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ORIGINAL ARTICLE

Visco-thermal and structural characterization of water chestnut flour

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Abstract In order to increase flour recovery, resistant starch content and to lower the glycemic index and glycemic load, the water chestnuts were subjected to pre-optimized conditions of pre-conditioning. The low glycemic index water chestnut flour (F1) obtained thereafter was analyzed for different functional, viscous, thermal and structural properties. F1 exhibited improved functional properties due to gelatinization of starch followed by retrogradation during pre-conditioning which confirms its feasibility for development of diverse food products in comparison to commercially available market flour (F2). Pasting properties—peak viscosity, hold viscosity, breakdown viscosity, final viscosity and set back viscosity (SBV) were found significantly ($p < 0.01$) higher in case of F1 than F2. Higher peak viscosity of F1 can be accorded to its higher swelling capacity than F2. Further, higher SBV of F1 suggests its susceptibility towards retrogradation and gel formation. Differential scanning calorimetry results revealed that gelatinization temperature, endothermic peak width, onset, peak and conclusion temperatures were significantly ($p < 0.01$) lower, whereas enthalpy of gelatinization and peak height index were significantly ($p < 0.01$) higher in case of F1 as compared to F2. Lower gelatinization transition temperatures of F1 could be attributed to its more water absorption ability than F2 which suggests its potential as a thickening agent in foods. ATR-FTIR studies revealed high absorbance ratio at 1047/1022 cm^{-1} in F1 as compared to F2 which confirmed the presence of packed double helices within the starch crystalline regions in F1 sample. Scanning electron microscopy showed the smooth, plumper and fused granules in F1 whereas disintegrated granules were observed in F2.

Keywords Low GI · Water chestnut flour · Functional behavior · DSC · ATR-FTIR

Introduction

The consequent rise in non-communicable diseases such as type II diabetes has incited research interventions for development of low GI food ingredients. The pandemic status of diabetes is a point of great concern nowadays which has created a paradigm shift from the interest in quantity of carbohydrates towards their quality. In this regard, low glycemic index food ingredients are credited with various health benefits especially for maintaining the glucose levels and reducing the insulin resistance in diabetic (Brand-Miller et al. 2003). Water chestnut is an aquatic angiosperm of *Trapaceae* family. Its kernels are tender, sweet, low in fats and a source of gluten-free starch. The dried kernels are ground to flour which is rich in macronutrients, minerals, vitamins, bioactive flavonoids and antioxidants (Rani et al. 2016).

Despite the high nutritional profile of water chestnut flour, its use as a major ingredient in food processing industries is still limited. However, in view of its low glycemic index (GI) score, water chestnut flour can serve as a favorable carrier for development of various functional bakery products, ready-to-eat snacks, desserts and other foods. The inclusion of low GI water chestnut flour in the therapeutic diets can also help to reduce the severity of

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type II diabetes, cardiovascular diseases and many other related metabolic risks (Shafi et al. 2016). Therefore, water chestnut flour un-doubtedly can serve as a suitable candidate in dietary management of diabetes due to its low GI but detailed studies on functional, viscous, thermal and structural properties are needed in this regard, so as to explore its use at commercial scale.

The physico-chemical properties of water chestnut starch as well as flour have been studied by many researchers in past (Shafi et al. 2016; Gani et al. 2010). However, the functional, viscous, thermal and structural characterization of water chestnut flour which could demonstrate its suitability for product development has remained unexplored so far. Starch interacts with other minor and major ingredients via various biochemical reactions during food processing which governs the overall acceptability of final product. Therefore, such complex molecular and structural interaction of food components in a system are essential and can be understood only through functional, viscous, thermal and structural properties of the raw material. Thus, there arises a need to conduct a systematic study on functional, viscous, thermal and structural properties of low GI water chestnut flour in order to decide its suitability for end use and to predict the dough characteristics suitable for processing so that simulation with acceptable market products can be accomplished.

Therefore, this study was envisaged as an extension of our previous work undertaken to optimize the pre-conditioning process for development of low GI water chestnut flour under All India Coordinated Research Project on Post-Harvest Engineering and Technology (Hussain et al. 2019). The aim of this work was to evaluate the functional, viscous, thermal and structural properties of aforementioned water chestnut flour in comparison to commercially available water chestnut flour so as to access its suitability to serve as potential ingredient for development of low GI foods.

Material and methods

Flour preparation

Water chestnuts harvested from Walur lake located in Jammu and Kashmir, India were subjected to pre-optimized conditions of pre-conditioning i.e., water chestnut: water ratio 1:1.5, water temperature 87.85 °C and pre-conditioning time 45 min (Hussain et al. 2019). The pre-conditioned samples were decorticated manually to extract the kernels. The kernels were dried in a tray drier (NSW-154, Scientific works New Delhi) at 45 ± 5 °C till the moisture content of 9.5 ± 0.5% was achieved. The dried kernels were ground to flour in a lab mill (3303, Perten, Sweden). The developed flour was evaluated for glycemic index, glycemic load and resistant starch content. Glycemic index (GI) of the samples was determined by following the procedures of Goni et al. (1997). The rate of starch digestion was expressed as the percentage of total starch hydrolyzed at a time difference of 30 min. The area under curve (AUC) for hydrolysis of all the samples was calculated for the release of glucose concentration against time. The hydrolysis index (HI) was calculated as the relation between the AUC for a sample and the AUC for a reference material- white bread, expressed as percentage. The kinetics of starch hydrolysis, the area under the hydrolysis curve (AUC), hydrolysis index (HI) and glycemic index (GI) were calculated by the equations given below:

