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Effect of some process variables on flowability and thermal properties of sprouted tigernut (*Cyperus esculentus*) flour

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ABSTRACT

The effect of drying temperature, drying time and post sprouting sampling time on flowability and thermal properties of tigernut flour was investigated using response surface methodology. Fresh and cleaned tigernut seeds were subjected to different post sprouting time (PSTm), drying temperature (DTp) and drying time (DTm) based on the experimental design, and thereafter milled into flour. The flowability and thermal properties of the flour samples were determined using standard analytical methods. The result showed that increasing drying temperature (from 46.59 ^{o}C to 56.00 ^{o}C) and drying time (from 11.5 h to 13.0 h) increased flow rate from 1.5×10^{-3}kg/s to 2.1×10^{-3}kg/s. Similarly, increasing the post sprouting sampling time (from 35 h to 43.5 h) and drying temperature (from 46.59 ^{o}C to 56.44 ^{o}C) increased the flow rate from 1.75×10^{-3} kg/s to 2.5×10^{-3} kg/s. An increase in drying temperature and post sprouting sampling time caused significant ($p\leq0.05$) reduction in the onset, peak and conclusion temperatures as well as enthalpy value of the tigernut flour. Optimized process conditions of 56.44 ^{o}C drying temperature, 12.03 h drying time and 43.53 h post sprouting sampling time produced flour of 2.75 % moisture content with flow rate of 2.5×10^{-3}kg/s and better thermal properties (60.58 ^{o}C onset temperature, 65.43 ^{o}C peak temperature, 68.03 ^{o}C conclusion temperature and enthalpy of 12.06 J/g indicating less heat energy for processing or cooking).

Keywords: Tigernut flour, process variables, flow rate, thermal properties

INTRODUCTION

Tiger nut (*Cyperus esclentus*) is a tuber crop that belongs to the family *cyperaceae*. It is cultivated throughout the world and is widely found in the Northern parts of Nigeria (Chinma *et al.*, 2010). Tigernut though, an underutilized crop, has been reported to be of high nutritional quality. Research studies have shown that 100 g tigernuts contain 386 kcal (1635 KJ), 7 % proteins, 35 % fat, 31 % starch, 21 % glucose and 6 % fibre (Muhammad *et al.*, 2011), minerals (mainly phosphorous and potassium) and vitamins E and C (Belewu and Belewu, 2007).

Nowadays, tigernuts are also cultivated in northern Nigeria, Niger, Mali, Senegal, Ghana and Togo where they are used primarily uncooked as side dish. In Africa, Niger Republic is the major producer of tigernut, with 125 metric tons, followed by

Ghana with 50 metric tons and about 36.3 metric tons in Nigeria, where production is concentrated in the Northern parts of the country (Balami *et al.,* 2015). In Nigeria, tiger nut is popularly grown around Zaria, Katsina, Mubi and Kastina-Ala (Abano and Amoah, 2011).

However, tigernut is underutilized (Adejuyitan, 2011; Ukwuru and Ogbodo, 2011). Tigernut is not widely known as nutritive food crop. Therefore, it has been poorly investigated (Shikhov *et al.,* 2011). Harvest usually occurs in November or December and after harvesting and cleaning, it is spread on the ground to dry. The drying occurs usually in the sun and take up to three months with the tigernuts having to be turned manually every day for uniform drying (https://en.wikipedia.org/wiki/cyperusesculentus accessed September 2015).

Sun- drying has been reported to have drawbacks of both requiring long drying time and poor product quality (Chou and Chua 2001; Soysal *et al.,* 2006; Therdthai and Zhou, 2009). An increase in the utilization of the crop and formulation of a wide range of possible products from tiger nut will require that the traditional drying technique be replaced with efficient drying systems like the use of industrial dryers such as solar and hot air dryers (Ertekin and Yaldiz, 2004). Processing of local crop increases its economic value and it's country gross domestic products.

