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THERMAL, PASTING, AND RHEOLOGICAL PROPERTIES OF PROCESSED BUCKWHEAT (FAGOPYRUM ESCULENTUM)

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ABSTRACT

Objective: The aim of the study was to analyze the effect of various processing treatments on thermal, pasting, and rheological properties of buckwheat flour.

Methods: Buckwheat seeds were processed through different processing treatments including cooking, germination, and fermentation, and their flours were produced. The processed flours were analyzed for their thermal properties using differential scanning calorimeter, pasting properties using rapid visco-analyzer, and rheological properties using rotational rheometer.

Results: Fermented buckwheat flour showed significantly ($p \le 0.05$) higher onset temperature ($T_a = 66.6^{\circ}$ C), peak temperature ($T_p = 71.15^{\circ}$ C), conclusion temperature ($T_c = 78.03^{\circ}$ C), and enthalpy of gelatinization (1.89 J/g). The peak viscosity ranged from 39 to 1299 cp, lowest for cooked buckwheat flour and highest for fermented buckwheat flour. The native buckwheat flour showed the highest value, whereas cooked buckwheat flour showed the lowest value for storage modulus (G') and loss modulus (G''). The value of tan ∂ was lower than 1 for native and processed buckwheat dough.

Conclusion: The changes observed in physicochemical properties of buckwheat flour after processing treatments provided a crucial basis for its potential applications on an industrial scale. Furthermore, buckwheat seeds are gluten-free; therefore, their flour or products can be used for persons suffering from celiac diseases.

Keywords: Germination, Fermentation, Thermal properties, Pasting properties, Rheology.

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INTRODUCTION

Buckwheat has been gaining importance because of its potential health benefits. Buckwheat seed is triangular in shape and belongs to the Polygonaceae family. Buckwheat is produced and consumed worldwide, and main species of buckwheat include tartary buckwheat (Fagopyrum tataricum) and common buckwheat (Fagopyrum esculentum). Buckwheat is an important source of various health beneficial components, including dietary fiber, minerals, proteins, carbohydrates, vitamins, and certain essential minerals [1]. Furthermore, buckwheat protein has well-balanced amino acids and free from gluten which makes it useful for a person suffering from celiac disease [2]. Buckwheat protein suppresses cholesterol level and gallstone formation more strongly than soy protein isolate. Buckwheat is a significant source of various bioactive components including phytosterols, polyphenols, saponins, fagopyritols, and rutin. Phenolic acids are known to act as antioxidants by donating hydrogen or electrons. Incorporation of food with components rich in phenolic acids bestows antimutagenic, antiglycemic, and antioxidative properties [3]. The products prepared from buckwheat are famous for their high level of resistant starch. Resistant starch has significant health benefits such as decline the risk of colon cancer, the rise in fecal bulking, hemorrhoids, and modulation of blood cholesterol and blood glucose levels.

Buckwheat is rich in mineral content, but the bioavailability of some minerals is reduced due to the presence of various antinutritional components such as tannin, phytic acid, and polyphenols. The processing of grains is associated with an improvement in bioavailability of nutrients and decrease in antinutrients. The processing methods such as cooking, germination, and fermentation have been found to improve the nutritional and functional quality of the seed. It is well known that processing causes many biochemical changes in food products. In recent years, medicinal effects in clinical applications of buckwheat have been widely investigated, and importance of buckwheat as food has been increasingly recognized in the prevention of several degenerative diseases such as cancer, diabetes, heart diseases, hypertension, and gallstone. Regular buckwheat consumption can thus be recommended as a lifestyle intervention to alleviate hypercholesterolemia and diabetes mellitus [4]. The isolated buckwheat starch has adaptability for many industrial processes in pharmaceuticals such as the use as a binding agent in tablet formation.

