

**INFLUENCE OF HEAT-MOISTURE TREATMENT ON FUNCTIONAL, COLOUR AND
THERMAL PROPERTIES OF BAMBARA GROUNDNUT STARCH**

MSc dissertation

By

Mathobo Vhulenda Melinda (11620018)

BSc (Hons) Food Science and Technology



University of Venda

Department of Food Science and Technology, School of Agriculture, University of Venda,
Thohoyandou 0950, South Africa

Supervisor: Dr. Henry Silungwe

Co-supervisors: Dr. Shonisani E. Ramashia

Dr. Tonna A. Anyasi

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ABSTRACT

Heat-moisture treatment (HMT) is a physical modification that alters the physicochemical properties of starch without changing its molecular structure. The objective of the study was to investigate the influence of HMT on the functional, colour and thermal properties of bambara groundnut (BG) starch. A central composite rotatable design comprising two independent factors (temperature and time) was used for the study. The central composite rotatable design was generated using Design-Expert software version 8.0.1.0. Bambara starch extraction was done by milling BG into flour (5 Kg), suspension in 15 L, 0.3% sodium hydroxide and centrifugation followed by washing using distilled water. The starch was then HMT treated in an air oven at 80 - 120 °C for 30 - 90 min under 15 % moisture content (MC) (HMT 15), 25% MC (HMT 25) and 35% MC (HMT 35). The highest L^* and WI values for HMT treated BG starch were observed at HMT 80 °C for 30 min under 15% MC; 100 °C for 60 min (25% MC); and 100 °C for 17.57 min (35% MC) while the lowest was observed in HMT 100 °C for 102.43 min (15% MC); 120 °C for 90 min (25% MC); and 120 °C for 90 min (35% MC). In HMT 15-BG starch, the gelatinisation parameters onset (T_o), peak (T_p) and concluding temperature (T_c) of the samples decreased as treatment time and temperature increased whereas gelatinisation enthalpy of BG starch increased with increase in HMT treatment temperature and time. In HMT 25-BG starch T_p , and gelatinisation enthalpy of the starch increased with increase in HMT treatment temperature and time. While in HMT 35-BG starch, T_o , T_p , T_c and gelatinisation enthalpy of the starches decreased with increase in HMT treatment temperature and time. In HMT 15-BG starch, the water absorption capacity (WAC), solubility and swelling power (SP) decreased as treatment time and temperature increased while oil absorption capacity (OAC) of the starch increased with increase in HMT treatment temperature and time. In terms of HMT 25-BG starch, the WAC and OAC increased as HMT treatment time and temperature increased while SP and solubility of the starch decreased with increase in HMT treatment temperature and time. In HMT 35-BG starch, OAC, solubility and SP decreased as treatment time and temperature increased while WAC of the starch increased with increase in HMT treatment temperature and time. The optimum HMT conditions for BG starch were found to be 80 °C for 30 min (HMT 15), 105.74 °C for 30 min (HMT 25), and 113.16 °C for 30 min (HMT 35). Desirability of the obtained optimum conditions were 0.63 (HMT 15), 0.56 (HMT 25) and 0.64 (HMT 35). Information obtained from scanning electron micrograph indicates oval and round shape for bambara starch granules, with varying sizes. The range of the granule size width was 4.2 – 4.7 mm and 10 µm for length. The modified starches showed some changes in granule morphology as they seem to disintegrate with application of HMT. Unmodified and HMT - BG starches showed characteristic FTIR bands linked with common starches. All the samples

displayed complex vibrations in the region below 1000 cm^{-1} due to the skeletal vibrations of the glucose pyranose ring. Statistical analysis on colour, thermal and functional properties of HMT 15-BG, HMT 25-BG and HMT 35-BG starch showed that effects of temperature and treatment time had no significant ($p \geq 0.05$) effect on these properties of HMT-BG starch. However, treatment time had a significant linear effect ($p \leq 0.05$) on swelling power, for HMT 15-BG starch. In HMT 35-BG starch, WAC was significantly affected by quadratic effect of temperature and time while solubility was significantly affected by linear effect of time and quadratic effect of temperature.

Keywords: Bambara groundnut starch; heat moisture treatment; colour characteristics; functional properties; thermal properties.

DECLARATION

I, Mathobo Vhulenda Melinda hereby declare that the dissertation titled “Influence of heat-moisture treatment on functional, physicochemical and thermal properties of bambara groundnut starch” submitted for the degree MSc Food Science and Technology at the University of Venda is my work, except where stated and has not been submitted by me for a degree at any other university or institution of higher learning.

Student’s signature.....

Date.....

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CHAPTER 1: INTRODUCTION

1.1. Introduction

Bambara groundnut (BG) is a self-pollinating legume which is preferred by farmers living in rural areas with limited resources (Azam-Ali *et al.*, 2001). Its annual global production is 330,000 tons where in 45 - 50% of BG is produced in Western Africa with Nigeria being the highest producer (33,000 to 49,000 tons) (Bamshaiye *et al.*, 2011). However, according to Greenhalgh (2000) there are no reliable production figures for BG in South Africa (SA). The crop is tolerant to drought, pests and produces a reasonable yield even when grown under poor soil conditions (Berchie *et al.*, 2010). BG is resilient to adverse environmental conditions and grow in low fertility soils and minimal rainfall (Azam-Ali *et al.*, 2001). BG which is botanically known as *Vigna subterranean* (L.) Verdc. and cowpeas are from the same *Fabaceae* family. Although BG is a common food staple in semi-dry areas of Africa, this legume is still not well researched despite its great nutritional potential (Bamshaiye *et al.*, 2011). Unfortunately, the production and cultivation of BG has decreased in most regions of Africa as there is arise in other crop productions (Yao *et al.*, 2015).

A study of this kind can, hopefully, motivate another look at the legume. The crop is basically grown for eating by humans as it is referred to as a 'complete food', as for example, it is a 'good supplement' for cereal-based diets (Quedraogo *et al.*, 2012). BG is produced mostly for its seeds, usually incorporated in various types of food. Matured dried seeds can be boiled and consumed as a pulse or used whole or split and then combined with maize or plantains, milled into flour, spiced and steamed (Alhassan & Egbe, 2013). In SA, BGs are mainly produced in Limpopo, Mpumalanga and KwaZulu-Natal provinces (Swanevelder, 1998). According to Masindeni (2006), the information about BG in SA is limited and very little research has been done to ensure its development, commercially. Research has established that the seed contains protein (11.4%), carbohydrate (53.1%), fat (6.1%), fibre (6.1%), ash (4.4%), calcium (0.097%), iron (0.007%), potassium (1.2%) and sodium (0.003%) (Amarteifio *et al.*, 2006).

The high content of carbohydrate in BG presents this crop as a potential source of starch for food manufacturing. Some of the botanical sources of starch are cereal, tuber, legume, unripe fruit and roots (Ashogbon, 2014). Starch adds significantly to textural qualities of different food products and numerous modern applications as a thickening, bulking and gelling agent, colloidal stabilizer and a water holding ingredient (Singh *et al.*, 2003). The industrial exploitation of native starch is restricted due to its intrinsic poor nature, such as being insoluble in water and its propensity to effortlessly retrograde and endure syneresis and form wobbly paste and gel (Ashogbon, 2014). Starch characteristics can be improved through the meticulous application of

heat and moisture which results to physical changes inside the starch granules (Anderson & Guraya, 2005).

Heat-moisture treatment is a physical alteration that changes the physicochemical characteristics of starch without triggering variations in its molecular structure. The improved starches can be advantageous for nutritive aims in view of the reduced digestibility arising from the heat-moisture treatment (Arns *et al.*, 2014). Diets comprising resistant starch, which is assimilated and absorbed sluggishly, may be valuable in the treatment of diabetes and obesity, by dropping the upsurge in blood sugar levels, after a meal (Anderson & Guraya, 2005).

Heat-moisture treatment does not include the usage of chemicals; hence this kind of starch can be an imperative constituent in the production of harmless food and may be introduced in food deprived of any limitation of quantity. Starch is a major bio-polymeric component of plants, such as grains, seeds and tubers; it is characteristically granular in terms of shapes and sizes (Tharanathan, 1995). Starch is the chief foundation of physiological energy in human food and is largely classified as accessible carbohydrate (Tharanathan & Mahadevamma, 2003). Response surface methodology (RSM) is a collection of mathematical and statistical techniques for empirical model building. By careful design of experiments, the objective is to optimize a response (output variable) which is influenced by several independent variables (input variables) (Bezerra *et al.*, 2008).

1.2. Problem statement

There is paucity of literature on colour, functional, and thermal properties of heat-moisture treated BG starch. Despite the unlimited applications of starch available, unmodified starches are unusable as they are unable to produce desirable properties, however, they can be altered to obtain those desirable qualities through various methods of modification. A study of functional, colour and thermal properties of BG starch, as affected by heat modified treatment (HMT) using response surface methodology is, thus, essential before food manufacturers and processors can think of its potential usage in food systems and large-scale production. Furthermore, there is also an increased demand by consumers for new and unique products and starch modification has brought about an evolution of new processing technologies and market trends. The usefulness of native starch cannot be over emphasised; however the modification of the native starch from agricultural products plays a major role in improving its quality and diversifying its use in the food industry.

1.3. Justification of the study

Bambara contains high levels of carbohydrates which make it a possible starch source and a low cost raw material for food industries; however, legume starches require some modifications, such as HMT to diversify its functionality (Kaur *et al.*, 2012; Oyeyinka *et al.*, 2016; Oyeyinka & Oyeyinka, 2017). The use of the starch from Bambara groundnut in the production of hydrocolloids could be advantageous because the legume is cost effective and drought tolerant. Bambara groundnut starch can contribute greatly to textural properties of various foods motivating an increase in the cultivation, processing and usage of BG in SA.

1.4. Research aim

The aim of this research work is to investigate the influence of heat-moisture treatment on functional, colour and thermal properties of starch from bambara groundnut.

1.5. Specific objectives

1. To determine the functional properties: water absorption, oil absorption, swelling power and solubility capacity of heat-moisture treated BG starch.
2. To evaluate the thermal properties of heat-moisture treated BG starch.
3. To determine the colour properties lightness/darkness (L^*), redness/ greenness (a^*), yellowness/blueness (b^*), Chroma (C^*), hue (H°), total colour difference (ΔE) and whiteness index (WI) of heat-moisture treated BG starch.
4. To optimise and determine the scanning electron microscopy (SEM) and Fourier Fourier transform infrared spectroscopy (FTIR) of functional, thermal and physical properties of heat-moisture treated BG starch.

1.6. Hypotheses

1. Heat-moisture treatment could improve the functional properties (water absorption, oil absorption, swelling power and solubility capacity) of bambara groundnut starch.
2. Heat-moisture treatment may influence the thermal properties (onset temperature, peak temperature, concluding temperature and enthalpy of gelatinisation) of bambara groundnut starch.
3. Heat-moisture treatment may influence the colour properties (L^* , a^* , b^* , C^* , H° , ΔE and WI) of Bambara groundnut starch.

CHAPTER 2: LITERATURE REVIEW

2.1. Background to the study

Grain legumes are foodstuff of high nutritional value to people in developing countries; however, most of these legumes lack adequate processing techniques to convert them into commercially acceptable products. BG (*Vigna subterranea*) is an indigenous leguminous plant that has been cultivated all over sub-Saharan Africa and has potential benefit to food and pharmaceutical industries (Gabriel *et al.*, 2013). According to farmers, there has been a decline in BG production due to lack of adequate processing techniques to promote the legume's wider utilisation and commercialisation (Mazahib *et al.*, 2013). In South Africa, BG is not only produced as a subsistence crop, but has recently been explored as an income-generating crop (Masindeni, 2006). In other Southern Africa countries, like Malawi, Tanzania, Zimbabwe and Mozambique, workshops towards the improvement, conservation and promotion of Bambara utilisation have recently been carried out aimed at enhancing the potential of BG (Hillocks *et al.*, 2012).

There are different methods of preparation that can be applied on BG to overcome its hard-to-cook effect and reduce its anti-nutrient factors. The seeds can be processed to develop acceptable flour, paste, slurry and can also be used as a complementary ingredient for maize porridge and stable shelf life products, such as extruded snacks (Okafor *et al.*, 2014; Adefalu & Fawole, 2014). In most countries it is commonly used as a food however, it has also been utilised in the manufacture of different products including, vegetable milk (Murevanhema & Jideani, 2015), puddings (Ukegbu & Uwaegbute, 2014), low fat yoghurt (Falade *et al.*, 2015), protein hydrolysate as well as peptide fractions (Arise *et al.*, 2017). BG is also a great starch source, which can potentially be incorporated by food manufacturers (Kaptso *et al.*, 2014; Oyeyinka & Oyeyinka, 2017).

2.1.1. Nutritional composition of bambara groundnut

The total nutritional composition of BG reflects an excellent balance that could play a significant part in meeting people's nutrition requirements in combined meals, particularly, in emerging countries. In a study of the nutritive evaluation of BG Ci12 Landrace [*Vigna subterranean* (L.) Verdc. (*Fabaceae*)] produced in Côte d'Ivoire, Yao *et al.* (2015) reported the presence of relatively high essential amino acids, such as lysine, isoleucine, leucine, methionine, valine, phenylalanine, and threonine. In another study conducted, Amarteifio *et al.* (2006) reported high linoleic acid content (18:2 n-6) 36% of the total fatty acids and low levels of calcium, iron, sodium and potassium. Mensah (2011) reported the essential amino acid content of BG where it was found to be 6.82 g lysine/ 16 g of nitrogen, 1.85 g methionine /16 g of nitrogen and 1.24 g

cystein/16 g nitrogen and was comparable to that of soya bean which was 6.24 g lysine/16 g of nitrogen, 1.14 g methionine/16 g of nitrogen and 1.80 g cystein/16 g of nitrogen.

Bambara is nutritionally superior to other legumes and is the preferred food crop for most people in the sub-Sahara Africa region (Brough *et al.*, 1993). The high carbohydrate (53.1%) of the seed thus, if found suitable as starch source for food products, could have tremendous nutritional advantage. De Kock (2004) also reported high carbohydrate content (54.5 - 69.3%), protein (17 - 24.6%), fat (5.3 - 7.8%) and calories (367 - 414 Kcal per 100 g). The seed was also found to be a good source of fiber (6.1%) (Sirivongpaisal, 2008). BG can essentially provide a balanced diet in rural areas where people cannot afford meat and the production of other legumes is affected by climate.

There are several types of BG, namely, black seed, red-coloured, cream-black eye, cream-no eye, brown and spotted seeds. The black seeds mature earlier than other varieties, are usually small to medium size and mainly one-seeded. Red-coloured seeds mature much later but consist of large kernels and are known for a good yield. Cream-black eyed Bambara seeds, have large kernel sizes and give a good yield. Cream-brown eyed ones, have moderate kernel size and are known to produce a good yield. Cream-no eye, consist of very small pods and kernels, it is mainly one-seeded and produce lower yields. Spotted seeds have a predominant purple colour and contain small one-seeded pods (Figure 1).

The brown Bambara variety consists of kernels of medium to large size and varies between light and dark brown (Department of Agriculture, Forestry & Fisheries, 2011). The red-coloured seeds have been reported to be beneficial in regions where deficiency in iron is a problem since they have almost doubles as much iron as the cream-coloured seeds (De Kock, 2004).



Figure 1: Varieties of bambara groundnut

Sources: <http://healthbubbles.com/gb/?p=2267>; <https://www.richters.com/show.cgi?page=SeedZoo/seedzoo4.html>; http://www.cffresearch.org/Updates-@_Growing_Bambara_ground_nut_in_Malaysia.aspx

2.2. Starch

Starch is the key and most significant energy spring in tubers, cereals, and legumes. The structure and useful characteristics of BG starch have been assessed by previous scholars (Sirivongpaisal, 2008; Ashogbon, 2013; Oyeyinka *et al.*, 2017). The starch granules of BG were found to be more circular and lesser than those existing in legumes and tend to display a little amylose content (21.7%) for legumes (Yao *et al.*, 2015). Starch is the foremost storage carbohydrate of legume seeds (Klamczynska *et al.*, 2001) and since it is the most essential carbohydrate in the human food and works as an energy basis, its health worth should be taken into account. Processing starch and starch spinoffs from conservative plants like cassava, potato, maize, and rice is a way to harness under utilised legumes as valuable sources of starch and protein concentrates.

Despite the array applications of starch obtainable, unmodified starches are unusable owing to their incapability to produce anticipated characteristics, however, they can be altered to get those desired qualities through starch alteration methods. For example, through chemical amendments and using specific moisture and temperature conditions, some physicochemical characteristics of starch can be changed. The implication of several alterations, both physical and chemical on functional and physicochemical characteristics of some under utilised legume starches have been formerly studied by researchers like, Adebowale & Lawal (2002); Adebowale & Lawal (2003); Lawal *et al.*, (2004); Adebowale & Lawal (2004).

2.2.1. Sources of starch

Cereal grains, tubers and legume seeds, are the main starch sources. Other sources of starch that have been studied include sweet potato, sorghum (white and red variety), barley and unripe fruits.

2.2.1.1. Maize

Maize (*Zea mays L*) is a vital cereal grain in the world, providing nutrients for human and animals (Dar, 2014). There are various species of maize with diverse characteristics - colour, shape and kernel size (Serna-Saldivar, 2010). Maize is processed and consumed in diverse forms in diverse parts of the world and also serves as the raw material for making corn starch, corn oil, alcoholic beverages and food sweeteners (Mishra *et al.*, 2012). Maize is a rich starch source in the world; the starch obtained for commercial purposes is found in the seed. Maize is said to contain carbohydrates (19%), sugar (3.22%) (Haytowitz *et al.*, 2011) and amylose (50%) (Be Miller, 2014).

2.2.1.2. *Potato*

The starch is obtained from the tuber of potato. The most commonly used species for starch purposes is *Solanum tuberosum*; which contains starch content of about (15-18%). Potatoes contain carbohydrates (17%), sugar (0.78%) (Haytowitz, *et al.*, 2011) and amylose content of (21-25%) (Be Miller, 2014). Potato starch contains approximately 800 ppm of phosphate ions which are bound to the starch. This gives potato starch unique characteristics including a relationship between viscosity and ionic content.

2.2.1.3. *Wheat*

Wheat is the utmost cultivated, consumed and sold grains in the world. It has varieties and constitutes a major portion of many diets due to its agronomic flexibility. Wheat can be stored easily, is highly nutritious and the ability of its flour to produce several palatable and satiating foods (Shevkani *et al.*, 2016). The industrial use of wheat includes producing starch and gluten (Maningat *et al.*, 2009). Wheat contains high carbohydrates (71%) (Haytowitz *et al.*, 2011) and this makes it a suitable source of starch.

2.2.1.4. *Rice*

Rice (*Oryza sativa*) is a widely eaten and vital cereal crop. As a grain, it is a commonly consumed staple food for most people in the world (Serna-Saldivar, 2010); it is also a great starch source. The starch extracted from rice is commonly utilised for various food and non-food uses (Reddy & Bhotmange, 2013). Regular rice contains amylose (20%), high carbohydrates (80%) and sugar of (12%) (Be Miller, 2014).

2.2.1.5. *Cassava*

Cassava (*Manihot esculenta*) is one of the major sources of food in the tropics. The cassava plant gives a high yield of carbohydrates per cultivated area against other crop plants. It also plays a significant role in agriculture in developing countries (Montagnac *et al.*, 2009). The starch is obtained from the plant root. Cassava is known to contain carbohydrate (38%), sugar (17%) and amylose content of (17%) (Be Miller, 2014).

2.2.1.6. *Legumes*

Different legumes such as pea, locust bean and Bambara are good sources of starch. This is due to their high carbohydrate and amylose content. For example peas (*Pisum sativum*) contain carbohydrates (58%) and amylose (61%) (Costa *et al.*, 2006). Bambara contains carbohydrate

content of (54.5 - 69.3%) (Mensah, 2011), while locust bean (*Parkia biglobosa*) was found to be a rich source of starch, protein, lipids, carbohydrates and soluble sugars (Sankhon *et al.*, 2013). Sankhon *et al.* (2013) also discovered that the purity of the starch obtained in the locust bean is suitable for the production of syrups with high glucose content. This implies that legume starches have the potential in the food industry.

2.2.2. Bambara groundnut starch

BG starch has been investigated and was found to contain high content of amylose with their starch granules being round (A-type pattern) and oval in shape. The starch obtained from BG shows a two stage swelling pattern with a related viscosity profile to cereal starches (Lawal *et al.*, 2004). It also displays a great resistance to acid at a pH range between 4.6 and 7.0 (Mensah, 2011). BG starch exhibits higher SP, breakdown and set back but lesser gelatinisation temperature, pasting temperature, WAC and OAC (Lawal *et al.*, 2004). Ashogbon (2014) in the study on ' physicochemical properties of Bambara starch and cassava starch blends', discovered that the starch extracted from BG contained more lipids (0.31%) and protein (0.18%) when compared to cassava starch.

The apparent content of amylose in the starch was established to be higher at (37.31%). Higher amylose contents in a starch are usually linked with formation of starch gels that are firm and hard (Novelo-Cen & Betancur-Acona, 2005). Sirivongpaisal (2008) reported protein (0.61%), fat (0.44%), ash (0.47%), fiber (0.60%) and amylose content of (21.67%, db) from BG starch which was extracted from the flour by the alkaline technique. The starch granules seemed to have an oval and round shape and the ones with round shape were small in size with a smooth granule surface. BG starch exhibited similar patterns of uneven distribution of size. These were like the findings reported by Adebowale & Lawal (2002).

Gelatinisation temperature of starch obtained from BG has been reported as 71.69 °C at the onset temperature (T_o), (75.33 °C) at the peak temperature (T_p), (79.17 °C) at the end of gelatinisation temperature (T_c) and 11.73 (J/g) for enthalpy of gelatinisation (ΔH) (Sirivongpaisal, 2008). The higher gelatinisation temperature for Bambara groundnut starch may be due to the more firm granular starch structure (Singh *et al.*, 2003). This is associated with some structural variations within the starch granules involving amylose-amylose and amylose-lipid interactions. These interfaces lessen the mobility of the amorphous region, leading to a higher temperature required for the swelling of starch hence, the disruption of the crystalline regions (Majzoobi *et al.*, 2017).

2.2.3. Starch extraction techniques

There are different methods of starch extraction which are largely classified into wet and dry milling. The process of extracting and purifying consist of the removal of protein, fat and other components to guarantee that the carbohydrate component is concentrated in the final starch product (Be Miller, 2014). Wet milling is normally applied in laboratories to obtain high purity starch whereas dry milling has broader utilisation in manufacturing industries. Dry milling encompasses the usage of ball mills, hammer mills by application of friction, collision and shear to attain higher degree of size reduction, which impacts the structure and characteristics of the starch granules (Huang *et al.*, 2008).

Dry milling has been utilised as a technique of starch separation in various legumes as well as navy bean, black bean, chick pea, lentil, pinto bean, mung bean, cow pea, smooth pea and wrinkled pea (Hoover & Ratnayake, 2002; Ovando-Martínez *et al.*, 2011; Abu *et al.*, 2006; Ratnayake *et al.*, 2001; Aggarwal *et al.*, 2004). In previous works of starch extracted from non-legume springs, ball-milling application has been described to lessen the gelatinisation temperature and enthalpy but upsurge the apparent content of amylose, solubility in water and clearness (Huang *et al.*, 2008). The limpiness of isolated starch gotten by wet milling is lower than that of dry milling. Wet milling procedure includes soaking of dehulled seeds in distilled water and the process is usually used for the retrieval of starch from legumes (Abu *et al.*, 2006).

Starch extraction in dissimilar legume sources has been previously done using dry and wet milling; wet milling has been applied in diverse studies for extraction of starch in beans, lentil and cowpea. A yield of isolated legume starches from kidney bean, chick pea, green gram, lentil, moth bean, smooth pea, black bean, pinto bean, navy beans, cow pea, faba bean and black gram has been reported by Wani *et al.* (2016) and ranged from 16.4 - 47.1%, being highest in kidney bean, chick pea, lentil and green gram. Cereals have starch in abundance as compared to legumes (Xue *et al.*, 1996). This disparity in yield from various sources is possibly due to the dissimilarities in species and cultivars, starch granule relations with other components, physical state of the seed and the techniques for extraction.

2.2.4. Applications and uses of starch

Starch is also one of the utmost imperative raw materials for industrial use. It has limited use in its native granular form, however, through the use of chemical and physical alterations, starch has been applied in a broad assortment of industrial uses including, as food ingredients, sizing agents for paper, textiles, and starch-based plastics. The contribution of starch is related to water absorption, gelatinisation and retrogradation capacity. There are several factors that

influence starch which need to be considered; the factors include, the desired texture of a food, viscosity and consistency during the processing and food formulation including other ingredients used. Overall water concentration has a significant impact as starch requires free and available water to gelatinise (Dar, 2014).

In food industries, unmodified and modified starches are used for one or more of the following purposes: in formulation of baked goods, such as cakes, muffins, pastry and cookies to manage viscosity and texture. It is also used in batters and breading to improve adhesion of coating and in beverages such as coffee creamer for enhancing creaminess. Starches provide crispiness and crunchiness in extruded cereal products and help manage moisture during production and storage of confectionery (Stephen *et al.*, 2006). It is used directly in cooked starch food, such as custard, as a filler and thickener in paste, soups, baby foods, sauces and gravies and a filler in pills, tablets as well as other pharmaceutical products. In dairy products such as yoghurt and sour cream, starches maintains their shelf life and give textural characteristics while working as a binding component to combine mass and inhibit drying out while cooking in sausages and processed meats (Dar, 2014). Starch is also applied as a glazing agent, viscosity and texture modifier. Starch and its derivatives are also used in snacks to help achieve various textural qualities.

Different starches are responsible for different functional qualities in snack products, including hardness, cohesiveness, melting ability and overall eating quality (Haung & Rooney, 2001). Normal maize and high amylose maize starches are added when more crunchiness and strength are desired for the final product (Liu, 2005). Other industrial applications of starch include paper making, corrugated paper board for packaging and in clothing for maintaining stiffness. Starches are used as binders in book binding, wall paper, paper sack production, bottle labeling and glue production. They are also used as a thickener in personal care products such as hair creams (Dar, 2014). Starch is used in cosmetics, sanitary products, plastics used for food packaging and as biodegradable filler (Xie *et al.*, 2005). Table 1 below shows major sources of starch and their applications.

Table 1: Major sources of starch and their applications.

Starch source	Application of starch	References
Maize	Fillers in capsules, texture, moisture and cooking yield	Olu-Owolabi <i>et al.</i> , 2010; Altunakar <i>et al.</i> , 2004
Cassava	Glucose production	Hermiati <i>et al.</i> , 2012
Sorghum	Gelling agent, thickening in sauces and strength enhancing	Adebowale <i>et al.</i> , 2005; Olayinka <i>et al.</i> , 2006
Rice	Food, and industrial uses	Reddy & Bhotmange, 2013
Potato	Gel and paste formation	Be Miller & Whistler, 2009
Legumes	Modified to resistant starch, improve organoleptic properties of food such as texture	Tharanathan & Mahadevamma, 2003
Tapioca	Texture, moisture, oil pickup and improve volume	Altunakar <i>et al.</i> , 2004
Wheat	Thickening, stabilizing, adhesive, gel forming agent, water-holding and bulking agent	Shevkani <i>et al.</i> , 2017
Sweet potato	Noodle production, texture and appearance	Tsakama <i>et al.</i> , 2013

2.3. Starch properties

2.3.1. Chemical properties

Starch granules are consisting of amylose and amylopectin that make up to 98-99% of the starch's dry weight (Sulaiman, 2011). Amylose is a direct sugar component linked by alpha (1 - 4) linkages and amylopectin is a split structure with sugar blocks merged by alpha (1 - 6) linkages at the branch points and alpha (1 - 4) linkages in the linear parts (Pepe *et al.*, 2015). The lipid component of native starches is relative to the amylose component as the complex the amylose content, the more lipids are present in the native cereal starches (Copeland *et al.*, 2009). Based on most studies reported, amylose, protein, lipid, ash and moisture contents of different legume starches varies. These disparities are known to occur because of numerous reasons, such as cultivar differences, methods of estimation and environments of growth (Ashogbon, 2014). Table 2 gives a summary of some chemical properties of cereal and legume starches

Table 2: Chemical composition of cereal and legume starches (%)

Starch source	Ash	Lipid	Moisture	Amylose	Protein	Reference
Bambara groundnut	0.1-0.2	0.1	12.3-12.8	25-27.8	0.9-1.1	Kaptso <i>et al.</i> , 2015
Chick pea	0.05–0.06	0.29–0.50	8.78–11.45	23.00–33.81	0.03	Wani <i>et al.</i> , 2016; Haung <i>et al.</i> , 2008
Kidney bean	0.25–0.50	0.10	10.88–11.00	42.96–49.28	0.08–0.10	Wani <i>et al.</i> , 2016.
Corn	–	0.7-0.8	–	28	0.35	Yuryev <i>et al.</i> , 2004
Wheat	–	0.8-0.9	–	28	0.4	Yuryev <i>et al.</i> , 2004
Lentil	0.03–0.25	0.09–0.40	8.90–9.40	22.10–33.90	0.03–0.09	Wani <i>et al.</i> , 2016
Green gram	0.25–0.50	0.10–0.32	10.63–11.96	34.47–45.30	0.02–0.05	Wani <i>et al.</i> , 2016; Andrabi <i>et al.</i> , 2015
Moth bean	0.62	0.87	9.48	26.42	0.06	Wani <i>et al.</i> , 2016.
Smooth pea	0.62	0.04	3.12	22.00–27.90	0.03–0.04	Wani <i>et al.</i> , 2016
Black bean	0.14	0.20–0.50	16.00	27.20–39.30	0.04–0.07	Wani <i>et al.</i> , 2016
Pinto bean	0.06	0.12	16.00	31.30–37.40	0.05–0.07	Wani <i>et al.</i> , 2016
Waxy corn	–	0.15-0.2	–	0	0.25	Yuryev <i>et al.</i> , 2004
Potato	–	0.05-0.1	–	21	0.06	Yuryev <i>et al.</i> , 2004
Tapioca	–	0.1	–	17	0.1	Yuryev <i>et al.</i> , 2004
Faba bean	0.03	0.08–40	–	17.00–42.00	0.33–0.43	Wani <i>et al.</i> , 2016; Gunasekera <i>et al.</i> , 1999
Cow pea	–	0.20–1.33	–	25.80–33.00	0.06–0.09	Wani <i>et al.</i> , 2016; Gunasekera <i>et al.</i> , 1999; Haung <i>et al.</i> , 2008

2.3.2. Functional properties

2.3.2.1. Swelling and solubility index

Swelling power (SP) is the hydration ability of starch at raised temperatures. SP of starch is prejudiced by several aspects such as the relations between starch chains inside the amorphous region and crystalline region of the granule, amylose content, amylose–lipid complexes and the molecular arrangement of amylopectin (Wani *et al.*, 2016; Zhou *et al.*, 2004). When starch is placed in hot water, the granules absorb water and swells; the inner bonds and binding forces of starch granule are then weakened by the attained thermal energy. Swelling and solubility of the starches are directly influenced by the ratio of the crystalline and amorphous constituents of the starch granules (Wani *et al.*, 2010).

