ARPN Journal of Engineering and Applied Sciences
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OPTIMIZATION OF ANNEALING TREATMENT IN PRODUCTION OF FUNCTIONAL WHEAT FLOUR

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ABSTRACT

The functionalization of native wheat flours was carried out by using annealing (ANN) process. This ANN process was executed according a full factorial design and the independent variables selected were temperatures and the duration of treatment when initial moisture content of flours was fixed at 70 % (wet basis). Then ANN treatment was optimized by using response surface method (RSM) for three different responses: gelatinization temperature, swelling capacity and lightness color of flours. The effects of two factors were found to be more significant at different level for all responses. The optimal ANN process conditions for having functional flours with high gelatinization temperature, high swelling capacity and high lightness of color, were found at 70% (wb) for initial moisture content of native flour treated at 54°C during25 hours 34 min. The predictable values of the response variables were 70.70 °C for gelatinization temperature, 18.7 N for hardness, 7.3 g/g for Water Bound Capacity and 62.5 for lightness of color.

Keywords: optimization, annealing (ANN), wheat flour, full factorial design, response surface method (RSM).

1. INTRODUCTION

Wheat flour is a component of many culinary meals and the main ingredient in bread making and infant food formula (Despre *et al.,* 2002). Depending on the variety of wheat from which it was formed, and the milling process used, various kind of native wheat flours can be produced. Despite of the diversity of native wheat flour, they do not cover all needs of wheat-based food industry. Their incorporation in some formulations is limited by the lack of desirable characteristics and the excessive development of undesirable functionalities (Martínez *et al.*, 2014).

To overcome these limitations, wheat industry performs development of atypical wheat varieties, or modification of some properties of native flours, by eliminating undesired fractions, or by correcting insufficient functionalities of nature flour. This modification could be performed by internal and external functionalization of flours. Internally functionalized flours are flours, which are supplemented with various additives to improve their qualities. Whereas in externally functionalized flours the same qualities are obtained through chemical, biochemical and physical processes. These functionalized flours bring benefits such as better dough hydration, machinability, cold and hot water or oil binding capacity, textural enhancement, and may substitute some hydrocolloid used as additives or replace fats in some food formulations.

The cereal flours where initial properties have been modified without using chemical reaction and by environmentally friendly processes are designed as clean label functionalized cereal flour. Most of commercial clean label functionalized flours are made by heat, mechanical and/or enzymatic processes which allow users to claim their natural character.

Many research and development studies have shown that, during thermal functionalization of flours, the extent of modifications obtained depends on heat treatment applied and water environment of wheat kernels major components (Kulp and Lorentz, 1981; Jacobs and Delcour, 1998; Stute, 1992; Maache - Rezzoug *et al.*, 2008; Jayakody and Hoover, 2008; Chung *et al.*, 2009). Depending on temperature applied and water content to which flours are put, we distinguish either:

- heat treatments leading to production of completely gelatinized products where particles can form aqueous suspensions, stable and viscous at cold;
- and heat treatments that do not alter the granular character of these flours starches and which don't lead to stable suspensions at cold.

Among heat treatments, those functionalize cereal flour without destroying completely structural feature of their starch granules, one may distinguish annealing (ANN) treatment which is carried out in a medium saturated with water (> 60%) and at relatively low temperatures that do not allow a complete starches gelatinization (Maache-Rezzoug *et al*., 2008).

The annealing (ANN) treatment of cereal flour involves three key parameters: moisture content of the system temperature, and the treatment duration. To date, there is not enough information on how these parameters rates affect wheat flours characteristics. Most of publications in this field being focused on the ANN of extracted starches. Only few research papers have addressed the effect of hydrothermal treatment processes parameters on complex starchy systems like cereal flour (Sun *et al*., 2014; Li *et al.,* 2018).

In the present study some changes induced upon annealing treatment of native wheat flours are

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characterized. Data collected during experimental study allow determining some parameters level that lead to optimal characteristic of functional wheat flour using the method of responses surfaces.

2. MATERIAL AND METHODS

2.1 Preparation of wheat flours

Native wheat flour (Natura -11/680) was provided by Moulins de Statte" from Huy (Belgium). For experimental design, 250 grams of aqueous suspensions of 30% flour contained in Duran® glass bottles are heated to 45, 50 and 55 ° C in a water bath. For each temperature, incubation times of 24, 36 and 48 hours are applied according to a full factorial design. The suspensions of flours thus treated were frozen, then lyophilized, before being reduced to flour using an IKA M20® laboratory mill and finally stored in sealed plastic bottles in a room at 10 °Cbefore analysis.

2.2 Annealing process (ANN) and experimental design

Annealing (ANN) is a hydrothermal process for treating starch and starch compounds. In this process starchy material is suspended in excess water and then incubated for a specified time at temperatures below gelatinization ones. 250 grams of aqueous 30% flour suspensions contained in Duran® glass bottles are heated to 45, 50 and 55 ° C in a water bath. For each temperature, incubation times of 24, 36 and 48 hours are applied.