Many methods are developed for characterization of flours, which could be utilized for screening a large number of genotypes for their unique properties. Gelatinization is the phenomenon of disruption of molecular order of granules in the presence of water during heating. The process of gelatinization can be monitored using differential scanning calorimeter (DSC). Henshaw et al. [5] studied that apart from starch flours also contain lipids, protein, and minerals that may interact with starch to varying degrees and effect the gelatinization and pasting properties. The rheological properties of flours are very useful for the preparation of different products with desirable attributes. Dynamic oscillatory measurements, stress relaxation, strain sweep test, frequency sweep test, and creep and recovery tests are the different methods that have been used to analyze the viscoelasticity of flour. To transform buckwheat flour into a novel product, analysis of its functional properties is essential to promote the acceptability of the end product. However, despite health and nutritional importance, very limited information is available on effects of processing treatments such as germination, cooking, and fermentation on the thermal, pasting, and rheological properties of buckwheat flour. Therefore, the present investigation was aimed to study the effect of different processing treatments on thermal, pasting, and rheological properties of buckwheat flour which would further be utilized for production of the varied range of novel products.

METHODS

Seeds of buckwheat (*F. esculentum -* VL Ugal 7) were procured from Vivekanand Parvatiya Krishi Anusandhan Sansthan, Almora, Uttarakhand.

Processing treatments

Cooking (autoclaving)

Cooking of seeds was done according to the method of Jood *et al.* [6] with slight modifications. The seeds (100 g) were soaked in distilled water (1:3 w/v) for 12 hrs at room temperature. Pre-soaked seeds were cooked (1:2 w/v) at 1.05 kg/cm^2 (15 psi) pressure for 10 minutes in an autoclave and then dried at 40°C for 14 hrs. The dried seeds were milled using laboratory mill (Milcent Flour Mill), sieved through 0.15 mm screen, packed in airtight container, and stored in refrigerator for further analysis.

Fermentation

Fermentation of seeds was carried out according to the method described by Hassan *et al.* [7] with slight modifications. Seeds were milled and mixed with distilled water (1:3 w/v). The suspension was incubated at 37°C for 16 hrs. The fermented paste was dried in hot air oven at 40°C for 14 hrs, after which it was milled using laboratory mill (Milcent Flour Mill), sieved through 0.15 mm screen and packed in airtight container and stored in refrigerator for further analysis.

Germination

The germination of seeds was carried out according to the method described by de Ruiz and Bressani [8] with slight modifications. Seeds were washed thoroughly and soaked in distilled water (1:5 w/v) for 12 hrs at ambient temperature. The seeds were spread on petriplate lined with tissue paper, covered with moist muslin cloth, and allowed to germinate at 28°C for 48 hrs. The germinated seeds were drained and oven dried at 40°C for 14 hrs. The dried seeds were milled using laboratory mill (Milcent Flour Mill), sieved through 0.15 mm screen and packed in airtight container and stored in refrigerator for further analysis.

Thermal properties

Thermal properties of flours were analyzed using a DSC (500 TA Instruments, new castle, DE, USA) as explained earlier by Yadav *et al.* [9]. Onset temperature (T_o), peak temperature (T_p), conclusion temperature (T_c), and enthalpy of gelatinization (ΔH_{gel}) were computed automatically. Gelatinization range ($R=T_c-T_o$) and peak height index ($=\Delta H_{gel}/T_n-T_o$) were also calculated from observed data.

Pasting properties

Pasting properties of flours were analyzed using RVA (Rapid viscoanalyzer, Newport Scientific, Warriewood, Australia). The temperature profile was started from 50°C for 1 minute, followed by ramping the temperature linearly to 95°C in 3 minutes and 42 seconds, holding for 2 minutes and 30 seconds, cooling the system to 50°C in 3 minutes and 48 seconds, and ending the process in 13 minutes. Peak viscosity (PV), temperature at PV (P_{Temp}), hot paste viscosity (HPV) or holding strength, cool paste viscosity (CPV), breakdown (BD=PV-HPV), and set back (SB=CPV-HPV) were determined from the viscosity profile curve.

