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Analytical Methods

Use of response surface methodology to investigate the effects of milling conditions on damaged starch, dough stickiness and chapatti quality

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ABSTRACT

Response surface methodology was used to investigate the influence of three factors namely mill aperture, feed rate and moisture content of wheat grain on the damaged starch content of whole wheat flour from Lokwan wheat cultivar, dough stickiness and chapatti (Indian unleavened flat bread) quality. Each predictor variable was tested at three levels. Aperture was varied as 2, 3 and 4 mm in a stone mill, feed rate as 0.21, 0.63 and 1.05 min for 200 g wheat grains and grain moisture content as 8.6, 14.3 and 20% w/w. Flours containing varying amounts of damaged starch ranging from 6.1% to 26.90% were obtained and these were evaluated for dough stickiness and chapatti quality. Dough stickiness was increased with decreased aperture. With decreased aperture and increased grain moisture content softness of the chapatti was improved.

1. Introduction

Almost 90% of the wheat produced in India is consumed in the form of chapatti and only 10% is consumed in the form of bread, biscuits and cakes. Chapatti is unleavened flat bread produced traditionally from plain whole-wheat flour dough generally baked fresh twice a day for lunch and dinner, and unless eaten immediately after baking, these stale rapidly and become difficult to chew (Ghodke & Ananthanarayan, 2007). Chapattis are an economical source of protein, and contribute to satiety through abundant dietary fiber, that reduces constipation and diverticular disease, rates of chronic bowel disease and diet-related cancers (Inglett, Carriere, & Maneepun, 2005). Ideally the chapatti is creamy in color, cooked with a minimum of small brown spots and fully puffed so that two distinct layers are present. The chapatti should be easily torn and pliable so that it can be folded by the thumb and forefinger to produce a small scoop for holding vegetables or curried preparations. A wheatish aroma and taste is desirable with a non sticky, soft chewing feel in the mouth.

Damaged starch refers to small particles of starch broken away from the main starch granules in wheat during milling. These smaller particles hydrate more easily during dough preparation. The level of starch damage therefore affects the water absorption and dough mixing properties of flour and is of technological significance. Damaged starch has much greater water retention capacity; however, too much starch damage leads to sticky dough, strong proofing, and undesirable red crust colour (Bettge, Morris, & Greenblatt, 1995). Two factors which lead to damaged starch are: the surface factor corresponding to the scratching effect by the surface of grooved mill, the internal factor appearing during the reduction phase when granules are broken or flattened.

Despite numerous studies about the role of damaged starch on dough properties and baking quality of bread, scarce information has been reported describing their effect on the chapatti quality. Haridas Rao, Leelavathi, and Shurpalekar (1986) reported that the quality characteristics of chapatti are mainly governed by the quality of wheat used and the processing conditions employed for converting it into flour. Some of the factors reported to influence the chapatti quality are damaged starch content, water absorption of the flour. Prabhasankar, Manohar, and Gowda (2002) studied physicochemical characterization of nine wheat cultivars and their correlation to chapatti making quality. From their study they concluded that dough water absorption and damaged starch correlated most significantly and flour with highest damaged starch produced softest chapatti. Prabhasankar and Haridas Rao (2001) studied effect of different milling methods on chemical composition of whole wheat flour. In this study, whole wheat flour obtained from stone mill with highest damaged starch content yielded chapatti with highest sensory score.

RSM can be used to evaluate the relative significance of several affecting factors even in presence of complex interaction. RSM is an empirical statistical modeling technique employed for multiple regression analysis using quantitative data obtained from properly designed experiments to solve multivariable equations simultaneously (Mason, Gunst, & Hess, 1989; Montgomery, Runger,

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& Hubele, 2001; Rao, Chul-Ho, & Rhee, 2000). In several food research studies, RSM is almost routinely used to optimize the efficiency of the ingredients such as fibers, improvers (Collar, Andreu, Martínez, & Armero, 1999; Collar, Santos, & Rosell, 2007) composite flours (Shittu, Raji, & Sanni, 2007), and optimization of various food processes such as extractions, product development, functional food preparation etc (Flander, Salmenkallio-Marttila, Suortti, & Autio, 2007; Katina, Heiniö, Autio, & Poutanen, 2006).