High SP and solubility index are ascribed to the moveable granule configuration, low molecular mass of amylose of the starch, amylopectin ratio and molecular features like chain length, branching, micellar structure in the starch granule and the existence of lipids that form complexes (Nwokocha *et al.*, 2010). Adebowale *et al.* (2006) in a study on ‘the functional, physicochemical and retrogradation features of sword bean’s (*Canavalia gladiata*) acetylated and oxidized starch’, observed that starch granules with a wide and strongly-bonded micellar structure demonstrate a comparatively high resistance towards swelling, which may eventually result to a lesser solubility index.

The SP of starch from dissimilar legumes has been reported to be exaggerated by the temperature and pH of the liquid starch slurries. This is because the application of thermal energy relaxes the strong intra-granular interactions, which therefore cause disruption in molecules of starch granules (Hoover *et al.*, 2010; Wani *et al.*, 2016). Studies have reported that legume starches better improve functional properties than cereal and potato-based starches (Adebowale & Lawal, 2004). It can be deduced that starches from legumes may attain a comparable extent of swelling and solubility as that of starches from cereals at higher temperatures. SP and solubility index provide indication of the extent of associations between starch chains in the amorphous and crystalline region.

2.3.2.2. Water absorption capacity

Water absorption capacity (WAC) is the quantity of water that can be apprehended by starch. WAC is a function of numerous structures comprising of the existence of hydrogen bonds, level of accessibility of water binding sites between the starches, conformational properties, stearic factors and hydrophilic–hydrophobic balance (Wani *et al.*, 2010; Wani *et al.*, 2016). Studies have revealed that water binding can be affected by the physical and chemical environment of

starch suspensions which include pH, temperature, ionic strength, vapor pressure, and the existence or nonexistence of a surfactant (Hoover & Sosulski, 1991). WAC is a starch property as it contains hydrophilic sites in its molecular chains.

When more hydrophilic sites are easily available to water for bonding during hydrogen bonding, the higher the WAC. Temperature greatly affects water absorption of starch because when the temperature rises, the molecules obtain enough thermal energy. The thermal energy makes the intermolecular hydrogen bonds weak and increase bonding and affinity towards water; due to the starch granules integrate with water (Garg & Jana, 2011). Wani *et al.* (2016) reported the water absorption capacity of blackgram starch, cowpea starch, sword bean starch, mucuna bean starch and ground nut starch. It ranged from 1.21 - 3.04 g/g with mucuna bean having the lowest and blackgram starch having the highest WAC.

2.3.3 Thermal properties

2.3.3.1 Gelatinisation parameters

When starch is heated in water over a certain temperature range, it undergoes a disorder-phase change called gelatinisation. The phase change is related to the dispersion of water into the granule. This is shadowed by water uptake, hydration and radial swelling of the starch granules, damage of birefringence and crystallinity, heat uptake, detachment of double helices and amylose leaching (Maaran *et al.*, 2014). The gelatinisation transition processes' temperatures: onset, peak, end set and enthalpy, have been known to be prejudiced by the molecular makeup of the crystalline region (Waigh *et al.*, 2000). The gelatinisation features of starch are usually ascertained by differential scanning calorimeter (DSC).

The gelatinisation temperatures of starch from non-legume sources such as sorghum, cassava, maize and arrowroot have been investigated (Olayinka *et al.*, 2008; Moraes *et al.*, 2013; Olu-Owolabi *et al.*, 2010; Cahng *et al.*, 2001; Pepe *et al.*, 2015). Comparable gelatinisation physiognomies amid the legume, cereal and tuber starches indicate that dissimilar temperatures are essential to begin and finish gelatinisation process in starch, from diverse sources (Ubwa *et al.*, 2012). The variations in the gelatinisation parameters as observed in Table 3 owing to factors such as inconstant amylopectin content and distribution, level of crystalline perfection and fraction of lipid complexed amylose chains (Waigh *et al.*, 2000). Starch pastes have characteristics such as clearness, viscidness, texture, firmness and tastefulness; these characteristics also rest on the extent of gelatinisation. The gelatinisation parameters of a starch, therefore, is significant in relating and regulating the performance of diverse starches in industrial cooking and processing.

Table 3: Thermal properties of legume starches

Starch source	Gelatinisation Parameters			
	T _o (°C)	T _p (°C)	T _c (°C)	ΔH J/g
Kidney bean	50.8–66.9	66.7–116.3	71.0–142.4	8.6–15.4
Green gram	54.8–69.6	67.0–118.1	72.1–137.6	7.9–18.5
Black bean	61.0–70.59	69.9–82.15	80.43–88.13	10.43–13.96
Black gram	66.1–71.3	71.0–76.2	76.0–80.4	6.7–9.4
Lentil	57.8–68.4	66.0–76.1	71.0–82.0	3.0–13.3
Navy bean	61.0–66.8	68.6–75.1	71.0–91.0	8.8–15.3
Cow pea	70.5–72.7	75.4	81.0	15.2–16.9
Faba bean	62.0–65.2	66.67–70.59	73.62–76.46	7.84–10.25

T_o = Onset temperature; T_p = Peak temperature; T_c = Concluding temperature; ΔH = Gelatinisation enthalpy
 Source: Wani *et al.*, 2016.

2.3.3.2. Retrogradation

Starch retrogradation is defined as variations in the physical behavior next to gelatinisation. The process happens when molecules of the starch re-associate and become an organised structure, such as double helices during storage (Liu, 2005; Wang & Copeland, 2013). Retrogradation is essential when starch is utilised in food industries, as it is a wanted end point in most applications (Wang *et al.*, 2015; Wang & Copeland, 2016). The process can also cause variability in starch pastes therefore, some structural modifications by genetic means, chemical or physical alteration of starch to change the pathway of starch biosynthesis must be applied to alter this process (Liu, 2005).

Starch retrogradation is prejudiced by the vegetal source, the molecular structure of amylopectin, granular architecture and amylose-amylopectin ratio (Gunaratne & Hoover, 2002). Size of the molecules and size dispersal of starch, moisture content in the starch gel and storage temperature can also influence the rate and degree of starch retrogradation. Liu & Thompson (1998) reported that retrogradation of various maize starches can be affected by 50 to 80% moisture content and preliminary heating temperature. The variation in retrogradation characteristics of these starches is probably connected to the dissimilar amylopectin structures.

2.3.4. Colour characteristics

Colour characteristics depict quality parameters and indicate some internal ingredients of food materials (Pathare *et al.*, 2013). Hue, saturation and brightness are three features of light through which a colour is identified. Hue is a feature connected to the overriding wavelength in a combination of light waves and signifies the overriding colour as observed by an observer (Grano & Masson, 2009).

Saturation is defined as the quantity of white light fused with a hue or relative purity. Brightness is an independent term, which represents the chromatic view of concentration. Hue and saturation are together regarded as 'chromaticity' (Jha, 2010). A colour may be categorized, therefore, by means of brightness and chromaticity. All colours that can be visually seen can be plotted in this L^* , a^* , b^* , rectangular colour space. L^* designates lightness, 0 - 100 with 0 depicting black and 100 signifying white. Coordinate a^* relates to red (positive values) and green (negative values) while b^* indicates yellow (positive values) and blue (negative values) (Macdougall, 2002). Hue angle (H°) indicates quality of lightness or darkness while C^* (Chroma) represents the quality of colour purity. Hue indicates 100 quality spread out hues round a circle; saturation represents elements of chroma starting at 0 on the center line and snowballing to values of 10 to 18 dependent upon the hue. Some hues contain unique stages of saturation; brightness represents value from 0 for black to 10 for white (Granato & Masson, 2009).

Availability of an extensive range of methods for measuring quality parameters requires one to recognize the appropriateness and the nature of samples (liquid, solid, paste, semi-solid, transparent, opaque or translucent) for a specific method to be used. This is because light trapped in a luminous sample, for example, may alter the colour, therefore, the sample's thickness should be selected to maximize the haze (Jha, 2010). Studies on colour have been done by Tijskens *et al.* (2001); Schouten *et al.* (2007); Jha *et al.* (2007) on different Food produce. Tijskens *et al.* (2001) reported a significant change in green vegetables' colour during heat treatments (blanching). Schouten *et al.* (2007) observed a limit in postharvest life of tomatoes with colour change and also correlated the level of colour and biological harvest age. Jha *et al.* (2007) studied the maturity index of mango. There is, however, very limited literature on works done on starch colour characteristics.

2.4. Modification of starch properties

The molecular prearrangement in a starch granule can be modified by applying numerous physical treatments. Such treatments include annealing and heat-moisture treatment (HMT). These physical treatments can alter certain starch features by means of simple and ecologically harmless processes (Adebowale *et al.*, 2005). Annealing is commonly usually done by heating starch with a huge amount of water at a temperature lower than the melting point of starch and HMT is carried out at limited moisture contents between 18% and 35% but at an elevated temperature (Kittipongpatana & Kittipongpatana, 2015).

2.4.1. Annealing

Starch annealing is defined as a physical treatment of starch granules in excess water (> 60 % w/w) or WC of (40 % w/w) and heat. During this process, starch granules are kept at a temperature higher than the glass transition temperature but lower than the T_0 of gelatinisation for a certain period of time (Tester & Debon, 2000). It has been reported that annealing enhances the thermal steadiness and lessen the amount of set-back in starches (Adebowale & Lawal, 2002). Annealed starches can be used for the improvement of frozen and canned food products. The process can also be used in preparation of rice noodles, produced from long-grain rice that has been in storage for a very longtime.

This storage is known to limit the swelling of the starch granules but enhance the quality of starch gel and paste. The decrease of swelling in starch granules and amylose leaching, as values of 10 to 18 dependent upon the hue. Some hues contain unique stages of saturation; brightness represents value from 0 for black to 10 for white (Granato & Masson, 2009).

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This storage is known to limit the swelling of the starch granules but enhance the quality of starch gel and paste. The decrease of swelling in starch granules and amylose leaching, as well as the improved shear and heat stability caused by annealing all form part of the desired properties in the production of noodles (Zhou *et al.*, 2004). Horndok & Noomhorm (2007) explored the use of annealed and native rice starches as substituting ingredients for the production of high quality noodles. In the study, the textural quality such as stretchability, chewability and adhesiveness of the rice noodle produced from annealed rice starch was similar to that of commercial noodles.

2.4.2. Heat moisture treatment

Heat-moisture treatment (HMT) is defined as a hydrothermal alteration in low moisture contents of not more than 35% W/W, and subjection of starch granules at temperatures higher than the glass transition temperature (T_g) but lower than the onset (T_o) temperature of gelatinisation for a certain period of time (15 min to 6 h) (Gunaratne & Hoover, 2002). Changes caused by heat moisture treatment have been studied in maize, cassava, potato, sweet potato, waxy corn, wheat, amylo-maize, barley, oat, and some legumes starches. The importance of the effect of the process was associated to the MC level during HMT and the source where starch was extracted (Lawal *et al.*, 2004). Two types of HMT are generally used in the modification of physical and chemical properties of starch. Annealing is a type of HMT which includes subjecting the starch to excess MC levels while the other involves the addition of restricted MC levels for the modification of starch (Adebowale *et al.*, 2005). Depending on the conditions applied in HMT, starch solubility, water absorption, oil absorption, swelling power, gelatinisation temperature and changes in colour characteristics may occur.

2.4.2.1. Uses and applications of heat moisture treatment (HMT)

HMT changes some starch properties by applying simple and environmentally safe procedures which are commonly utilised in the food industry to produce quality products (Olayinka *et al.*, 2006). Such products include noodles, baked goods and pastes.

Noodle Manufacture

The use of HMT on production of different starch noodles has been investigated. These include, unmodified starch noodles, sautéed noodles as well as plain boiled noodles prepared from 100% HMT (27-30% MC for 3 h, at 110 °C). Normally sweet potato starch is shown to be unacceptable for the production of Bihon type noodles due to their sticky nature and adherence to each other when dried and on rehydration (Collado *et al.*, 2001). However, when 50% HMT sweet potato starch and 50% maize starch were used in production of noodles, the quality obtained was shown to be comparable to that of commercial Bihon-type noodles with regard to color and texture. Some noodles have been produced from sweet potato starch by HMT (with 30% moisture at 110 °C) (Tsakama *et al.*, 2013).

Sago starch has also been used in the manufacture of noodles prepared from HMT (25% moisture, at 110 °C, for 16 h); the end product displayed a higher elasticity and firmness with lower stickiness in comparison to those prepared from native starch. Lower cooking loss, increased cooking time, and lower rehydration weight was also reported (Purwani *et al.*, 2006). From the sensory evaluation of the sautéed noodles, the 100% HMT sweet potato starch noodles was the most liked due to their bursting flavor and distinct chewiness (Collado *et al.*, 2001). This shows that application of heat moisture treatment on noodles produced from different starch sources can improve not only the quality but the acceptance of the final product.

Baking

Heat moisture treatment has been applied on cake and bread production to improve quality and texture of the final product. Different scholars have investigated the impact of HMT on certain attributes of cakes and bread. Miyazaki & Morita (2005) investigated the effect of heat moisture-treated maize starch on the properties of dough and bread. The study showed that the bread specific volume and the softness of crumbs baked with HMT maize increased. The firmness of crumb baked with HMT maize in the presence of shortening and absence of shortening remained the same. The interactions between starch chains and amylose-lipid interactions in HMT may have decreased the gluten-starch interactions.

Processed Foods

HMT enhances the thermal stability, resistance to shear, and acid stability in most starches. Therefore, it is used as an alternative to chemical modification in canned foods, dressings, batter products and confections (Hoover, 2010).

2.4.2.2. Effect of heat moisture treatment on some properties of various starches

Heat moisture treatment induces changes in the crystalline regions as well as in the amorphous regions in starch granules; this leads to modification of some properties of the starch. This process helps in the formation of double helices, and thus restricts solubility and swelling of starches (Lawal, 2005). According to Jyothi *et al.* (2010), the process also causes further relations among amylose to amylose and amylose to amylopectin. This results in a solid granule structure relatively responsible for the decrease in swelling power. The formation of amylose-lipid complexes in starches has also been associated with reduction of the swelling capacity as amylose inhibits swelling of the starch where amylose-lipid complexes may be formed (Morrison *et al.*, 1996). According to Hoover (2010) the structural modifications in the starch, after HMT might also be the cause of the reduction of starch solubility and swelling power.

Adebowale *et al.* (2005) in their study on the effect of heat moisture treatment and annealing on physicochemical properties of red sorghum starch, reported that different types of starch swells in different ways, demonstrating dissimilarities in the molecular organisation within the granules. The authors further suggested an association between temperature and swelling power; this shows an increase in SP of starch as temperature increases. HMT decreases the solubility of starch due to interactions between amylose-amylose and amylopectin-amylopectin chains during hydrothermal treatment (Olayinka *et al.*, 2008; Sarkar, 2016). HMT allows the amylose molecules positioned in the bulk amorphous regions to bond with the branched amylopectin segments in the crystalline regions (Hoover & Manuel, 1996). Sarkar (2016) reported an increase in water absorption capacity of starches by HMT. This suggests that hydrophilic tendency increases with an increase in levels of HMT. In the study of arrowroot starch, HMT hardly changed the WAC and solubility index values; however, decreased in SP and solubility at 90 °C.

This suggests that HMT caused a reordering of starch chains which acceptable for the repositioning of bond forces of the starch chains (Chung *et al.*, 2009; Gunaratne & Hoover, 2002). Pepe *et al.* (2015) reported a decrease in solubility at 90°C but an increase when treatment time was increased. HMT also decreased oil absorption capacity in wheat and potato starches (Kulp & Lorenz, 1981) but, led to an increase in buckwheat and cassava starches because of the

amorphous region in the starch granules (Sarkar, 2016). Collado & Corke (1999) modified sweet potato starch and reported a short and sheer-stable starch paste, as well as a hardened and highly adhesive starch gel after modification.

2.4.2.3. Effect of heat moisture treatment on thermal properties

The effect of HMT has been investigated on different starches including normal, dull waxy, waxy, and amylo maize (Jaranuntakul *et al.*, 2011; Chung *et al.*, 2009) and rice starches (Anderson & Guraya, 2006; Arns *et al.*, 2014; Khunae *et al.*, 2007). The thermal properties, gelatinisation and retrogradation are affected by application of HMT in varying ways depending on the starch being investigated. HMT has been reported to increase gelatinisation temperatures but decrease enthalpy change of modified starches, despite the treatment time used. These suggest that HMT increases the relations between chains of the starch, and higher temperatures are required for the reordering of the crystals. According to Cooke & Gidley (1992), enthalpy characterises mainly the loss of the double helices order other than the loss of crystallinity during gelatinisation.

Pepe *et al.* (2015) reported a decrease in the enthalpy change of arrowroot starch after HMT. The study showed that the treatment possibly leads to an unwinding of double helices. HMT increased the onset, peak and concluding temperature of pea and lentil starches with increase in temperature (Chung *et al.*, 2010). This has been due to the changes in the structure of granules of the starch and the interaction between branched fragments of amylopectin in the crystalline regions and amylose molecules located in the bulk amorphous regions (Hoover & Vassanthan, 1994). Such interactions, therefore, reduce the movement of the amylopectin chains, leading to increase in onset, peak and concluding temperatures.

Lim *et al.* (2001) suggest that the onset temperature of HMT starches increases due to the modification of the inter-crystalline parts into amorphous phases. This is because fractional helices are formed by amylose chains with the shorter chains on the edges of the crystalline micelles; therefore, giving rise to a higher onset temperature. A decrease in enthalpy change of rice starches with increasing MC level of HMT suggest that some double helices in the crystalline and in non-crystalline regions of the starch may be disordered during HMT; therefore, fewer double helices would untangle and dissociate during gelatinisation of HMT starches (Gunaratne & Hoover, 2002).

The decreased enthalpy may also be as a result of the modification in the inter-crystalline region to an amorphous state, and so the crystalline regions can liquefy easily (Lim *et al.*, 2001). A major increase in the gelatinisation range has been reported for most of treated rice starches.

The gelatinisation range enlarged with an increase in MC of HMT. This is due to the occurrence of a biphasic endotherm in heat moisture-treated rice. According to Lim *et al.* (2001) increase in gelatinisation was caused by modification of the inter-crystalline portions into amorphous phases during HMT. An expansion of the gelatinisation endotherm has also been reported for HMT potato, wheat, mucuna bean and corn starches (Khunae *et al.*, 2007).

When a gelatinised starch mixture is cooled, it begins to retrograde. Starch retrogradation is a non-equilibrium thermo adjustable recrystallisation procedure, which takes place in three successive stages known as nucleation, propagation and maturation (Ambigaipalan *et al.*, 2013). The process is accompanied by an increase in crystallinity, turbidity and firmness of the gel as well as formation of a 'B' X-ray diffraction pattern. This is because crystallisation increases granule rigidity and improves their strengthening of the amylose matrix (Hoover, 1995; Ambigaipalan *et al.*, 2013). Retrogradation during HMT (at 100 °C, for 16 h, with 30% MC) on wheat, potato, oat, and lentil starches by DSC has shown that the retrogradation endotherm appeared after 3 days (Hoover & Vasanthan, 1994).

During storage the development and adjacent connection of double helices linking amylopectin chains is stronger and occurs quickly in HMT starches than in native starches. HMT enhances amylose-amylopectin interactions due to an increase in chain mobility, resulting in less disruption of hydrogen bonds of the starch during gelatinisation (Hoover, 2010). The DSC studies by Takaya *et al.* (2000) also showed that HMT (under high humidity, for 20 min, at 120 and 130 °C) of maize starch increased the level and rate of retrogradation during storage at 5 °C, while X-ray diffraction studies displayed that retrogradation of potato and maize starches is significantly increased by HMT (under high humidity, for 20 min at 125 °C for maize and potato at 110 °C potato for 30 min).

This is possibly due to recrystallisation and a link of short chains resulting from the degradation of the exterior chains of amylopectin during the treatment (Miyoshi, 2002). Adebowale & Lawal (2003) reported during DSC studies on mucuna bean starch, that HMT (18 - 27% moisture, at 60 - 90 °C) decreased retrogradation in starches stored at 30 °C for two and seven days. It can be recognised that the conditions of storage and those of heat moisture treatment have an impact on retrogradation of starch from different sources.

CHAPTER 3: MATERIALS AND METHODS

3.1. Source of material

Bambara groundnut used for this research were matured, dried and obtained from Tshimbupfe area, Limpopo province. The crop was planted in the first week of January 2017 and harvested in the second week of April 2018. No special irrigation was used for growing the BG crops. Compost from dried maize plant and chicken manure were used on the soil where BG was planted as it was intercropped with other crops namely; maize and peanuts. When the seeds were harvested they were simply sun dried. The seeds were sorted, cleaned and starch was extracted.

3.2. Experimental design

A central composite rotatable design comprising of two independent factors (temperature and time) was used for the study. Dependent response variables for this study consist of colour characteristics, functional properties and thermal properties. Table 4 shows the upper and lower limits of the independent factors as required for the generation of experimental runs for the study. The central composite rotatable design was generated using Design-Expert software version 8.0.1.0.

Table 4: Levels of independent variables used for central composite rotatable design

Sample	Code	Temperature (°C)	Time (min)
Bambara starch	-1	80	30
	1	120	90

3.3. Starch extraction and heat-moisture treatment

Bambara groundnut starch was extracted according to the method by Adebowale & Lawal (2002) while HMT was done according to the methods of Kittipongpatana & Kittipongpatana (2015) and Sacilik *et al.* (2003) as shown in Figure 2.

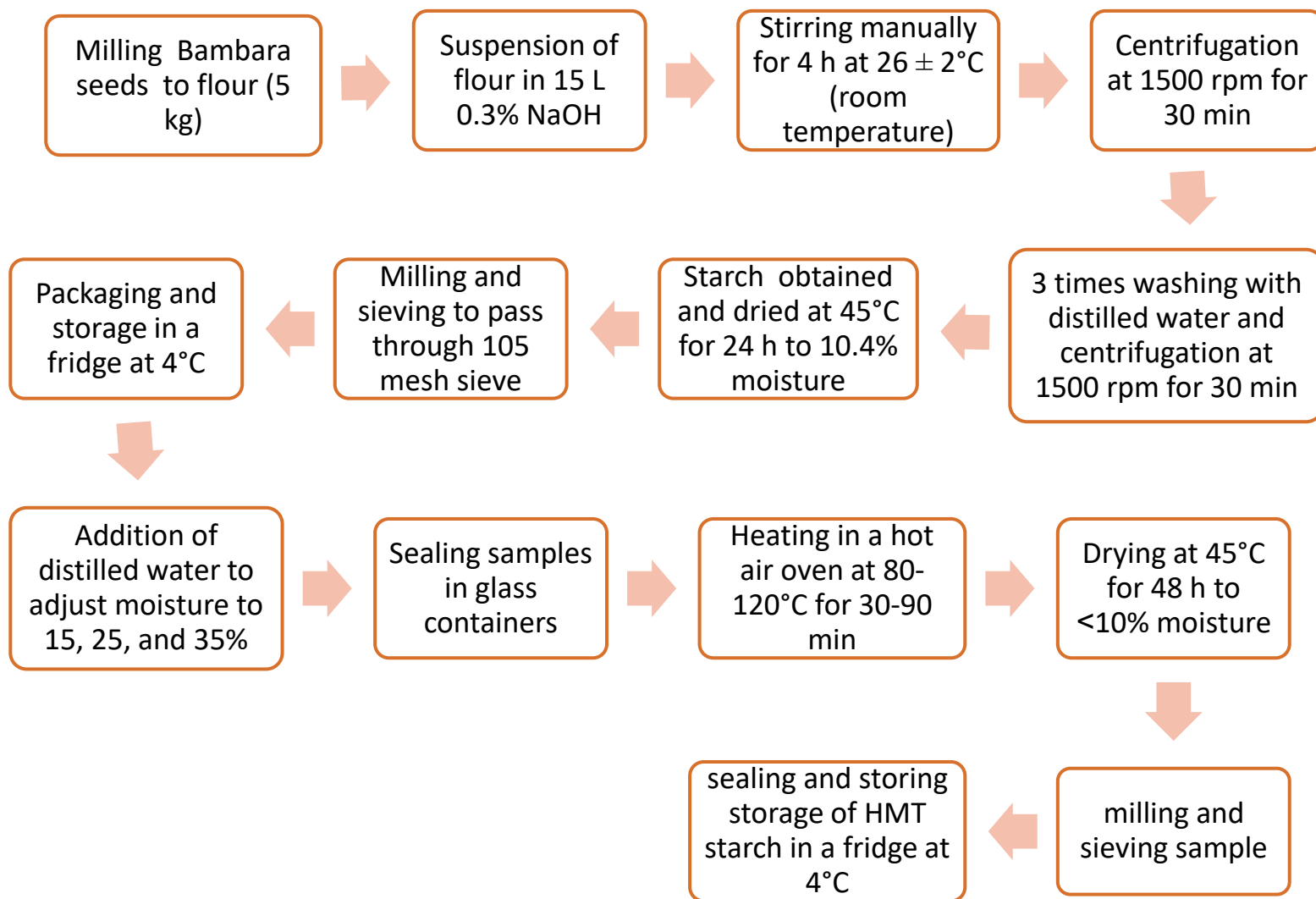


Figure 2: Process diagram illustrating extraction and heat moisture treatment of bambara groundnut starch

Figure 3 is the starch extracted from BG seeds while Figures 4 - 6 represents the heat moisture treated starches under different HMT conditions of moisture (15 - 35%), temperature (80 - 120 °C) and time (30 - 90 min).

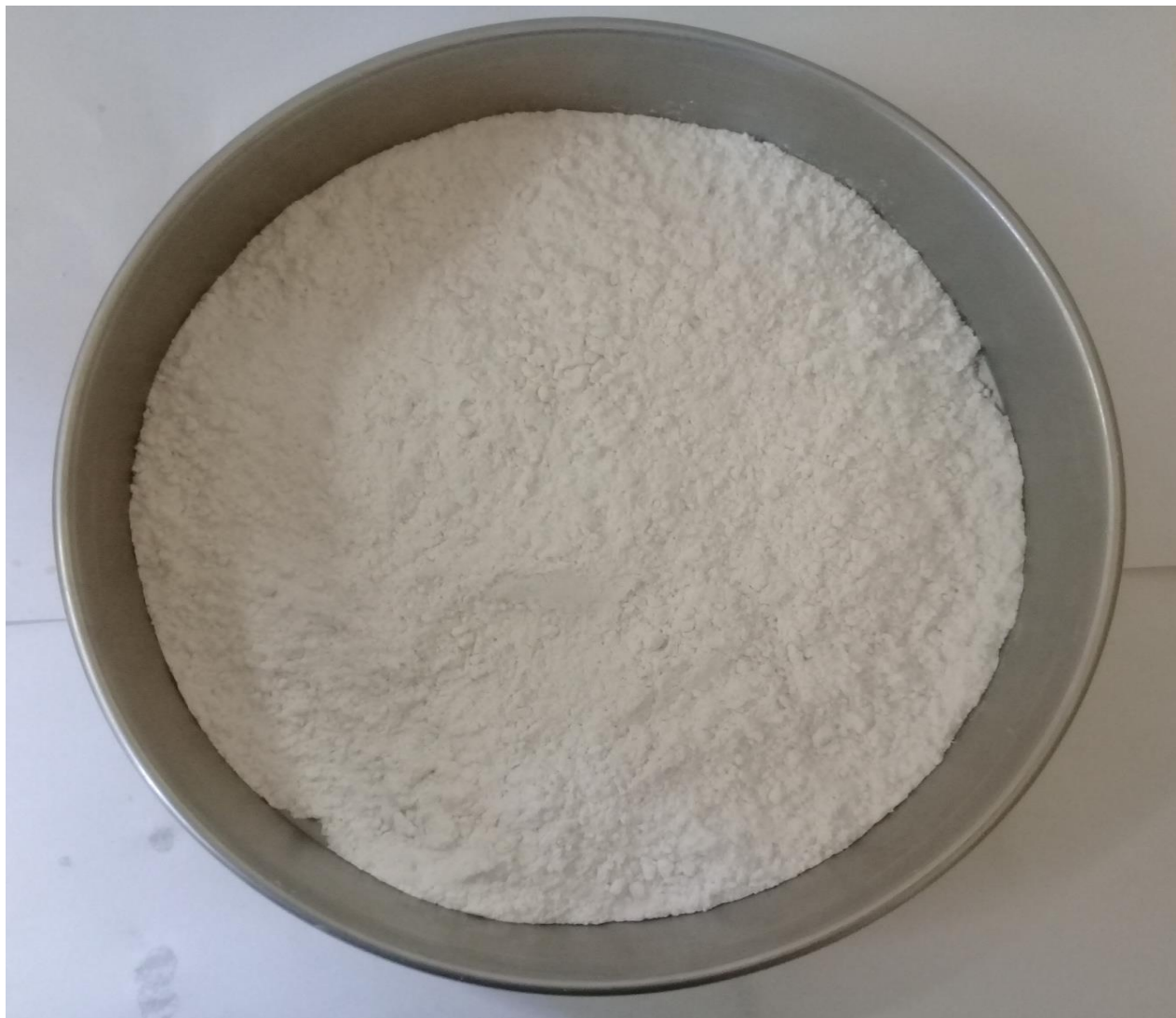


Figure 3: Extracted Bambara groundnut starch (unmodified)

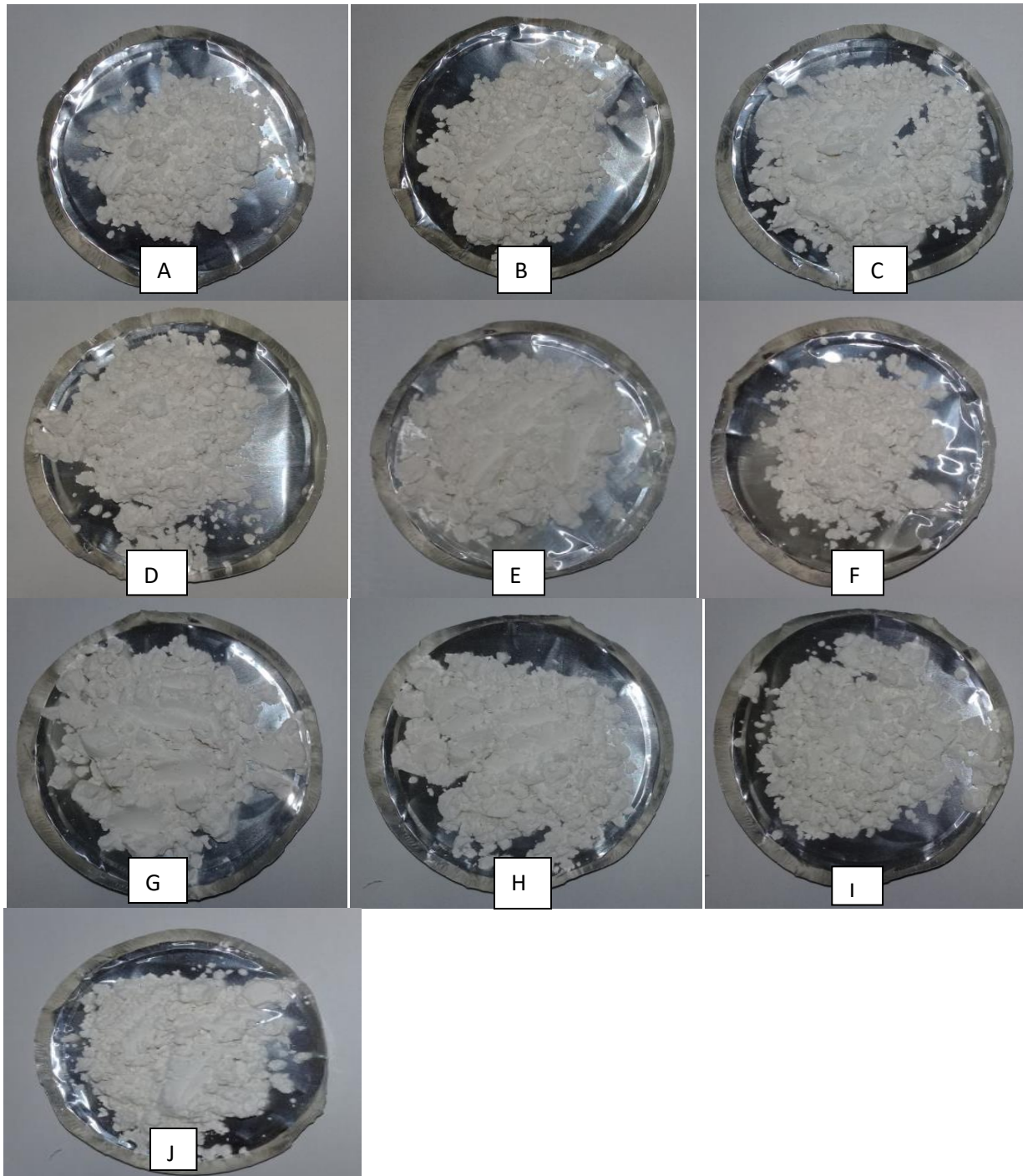


Figure 4: Bambara groundnut starch (HMT 15) heat treated under different conditions. A = 100 °C for 17.57 min; B = 100 °C for 60 min; C = 100 °C for 60 min; D = 100 °C for 102.43 min; E = 120 °C for 30 min; F = 120 °C for 90 min; G = 80 °C for 30 min; H = 80 °C for 90 min; I = 71.72 °C for 60 min and J = 128.28 °C for 60 min HMT conditions.