Considering the well documented health benefits of tiger nut, substitution of wheat flour with tigernut flour for cake production is advocated as cakes prepared from such composite flour could help in reducing protein energy and micronutrient deficiency prevalent in developing countries such as Nigeria (Chinma *et al.,*2010). Tigernut flour has also been reported to have potential for food applications such as in cakes, biscuits, cookies, pasta, beverage powder production, inclusion in ice-cream, soup thickener and complementary foods production (Elena *et al*., 2012; Gambo and Da'u 2014). The inclusion of tigernut flour in composite flour programme especially in developing countries such as Nigeria has the potential to conserve foreign exchange, provide nutritious food to more people at affordable cost, and widen utilization of indigenous crops in food formulation (Ade-Omowaye *et al.,*2008). The development of tigernut flour is encouraged, as it will increase the economic value of the crops and provide health benefits to consumer (Sanchez *et al.,* 2012). Sprouting of tiger nut seeds improved the chemical, functional and pasting properties of tiger nut flour with reduction anti-nutrients, and could be employed to potentially improve tiger nut nutritional quality, digestibility and utilization in baking, confectioneries as well as complementary food formulations (Chinma *et al.,* 2009). The best known application of tigernut in food technology is the production of tigernut milk beverage, but limited by its very short shelf life (Elena *et al.,* 2012; Sa'id *et al.,* 2016).

Flowability as the measurement of the physical properties of granular materials is an essential step during the development on the optimization of an industrial process (Lumay *et al.,* 2012). Flowability of granular material can be defined as its ability to flow in a desired manner in a specific piece of equipment (Bumiller *et al.,* 2012). The flow characteristics of granular solids have recently gained special importance as measures of the quality of final product online as well as during the later handling and on-shelf storage (Molenda and Stasiak 2002). Flow properties are the specific bulk characteristics and properties of the granular materials that affect flow, which can in principle be measured. On the other hand, the thermal analyses are commonly used to evaluate the physical properties of food samples as a function of temperature. The objective of this study was to determine the effect of drying temperature, drying time and post sprouting sampling time on the flowability and thermal properties of tigernut flour.

MATERIALS AND METHODS

Source of raw material

Fifty kilograms of yellow variety of tigernut seeds was purchased from Sheihk Gumi Central Market, Bakindogo, Kaduna, Kaduna State, Nigeria.

Experimental design

Response surface methodology according to Myers and Montgomery (2002) was adopted. The experimental design is as shown in Table 1.

Sprouting of tigernut seeds

Tigernut seeds were sorted, cleaned and washed with portable water. The seeds were drained, each part spread separately on clean jute bags, covered with damp cotton cloth and then left to sprout for 31.91, 36, 42, 48 and 52.09 h. Water was sprinkled at 12 h interval to facilitate the sprouting process. Root hairs were removed from the sprouted seeds. The flow chart for the preparation of sprouted tigernut flour is shown in Figure 1.

Drying of sprouted tigernut seeds

The sprouted tigernut seeds were dried at the varied temperature and the drying time in a tray dryer (LEECHWA Model LH – 1300) using hot air at 2.0m/s velocity (Figure 1). At the end of the drying period, dried samples were separately milled into flour and passed through a 212 (micron number 70 sieve) (Laboratory Endecott test sieve), cooled and package in an air tight low density polyethene of 1.2mm thickness with 0.920 g/cm^3 density and further stored in covered plastic containers. The samples were then stored in a freezer at -18 ^{o}C from where samples were drawn for analysis.

Analytical methods

Determination of flowability

Flowability test was carried out at the Federal Institute of Industrial Research (FIIRO) Oshodi, Lagos as described by the Metal Powder Industries Federation (MPIF standards 03) using Hall flow-meter apparatus and by application of the principle prescribed by Barbosa-Canovas and Tan (2003). About 50g of the flour sample was weighed and carefully loaded into the Hall flow meter funnel, which was supported with a stand while keeping the discharge orifice at the bottom of the funnel closed by placing a dry finger under it. Care was taken to ensure that the short stem of the funnel was filled. A stop watch was started simultaneously as the removal of the finger from the discharge of the sample and stopped at the instant the last of the powder left the funnel. The elapsed time in seconds was recorded. The flow rate was then calculated as follows:

Flow rate = Elapse time × correction factor (40).