Rheological properties of dough

The rheological properties of native and processed dough were studied at 25°C using a modular compact rheometer (MCR02, Anton Paar, Germany, Gmbh). The parallel plate geometry (25 mm diameter, 1mm gap) was used. The temperature was regulated at 25°C by a controlled peltier system. The dough sample was placed in the rheometer measuring system, the excess dough was trimmed, and a thin layer of paraffin oil was applied to the edge of exposed sample to prevent the moisture loss during measurement. The dough was left for 15 minutes to equilibrate stresses, before starting the test. The following tests were performed for each sample: (1) Strain sweep test, the range of linear viscoelasticity was determined by checking the dependence of storage modulus (G') and loss modulus (G') on the applied stress in the range of 0.01-100 Pa and at a constant frequency of 1 Hz. (2) Frequency sweep test, in the range of 1-20 Hz at 0.1% strain within the linear viscoelastic region. Creep and recovery tests were performed at a controlled stress of 70 Pa, creep phase continued for 150 seconds, and recovery phase for 300 seconds. The measurements were conducted using a 25-mm diameter parallel plate configuration with 1 mm gap between the plates. As a result of these measurements, strain values were obtained as a function of time.

Statistical analysis

All observations were carried out in triplicate and analyzed using one- and two-way ANOVA (SPSS 19.0). *Post hoc* comparison test (Tukey's HSD) at p<0.05 was carried out to determine the significant difference among mean values of experimental data.

RESULTS AND DISCUSSION

Thermal properties

Gelatinization involves irreversible changes that occur when starch granules are heated in presence of water. The gelatinization properties of native and processed buckwheat flour are presented in Table 1. The peak temperature (T_n) of native and processed buckwheat flour were ranged from 69.56°C to 71.15°C. Fermented buckwheat flour showed highest T_n followed by germinated and native buckwheat flour. The increase in peak temperature after fermentation might be due to the growth of natural bacteria releasing more proteolytic enzymes that break down the grain cell walls losing more starch, hence increased crystalline structural proportions within the sample. A similar increase in peak temperature after fermentation was reported for foxtail millet flour [10]. The onset temperature (T_a), peak temperature (T_a), and conclusion temperature (T_) of fermented buckwheat flour were significantly higher than others. Enthalpy of gelatinization (Δ Hgel) was observed to be highest (1.89 J/g) for fermented buckwheat flour, whereas native buckwheat flour showed the lowest value (1.44 J/g). The enthalpy of gelatinization and gelatinization temperature range are believed to be increased with the crystallinity of starch granules. No endothermic peak of starch gelatinization in the cooked buckwheat flour was observed indicating that the starch was fully gelatinized during cooking.

Pasting properties

The data pertaining to pasting properties of native and processed buckwheat flour are presented in Fig. 1. The PV varied from 39 to 1299 cp, lowest for cooked buckwheat flour and highest for fermented buckwheat flour. The studies have shown that flour with low PV has a lower thickening power than flour with high PV. The increase in viscosity of fermented buckwheat flour might be due to the BD of complex carbohydrates to simple sugars [11]. The HPV ranged from 36 cp for cooked buckwheat flour to 1203 cp for fermented buckwheat flour. The CPV varied from 53 to 1763 cp. HPV is influenced by the formation of amylose-lipid complexes, the rate of amylose exudation, and extent of granule swelling while CPV is explained as the tendency of soluble

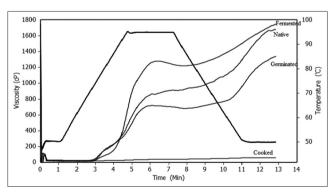


Fig. 1: Viscosity profiles of native and processed buckwheat flour

Table 1: Thermal properties of native and processed buckwheat flour

Т _. (°С)	T _p (°C)	T _c (°C)	$\Delta H_{gel}(J/g)$	PHI	R
64.39±0.45ª	69.56±0.35ª	76.37±0.17ª	1.44±0.08ª	0.29	11.96
-	-	-	-	-	-
66.60±0.26 ^c	71.15±0.24 ^c	78.03±0.27°	1.89±0.06°	0.42	11.37
65.79±0.29 ^b	70.11 ± 0.24^{b}	77.44±0.29 ^b	1.60 ± 0.08^{b}	0.37	11.63
	64.39±0.45 ^a - 66.60±0.26 ^c	64.39±0.45 ^a 666.60±0.26 ^c 666.60±0.26 ^c 666.60±0.24 ^c	64.39±0.45° 69.56±0.35° 76.37±0.17° - - - - 66.60±0.26° 71.15±0.24° 78.03±0.27°	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