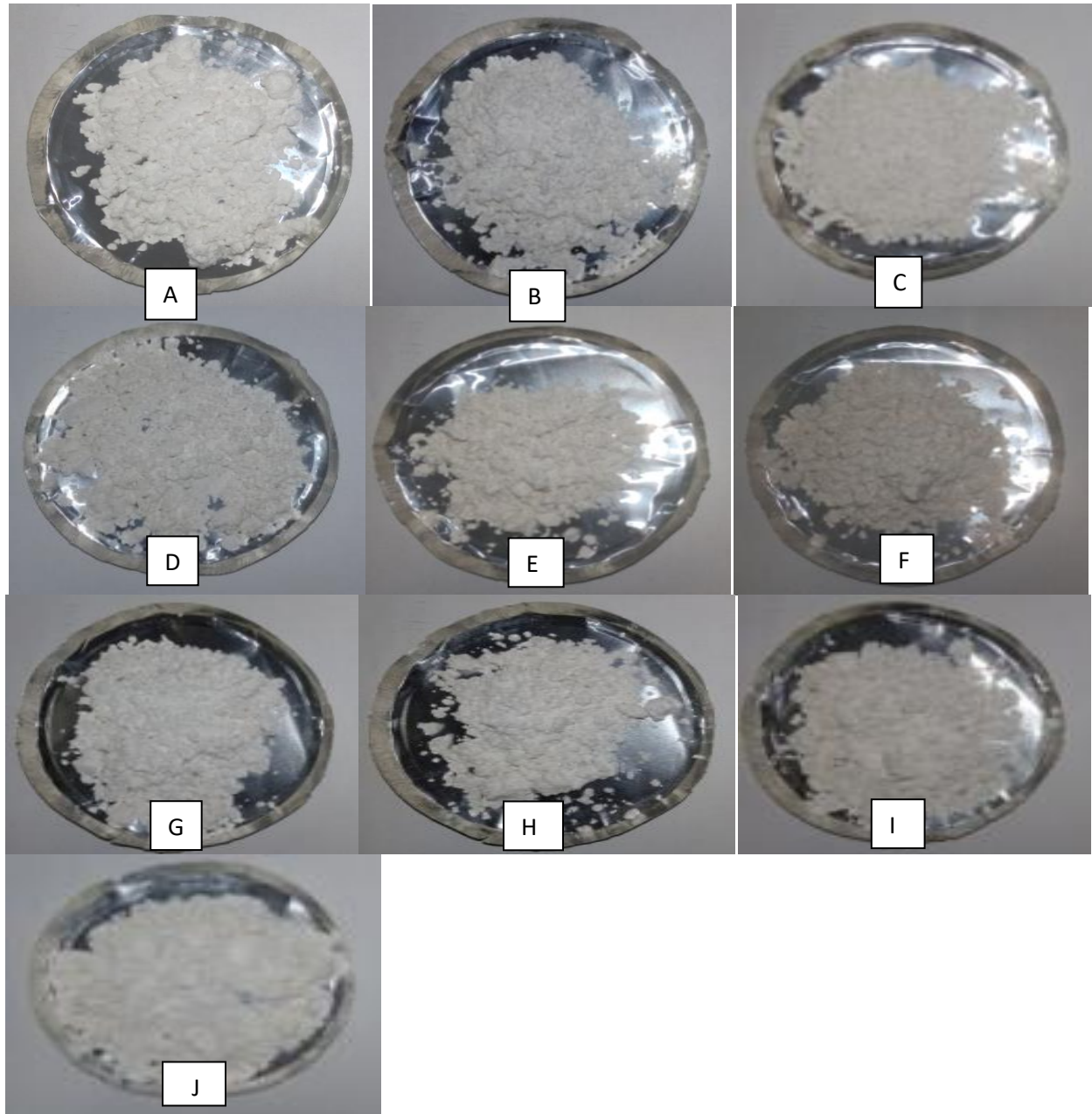


Figure 5: Bambara groundnut starch (HMT 25) heat treated under different conditions. A = 100 °C for 17.57 min; B = 100 °C for 60 min; C = 100 °C for 60 min; D = 100 °C for 102.43 min; E = 120 °C for 30 min; F = 120 °C for 90 min; G = 80 °C for 30 min; H = 80 °C for 90 min; I = 71.72 °C for 60 min and J = 128.28 °C for 60 min HMT conditions.

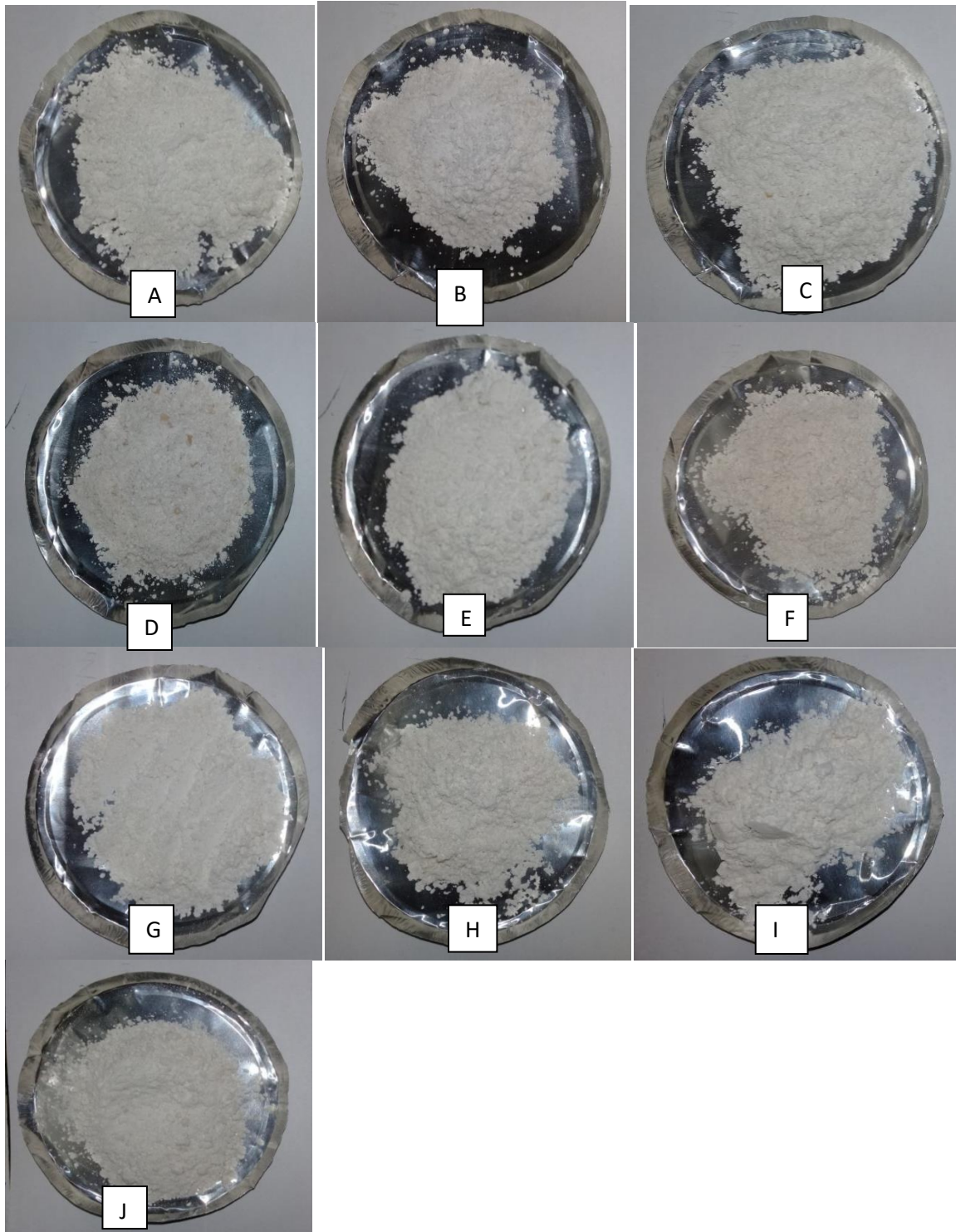


Figure 6: Bambara groundnut starch (HMT 35) heat treated under different conditions. A = 100 °C for 17.57 min; B = 100 °C for 60 min; C = 100 °C for 60 min; D = 100 °C for 102.43 min; E = 120 °C for 30 min; F = 120 °C for 90 min; G = 80 °C for 30 min; H = 80 °C for 90 min; I = 71.72 °C for 60 min and J = 128.28 °C for 60 min HMT conditions.

3.4. Colour characteristics

Colour characteristics of the samples was determined with the Hunterlab Scan, XE Spectrophotometer, CIELAB colour scale (Hunterlab, USA) with the parameters L^* , a^* , b^* , C^* , H° and colour difference (ΔE). Where L^* designates lightness, 0 - 100 with 0 defining black and 100 defining white. Coordinate a^* designates red (positive values) and green (negative values) while b^* designates yellow (positive values) and blue (negative values). H° (hue angle) indicates quality of lightness or darkness while C^* (Chroma) represents the quality of colour purity. Whiteness index (WI) of the starch samples was obtained from L^* , a^* , and b^* values using equation 1.

$$WI = \sqrt{(100 - L^*)^2 + a^{*2} + b^{*2}} \quad (1)$$

3.5. Thermal properties

Differential scanning calorimeter (DSC, DSC 4000, Perkin-Elmer, Shelton, CT, USA) was utilised to obtain thermal properties. The gelatinisation temperatures: onset temperature, peak temperature, concluding temperature and gelatinisation enthalpy were determined as described by Arns *et al.* (2014). Where a starch sample (2.5 mg) was measured in an aluminium pan and distilled water was added to attain a starch-water ratio 1:3 (g/g). The pan was tightly sealed and left to equilibrate overnight before analysis. The sample pans were then heated from 20 °C to 120 °C at a rate of 10 °C min⁻¹. Onset temperature, peak temperature, concluding temperature and enthalpy change were recorded in triplicate and Pyris software (Perkin-Elmer, Shelton, CT, USA) was utilised to calculate the obtained data.

3.6. Functional properties

3.6.1. Water absorption capacity

Determination of water absorption capacity (WAC) was achieved by the method of Herlina & Tensiska (2017), through mixing starch (1 g) sample and distilled water (10 mL) in a centrifuge tube. Subsequently the sample was cooled at room temperature (26 ± 2 °C) for 1 h thereafter centrifuged at 1500 rpm for 30 min. The volume of supernatant was measured and WAC calculated using equation 2.

$$\%WAC = \frac{\text{Volume of absorbed water} \times 100}{\text{weight of sample}} \quad (2)$$

3.6.2. Oil absorption capacity

Determination of OAC was achieved by method of Herlina & Tensiska (2017) through mixing starch (1 g) sample with of vegetable oil (10 mL) in a centrifuge tube. Subsequently the sample was cooled at room temperature (26 ± 2 °C) thereafter centrifuged at 1500 rpm for 30 min. The resultant supernatant was weighed and WAC calculated using equation 3.

$$\%OAC = \frac{\text{Volume of absorbed oil} \times 100}{\text{weight of sample}} \quad (3)$$

3.6.3. Swelling power and solubility

Determination of swelling power (SP) and solubility was achieved by method of Sankhon *et al.* (2013) by mixing starch (0.6 g) with distilled water (30 mL). It was then placed in a water bath at 85 °C for 30 min, with occasional stirring. The mixture was centrifuged at 3000 rpm for 15 min. Subsequently the resultant supernatant was prudently detached and the swollen starch sediment measured and recorded as M_1 . The supernatant was then evaporated and dried at 105 °C overnight in a drying oven (Prolab Instrument – model OTE 80, Vancouver, Canada) until constant weight which was obtained and recorded as M_2 . Swelling power (SP) and solubility was calculated using equation 4 and 5,

$$SP = \frac{M_1}{M_0} \quad (4)$$

$$\text{Solubility \%} = \frac{M_2}{M_0} \times 100 \quad (5)$$

Where M_0 = the initial dry weight of the starch sample

3.7. Scanning electron microscopy

Morphology of starch was examined using a scanning electron microscope (SEM) as applied by Oyeyinka *et al.*, (2017). Where samples were attached onto SEM stubs with a double-sided carbon tape, and gold-coated using an Edwards S150A sputter-coater in order to enhance conductivity. Samples were then visualised with a Zeiss Merlin Field Emission Scanning Electron Microscope (FESEM, Carl Zeiss Microscopy, Germany) at the Electron Microscopy Unit of Stellenbosch University's Central Analytical Facility (CAF). SEM images were generated with the

aid of Zeiss In Lens SE (Secondary Electron) and SE2 detectors and Zeiss Smart SEM software at 3kV accelerating voltage and 100 pA beam current with a working distance of 4.2 to 4.8 mm.

3.8. Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) spectra of unmodified and heat- moisture treated Bambara groundnut starches were attained using a spectrophotometer (120HR, Bruker, Germany) as used by Oyeyinka *et al.*, (2017) . The background spectra of the instrument were recorded before 0.5 g of starch samples were attached on the instrument and the spectra were noted with characteristic peaks in wave numbers from 450 to 4000 cm^{-1} at 16 runs per scan.

3.9. Statistical analysis

All experiments were conducted in triplicate. Design-Expert software version 8.0.1.0. Was used to conduct analysis of variance (ANOVA), regression models, optimisation and contour plots response surface graphs.

CHAPTER 4: RESULTS AND DISCUSSION

4.1. Effect of heat moisture treatment on colour characteristics of bambara groundnut starch.

The colour characteristics, of heat moisture treated BG starch under different HMT conditions of moisture (15, 25 and 35%), temperature (71.72 - 128.28 °C) and time (17.57 - 102.43 min) are shown on Table 5. Colour is a major attribute that is commonly evaluated by consumers when they buy foods and can influence consumption (Hutchings, 2015). L* value is defined as psychometric index of lightness and a higher whiteness value of starch is ideal for consumer acceptability (Barau & Srivastav, 2017). Whiteness index (WI) suggests whiteness of a food product and shows the degree of discolouration during treatment process (Anyasi *et al.*, 2015).

Table 5: Levels of process variables and values for colour characteristics of heat-moisture treated bambara groundnut under different treatment conditions

Independent variables		Response variables						
Temperature (°C)	Time (min)	L*	a*	b*	C*	H°	WI	ΔE
HMT 15								
100	17.57	81.9	1.5	3.4	3.7	66.6	81.5	19.0
100	60	80.7	2.5	4.6	5.2	61.6	80	20.1
100	60	82.2	1.8	3.3	3.7	61	81.8	18.6
100	102.43	79.3	1.2	3.1	3.3	68.2	79	21.9
120	30	81.8	1.8	3.6	4.0	63.9	81.4	19.5
120	90	80.6	2.2	3.9	4.5	60.2	80.1	20.1
80	30	82.3	1.3	3.2	3.5	68.0	81.9	18
80	90	82.1	1.4	3.5	3.8	69	81.7	18.4
71.72	60	81.8	1.7	3.1	3.6	62	81.5	18.4
128.28	60	81.8	1.9	4.7	5.0	68.1	81.1	19
HMT 25								
100	17.57	81.5	1.8	3.4	3.8	61.7	81.1	18.9
100	60	81.1	1.7	3.6	4.0	64.4	80.7	19.4
100	60	82	1.8	3.6	4	64.3	81.6	19.1
100	102.43	80.2	2.3	4.2	4.7	60.2	79.6	19.9
120	30	81.8	1.6	3.3	3.6	64.6	81.4	18.6
120	90	78.4	2.3	4.5	5.1	63.5	77.8	22.3
80	30	81.3	1.9	3.8	4.2	63.3	80.8	19.7
80	90	81.2	1.8	3.4	3.8	61.3	80.8	18.5
71.72	60	81.2	1.7	4	4.4	66.6	80.7	18.9
128.28	60	80.5	2.3	3.9	4.6	59.1	79.9	20.1
HMT 35								
100	17.57	80.8	1.7	4.2	4.5	67.9	80.3	19.7
100	60	75.7	3.6	5.8	6.9	58.1	74.8	23.7
100	60	77.9	2.4	4.3	4.9	61	77.4	23.2
100	102.43	77.8	3.2	5.3	6.2	59.1	76.9	22.7
120	30	79.7	2.4	5	5.5	64.6	78.9	20.3
120	90	74.2	4.4	5.8	7.3	53.2	73.2	27.3
80	30	80.1	2.4	4.3	4.9	60.9	79.5	21
80	90	78.9	2	4.7	5.1	67	78.3	21
71.72	60	80.6	1.6	4.8	5.1	71.1	79.9	20
128.28	60	75.6	2.1	4.8	5.2	66.6	75	23.7

L* = Lightness/darkness; a* = Redness/greenness; b* = Yellowness/blueness; C* = Chroma; H°= Hue angle; WI = Whiteness index; ΔE = Total colour difference.

Total colour difference (ΔE) is known as a noticeable difference, were the observer does not notice the difference (Jha, 2010). The colour characteristics of HMT 15-BG starch ranged from 79.3 - 82.3 (L^*), 1.2 - 2.5 (a^*), 3.1 - 4.7 (b^*), 3.3 - 5.2 (C^*), 60.2 - 69 (H°), 79 - 81.9 (WI) and 18 - 21.9 (ΔE). The colour characteristics of HMT 25-BG starch ranged from 78.4 - 82 (L^*), 1.6 - 2.3 (a^*), 3.3 - 4.5 (b^*), 3.6 - 5.1 (C^*), 59.1 - 66.6 (H°), 77.8 - 81.6 (WI) and 18.5 - 22.3 (ΔE). HMT 35-BG starch colour characteristics ranged from 74.2 - 80.8 (L^*), 1.6 - 4.4 (a^*), 4.2 - 5.8 (b^*), 4.5 - 7.3 (C^*), 53.2 - 71.1 (H°), 73.2 - 80.3 (WI) and 19.7 - 27.3 (ΔE) as shown in Table 5.

The inconsistencies in the colour characteristics of the starch samples were expected as dissimilar HMT conditions (combination of temperature and time) were applied. In this study, colour characteristics in terms of L^* and WI were considered as they are more applicable to starch. The highest L^* and WI values for heat moisture treated BG starch were observed at HMT 80 °C for 30 min under 15% moisture content (MC); 100 °C for 60 min (25% MC) and 100 °C for 17.57 min (35% MC) while the lowest was observed in HMT 100 °C for 102.43 min (15% MC); 120 °C for 90 min (25% MC) and 120 °C for 90 min (35% MC). This suggests that the lower the treatment time the higher the lightness and whiteness of the starch and vice versa.

The low a^* and b^* values obtained for all heat moisture treated BG starch samples suggests that samples were less red and yellow in colour, hence vindicating the moderately higher WI and L^* values of the starch samples obtained in this study. Analysis of variance (ANOVA) of the effect of model parameters on colour characteristics of HMT-BG starch (Table 6) showed that effects of temperature and treatment time, had no significant ($p \geq 0.05$) effect on the colour characteristics (L^* , a^* , b^* , C^* , H° , WI, ΔE) of the samples.

Table 6: Analysis of variance for the effect of model parameters on colour characteristics of heat-moisture treated bambara groundnut starch

Model terms	L*	a*	b*	C*	H°	WI	ΔE	P-Value
HMT 15								
A	0.4436 [^]	0.1890 [^]	0.1242 [^]	0.1342 [^]	0.7228 [^]	0.3328 [^]	0.2099 [^]	
B	0.0973 [^]	0.9433 [^]	0.9167 [^]	0.8968 [^]	0.9708 [^]	0.1061 [^]	0.1332 [^]	
A ²	0.4841 [^]	0.4161 [^]	0.9104 [^]	0.8346 [^]	0.4275 [^]	0.4626 [^]	0.3550 [^]	
B ²	0.4841 [^]	0.0862 [^]	0.2439 [^]	0.1746 [^]	0.2043 [^]	0.6437 [^]	0.4164 [^]	
AB	0.5805 [^]	0.6935 [^]	1.0000 [^]	0.8757 [^]	0.5856 [^]	0.5556 [^]	0.9220 [^]	
HMT 25								
A	0.7570 [^]	0.0509 [^]	0.5786 [^]	0.4403 [^]	0.0808 [^]	0.6927 [^]	0.5041 [^]	
B	0.4726 [^]	0.1228 [^]	0.6394 [^]	0.5797 [^]	0.0746 [^]	0.4602 [^]	0.6093 [^]	
A ²	0.4684 [^]	0.8396 [^]	0.7464 [^]	0.9820 [^]	0.6644 [^]	0.4939 [^]	0.4298 [^]	
B ²	0.2039 [^]	0.1731 [^]	0.4429 [^]	0.4984 [^]	0.3953 [^]	0.2154 [^]	0.2366 [^]	
AB	0.4663 [^]	0.3419 [^]	0.2645 [^]	0.3081 [^]	0.7357 [^]	0.4277 [^]	0.6560 [^]	
HMT 35								
A	0.9412 [^]	0.4669 [^]	0.3733 [^]	0.3892 [^]	0.6999 [^]	0.9745 [^]	0.9082 [^]	
B	0.4873 [^]	0.6487 [^]	0.9654 [^]	0.8739 [^]	0.5376 [^]	0.5310 [^]	0.4968 [^]	
A ²	0.6381 [^]	0.8934 [^]	0.6630 [^]	0.8293 [^]	0.7684 [^]	0.6792 [^]	0.4594 [^]	
B ²	0.2191 [^]	0.5419 [^]	0.4454 [^]	0.4777 [^]	0.6660 [^]	0.2464 [^]	0.4136 [^]	
AB	0.5197 [^]	0.7710 [^]	0.6038 [^]	0.6890 [^]	0.8333 [^]	0.5304 [^]	0.4625 [^]	

[^]Not significant at $p \geq 0.05$; A = Linear effect of treatment temperature; B = Linear effect of treatment time; A² = Quadratic effect of temperature; B² = Quadratic effect of time. L* = Lightness/darkness; a* = Redness/greenness; b* = Yellowness/blueness; C* = Chroma; H° = Hue angle; WI = Whiteness index; ΔE = Total colour difference.

The regression models for predicting colour characteristics of heat moisture treated BG starch is shown in Table 7.

Table 7: Regression models relating colour characteristics and model parameters of heat-moisture treated bambara groundnut starch

Responses	Models	Lack of fit P-value
HMT 15		
L*	+87.76924-0.13750*A+0.060513*B+7.50000E-004*A ² -3.33333E-004*B ² -4.16667E-004*A*B	0.7522 [^]
a*	-3.35821+0.077393*A+0.037816*B-3.75000E-004*A ² -4.16667E-004*B ² +1.25000E-004*A*B	0.8112 [^]
b*	-0.21440+0.034767*A+0.048232*B-7.81250E-005*A ² -3.95833E-004*B ² -2.31296E-019*A*B	0.9137 [^]
C*	-0.50852+0.046124*A+0.054310*B-1.56250E-004*A ² -5.13889E-004*B ² +8.33333E-005*A*B	0.9535 [^]
H°	+104.51763-0.72796*A-0.18099*B+4.09375E-003*A ² +3.12500E-003*B ² -1.95833E-003*A*B	0.0680 [^]
WI	+88.39994-0.15166 *A +0.051519 *B +8.12500E-004 * A ² -2.22222E-004 * B ² -4.58333E-004*A*B	0.8500 [^]
ΔE	+5.95062+0.25468*A-0.041245*B-1.17188E-003*A ² +4.51389E-004*B ² +8.33333E-005*A*B	0.6663 [^]
HMT 25		
L*	+90.37586-0.17093*A-0.041280*B+1.04688E-003*A ² +8.81944E-004*B ² -7.50000E-004*A*B	0.9965 [^]
a*	+0.84485+8.42830E-003*A+6.76777E-003*B-4.68750E-005*A ² -1.59722E-004*B ² +1.66667E-004*A*B	0.9876 [^]
b*	+3.70181+0.011402*A-0.020804*B-1.71875E-004*A ² -1.87500E-004*B ² +4.58333E-004*A*B	0.9716 [^]
C*	+5.90129-0.027197*A-0.024381*B+1.56250E-005*A ² -2.15278E-004*B ² +5.41667E-004*A*B	0.9874 [^]
H°	+69.10670+0.083154*A-0.19516*B-9.06250E-004*A ² +8.19444E-004*B ² +5.00000E-004*A*B	0.4766 [^]
WI	+89.52549-0.16582*A-0.033119*B+1.04688E-003*A ² +9.09722E-004*B ² -8.75000E-004*A*B	0.9966 [^]
ΔE	+5.42987+0.24244*A+0.066482*B-1.28125E-003*A ² -9.02778E-004*B ² +5.00000E-004*A*B	0.9914 [^]
HMT 35		
L*	+63.10876+0.35737*A-0.096082*B-1.39062E-003*A ² +1.77083E-003*B ² -1.37500E-003*A*B	0.5906 [^]
a*	+6.229750.070954*B+0.024011*B+1.87500E-004*A ² -3.88889E-004*B ² +2.91667E-004*A*B	0.6128 [^]
b*	+3.50086+0.045536*A+3.77961E-003*B-3.90625E-004*A ² -3.12500E-004*B ² +3.33333E-004*A*B	0.6598 [^]
C*	+5.51725+0.017688*A+0.017315*B-3.12500E-004*A ² -4.72222E-004*B ² +4.16667E-004*A * B	0.6610 [^]
H°	+32.67360+0.65719*A-0.12988*B-2.62500E-003*A ² +1.72222E-003*B ² -1.33333E-003* A * B	0.5435 [^]
WI	+62.07592+0.35466*A-0.090594*B-1.32812E-003*A ² +1.79861E-003*B ² -1.45833E-003*A*B	0.5925 [^]
ΔE	+48.70864-0.56619*A+0.051743*B+2.45313E-003*A ² -1.21528E-003*B ² +1.16667E-003*A*B	0.4625 [^]

[^]Not significant at $p \geq 0.05$; A = Linear effect of treatment temperature; B = Linear effect of treatment time; AB = Interaction of temperature and treatment time; A² = Quadratic effect of temperature; B² = Quadratic effect of treatment time; L* = Lightness/darkness; a* = Redness/greenness; b* = Yellowness/blueness; C* = Chroma; H° = Hue angle; WI = Whiteness index; ΔE = Total colour difference.

The regression models for colour characteristics of HMT-BG starches are characterised by non-significant lack of fit ($p \geq 0.05$). Non-significant lack of fit is good as it guarantees a good fit of the models for the experimental data. Contour plots illustrating the effects of HMT treatment temperature and time on L^* , a^* , b^* , C^* , H^0 , WI and ΔE is shown in Figures 7 - 9. In HMT 15-BG starch, the lightness (L^*) and whiteness index of the samples decreased as treatment time and temperature increased whereas a^* , b^* , C^* , ΔE and H^0 of the starch samples all increased with increase in HMT treatment temperature and time (Figure 7 a-g).

