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Morphological, Pasting And Thermal Properties Of Common Buckwheat (Fagopyrum Esculentum Moench) Flour And Starch

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Abstract: Common buckwheat (F. esculentum Moench) is a major pseudo- cereal used in the form of groats and flour in many processed functional foods. Starch is the main component in buckwheat flour which plays an important role in the functional and technological properties of end-use food products. In the present study starch was isolated; flour and starch isolates of buckwheat were studied for their pasting properties using RVA and thermal properties using DSC. Micro-structural characteristics starch granules were analysed by SEM. Granules of starch were oval, spherical and polygonal in shape and having noticeable flat surface areas. Scanned image of flour showed more agglomeration of granules. Results revealed that, presence of protein, lipids and other components affects the pasting and thermal properties of flour and starch. Pasting temperature of flour was higher than starch indicates the higher temperature requirement for gelatinization. RVA analysis showed higher peak, trough, and final and set back viscosity of starch than corresponding viscosities of flour. Slightly higher onset temperature was observes for flour than starch sample while conclusion temperature of starch was higher than flour. Difference in enthalpy change was noticed and starch showed higher values for enthalpy change. Understanding of these properties of buckwheat starch and flour supports in the selection of processing parameters in the food industry, and opens up new opportunities of industrial applications.

Keywords: Common buckwheat; starch; RVA; DSC; SEM.

I. INTRODUCTION

The most abundant polysaccharide in plants existing naturally within the plant cells in the form of granules is starch. It is the important source of carbohydrate utilized in food industries in the form of thickener, bulking agent and gelling agent. Pasting, gelatinization, and solubility, swelling and morphological properties of starch mainly govern the application of starch. Particle size of starch granules contributes to specific functional properties of starch such as texture, volume, consistency, and moisture and shelf stability (Raeker and others, 1998). Additionally, the size of starch granules affects the temperatures of gelatinization, which influences the properties of food (Soral-Smietanaet al., 1984). Gelatinization of starch describes the disruption of the molecular order within the starch granules during heating in the presence of water. Indication of the loss of an organized structure includes irreversible swelling of starch granules, loss of birefringence and crystallinity. The process of gelatinization is energy absorbing than can be monitored through Differential Scanning Caloriety (DSC) which has been used for analysis of starch (Freitas et al., 2004). In DSC thermogram, thermal stability of samples can be observed by the onset temperature or peak temperature, while the transformed proportion is reflected by the area under the endothermic peak, representing the enthalpy change (Li-Chan and Ma, 2002). It is generally accepted that swelling of granules increase the viscosity during heating of starch in water. Breakdown refers the rigidity/fragility of the swollen granules. The properties of the swollen granules and the soluble materials leached out from the granules are cooperatively ruling the viscosity parameters during pasting (Doublier et al., 1987). Growing demand of starch has created interest in searching the non-conventional starch sources and buckwheat (*Fagopyrum*) is a promising new starch source as it produces starch with diverse physicochemical, structural and thermal properties that give it a broad range of potential industrial applications. Generally, common buckwheat (*F. esculentum* Moench) is grown more in Asian buckwheat growing countries, such as Japan, Korea, and the central and northern parts of China and the same is true in Europe, USA, Canada, Brazil, South Africa and Australia. In India common buckwheat is grown in mountainous regions and in some states like Gujrat and Rajasthan. Because of the nutritious advantages of buckwheat, this crop has been considered as a raw material for functional food and medicine. Buckwheat is consumed in the form of flour for making noodles, pancakes, and pasta; and, as whole-seed groats for making porridge, salad, and tea. Minor crops are usually less investigated and hence the information on flour and on the ultra-structural details starch of buckwheat starch is limited. The size and shape of starch granules are species specific (Stark and Lynn 1992). Kim et al. (1977) reported that buckwheat starch granules are polygonal but slightly larger than rice; have a 25% amylose content by iodine colorimetry; a gelatinization temperature of 61–65ºC; and high cool paste viscosity in a Brabender ViscoAmylograph. Better understanding of the structure and morphology of starch granules and examination of thermal properties of starch can present the important information to direct the processing and the application of starch. Even though lots of work has been done on starch systems, few studies have evaluated starch of common buckwheat. Thus, the present investigation focuses on pasting, thermal and micro-structural analysis of flour and starch of common buckwheat.

II. MATERIALS AND METHODS

A. MATERIALS

Grains of common buckwheat of cultivar named VL-7 were used in this study and procured from National Bureau of Plant Genetic Resources Regional Station, Shimla, India. The grains were screened to remove defective grains and foreign matter if present and stored in sealed container at room temperature previous to their use. The flour were prepared by grinding seeds on laboratory mill and stored in polyethylene bags at 10ºC. Chemicals used for the analysis purpose were of analytical grade.