Determination of thermal properties

The thermal properties of flours were determined using a digital scanning calorimeter (DSC Model 204, Nietzsche, Germany) according to the method of Escamilla-Silva *et al*. (2003). A 5 mg of the sample was dispersed in 1:3 ratio of sample to deionized water and was allowed to equilibrate in a beaker for 2 h at room temperature. The suspension (approximately 10 mg) was weighed into DSC aluminuim pans, which was hermetically sealed, and allowed to stand for 30 minutes and heated from 25 °C to 120 °C at rate of 10^0 C/ minute. An empty DSC pan was used as a reference. The thermal properties determined were the Onset temperature (T_O), Peak temperature (T_P), Conclusion temperature (T_C) and gelatinization. Enthalpy of gelatinization (ΔH) was recorded. Determinations were done in three replicates.

Statistical analysis

Data obtained were subjected to analysis of variance (ANOVA) using Minitab 16. Significant difference was accepted at $p < 0.05$.

RESULTS

The effect of process variables on flow rate of tigernut flour is as presented in Table 2. Tigernut dried at 63.41°C for 13 h had the highest flow rate of 2.645 kg/s while samples dried at 46.59 °C for 11.32 h had flow rate of 1.662 kg/s, indicating increase in flour flow rate with increase in temperature. The result of the regression coefficients for flow rate as presented in Table 3 showed that ignoring values not significant at $p \leq 0.05$. The developed model is as shown below. The result of the experimental data obtained from the responses of flow rate is shown in Table 3, and was used to give equation 1.

$$Y= \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{11}X_1^2 + \beta_{22}X_2^2 + \beta_{33}X_3^2 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2X_3 + \varepsilon ..1$$

The model for tigernut flour flow rate as developed from the result of the regression coefficients for flow rate shown in Table 3 while ignoring values not significant at $p \leq 0.05$ is as follows:

$$Y(F_r) = -48.0438 + 0.7783X_1 + 3.5686X_2 + 0.1879X_3 - 0.0062X_1^2 - 0.1338X_2^2 - 0.0018X_3^2 \text{----}2$$

The model showed a synergetic effect on flour flow rate as drying temperature, drying time and post sprouting time increased.

The ANOVA result (Table 4) showed F-calculated value of 135.80 indicating that the model is adequate in explaining the variations in the responses of flow rate obtained. The 3-dimensional graphical optimization for flour flow rate is presented in Figures 2a-c indicating that increasing drying at 46.59°C for 11.5 h to 56 °C for 13 h increased the flow rate from 1.5×10^{-3} kg/s to 2.1×10^{-3} kg/s. Increasing sprouting time from 35 hours to 43.5 hours while increasing drying temperature from 46.59 °C to 56.44°C increased flow rate from 1.75×10^{-3} kg/s to 2.5×10^{-3} kg/s.

The thermal properties of the flour as affected by the varied sample treatments as presented in Table 5 indicated that optimized tigernut sample (sprouted for 43.53 h, dried at 56.44°C for 12.03 h had lower onset, peak and conclusion temperatures and enthalpy value.

Table 1: Variables and values for the tiger nut flour process parameters

Variable	Code	Unit	Variable levels				
			-α	-1	0	+1	+ α
Drying Temperature	X_1	^{0}C	46.59	50	55	60	63.41
Drying time	X_2	Hour	11.32	12	13	14	14.68
Post sprouting time	X_3	Hour	31.91	36	42	48	52.09

X_1, X_2, X_3 and their levels of variation are based on literature (Chinma *et al.*, 2009; Chinma *et al.*, 2010; Komolafe *et al.*,2012) and preliminary experiment.