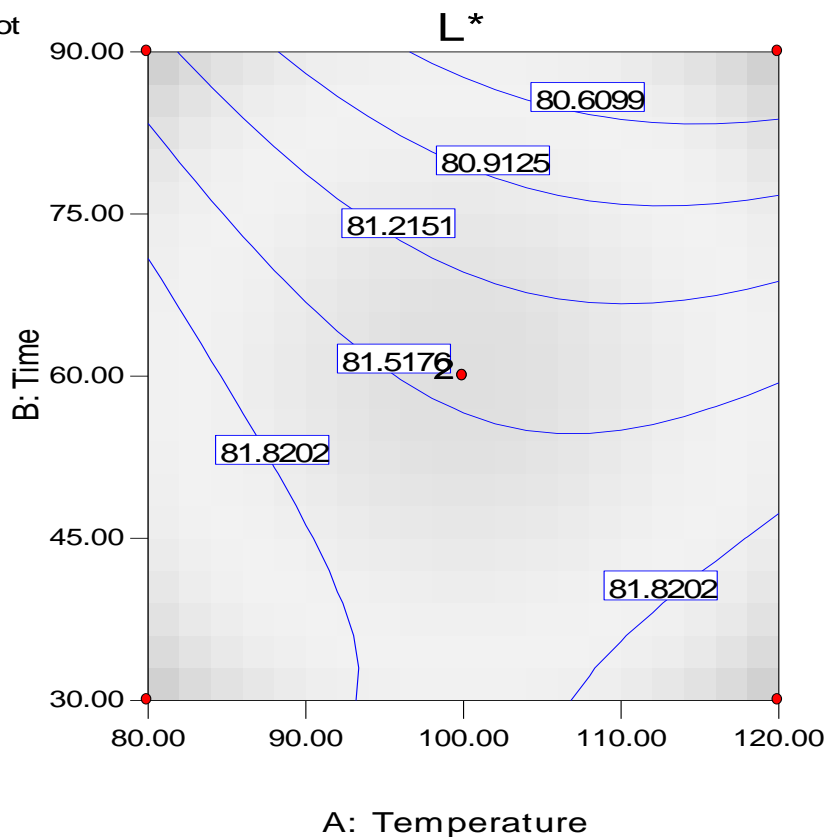
DESIGN-EXPERT Plot

L^*

● Design Points

X = A: Temperature

Y = B: Time



(a)

DESIGN-EXPERT Plot

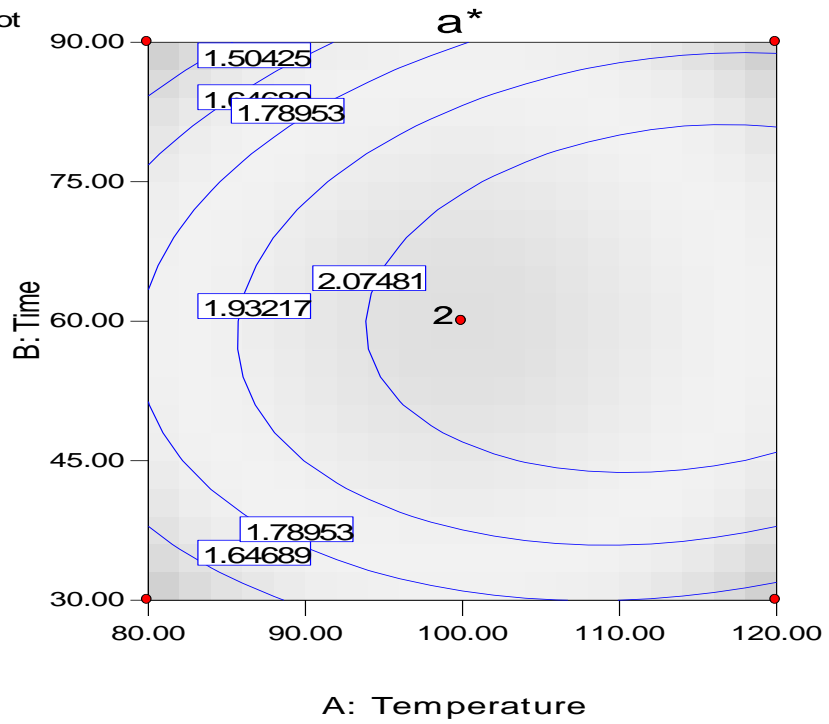
a^*

● Design Points

X = A: Temperature

Y = B: Time

(b)



DESIGN-EXPERT Plot

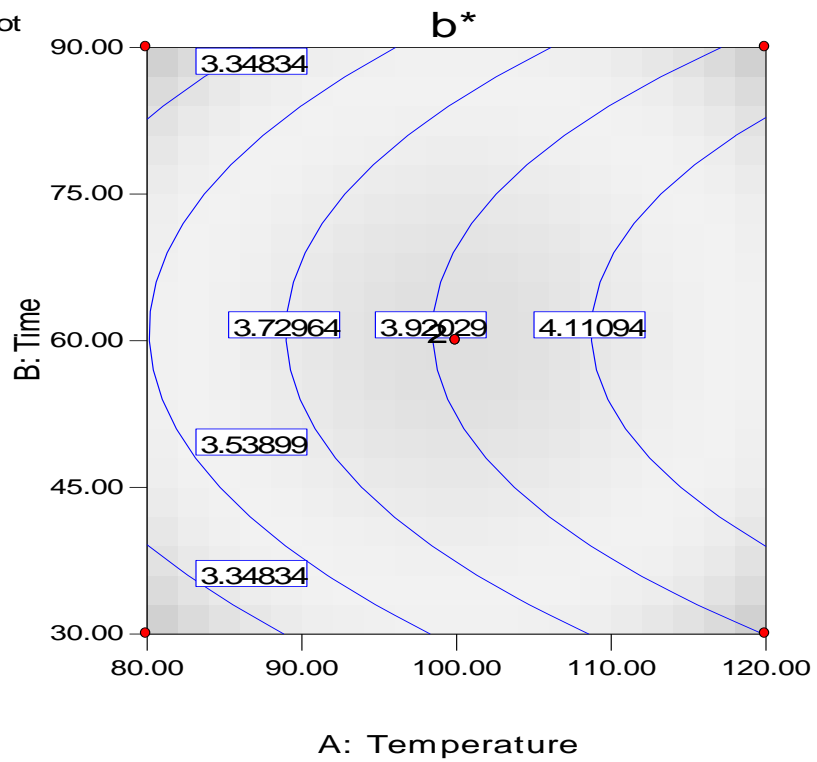
b^*

● Design Points

X = A: Temperature

Y = B: Time

(c)



DESIGN-EXPERT Plot

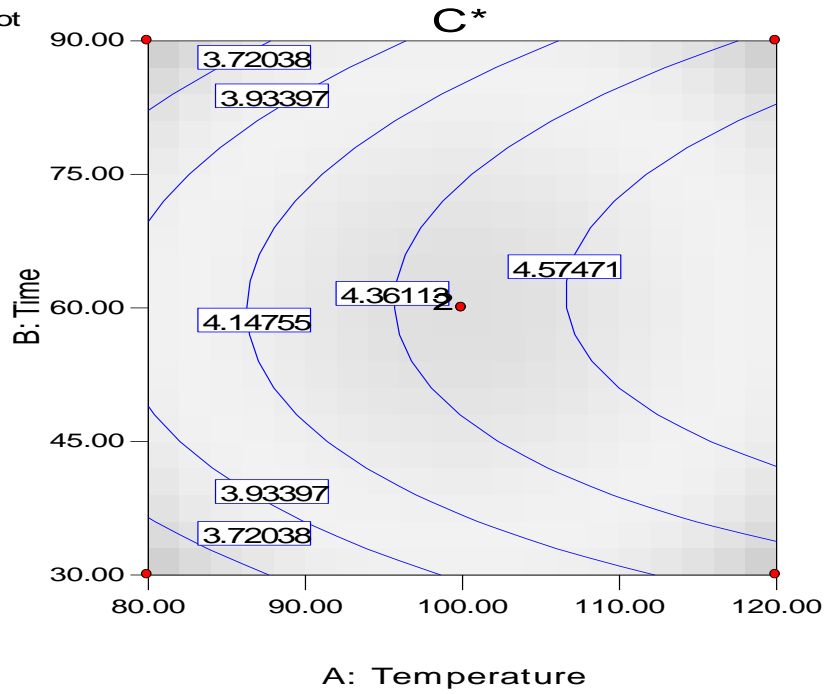
C*

● Design Points

X = A: Temperature

Y = B: Time

(d)



DESIGN-EXPERT Plot

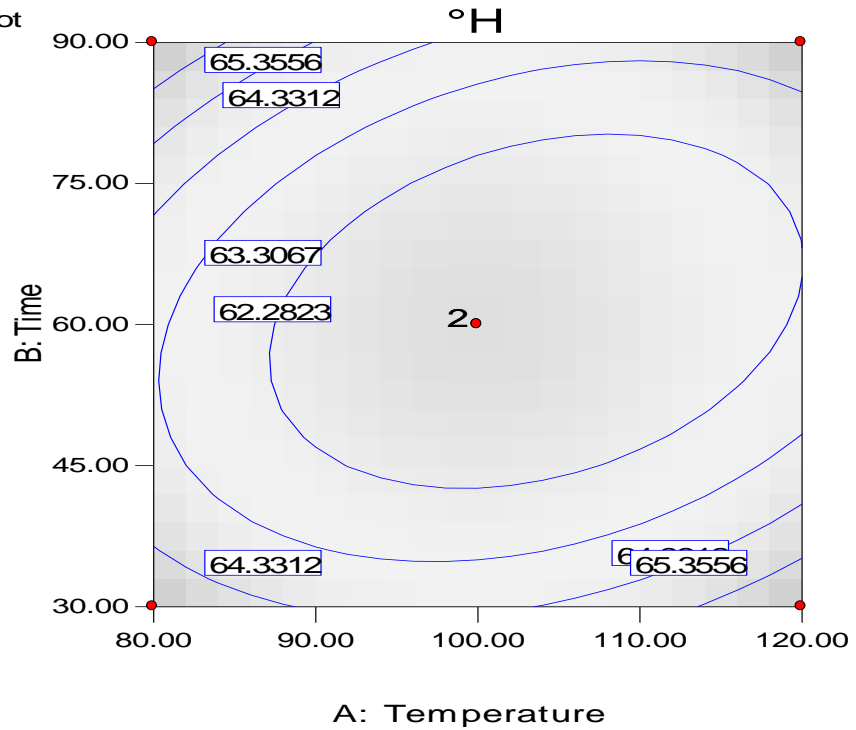
°H

● Design Points

X = A: Temperature

Y = B: Time

(e)

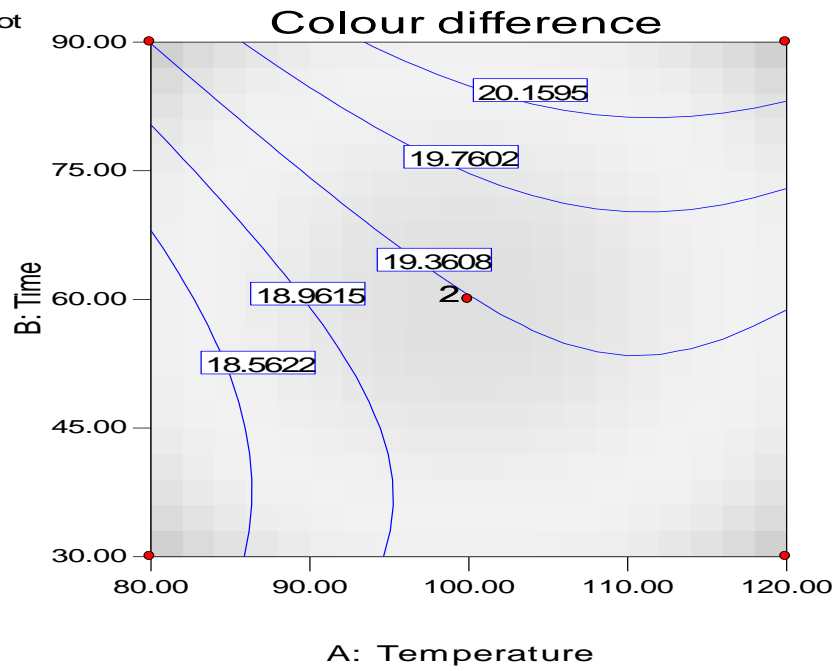


DESIGN-EXPERT Plot

Colour difference
● Design Points

X = A: Temperature
Y = B: Time

(f)

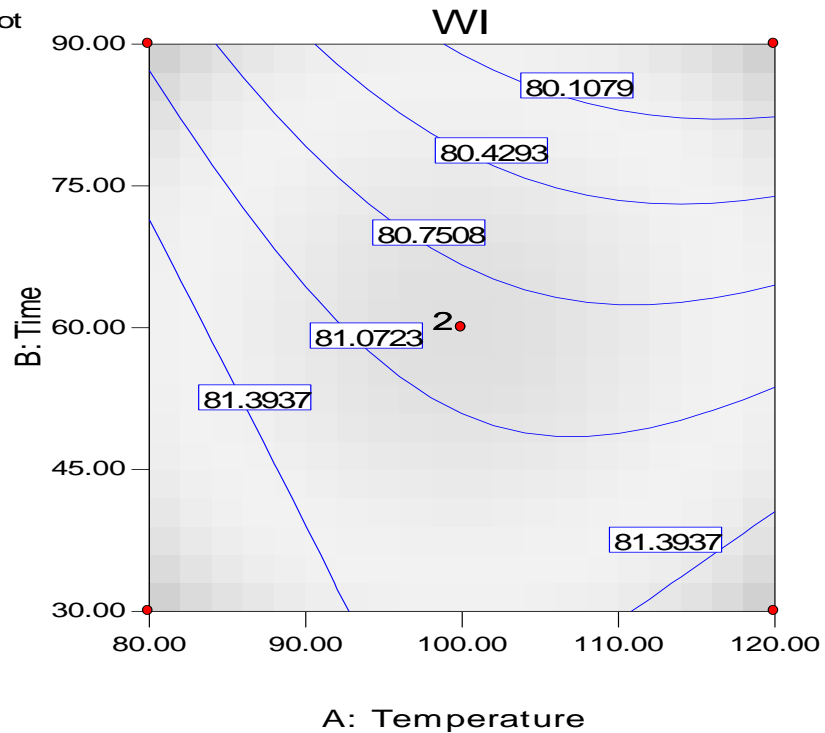


DESIGN-EXPERT Plot

WI
● Design Points

X = A: Temperature
Y = B: Time

(g)

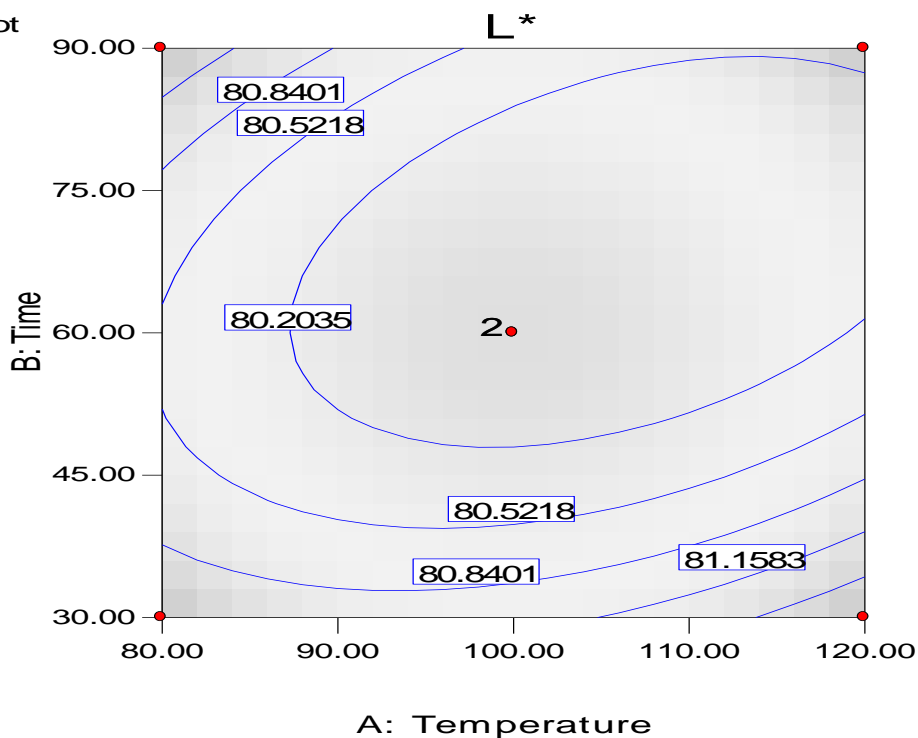


Figures 7 (a-g): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = L^* ; (b) = a^* ; (c) = b^* ; (d) = C^* ; (e) = H^* ; (f) = Total colour difference; (g) = WI of HMT 15-BG starch

In HMT 25-BG starch, a^* and total colour difference increased with increase in treatment temperature and time. Consequently, b^* value, hue angle and whiteness index of the samples decreased as treatment time and temperature increased. This suggests that as temperature and time increase the discolouration of the starch samples is inevitable. The lightness and chromaticity of the starch samples decreased with increase in HMT treatment temperature and time but later increased as treatment time progressed (Figures 8 a-g).

DESIGN-EXPERT Plot

L^*
● Design Points
 X = A: Temperature
 Y = B: Time



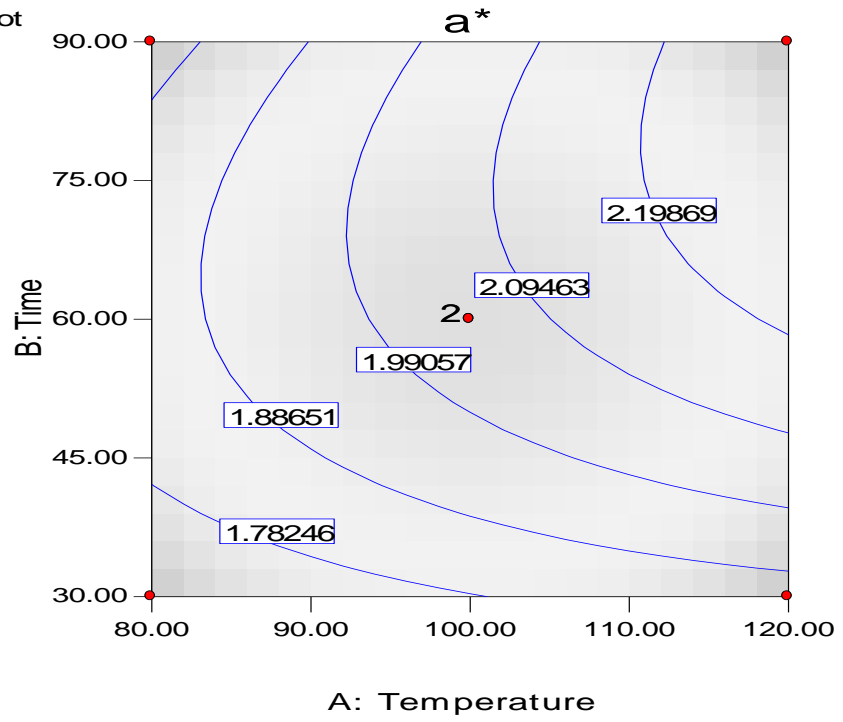
(a)

DESIGN-EXPERT Plot

a^*
● Design Points

X = A: Temperature
Y = B: Time

(b)



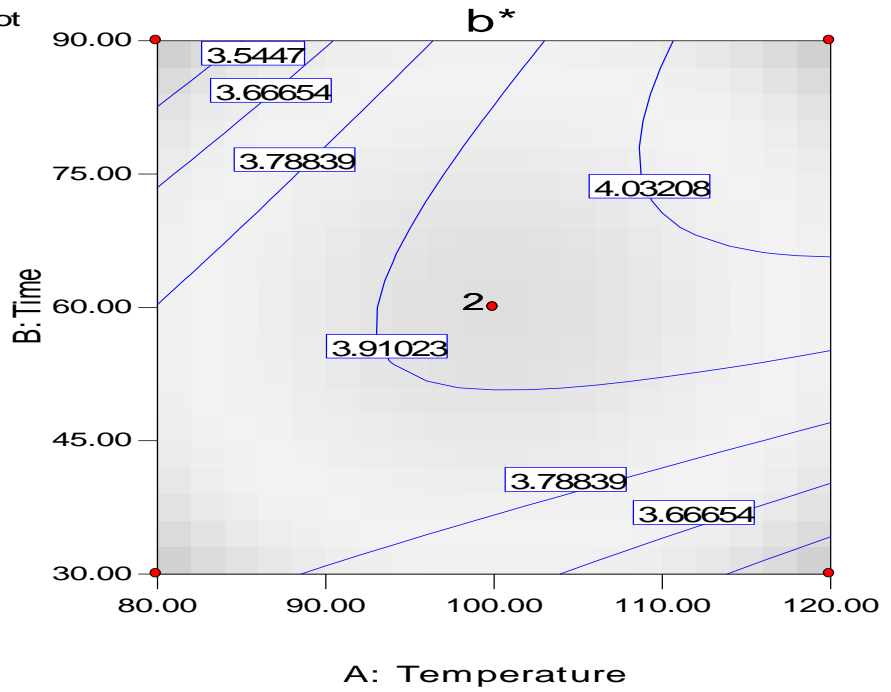
DESIGN-EXPERT Plot

b^*

● Design Points

X = A: Temperature
Y = B: Time

(c)



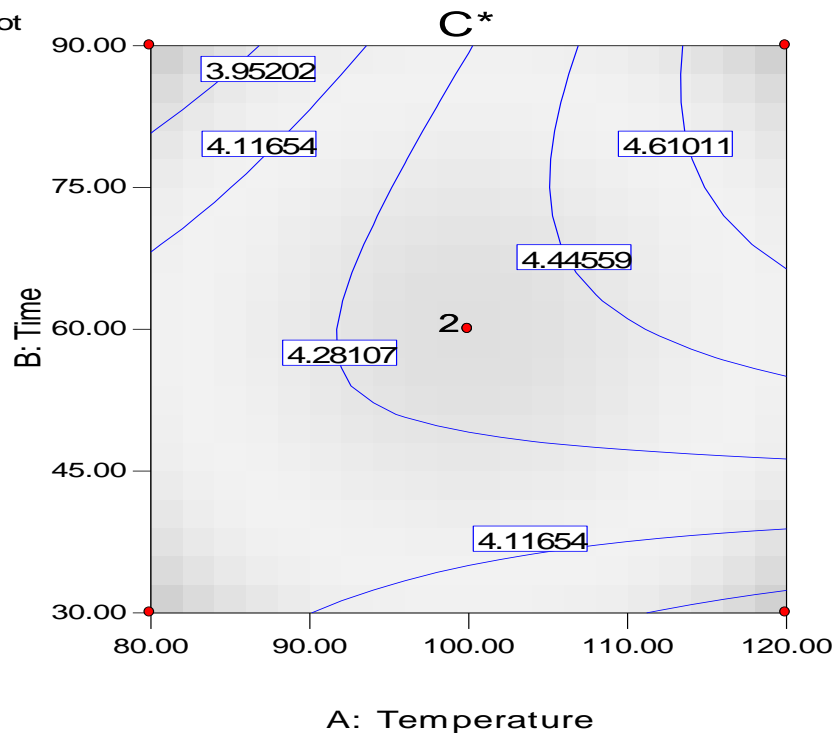
DESIGN-EXPERT Plot

C^*

● Design Points

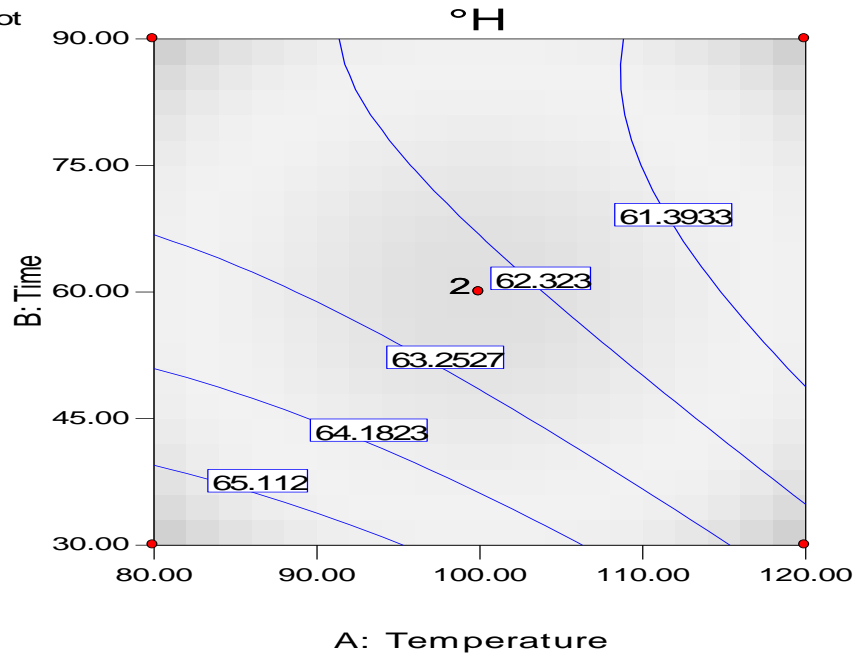
X = A: Temperature
Y = B: Time

(d)



DESIGN-EXPERT Plot

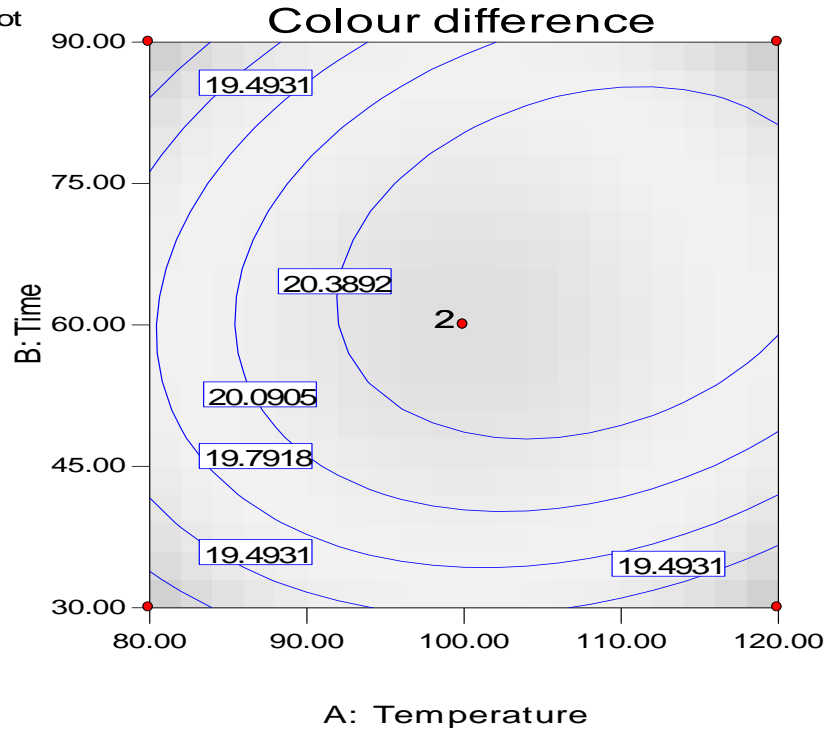
°H
 ● Design Points
 X = A: Temperature
 Y = B: Time



(e)

DESIGN-EXPERT Plot

Colour difference
 ● Design Points
 X = A: Temperature
 Y = B: Time



(f)

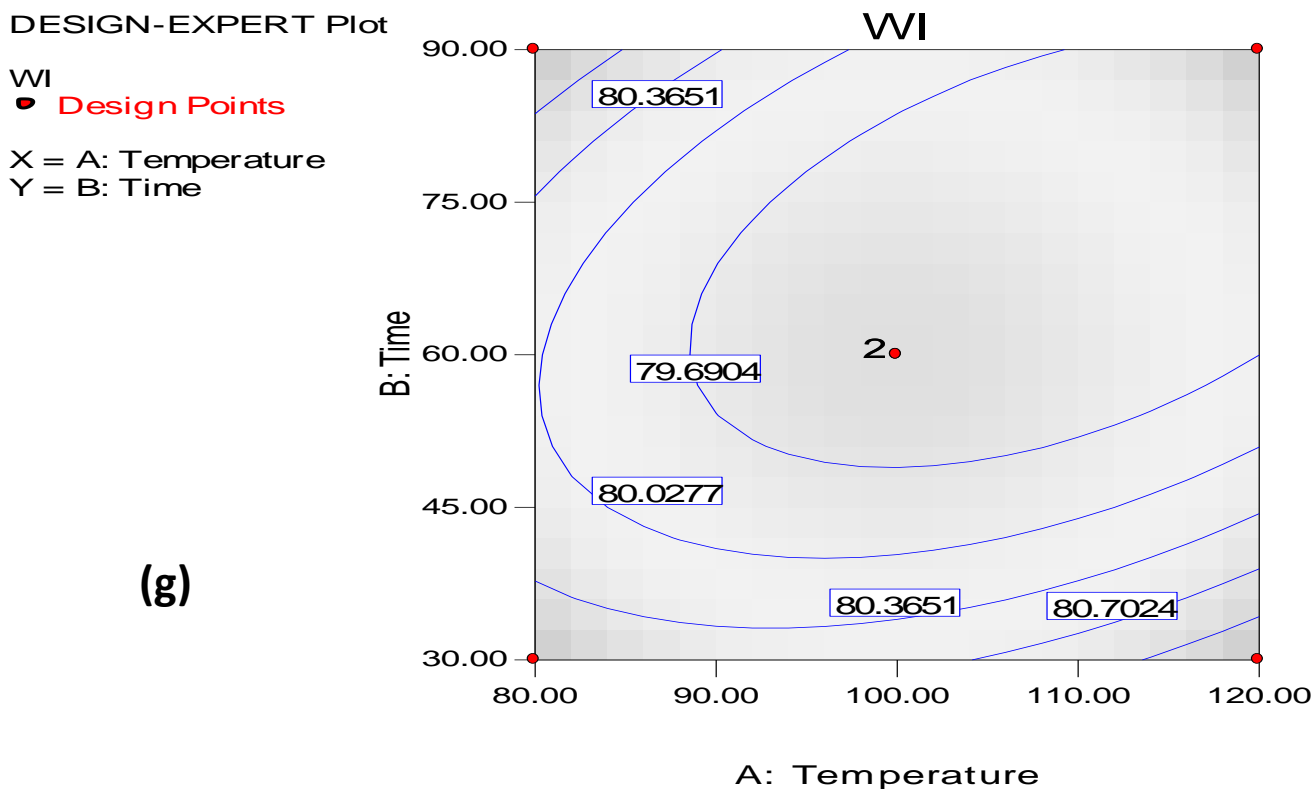


Figure 8 (a-g): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = L^* ; (b) = a^* ; (c) = b^* ; (d) = C^* ; (e) = H° ; (f) = Total colour difference; (g) = WI of HMT 25-BG starch

In HMT 35-BG starch the L^* , a^* and total colour difference of the samples increased as treatment time and temperature increased, whereas b^* , chromaticity, whiteness index and Hue angle of the starch samples all decreased with increase in HMT treatment temperature and time (Figure 9 a-g). This decrease as the treatment conditions increase suggests that higher treatment temperature and time can lead to the discoloration of the starch samples.