$$C = C_{\infty}\left(1 - e^{-kt}\right) \quad (1)$$

$$AUC = C_{\infty}(t_f - t_0) - (C_{\infty}/k)(1 - \exp(-k(t_f - t_0))) \quad (2)$$

$$HI = \left(AUC_{sample}/AUC_{white\ bread}\right) \times 100 \quad (3)$$

$$GI = 39.71 + 0.549HI \quad (4)$$

where C is the percentage of starch hydrolyzed at time t, C_{∞} is the percentage of starch hydrolyzed after 180 min, k is the kinetic constant (min^{-1}), t is the time (min), t_f is the final time (180 min), t_0 is the initial time (0 min). Glycemic load (GL) was estimated indirectly by multiplying the amount of available carbohydrate contained in a nominal serving (50 g) of water chest flour with glycemic index (GI) value of flour, divided by 100 (Salmeron et al. 1997).

$$GL = \frac{\text{Available carbohydrate}}{100} \times GI \quad (5)$$

The available carbohydrate per serving was calculated by subtracting dietary fiber from the total carbohydrate content of flour sample. Resistant starch was determined using the Megazyme Assay Kit (Megazyme International, Wicklow, Ireland) according to approved AACC method 32–40 (AACC 2000). The water chestnut flour obtained after pre-conditioning had a GI of 30.21, glycemic load of 22.97 and resistant starch content of 40.24% (Hussain et al. 2019). Both developed low GI water chestnut flour (F1) as well as water chestnut flour commercially available in the market (F2) were evaluated for various physico-chemical and structural properties. In the entire region of Jammu and Kashmir, the commercially available water chestnut flour in the market is extracted from water chestnuts after subjecting them to dry heating over traditional chulas (made of mud) for about 10–12 days.

Particle size distribution

Particle size distribution test was carried out by following the procedures of Sakhare et al. (2014). Flour samples (200 g each) were sifted for 5 min in a Lab Sifter (Buhler,

Switzerland) using the sieves with opening of 180, 150,118 and 75 µm. The flour fractions passing through sieve openings of 180 and 150 µm were considered as coarser fractions and those passing through 118 and 75 µm sieve openings as finer fractions.

Functional properties

Bulk density of samples was determined by the method reported by Shafi et al. (2016). Flour sample was allowed to fall freely into a pre-weighed and dried 50-mL measuring flask. The flask was weighed again along with the sample. Bulk density was calculated as weight of the flour sample per unit volume and results were reported as g/cm^3.Water absorption capacity was determined by the method described by Sosulski et al. (1976). One gram suspension of water chestnut flour in 10 ml of distilled water was vortexed at room temperature for 30 min. The suspension was transferred into a pre-weighed tube and centrifuged at 3000 rpm for 30 min. The supernatant was decanted and wet sample in tube was weighed for determination of WAC as

$$\mathrm{WAC}(\%) = \frac{W_2 - W_1}{W_0} \times 100$$

where W_0 is weight of the flour; W_1 is weight of the centrifuge tube with sample; W_2 is weight of the centrifuge tube along with wet sample.

The methods described by Shad et al. (2011) were used for determining the solubility index and swelling capacity. One gram of water chestnut flour was dispersed in 50 ml distilled water and heated at 90 °C in water-bath (Gupta Pvt Ltd, Ambala Cantt, India) followed by 30 min of cooling. The slurry was then centrifuged at 2200 rpm for 15 min and the supernatant was decanted and dried in pre-weighed petri dish at 120 °C to complete dryness. Solubility index of flour was measured as:

$$\mathrm{SI}(\%) = \frac{W_1 - W_2}{W_1} \times 100$$

where W_1 is weight of the flour; W_2 is weight of the residue obtained after drying.

The wet sediment retained in the centrifuge tube after centrifugation was weighed for measurement of swelling capacity as

$$\mathrm{SC}(\%) = \frac{W_3 - W_1}{W_1} \times 100$$

where W_1 is weight of the flour; W_3 is weight of the wet sediment in centrifuge tube.

The least gelation concentration (LCG) was determined using the method described by Coffman and Garcia (1977). Water chestnut flour suspensions of 2, 4, 6, 8, 10, 12, 14, 16, 18 and 20% (m/v) were prepared in 10 ml distilled water in glass tubes followed by heating of suspensions in boiling water bath for 1 h. The suspensions were cooled to 4 °C and tubes were inverted. The least gelation capacity (LGC) was taken as the concentration at which the inverted suspension did not fall or slip.

The foaming capacity (FC) and foaming stability (FS) were determined as described by Narayana and Narsinga (1982). One gram of water chestnut flour was dispersed in 50 ml of distilled water in a graduated glass tube. The suspension was shaken for 2–5 min at room temperature. The volume of the suspension was measured before and after whipping. FC was calculated as:

$$\mathrm{FC}(\%) = \frac{V_2 - V_1}{V_1} \times 100$$

where V_1 is the volume before whipping; V_2 is the volume after whipping.

The foam was allowed to stand for one hour at room temperature and volume of foam was recorded after one hour to determine the foam stability as

$$\mathrm{FS}(\%) = \frac{V_t - V_0}{V_0} \times 100$$

where V_t is the foam volume after one hour; V_0 is the initial foam volume.