Values are means ±SD of three (n=3) independent determinations. Values in the same column with different letters are significantly different (p≤0.05). T_{o} : Onset temperature, T_{o} : Peak temperature, T_{c} : Conclusion temperature, ΔH_{out} : Enthalpy of gelatinization, R: Gelatinization range (T_{c} - T_{o}), PHI: Peak height index (ΔH_{out})/ T_{n} - T_{o})

Table 2: Creep-recovery parameters of native and processed buckwheat flour

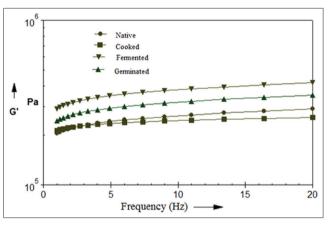
Samples	Creep phase			Recovery phase			
	J _o (10 ⁻⁵) (Pa ⁻¹)	J ₁ (10 ⁻⁴) (Pa ⁻¹)	R ²	J _{max} (10 ⁻⁴) (Pa ⁻¹)	J _o (10 ⁻⁵) (Pa ⁻¹)	J ₁ (10–4) (Pa ⁻¹)	R ²
Native	0.24±0.02ª	0.12±0.02 ^{ab}	0.99	0.34±0.05 ^{ab}	0.26±0.01ª	0.61±0.02 ^b	0.99
Cooked	0.28 ± 0.03^{b}	0.08 ± 0.02^{a}	0.99	0.24 ± 0.06^{a}	0.46 ± 0.01^{b}	0.32±0.06ª	0.99
Fermented	0.38 ± 0.02^{b}	0.17 ± 0.01^{b}	0.99	0.45 ± 0.03^{b}	0.34 ± 0.05^{ab}	0.75 ± 0.09^{b}	0.99
Germinated	0.23 ± 0.01^{a}	0.10 ± 0.01^{ab}	0.99	0.26 ± 0.01^{a}	0.22±0.03ª	0.59 ± 0.04^{b}	0.99

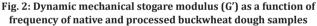
Values are means ±SD of three independent determinations. Values in the same column with different letters are significantly different ($p \le 0.05$). J_0 : Instantaneous compliance, J_1 : Viscoelastic compliance, R: Correlation coefficient, J_{mux} : Maximum creep compliance, SD: Standard deviation

amylose to retrograde on cooling. A significant (p<0.05) increase in PV, HPV, and CPV was observed in fermented buckwheat flour. The value of BD viscosity ranged from 3 to 96 cp. BD is the measure of the resistance of swollen granules to disintegrate at high temperature and gives the indication of the stability of the flour product. This shows that higher the BD, the lower the ability of the sample to withstand shear stress and heating during cooking. A significant (p<0.05) increase in BD was observed in fermented buckwheat flour. The result suggests that increased BD after fermentation in buckwheat would enable easy cooking but susceptible to stress when processed into the solid form. Cooked buckwheat flour had the lowest value of PV, HPV, CPV, and BD. This might be because of high temperature during cooking could complete the gelatinization of the starch in buckwheat flour [12]. The pasting temperature of the flour samples varied between 75.05°C and 85.81°C. The minimum temperature required to cook the flour is pasting temperature. The lowest pasting temperature was reported for native buckwheat flour, and highest was reported for fermented buckwheat flour.