DESIGN-EXPERT Plot

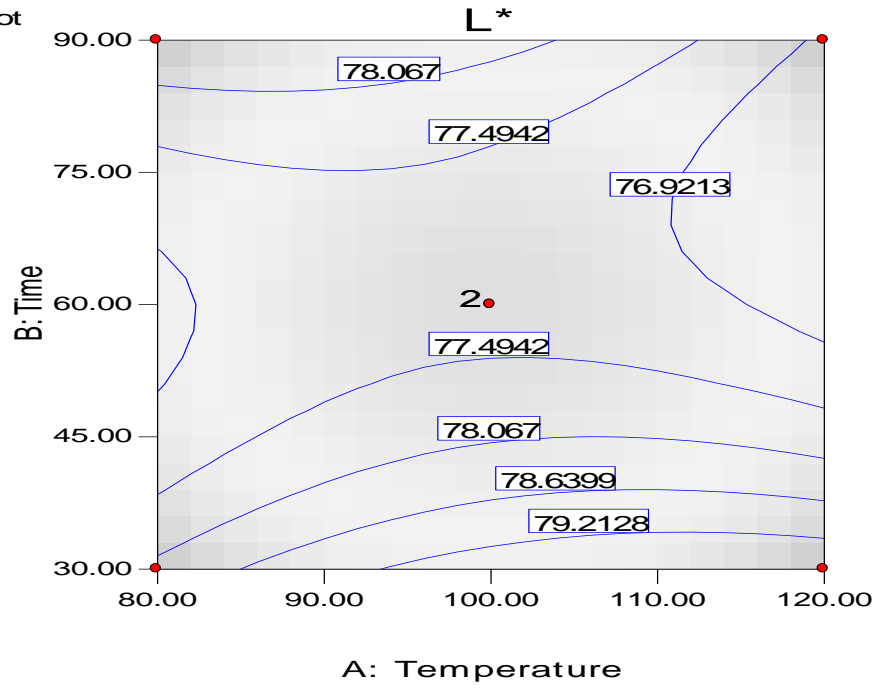
L*

● Design Points

X = A: Temperature

Y = B: Time

(a)



DESIGN-EXPERT Plot

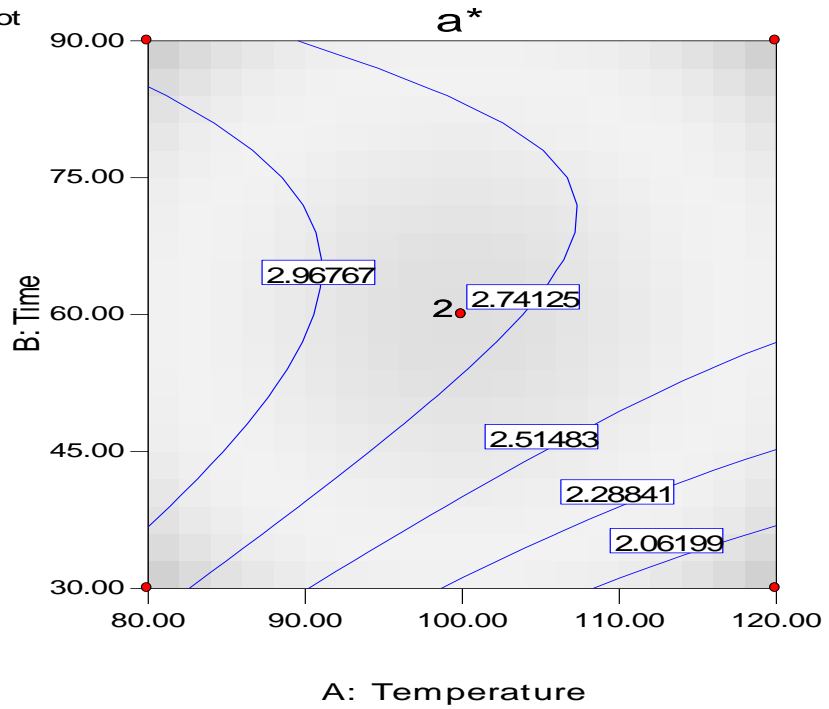
a*

● Design Points

X = A: Temperature

Y = B: Time

(b)



DESIGN-EXPERT Plot

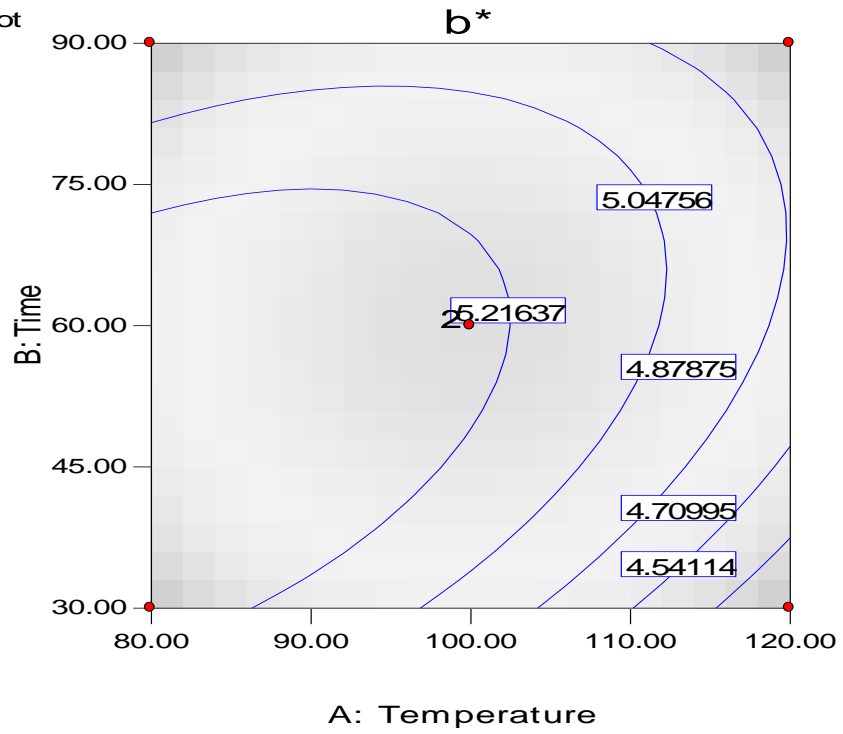
b^*

● Design Points

X = A: Temperature

Y = B: Time

(c)



DESIGN-EXPERT Plot

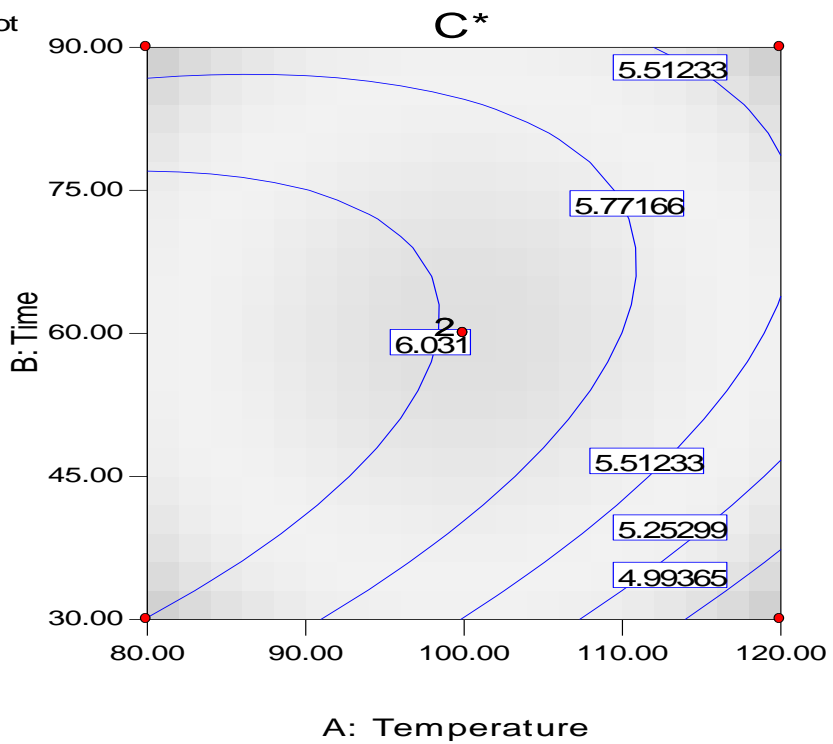
C^*

● Design Points

X = A: Temperature

Y = B: Time

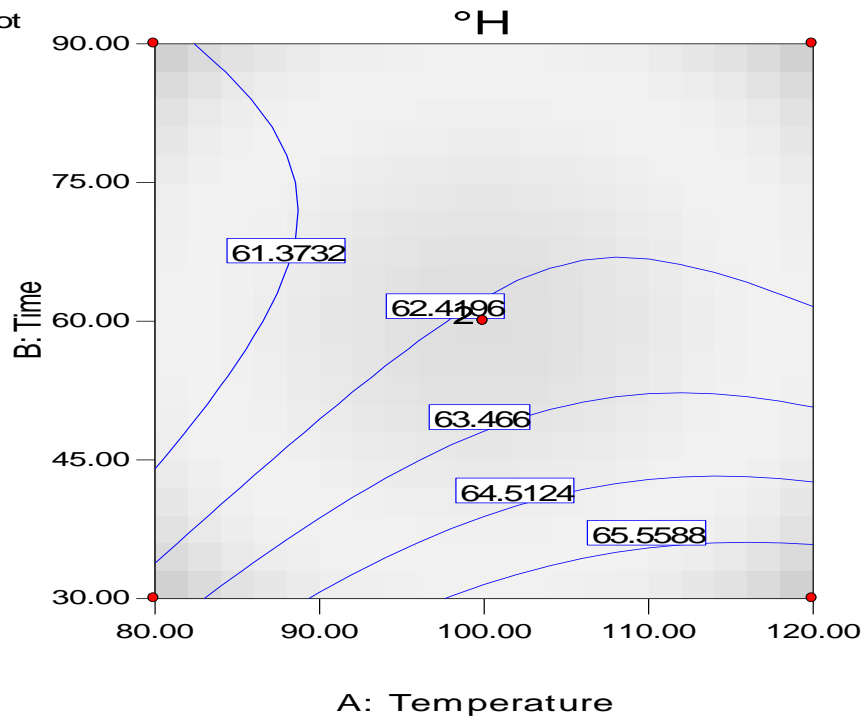
(d)



DESIGN-EXPERT Plot

°H
 ● Design Points
 X = A: Temperature
 Y = B: Time

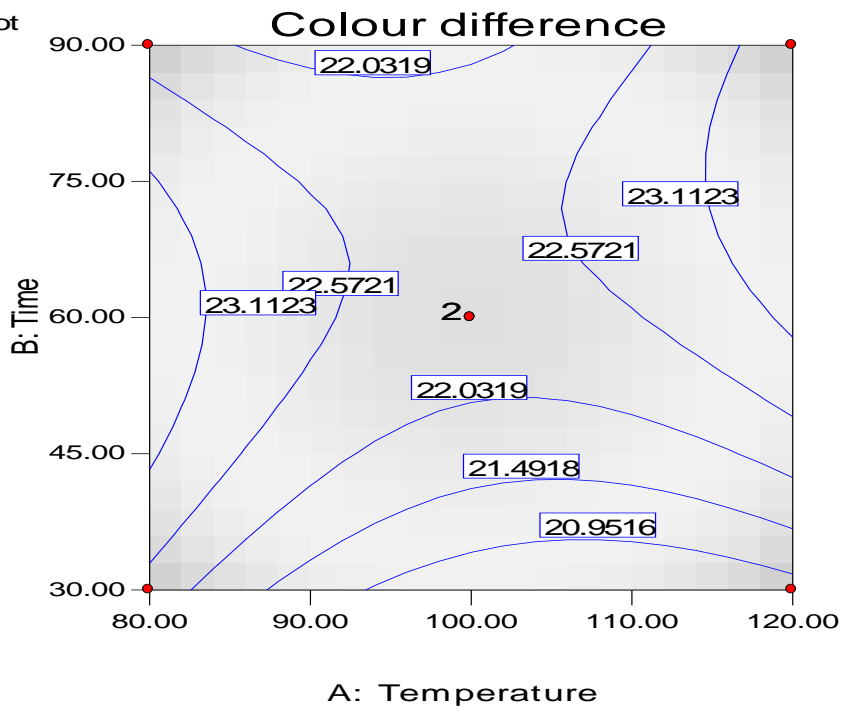
(e)



DESIGN-EXPERT Plot

Colour difference
 ● Design Points
 X = A: Temperature
 Y = B: Time

(f)



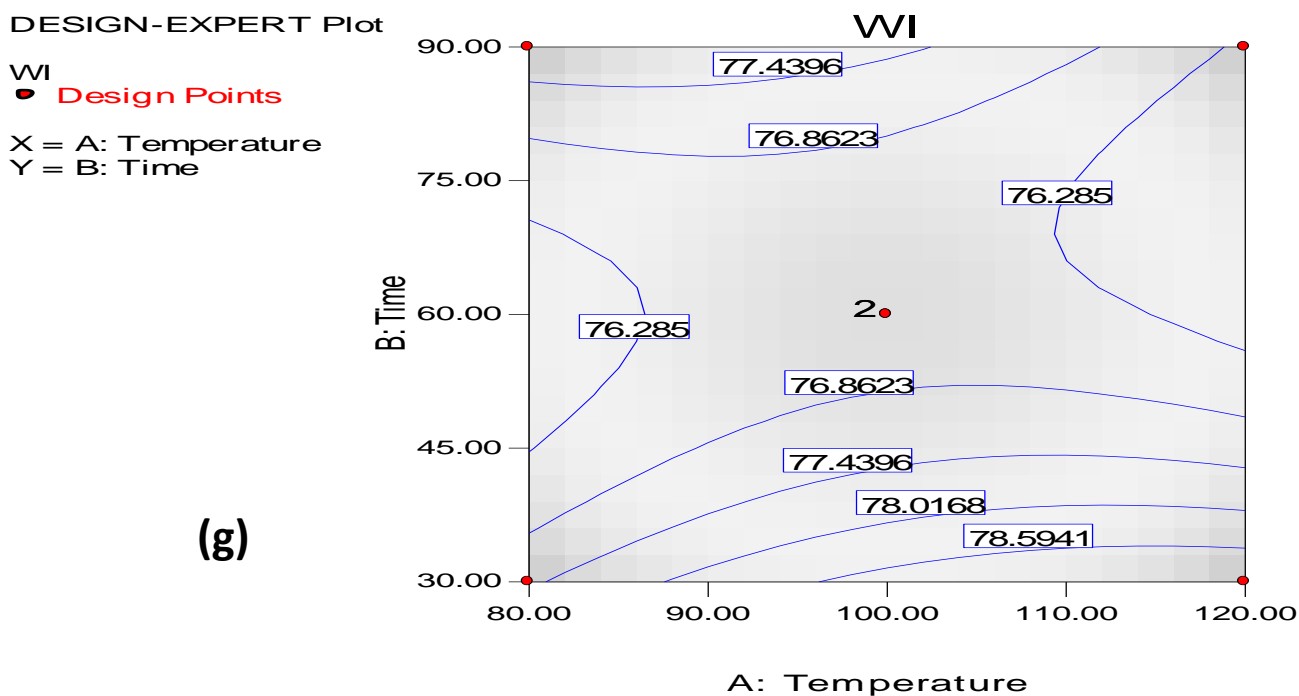


Figure 9 (a-g): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = L^* ; (b) = a^* ; (c) = b^* ; (d) = C^* ; (e) = H° ; (f) = total colour difference; (g) = WI of HMT 35-BG starch

Barau & Srivastav (2017) reported L^* , a^* and b^* values ranging between 83.66 - 87.68, 1.73 - 2.16 and 14.24 - 16.74 respectively, on mung bean starch heat moisture treated at 30% moisture, for 16 h at 80 – 120 °C. The L^* (63.71), a^* (4.19), b^* (10.28) values, H° (81.20) and total colour difference (7.51) has also been reported for HMT potato starch with 20-25% at 120 °C (Nadir *et al.*, 2015). Liestianty *et al.* (2016) reported colour characteristics of heat moisture treated sago starch at 28% moisture, and 110°C for 2 - 6 h. They observed that L^* , a^* and b^* values ranged from 80.58 - 82.17, 0.75 - 1.25 and 6.86 - 7.41 respectively. L^* , a^* and b^* values of 92.29, 1.725 and 6.549 were reported for buckwheat starch heat moisture treated at 25% moisture at 110 °C for 4 h (Sarkar, 2016).

An increase in L^* values after modification of buckwheat starch by HMT was reported by Sarkar (2016). Nadir *et al.* (2015) reported a decrease in L^* and b^* values with an increase in a^* value, but a notable decrease in Hue angle and colour difference after HMT. It can be observed that different HMT conditions have varying effects on colour properties (Figures 7 - 9). It has been reported that changes in colour characteristics of starches during physical methods like HMT could be as a result of the separation and purification of heterogeneous materials including

sugars, proteins, salts and other elements (Falade & Ayetigbo, 2015; Nadir *et al.*, 2015). Variations in colour are often attributed to the browning reaction which could take place during HMT. This also leads to the breakdown of starch molecules and consequently produces simple sugars (Kim *et al.*, 2017; Liu *et al.*, 2002; Barau & Srivastav, 2017).

4.2. Effect of heat-moisture treatment on the gelatinisation parameters of bambara groundnut starch

Starch gelatinisation is an endothermic alteration significant for the separation of amylopectin double helices from the ordered structure to a disordered structure (Chen *et al.*, 2017). This phenomenon is significant in the processing procedures as they form unique textural and structural characteristics in food products. Gelatinisation temperature also characterises starch type and is dependent on the glass transition of the amorphous fraction of the starch (Zavareze *et al.*, 2012). The onset temperature (T_o) is defined as the melting temperature of the weakest crystalline in starch granules, while the conclusion temperature (T_c) is the melting temperature of high perfection crystalline (Chen *et al.*, 2017). The gelatinisation enthalpy (ΔH) primarily represents the loss of double helical components within starch granules (Singh *et al.*, 2003). The thermal properties (gelatinisation) of BG starch under different HMT conditions are shown in Table 8.

Table 8: Levels of process variables and values of gelatinisation for heat-moisture treated bambara groundnut under different treatment conditions

Independent variables		Response variables			
Temperature (°C)	Time (min)	T _o (°C)	T _p (°C)	T _c (°C)	ΔH (J/g)
HMT 15					
100	17.57	75.86	81.52	87	5.36
100	60	70.09	77.81	87.54	5.19
100	60	94.63	97.03	94.63	2.95
100	102.43	83.39	86.79	90.42	1.26
120	30	93.20	96.11	98.59	2.8
120	90	78.26	84.19	90.54	1.17
80	30	89.18	93.61	98.92	4.24
80	90	78.02	85.83	94.75	7.93
71.72	60	92.91	97.85	102.76	9.21
128.28	60	78.13	87.35	97.74	8.27
HMT 25					
100	17.57	59.12	72.21	86.86	7.73
100	60	76.40	81.26	87.1	3.57
100	60	78.58	86.58	96.97	17.90
100	102.43	71.80	81.24	89.99	3.79
120	30	62.62	75.87	92.26	1.20
120	90	76.85	90.25	104.71	18.14
80	30	77.76	90.56	102.34	11.86
80	90	87.77	96.14	100.28	12.26
71.72	60	82.14	92.63	103.23	3.48
128.28	60	92.64	99.47	102.92	12.26
HMT 35					
100	17.57	80.45	88.63	97.52	9.37
100	60	66.62	80.52	92.43	9.21
100	60	70.39	81.44	92.89	11.35
100	102.43	68.28	78.55	91.32	1.24
120	30	66.45	78.36	90.50	11.11
120	90	70.53	83.43	96.56	6.17
80	30	78.37	90.33	102.21	6.03
80	90	74.95	89.67	105.42	8.26
71.72	60	85.53	97.18	108.88	6.08
128.28	60	85.23	93.83	103.43	1.66

T_o (°C) = Onset temperature; T_p (°C) = Peak temperature; T_c (°C) = Conclusion temperature; ΔH (J/g) = Gelatinisation enthalpy.

The thermal properties of HMT 15-BG starch ranged from 70.09 - 94.63 °C (T_o), 77.81 - 97.85 °C (T_p), 87 - 102.76 °C (T_c) and 1.17 - 9.21 (ΔH) whereas, the thermal properties of HMT 25-BG starch ranged from 59.12 - 92.64 °C (T_o), 72.21 - 99.47 °C (T_p), 86.86 - 104.71 °C (T_c) and 1.20 - 18.14 (ΔH). In terms of HMT 35-BG starch, thermal properties ranged from 66.45 - 85.53 °C (T_o), 78.36 - 97.18 °C (T_p), 91.32 - 108.88 °C (T_c) and 1.24 - 11.35 (ΔH).

The highest values of T_o , of BG starch with 15% moisture content (MC) were obtained at HMT; 100 °C for 60 min while the highest values of T_p , T_c and ΔH were obtained at HMT 71.72°C for 60 min. Similarly the highest values of T_o and T_p of BG starch with 25% MC were obtained at HMT 128.28°C for 60 min and the highest values of T_c and ΔH were obtained at HMT 120 °C for 90 min. Consequently, the highest values of T_o , T_p and T_c were obtained at HMT 71.72 °C for 60 min while the highest ΔH values of BG starch with 35% MC were obtained at HMT 100 °C for 60 min. The effect of heat moisture treatment on thermal properties has been previously investigated on starch from other crops such as maize, cassava, millet, locust bean, lentil, yam bean and pea. The comparison of these reports to the findings of the present study is shown in Table 9.

Table 9: Comparison of gelatinisation temperatures of the present study with previous studies

Source of starch	Temperature (°C)	Time (min)	T _o (°C)	T _p (°C)	T _c (°C)	ΔH (J/g)	Reference
Present study							
BG	71.72-128.28	17.57-102.43	70.09-94.63	77.81-97.85	87-102.76	1.17-9.21	
BG	71.72-128.28	17.57-102.43	59.12-92.64	72.21-99.47	86.86-104.71	1.20-18.14	
BG	71.72-128.28	17.57-102.43	66.45-85.53	78.36-97.18	91.32-108.88	1.24-11.35	
Previous studies							
African yam bean	100	960	73.57	76.49	79.23	0.543	Adebowale <i>et al.</i> , 2009
Locust bean	110	1440	63.7	82.5	103.6	8.94	Sankhon <i>et al.</i> , 2013
Pea	100-120	120	64.3	88.6	97.5	7.1	Chung <i>et al.</i> , 2009
Lentil	100-120	120	65.9	86.4	96.3	8.0	Chung <i>et al.</i> , 2009

T_o = Onset temperature; T_p = Peak temperature; T_c = Conclusion temperature; ΔH = Gelatinisation enthalpy.

The variation in the starch gelatinisation temperatures could be attributed to the effects on the molecular structure of amylopectin, starch composition as well as the granular arrangement of the starch particles (Gunaratne & Hoover, 2002). Furthermore, these differences in gelatinisation temperatures could also be due to the content of amylose, size and also the re-alignment of starch helices within the starch (Barau & Srivastav, 2017). Analysis of variance (ANOVA) of the effect of model parameters on thermal properties of HMT 15-BG, HMT 25-BG and HMT 35-BG starch (Table 10) show that effects of HMT treatment temperature and time, had no significant ($p \geq 0.05$) effect on thermal properties (T_o, T_p, T_c and ΔH) of the HMT bambara groundnut starch.

Table 10: Analysis of variance of the effect of model parameters on gelatinisation properties of heat-moisture treated bambara groundnut starch

Model terms	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)
	P-Value			
HMT 15				
A-temperature	0.6445 [^]	0.6057 [^]	0.3660 [^]	0.2414 [^]
B-time	0.6677 [^]	0.6499 [^]	0.5534 [^]	0.6185 [^]
A ²	0.7227 [^]	0.5110 [^]	0.0609 [^]	0.1844 [^]
B ²	0.8859 [^]	0.7786 [^]	0.6648 [^]	0.4868 [^]
AB	0.8806 [^]	0.8264 [^]	0.6562 [^]	0.3390 [^]
HMT 25				
A	0.9163 [^]	0.8878 [^]	0.8516 [^]	0.3050 [^]
B	0.5426 [^]	0.5851 [^]	0.8894 [^]	0.5640 [^]
A ²	0.1493 [^]	0.3843 [^]	0.9966 [^]	0.4841 [^]
B ²	0.7418 [^]	0.7795 [^]	0.8274 [^]	0.3570 [^]
AB	0.5941 [^]	0.7152 [^]	0.8547 [^]	0.1053 [^]
HMT 35				
A	0.3403 [^]	0.3571 [^]	0.4320 [^]	0.4957 [^]
B	0.4298 [^]	0.2536 [^]	0.2313 [^]	0.8069 [^]
A ²	0.4825 [^]	0.7910 [^]	0.8754 [^]	0.6592 [^]
B ²	0.5614 [^]	0.8807 [^]	0.8875 [^]	0.6199 [^]
AB	0.2119 [^]	0.2217 [^]	0.2960 [^]	0.4713 [^]

[^]Not significant at $p \geq 0.05$; A = Linear effect of treatment temperature; B = Linear effect of treatment time; A² = Quadratic effect of treatment temperature; B² = Quadratic effect of treatment time; T_o = Onset temperature; T_p = Peak temperature; T_c = Conclusion temperature; ΔH = Gelatinisation enthalpy.

The regression models for predicting thermal properties of heat moisture treated BG starch (HMT 15, HMT 25 and HMT 35) is shown in Table (11).

Table 11: Regression models relating gelatinisation parameters and model parameters of heat-moisture treated bambara groundnut starch

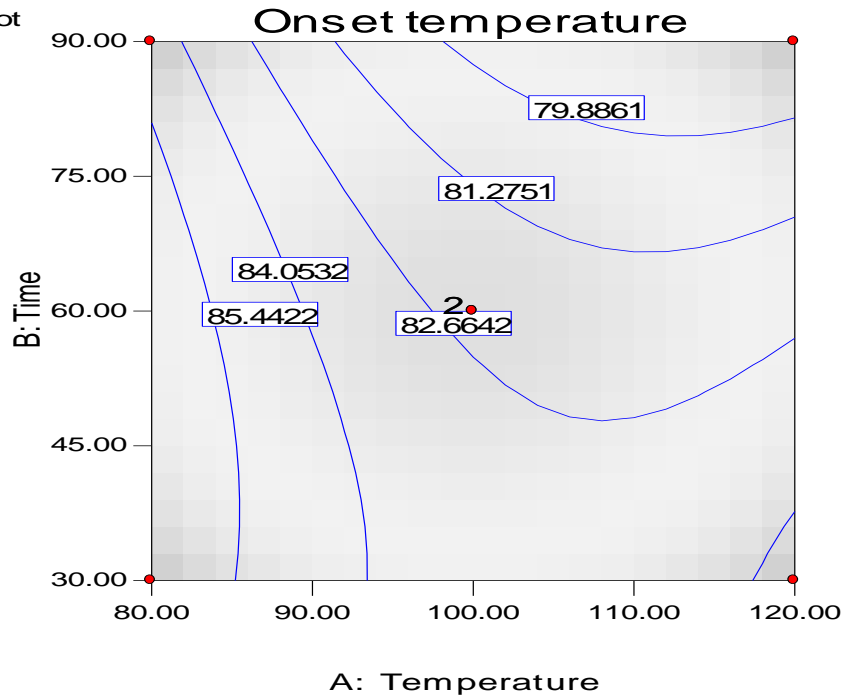
Responses	Model	Lack of fit p-value
HMT 15		
T _o (°C)	+136.37467-1.06108*A+0.20570*B+5.25781E-003*A ² -9.38194E-004*B ² -1.57500E-003*A*B	0.8427 [^]
T _p (°C)	+158.38693-1.47362*A+0.28722*B+7.44844E-003*A ² -1.38125E-003*B ² -1.72500E-003*A*B	0.8712 [^]
T _c (°C)	+209.16294-2.41950*A+0.24857*B+0.012219*A ² -9.80556E-004*B ² -1.61667E-003*A*B	0.7358 [^]
ΔH (J/g)	+40.08792-0.84531*A+0.32309*B+4.59375E-003*A ² -9.75000E-004*B ² -2.21667E-003*A*B	0.4040 [^]
HMT 25		
T _o (°C)	+260.50941-4.14103*A+0.36482*B+0.022362*A ² +1.96944E-003*B ² -5.17083E-003*A*B	0.6990 [^]
T _p (°C)	+186.61641-2.32717*A+0.23677*B+0.012892*A ² +1.75764E-003*B ² -3.68750E-003*A*B	0.6787 [^]
T _c (°C)	+84.93502+0.14808*A+0.036189*B-5.46875E-005*A ² +1.24792E-003*B ² -1.67917E-003*A*B	0.7269 [^]
ΔH (J/g)	-118.20733+1.67437*A+1.35019*A-4.94062E-003*A ² -2.96528E-003*B ² -9.53750E-003*A*B	0.7849 [^]
HMT 35		
T _o (°C)	+81.98883-0.64801*A+0.54719*B+6.69531E-003*A ² +2.43403E-003*B ² -9.15833E-003*A*B	0.4927 [^]
T _p (°C)	+55.19244+0.16174*A+0.62822*B+2.19531E-003*A ² +5.50694E-004*B ² -7.98333E-003*A*B	0.5960 [^]
T _c (°C)	+39.14674+0.77941*A+0.63169*B-1.32969E-003*A ² -5.32639E-004*B ² -6.80833E-003*A*B	0.7888 [^]
ΔH (J/g)	+3.89626+0.25995*A-0.15981*B-2.48125E-003*A ² -1.24444E-003*B ² +2.95417E-003*A*B	0.0958 [^]

[^]Not significant at $p \geq 0.05$; A = Linear effect of treatment temperature; B = Linear effect of treatment time; AB = Interaction of temperature and treatment time; A^2 = Quadratic effect of treatment temperature; B^2 = Quadratic effect of treatment time; T_o = Onset temperature; T_p = Peak temperature; T_c = Conclusion temperature; ΔH = Gelatinisation enthalpy.

The regression models for thermal properties of HMT-BG starches are characterised by non-significant lack of fit ($p \geq 0.05$). Non-significant lack of fit is good as this guarantees a good fit of the models for the experimental data. Contour plots illustrating the variations in T_o , T_p , T_c and gelatinisation enthalpy under different HMT treatment conditions are shown in Figures 10 - 12. In HMT 15-BG starch (Figure 10 a-d), the T_o , T_p and T_c of the samples decreased as treatment time and temperature increased whereas gelatinisation enthalpy of the starch samples increased with increase in HMT treatment temperature and time. The decreased gelatinisation temperatures suggest a low degree of crystallinity, stability and low resistance of the granule structure of BG starch to gelatinisation (Alcazar-Alay & Meireles, 2015).