Present study was undertaken with the objective of using RSM to investigate the influence of milling conditions on the formation of damaged starch in whole wheat flour and also to study the effect of milling conditions on dough stickiness and chapatti tear force prepared from these flours.

2. Materials and methods

2.1. Materials

Lokwan wheat cultivar was procured from local market, Mumbai, India. Double filtered groundnut oil (Dhara), and table salt (Tata salt) were procured from the local market. SPEZYME®FRED (*Thermostable α-amylase*) 40,000 SKBU, per ml (one SKBU unit is the activity which dextrinizes 1 g of limit dextrin substrate per hour), was gifted by ©Genencor International, Inc., USA. All the other chemicals used for the analysis were of analytical grade.

2.2. Methods

2.2.1. Design of experiment for milling of whole wheat grains

An RSM study as described by Box and Wilson (1951) was conducted to determine the relative contributions of three predictor variables (aperture, feed rate, moisture content) to the damaged starch content, dough stickiness and chapatti tear force and extensibility. A central composite design was constructed using the software Design Expert Version 6.0.10 (Stat-Ease Corporation, Minneapolis, MN, USA) and was used to analyze the results. Maximum and minimum predictor variable levels were chosen by carrying out preliminary trials of milling. Three levels of each predictor variable were incorporated into the design. To study three factors (predictor variables) at five levels would require 5^3 or 125 experiments, whereas use of the central composite design required 20 runs or experiments. Response variables measuring flour quality were damaged starch, dough stickiness and chapatti (Table 1).

2.2.2. Wheat grain milling

Initially 5 kg of grain sample was cleaned and milled at 20 different milling conditions to obtain flours with different percentage of damaged starch content. Aperture (mm) was varied as 2, 3 and 4 mm in a stone mill by varying distance between two stones, feed rate was changed as 0.21, 0.63 and 1.05 min per 200 g grains by adjusting the inclination of the feed chute and grain moisture content was varied by adding water externally to adjust moisture at 8.6%, 14.3% and 20%. Milled flour samples were cooled immediately and stored in air tight plastic containers at 4 °C.

2.2.3. Determination of damaged starch content of whole wheat flour

Damaged starch was estimated by the AACC method 76-30A (AACC, 2000). The milled whole wheat flours of various trials were taken and evaluated for its damaged starch content. Three determinations per flour lot were made and results were expressed as mean ± standard deviation (SD).

2.2.4. Dough preparation and evaluation of dough stickiness

For this study flours of various trials were taken and thoroughly mixed with addition of required amount of water to form into dough suitable for making into chapatti. The prepared dough was evaluated using The Stable Micro Systems Chen–Hoseney Dough Stickiness Rig test, using accessories such as 25 mm perspex cylinder probe (P/25P), 50 kg load cell and SMS/Chen–Hoseney Dough Stickiness Cell (A/DSC) (Hoseney & Smewig, 1999).

Table 1
Experimental design (actual and coded values) and responses for damaged starch and dough stickiness obtained by milling trials