Rheological properties of the dough

The dynamic oscillatory tests of all the samples were analyzed by performing frequency sweeps in the linear viscoelastic region. The linear viscoelastic region for buckwheat dough was determined by strain sweep experiments and limited up to 0.1% strain, indicating the deformation of buckwheat dough structure beyond this level. Similarly, it has been previously reported that wheat dough also exhibits linear viscoelasticity at strain levels lower than 0.1-0.25% [13]. The frequency sweep shows the elastic and viscous behavior of the material with the rate of application of stress or strain, while the amplitude is kept constant [14]. The frequency sweep curves of native and processed buckwheat flour were presented in Figs. 2-3. For all the samples, during the whole range of frequency, storage modulus G' (elastic property) was greater than loss modulus G" (viscous property) showing solid elastic-like behavior of native and processed buckwheat dough. Similar behavior was reported for gluten-free flours including buckwheat flour dough [15] and rice flour dough [16]. Storage modulus and loss modulus were also frequency dependent as they increase with the increase in frequency. Fermented buckwheat flour showed the highest value for storage and loss modulus, while cooked buckwheat flour showed the lowest value for storage and loss modulus. Therefore, cooked buckwheat flour exhibited weak viscoelastic properties among native and processed buckwheat flours. The damping factor can be used to determine the viscoelastic character of polysaccharides. Furthermore, higher is the difference between storage modulus and loss modulus and lower is the value of damping factor (tan $\partial = G''/G'$).





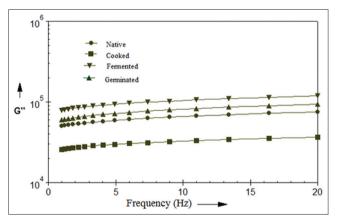


Fig. 3: Dynamic mechanical loss modulus (G") as a function of frequency of native and processed buckwheat dough samples

The fermented buckwheat flour had the highest value for damping factor, whereas cooked buckwheat flour had lowest value of damping factor. The value of tan δ less than one shows elastic behavior, whereas the value of tan δ greater than one shows viscous behavior. The value of tan ∂ for native and processed buckwheat dough was lower than 1; the observed rheological properties were typical of weak

gels also indicate that buckwheat dough has more elastic character than viscous character [17]. The similar observation was reported by Sivaramakrishnan *et al.* [18] for gluten-free dough of rice flour.

The comparison of creep-recovery data of native and processed buckwheat flour dough is presented in Table 2. Creep-recovery phenomenon is caused by the reorientation of the intermolecular bonds in a viscoelastic material [19]. Instantaneous compliance is related to the energy of elastic stretching of the bonds, when stress is applied and vanishes immediately after its removal. On the other hand, viscoelastic compliance is related to the disturbance and conversion of the bonds [20]. The instantaneous compliance ([_) of native and processed buckwheat flour varied from 0.23×10^{-5} to 0.38×10^{-5} . The highest instantaneous compliance was observed for fermented buckwheat dough, whereas lowest was reported for germinated buckwheat flour. The zero shear viscosity is the flowability of material at the end of applied load and was highest for native buckwheat flour and lowest was observed for germinated buckwheat flour. The high value of zero shear viscosity leads to lower in the fluidity of the dough. Creep compliance value is mainly related with softness [21]. During the recovery phase, the maximum creep compliance (J_{max}) of buckwheat flour varied from 0.24×10^{-4} to 0.45×10^{-4} . The cooked buckwheat flour had significantly lower maximum creep compliance while fermented buckwheat flour had highest value for maximum creep compliance. The flour having high creep values with time has weak structures; however, low values are indication of strong structures. The mean retardation time (λ_1) during the recovery phase ranged from 35.57 to 72.79 seconds. The highest mean retardation time was reported for germinated buckwheat flour, and lowest was reported for native buckwheat flour. Therefore, from the present study, it was concluded that fermented buckwheat flour had a higher value of maximum creep compliance results in weakened the dough structure. In native buckwheat dough, the amount of elastic component during creep-recovery test was 36.38% and the viscous part was 63.62%.

CONCLUSION

Fermented buckwheat flour showed the highest value for gelatinization and pasting temperature. The highest PV was reported for fermented buckwheat flour and lowest for cooked buckwheat flour. The low value of tan ∂ (<1) indicates that buckwheat dough prepared from native and processed flour has more elastic character than viscous character. The results of the study revealed that the processing treatments, i.e., cooking, fermentation, and germination had a significant effect on thermal, pasting, and rheological properties of flour. Therefore, the processed buckwheat grain can be utilized in the development of novel and functional food products with enhanced nutritional qualities.

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