DESIGN-EXPERT Plot

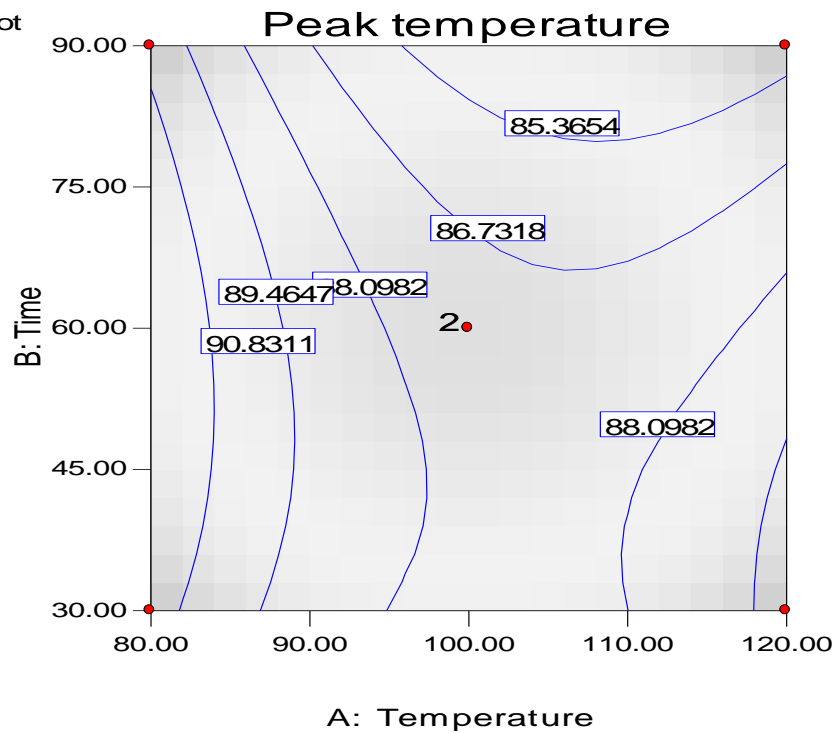
Onset temperature
 ● Design Points
 X = A: Temperature
 Y = B: Time



(a)

DESIGN-EXPERT Plot

Peak temperature
 ● Design Points
 X = A: Temperature
 Y = B: Time



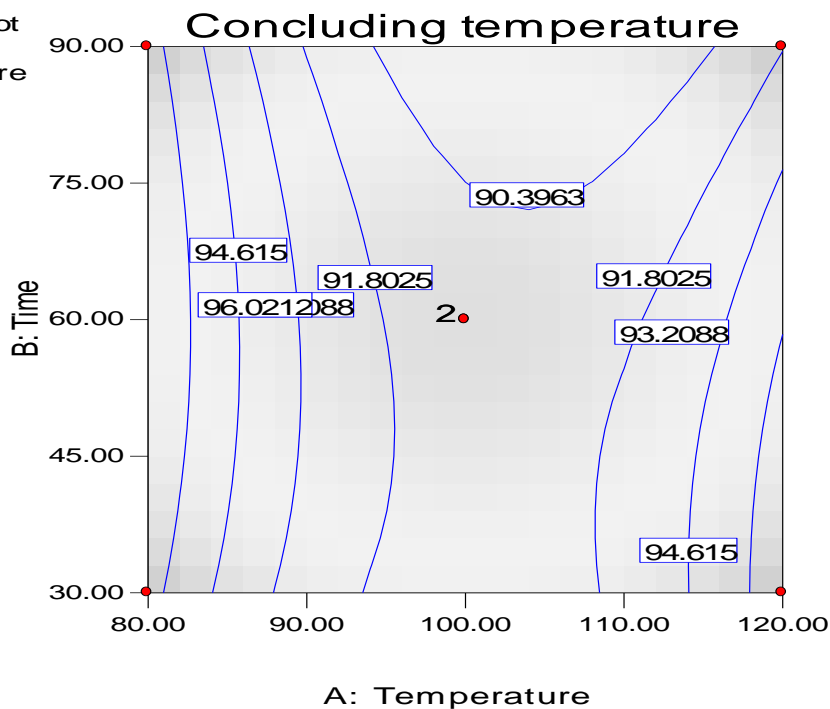
(b)

DESIGN-EXPERT Plot

Concluding temperature
 ● Design Points

X = A: Temperature
 Y = B: Time

(c)



DESIGN-EXPERT Plot

Enthalpy change
 ● Design Points

X = A: Temperature
 Y = B: Time

(d)

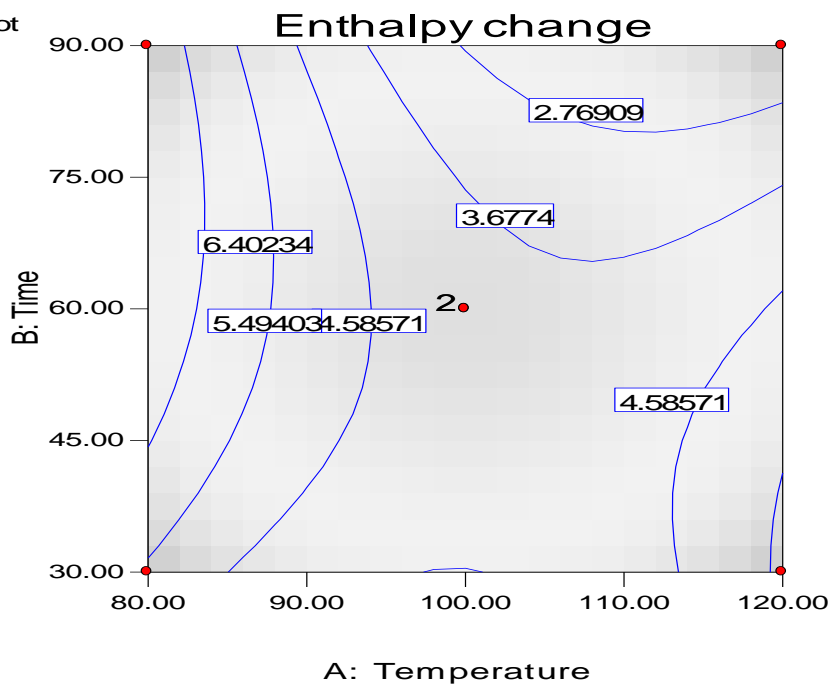
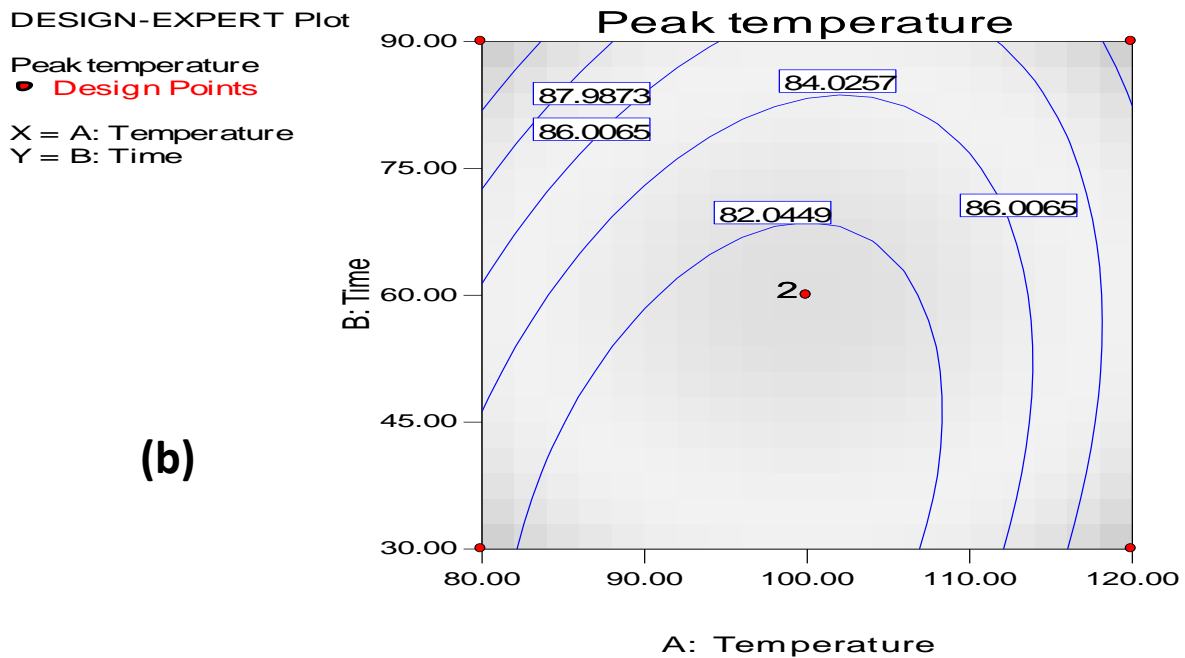
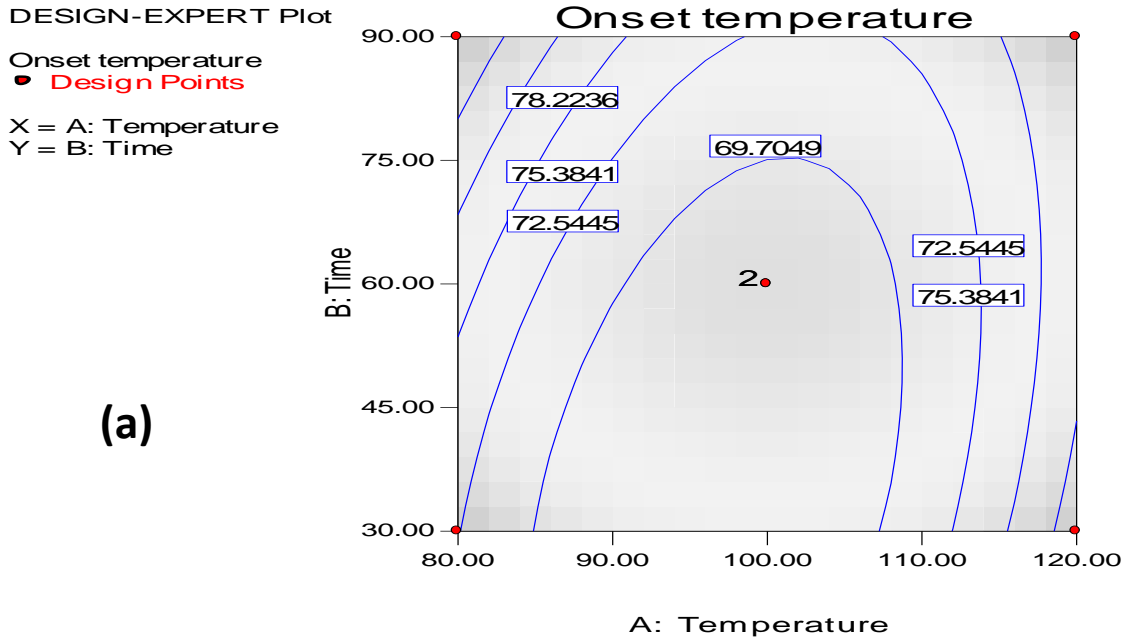


Figure 10 (a-d): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = Onset temperature; (b) = Peak temperature; (c) = Concluding temperature; (d) = Enthalpy change of HMT 15-BG starch.

In HMT 25-BG starch (Figure 11 a-d), the T_p and gelatinisation enthalpy of the starch samples increased with the increase in HMT treatment temperature and time. Whereas T_o and T_c increased as HMT treatment time and temperature increased but later decreased.



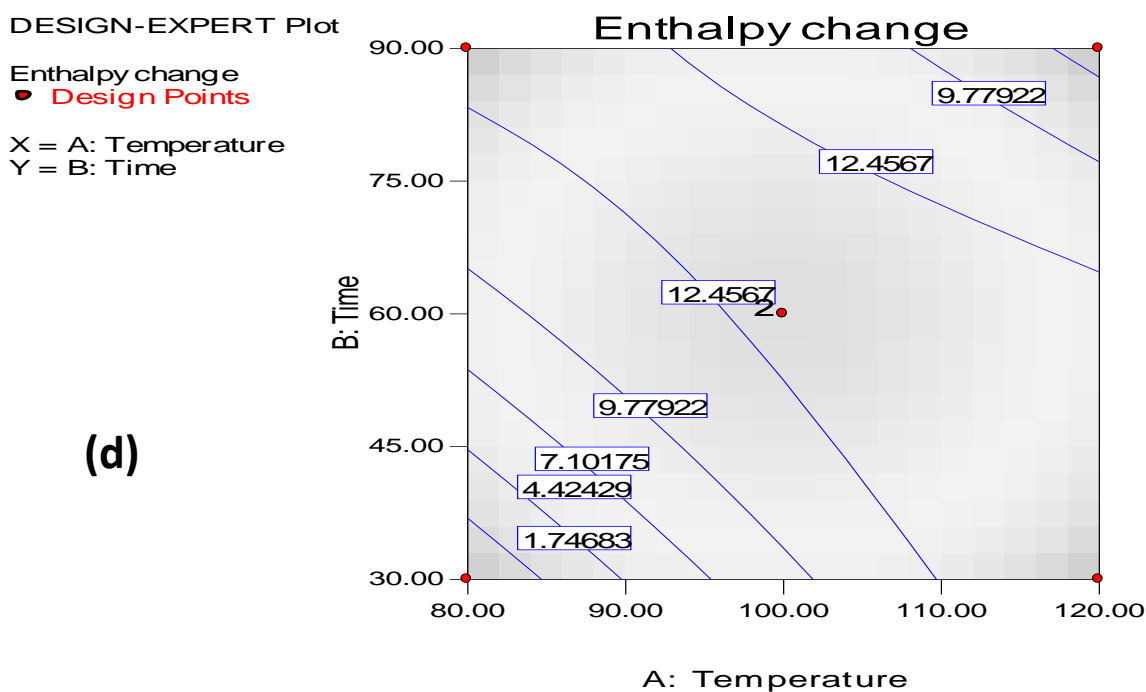
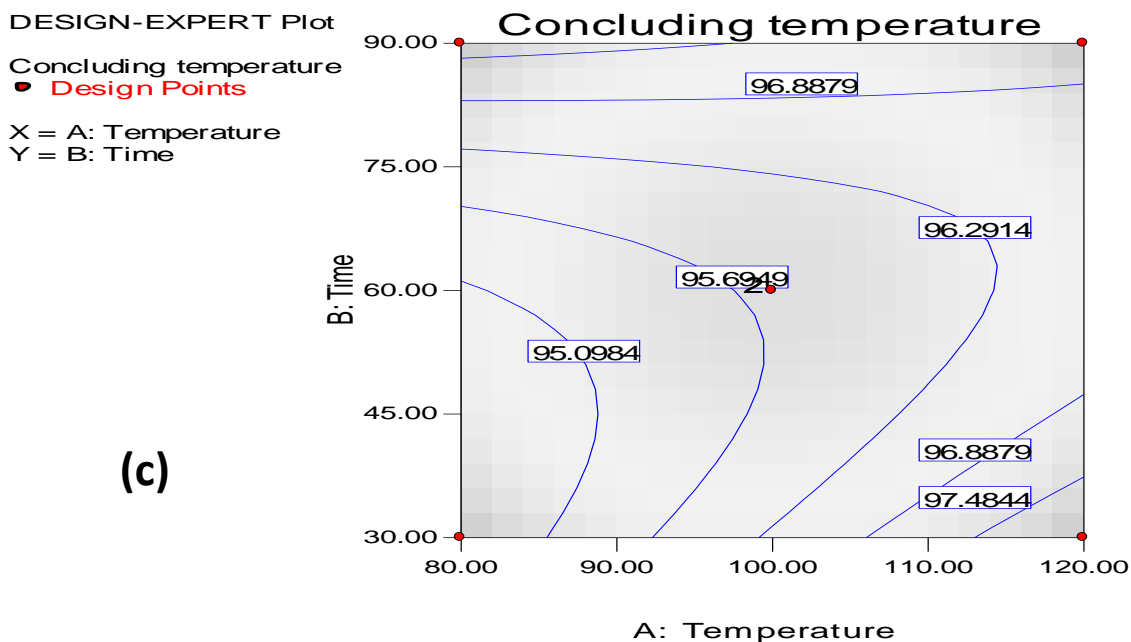


Figure 11 (a-d): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = Onset temperature; (b) =Peak temperature; (c) = Concluding temperature (d) = Enthalpy change of HMT 25-BG starch.

In HMT 35-BG (Figure 12 a-d) starch, the T_o , T_p , T_c and gelatinisation enthalpy (ΔH) of the starch samples all decreased with increase in HMT treatment temperature and time. Generally,

heat moisture treated starch, exhibits an increased ΔH . However, if the incubation temperature is above starch gelatinisation temperature during the treatment, it can cause partial gelatinisation of starch and decrease ΔH (Ai & Jane, 2002; Tester & Debon, 2000; Hoover, 2010). The increase in T_o , T_p , and T_c by HMT is due to amylose-amylose, amylose-amylopectin and amylose-lipid interactions.

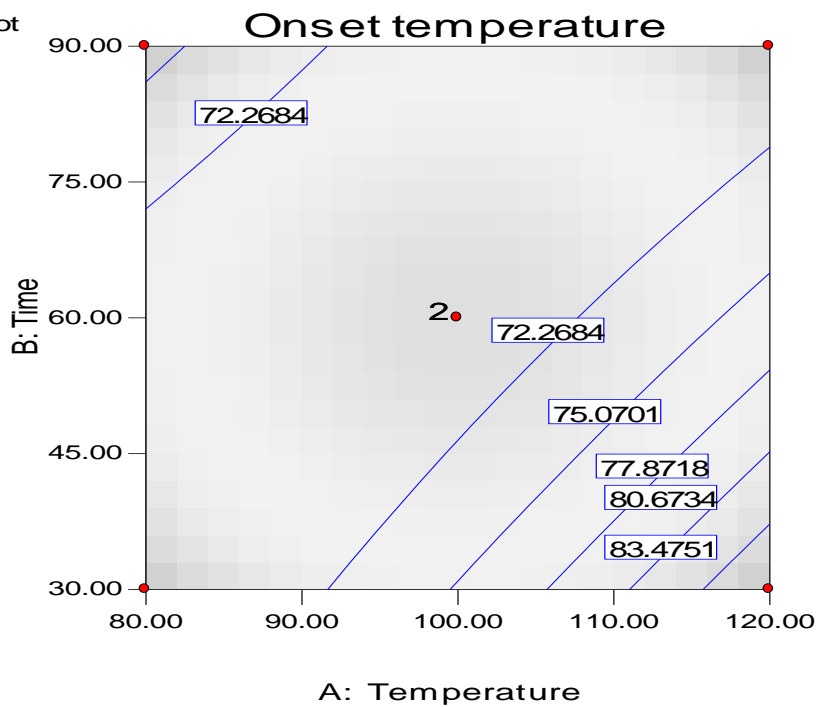
These interactions restrain the mobility of starch chains in the amorphous regions. Consequently, the amorphous regions would require a higher temperature to swell which could contribute to the disruption of the crystalline regions (Adebowale *et al.*, 2005; Zeng *et al.* 2015). However, the extent to which, amylose-amylose, amylose-amylopectin and amylose-lipid interactions associate during HMT, has been shown to be influenced by starch source, amylose chain length, and by the moisture content prevailing during HMT (Hoover, 2010).

DESIGN-EXPERT Plot

Onset temperature
● Design Points

X = A: Temperature
 Y = B: Time

(a)

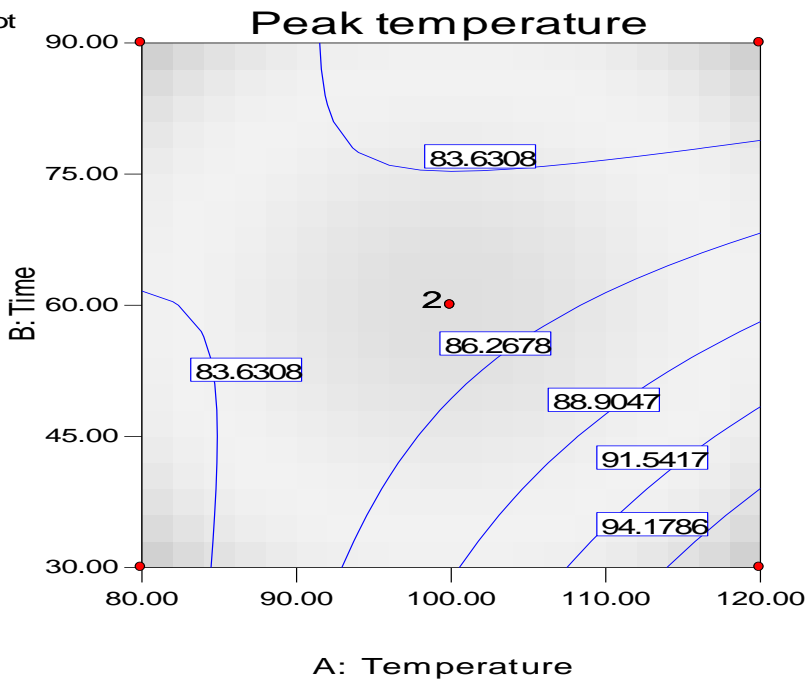


DESIGN-EXPERT Plot

Peak temperature
● Design Points

X = A: Temperature
 Y = B: Time

(b)



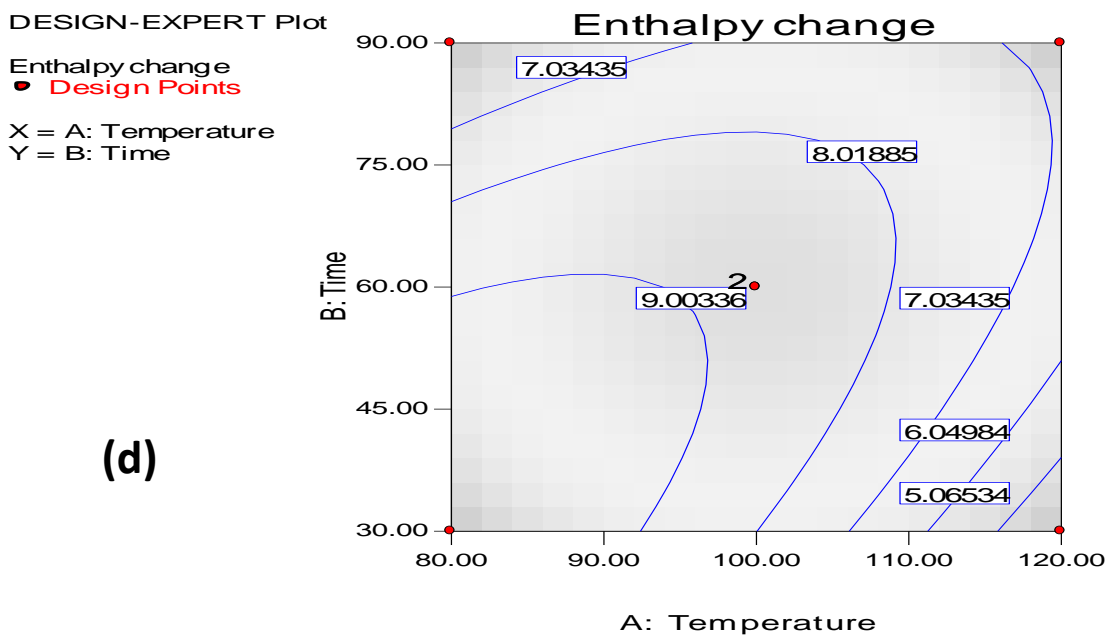
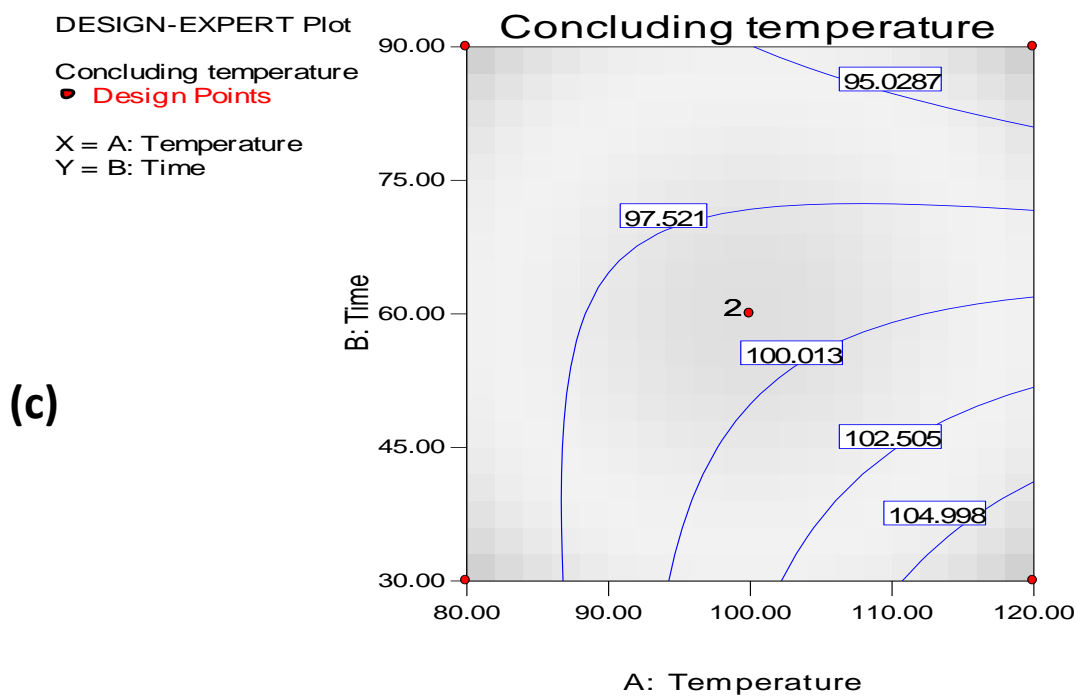


Figure 12 (a-d): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = Onset temperature; (b) = Peak temperature; (c) = Concluding temperature (d) = Enthalpy change of HMT 35-BG starch.

Zeng *et al.* (2015) and Sharma *et al.* (2015) reported an increase in T_o , T_p , and T_c with a decrease in ΔH during HMT treatment of waxy rice and pearl millet starch respectively. A decrease in ΔH after HMT treatment has also been reported for pea, navy bean, normal maize starch, lentil and waxy potato starches (Chung *et al.*, 2009; Chung *et al.*, 2010; Lee *et al.*, 2012). Increase in gelatinisation parameters on HMT have been linked to interaction between amylose–amylose, amylose–amylopectin, amylopectin–amylopectin chains, as well as the formation of other complexes between starch amylose and lipids (Waduge *et al.*, 2006).

Moreover, increase in gelatinisation parameters could also suggest that crystallites disrupted by HMT may have combined to form larger crystallites. The strength of intermolecular bonds in starch promoted by HMT requires a higher temperature to gelatinise the starch granules (Zavereze *et al.*, 2012). The reduction in ΔH promoted by HMT has also been reported in jack bean, corn, potato and cassava starches (Lawal & Adebowale, 2005; Pukkahuta *et al.*, 2008; Gunaratne & Hoover 2002; Sharma *et al.*, 2015). The reduction in gelatinisation enthalpy by HMT is also as a result of the disruption of the crystallites, which are unstable or slightly small, leading to a lower degree of crystallinity and therefore, requiring less energy for the disruption of these crystals (Gunaratne & Hoover, 2002).

According to Chung *et al.* (2009), this reduction by the HMT also suggests that the high temperature during the HMT may increase the mobility of double helices forming the crystalline structure there by disrupting the hydrogen bonds between the helices. In addition, Horndok & Noomhorm (2007) reported that the reduction in gelatinisation after HMT may be due to the partial gelatinisation of amylose and amylopectin molecules, which become highly unstable during heating. Cheng *et al.* (2017) reported that HMT at 25% moisture had a significant effect on gelatinisation parameters of maize starch. However, the findings of this present study negate the observation of Cheng *et al.* (2017). This may be due to the differences in HMT conditions used in Cheng *et al.* (2017) (120 °C, 3 - 9 h) and those used in the present study.

Gelatinisation is necessary for particular processes, such as textile and hydrolyzed starch industries. It also has an impact on the rheological properties and viscosity of pastes, making the starch granule more accessible to enzymatic action. In food, starch is typically in combination with other polymeric ingredients, such as proteins and other polysaccharides, forming different phases (Conde-Petit *et al.*, 2001). Gelatinisation is caused the processing of products from raw materials based on starch. Therefore, the knowledge of the gelatinisation properties of BG starch is necessary for the processing and commercialization of starch.

4.3. Effect of heat-moisture treatment on functional properties of bambara groundnut starch

Application of HMT to BG starch is dependent on its functionality which can be deduced from characteristics such as water absorption, oil absorption capacity, swelling power (SP) and solubility of the starch. The functional properties of BG starch heat moisture treated under different HMT conditions are shown in Table 12.

Table 12: Levels of process variables and values of functional properties for heat-moisture treated bambara groundnut under different treatment conditions

Independent variables		Response variables			
Temperature (°C)	Time (min)	WAC (%)	OAC (%)	SP (g/g)	Solubility (%)
HMT 15					
100	17.57	200	180	12.2	20
100	60	190	240	12.2	10
100	60	170	180	11.2	11.7
100	102.43	200	240	8.7	11.7
120	30	190	200	11.7	21.7
120	90	200	240	11.6	18.3
80	30	180	200	13.7	16.7
80	90	200	240	7	15
71.72	60	180	280	13.3	10
128.28	60	190	200	10.8	10
HMT 25					
100	17.57	210	320	8	13.3
100	60	190	300	8.2	18.3
100	60	180	300	8.5	15
100	102.43	170	320	7.9	15
120	30	170	220	10.7	18.3
120	90	190	200	8	8.3
80	30	210	300	12.2	5
80	90	180	200	4.5	5
71.72	60	180	200	13.2	15
128.28	60	170	220	9.7	10
HMT 35					
100	17.57	170	200	9.2	8.3
100	60	200	300	6.25	11.7
100	60	260	200	7.0	6.7
100	102.43	180	260	6.3	8.3
120	30	200	280	7.8	11.7
120	90	290	100	7	5
80	30	200	280	7.5	13.3
80	90	210	240	7.9	15
71.72	60	100	300	9.9	13.3
128.28	60	290	240	8	8.3

WAC = Water absorption capacity; OAC = Oil absorption capacity; SP = Swelling power.