The experiments were realized according a Full Factorial Design (FFD) in order to optimize ANN process by responses surfaces methodology (RSM). Three levels of each factors were applied according to the experimental domain presented in Table-1. The design consists of 11 experimental conditions (Kumar *et al*., 2015).

Table-1. Experimental domain of full factorial design.

Coded level	T (°C)	D (h)
Lower: -1	45	24
Central: 0	50	36
Higher: +1	55	48

T: temperature of ANN; D: Time of ANN

Two independents variables were considered: the temperature (T) and the time (D) of ANN, when the final moisture content of wheat flours gels were fixed at 70% (wet base). Responses analyzed were gelatinization temperature (Tp) of functionalized flour, the hardness de gel flours (Hardness), the Water Binding Capacity (WBC) of flours, and intensity of wheat flour gel color (L*). This study allowed to investigate the influence of two factors simultaneously (i.e. temperature (T) and heating time (D) of water bath (inANN-process)) on responses related to flour functionality.

Experimental data were used to model the responses by a multiple linear regression. For models quality assessment and significance of quadratics equations coefficients, an analysis of variance (ANOVA) was used.

2.3 Measurement of gelatinization temperature

The flours thermal properties were examined with a DSC 2920 TA Instruments (New Castle, Delaware, USA) with a Refrigerated Cooling Accessory and modulated capability. Samples (around 2.5 mg, db) with distilled water (moisture 70%), were hermetically sealed in aluminum pan and equilibrated at temperature of 10°C for 24 h. Then sample pans were heated from 20 °C to 120 °C at a heating rate of 2°C/min modulated at ± 0.5 °C every 100 s. The instrument was calibrated with indium (Tonset: 156.6°C, ΔH: 28.7 Jg^{-1}) and eicosane (Tonset: 36.8°C, ΔH: 247.4 Jg^{-1}) and an empty pan was used as reference. Gelatinization enthalpy ΔH was calculated by integrating area of the gelatinization endothermic signal divided by the amount of dry flour used for measurement. The onset temperature (To) and peak temperatures (Tp) were computed using NV Thermal analysis software (Universal analysis, New Castle, Delaware, USA) (Malumba *et al*., 2010).

2.4 Measurement of samples hardness

2.4.1 Gel preparation

60 mL of suspensions (at 20% wheat flour) subjected to ANN treatment were prepared in metal cans 1/4b (30 mm high and 73 mm wide) hermetically crimped. For each essay, a can containing a thermocouple enables to follow temperature evolution inside the thermocouple during autoclave (FMC® A091, Zwevegem, Belgium) heating.

Temperature program provides a gradual rise in temperature of 5 ° C / min between 30 and 95 ° C. During heating, cans are subjected to a rotation up to 10 rpm. When set temperature reaches 65 ° C, rotation of cans is stopped. The heating is continued until set temperature (95 ° C) which, once reached, is maintained for 10 minutes. Boxes are then cooled to room temperature and stored for 1 hour before proceeding texturometer measurement. Compression was applied over 0.005 m, while probe advancing at a speed of 0.001m / second. TPA parameters were determined on 3 different samples of each gel.

2.4.2 Characterization of gels texture

Gels textural profile was characterized according to Texture Profile Analysis (TPA) procedure presented by Bourne (1968). It consists of applying two compression cycles on product using an SMS texturometer TAXT2 (Surrey, Great Britain) equipped with a cylindrical probe 0.036 m in diameter (TA / XT2 3.6 R).

2.5 Determination of samples Water Binding Capacity

Samples of 0.1 g of flour (ANN) were accurately weighed into 15 mL Falcon® tubes previously tared. 5 mL of distilled water were added to each tube followed by their incubation at 70 ° C for 10 minutes in a shaking water bath. Tubes were subsequently transferred to a boiling water bath for 10 minutes and then cooled for 10 minutes in a cold

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water bath. Tubes were then centrifuged at 3220 g for 30 minutes at a temperature of -10 ° C. After centrifugation, supernatant was frayed and the pellet in the falcon weighed. Water Binding Capacity of the flour WBC is determined by the following expression:

$$\mathrm{WBC}(g/g) = \frac{m - T}{S} \qquad (1)$$

With T: tare (g); m: Weight of the pellet (g) and S: sample (g)

2.6 Measurement of color

60 mL of suspension at 70% of ANN-flour was prepared in cans (1 / 4b) tightly crimped. For each test, the can was heating in an autoclave FMC® A091 (Zwevegem, Belgium). The temperature program included a gradual temperature rise of 5 ° C / min between 30 and 95 ° C. Cans were then cooled to room temperature and stored for 1 hour prior to color measurement. Gels flour-HMT obtained have undergone color measurement using the Spectrocolorimeter Hunterlab Miniscan® XE (Reston, USA). This allowed to determine gels Lab parameters on white backgrounds. Only L*value was used as response variable.