The emulsifying activity (EA) and emulsion stability (ES) were measured by the methods described by Yasumatsu et al. (1972). One gram of water chestnut flour was dispersed in 10 ml of distilled water in a graduated tube. 10 ml of soyabean oil was added to the suspension followed by homogenization to form an emulsion. The emulsion was centrifuged at 2000 rpm for 5 min and EA was calculated as:

$$\mathrm{EA}(\%) = \frac{H_2 - H_1}{H_1} \times 100$$

where H_1 is the initial height of the solution before centrifugation; H_2 is the height of the emulsified solution;

The emulsion was heated at 80 °C for 30minutes in a water bath followed by cooling to room temperature. The emulsion was centrifuged at 2000 rpm for 15 min and ES was calculated as

$$\mathrm{ES}(\%) = \frac{H_t}{H_2} \times 100$$

where H_t is the height of emulsified layer after heating; H_2 is the total height of the emulsified layer before heating;

Pasting properties

Pasting properties were determined with Rapid Visco analyzer (RVA Starch TM, Scientific Warrie Wood,

Australia) in accordance with the methods described by Batey et al. (1997). The slurry of water chestnut flour (3 g at 12% moisture content) was prepared by adding 25 ml of distilled water and was cooked in a temperature-controlled heating–cooling regime. The temperature was gradually raised to 50 °C and then increased further to 95 °C, held at 95 °C for 2.5 min, then cooled back to 50 °C and held for 2 min. The different recorded parameters were pasting temperature, peak viscosity, hold viscosity (minimum viscosity at 95 °C), final viscosity (viscosity at 50 °C), break down viscosity (peak viscosity–hold viscosity) and set back viscosity (final viscosity–hold viscosity).

Thermal properties

Thermal properties were determined by using differential scanning calorimeter, model-DSC-822^e (Mettler Toledo, Switzerland) as described by Shah et al. (2016). Flour samples (3.5 mg) were weighed into aluminum pans and deionized water (8 µl) was added. The pans were sealed hermetically and kept at room temperature overnight before analysis. The samples were heated at 10 °C/min from 20 to 150 °C. An empty aluminum pan was used as a reference. The endothermic peak width, gelatinization temperature range (R), and peak height index (PHI) were calculated as

$$\text{Endothermic peak width} = (T_c - T_o)$$

$$\text{Gelatinization temperature range (R)} = 2(T_p - T_o)$$

$$\text{Peak height index (PHI)} = \frac{\Delta H}{T_p - T_0}$$

Attenuated total reflection-Fourier transform infrared spectroscopy (ATR-FTIR)

ATR-FTIR spectra of the flour samples were recorded in the range of 4000–400 cm^{-1} by a FTIR spectrometer system (Cary 630 FTIR, Agilent Technologies, USA), coupled with an ATR accessory. Analysis was carried out at room temperature, at a resolution of 4 cm^{-1}, using Resolution Pro software version 2.5.5 (Agilent Technologies, USA) (Ashwar et al. 2016).

Scanning electron microscopic (SEM) imaging

SEM was used to study the morphology of water chestnut flour samples. The samples were glued onto a sample holder using double-sided cellophane tape and then coated with gold. The coated samples were photographed using a scanning electron microscope (Hitachi S-300H- Japan), at an accelerator potential of 5 kV to visualize the structure of water chestnut flour samples.

Statistical analysis

In case of functional, pasting and thermal properties, each measurement was replicated three times and data was expressed as mean ± standard deviation. The results obtained for all the parameters were statistically analyzed by employing paired Students t test. The statistical difference was determined with p value < 0.01.

Results and discussion

Particle size distribution

Particle size fractionation of the flour is critical for determining its functionality in processing and packaging (Sakhare et al. 2014). Particle size distribution of F1 and F2 were significantly ($p < 0.01$) different. After passing the two flour samples through the set of sieves, 11.60% and 15.67% of the flour was coarse (with an average particle size between 150 and 180 µm); 42.30% and 41.30% was fine (with an average particle size between 75 and 118 µm) and 46.10% and 43.03% was very fine fraction (with an average particle size less than 75 µm) in case of F1 and F2 respectively. Lower percentage of fine particles in F2 are due to the existing practice of prolonged dry heating of water chestnuts prior to flour preparation which hardens the endosperm and leads to large particle size in F1 compared to the F2. Mir et al. (2014) reported that in addition to grinding conditions, botanical source and texture can also be responsible for variations in particle size distribution.

Functional properties

Bulk density

Bulk density (BD) of two flour samples were significantly ($p < 0.01$) different than each other (Table 1). Lower bulk density of F1 (0.54 g/cm^3) than F2 (0.60 g/cm^3) can be attributed to higher percentage (46.10%) of very fine particles in F1. The fine particle size tends to lower the BD of flour. Low BD of F1 demonstrates a desirable storage trait as well as its feasibility for development of complementary food products (Awolu 2017).