Water and oil absorption capacity is the ability of starch to soak up water or oil (Sharma *et al.*, 2015). Water absorption capacity (WAC) is an indicator of water binding and emulsifying potential of that particular starch (Sharma *et al.*, 2015; Herlina & Tensiska, 2017). The functional properties of HMT 15-BG starch ranged from 170 - 200 (WAC), 180 - 280 (OAC), 7 - 13.7 (SP) and 10 - 21.7 (solubility), while the functional properties of HMT 25-BG starch ranged from 170 - 210 (WAC), 200 - 320 (OAC), 4.5 - 13.2 (SP) and 5 - 18.3 (solubility). The functional properties for HMT 35-BG starch ranged from 100 - 290 (WAC), 100 - 300 (OAC), 6.25 - 9.9 (SP) and 5 - 13.3 (solubility).

HMT 25-BG treated at 120 °C for 90 min exhibited the lowest WAC (190) than HMT 15-BG (200) and HMT 35-BG (290) at the same temperature for 90 min. Similarly, HMT 35-BG treated at 120 °C for 90 min exhibited the lowest OAC (100) than HMT 15-BG (240) and HMT 25-BG (200) at the same treatment temperature for 90 min. HMT 35-BG treated at 128.28 °C for 60 min also exhibited the lowest SP (8 g/g) than HMT 15-BG (10.8 g/g) and HMT 25-BG (9.7 g/g) at the same treatment temperature for 60 min. Similarly, HMT 35-BG treated at 100 °C for 17.57 min exhibited the lowest solubility (8.8%) than HMT 15-BG (20%) and HMT 25-BG (13.3%) at the same treatment temperature for 17.57 min. This could suggest that higher moisture contents coupled with short HMT treatment time lessen the water binding and emulsifying potential of BG starch.

Analysis of variance (ANOVA) of the effect of model parameters on functional properties of HMT 15-BG, HMT 25-BG and HMT 35-BG starch is shown in Table 13. Treatment time had a significant linear effect ($p \leq 0.05$) on swelling power, for HMT 15-BG starch. In HMT 35-BG starch, WAC was significantly affected by quadratic effect of temperature and time while solubility was significantly affected by linear effect of time and quadratic effect of temperature. However, temperature and treatment time had no significant ($p \geq 0.05$) effect on the functional properties (OAC, SP and solubility) of the HMT 25-BG starch.

The comparison of functional properties of HMT-starches from different sources and the present study is as shown in Table 14. The Table reflects disparities in findings obtained from the literature and the present study. These disparities could be attributed to the rearrangement of molecular chains during modification and the strengthening of inner forces, which prohibits water absorption within starch matrices (Li & Gao, 2010). HMT treatment has been reported to cause some alterations in the crystalline regions and the amorphous regions of starch granules. Amylose content and starch chain length is a factor that greatly affects the functional properties of the final

product (Adebowale *et al.*, 2009; Olayinka *et al.*, 2008; Chung *et al.*, 2009; Gunaratne & Hoover, 2002; Zeng *et al.*, 2015).

The hydrophilic group of modified starches is more capable to bind with water and therefore facilitates the absorption of water (Herlina & Tensiska, 2017). The regression models for predicting the functional properties of heat moisture treated BG starch is shown in Table 15. The regression models for these functional properties of HMT-BG starches are characterised by non-significant lack of fit ($p \geq 0.05$). Non-significant lack of fit is good as this guarantees a good fit of the models for the experimental data.

Table 13: Analysis of variance of the effect of model parameters on functional properties of heat-moisture treated bambara groundnut starch

Model terms	WAC (%)	OAC (%)	SP (g/g)	Solubility (%)
	P-value			
HMT 15				
A-temperature	0.5475 [^]	0.2453 [^]	0.7965 [^]	0.5087 [^]
B-time	0.9650 [^]	0.1018 [^]	0.0258 ^v	0.2154 [^]
A ²	0.8472 [^]	0.0720 [^]	0.8509 [^]	0.6855 [^]
B ²	0.0867 [^]	0.3314 [^]	0.2877 [^]	0.1187 [^]
AB	0.7202 [^]	0.7007 [^]	0.0514 [^]	0.8440 [^]
HMT 25				
A-temperature	0.4198 [^]	0.6388 [^]	0.9102 [^]	0.8983 [^]
B-time	0.3359 [^]	0.5619 [^]	0.3531 [^]	0.0871 [^]
A ²	0.1673 [^]	0.9731 [^]	0.9627 [^]	0.4973 [^]
B ²	0.3845 [^]	0.9731 [^]	0.3123 [^]	0.8852 [^]
AB	0.7567 [^]	0.8997 [^]	0.1692 [^]	0.3303 [^]
HMT 35				
A-temperature	0.2315 [^]	0.2032 [^]	0.7967 [^]	0.4033 [^]
B-time	0.0885 [^]	0.1718 [^]	0.3477 [^]	0.0269 ^v
A ²	0.0183 ^v	0.0983 [^]	0.7408 [^]	0.0214 ^v
B ²	0.0312 ^v	0.7054 [^]	0.4609 [^]	0.3117 [^]
AB	0.1986 [^]	1.0000 [^]	0.5330 [^]	0.4164 [^]

[^]Not significant at $p \geq 0.05$; ^vSignificant at $p \leq 0.05$; A = Linear effect of treatment temperature; B = Linear effect of treatment time; A² = Quadratic effect of temperature; B² = Quadratic effect of treatment time; WAC = Water absorption capacity; OAC = Oil absorption capacity; SP = Swelling power.

Table 14: Comparison of some functional properties of various heat-moisture treated starches in previous and present studies

Source of starch	Temperature (°C)	Time (Min)	WAC (%)	OAC (%)	SP (g/g)	Solubility (%)	References
Present study							
BG	71.72-128.28	17.57-102.43	170 – 200	180 – 280	7 - 13.7	10 - 21.7	
BG	71.72-128.28	17.57-102.43	170 – 210	200 – 320	4.5 - 13.2	5 - 18.3	
BG	71.72-128.28	17.57-102.43	100 – 290	100 – 300	6.25-9.9	5 - 13.3	
Previous studies							
Locust bean	110	1440	–	–	5.57	39.5	Sankhon <i>et al.</i> , 2013
Mung bean	80-120	960	–	–	9.4 - 10.94	5.22 - 6.58	Barau & Srivastav, 2017

WAC = Water absorption capacity; OAC = Oil absorption capacity; SP = Swelling power.

Table 15: Regression models relating functional properties and model parameters of heat-moisture treated bambara groundnut starch

Responses	Model	Lack of fit p-value
HMT 15		
WAC (%)	$+225.73223-0.22411*A-1.40952*B+3.12500E-003*A^2+0.015278*B^2-4.16667E-003*A*B$	0.6564 [^]
OAC (%)	$+894.49747-13.83211*A-0.22978*B+0.068750*A^2 +0.013889*B^2-8.33333E-003*A*B$	0.1853 [^]
SP (g/g)	$+31.78465-0.22710*A-0.23229*B+2.81250E-004*A^2-7.63889E-004*B^2+2.75000E-003*A*B$	0.3697 [^]
Solubility (%)	$+41.24699-0.31813*A-0.49932*B+2.06250E-003*A^2+4.16667E-003*B^2-7.08333E-004*A*B$	0.1883 [^]
HMT 25		
WAC (%)	$-137.08947+5.94822*A+1.52737*B-0.02968*A^2-7.63889E-003*B^2-4.16667E-003*A*B$	0.5748 [^]
OAC (%)	$+173.67641+0.79289*A-0.11193*B-3.12500E-003*A^2-1.38889E-003*B^2+8.33333E-003*A*B$	0.6836 [^]
SP(g/g)	$+36.16259-0.22549*A-0.54158*B+1.40625E-004*A^2+1.45139E-003*B^2+3.37500E-003*A*B$	
Solubility (%)	$+84.76349-1.04836*A-0.58004*B+3.93750E-003* A^2+3.61111E-004*B^2+4.16667E-003*A*B$	0.4853 [^]
HMT 35		
WAC (%)	$+1216.09073-22.00000*A+1.42974*B+0.10156*A^2-0.038194*B^2+0.037500*A*B$	0.1986 [^]
OAC (%)	$-920.85155+24.36244*A-2.06904*B-0.11563*A^2+9.72222E-003*B^2-6.75016E-016*A*B$	0.5649 [^]
SP (g/g)	$+11.95766-0.074911*A-9.12987E-003*B+5.78125E-004*A^2+5.90278E-004*B^2-7.91667E-004*A*B$	0.5168 [^]
Solubility (%)	$-72.90530+1.62479*A+0.18970*B-7.57813E-003* A^2-1.06250E-003*B^2-1.33333E-003*A*B$	0.7748 [^]

[^]Not significant at $p \geq 0.05$; A = linear effect of treatment temperature; B = linear effect of treatment time; AB = interaction of temperature and treatment time; A^2 = quadratic effect of treatment temperature; B^2 = quadratic effect of treatment time; WAC = water absorption capacity; OAC = oil absorption capacity; SP = swelling power.

Contour plots illustrating the effects on WAC, OAC, SP and solubility under different HMT treatment conditions are shown in Figures 13 - 15. In HMT 15-BG starch (Figure 13 a-d), the WAC, solubility and SP decreased as treatment time and temperature increased while OAC of the starch increased with increase in HMT treatment temperature and time.

DESIGN-EXPERT Plot

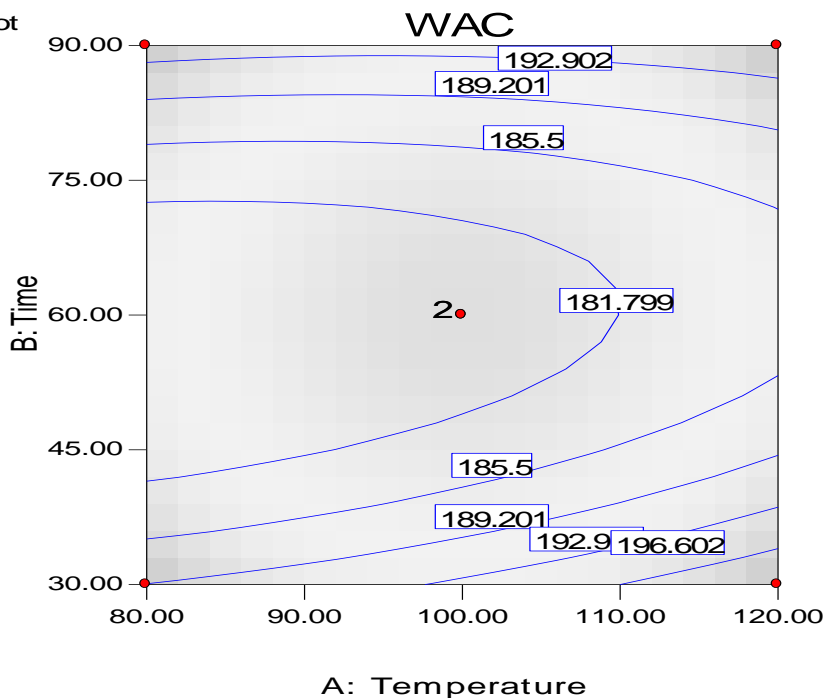
WAC

● Design Points

X = A: Temperature

Y = B: Time

(a)



DESIGN-EXPERT Plot

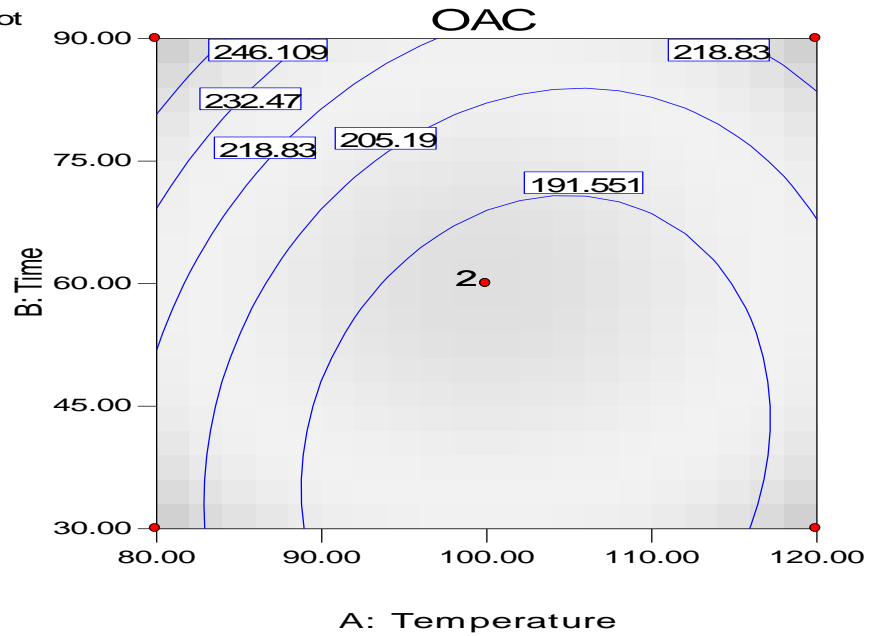
OAC

● Design Points

X = A: Temperature

Y = B: Time

(b)



DESIGN-EXPERT Plot

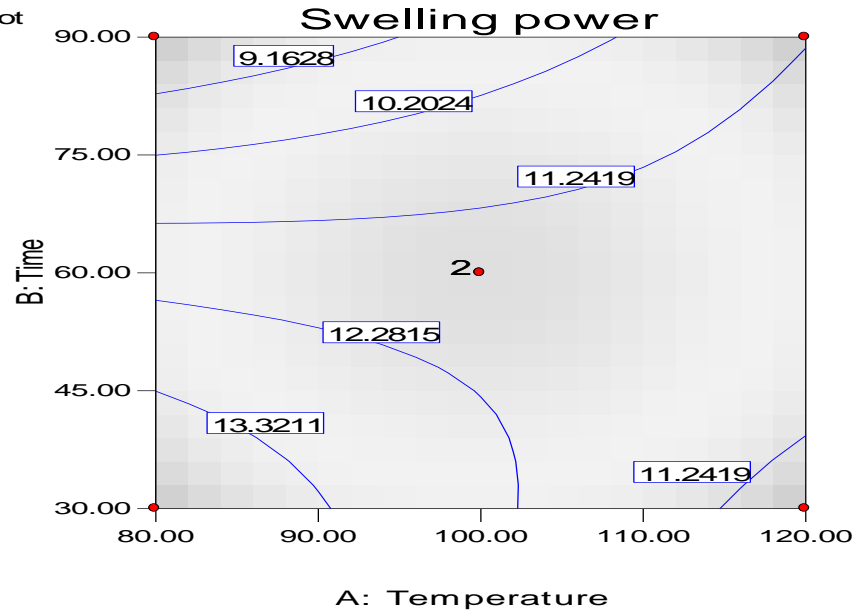
Swelling power

● Design Points

X = A: Temperature

Y = B: Time

(c)



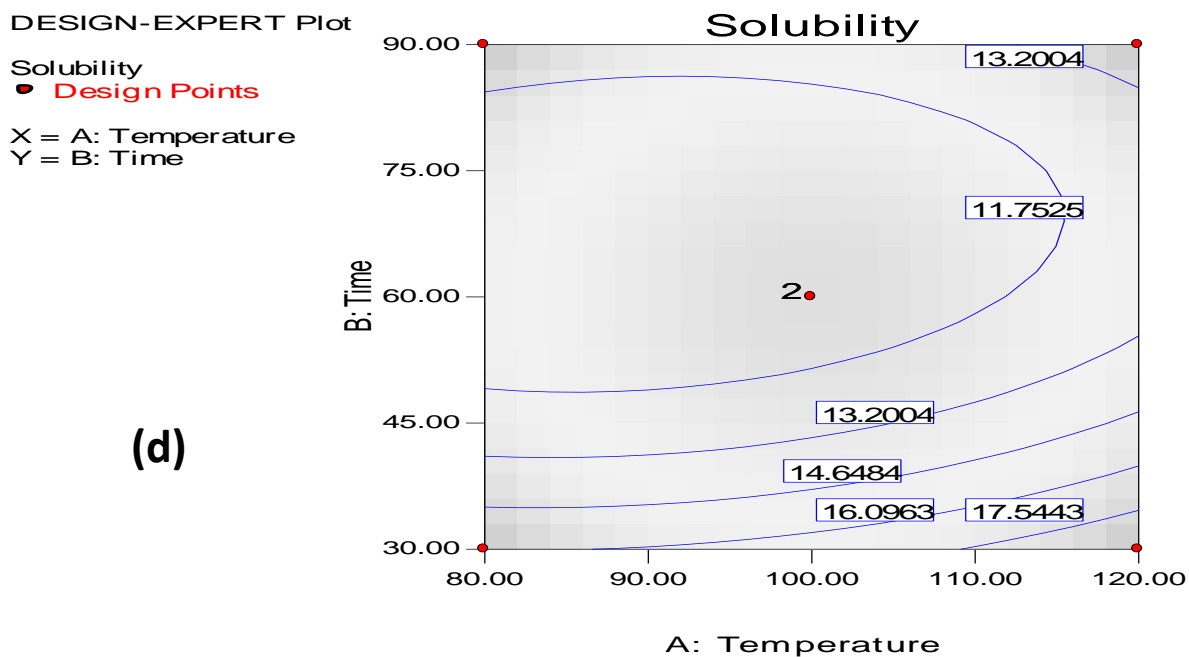


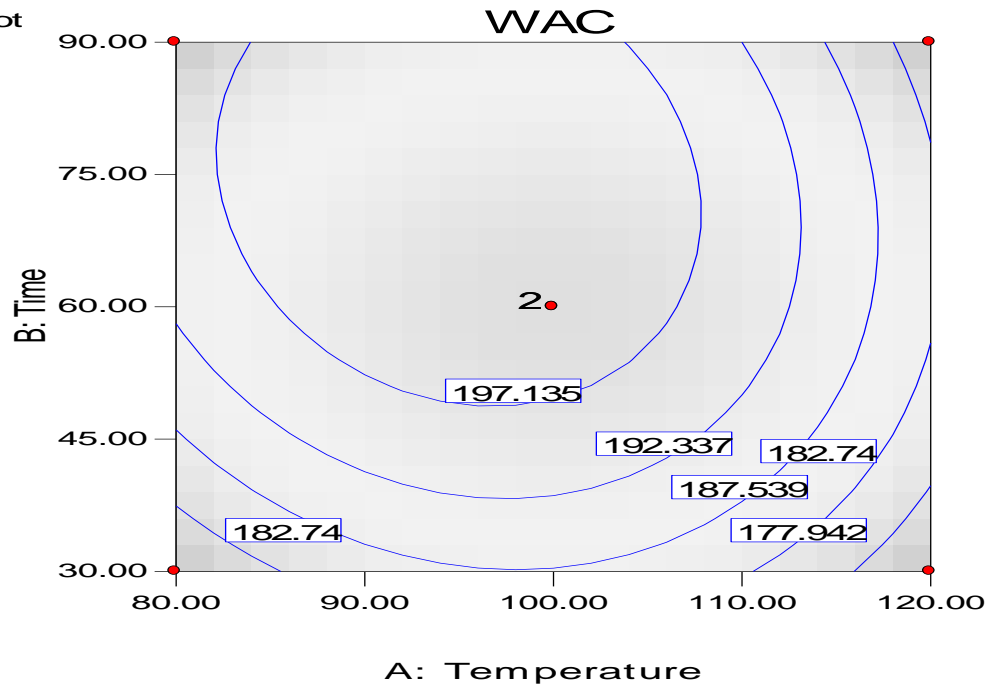
Figure 13 (a-d): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = WAC; (b) = OAC; (c) = SP; (d) = solubility of HMT 15 BG starch.

In HMT 25-BG starch (Figure 14 a-d), WAC and OAC increased as HMT treatment time and temperature increased while SP and solubility of the starch decreased with increase in HMT treatment temperature and time.

DESIGN-EXPERT Plot

WAC
 ● Design Points
 X = A: Temperature
 Y = B: Time

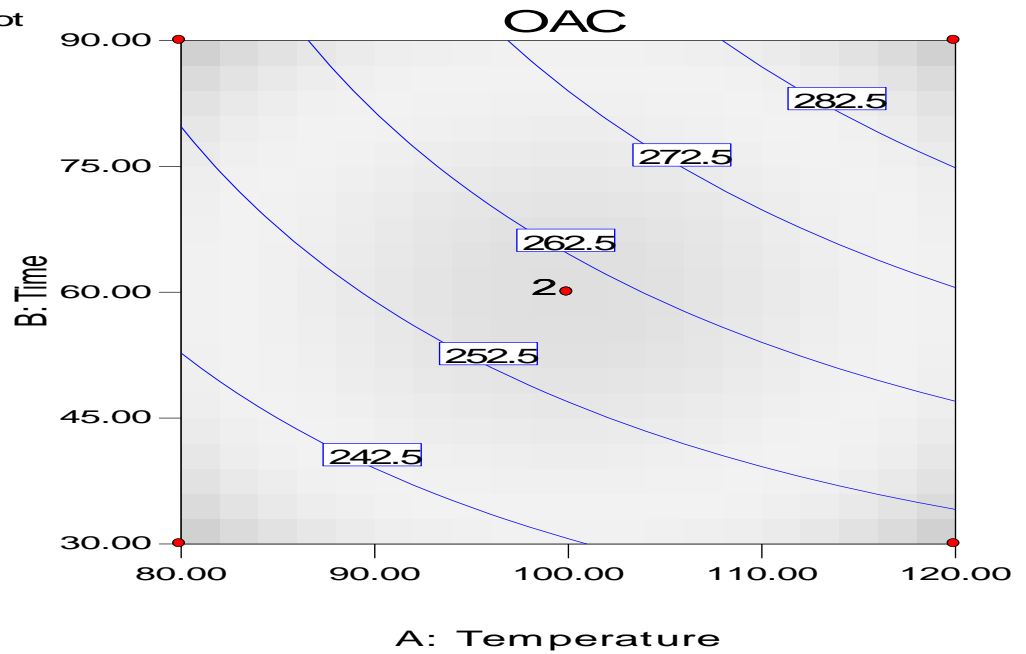
(a)



DESIGN-EXPERT Plot

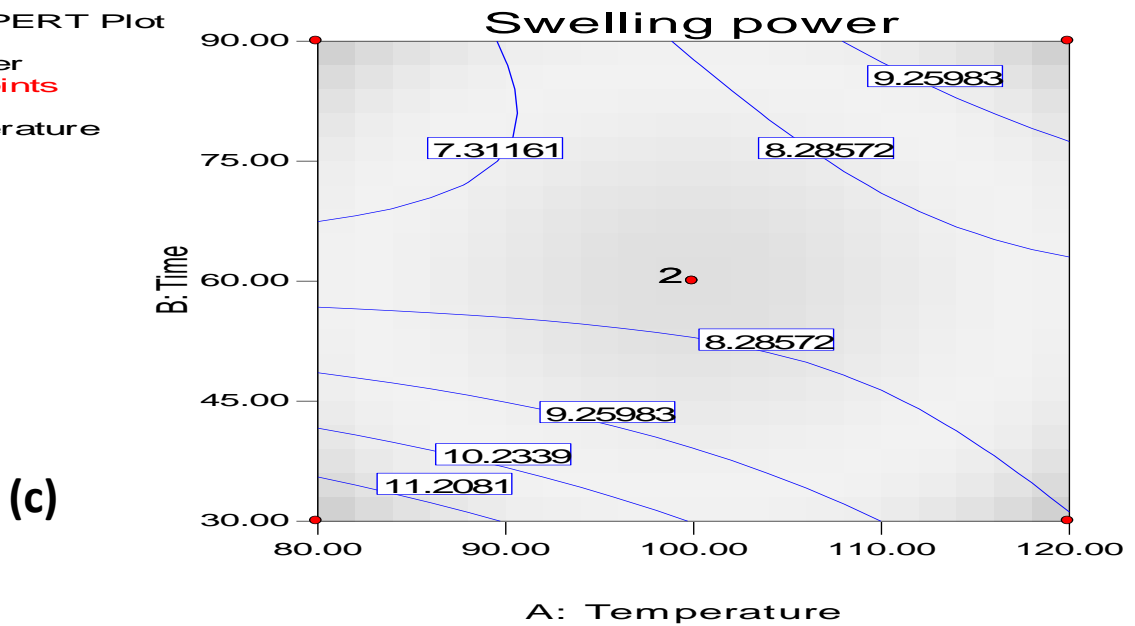
OAC
 ● Design Points
 X = A: Temperature
 Y = B: Time

(b)



DESIGN-EXPERT Plot

Swelling power
 ● Design Points
 X = A: Temperature
 Y = B: Time



DESIGN-EXPERT Plot

Solubility
 ● Design Points
 X = A: Temperature
 Y = B: Time

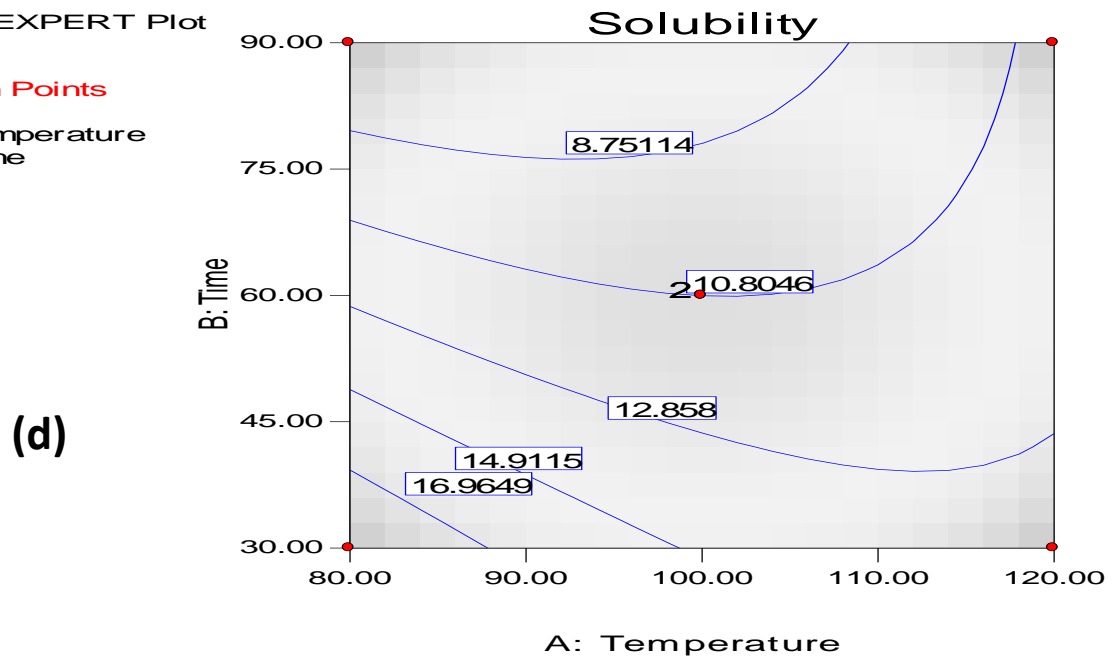


Figure 14 (a-d): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = WAC; (b) = OAC; (c) = SP; (d) = Solubility of HMT 25-BG starch.

In HMT 35-BG starch (Figure 15 a-d), the OAC, solubility and swelling power decreased as treatment time and temperature increased while WAC of the starch increased with increase in HMT treatment temperature and time.