Table 2: Design matrix, observed predicted values and responses for tigernut flour flow rate

R	**Series**	**Coded**			**Actual**			**Response flow rate (Kg/s)**	
		X_1	X_2	X_3	X_1	X_2	X_2	**Observed**	**Predicted**
1	Factorial	-1	-1	-1	50	12	36	1.886	1.832
2	Factorial	-1	-1	-1	60	12	36	2.538	2.475
3	Factorial	-1	-1	-1	50	14	36	1.938	1.975
4	Factorial	-1	-1	-1	60	14	36	2.538	2.605
5	Factorial	-1	-1	-1	50	12	48	1.894	1.862
6	Factorial	-1	-1	-1	60	12	48	2.427	2.416
7	Factorial	-1	-1	-1	50	14	48	1.930	2.015
8	Factorial	-1	-1	-1	60	14	48	2.487	2.555
9	Axial	$-\alpha$	0	0	46.59	13	42	1.662	1.636
10	Axial	α	0	0	63.41	13	42	2.645	2.632
11	Axial	0	$-\alpha$	0	55	11.32	42	1.968	2.073
12	Axial	0	α	0	55	14.68	42	2.451	2.311
13	Axial	0	0	$-\alpha$	55	13	31.91	2.380	2.393
14	Axial	0	0	α	55	13	52.09	2.252	2.377
15	Centre	0	0	0	55	13	42	2.567	2.571
16	Centre	0	0	0	55	13	42	2.569	2.571
17	Centre	0	0	0	55	13	42	2.569	2.571
18	Centre	0	0	0	55	13	42	2.572	2.571
19	Centre	0	0	0	55	13	42	2.572	2.571
20	Centre	0	0	0	55	13	42	2.572	2.571

X_1 =Drying temperature; X_2 =Drying time; X_3 =Post sprouting sampling time; R=Experimental runs

Table 3: Regression coefficients for flow rate (kg/s)

Term	**Coef**	**SE Coef**	**T**	**P**
Constant	-48.0438	3.4621^{-}	-13877	0.000
DTp	0.7783	0.06057	12.850	0.000
DTm	3.5686	0.32052	11.134	0.000
PSTm	0.1879	0.04570	4.111	0.000
DTp.DTp	-0.0062	0.00041	-15.105	0.000
DTm.DTm	-0.1338	0.01022	-13.093	0.000
PSTm/PSTm	-0.0018	0.00028	-6.429	0.000
DTp.DTm	-0.0007	0.00274	-0.240	0.811*
DTp.PSTm	-0.0007	0.00046	-1.625	0.110*
DTm.PSTm	0.0004	0.00229	0.185	0.854*

R-Sq =96.07% R-Sq(pred) = 93.82% R-Sq(adj) = 95.36%
Drying temperature (DTp) (^{o}C) = X_1, Drying time (DTm) (hour) = X_2
Post sprouting sampling time (PSTm) (hour) = X_3, *values not significant $p<0.05$

Table 4: Analysis of variance for flow rate (kg/s)

Source	**DF**	**Seq SS**	**Adj SS**	**Adj MS**	**F**	**P**
Regression	9	5.520	5.52089	0.61343	135.80	0.01
Linear	3	3.79549	0.98081	0.32694	72.38	0.01
DTp	1	3.59046	0.74592	0.774592	65.13	0.01
DTm	1	0.20406	0.55993	0.55993	123.96	0.01
PSTm	1	0.00098	0.07636	0.07636	16.90	0.01
Square	3	1.71306	1.71306	0.57102	126.41	0.01
DTp.DTp	1	0.81871	1.03060	1.03060	228.16	0.01
DTm.DTm	1	0.70765	0.77434	0.77434	171.43	0.01
PSTm.PSTm	1	0.18669	0.18669	0.18669	41.33	0.01
Interaction	3	0.01234	0.01234	0.00411	0.91	0.443*
DTp.DTm	1	0.00026	0.00026	0.00026	0.06	0.811*
DTp.PSTm	1	0.01193	0.01193	0.01193	2.64	0.110*
Residual Error	50	0.22585	0.22585	0.00452		
Lack-of-Fit	5	0.16769	0.16769	0.03354	25.95	0.010
Pure Error	45	0.05817	0.05817	0.00129		
Total	59	574674				

Drying temperature (DTp) (^{o}C) = $X_{1,}$ Drying time (DTm) (hour) = X_2
post sprouting sampling time (PSTm)(hour) = X_3, *values not significant $p<0.05$

Table 5: Analysis of variance for Thermal Properties of Tigernut Flour

	T_0 (^{o}C)		T_p (^{0}C)		T_c (^{0}C)		ΔH (J/g)	
Sample	Mean	SD	Mean	SD	Mean	SD	Mean	SD
R6	62.140	0.0964	66.447	0.0737	69.757	0.196	13.610	0.270
OPS	60.583	0.240	65.427	0.315	68.027	0.488	12.060	0.0964

SD = Stand Deviation; T_0 = Onset temperature, T_p = Peak temperature, T_c = Conclusion temperature, ΔH = Enthalpy, OPS = Optimized sample.