Aperture (mm)	Feed rate[b] (min^{-1})	Moisture (%)	Damaged starch[a] (%)	Dough stickiness[a] (g)	Tear force[a] (g) 0 day	Tear force[a] (g) 2 day
3 (0)	0.63 (0)	20 (1)	21.76	40.71	242.07	428.79
3 (0)	0.63 (0)	8.6 (−1)	13.25	30.45	398.63	654.90
2 (−1)	1.05 (1)	20 (1)	26.9	46.04	192.32	309.92
3 (0)	0.63 (0)	14.3 (0)	14.46	41.72	354.71	541.45
3 (0)	0.63 (0)	14.3 (0)	14.62	41.08	349.55	465.68
4 (1)	0.63 (0)	14.3 (0)	6.14	35.89	715.94	989.95
4 (1)	1.05 (1)	20 (1)	10.2	32.75	441.07	830.06
3 (0)	0.63 (0)	14.3 (0)	14.6	40.87	353.21	531.34
2 (−1)	0.63 (0)	14.3 (0)	23.64	42.76	240.55	347.52
3 (0)	0.63 (0)	14.3 (0)	14.44	41.89	356.69	599.77
2 (−1)	0.21 (−1)	20 (1)	20.91	35.74	270.46	451.76
3 (0)	0.63 (0)	14.3 (0)	14.62	39.92	300.57	467.13
4 (1)	0.21 (−1)	8.6 (−1)	6.1	28.89	901.86	1218.63
2 (−1)	1.05 (1)	8.6 (−1)	21.27	38.05	265.67	446.04
3 (0)	0.63 (0)	14.3 (0)	14.36	41.18	385.53	633.91
3 (0)	1.05 (1)	14.3 (0)	15.97	30.98	287.03	464.51
2 (−1)	0.21 (−1)	8.6 (−1)	16.15	35.61	275.20	458.16
4 (1)	1.05 (1)	8.6 (−1)	6.97	27.14	708.5	867.71
3 (0)	0.21 (−1)	14.3 (0)	11.6	34.75	425.72	735.31
4 (1)	0.21 (−1)	20 (1)	8.01	29.62	484.26	839.87

[a] The damaged starch, dough stickiness and tear force is taken as response. The experiments were carried out in triplicate.
[b] Feed rate 0.21, 0.63, 1.05 min/200 g grains.

2.2.5. Response surface methodology (RSM)

For each of the response variables, model summaries and lack of fit tests were analyzed for linear or quadratic models. The cubic model was aliased because there were not enough points for this type of model. From this information, the most accurate model was chosen via the sequential *F*-tests, lack-of-fit tests and other adequacy measures. Three-dimensional response surface plots were generated for each quality parameter. Calculation of optimal processing conditions for optimum damaged starch content of whole wheat flour was performed using a multiple response method called desirability (Box & Wilson, 1951; Myers and Montgomery, 1995). This optimization method incorporates desires and priorities for each of the variables. In this study, predictor variables were permitted to be at any level within the range of the design. Statistical experimental design was used to optimize the milling conditions to obtain flour of various damaged starch content, which was checked with respect to effect on dough stickiness and chapatti making quality.

For statistical calculations, the relationship between the coded values and actual values are described by the following equation:

$$X_i = \frac{A_i - A_0}{\Delta A}$$

where X_i is coded value of variable; A_i the actual value of variable; A_0 the actual value of the A_i at the center point; and ΔA the step change of variable. The design matrix is shown in Table 1. Damaged starch, dough stickiness and chapatti tear force were taken as response variables Experiments were carried out in triplicate.

Quadratic equation for the variable was as follows:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i X_j + \sum_i \sum_j \beta_{ij} X_i X_j$$

Y is the predicted response; β_0 a constant; β_i the linear coefficient; β_{ii} the squared coefficient; and β_{ij} the cross-product coefficient.

The above quadratic equation was used to build surfaces for the variables. The software Design Expert Version 6.0.10 was used to analyze the results. By keeping one variable at its optimum level, three-dimensional plots of two factors versus damaged starch, dough stickiness and tear force were drawn, and corresponding contour plots were obtained.

2.2.6. Preparation of chapatti

In the present study chapattis were prepared according to the method described by Ghodke and Ananthanarayan (2007). These prepared chapattis were packed in self-sealable low density polyethylene plastic bags and stored for 2 days at 30 ± 1 °C and 70% RH.

2.2.7. Evaluation of chapatti for tear force and extensibility

Fresh as well as stored chapattis were evaluated for tear force as described by Ghodke and Ananthanarayan (2007). Three samples from each batch of RSM design were analyzed and averaged.

