter-Scotfield equation was used:

$$\Delta E = \sqrt{(\Delta a)^2 + (\Delta b)^2 + (\Delta L)^2} \quad \textit{Equation 7}$$

where $\Delta a = a^* - a_0^*$; $\Delta b = b^* - b_0^*$ and $\Delta L = L^* - L_0^*$, the subscript 0 indicates the sample dried with an air temperature of 60 °C.

5.2.2 Chemical analysis and protein testing

SGS Canada Inc. Agriculture and Food Laboratory in Guelph, Ontario was selected to carry out the brewers' spent grains characterization and determine protein levels (Appendix A). Due to lack of untreated sample available for a baseline control characterization test, the sample dried at 60 ºC hot air convection was used for the complete characterization since 60 ºC is low enough not to alter the composition of the grains (Santos et al., 2003; Aboltins and Palabinski, 2015). This sample was analyzed using a net carbohydrate-protein system (NCPS) nutrition analysis. The

NCPS test uses near-infrared (NIR) and wet chemistry techniques to determine dry matter, crude protein, soluble protein, acid-detergent fiber, neutral detergent fiber, lignin, fat, ash, starch, minerals (Ca, P, K, Mg, Na, Cu, Mn, Zn, Fe) and calculated values such as total digestible nutrients (TDN) (Table 15). It should be noted that the complete brewery grain composition characterization may vary with grain type (barley, rice, sorghum, maize) used in the brewing process, time of grain harvest, types of adjuncts added in the brewing process and brewing techniques (Muthusamy, 2014; Mussatto et al., 2004; Lynch et al., 2016). All treated samples (9) and untreated control (1) were tested for dry matter (%), moisture (%), crude protein (N x 6.25) (%), acid detergent fiber as % crude protein (ADF-CP or ADP) (%), and acid detergent fiber (ADF) (%) as part of the experimental design.

5.2.3 Statistical Analysis

Drying time, color and percentage of unbound protein responses were examined as a function of air temperature and microwave power during the drying process. A central composite design was followed for the experimental design and included 2 factors at 3 levels (Table 12).

Table 12. Description of the experimental design.

Factors	Levels	Description
Temperature	1	60 °C
	2	75 °C
	3	90 °C
Microwave Power Density	1	0 W g^{-1}
	2	1 W g^{-1}
	3	2 W g^{-1}

The experimental design was organized to find the optimal drying conditions to reduce the drying time and yield optimal quality characteristics. Optimal quality characteristics were increased lightness (L^*) and a decrease in the percentage of unbound proteins (ADP). Therefore a central composite design (CCD) was followed which included 10 experimental units where the central point (75°C, 1 W g^{-1}) was repeated twice. All trials were performed at room temperature and the statistical analysis was performed with the software JMP® Pro 13.2.0 (SAS Institute Inc., Cary, NC, USA).

5.3 Results

Two parameters were examined post drying to conclude on product quality as a result of drying conditions. First, color change was examined, and specifically lightness (L^*) in order to determine if the material was visibly browning or burning. Following a color analysis, protein availability in the form of ADF-CP (ADP) was determined.

5.3.1 Color Test

Averaged color test results for each color coordinate was recorded from 2 subsamples within each sample, and measurements were performed in triplicates (Table 13). The change in color (ΔE) compared to the sample dried at 60 ºC varied from 0.20 - 5.32 and is reported in Table 13. Lightness (L^*) varied from 34.90 – 41.19. The sample 90 ºC hot air + 1 W g^{-1} power density saw the greatest color change (ΔE) and was also the darkest sample (L^* = 34.90). Darkness increased as temperature and power density increased, except for 60 °C in this case, where the lightest sample was reported for 60ºC + 2W g^{-1} power density (Table 13).

*Table 13. CIE L*a*b* color coordinate and color change (ΔE). Results were recorded as a function of air temperature (°C) and microwave power density (W g^{-1}).*

Air Temperature (ºC)	Microwave Power Density (W g^{-1})	CIE L*a*b* Coordinates			
		L*	a*	b*	Δ*E*
60	0	39.41 ± 2.42	5.81 ± 0.55	20.79 ± 1.25	NA
60	1	39.34 ± 0.93	5.37 ± 0.28	19.44 ± 0.40	1.41
60	2	41.12 ± 2.71	5.43 ± 0.41	20.22 ± 1.07	1.85
75	0	41.19 ± 2.53	5.19 ± 0.37	20.69 ± 0.75	1.89
75	1	40.16 ± 3.01	5.60 ± 0.51	20.63 ± 0.85	0.80
75	2	39.46 ± 2.91	6.00 ± 0.51	20.82 ± 1.00	0.20
90	0	38.95 ± 3.02	5.50 ± 0.36	20.14 ± 0.81	0.85
90	1	34.90 ± 2.76	5.00 ± 0.67	18.08 ± 1.81	5.32
90	2	37.98 ± 3.12	6.05 ± 0.85	20.58 ± 0.96	1.46

5.3.2 Regression model and model fitness

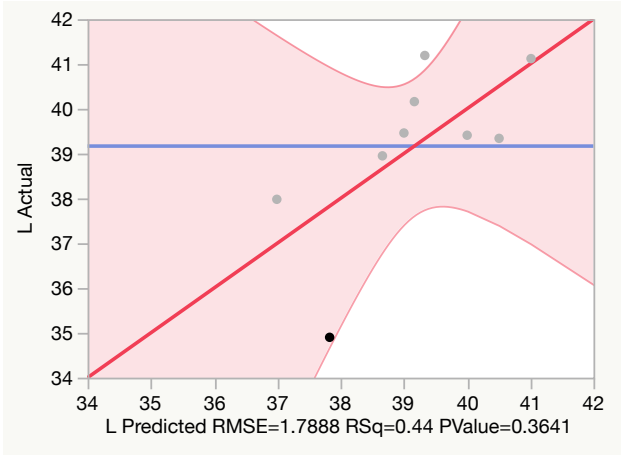
A central composite design with 2 factors having 3 levels was carried out to determine the significance of the main effects (i.e., air temperature and microwave power) and their interaction, using a regression model analysis and ANOVA to find any linear dependencies for these two main effects independently and as an interaction on grain color.

All model effects were highly statistically insignificant ($p > 0.10$) and their respective p-values are listed in Table 14. The effect of drying temperature on color was statistically insignificant ($p = 0.1260$). Removing microwave watts from the model did not result in drying temperature to be statistically significant alone, however its' significance in the model did improve ($p = 0.0796$). The regression model for each color response resulted in a poor fit and the ANOVA was statistically insignificant ($R^2 < 0.999$; $p > 0.001$) (Table 14; Figure 12). It can be summarized that neither drying temperature nor microwave power had a significant effect on grain color throughout the drying process.