B. ISOLATION OF STARCH

The alkaline steeping method of Choi et al. (2000) was followed to isolate the starch from common buckwheat grains. Grains were steeped in 0.25% aqueous NaOH solution for 18 h at room temperature and stirred three times during this period. Grains were washed 2-3 times with distilled water and ground in a blender at full speed for 2 min. Slurry was filtered step wise through 100 mash (150µm) and 270 mesh (53µm) sieves. The starch was isolated from the filtrate by centrifugation at 25,000g for 20 min. The supernatant was discarded, and the top yellowish layer of protein was removed. This step was repeated to obtain a white starch layer. The starch layer was resuspended in distilled water, shaken and centrifuged as described above. Thereafter, the isolated starch was dried in hot air oven at below 40ºC for 8-10 hours and stored at room temperature in sealed container.

C. DETERMINATION OF AMYLOSE CONTENT

The amylose content of starch and flour of common buckwheat was determined according to modified methods of Williams et al. (1970). The standard curve used for amylose was Y = 0.0089X + 0.0528 (r= 0.99), where X = amylose content (%), and Y = absorbance at 680 nm, based on fractionation of rice starch by Montgomery and Senti (1958).

D. SCANNING ELECTRON MICROSCOPY

The granule morphology of flour and starch sample was observed in a scanning electron microscope (SEM). The sample was mounted on an aluminium stub using a double sided copper tape and coated with gold in sputter coater. The samples were examined at suitable magnification at an accelerated voltage of 10kV. The selected regions were photographed and saved.

E. PASTING PROPERTIES

Pasting properties of starch were determined using a Rapid Visco Analyser (Perten Instruments, Australia). Starch (3 g, 14% moisture basis) was mixed with calculated amount of distilled water in the RVA sample canister. The slurry was manually homogenized using plastic paddle to avoid lump formation before RVA run. A programmed heating and cooling cycle was used where the samples were held at 50°C for 1 min, heated to 95°C in 3.30 min, held at 95°C for 3 min before cooling to 50°C in 3.30 min and holding at 50°C for 2 min. The mixture was stirred at a constant speed of 160 rpm during the test. A RVA plot of viscosity (cP) versus time (s) was used to determine peak viscosity (PV), trough viscosity (T), breakdown viscosity(BD), final viscosity (FV), set back (SB), peak time (P time) and pasting temperature (P temp).

F. THERMAL PROPERTIES

Thermal properties of starch and flour of buckwheat were analysed using a Differential Scanning Calorimeter 2920 (TA Instruments, New Castle, DE, USA) according to Bao et al. (2004) with some modifications. Starch (6.0 mg, db) was weighed into an aluminium pan and 6µl distilled water was added. The pan was hermetically sealed and equilibrated at room temperature for 1 h, then heated at the rate of 10°C/min from 30°C to 120°C with an empty sealed pan as a reference. Parameters such as onset (To), peak (Tp), conclusion (Tc) temperature and enthalpy (ΔH) of gelatinisation were determined by software provided in the system.

III. RESULTS AND DISCUSSION

Amylose content is an important factor affecting functional properties like swelling power and solubility of flour and starch. The amylose content in buckwheat flour was 18.48% which was in range (19-28%) reported by Qin et al. (2010) for thirty nine varieties of buckwheat flour. Amylose content of starch (35.66%) was slightly higher than the range (22%-33%) registered for the amylose content of buckwhwat starch in earlier studies (Li et al., 1997; Pandey et al., 2015) , and comparable to the amylose content of cereal, root, tuber and legume starches. However, amylose content of buckwheat was also reported as high as 46.6% (Qian et al., 1998).

	Flour	Starch
Peakn Viscosity (cP)	1637.66±34.93	3106±103.71
Trough Viscosity (cP)	1528.33±60.66	1904.33±144.07
Breakdown viscosity (cP)	86±17.08	1201.66±46.30
Final Viscosity (cP)	3028.33±191.26	3514.33±219.06
Setback Viscosity (cP)	1510.33±113.7	1610±80.55
Peak Time (min)	7.0±0.0	4.0±0.0
Pasting temperature (°C)	72.63±0.02	71.33±0.49