Water absorption capacity

Water absorption capacity (WAC) determines the ability of a known weight of flour to absorb water depending upon the presence of hydrophilic/polar proteins and polysaccharides (Shad et al. 2011). The WAC of F1 was recorded to be 110 g/100 g which was significantly ($p < 0.01$)

Table 1 Functional properties of developed low GI water chestnut flour and commercially available water chestnut flour

Sample	Bulk density (g/cm^3)	Water absorption capacity (g/100 g)	Solubility index (%)	Swelling capacity (%)	Least gelation concentration (%)	Foaming capacity (%)	Foaming stability (%)	Emulsifying activity (%)	Emulsion stability (%)
Developed low GI water chestnut flour (F1)	0.54 ± 0.02	110 ± 0.05	10.56 ± 0.31	8.67 ± 0.33	14 ± 0.52	23.15 ± 0.42	80.12 ± 0.51	1.60 ± 0.10	50.65 ± 0.20
Commercially available water chestnut flour (F2)	0.60 ± 0.05	87 ± 0.3	12.40 ± 0.41	7.4 ± 0.32	18 ± 0.41	21 ± 0.52	75 ± 0.4	1.55 ± 0.32	49.03 ± 0.25
t value	8.57*	89.80*	89.50*	25.19*	13.35*	7.61*	46.14*	NS	17.46*

*Significant calculated *t* value at $p < (0.01)$

higher than F2 (87 g/100 g) (Table 1). Prior to flour preparation, gelatinization taking place during pre-conditioning which weakens the intermolecular association between amylose and amylopectin molecules and possibly resulted in higher WAC of F1 as compared to F2. High WAC demonstrates the desirability of developed WCF for making products like gravies, confectionary, soups, batters and bakery doughs where high viscosity and consistency is required as well as its possible use as bulking and texturizing agent (Adebowalea et al. 2005). Lower WAC of F2 can be attributed to strong hydrogen bonding between fragmented starch structure because of repolymerization due to dextrinization during prolonged dry heating prior to flour preparation which limits the availability of hydroxyl groups for water binding. Yadav et al. (2012) has reported that excessive heat treatment changes the availability of water binding sites due to changes in carbohydrate and protein structures. The WAC of developed WCF (110 g/100 g) was found higher than the WAC of horse chestnut flour (85 g/100 g) reported by Rafiq et al. (2015) which indicates the presence of more water binding polar groups in the developed WCF flour as compared to other non-conventional sources.

Solubility index

Solubility index measures the water-soluble components present in the flour matrix (Rafiq et al. 2015). Solubility index of F1 (10.56%) was found to be significantly ($p < 0.01$) lower than F2 (12.40%) (Table 1). Water chestnuts lose the ordered molecular structure due to gelatinization during pre-conditioning. On the other hand, prolonged dry heating of water chestnuts prior to flour preparation in case of F2 increased the damaged starch content. Starch damage causes exposure of hydrophilic groups with an increased leaching of soluble starch remnants including soluble non-starch components which increases the solubility index (Hasjim et al. 2012).

Swelling capacity

Starch granules when heated in aqueous solution imbibe water due to rupture of intermolecular hydrogen bonds which leads to granular swelling, gel formation and loss of native starch structure (Rafiq et al. 2015). The swelling capacity of F1 was significantly ($p < 0.01$) higher than F2. The higher swelling capacity of F1 (8.67%) than F2 (7.4%) (Table1) indicates good eating quality of F1 compared to F2. Eating quality of various processed products is correlated with retention of water in swollen starch granules (Falade and Okafor 2015). Swelling of starch granules denotes hydration ability which takes place during the gelatinization process. Thus, higher

WAC of F1 is responsible for its higher swelling capacity.

Least gelation concentration

Least gelation concentration (LGC) is used as an index of gel forming ability of a flour. Gelation is favored at high protein concentration owing to greater electrostatic and hydrophobic interactions (Shad et al. 2011). The LGC of F1 was significantly ($p < 0.01$) lower than F2. LGC of F1 was obtained at 14% while that of F2 was obtained at 18% (Table 1). A flour with good gelling property has lower LGC. Gelation ability is also positively associated with water absorption and swelling capacity. Lower LGC of F1 can be attributed to partial denaturation of proteins and starch structural changes which compete for available water binding sites for gelation and gelatinization respectively. It is also reported that heat-moisture treatment improves the gelation properties of flour by gelatinization and partial denaturation of proteins Olu-Owolab et al. (2011), thus, pre-conditioning water chestnuts prior to flour preparation under high moisture and temperature conditions might be the reason for lower LGC of F1 compared to F2 where water chestnuts are subjected to prolonged dry heating prior to flour preparation leading to complete denaturation of proteins. Developed WCF can be thus a suitable carrier to be used in food thickening applications and for retention of flavors. Low LCG values are desirable for use in food thickening applications and in new product development by providing a structural matrix for holding water and flavoring ingredients.

Foaming capacity

Proteins have a tendency to reduce the interfacial tension between the air–water interface in the foams (Shad et al. 2011). Foaming capacity (FC) of F1 was found to be 23.15%, while as that of F2 was 21% (Table 1). High FC of F1 in comparison to F2 might be related to flexibility of proteins which reduce the interfacial tension by rapid adsorption and conformational rearrangement (Asghari et al. 2016) while as prolonged dry heating of water chestnuts prior to flour preparation leads to almost complete denaturation of proteins rendering them insoluble and thus resulted in lower FC of F2.

Foaming stability

Foaming stability (FS) is the measure of how well a protein is able to stabilize the bubble formation by forming a thin viscoelastic film around the foam droplets (Chandra et al. 2015). FS of F1 was recorded to be 80.12% while that of F2 was 75% (Table 1). High FS of F1 indicates the partially denatured protein molecules are susceptible to rapid changes in their structures. The developed WCF can, thus act as good whipping agent in various processed products as foaming properties are important for any flour if intended to be used as whipping or aerating agents (Adesina and Adeyeye 2016). At the same time, prolonged dry heating of water chestnuts prior to flour preparation possibly reduced cohesiveness of proteins and thus, lowered the foaming stability of F2.