2.7 Optimization of ANN process

2.7.1 Mathematical modelling and statistical analysis

The experimental data were fitted to the following quadratic model (Eq. 2):

$$Y = \beta_0 + \sum_{i=1}^{3}\beta_i X_i + \sum_{i=1}^{3}\beta_{ii} X_i^2 + \sum_{i=1}^{2}\sum_{j=i+1}^{3}\beta_{ij} X_i X_j \qquad (2)$$

Where Y is the response variable, β_0, β_i, β_{ii}, β_{ij} are regression coefficients of variables for intercept, linear, quadratic and interaction terms, respectively. Xi and Xj are independent variables.

Results were completely analyzed using analysis of variance (ANOVA) by Design-Expert software version 11 (Stat-Ease Inc., Minneapolis, U.S.A).

The determination coefficient, R^2, is the proportion of variation in the response attributed to the model rather than to random error. It has been suggested that a good-fitting model should have R^2 no less than 80%. When R^2 is close to unity, the empirical model is suitable for fitting the actual data. A lower value of R^2 indicates that the model is inappropriate for explaining the relation between variables (Krishnaiah, 2015).

Design-Expert was also used to fit equations developed and for assessment of models statistical significance.

Model significance is proved by model F, Lack of fit F and Pvalues. Indeed, if P-value is< 0.05, F-value> 3 and Lack of fit F value< 3, the model is statistically significant and good (Azmi *et al.*, 2015).If not, the model is considered not suitable.

Coefficients of the independent variables Xi, were identified as significant when their absolute value was greater than the standard error (SE) (Morineau and Chatelin, 2005):

2.7.2 Optimization by RSM method

Numerical and graphical methods of the Design Expert 11 software were used. For numerical method, a multi-objective optimization was performed using the constraints described in Table-2.

Table-2. Constraint of numerical optimization

Name	Goal	Lower Weight	Upper Weight	Importance (in stars)
T (°C)	isin range	1	0.1	***
Duration (h)	minimize	1	0.1	***
Tp (°C)	maximize	0.1	1	*****
σ (N)	maximize	0.1	1	***
WBC (g/g dm)	maximize	0.1	1	****
L* (-)	maximize	0.1	1	*****

For graphical method, the four responses surfaces (gelatinization temperature, gel hardness, flour WBC and gel lightness (L*)) were superposed on a same graphical to determine the optimal area.

Desirability function (D(x)) was estimated for optimization of multiple responses (Giri and Prasad, 2007; Myers *et al.*, 2009. Majdi *et al*, 2018) applying Eq. (3).

$$D(x) = (Y1 \times Y2 \times Y3 \cdots Yn)^{1/n} \qquad (3)$$

3. RESULTS AND DISCUSSIONS

Table-3 summarizes real and coded values of responses according to Full Factorial Design used to optimize ANN process for functionalize wheat flour. It can be observed that, according to experimental parameters used, gelatinization peak temperature (Tg) varies between 65.58 and 71.97 °C. The highest Tg values being observed for samples treated at highest ANN temperature (55°C). On the other hand, the high or low treatment times do not necessarily give high or low Tg. These observations suggest

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that ANN treatment temperatures certainly affect flours gelatinization behavior compared to treatment duration.

For gel hardness, values ranged between 15.49 and 22.54 N. Maximal hardness (22.54 N) was obtained with a treatment low temperature and duration. The low or high treatment temperatures do not necessarily give a high or low gel hardness (i.e. 55 ° C gave 19.11 N and 55 ° C gave 15.49 N). However, it was noted that shortest treatment time (24 h) resulted in hardest wheat flour gels (20.91 and 22.54 N).And highest treatment duration (48 h) would give mean gel hardness (17.41-17, 68-17.71).

Table-3. Full factorial design: Two factors, three levels, real and coded data, experimental values.

Run	Factors		Responses			
	T: Temp.(°C)	D: Time (h)	Temperature of gelatinization (Tp) (°C)	Gel Hardness (σ) (N)	Water Binding Capacity (WBC) (g H_2O/g DM)	Gel lightness (L*) (-)
1	50 (0)	36 (0)	69.42	15.78	7.36	60.21
2	55 (1)	48 (1)	71.97	17.68	6.83	60.87
3	55 (1)	24 (-1)	71.03	19.11	6.70	63.41
4	50 (0)	36 (0)	69.50	18.12	8.50	59.69
5	50 (0)	36 (0)	69.46	16.95	7.93	59.95
6	45 (-1)	24 (-1)	65.58	22.54	7.47	62.03
7	50 (0)	24 (-1)	68.55	20.91	8.47	61.01
8	55 (1)	36 (0)	71.93	15.49	7.44	62.93
9	45 (-1)	48 (1)	67.98	17.41	6.73	61.41
10	45 (-1)	36 (0)	66.54	18.25	8.65	62.08
11	50 (0)	48 (1)	69.90	17.71	6.92	59.24