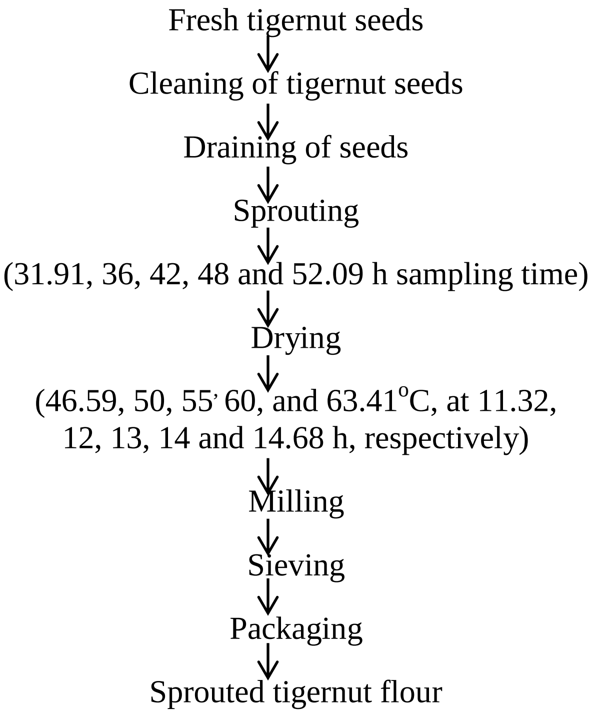
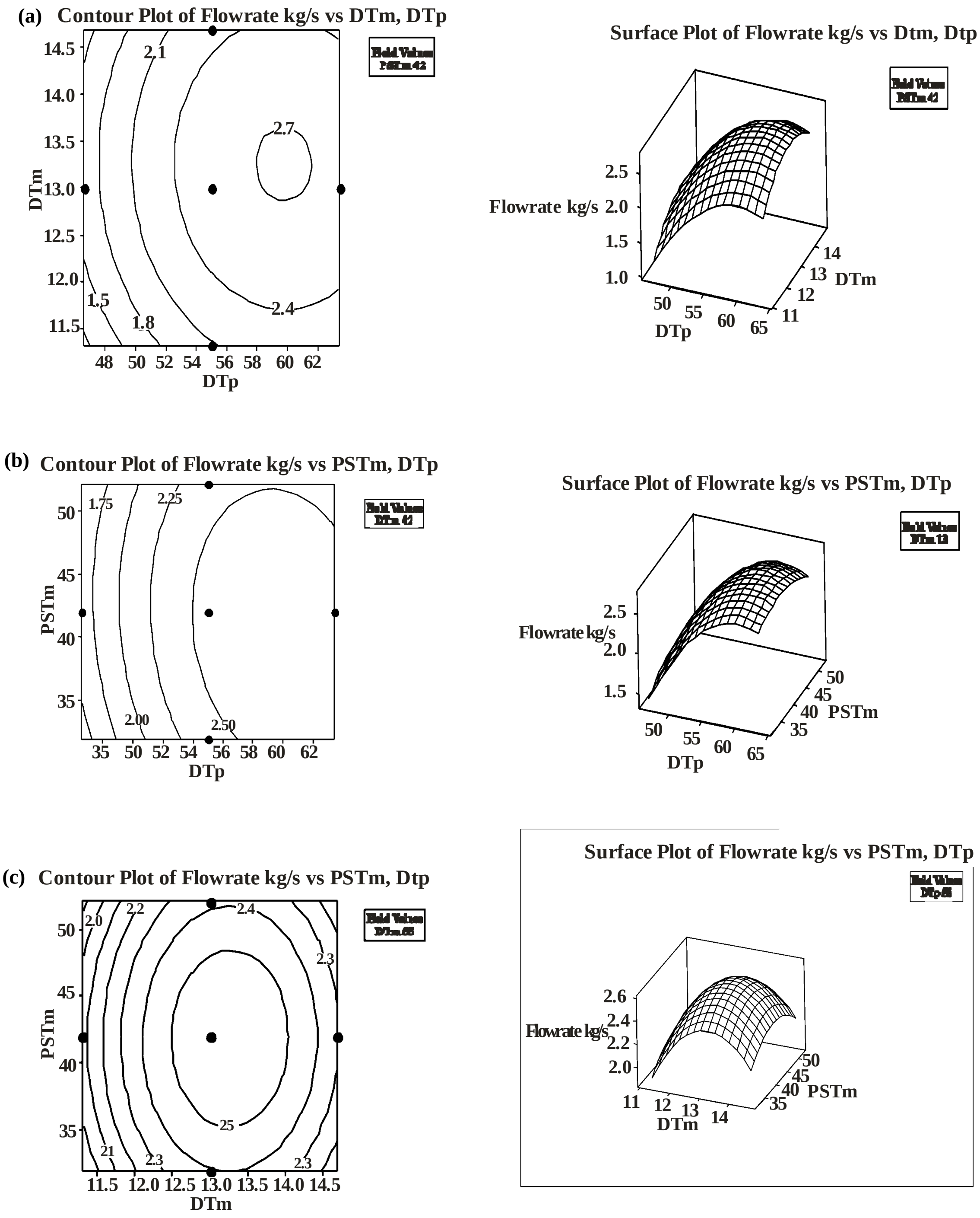