3. Results and discussion

3.1. Effect of milling conditions on formation of damaged starch and effect on dough stickiness and chapatti tear force

Table 1 shows the levels of selected predictor variables for the central composite design and summarizes the experimental design (actual and coded values) for optimization of milling conditions. To examine the combined effect of three different components (independent variables) on damaged starch, dough stickiness and chapatti quality, a central composite factorial design of 1–14 experiments were performed at different combinations, and those from 15 to 20 were under same conditions. Table 1 also includes data showing effect of milling condition on damaged starch (DS) and dough stickiness and chapatti tear force both fresh as well as stored for 2 day as a response. The application of RSM yielded following regression equation, which is empirical relation between damaged starch (X), dough stickiness (Y) and chapatti tear force 0 day (Z_1), chapatti tear force 2 day (Z_2), and the test variable (A – aperture, B – feed rate, C –moisture) in coded units.

For damaged starch:

$$X = +14.89 - 7.14A + 1.85B + 2.40C - 0.57A^2 - 1.67B^2 + 2.05C^2 - 1.01AB - 0.66AC + 0.27BC$$

For dough stickiness:

$$Y = +39.85 - 4.39A + 1.03B + 2.47C + 1.37A^2 - 5.09B^2 - 2.37C^2 - 1.42AB - 0.22AC + 1.59BC$$

For tear force (0 day):

$$Z_1 = +351.86 + 200.74A - 46.29B - 91.97C + 123.67A^2 + 1.80B^2 - 34.23C^2 - 8.61AB\,5.87AC + 10.19BC$$

For tear force (2 day):

$$Z_2 = +547.58 + 273.28A - 78.55B - 78.50C + 109.60A^2 + 40.77B^2 - 17.29C^2 - 25.85AB - 34.24AC + 26.42BC$$

Table 2
ANOVA for damaged starch, dough stickiness and chapatti tear force by response surface quadratic model[a]

Source	Damaged starch				Dough stickiness				Tear force (0 day)				Tear force (2 day)			
	SS[b]	DF	*F-value*	Prob > *F*	SS[b]	DF	*F-value*	Prob > *F*	SS[b]	DF	*F-value*	Prob > *F*	SS[b]	DF	*F-value*	Prob > *F*
Model	629.55	9	38.27	<0.0001	479.56	9	6.86	0.0029	6.165E+0.005	9	45.53	<0.0001	9.715E+005	9	15.63	<0.0001
A	510.51	1	279.3	<0.0001	192.81	1	24.81	0.0006	4.030E+00	1	267.85	<0.0001	7.468E+005	1	108.17	<0.0001
B	34.37	1	18.81	0.0015	10.71	1	1.38	0.2676	21428.57	1	14.24	0.0036	61699.45	1	8.94	0.0136
C	57.79	1	57.79	0.0002	61.11	1	7.86	0.0187	84581.13	1	56.22	<0.0001	61628.78	1	8.93	0.0136
A^2	0.88	1	0.88	0.5034	5.17	1	0.67	0.4336	42058.3	1	27.96	0.0004	33031.25	1	4.73	0.0536
B2	7.68	1	7.68	0.0676	71.21	1	9.16	0.0127	8.90	1	5.913E-003	0.9402	4571.34	1	0.66	0.4348
C^2	11.55	1	11.55	0.0307	15.49	1	1.99	0.1883	3221.47	1	2.14	0.1741	822.44	1	0.12	0.7371
AB	8.10	1	8.10	0.0615	16.13	1	2.08	0.1802	2770.66	1	1.84	0.2046	5344.23	1	0.77	0.3996
AC	3.45	1	3.45	0.1998	0.4	1	0.051	0.8259	46047.0	1	30.61	0.0003	9376.97	1	1.36	0.2709
BC	0.60	1	0.60	0.5795	20.29	1	2.61	0.1372	831.50	1	0.55	0.4743	5585.72	1	0.81	0.3896
Residual	18.28	10			77.71	10			15044.7	10			69044.7	10		
Lack-of- fit	18.22	5	3.64	<0.0001	75.25	5		0.0009	11261.6	5	2.98	0.1282	45742.52	5	1.96	0.2385
R^2	0.9718				0.8606				0.9762				0.9336			

[a] All the values are mean ± SD of three values.
[b] SS – sum of square.