Emulsifying activity

Emulsifying activity (EA) of flour is the measure of its ability to form a stable emulsion by interaction of polar and non-polar proteins with the oil droplets (Kaushal et al. 2012). EA of F1 was recorded to be 1.60% while that of F2 was 1.55% (Table 1). The difference in EA of F1 and F2 was non-significant.

Emulsion stability

Emulsion stability (ES) of flour reflects the ability of globular proteins to form rigid cohesive films around the fat droplets to prevent deformation under shear stress conditions (Kaushal et al. 2012). ES of F1 was recorded to be 50.65% while that of F2 was 49.03% (Table 1). Therefore, developed WCF can be explored well as a stabilizer in foods such as salad dressings, frozen confectionary, mayonnaise, desserts etc. The higher ES of F1 was possibly due to its higher percentage of very fine particles (46.10%). Timgren et al. (2013) reported that heat moisture treatment causes improvement in overall emulsion activity and stability by partial unfolding of globular proteins and exposure of non-polar amino acids, thus pre-conditioning of water chestnuts under high moisture-temperature conditions might be the reason for higher ES of F1. However, in case of F2, prolonged dry heating of water chestnuts prior to flour preparation might have changed the intermolecular interactions of proteins and starch molecules which renders these biomolecules less capable for surface adsorption. Similar findings have been reported by Timgren et al. (2013) for different starches.

Pasting properties

Pasting profile of flour samples is depicted in Table 2. Pasting profile is important to access the viscosity changes in flour suspensions. It has significant implications in product development and is known to affect the sensory and starch digestibility of final products.

Pasting temperature (PT) is the lowest temperature at which the starch granules become edible and gelatinize by

Table 2 Pasting properties of developed low GI water chestnut flour and commercially available water chestnut flour

Sample	Pasting temperature (°C)	Peak viscosity (cp)	Hold viscosity (cp)	Breakdown viscosity (cp)	Final viscosity (cp)	Set back viscosity (cp)
Developed low GI water chestnut flour (F1)	83.55 ± 0.27	2480 ± 0.40	1690 ± 0.23	790 ± 0.28	3233 ± 0.84	1543 ± 0.10
Commercially available water chestnut flour (F2)	87.35 ± 0.2	349 ± 0.3	325 ± 0.15	24 ± 0.31	481 ± 0.75	156 ± 0.15
t value	22.75*	2500*	1500*	1876*	3930*	2485*

* Significant calculated *t* value at $p < (0.01)$

forming a viscous paste (Shafi et al. 2016). Pasting temperature (PT) of F1 (83.55 °C) was significantly ($p < 0.01$) lower than that of F2 (87.35 °C) (Table 2). Low PT is desirable for starch-based materials intended to be used as thickening agents. Lower PT value of F1 (83.55 °C) implies lesser thermal energy is required to initiate starch gelatinization and paste formation due to weaker starch structure and intramolecular forces. At the same time, prolonged heating of water chestnuts in absence of moisture contributes to lower swelling ability and higher PT value (87.35 °C) of F2.

Peak viscosity (PV) is the maximum viscosity attained by the starch paste after irreversible swelling of intact starch granules takes place (Hussain et al. 2014). PV of F1 (2480 cp) was significantly ($p < 0.01$) higher than F2 (349 cp) (Table 2), which implies more availability of starch granules for hydration in case of F1. High peak viscosity of F1 may be ascribed to its high-water absorption as well as gelatinization of starch during pre-conditioning of water chestnuts under high moisture-temperature conditions prior to flour preparation which leads to more leaching of amylose molecules. At the same time, restricted swelling of F2 might have caused less leaching of amylose molecules which subsequently reduced its peak viscosity. High pasting temperature of F2 also confirms its low PV (Chen et al. 1998). Lower PV of F2 can also be justified by its more damaged starch molecules which are degraded easily and unable to develop viscous pastes (León et al. 2006).

Hold viscosity (HV) is attained when the flour suspension is subjected to high temperature under continuous shear conditions for a certain period of time (Hussain et al. 2014). F1 and F2 showed significantly ($p < 0.01$) different HV values of 1690 cp and 325 cp respectively (Table 2). Higher hold viscosity of FI can be attributed to its granular homogeneity which resulted in higher resistance to shearing force (Liu et al. 2016).

Breakdown viscosity (BDV) indicates the potential of flour suspension to withstand high temperature with continuous shear conditions which is an important parameter for stability of products during processing (Hussain et al. 2014). F1 showed a significantly ($p < 0.01$) higher BDV of 790 cp as compared to F2 (24 cp) (Table 2). Higher BDV of F1 sample indicates that swollen starch granules in developed WCF are highly susceptible to disintegration during cooking possibly because of weaker membranes, whereas lower BDV of F2 sample reflects its high textural rigidity.

Final viscosity (FV) is the ability of the flour suspension to form a viscous paste after being subjected to repeated heating and cooling cycles (Hamid et al. 2015). FV of F1 (3233 cp) was significantly ($p < 0.01$) higher than that of F2 (481 cp) (Table 2) which can be attributed to formation of amylose aggregates in developed WCF. High FV of F1 is evident from its high BDV which indicates that highly swollen starch granules have a tendency to form viscous and rigid gels upon cooling (Mir et al. 2014). At the same time, lower PV and BDV of F2 sample are consistent with its reduced FV possibly due to restricted swelling which hinders viscous paste formation in F2.