Figure 1: Flow chart for the production of sprouted tigernut flour



(c) **Figure 2 a-c:** Contours and surface plots for flowrate of tigernut flour samples

DISCUSSION

The effects of the production process variables (drying temperature, drying time and post sprouting sampling time) on the flow rate of the tigernut flour as shown in Table 2 indicated that the mass flow rate of the tigernut flour ranged from 1.662×10^{-3} Kg/s to 2.659×10^{-3} Kg/s corresponding to experimental conditions of 46.59 ^{o}C DTp, 13 h DTm and 42 h PSTm for the lowest flow rate; and 55 ^{o}C DTp, 13 h DTm and 42 h PSTm for the highest flow rate, respectively, indicating that increase in drying temperature as observed with the experimental domain significantly increased the tigernut flour flow rate while DTm and PSTm appeared not to have a significant effect the flow rate of the flour. This significant effect of increase in flow rate of the flour with increase in drying temperature might likely be as a result of higher temperature gradient between the moisture in the food material and the hot drying air which is responsible for reduction in moisture content of the product consequently enhancing the mass flow rate of the flour. Similar flow rate (volumetric flow rate) of 1.5 to 3.0g/cm^{3} was reported by Fadeyibi *et al.* (2014) for cassava flour. This indicates that the sole effect of drying temperature, drying time and post sprouting sampling time tend to enhance the flow rate of the flour, while their squared effect tend to be antagonistic to the flour flow rate. . The observed flow rate values were within predicted values at $p \leq 0.05$. The results of the adequacy test of the fitted model for model adequacy being 96.07 % and 95.36 % for R^{2} and R^{2} adjusted, respectively. A suitable model is said to be the one with highest polynomial order and statistically significant if its coefficient of determination R^{2} and R^{2} adjusted is close to unity (Zaibunnisa *et al.*, 2009). The results obtained in this study showed that the model for the response variable is highly adequate to explain the variability in response. According to Chauchan and Gupta (2004), $R^2 \geq 75$ % is acceptable for fitting a model. The probability values obtained in the regression coefficient table indicated a significant model. In general, the smaller the magnitude the more significant the corresponding coefficient term (Mason *et al.*, 2003). The result of the application of Fisher's test on the developed model is shown in the table of analysis of variance (ANOVA)(Table 4).