DESIGN-EXPERT Plot

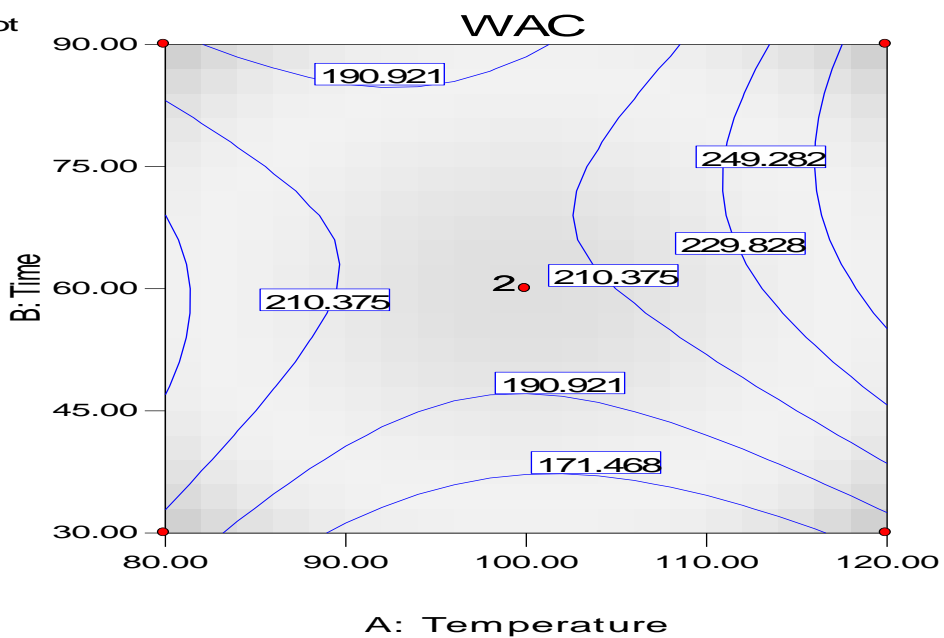
WAC

● Design Points

X = A: Temperature

Y = B: Time

(a)



DESIGN-EXPERT Plot

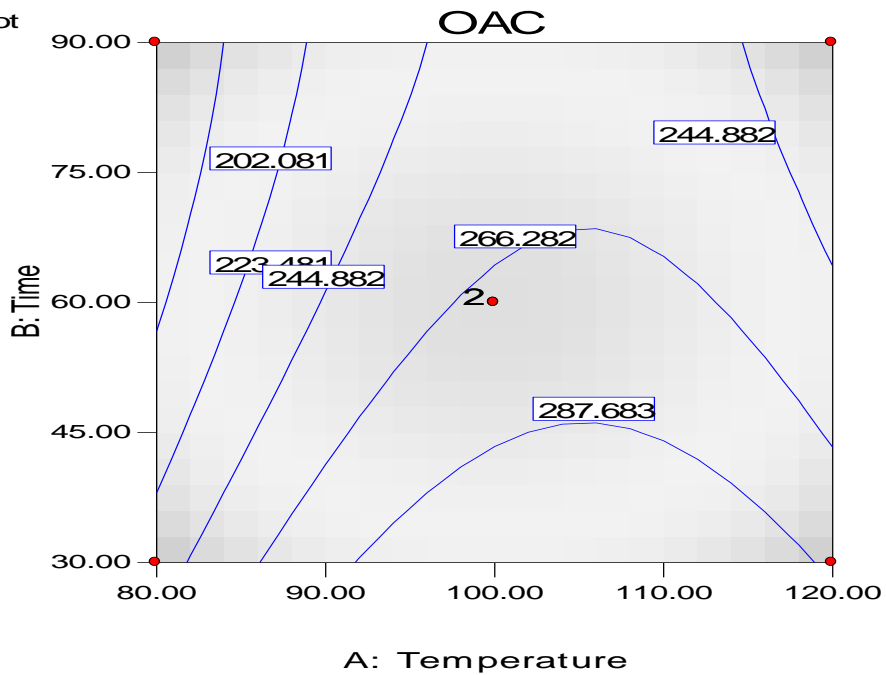
OAC

● Design Points

X = A: Temperature

Y = B: Time

(b)

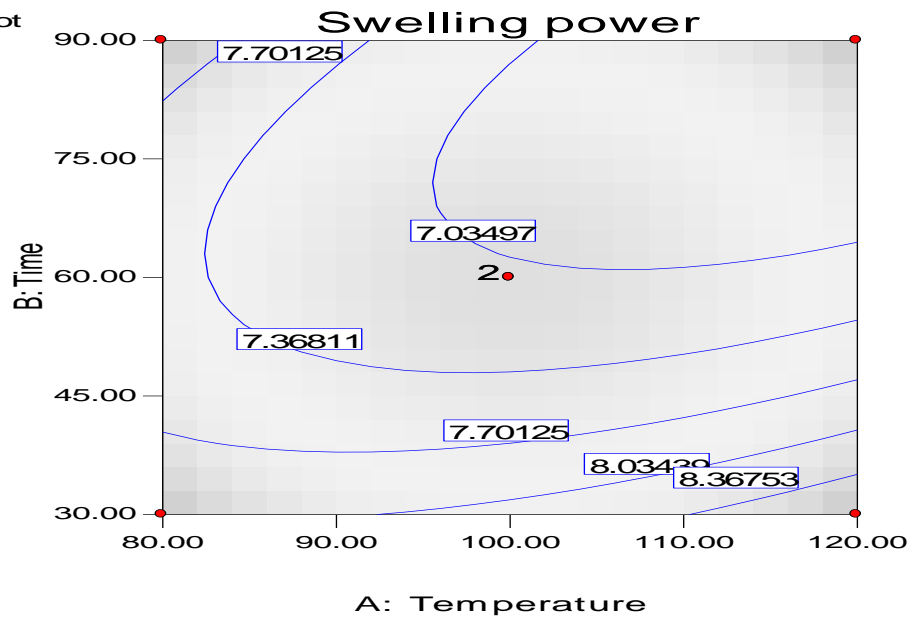


DESIGN-EXPERT Plot

Swelling power
● Design Points

X = A: Temperature
Y = B: Time

(c)



DESIGN-EXPERT Plot

Solubility
● Design Points

X = A: Temperature
Y = B: Time

(d)

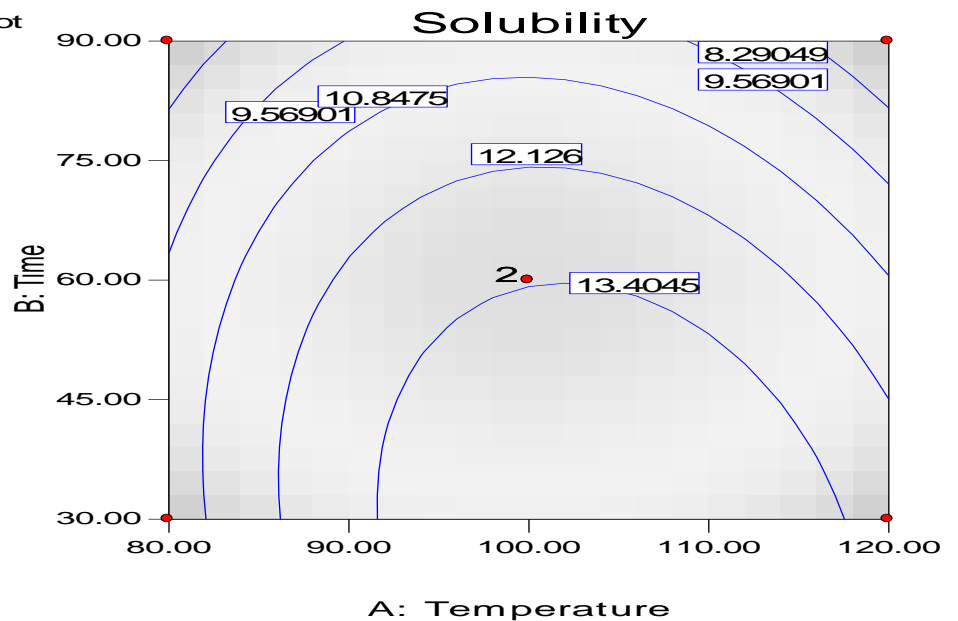


Figure 15 (a-d): Contour response surface plots illustrating the effects of HMT treatment temperature and time on (a) = WAC; (b) = OAC; (c) = SP; (d) = solubility of HMT 35-BG starch

Chung *et al.* (2009); Gunaratne & Hoover (2002); Senanayake *et al.* (2013); Olayinka *et al.* (2008); Zeng *et al.* (2015); Sharma *et al.* (2015) ; Adebowale *et al.* (2005) all reported a decrease in swelling power and solubility for corn, cassava, sweet potato sorghum, waxy rice, pearl and finger millet starches due to application of HMT. The decrease in solubility suggests

that there was a strengthening of bonds and an increase of interactions among amylopectin-amylopectin molecules there by slowing them down from leaching out of the starch granules. Consequently, the reduction in swelling power by HMT is caused by internal reorganisation of starch granules, resulting in interaction between starch functional groups, making it form more ordered double helical amylopectin side chain clusters (Hoover & Manuel, 1996).

The decrease in swelling capacity can also be attributed to the structural re-association of starch chains caused by the HMT and thus restriction for hydration, while starch solubility results from the leaching of amylose, which separates from starch granules and thus spreads out of starch granules during swelling (Zavareze *et al.*, 2012). Sarkar (2016) reported an increase in WAC and OAC in HMT buckwheat starch. Water absorption capacity may increase in the starch when amylose and amylopectin are loosely associated (Sarkar, 2016).

Nadir *et al.* (2015) also reported high WAC but a decrease in OAC by HMT in potato starch. This variation by HMT is due to an increase or decrease in hydrophobic and hydrophilic tendencies of the starch which affects oil and water absorption capacity of the starch (Adebowale *et al.*, 2009). The hydrophobic sites of starch promote oil absorption, while the hydrophilic site of the starch promotes water absorption (Alcazar-Alay & Meireles, 2015; Adebowale *et al.*, 2009). Therefore, the ability of starch to absorb oil and water is a good indication of the emulsifying potentials of the starch (Adebowale *et al.*, 2005).

4.4. Correlation analysis

Correlation analysis was carried out to determine the correlation between colour characteristics, functional properties and thermal properties of heat moisture treated BG starch as shown in Tables 16 (HMT 15), Table 17 (HMT 25) and Table 18 (HMT 35) respectively. The Tables revealed a mixture of positive and negative correlations among the variables. Generally, negative correlation is a relationship between two variables in which one variable increases as the other decreases, and vice versa. Whereas, a positive correlation exists when one variable decreases as the other variable decreases, or one variable increases while the other increases. In HMT 15, a correlation existed between WI and L^* ; ΔE and WI; ΔE and L^* ; H^0 and a^* ; T_o and T_p ; T_p and T_c as well as T_o and T_c . However, there was no correlation between the thermal properties and colour characteristics except for T_o which had a significant correlation with b^* .

Furthermore, it could be observed that WAC correlated with T_o and T_p . A correlation between WI and L^* ; ΔE and WI; ΔE and L^* ; H^0 and a^* , T_o and T_p ; T_c and T_p ; OAC and T_c was observed under HMT 25. However, there was no correlation between the thermal properties and colour characteristics (Table 16). Furthermore, thermal properties except for T_c correlated with

solubility. Solubility is normally affected by higher temperatures and is increased by the weathering of the starch granules by heating during HMT (Adebowale *et al.*, 2005). However, T_o is very low to have a notable effect on solubility. In HMT 35, a correlation was observed between WI and L^* ; and WI; ΔE and L^* ; H^o and a^* ; WAC and OAC; OAC and solubility; SP and T_o ; SP and T_c ; T_o and T_p ; T_o and T_c ; as well as between T_p and T_c .

Table 16: Correlation matrix of colour, functional and thermal properties of HMT 15 bambara groundnut starch

	L*	a*	b*	C*	WI	ΔE	H°	WAC (%)	OAC (%)	SP (g/g)	Solubility (%)	T _o (°C)	T _p (°C)	T _c (°C)	ΔH (J/g)
L*	1														
a*	-0.113	1													
b*	-0.088	0.752*	1												
C*	-0.094	0.860**	0.981**	1											
WI	0.989**	-0.221	-0.232	-0.235	1										
ΔE	-0.963**	0.114	0.121	0.108	-0.956**	1									
H°	0.076	-0.734*	-0.120	-0.294	0.093	-0.052	1								
WAC (%)	-0.508	-0.078	0.180	0.130	-0.512	0.528	0.365	1							
OAC (%)	-0.428	0.134	-0.058	0.031	-0.401	0.235	-0.200	0.194	1						
SP (g/g)	0.281	0.306	0.008	0.100	0.264	-0.322	-0.459	-0.514	-0.121	1					
Solubility (%)	0.212	-0.178	-0.250	-0.240	0.251	-0.086	0.060	0.353	-0.410	0.109	1				
T _o (°C)	0.349	-0.362	-0.642*	-0.608	0.438	-0.314	-0.176	-0.686*	-0.103	0.262	0.084	1			
T _p (°C)	0.448	-0.331	-0.549	-0.522	0.522	-0.429	-0.132	-0.715*	-0.059	0.257	0.007	0.979**	1		
T _c (°C)	0.517	-0.244	-0.248	-0.243	0.549	-0.558	0.072	-0.56275	0.168	0.231	-0.124	0.726*	0.842**	1	
ΔH (J/g)	0.519	-0.025	0.198	0.170	0.485	-0.603	0.263	-0.132	0.265	0.012	-0.405	-0.123	0.047	0.433	1

** Correlation is significant at the 0.01 level; * correlation is significant at 0.05 level; WAC = Water absorption capacity; OAC = Oil absorption capacity; SP = swelling power; ΔH = Gelatinisation enthalpy; ΔE = Total colour difference; WI = Whiteness index; L* = Lightness/whiteness; a* = Yellowness/redness; b* = Blueness/greenness; C* = Chroma; H° = Hue.

Table 17: Correlation matrix of colour, functional and thermal properties of HMT 25 bambara groundnut starch

	L*	a*	b*	C*	WI	ΔE	H°	WAC (%)	OAC (%)	SP (g/g)	Solubility (%)	T _o (°C)	T _p (°C)	T _c (°C)	ΔH (J/g)
L*	1														
a*	-0.788**	1													
b*	-0.854**	0.773**	1												
C*	-0.876**	0.853**	0.983**	1											
WI	0.999**	-0.811**	-0.872**	-0.895**	1										
ΔE	-0.924**	0.775**	0.846**	0.870*	-0.925**	1									
H°	0.251	-0.669*	-0.077	-0.213	0.268	-0.164	1								
WAC (%)	0.057	-0.188	-0.105	-0.140	0.073	0.080	0.145	1							
OAC (%)	0.312	-0.027	-0.164	-0.191	0.308	-0.155	-0.163	0.425	1						
SP (g/g)	0.189	-0.191	0.163	0.103	0.161	-0.036	0.507	0.090	-0.055	1					
Solubility (%)	0.321	-0.377	-0.210	-0.257	0.324	-0.301	0.387	-0.346	0.269	0.209	1				
T _o (°C)	-0.201	0.323	0.285	0.365	-0.217	0.161	-0.214	-0.331	-0.469	-0.062	-0.487	1			
T _p (°C)	-0.287	0.369	0.368	0.436	-0.305	0.252	-0.180	-0.249	-0.581	0.034	-0.637*	0.960**	1		
T _c (°C)	-0.368	0.321	0.469	0.491	-0.383	0.398	0.071	-0.097	-0.679*	0.273	-0.659*	0.701*	0.863**	1	
ΔH (J/g)	-0.334	0.413	0.281	0.342	-0.328	0.500	-0.195	0.205	-0.122	-0.306	-0.632*	0.421	0.497	0.570	1

** Correlation is significant at the 0.01 level; * Correlation is significant at 0.05 level; WAC = Water absorption capacity; OAC = Oil absorption capacity; SP = Swelling power; ΔH = Gelatinisation enthalpy; ΔE = Total colour difference, WI = Whiteness index; L* = Lightness/whiteness; a* = Yellowness/redness; b* = Blueness/greenness; C* = Chroma; H° = Hue.

Table 18: Correlation matrix of colour, functional and thermal properties of HMT 35 bambara groundnut starch

	L*	a*	b*	C*	WI	ΔE	H°	WAC (%)	OAC (%)	SP (g/g)	Solubility (%)	T _o (°C)	T _p (°C)	T _c (°C)	ΔH (J/g)
L*	1														
a*	-0.779**	1													
b*	-0.722*	0.830**	1												
C*	-0.773**	0.939**	0.969**	1											
WI	0.998**	-0.807**	-0.761*	-0.809**	1										
ΔE	-0.950**	0.844**	0.662*	0.772**	-0.951**	1									
H°	0.688*	-0.946**	-0.617	-0.783**	0.706*	-0.799**	1								
WAC (%)	-0.754*	0.465	0.188	0.299	-0.726*	0.755*	-0.535	1							
OAC (%)	0.464	-0.433	-0.119	-0.270	0.452	-0.629	0.453	-0.634*	1						
SP (g/g)	0.637*	-0.770**	-0.535	-0.641*	0.647*	-0.628	0.849**	-0.511	0.123	1					
Solubility (%)	0.553	-0.477	-0.200	-0.328	0.544	-0.665*	0.512	-0.592	0.757*	0.303	1				
T _o (°C)	0.321	-0.664*	-0.525	-0.590	0.349	-0.338	0.692*	-0.211	0.094	0.786**	0.183	1			
T _p (°C)	0.089	-0.389	-0.134	-0.226	0.107	-0.196	0.491	-0.277	0.335	0.525	0.454	0.748*	1		
T _c (°C)	0.271	-0.539	-0.351	-0.424	0.289	-0.281	0.604	-0.243	0.134	0.683*	0.476	0.855**	0.793**	1	
ΔH (J/g)	0.298	-0.125	-0.214	-0.183	0.303	-0.263	0.056	-0.082	-0.033	0.086	0.147	-0.368	-0.182	-0.293	1

** Correlation is significant at the 0.01 level; * Correlation is significant at 0.05 level; WAC = Water absorption capacity; OAC = Oil absorption capacity; SP = Swelling power; ΔH = Gelatinisation enthalpy; ΔE = Total colour difference, WI = Whiteness index; L* = Lightness/whiteness; a* = Yellowness/redness; b* = Blueness/greenness; C* = Chroma; H° = Hue.

4.5. Optimisation of heat moisture treatment of bambara groundnut starch HMT 15, HMT 25, and HMT 35

The optimum heat moisture treatment conditions for BG starch HMT 15, HMT 25, and HMT 35 were found to be 80 °C for 30 min, 105.74 °C for 30 min and 113.16 °C for 30 min respectively. The predicted values of colour characteristics, functional and thermal properties for BG starch HMT 15, HMT 25, and HMT 35 are as reflected in Table 19. Desirability of the obtained optimum conditions were 0.63, 0.56 and 0.64 for HMT 15, HMT 25, and HMT 35 respectively.

Table 19: Predicted values of colour, functional and thermal properties of bambara groundnut starch HMT 15, HMT 25 and HMT 35

Responses	HMT 15	HMT 25	HMT 35
L*	82.08	81.18	79.79
a*	1.49	1.80	1.96
b*	3.16	3.65	4.62
C*	3.55	3.99	5.03
H°	65.16	64.23	66.55
WI	81.71	80.75	79.15
ΔE	18.19	19.51	20.47
WAC (%)	189.27	185.65	162.90
OAC (%)	213.51	244.42	302.10
SP (g/g)	14.36	9.65	8.45
Solubility (%)	16.06	14.07	14.13
T _o (°C)	86.68	69.01	81.90
T _p (°C)	91.40	81.69	93.84
T _c (°C)	96.50	96.87	105.67
ΔH (J/g)	5.36	11.20	5.66

WAC = Water absorption capacity; OAC = Oil absorption capacity; SP = Swelling power; ΔH = Gelatinisation enthalpy; ΔE = Colour difference, WI = Whiteness index; L* = Lightness/whiteness; a* = Yellowness/redness; b* = Blueness/greenness; C* = Chroma; H° = Hue; T_o = Onset temperature; T_p = Peak temperature; T_c = Concluding temperature.

4.6. Microscopy of the optimised heat-moisture treated bambara groundnut starches

Scanning electron micrographs were used to observe the shape and surface characteristics of the unmodified and optimised HMT starches. Images of the unmodified and optimised heat-moisture treated Bambara groundnut starch granules at a magnification of x1.00 to x1.05 are shown in Figure 16. Only the micrographs of the unmodified starch and HMT 15 (80 °C for 30 min), HMT 25 (105.7 °C for 30 min) and HMT 35 (113.16 °C for 30 min) starches were shown as representative samples. Information obtained from scanning electron micrograph indicates oval and round shape for Bambara starch granules, with varying sizes. The range of the granule size width was 4.2 – 4.7 mm and 10 µm for length. Adebowale *et al.* (2009) also observed oval and round shape for African yam bean starch.

The modified starches showed some changes in granule morphology as they seem to disintegrate with application of heat-moisture treatment. Similar observations were made in Zavareze *et al.* (2012) for amylose rice starches. The unmodified starch presented a more agglomerated and cracked surface than the heat moisture treated starches. The HMT starches seem to have smaller cracks and a smoother surface was formed as HMT conditions (moisture and temperature) increased.

Kawabata *et al.* (1994) reported the development of cracks on the surface of treated maize and potato starches and hollowing of starch granules. It has been suggested that cracks may be due to internal cracking occurring during air drying of the starch samples. Heat moisture treatment did not alter the size of the native starch. Generally, HMT has been reported to have no viable effect on the morphology of most starches such as cassava, potato, taro, true and new coco yam, finger millet and rice starches (Gunaratne & Hoover, 2002; Adebowale *et al.*, 2005; Khunae *et al.*, 2007). However, studies show that HMT decreases the relative crystallinity with an increase in the moisture of HMT starches (Zavareze *et al.*, 2010).

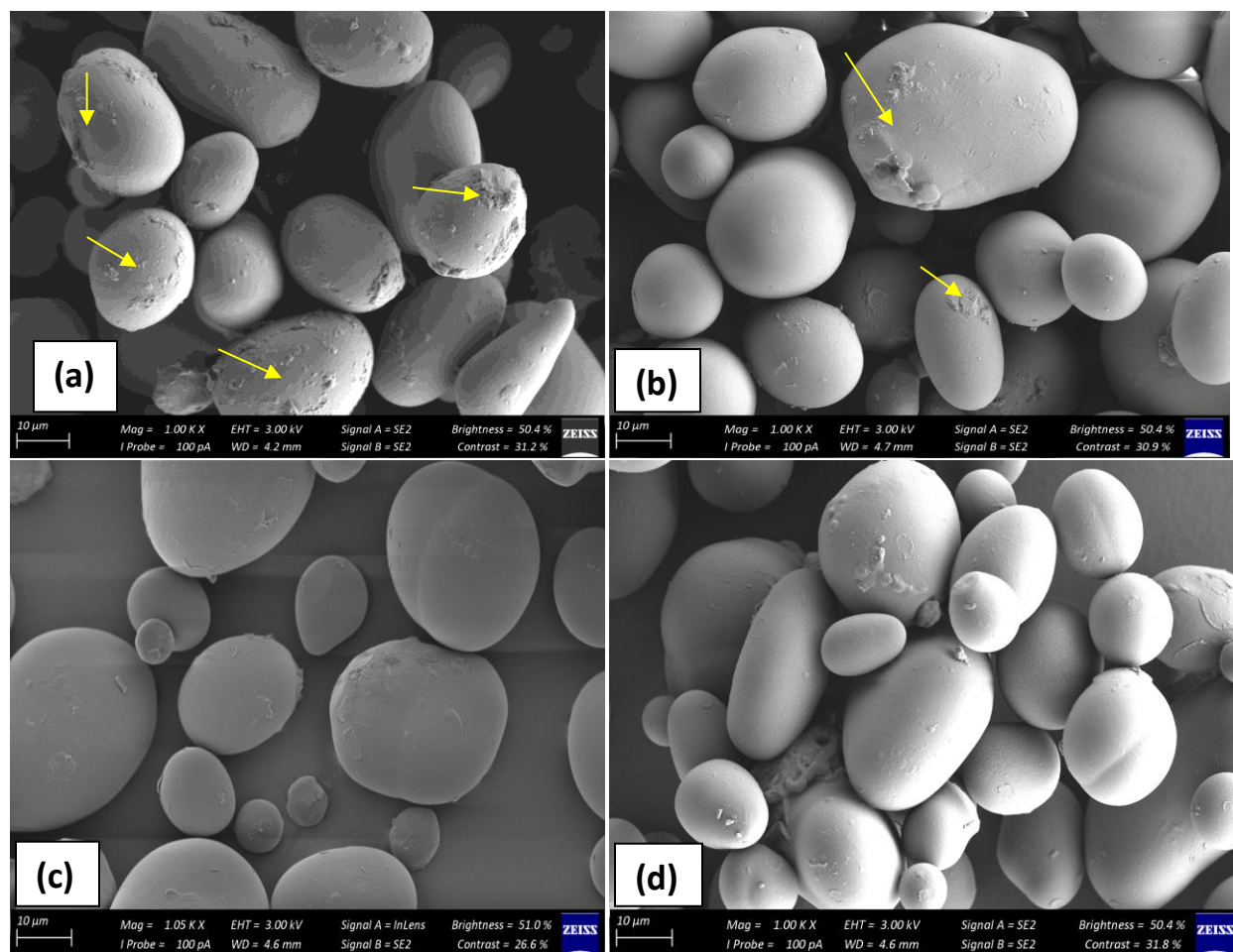


Figure 16: (a)= Scanning electron micrograph of unmodified BG starch (x1.00); (b)= Scanning electron micrograph of heat moisture treated (HMT 15) BG starch (x1.00); (c)= Scanning electron micrograph of heat moisture treated (HMT 25) BG starch (x1.05); (d)= Scanning electron micrograph of heat moisture treated (HMT 35) BG starch (x1.00).

4.7. Fourier transform infrared spectroscopy of the optimised heat-moisture treated bambara groundnut starches

FTIR has been suggested to be sensitive to changes in structure on a molecular level (short-range order). Furthermore, it provides information on the structural arrangement of starch chains near the granule surface, because the infrared beam penetrates only to a certain extent into the granule (Zeng *et al.*, 2015). Unmodified and heat moisture treated Bambara starches showed characteristic FTIR bands linked with common starches. All the samples displayed complex vibrations in the region below 1000 cm^{-1} due to the skeletal vibrations of the glucose pyranose ring. A broad band in the region of $3000\text{--}3600$ was observed with peak at approximately

3425 cm^{-1} (Figure 17). This peak could be attributed to OH stretching (Oyeyinka *et al.*, 2017; Zhang & Han 2006; Ottenhof *et al.*, 2003).

Similar FTIR band patterns were reported for different Bambara cultivar starches by Oyeyinka *et al.* (2017). In the C-H stretching region (2800-3000 cm^{-1}), unmodified and HMT 15 and showed lower peak intensities than HMT 25 and HMT35-BG starches. The variations in peak intensities could be related to the difference in amylose composition (Kizil *et al.*, 2002). Other peaks were observed around 1650 cm^{-1} wavelength, which is probably associated with bending vibrations of H_2O absorbed in the amorphous regions of the starch (Oyeyinka *et al.*, 2017; Zeng *et al.*, 2011). Similar observations were reported for potato, corn and wheat starches (Kizil *et al.*, 2002).

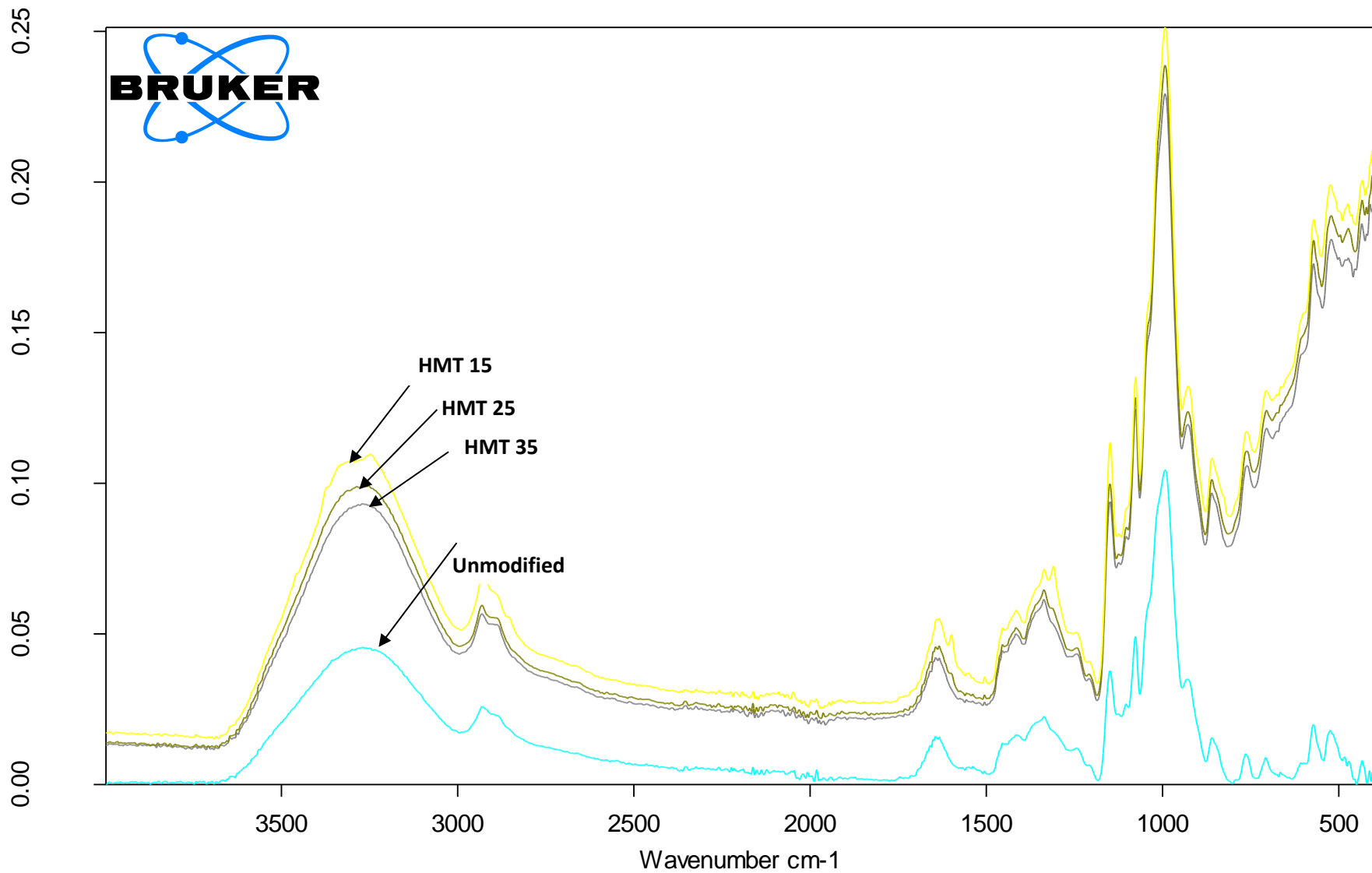


Figure 17: FTIR spectra of unmodified and HMT BG starches

CONCLUSION AND RECOMMENDATION

Starch obtained from Bambara groundnut was subjected to varying heat moisture treatments and colour, functional, and thermal properties were investigated. The treatments seemed to have had a slight effect on colour, functional properties and thermal properties. Heat moisture treatment had a non-significant change on colour properties L^* , whiteness index and colour difference. Reduced colour change and high whiteness values are desired in processed food as it can influence consumer acceptability. Among the functional properties, oil absorption was enhanced as moisture and temperature increased, while water absorption of the starches reduced with increase in moisture and temperature increase. This is desirable in food production where starch is applied as a thickener in food products like sauces and gravies. Heat-moisture treated starches possessed differences in thermal properties.

Treatment time had a significant linear effect ($p \leq 0.05$) on swelling power, for HMT 15-BG starch. In HMT 35-BG starch, WAC was significantly affected by quadratic effect of temperature and time while solubility was significantly affected by linear effect of time and quadratic effect of temperature. However, temperature and treatment time had no significant ($p \geq 0.05$) effect on the functional properties (OAC, swelling power and solubility) of the HMT 25-BG starch. In HMT 15-BG starch WAC, solubility and SP decreased as treatment time and temperature increased while OAC of the starch increased with increase in HMT treatment temperature and time. In HMT 25-BG starch WAC and OAC increased as HMT treatment time and temperature increased while SP and solubility of the starch decreased with increase in HMT treatment temperature and time.

In HMT 35-BG starch, OAC, solubility and swelling power decreased as treatment time and temperature increased while WAC of the starch increased with increase in HMT treatment temperature and time. Information obtained from scanning electron micrograph indicates oval and round shape for Bambara starch granules, with varying sizes. The range of the granule size width was 4.2 – 4.7 mm and 10 μm for length. The unmodified starch presented a more agglomerated and cracked surface than the heat moisture treated starches. The HMT starches seemed to have smaller cracks and a smoother surface was formed as HMT conditions (moisture and temperature) increased. Unmodified and heat moisture treated Bambara starches showed characteristic FTIR bands linked with common starches. All the samples displayed complex vibrations in the region below 1000 cm^{-1} due to the skeletal vibrations of the glucose pyranose ring.

A broad band in the region of 3000-3600 cm^{-1} wave length was observed with peak at approximately 3425 cm^{-1} . The optimum heat moisture treatment conditions for BG starch HMT15,

HMT25, and HMT35 were found to be 80 °C for 30 min; 105.74 °C for 30 min and 113.16 °C for 30 min respectively. Desirability of the obtained optimum conditions were 0.63, 0.56 and 0.64 for HMT 15, HMT 25, and HMT 35 respectively. The findings of this study will help food processors to tailor the process conditions of heat moisture treated Bambara groundnut starch for industrial application and also promote Bambara groundnut as it is an under utilised crop. Further research is required to understand the impact of HMT on other colour attributes such as browning index and on the retrogradation of Bambara starch. This may provide insight on which best HMT conditions could be favourable for industrial application and possible commercialisation of BG starch in SA.

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