The results of the second order response surface model fitting in the form of ANOVA for damaged starch, dough stickiness and tear force is given in Table 2. The ANOVA of quadratic regression model demonstrates that the model is significant, as is evident from Fisher's *F*-test value being 38.27, 6.86, 45.53 and 15.63 for damaged starch, dough stickiness and tear force 0 and 2 day, respectively, with a very low probability value for damaged starch [(P model > *F*) = 0.0001] and for dough stickiness [(P model > *F*) = 0.0029], tear force 0 day [(P model > *F*) = 0.0001] and tear force 2 day [(P model > *F*) = 0.0001]. The goodness of the fit of the model was checked by regression coefficient (R^2). In this case, the value of regression coefficient for damaged starch was $R^2 = 0.9718$, for dough stickiness it was $R^2 = 0.8606$, for chapatti tear force 0 day $R^2 = 0.9762$ and tear force 2 day $R^2 = 0.9336$.

The *P*-values were used as a tool to check the significance of each of the coefficients, which, in turn, are necessary to understand the pattern of mutual interactions between the test variables. The smaller the magnitude of the *P*, the more significant is the corresponding coefficient. Values of *P* less than 0.05 indicate model terms are significant. The coefficient estimates and the corresponding *P*-values suggest that, among the test variables used in the study, A (aperture), B (feed rate), C (moisture content) are significant model terms. Aperture ($P < 0.0001$) has the largest effect on damaged starch formation followed by moisture content ($P < 0.0002$), feed rate ($P < 0.0015$). The mutual interaction between aperture and feed rate ($P < 0.0615$), aperture and moisture content ($P < 0.1998$) and feed rate and moisture content were found to be insignificant. The fit of the model was also expressed by the coefficient of determination R^2, which was found to be 0.9718, indicating that 97.180% of the variability in the response could be explained by the model. The R^2 value was always between 0 and 1 and the closer the R^2 was to 1.0, the stronger the model and the better it predicted the response (Haaland, 1989).

For the dough stickiness response, aperture ($P < 0.0001$) has the largest effect on dough stickiness followed by moisture content ($P < 0.0187$). Feed rate and other mutual interaction between all variables were found to be insignificant. For chapatti tear force (0 day) response, aperture ($P < 0.0001$), moisture ($P < 0.0001$) followed feed rate ($P < 0.0036$) and on 2nd day of storage aperture ($P < 0.0001$) feed rate and moisture content ($P < 0.0136$) were found to be significant.

The 3D response plots are generally the graphical representations of the regression equation from which the values of damaged starch, dough stickiness and chapatti tear force for different settings of variables, respectively, can be predicted. Each contour curve represents an infinite number of combinations of two least variables with the other maintained at zero level. The maximum predicted value is indicated by the surface confined in smallest ellipse in the contour diagram.

Fig. 1 shows the interaction between feed rate and aperture. From the plot it can be seen that when feed rate (min/200 g grains) was increased from 1.05 min/200 g grains to 0.21 min/200 g grain, damaged starch content decreased, however, at minimum feed rate (1.05 min/200 g grain) whole wheat flour of 22.60% damaged starch was obtained. This response graph showed increase in feed rate and aperture (distance between two stones) led to decrease in damaged starch content and vice versa. Tightening the rolls, increasing the rate of feed, lead to an increase in the level of starch damage (Williams & Fegol, 1969). During milling some starch granules are mechanically damaged, hence the level of starch damage varies with the severity of grinding (Hoseney, 1994). Fig. 2 shows the "mound shaped" surface at the center. The contour line indicated that dough stickiness was dependent on moisture content and feed rate. Highest dough stickiness values occurred at the slowest feed rate (1.05 min/200 g grains). Fig. 3 shows the interaction between moisture content and aperture on chapatti tear force