Similarly, the F-values observed in the ANOVA Table for the tigernut flour flow rate showed values that ranged from 16.90 to 228.16 which are greater than 3.02. Therefore, the predicted quadratic model was statistically significant ($p \leq 0.05$), and most of the variations in the response excluding the insignificant values can be explained by the regression equation for tigernut flour flow rate. The result of the contour plots and surface plot of tigernut flour showed that tigernut flour flow rate increased (from 1.5×10^{-3} kg/s to 2.7×10^{-3} kg/s) as drying temperature and drying time increased from 46.59 ^{o}C to 60 ^{o}C, and 11.5 h to 13 h, respectively.

Holding drying time at 13 h, the effect of the interactions of drying temperature (DTp) and post sprouting sampling time (PSTm) indicated that increase in PSTm (35 h to 43.5 h) and increase in DTp (46.59 ^{o}C to 56.44 ^{o}C) showed increase in flour flow rate from 1.5×10^{-3}kg/s to 2.5×10^{-3}kg/s. Similarly, holding DTp at 55 ^{o}C, the interactive effect of PSTm and DTm on flour flowrate showed that increase in PSTm (31.91 h to 48 h) and increase in DTm (11.5 h to 13 h) showed increase in flow rate from 2.0×10^{-3} kg/s to 2.5×10^{-3} kg/s, but slightly decreased to 2.4×10^{-3}kg/s as the PSTm is beyond 48 h.

Thermal properties enhance investigation into phase transition of starch based foods during heating or cooling and the quantity of heat that is either absorbed or released by the food substance undergoing physical transition.

From the results obtained in this study, the optimized sample (OPS) which corresponded with processing condition of 56.44 ^{o}C drying temperature , 12.03 h drying time and 43.53 h post sprouting sampling time had better thermal properties of 60.58 ^{o}C onset temperature, 65.43 ^{o}C peak gelatinization temperature, 68.03 ^{o}C gelatinization temperature and enthalpy of 12.06 ^{o}C as against 62.14 ^{o}C, 66.45 ^{o}C , 69.76 ^{o}C, and 13.61 ^{o}C onset temperature, peak gelatinization temperature, gelatinization temperature, and enthalpy respectively, for R6 sample indicating that the optimized sample (OPS) will require less heat energy for processing or cooking. The result obtained in this study agreed with Jasim *et al*. (2015). Gelatinization temperature is not an intrinsic property of starchy food, but depends on the water content as a process parameter (Jasim *et al.*, 2015). The thermal transitions of chestnut flour (powder and in dispersion) reported by Jasim *et al*. (2015) showed that the two distinct glass transitions were detected for dry chestnut flour; the first one ranged between 42 and 43 ^{o}C, and the second one ranged from 116 to 121^{o}C. The result obtained in this study is in agreement with Chinma *et al.* (2009) for germinated tigernut seeds. The peak temperature of the optimized sample could be as a result of the presence of protein and fiber in the flour which has been reported to significantly increase the competition for water in the food material, thus, decreasing the amount of water available for food starch gelatinization and the food starch endothermic transition temperatures increased. The reduction in onset, peak and conclusion temperatures could be attributed to starch modification which probably caused a reduction in gelatinization temperature. During sprouting (germination) of seeds, enzymes become active and alpha-amylase activity increases catalyzing starch degradation and consequently increasing the amount dextrin and fermentable sugars (Cornejo *et al.*, 2015). Increase in drying temperature with increase in post sprouting sampling time caused a significant ($p \leq 0.05$) decrease in onset, peak, and conclusion temperatures, and enthalpy value.

CONCLUSION

Based on the results of this study, it is concluded that increasing the drying temperature, drying time and post sprouting sampling time increased the flow rate of tigernut flour. The interactive effect of post sprouting sampling time and drying temperature in enhancing flour flow rate was higher than that of drying temperature and drying time. On the other hand, increase in drying temperature along with increase in post sprouting sampling time significantly reduced the onset, peak and conclusion temperatures as well as the enthalpy value of tigernut flour.

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