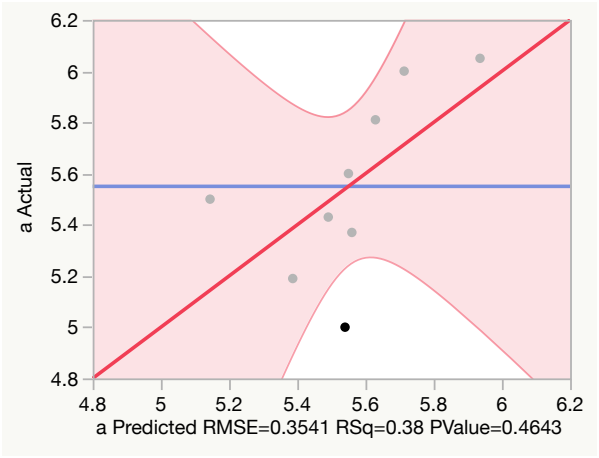
Table 14. Color model effects and significance summary. Displayed are the (p-values) for the color response regression model.

P-value Source	L*	a*	b*
Model	0.3641	0.4643	0.8861
Drying Temperature	0.1260	0.9475	0.5542
Drying Temperature*Microwave Watts	0.2462	0.3099	1.0000
Microwave Watts	0.3099	0.2462	0.6548

a.



b.



c.

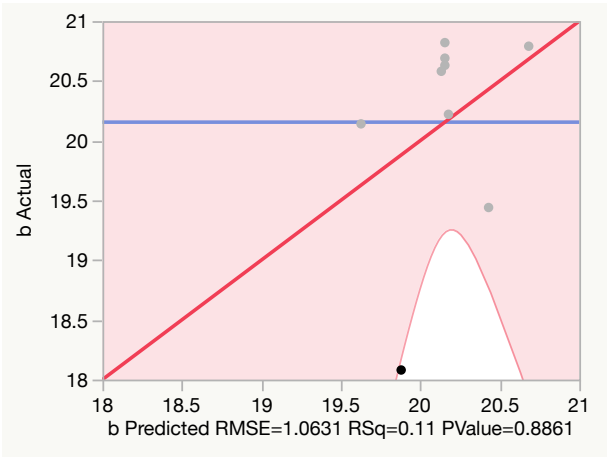


Figure 12. Color actual versus predicted plots. Graphs display model fitness significance scores demonstrated through an ANOVA analysis for each color response. A = L, B = a*, C= b**

5.3.3 BSG Grain characterization and protein

The grains analyzed in the current study exhibit an average amount of protein, however fiber was lower than expected and starch was much higher compared to a pool of samples from the DairyOne Forage Lab in Ithaca, New York (Table 15).

Table 15. NCPS nutrition characterization for BSG. Table includes results from SGS Laboratories for BSG sample oven dried at 60 °C hot air convection compared to reported accumulated average from DairyOne Forage Laboratory, Ithaca, NY between the years May 1, 2000 – April 30, 2018.

Nutrient Measured	Unit	Current BSG Dried at 60°C [1]	Dried BSG Average[2]	Range Min[2]	Range Max[2]
Dry Matter	%	91.23	92.82	89.93	95.70
PROTEIN					
Crude Protein	%	17.68	26.35	20.34	32.35
Soluble Protein,	% of CP	22.08	17.33	2.78	31.89
ADICP	%	0.71	2.93	1.29	4.56
NDICP	%	5.61	8.51	4.92	12.09
FIBER					
Lignin	%	3.43	6.49	4.44	8.54
Acid Detergent Fiber	%	9.4	24.47	18.78	30.16
Neutral Detergent Fiber	%	24.3	50.27	41.23	59.31
NON-FIBERS					
Starch	%	39.99	6.84	0.00	14.88
Crude Fat	%	2.37	8.94	6.22	11.66
MINERALS					
Ash	%	2.59	4.98	3.06	6.90
Calcium	%	0.07	0.32	0.12	0.53
Phosphorus	%	0.43	0.66	0.48	0.84
Magnesium	%	0.17	0.22	0.16	0.28
Potassium	%	0.23	0.30	0.00	0.71
Sodium	%	0.02	0.03	0.00	0.08
Iron	PPM	94.23	367.65	0.00	1303.10
Zinc	PPM	54.93	115.13	0.00	235.56
Copper	PPM	21.40	14.78	6.64	22.93
Manganese	PPM	25.12	51.64	28.36	74.92
ENERGY (ADF BASED)					
TDN	%	81.67	72.74	67.89	77.58
NEL	Mcal/Lb.	0.81	0.80	0.74	0.86
NEM	Mcal/Lb.	0.99	0.79	0.71	0.87
NEG	Mcal/Lb.	0.68	0.51	0.44	0.58

Abbreviations: ADICP, acid detergent insoluble crude protein; NDICP, neutral detergent insoluble crude protein; TDN, Total Digestible Nutrients; NEL, Net Energy for Lactation; NEM, Net Energy for Maintenance; NEG, Net Energy for Gain.

[1]SGS Laboratory, Guelph, Ontario

[2] DairyOne Forage Laboratory, Ithaca, New York

Protein was of major interest due to its susceptibility to heat damage and value as a commodity. Crude protein values pre and post drying treatments, as a percentage of dry matter, are reported in Table 16. Protein varied between 17.68 - 20.26 % and fell within the anticipated range reported in previous studies, which varied from 14 – 31 % (Mussatto et al 2006; Lynch et al 2016; Santos et al 2003; Westendorf et al., 2002). Acid detergent insoluble protein, or bound protein (ADP), was quantified with acid detergent fiber (ADF) since they are common indicators of grain overheating (Table 16) (Sniffen et al., 1992). ADP values as a percentage of crude protein were specifically targeted since values higher than 12% suggests that some overheating has occurred, leaving said percentage of protein unavailable to the animal and microbes in the gut for dairy animals (SGS, 2018). Current ADP as a percentage of crude protein ranges from 3.30-12.39% (Table 16).

Table 16. Acid detergent protein and fiber values. Values are displayed as drying temperature and microwave power density increases.

Temperature (°C)	Power Density (W g^{-1})	PROTEIN (CP)	ADP	ADP as % CP	ADF
Untreated	Untreated	19.67	0.65	3.30	8.55
60	0	17.68	0.71	4.01	9.40
60	1	18.50	0.76	4.10	10.02
60	2	18.79	0.80	4.28	9.96
75	0	18.75	0.74	3.92	10.56
75	1	18.69	1.00	5.37	10.42
75	2	18.52	0.78	4.20	9.73
90	0	18.94	0.63	3.34	10.35
90	1	20.26	2.51	12.39	14.17
90	2	18.82	1.80	9.59	13.73

Finally color *L** saw a greater change at 90 ºC+ 1W g^{-1} similar to ADF and ADP (Figure 13). However, the change in lightness was considerably greater at 60 °C + 2W g^{-1} and 75 °C compared to ADF and ADP. Therefore a color detection method may not be able to quickly indicate a loss in fiber and protein digestibility.