After the starch paste attains peak viscosity, cooling of such suspensions leads to molecular reassociation which is measured by setback viscosity (SBV) (Hussain et al. 2014). F1 showed SBV of 1543 cp which was significantly ($p < 0.01$) higher than that of F2 (156 cp) (Table 2). Higher SBV of F1 sample can be correlated with the higher tendency of amylose-amylopectin realignments to form retrograded resistant starch due to high moisture-temperature pre-conditioning of water chestnuts prior to flour preparation. Higher SBV thus reflects that F1 is more susceptible to amylose reassociation and has good gel formation ability than F2. Overall, findings of pasting properties observed in the present study are in accordance with the findings reported by Wani et al. (2017) in sweet chestnut flour.

Thermal properties

DSC was used to evaluate and compare the gelatinization behavior of F1 with F2. Thermal properties determined differed significantly ($p < 0.01$) in both the flour samples (Table 3).

In F1, the T_o, T_p and T_e were recorded as 50.33 °C, 76.77 °C and 87.13 °C while in case of F2, they were

Table 3 Thermal properties of developed low GI water chestnut flour and commercially available water chestnut flour

Sample	Onset temperature (°C)	Peak temperature (°C)	Conclusion temperature (°C)	Enthalpy of gelatinization (J/g)	Endothermic Peak width (°C)	Gelatinization temperature range (°C)	Peak height index
Developed low GI water chestnut flour (F1)	50.33 ± 0.37	76.77 ± 0.26	87.13 ± 0.22	7.02 ± 0.33	36.8 ± 0.20	52.88 ± 0.29	0.26 ± 0.21
Commercially available water chestnut flour (F2)	60.0 ± 0.43	250.29 ± 0.7	292.41 ± 1.05	5.54 ± 0.67	232.41 ± 0.33	380.68 ± 1.45	0.030 ± 0.01
t value	91.81*	1502*	1611*	18.81*	933.36*	2022.2*	64.06*

*Significant calculated *t* value at $p < (0.01)$

recorded as 60.0 °C, 250.29 °C and 292.41 °C respectively. The lower T_o, T_p and T_e values of F1 suggests that low temperature is required to gelatinize the starch in F1 than F2. This is due to more water absorption ability of F1, since the water acts as a plasticizer during gelatinization. Lower T_o, T_p and T_e values of F1 can also be justified by interlinking of amylose molecules within amylopectin branches which disrupts the crystalline regions and thus leads to lower gelatinization transition temperatures. On the other hand, prolonged dry heating of water chestnuts prior to flour preparation in F2 possibly leads to dehydration and higher gelatinization transition temperatures. Higher coarse particle size percentage in F2 could be another reason for its higher gelatinization temperature compared to F1. Lan et al. (2008) reported that T_o, T_p and T_e usually depends upon the amorphous and crystalline regions in the starch, their orientation and chain length.

Enthalpy of gelatinization (ΔH_{gel}) reflects the amount of energy required to melt and gelatinize the amorphous regions of the starch (Coral et al. 2009). ΔH_{gel} recorded for F1 (7.02 J/g) was significantly ($p < 0.01$) higher than that of F2 (5.54 J/g) (Table 3). The reason behind higher ΔH_{gel} of F1 was the pre-gelatinization of starch due to pre-conditioning which leads to less melting of double helical regions within the starch granules. At the same time, lower ΔH_{gel} value of F2 demonstrate its excessive structural breakdown due to prolonged dry heating of water chestnuts prior to flour preparation.

Endothermic peak width of F1 and F2 were recorded as 10.36 °C and 42.12 °C respectively (Table 3). Lower endothermic peak width of F1 reflects its perfect crystalline regions compared to F2. Gelatinization temperature range (R) of F1 (52.88 °C) was significantly ($p < 0.01$) lower than that F2 (380.68 °C) (Table 3). Gelatinization temperature range reflects the homogeneity of crystallites in the starch granular structure. Higher GT of F2 is well correlated with lower peak viscosity of F2 than F1. Peak height index of F1 (0.26) was significantly ($p < 0.01$) higher than F2 (0.03). High PHI of F1 was possibly due to lesser percentage of coarse particles and higher percentage of fine particles in F1 than F2. Similar findings were reported by Chen et al. (1998) for different starches.

ATR-FTIR analysis

ATR-FTIR analysis of the samples was done in order to characterize the chemical groups/bonds associated with specific structural organization of starch granules. FTIR spectrum of F1 is depicted in Fig. 1a and that of F2 is depicted in Fig. 2b.