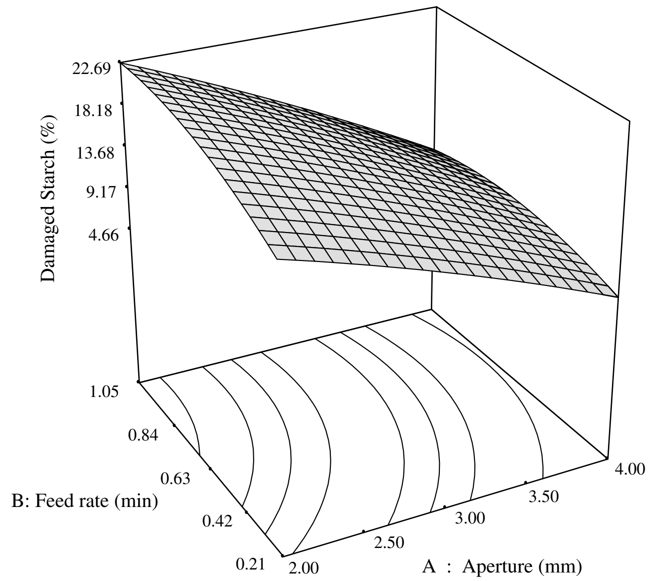

Fig. 1. Response surface plot of damaged starch; the effect of feed rate and aperture on damaged starch. Other variables are held at zero level.

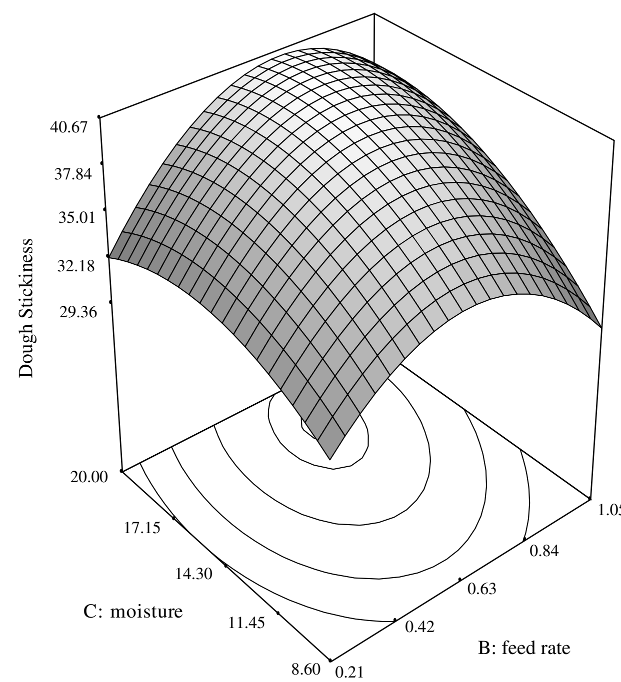

Fig. 2. Response surface plot of dough stickiness; the effect of moisture and feed rate on dough stickiness. Other variables are held at zero level.

on 0 day. When moisture content increased from 8.6% to 20% there was decrease in tear force indicating increased softness in chapatti. At lowest aperture (2 mm) and highest moisture (20%) content lowest tear force (192.32) was observed. This was due to lower aperture caused more damaged flour starch and hence increased the water absorption and yielded softest chapatti. Similar findings were observed byPrabhasankar et al. (2002) and Prabhasankar and Haridas Rao (2001). Fig. 4 shows the response and contour plot for effect of moisture and aperture on chapatti tear force stored for 2 days. Tear force at (2/0.21/20) RSM setting was 270.46 g on 0 day whereas it increased to 451.76 g after two days of storage. Chapatti