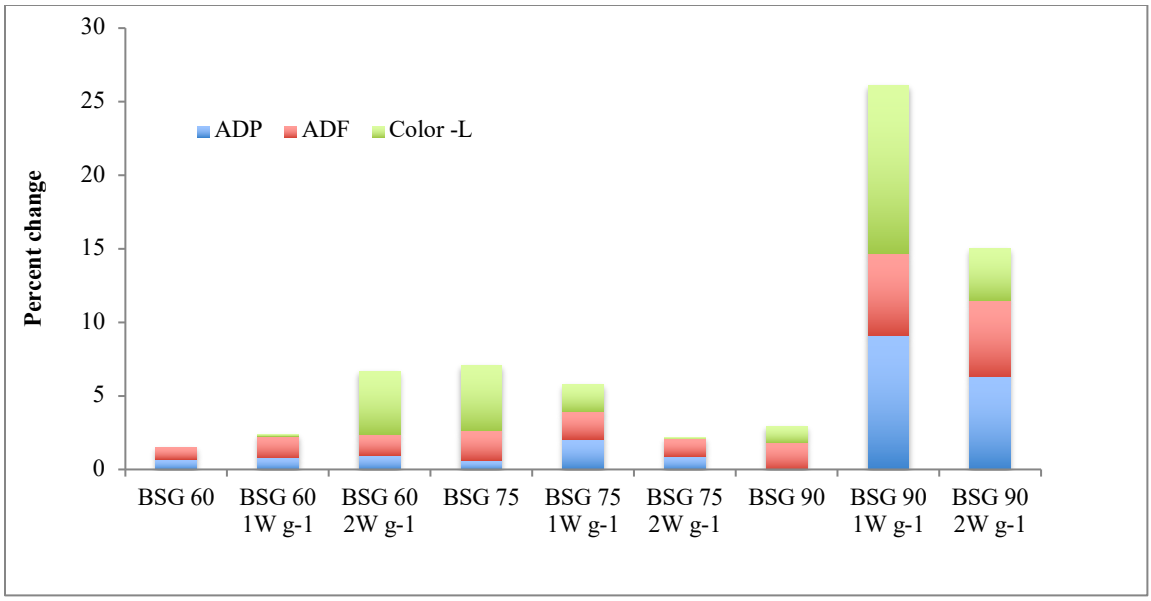


Figure 13. Change in ADF, ADP and color L percentages from untreated grains as a function of drying treatments.*

5.3.4 Response surface analysis and model fitness

A central composite design from a quadratic response surface methodology of JMP's software was used in order to determine optimal drying conditions to avoid negative overheating effects on brewers' spent grains, such as decreasing protein availability and reducing grain digestibility for dairy animals. The central composite design pattern, factors and experimental data collected to model optimal drying conditions are listed in Table 17.

Table 17. CCD data table. The experimental data and central composite design pattern were used to model optimal drying conditions and limit grain overheating.

Pattern	Temp., °C	Power Density (W g^{-1})	Time, Min	Color - L	Color - a	Color - b	Protein	ADP (% CP)	ADF
++	90	2	19	37.98	6.05	20.58	18.82	9.59	13.73
+−	90	0	84	38.95	5.50	20.14	18.94	3.34	10.35
−+	60	2	36	41.12	5.43	20.22	18.79	4.28	9.96
−−	60	0	131	39.41	5.81	20.79	17.68	4.01	9.40
0	75	1	42	40.16	5.60	20.63	18.69	5.37	10.42
0	75	1	47	40.16	5.60	20.63	18.69	5.37	10.42
0a	75	0	101	41.19	5.19	20.69	18.75	3.92	10.56
0A	75	2	33	39.46	6.00	20.82	18.52	4.20	9.73
a0	60	1	60	39.34	5.37	19.44	18.50	4.10	10.02
A0	90	1	41	34.90	5.00	18.08	20.26	12.39	14.17

The model was significant (p = 0.0006) for the response Time (min) according to the analysis of variance results (Table 18) in effectively predicting drying time as a function of temperature and microwave power density. The predicted versus actual experimental data fit the model well (R^2 = 0.99) (Figure 14).

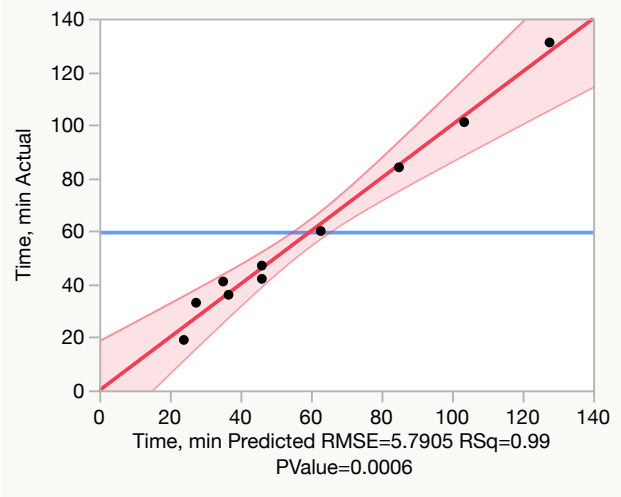


Figure 14. Drying time actual vs predicted plot. A strong correlation was found (R^2 = 0.99) between experimental and modeled data as a function of drying temperature and power density.

Table 18. Drying time ANOVA table. Analysis of variance for the response Time was statistically significant (p <0.001).

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	11000.28	2200.06	65.62
Error	4	134.12	33.53	**Prob > F**
C. Total	9	11134.40		0.0006

Temperature and power density model effects were statistically significant (p = 0.0043 and p = <0.0001), where microwave power density was highly significant over temperature. The interaction between temperature and power density, however, was insignificant (p = 0.0607) (Table 19). Finally, the modeled CCD solution as a function of these main effects was a minimum and predicted 22 minutes 31 seconds to reach 15% MC (w.b.) with an air temperature of 97.8 ºC and power density of 1.69 W g^{-1} (Table 20; Figure 15).

Table 19. Drying time model main effects. Power density was highly significant statistically (p <0.001) and temperature was statistically significant (p <0.01). The interaction between temperature and power density was insignificant (p >0.01).