This would presume a greater influence of treatment time than treatment temperature on gel hardness (firmness). Values of Water Binding Capacity (WBC) presented in table 3 are ranged between 6.70 and 8.65. The highest value of WBC was obtained for the treatment low temperature (level -1) and middle time (level 0). And we observed that for a same treatment temperature (i.e. 50 °C), we didn't have stables values of WBC (6.92; 7. 93; 8.50 g H2O/g DM). Moreover, treatment high time (48 h) gives low values of WBC (6.73; 6.83; 6.92 g H2O/g DM).

After experiments, gel lightness was ranged between 59.24 and 63.41. Compared to native gel of wheat flour with a lightness of 66.86 ±0.52, it can be concluded at a low color degradation of treated gel with ANN process. The highest value of lightness (63.41) was obtained for treatment low time (level -1) and high temperature (level +1). In the analysis of Table-3, temperature parameter of treatment could influence with a curvature effect the luminescence of gel treated by ANN process. Indeed, when the temperature varies from 45 to 55 ° C via 50 ° C, the average values of L* change from 61.84 ± 0.37 to 62.40 ± 1.35 via 59.63 ± 0.36, respectively.

Before analysis of the responses surfaces curves, a simultaneous influence of treatment temperature and time on the 4 responses related to the functionalization of the wheat flour is overall observed.

3.1 Model fitting and evaluation of responses parameters

In this chapter, for each response studied, focus will be on the analysis of model quality, model coefficients significance and the effect of factors on responses.

3.1.1 Temperature of gelatinization of ANN wheat flour

Fitting quadratic models to experimental data, the most significant model that better described the change of gelatinization temperature (Tp) can be exhibit as follows:

$$Tp = 69.43 + 2.47T + 0.78D - 0.37T*D - 0.15T^2 - 0.16D^2 \quad (4)$$

Table-4 presents the characteristics of Tp model quality and the significant of model coefficients.

 ISSN 1819-6608

ARPN Journal of Engineering and Applied Sciences

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Table-4. Coefficients, standard error, and ANOVA for gelatinization temperature model.

Tp: Temperature of gelatinization (°C)				
	SE of coeff.	F-value	P-value	R^2
Model	0.1024	205.79	< 0.0001***	0.99
T	0.0815	919.38	< 0.0001***	
D	0.0815	91.95	0.0002***	
T*D	0.0998	13.37	0.0147*	
T^2	0.1255	1.45	0.2817^{ns}	
D^2	0.1255	1.65	0.2548^{ns}	
Lack of fit		40.86	0.0240*	

Note: Temperature of treatment (T), Time of treatment (D)
*P <0.05; **P < 0.01; ***P < 0.001; ns: not significant.

According to Table-4, gelatinization temperature model (Tp) obtained is very significant (F-value = 205.79, high and P-value <0.001). This means that there is a strong correlation between Tp and both treatment temperature (T) andtime (D); that is confirmed by R^2 value (i.e. 0.99). Indeed, in the analysis of Table-4, treatment temperature and duration, very significantly influence Tp at a 99.9 % confidence level. Also, the effect of the interaction (T * D) influences gelatinization temperature at a 95% confidence level. However, (quadratic) curvature effects have no significant impact on Tp. The Lack of Fit F-value of 40.86 implies the Lack of Fit is significant. There is only a 2.40% chance that a Lack of Fit F-value this large could occur due to noise. Significant lack of fit is bad. The validity of the model is therefore to be questioned. However, the model can to fit.

The absolute value of each model coefficients is greater than its standard deviation (SE) (Morineau and Chatelin, 2005). This means that all the coefficients of the model will be retained for the final optimization.

The effects of parameters (T and D) of ANN treatment on gelatinization temperature (Tp) of the wheat flour are highlighted in Figure-1.

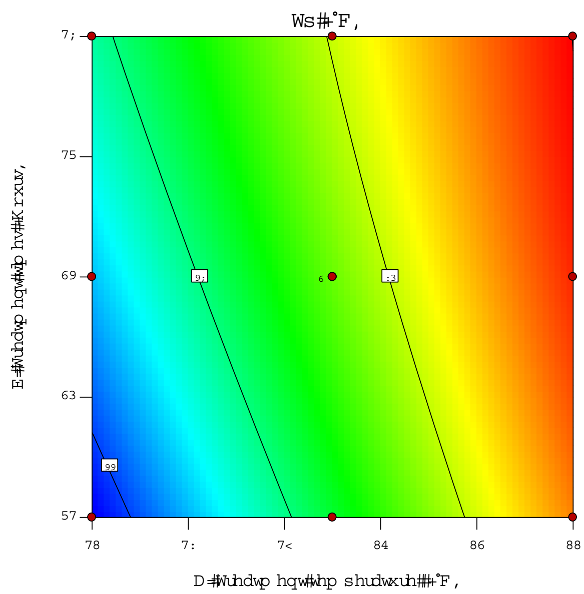

Figure-1. Estimated response surface for gelatinization temperature (Tp) of wheat flour.