Both the samples depicted absorption peaks in the range of 3400–800 cm^{-1} which are characteristic to starch structures. The peaks from 3272 to 3198 cm^{-1} in both the samples correspond to scissoring vibrations and stretching of poly-OH groups. A peak at 3428 cm^{-1} observed in F2 only indicates presence of strong intermolecular hydrogen bonding due to cross linking of fragmented lower molecular starch molecules. The band intensity at 3279 cm^{-1} in case of F1 decreased to 3266 cm^{-1} for commercial water chestnut flour which suggests a strong interlinking of hydrogen bonds with covalent bonds. Sharp band at 2930 cm^{-1} was shifted to 2927 cm^{-1} in case of F2 which is attributed to asymmetric stretching of C−H bonds. Various overlapping peaks observed in Fig. 1b between 2042 and 1965 cm^{-1} were possibly due to structural changes in amylose and amylopectin leading to vibrations in OH and CH bonding. Peaks at 1409, 1340, 1242 cm^{-1} in F1 sample corresponds to H−CH, C−O−H and CH_2OH bending vibrations. Absorption bands displayed between 852 and 929 cm^{-1} in F1 and F2 correspond to vibrations of α1,4 glycosidic bands in glucopyranosyl rings. The increase in band at 991 cm^{-1} in case of F1 to 993 cm^{-1} in case of F2 suggests gelatinization of F1 sample due to pre-conditioning prior to flour preparation which in turn might have led to variations in α-1,4 and α-1,6 glycosidic linkages. The band ratio of 1047/1022 cm^{-1} was calculated to be 0.89 for F1 and 0.82 for F2 which confirms the presence of smooth, compact and packed helices within the

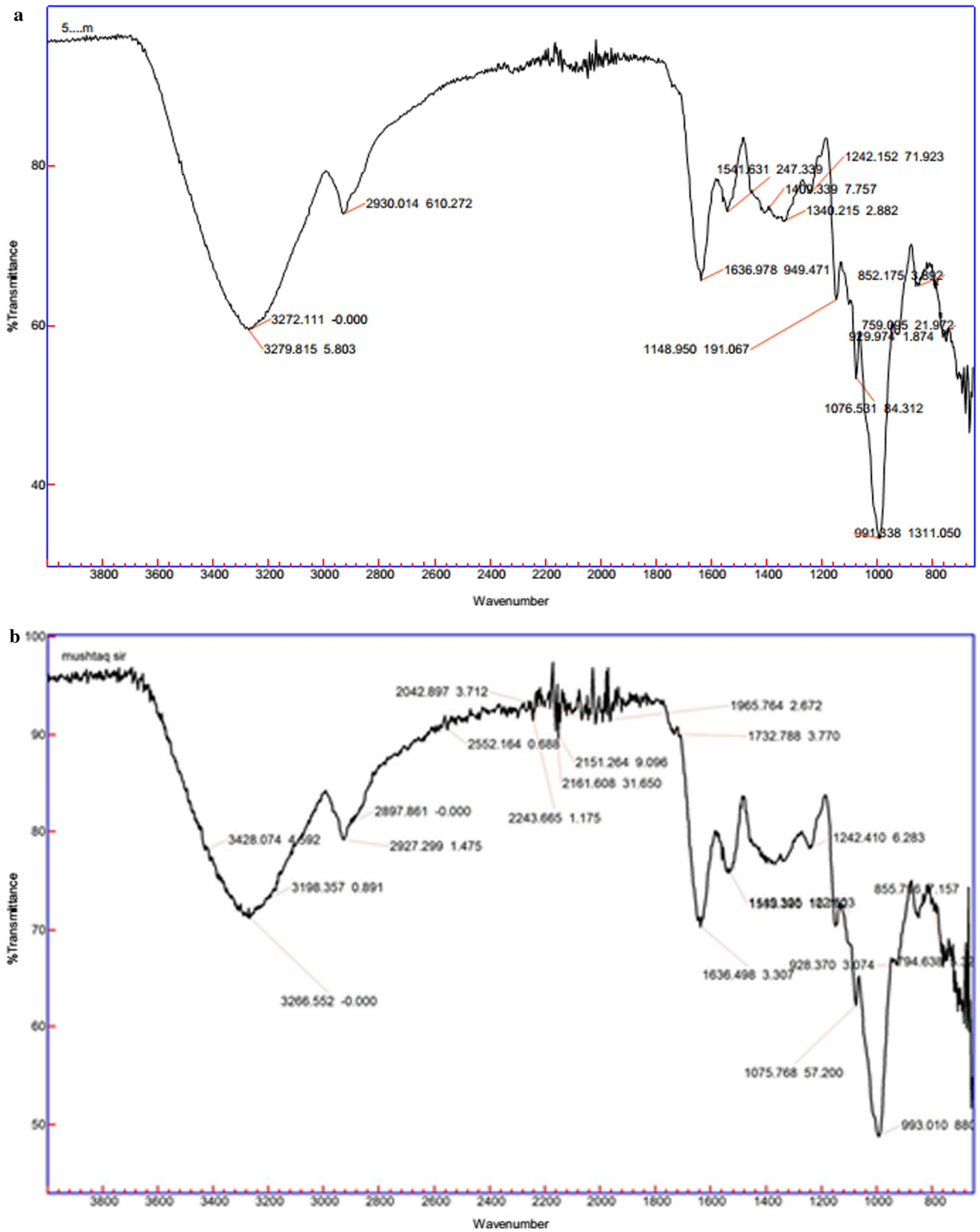

Fig. 1 ATR-FTIR spectrum of **a** developed low GI water chestnut flour (F1) and **b** commercially available water chestnut flour (F2)