Source	DF	Sum of Squares	F Ratio	Prob > F
Temperature (60,90)	1	1148.17	34.24	0.0043
Power Density (W/g)(0,2)	1	8664.00	258.40	<.0001
Temperature*Power Density (W/g)	1	225.00	6.71	0.0607
Temperature*Temperature	1	19.05	0.57	0.4930
Power Density (W/g)*Power Density (W/g)	1	874.30	26.08	0.0070

Table 20. Drying time modeled solution. Solution was a minimum in 22:31 minutes.

Variable	Critical Value
Temperature (60,90)	97.78
Power Density (W/g)(0,2)	1.69

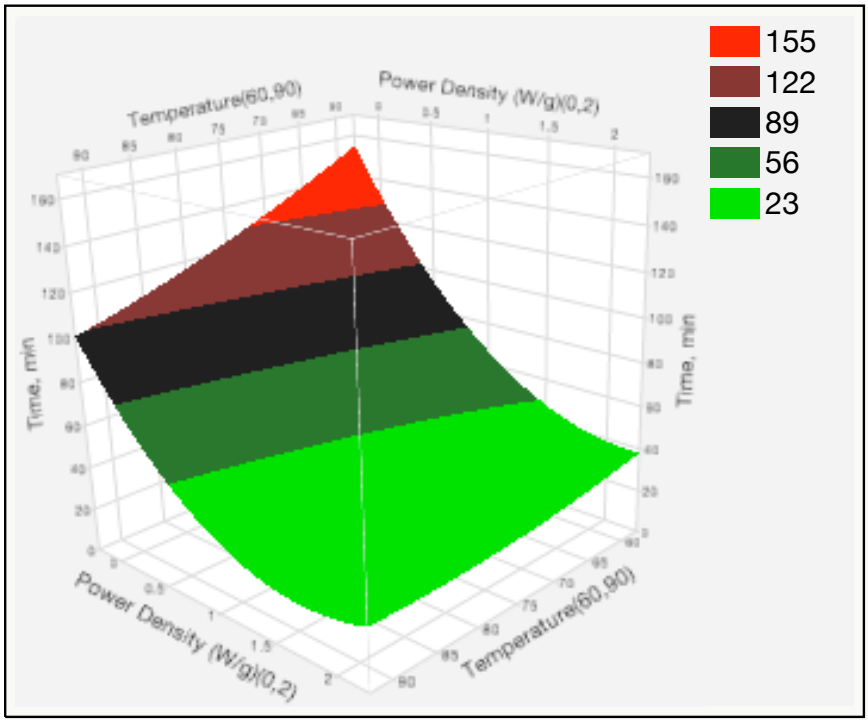


Figure 15. Drying time surface plot. Response surface analysis illustrating optimal parameter settings in order to minimize drying time. Modeled solutions drying time resulted in 22:31 minutes.

The resulting model equation that was used to predict the drying time response:

$$Drying\ time = 46.07 + -13.83 * ((Temperature - 75) / 15) + -38 * ("Power\ Density\ (W\ g^{-1})" - 1) + ((Temperature - 75) / 15) * (("Power\ Density\ (Wg^{-1})" - 1) * 7.5) + ((Temperature - 75) / 15) * ((Temperature - 75) / 15) * 2.86) + ("Power\ Density\ (Wg^{-1})" - 1) * ("Power\ Density\ (Wg^{-1})" - 1) * 19.36)$$

Equation 8

The model was suitable to predict drying time with temperature and power density as 2 significant main effects, however this was not the case for color, protein and fiber quality response variables ($p > 0.01$) (Table 21).

Table 21. Quality response variables ANOVA model p-values. Remaining quality indicator response variables were insignificant (p = >0.01).

ANOVA model *p*-values	
Response	**Prob > F**
Color - L	0.1561
Color - a	0.4708
Color - b	0.2604
Protein	0.2200
ADP (%CP)	0.1520
ADF	0.1049

Finally the prediction profiler was optimized for desirability to determine the optimal conditions to decrease drying time, increase Color L^*, decrease ADF as %CP and ADF. The parameter settings for a drying solution provided from the prediction profiler was 64.5 °C air temperature with a microwave power density of 2 W g^{-1} (Figure 16) Under these settings, the brewers' spent grains would dry in 33 minutes, have a color L^* value of 41.25, ADP as %CP at 3.12 % and ADF as 9.41%.