According to Figure-1, Tp is linearly related to treatment temperature (p <0.05) and treatment duration (p <0.05). It is the same for interaction effect (T*D) between T and D. Only the curvature effects (T^2 and D^2) of T and D are insignificant (P> 0.05). Figure-1 of the response surfaces also indicate that the Tp increases with increasing temperature and processing time during the ANN process. Indeed, several publications (Jacobs and Delcour, 1998; Adebowale *et al*., 2005; Gomes *et al*., 2005; Waduge *et al*., 2006; Chung *et al*., 2009) report a modification of physicochemical properties of starch by ANN. This increase is attributed to the strengthening of the crystallinity of the starch granules. ANN process induces a rearrangement of amylose chains in amorphous regions (Tester *et al*., 1999) and an increase in amylose-amylose and amylose-amylopectin interactions, which increases the crystallinity of certain granules (Hoover and Vasanthan,

 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences
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1994; Jacobs *et al*., 1998). During annealing process, the highest Tp(71.97 °C) was obtained with a treatment temperature of 55 °C and a treatment time of 48 h (Figure-1). The gelatinization temperature is related to the firmness and swelling capacity of the wheat flour. Indeed, following an ANN treatment, Jacobs, Eerlingen and Delcour (1996) observed an increase in the peak viscosity of rice starch and an increase in the final viscosity of wheat starch.

3.1.2 Hardness of ANN gel of wheat flour

The gel firmness model obtained from experimental data is presented in equation 4.

$$\text{Hardness } (\sigma) = 16.96 - 0.99T - 1.63D + 0.93T{*}D - 0.10T^2 + 2.34D^2 \quad (5)$$

The characteristics of this model quality and the significance of the model coefficients are presented and Table-5.

Table-5. Coefficients, standard error, and ANOVA for hardness model (σ).

σ: Hardness of gel (N)				
	SE of coeff.	**F-value**	**P-value**	**R²**
Model	0.4107	12.39	0.0076**	0.93
T	0.3269	9.11	0.0295*	
D	0.3269	24.77	0.0042**	
T*D	0.4003	5.34	0.0689^{ns}	
T^2	0.5030	0.0425	0.8448^{ns}	
D^2	0.5030	21.57	0.0056**	
Lack of fit		0.1138	0.9443^{ns}	

Note: Temperature of treatment (T), Time of treatment (D)
*P <0.05; **P < 0.01; ***P < 0.001; ns: not significant.

In analysis of Table-5, the model high F-value (12.39), indicates a significant ($P < 0.01$) dependence at 99%, between gel firmness and independent factors (T and D). The R^2 of 0.93confirms it. Then the Lack of Fit F-value is not significant relative to the pure error, because of is low value. There is a 94.43% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good.

When analyzing the coefficients of gel hardness standard error, it seems that, except T^2 coefficient, the others ones can be retained in the hardness (σ) model. Because the absolute value of these coefficients is greater than their standard error (Morineau and Chatelin, 2005).

Figure-2 shows the effect of annealing process temperature and time on wheat flour gel hardness.

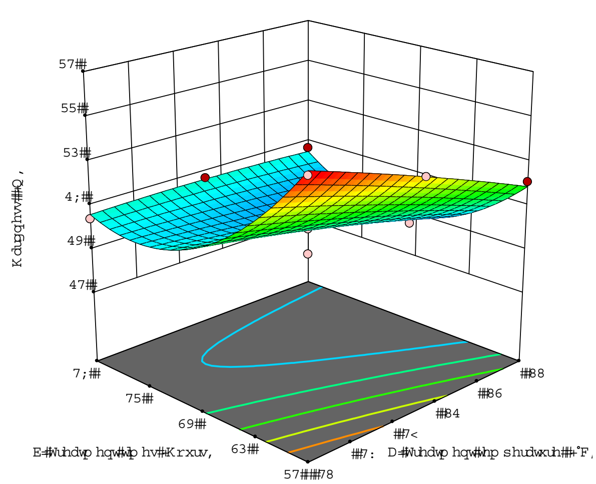

Figure-2. Estimated contour of response surface in 3D for gel hardness of wheat flour.