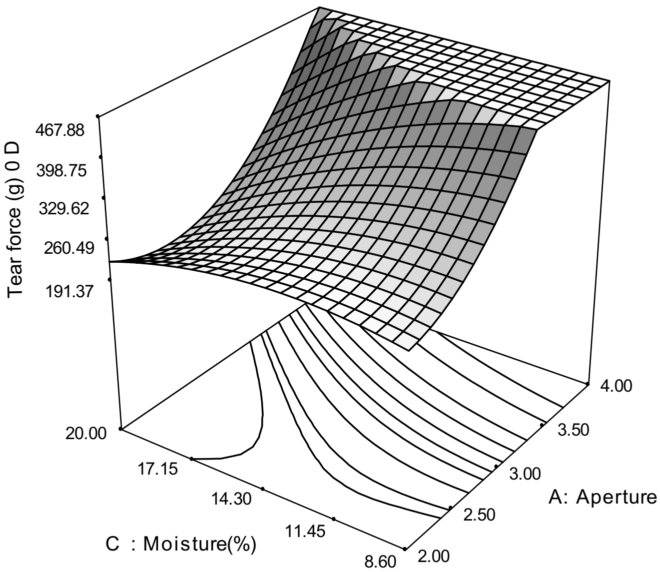

Fig. 3. Response surface plot of tear force (0 day); the effect of moisture and aperture on chapatti tear force. Other variables are held at zero level.

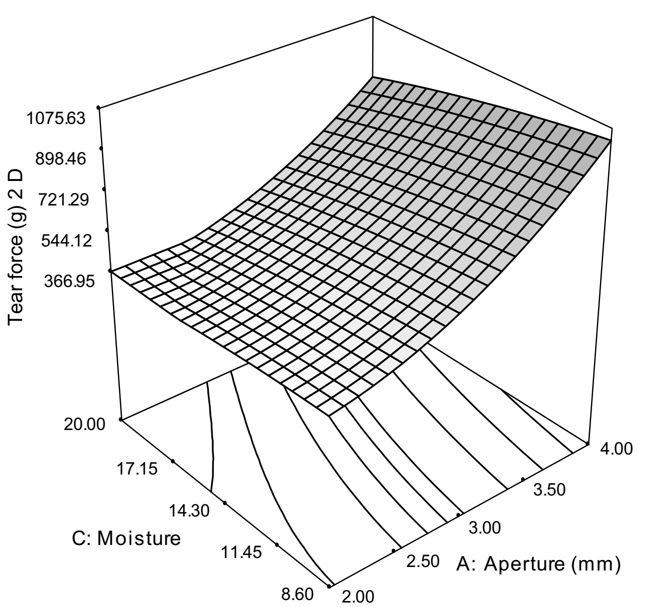

Fig. 4. Response surface plot of tear force (2 day); the effect of moisture and aperture on chapatti tear force. Other variables are held at zero level.

prepared from the highest aperture (4 mm) with high feed rate (0.21 min) and low grain moisture (8.6%) resulted into higher tear force (901.86 g), which increased to 1218.63 g after 2 days of storage. The tear force increase during storage could be partly attributed to recrystallization and retrogradation of starch in the chapatti, a very common phenomenon that occurs in bread (Guy, Hodge, & Robb, 1983). Formation of damaged starch in the flours during milling affects the staling in bread (Barrera, Pérez, Ribotta, & Léon, 2007). Léon, Barrera, Ribotta, and Rosell (2006) studied the effect of damaged starch levels on thermal behaviour of starch and bread staling. They observed an increase in enthalpy with increased damaged starch content. In addition, the retrogradation enthalpy increased with the amount of damaged starch at 1 and 3 days of storage time, although differences were dependent on the flour type.

Further, the model was validated by carrying out experiments under conditions predicted by it. The experimental values were found to be very close to the predicted values, and hence, the model was successfully validated.

4. Conclusions

From present study it was observed that, using RSM, flours of different damaged starch content ranging between 6.10% to 26.90% could be obtained by changing moisture, aperture and feed rate during wheat milling. Aperture, moisture followed by feed rate significantly affected damaged starch content whereas dough stickiness and chapatti tear force was significantly affected by mill aperture and grain moisture content. This study also confirmed the influence of damaged starch on chapatti dough and chapatti making quality. Damaged starch improved the chapatti quality; therefore, attention should be paid to content of damaged starch in flours since it affects the resulting fresh products and their shelf life. This study was preliminary and needs to be studied at molecular level in detail.

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