Table 1: Pasting properties of common buckwheat flour and starch

Change in viscosity of starch paste during heating and cooling is up to some extend governs the wide applications of starch in food application. The pasting curve corresponds to changes in behaviour of paste viscosity of flour and starch with change in temperature and mainly varies with composition of flour and characteristics of starch. Pasting properties of starch and flour of buckwheat are depicted in Table 1. Increase in viscosity during heating may be attributed to the swelling of granules, as a result of loss of crystalline order and absorption of water (Bao and Bergman, 2004). Initiation temperature for gelatinization of buckwheat flour was 72.63°C which was similar to the range of 71.3°C-72.1°C reported by Shevkani et al. (2014) for amaranth flour and higher than the value of 64°C for rice flour reported by Qui et al. (2015). The high pasting temperature of flour as compare to other cereals flour indicates its higher resistance towards swelling. Pasting temperature of starch was 71.33°C that was higher than that reported (62.8°C) by Li et al. (1997) for tartary buckwheat starch. Yoshimoto et al. (2004) studied eight kinds of buckwheat cultivars and reported 70°C as maximum pasting temperature of starches. The point of maximum swelling of starch granules is pointed out by peak viscosity. Peak viscosity of flour (1637.66cP) was found to be lower than the starch (3106cP). Yoshimoto et al. (2004) reported peak viscosities of starches of various cultivars of buckwheat range from 2712cP to 3132cP. Break down viscosity, is the measure of resistance of gel to disintegrate at high temperature, noticed 1201.66cP for starch was lower than the findings of Lui et al. (2014) observed 1612cP for buckwheat starch. Lower breakdown viscosity of flour than starch represents greater resistance to shear thinning and high stability of paste. Final viscosity represents the ability of starch/flour to form a viscous paste, was found to be 3028.33cP and 3514.33cP for flour and starch respectively. Final viscosity of starch in present observation was lower than the reports of Lui et al. (2014) noticed 4208cP for buckwheat starch. Increase in final viscosity from peak viscosity might be due to the aggregation of amylose molecules (Miles et al., 1985). Set Back viscosity is the measure of syneresis upon cooling of cooked paste. Set back viscosity of starch was lower than the range (2172cP -2712cP) registered by Yoshimoto et al. (2004) and comparable set back viscosities were noticed by Lui et al. (2014) for buckwheat starches. During RVA analysis viscosity of flour was lower than starch at every point, which might be due to presence of less percent of starch in flour than starch isolate, as the pasting properties depends mainly on starch present in sample. Hamaker and Griffin (1993) and Yang and Chang (1999) noticed that proteins in flour restrict starch granules swelling and reduce the amylogram viscosity. Protein removal was found to increase the paste viscosity of starch (Yang and Chang, 1999). The role of lipids on the pasting properties was observed as positive relation with pasting temperature while negative relation with viscosity (Shevkani et al., 2014; Singh et al., 2014). Hence, the results of present investigation agreed with these conclusions of earlier investigations.

	Flour	Starch
Onset temperature (°C)	65.73±1.3	64.13±1.20
Peak temperature(°C)	75.1±0.8	77.3±1.02
Conclusion temperature(°C)	79.9±1.4	83.1±0.1
Enthalpy change(J/g)	9.9±1.4	11.2±0.5
Amylose content (%)	18.48±0.3	35.66±0.1

Table 2: Thermal properties and amylose content of flour and starch of common buckwheat

Results of differential scanning calorimetry which measured the transition temperatures (T_0, T_P, T_C) and gelatinization enthalpy (ΔH) of starch and flour of buckwheat are given in Table 2. The value of onset temperature represents the internal structure of the granule during its disintegrations, which results in the release of polysaccharide into the immediate medium (Singh et al., 2006). DSC analysis of flour and starch of buckwheat showed that the gelatinization temperature of the flour (65.73°C) was slightly higher than that of the starch (64.13°C). The difference could be attributed to the presence of mucilage in the flour. In addition to protein, the mucilage also contains complex polysaccharides which could compete with starch for moisture and result in a higher onset starch gelatinization temperature in the flour (Taxi et al., 1972). Similar trend of transition temperatures were observed by Jane et al. (1992) for taro starches and flours. The peak temperature for flour (75.1°C) and starch (77.3°C) were slightly higher than paste temperature obtained by RVA for flour and starch (72.63°C and 71.33°C respectively). Similar observations were found in earlier studies (Li et al., 1997; Yoshimoto et al., 2004) reported onset temperature ranged from 59°C to 64°C for various cultivars of buckwheat. The peak temperature and conclusion temperature of starch was similar to the analysis by Lui et al. (1997) reported peak temperature 78°C and conclusion temperature 83.9°C for tartary buckwheat starch. During DSC analysis the transformed proportion of starch is reflected by the area under the endothermic peak, representing the enthalpy change (ΔH). The enthalpy of gelatinization of starch (11.2 J/g) was in the range (2.14-12.7 J/g) registered in literature by other authors (Li et al., 1997; Zheng et al., 1998; Qian and Kuhn, 1999) however, it was lowerr than the findings of some other investigations (Yoshimoto et al., 2004; Lui et al., 2014), reported range of enthalpy change 14.5-19.8 J/g. The differences in thermal properties could be due to the different varieties used and growing location of the observed buckwheat grains.