Figure-2 reveals that treatment temperature (T) and duration (D) then the curvature effect (quadratic) of time (D^2) influence significantly ($P < 0.05$) gel hardness (Figure-3). This time curvature effect means that gel hardness decreasing at the beginning of the treatment reaches a minimum (after 40 hours) and then increases with time towards the treatment end (48h). As for the effect of interaction (T* D) and temperature curvature (T^2), they are insignificant on gel hardness. The results also indicate that gel hardness decreases with increasing treatment temperature and time during annealing process. Indeed, Gomes *et al.,* (2005) observed a decrease in the retro gradation of cassava starch during ANN process. Tsutsui *et al.*, (2013) also observed that, ANN process tends to reduce the rheofludiant behavior and parameters (G'and G") of dynamic viscoelasticity of starch (rice). During runs, highest gel hardness was achieved with annealing temperature (level 0) of 45 ° C and annealing time (level -1) of 24 min. This result is inconsistent with Cham and Suwannaporn (2010) observations as well as those of Hormdok and Noomhorm (2007), who observed an increase in the hardness and cohesion of rice starch gels after ANN. Thus, the hardness decrease of ANN-flours would therefore be a result of the increase in their swelling capacity.

3.1.3 Water Binding Capacity of ANN wheat flour

The model WBC of wheat flour has been given by equation 6.

VOL. 14, NO. 19, OCTOBER 2019 ISSN 1819-6608
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$$WBC = 8.11 - 0.31T - 0.36D + 0.22T*D - 0.35T^2 - 0.70D^2 \quad (6)$$

The characteristics of this model quality and the significance of the model coefficients are presented and Table-6.

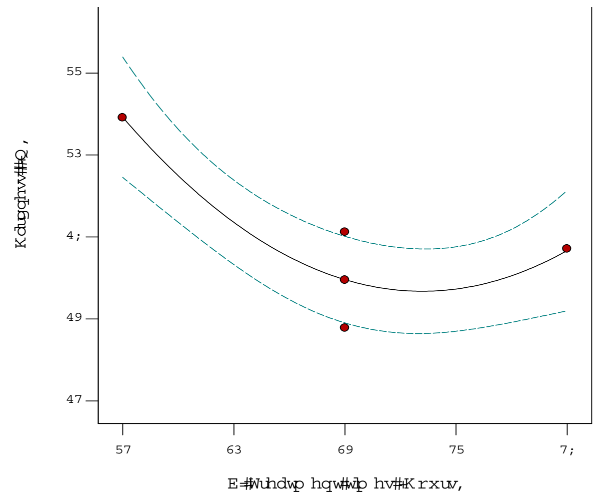

Figure-3. Curvature effect of ANN processing time on gel hardness.

Table-6. Coefficients, standard error, and ANOVA for water binding capacity (WBC).

WBC: water binding capacity (g H_2O/ g DM)				
	SE of coeff.	**F-value**	**P-value**	**R^2**
Model	0.3169	1.87	0.2551^{ns}	0.65
T	0.2522	1.54	0.2692^{ns}	
D	0.2522	2.04	0.2128^{ns}	
T*D	0.3089	0.4959	0.5128^{ns}	
T^2	0.3881	0.7986	0.4124^{ns}	
D^2	0.3881	3.22	0.1325^{ns}	
Lack of fit		1.29	0.4645^{ns}	

Note: Temperature of treatment (T), Time of treatment (D)
*P <0.05; **P < 0.01; ***P < 0.001; ns: not significant.

Table-6 indicates that WBC model is not significant, with its F-value (1.87) and R square (0.65) very smalls. However, the lack of fit is good (because not significant); meaning that experimental error and the model calculated error are very close. By comparing the absolute value of the coefficients estimated to their standard error (Morineau and Chatelin, 2005); only the coefficients relating to interaction effect (T * D) and curvature effect (T^2) will not be retained for WBC model.

For the effect of parameters T and D on the WBC of wheat flour, Figure-4 highlights it very well. In the Table-3, all parameters (T, D, TD, T^2 and D^2) P-value are not significant (P> 0.05), meaning that their effects are insignificant on wheat flour WBC. However, the WBC of wheat flour decreases lowly with increasing temperature and duration of ANN treatment (Figure-5). These results are consistent with the observations of Waduge *et al*. (2006), Jacobs *et al*., (1998) and Hoover and Vasanthan, (1994); who reporting a decrease in the swelling capacity of starches after ANN at 50 ° C (0.5 - 72 hours). Indeed, ANN is governed by the glass transition phenomenon whose limits are not very precise. Thus, at high temperatures, two contradictory phenomena could be in competition. On the one hand, structuring phenomena that improve the structural stability of starches and therefore limit their swelling capacity: it's the case here. On the other hand, the partial melting of crystallites, on the contrary, would be accompanied by an increase in the swelling capacity of granules. It is the result of these two phenomena that conditions the overall behavior of flour after ANN processing(Wang et *al.,* 2018).