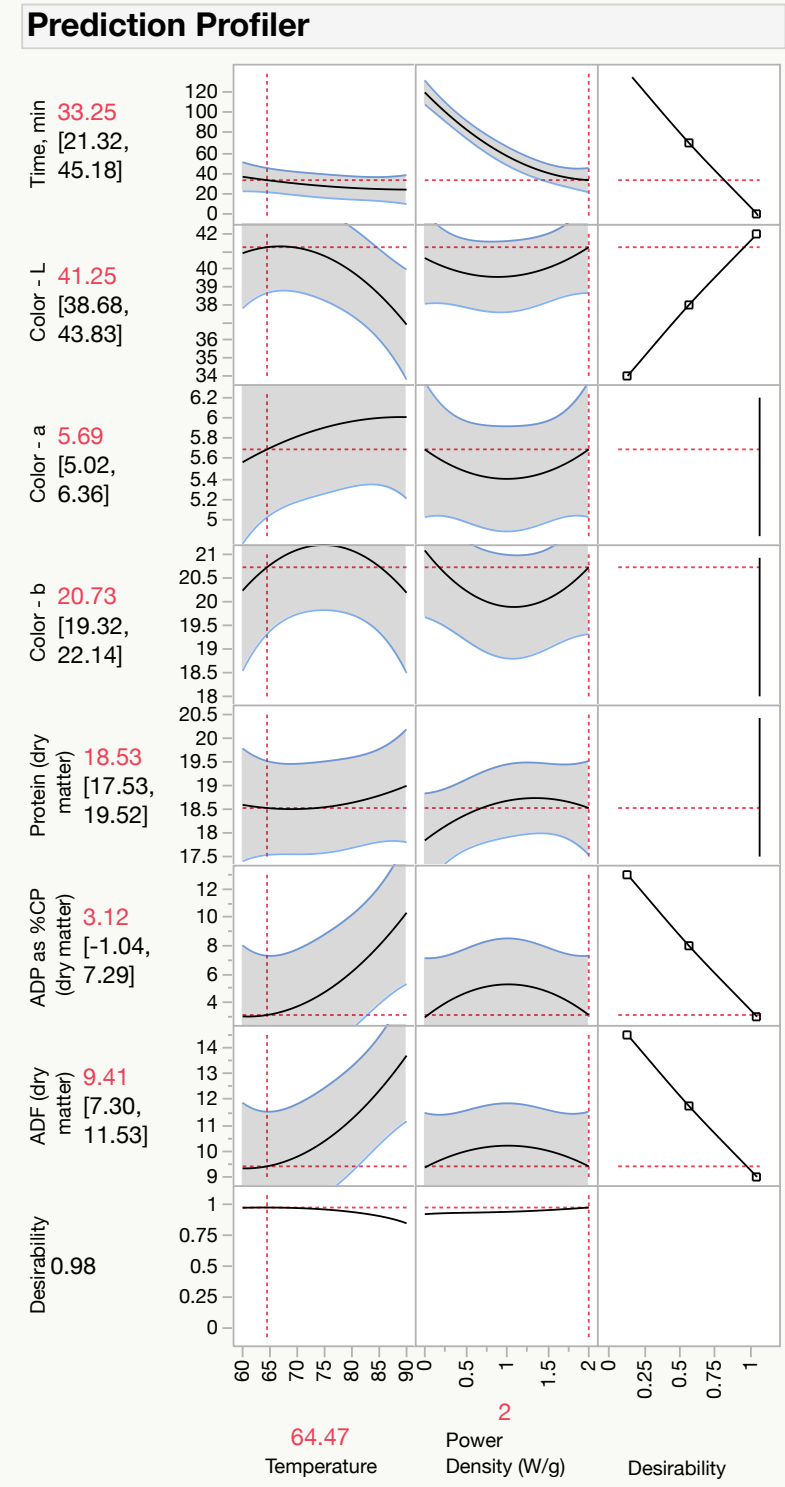


Figure 16. Prediction profiler to maximize quality and drying parameters. The prediction profiler demonstrates the optimal drying conditions (i.e., 64.47 °C + 2 W g-^{1}) to yield optimal product quality. The analysis set desirability standards to decrease ADF, ADP and time, while increasing L or lightness.*

5.4 Discussion

Color tests and regression model for grain color prediction

Figure 17 illustrates the final grain product from the resulting dryer treatment tests. No distinct color differentiation was visually observed. A clear trend between temperature, microwave power and lightness was not found (Table 21). The regression model for all color responses resulted in a poor fit and the ANOVA was statistically insignificant (R^2<0.999; p > 0.001) (Table 14; Figure 12). Similar results were found in a study with carrot slices where color did not change significantly, however color preservation was most successful using a 20 % sugar pretreatment solution (Hu et al., 2016). However this cited study used a temperature range between 40-60 °C and power density from 0.44-1.21 W g^{-1} as opposed to higher temperatures and power densities seen in the current study. On the contrary, Salim et al (2015) found less color change with broccoli samples dried between 40-60 °C using hot air drying microwave assisted. Although in Salim et al (2015), microwave power density was not a variable and remained constant at 100 W or 2 Wg^{-1}.

Higher temperatures and increasing power density would suggest a change in color, but as previously mentioned, the effect of temperature and power density on color was highly insignificant in the current study (p =>0.10). Specifically for lightness with temperature 60 °C, L^* values increased as power density increased, whereas L^* decreased suggesting grain darkening, as power density increased for 75 °C and 90 °C. The samples 90 ºC with power densities of 1 and 2 W g^{-1}, resulted in the lowest L^* values, and is believed to be as a result of isolated material burning observed during these two treatments (Figure 17, Figure 18). Isolated burns only occurred at 90 ºC and with both 1 and 2 W g^{-1} microwave power density settings. Under high air temperature conditions and the non-uniformity, or potential for excessive heating from microwave, increased the susceptibility of grain burning at higher temperatures and power densities in this study (Vadivambal and Jayas, 2001). The drying treatment 90 ºC + 1 W g^{-1} resulted in being the darkest sample (L^* = 34.90 ± 2.76) and highest change in color value (ΔE = 5.32).

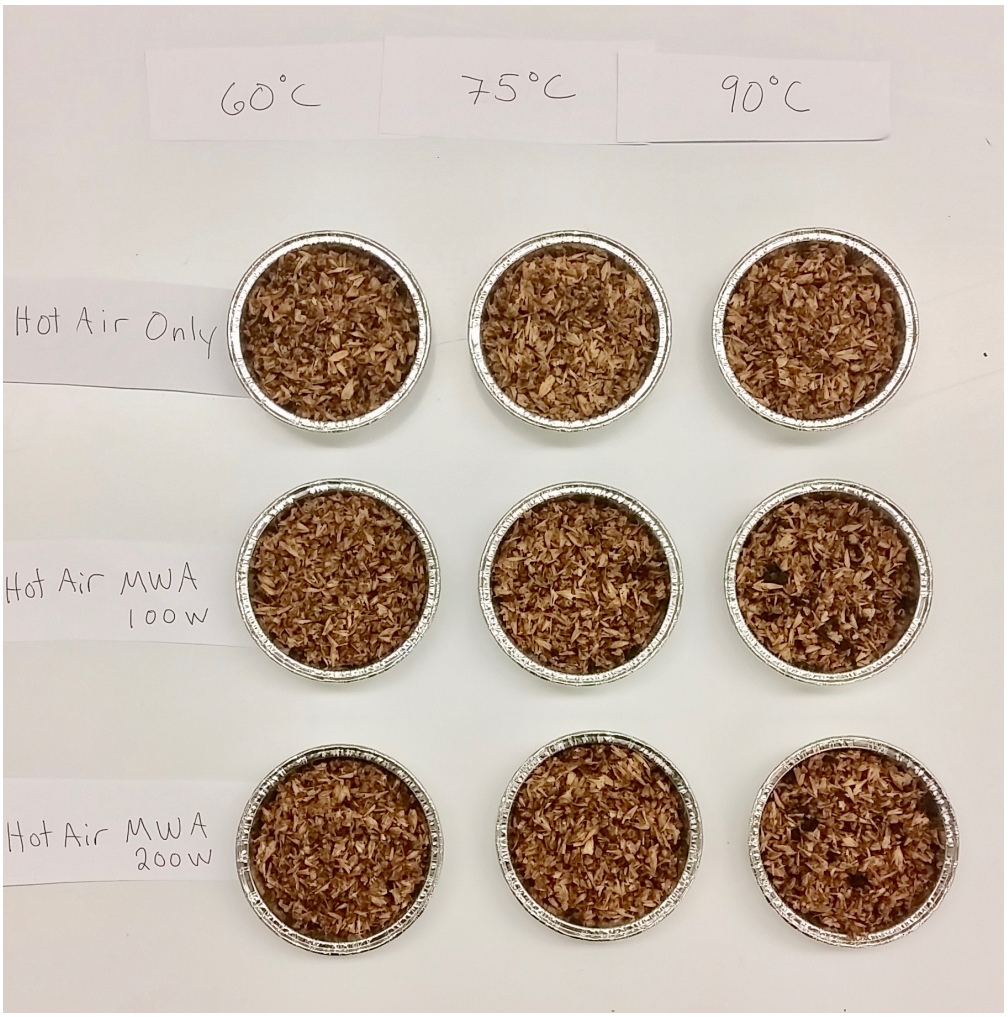


Figure 17. Dried brewers' spent grains. Image taken of all treated samples and organized according to air temperature and microwave power.