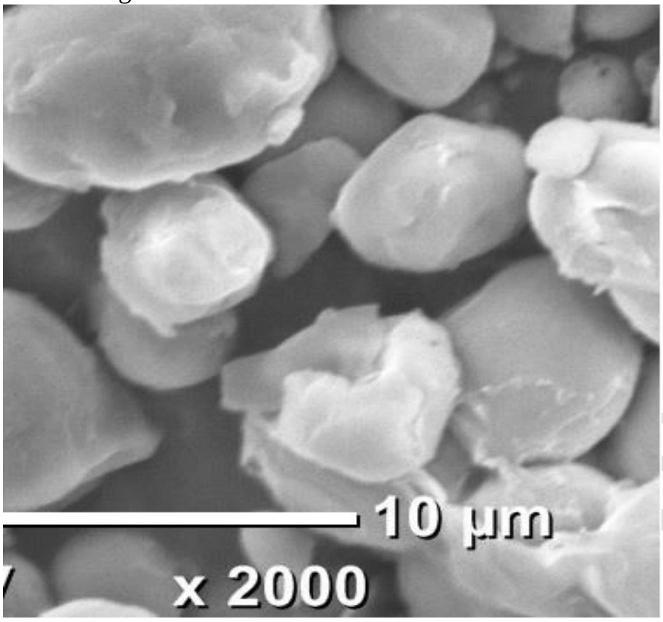

Figure 1: Scanning electron micrographs of common buckwheat flour A (X2000)

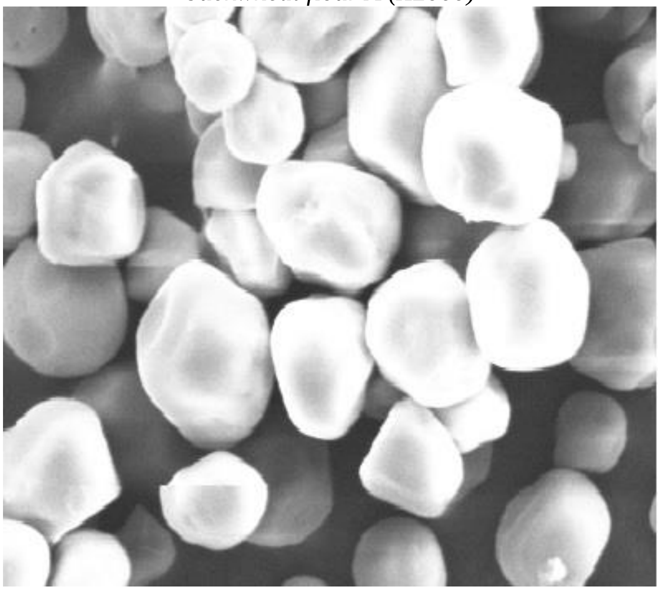

Figure 2: Scanning electron micrographs of common buckwheat starch B (X1000)

Scanned image of flour and starch was shown in Fig. 1 and Fig. 2 respectively. Morphological characteristics like size, shape and distribution of starch granules are attributed to the biological origin of the grain (Vandeputte and Delcour, 2004). The microstructure of the starch revealed that the shape of starch granules were polygonal, oval or spherical with visible flat areas due to compact packing in the grain endosperm. As regard to size, diameter of granules ranged from 5.6-7.3µm. The size and shape of granules were in agreement with previous reports (Qian et al., 1998; Qian and Kuhn, 1999). Particle size of starch granules were in range of 2-14 µm, which were 1.6-2.4 time smaller than corn and wheat starch granules responsible for low starch recovery during wet extraction (Zheng et al., 1998). Compared to starch, granules of buckwheat flour were more agglomerated, certainly due to the presence of proteins and lipids, which could be forming complexes with starch (Nierle, 1990). No fissures were observed in the surface of starch granules. The smooth surface of starch granules signified that the particles were not damaged during isolation of starch.

IV. CONCLUSION

In the present study, noticeable differences in the thermal and pasting properties were observed between flour and starch of common buckwheat. Lower viscosity was noticed for flour than starch at every stage of RVA analysis. The higher onset temperature of flour than starch indicated that protein and mucilage in flour interfere with water absorption of starch granules and form basis for more energy requirement for occurrence of gelatinization during DSC analysis. Granules of buckwheat starch were irregular in shape and having large flat surface areas indicating compact packing in endosperm. Size of granules was smaller than starch of cereal sources which make its wide applications in food and non-food industries. Scanning electron microscopy revealed that granules of flour were more agglomerated than starch granules. Future work is necessary to characterise fine structures of common buckwheat starch and analyse their relationships with starch properties.

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