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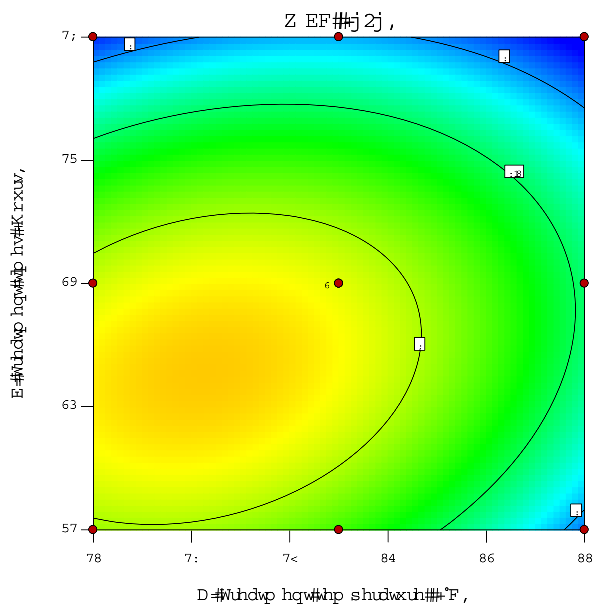

Figure-4. Estimated response surface for water binding capacity (WBC) of wheat flour.

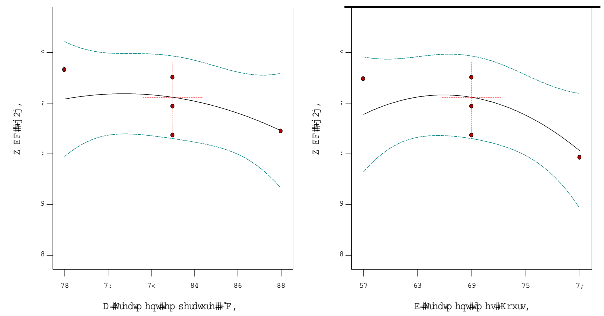

Figure-5. Weak effect of temperature and ANN process time on Water Binding Capacity of wheat flour.

3.1.4 Lightness of ANN wheat flour gel (L*)

Fitting quadratic models from experimental data, the most significant model that better described the gel color change can be exhibited as follows:

$$L^* = 60.11 + 0.28T - 0.82D - 0.48T^*D + 2.16T^2 - 0.22D^2 \quad (7)$$

Lightness model characteristics of ANN flour is highlighted in Table-7.

Table-7. Coefficients, standard error, and ANOVA for lightness of gel (L*).

L*: Lightness of gel (-)				
	SE of coeff.	**F-value**	**P-value**	**R^2**
Model	0.1723	31.23	0.0009***	0.97
T	0.1371	4.22	0.0952	
D	0.1371	35.90	0.0019**	
T*D	0.1679	8.17	0.0355*	
T^2	0.2110	104.78	0.0002***	
D^2	0.2110	1.08	0.3455^{ns}	
Lack of fit		2.12	0.3370^{ns}	

Note: Temperature of treatment (T), Time of treatment (D)
*P <0.05; **P < 0.01; ***P < 0.001; ns: not significant.

In Table-7, the Model F-value (i.e. 31.23) implies that the model is significant. There is only a 0.09% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case D, T*D, T^2 are significant model terms. The Lack of Fit F-value of 2.12 implies the Lack of Fit is not significant relative to the pure error. There is a 33.70% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good; thus, model can be used for predicting the gel lightness.

The absolute value of all coefficients is greater than their standard error (SE), meaning that the model will remain in state without being modified.

For the effect of T et D on gel lightness, the results are presented in Figure-6.

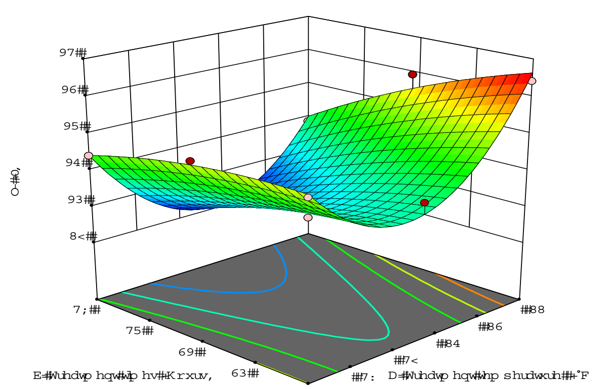

Figure-6. Estimated contour of response surface in 3D for gel color lightness of wheat flour.

VOL. 14, NO. 19, OCTOBER 2019 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences
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In the analysis of Figure-6, the curvature effect of temperature is more significant (P <0.001) than the linear one (P <0.1). Indeed, the gel lightness decreases with increasing temperature to a minimum value (L * = 60 at 50 °C). Above 50°C, this lightness increases to reach a maximum of 63.41 at 55 ° C. This loss of lightness at the treatment beginning (between 45 and 50 ° C) is due to Maillard reactions governed by Arrhenius law. This law stipulates that the lower the temperature, the less quickly the reaction evolves (Serpen *et al.*, 2007). The treatment taking place at low temperatures (40 to 50° C). Thereby, formation of colored compounds and color changes of product are less pronounced. Thus, this results in a low decrease of product lightness (L*).