Figure 18. Burned BSG. Isolated burned grains after drying treatment.

Grain characterization and protein

Total digestible nutrients (%TDN) was slightly higher (%TDN = 81.67) than the average (%TDN = 72.74) and additional details regarding initial grain variant and the brewing process would further explain these differences. Starch in the grains was much higher (% starch = 39.99) than the average (% starch = 6.84) suggesting starches remained high after the malting process and this could be why the concentration of fiber (%NDF = 24.30) and protein (%CP = 17.68) were lower in the spent grains compared to the average (%NDF = 50.27; %CP = 26.35) (Thomas et al., 2010). The gelatinization process, malt enzymes (e.g., α-amylase, β-amylase, limit dextrinase, and α-glucosidase) and temperature can all play a role in the efficiency of sugars extracted from the hydrolysis of starch to create wort (Gupta et al., 2010). As reported from the current characterization and comparing values from a sampled pool spanning 18 years with between 200-400 sampled grains at DairyOne Forage Lab in Ithaca, New York, the nutrient value can vary considerably. Due to this known variability, Westendorf et al. (2002) would suggest sampling and nutrient monitoring before reusing the grains as a feed source.

Acid detergent fiber (ADF) and bound protein (ADP) were monitored in order to assess protein quality and whether or not drying treatments affected the digestibility of the grains as an animal feed because as ADF increases, normally forage digestibility decreases (SGS, 2018). The current study demonstrated that ADF and ADP increased as temperature increased with microwave assisted drying (Table 16). Bound protein increased to 12.39 % with an air temperature of 90 ºC and power density 1 W g^{-1}. While under the same temperature condition (i.e., 90 °C) and a power density of 2 W g^{-1}, bound protein resulted in 9.59 %. Increasing acid detergent fiber and bound protein percentages at 90 ºC with power densities of 1 and 2 W g^{-1} would suggest that these samples began to experience overheating. These two samples also experienced material burning (Figure 18).

Response surface analysis to optimize drying efficiency and quality

The central composite design included 10 runs and 2 center points. A central composite design with 13 runs and 5 center points was the preferred method to increase precision, however insufficient sample remained to complete the preferred design. The model included temperature (°C) and power density (W g^{-1}) as predictor factors with 3 levels each. The response variables to minimize in the model were Time (min), ADP (%CP) and ADF. The Color – L* response variable

was maximized to optimize a lighter colored sample. The responses Color – a*, Color – b* and crude protein were left in the model to examine any significant effect in the model.

As previously stated the regression model was suitable to predict drying time with temperature and power density as 2 significant main effects, however this was not the case for color, protein and fiber quality response variables ($p > 0.01$) (Table 21). The model resulted in saddle point solution for these responses, meaning neither a maximum nor a minimum was found. The prediction model was statistically insignificant for Color L*, Color a*, Color b*, Protein, ADP as %CP and ADF ($p = >0.05$) (Table 21). This was anticipated for Color L*, Color a* and Color b* since these variables previously demonstrated that color change was insignificant as a function of temperature and microwave power density (Table 14). However, temperature as a main effect in each response was significant for the model ADF ($p =0.0242$) and near significant for Color L* ($p = 0.0586$), protein ($p = 0.0608$) and ADP as %CP ($p =0.0594$).

Finally the optimal conditions to decrease drying time, increase Color L*, decrease ADP as %CP and ADF was with using an air temperature of 64.49 °C and microwave power density of 2 W g^{-1}. ADF as %CP is known to decrease in wheat distillers dried grain with solubles samples with increasing air temperature in convection drying and microwave power (Mosqueda et al., 2013). Furthermore, protein quality and color are known to have a significant relationship in forced air convection, microwave and microwave-convection dried wheat distillers grains with solubles (Mosqueda et al., 2013). The current study did not attempt to analyze the additional costs of added microwave to enhance drying, and by including a cost parameter may alter the results to reduce microwave and increase temperature since microwave drying is known to be costly for food drying; although Vadivambal and Jayas (2007) would argue the increased efficiency in drying and product quality benefits render microwave drying a cost-effective solution.

5.5 Conclusion

The regression model for each color response as a function of drying temperature and microwave power density resulted in a poor fit, and the ANOVA was statistically insignificant ($R^2<0.999$; $p >0.001$) (Table 14; Figure 12). Therefore neither drying temperature nor microwave power had a significant effect on grain color throughout the drying process. The sample 90 ºC hot air + 1 W g^{-1} power density saw the greatest color change (ΔE) and was also the darkest sample ($L^* = 34.90$), however results from the current study were statistically insignificant to predict this response.

The model was significant ($p = 0.0006$) for the response Time (min) in order to predict the minimum drying time as a function of temperature and microwave power density (Table 18), however temperature and microwave power could not predict the response color, protein and fiber quality response variables ($p > 0.01$) (Table 21). The modeled CCD solution for drying time was a minimum and predicted 22 minutes 31 seconds to reach 15% w.b. with an air temperature of 97.8 ºC and power density of 1.69 W g^{-1} (Table 20; Figure 15).

The grains analyzed in the current study demonstrate an average amount of protein, however fiber was lower than expected and starch was much higher compared to a pool of samples from the DairyOne Forage Lab in Ithaca, New York (Table 15). In the experimental samples studied, ADF as a percentage of crude protein varied from 3.30-12.39 % (Table 16). Bound protein increased with an air temperature of 90 ºC and added microwave power, indicating that these samples began to experience overheating and a reduction in protein quality at 90 ºC with the addition of microwave power only.

Finally the optimal parameter settings for a drying solution to minimize time and maximize quality was with an air temperature of 64.49 °C + 2 W g^{-1} (Figure 16). Under these settings, brewers' spent grains would dry in 33 minutes, have a color L^* value of 41.25, ADP as %CP at 3.12% and ADF as 9.41%.

Chapter 6: Final summary and closing statement

6.1 Final summary

The current study was successful in determining efficient dewatering and drying conditions to maximize water removal from brewers' spent grain while preserving digestible protein. During the dewatering stage, maximum water loss was modeled using a response surface methodology, however the original model proved to be inadequate with a significant lack-of-fit report (p = 0.0012) despite experimental data correlating with modeled data well (R^2 = 0.98). The water loss model as a function of pressure and holding time may be improved by reducing the holding time's gap between each level. Pressure was highly significant in the model (p = <0.0001) and when the model was reduced to holding time only, it was statistically insignificant (p = 0.5535). The maximum water loss was predicted at 35 % as a result of 1.19 MPa of pressure and 3 minutes holding time. Therefore by applying the current press parameter settings, wet brewers' spent grains were reduced from 70 % MC w.b. to 60 % MC w.b. mechanically, but missing the 50 % MC target. From the first stage of drying an additional 88 % wt of the remaining water was to be removed thermally to reach 15 % MC w.b.

Drying times were modeled and predicted drying times to reach 15 % w.b. moisture were found. As suspected, as drying temperature and microwave power density increased, drying time decreased. The addition of microwave during hot air convection drying reduced or eliminated the falling rate drying period, and the microwave power density setting of 2W g^{-1} nearly tripled the drying rate during the initial constant rate of drying. In general drying rates improved by 40-60 % due to microwave assisted drying. The air velocity during these drying tests remained constant throughout experiments, however this parameter would be interesting to explore further to improve efficiency since it has been reported to have a significant effect on thin-layer drying of spent grains in super-heated steam drying (Tang et al., 2005). Finally, an air temperature setting of 90 ºC and 2 W g^{-1} microwave assisted power density was the most efficient drying method where 15 % w.b. moisture was predicted in only 19 minutes. Following a central composite design as part of a response surface methodology to optimize the drying time response, optimal conditions were discovered with hot air at 97.8 ºC and 1.69 W g^{-1} microwave power density resulting in 23 minutes to dry.

Although 90 ºC + 2 W g^{-1} microwave assisted drying conditions yielded the most efficient drying method, both microwave assisted drying treatments at 90 °C resulted in grain overheating and burning. Material darkness (L^*) increased for both of these samples, and 90 ºC + 1 W g^{-1} was the darker sample of the two ($L^* = 34.90$). However the color regression model analysis resulted in a poor fit ($R^2 < 0.50$) and proved that drying temperature and microwave power density did not have a significant effect on grain color ($p > 0.30$). Color was examined with protein quality in order to determine if color change, and specifically lightness (L^*), could correlate with reduced protein digestibility. However with no significant effect to color across drying treatments, color alone could not be used as a rapid and inexpensive grain-overheating indicator with BSG.

Finally bound protein (ADP) increased to levels that would suggest protein digestibility had been affected as a result of heat damage (> 12%) during the 90 ºC hot air microwave assisted drying treatments. Acid detergent fiber (ADF) also increased as expected during this time. The response surface analysis having a central composite design was employed to model the least amount of bound protein under the most efficient drying conditions. The response drying time could be modeled adequately ($p = 0.0006$; $R^2 = 0.99$), where other responses such as ADP, color lightness (L^*), crude protein and ADF could not ($p > 0.10$). Despite the lack of significant modeling capabilities with the current experimental results and design, the prediction profiler from the response surface analysis report was optimized to minimize drying time while maximizing color lightness and minimizing undigestible protein (ADP). Dryer settings to reach this goal was with hot air at 64.49 °C and 2 Wg^{-1} microwave power density resulting in 33.27 minutes to reach 15 % w.b. moisture.

6.2 Closing statement

The current study examines the importance of preserving digestible proteins for ruminants as an animal feed during processing conditions, while assessing optimal drying parameter conditions to yield a nutritional dried feed. Although this study was designed to develop a process for enhancing animal nutrition from BSG, human nutrition can benefit from the valorization of food waste as well. Kosseva (2013) discussed functional food and nutraceuticals derived from fruit and vegetable waste, which like brewers' spent grains, are known to contain functional food properties with health benefits related to disease prevention. Extraction and preservation methods to retain the nutritional and chemical value in food waste are important fields of continued research. The functional foods market itself is growing by ~8% each year (Kosseva, 2013).

Beyond the grains having high-valued nutritional properties, it is vital that technological innovation to improve societal and economical inefficiencies are continuously challenged or made available. Reducing food waste continues to draw more attention, and Canada itself is targeting a 50% reduction in food waste by 2030, where food scraps as an animal feed is part of the recovery strategy (NZWC, 2018).

To conclude, the importance of the food waste valorization work presented is not only to shed light on better methods to manage waste and reduce organics in landfills which generate GHG emissions, but to help create a product which advances the health and safety of both humans and animals, while harnessing scientific innovation to develop advanced processing systems.

6.3 Contribution to knowledge

- This study found that digestible protein was retained in the brewers' spent grains dried at 90 °C.
- The nutrient composition of BSG, specifically protein quantity, was demonstrated to be consistent with findings in literature.
- Drying models for BSG were provided.
- Optimized parameter settings at lab scale for a two-stage drying process were determined.
- Color measurements are not effective in determining BSG quality when dried between 60 °C and 90 °C with microwave assistance ranging from 1 to 2 W g^{-1}.

6.4 Future recommendations

- Microwave assisted drying may not be necessary for animal feed with regards to protein digestibility preservation since 90 °C was an efficient drying temperature and retained protein digestibility.
- Examine grains from breweries using different brewing methods and grain formulas since grains vary within and between breweries in order to generalize conclusions.
- Vary drying treatments to include air velocity and vacuum as variables.
- To include the financial impact of the varying drying processes as a factor in order to determine the most efficient drying methods for industry at a scalable level.
- Compare other methods against ADP measurements such as pH or colorimetry to quantify heat damage or Maillard reaction products.

- Improve press holding time by varying the quantity of grain to use in the press in order to manipulate and examine bed thickness as a variable.
- Reduce holding time gap between measurement intervals.

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Appendix A

Analysis Report

GF18-00431.001

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG UNT
Client Sample ID: 1

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	32.98
Moisture (%)	--	67.02
PROTEIN		
Protein {N x 6.25} (%)	19.67	6.49
ADF-CP [ADP] (%)	0.65	0.21
ADP as %CP	3.30	--
FIBRES		
ADF (%)	8.55	2.82

NOTE:
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Signed and dated in Guelph, ON
On 25-Oct-2018

For and on behalf of SGS Canada Inc., Agriculture and Food

Jack Legg
Branch Manager

Report File Reference Number: 0000095604

SGS Canada Inc. | Agriculture and Food
Suite 1 - 503 Imperial Road N Guelph ON N1H 6T9 t+1(519) 837-1600 f+1(519) 837-1242 www.sgs.com/agriculture