In conclusion, ANN process has made little change in flour gels color, probably because of the low temperatures applied during these treatments.

3.2 Optimization of annealing process conditions

The annealing (ANN) conditions of wheat flour for its functionalization would be considered optimal when gelatinization temperature, gel hardness, water retention capacity and gel lightness reached maximum values. Thus, temperature and time of the optimal annealing process were obtained by overlaying plots of responses contour curves. Figure-7 shows the optimal conditions area of ANN to produce wheat flour with good functional property.

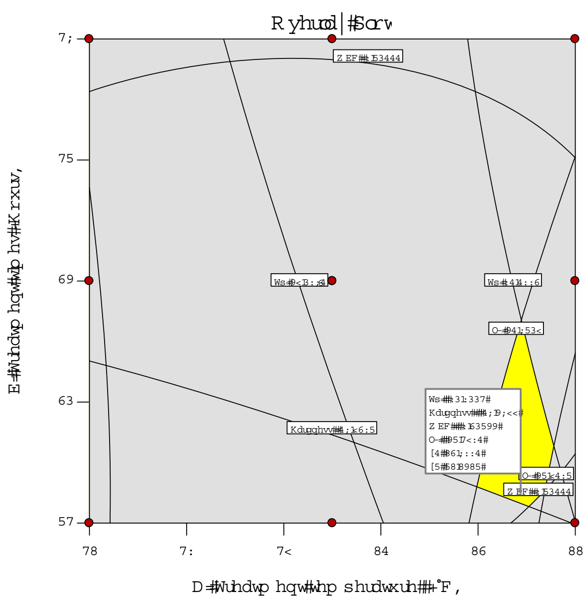

Figure-7. Overlay plot of contours of response surfaces of gelatinization temperature, gel hardness, water binding capacity and lightness when annealing wheat flour.

The yellow area within the overlay plot highlighted the most advantageous zone for a given set of variables. The results of Figure-7 show that optimal annealing temperature and time for developing wheat flour with good functional properties are 53.88 ° C and 25.57 minutes. These annealing process conditions (54 ° C / 25h34 min) were used to produce functional wheat flour. Under these conditions, the gelatinization temperature, gel hardness, water binding capacity, and gel lightness predicted by model were 70.70 ° C, 18.69 N, 7.30 g H_2O/g DM and 62.50 respectively for a calculated desirability of 0.953.

3.3 Validation of the optimum

Optimal parameters resulting from RSM were validated by carrying out three tests. Validation results are shown in Figure-8.

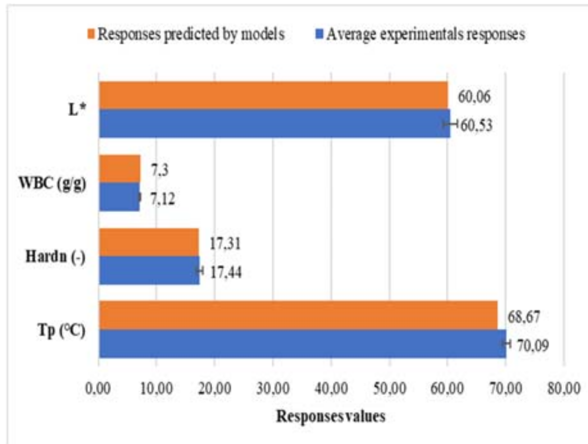

Figure-8. Validation by comparison of experimental with predicted values of RSM.

Figure-8 shows that application of ANN optimal parameters (final water content of the flour (70%), treatment temperature (54 ° C) and treatment time (25h34 min)) gives flours with functional properties close to those predicted by models with a determination coefficient $R^2$of 99.9 %.

4. CONCLUSIONS

The aim of this study was to optimize an hydrothermal process named annealing (ANN), for producing a wheat flour with desired functionalities. A full factorial design applied with response surface method was used for optimization. ANN process experiments have been conducted with different operating conditions: moisture content fixed at 70% (wb), T (45-55°C) and D (24-48 hours). The conventional graphical based on RSM and desirability function have been effective at determining the optimal zone within the experimental region. For each quadratic model, it has been found that conditions of ANN process (treatment temperature and duration) influenced them significantly and simultaneously. Except for WBC response where conditions were insignificant. The optimal conditions found allow having stable wheat flour with good functional characteristics for infant feeding. A hedonic evaluation through sensory consumer tests are necessary steps for a large-scale consumer endorsement of this new product.

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ACKNOWLEDGMENTS

The authors forward their deepest gratitude to the Department of Formation and research Chemical and Food Engineering, in INP-HB, Yamoussoukro (RCI), and everybody who supported them in all aspects of their research work.

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