Analysis Report

GF18-00431.002

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 60
Client Sample ID: 2

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	91.23
Moisture (%)	--	8.77
PROTEIN		
Protein {N x 6.25} (%)	17.68	16.13
Soluble Protein (%)	3.90	3.56
Soluble Protein as %CP	22.08	--
ADF-CP [ADP] (%)	0.71	0.65
ADP as %CP	4.01	--
NDF-CP [NDP] (%)	5.61	5.12
NDP as %CP	31.76	--
UIP Bypass Est. as %CP	38.96	--
FIBRES		
ADF (%)	9.40	8.58
aNeutral Detergent Fibre (%)	24.3	22.2
Lignin (%)	3.43	3.12
NON-FIBRES		
Fat (%)	2.37	2.17
Starch (%)	39.99	36.48

Analysis	Dry Basis	As Is
MINERALS		
Ash (%)	2.59	2.36
Calcium (%)	0.07	0.07
Phosphorus (%)	0.43	0.39
Potassium (%)	0.23	0.21
Magnesium (%)	0.17	0.15
Sodium (%)	0.02	0.02
Copper (ppm)	21.40	19.52
Iron (ppm)	94.23	85.96
Manganese (ppm)	25.12	22.92
Zinc (ppm)	54.93	50.11
ENERGY (ADF Based)		
TDN (%)	81.67	74.51
Net Energy {lac} (Mcal/kg)	1.79	1.63
Net Energy {gain} (Mcal/kg)	1.50	1.37
Net Energy {maint} (Mcal/kg)	2.18	1.99
ENERGY (OARDC)		
WTDN (%)	78.74	71.84
WNEL (Mcal/kg)	1.81	1.65
WNEG (Mcal/kg)	1.31	1.19
WNEM (Mcal/kg)	1.95	1.78

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Analysis Report

GF18-00431.003

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 60_100
Client Sample ID: 3

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	94.97
Moisture (%)	--	5.03
PROTEIN		
Protein {N x 6.25} (%)	18.50	17.57
ADF-CP [ADP] (%)	0.76	0.72
ADP as %CP	4.10	--
FIBRES		
ADF (%)	10.02	9.52

NOTE:
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Signed and dated in Guelph, ON
On 25-Oct-2018

For and on behalf of SGS Canada Inc., Agriculture and Food

Jack Legg
Branch Manager

Report File Reference Number: 0000095604

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Analysis Report

GF18-00431.004

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 60_200
Client Sample ID: 4

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	90.53
Moisture (%)	--	9.47
PROTEIN		
Protein {N x 6.25} (%)	18.79	17.01
ADF-CP [ADP] (%)	0.80	0.73
ADP as %CP	4.28	--
FIBRES		
ADF (%)	9.96	9.02

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Signed and dated in Guelph, ON
On 25-Oct-2018

For and on behalf of SGS Canada Inc., Agriculture and Food

Jack Legg
Branch Manager

Report File Reference Number: 0000095604

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Analysis Report

GF18-00431.005

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 75
Client Sample ID: 5

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	92.61
Moisture (%)	--	7.39
PROTEIN		
Protein {N x 6.25} (%)	18.75	17.37
ADF-CP [ADP] (%)	0.74	0.68
ADP as %CP	3.92	--
FIBRES		
ADF (%)	10.56	9.78

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On 25-Oct-2018

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Jack Legg
Branch Manager

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Analysis Report

GF18-00431.006

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 75_100
Client Sample ID: 6

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	89.36
Moisture (%)	--	10.64
PROTEIN		
Protein {N x 6.25} (%)	18.69	16.70
ADF-CP [ADP] (%)	1.00	0.90
ADP as %CP	5.37	--
FIBRES		
ADF (%)	10.42	9.31

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Analysis Report

GF18-00431.007

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 75_ 200
Client Sample ID: 7

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	83.78
Moisture (%)	--	16.22
PROTEIN		
Protein {N x 6.25} (%)	18.52	15.52
ADF-CP [ADP] (%)	0.78	0.65
ADP as %CP	4.20	--
FIBRES		
ADF (%)	9.73	8.15

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On 25-Oct-2018

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Jack Legg
Branch Manager

Report File Reference Number: 0000095604

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Analysis Report

GF18-00431.008

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 90
Client Sample ID: 8

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	91.03
Moisture (%)	--	8.97
PROTEIN		
Protein {N x 6.25} (%)	18.94	17.24
ADF-CP [ADP] (%)	0.63	0.58
ADP as %CP	3.34	--
FIBRES		
ADF (%)	10.35	9.43

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On 25-Oct-2018

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Jack Legg
Branch Manager

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Member of the SGS Group (Société Générale de Surveillance)

Analysis Report

GF18-00431.009

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 90_100
Client Sample ID: 9

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	90.60
Moisture (%)	--	9.40
PROTEIN		
Protein {N x 6.25} (%)	20.26	18.35
ADF-CP [ADP] (%)	2.51	2.27
ADP as %CP	12.39	--
FIBRES		
ADF (%)	14.17	12.83

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On 25-Oct-2018

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Jack Legg
Branch Manager

Report File Reference Number: 0000095604

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Analysis Report

GF18-00431.010

MCGILL UNIVERSITY
21 111 LAKESHORE RD
CINE BLD RM 204 MACDONALD CAMPUS MCGILL
STE ANNE DE BELLEVUE QC H9X 3V9
CANADA
Commodity/Product: BSG 90_200
Client Sample ID: 10

Job Number : GF18-00431
Received : 12-Oct-2018
Completed : 25-Oct-2018
Order Reference : Christine Crowe - BGS UNT...BSG90200

Analysis	Dry Basis	As Is
Dry matter (%)	--	88.01
Moisture (%)	--	11.99
PROTEIN		
Protein {N x 6.25} (%)	18.82	16.56
ADF-CP [ADP] (%)	1.80	1.59
ADP as %CP	9.59	--
FIBRES		
ADF (%)	13.73	12.08

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On 25-Oct-2018

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Appendix B

RE: Form submission from: Contact MAGB

Sue Capewell <sue@magb.org.uk>

Tue 1/8/2019 9:00 AM

Inbox

To:Christine Crowe <christine.crowe@mail.mcgill.ca>;

1 attachments (573 KB)

cornsection.jpg;

Hi Christine

Please find attached the image requested in the biggest size we have. Hope you find this useable.

Please acknowledge MAGB secretariat as the source.

Good luck with your thesis

Best regards
Sue

Sue Capewell MAGB Secretariat	Tel: 01636 700781 E: sue@magb.org.uk	
1st Floor, Exchange Business Centre, Water Lane, Newark, Notts, NG24 1HA www.ukmalt.com Twitter.com/MagbInfo Facebook.com/magb.maltsters		

From: Christine Crowe <christine.crowe@mail.mcgill.ca>
Sent: 08 January 2019 13:51
To: Sue Capewell <sue@magb.org.uk>
Subject: Re: Form submission from: Contact MAGB

Dear Sue,

Thank you for your email. The image I am requesting to use is at the bottom of the page and attached in this email.

End of document