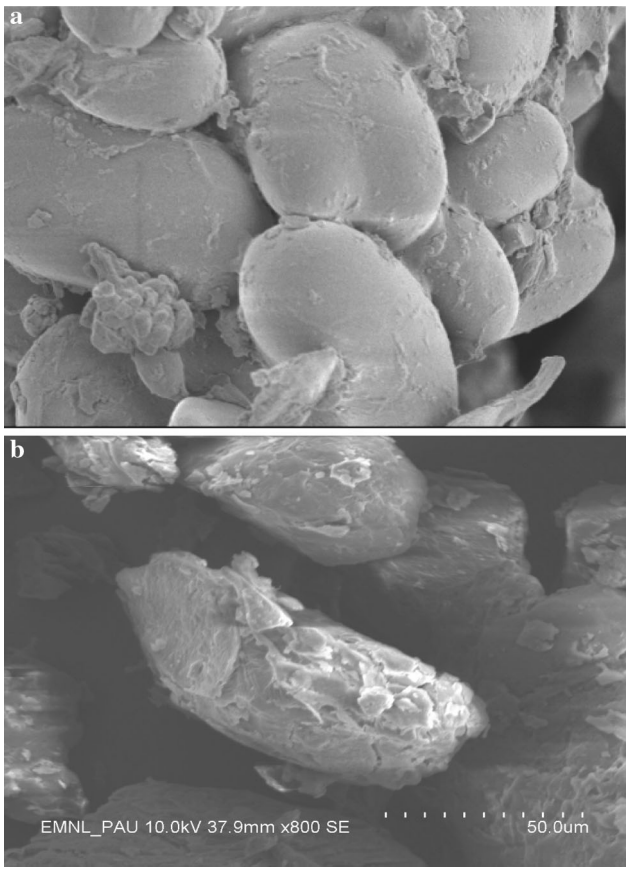

Fig. 2 SEM images of **a** developed low GI water chestnut flour (F1) and **b** commercially available water chestnut flour (F2)

crystalline regions of starch in F1 sample. Similar results were reported in potato starch by Soest et al. (1995).

Scanning electron microscopy

Scanning electron micrograph of F1 is depicted in Fig. 2a and that of F2 is depicted in Fig. 2b. The micrograph of F1 depicted round and oval shaped starch granules with smooth surfaces (Fig. 2a). The micrograph (Fig. 2b) indicates that starch granules in F2 were somewhat irregular and fragmented with rough surface. Although, the gelatinization during pre-conditioning destroys the organized starch structure, but retrogradation followed after gelatinization might have facilitated the fusion of degraded starch molecules which leads to formation of compact structures with smooth surfaces in case of F1. Changes in the morphology of starch granules are attributed to extent of gelatinization and retrogradation (Ashwar et al. 2016). Rough granular structures seen in Fig. 2b indicated that commercially available WCF flour was highly damaged due to prolonged dry heating prior to flour preparation. The fissures on the surface of granules in Fig. 2b indicates the presence of remnants of fragmented starch granules due to structural breakdown in F2.

Conclusion

The findings of the present study revealed that low GI WCF obtained after subjecting water chestnuts to optimized conditions of pre-conditioning has much better potential in food processing and product development than commercially available water chestnut flour obtained after subjecting water chestnuts to prolonged dry heating. The hypothesis was validated by studying the functional, pasting, thermal and structural properties of these flour samples. The functional behavior of F1 was significantly improved in terms of water absorption capacity, swelling capacity, least gelation concentration, foaming capacity, foaming stability and emulsifying stability as compared to F2 which demonstrates its possible use as a food thickening, foaming and stabilizing agent. Microstructural studies further verified the findings and showed improved granular homogeneity and ordered crystalline packing in F1 as compared to F2. Owing to well demonstrated functional, viscous, thermal and structural properties, it can be concluded that low GI water chestnut flour, is an ideal candidate to be explored in food formulations for development of different products like bakery, ready to eat snacks, weaning foods, confectionary etc. for diabetic patients and health conscious people. However, further studies on product development from developed low GI water chestnut flour and efficacy studies of developed products among diabetic patients as well as consumer acceptability tests are needed if commercial utilization of developed flour is desired.

Compliance with ethical standards

Conflict of interest The authors have declared no conflict of interest to this work.

References

AACC (2000) Approved Methods of the AACC International. American Association of Cereal Chemists, Methods, 10th edn. St Paul, MN

Adebowalea KO, Olu-Owolabi BI, Olayinka OO, Lawal OS (2005) Effect of heat moisture treatment and annealing on physicochemical properties of red sorghum starch. Afr J of Biotechnol 4(9).

Adesin AJ, Adeyeye EI (2016) Influence of Salts at Different concentrations on the Foaming Properties, Water and Oil Absorption Capacities of Raw and Treated Treculia africana Seeds Flour. Int J Innov Sci Eng Technol 3(12):73–88

Asghari AK, Norton I, Mills T, Sadd P, Spyropoulos F (2016) Interfacial and foaming characterisation of mixed protein-starch

particle systems for food-foam applications. Food Hydrocoll 53:311–319
Ashwar BA, Gani A, Wani IA, Shah A, Masoodi FA, Saxena DC (2016) Production of resistant starch from rice by dual autoclaving-retrogradation treatment: Invitro digestibility, thermal and structural characterization. Food Hydrocoll 56:108–117
Awolu OO (2017) Optimization of the functional characteristics, pasting and rheological properties of pearl millet-based composite flour. Heliyon 3(2):00240
Batey IL, Gras PW, Curtin BM (1997) Contribution of the chemical structure of wheat starch to Japanese noodle quality. J Sci Food Agric 74:503–508
Brand-Miller J, Hayne S, Petocz P, Colagiuri S (2003) Low–glycemic index diets in the management of diabetes: a meta-analysis of randomized controlled trials. Diabetes Care 26(8):2261–2267
Chandra S, Singh S, Kumari D (2015) Evaluation of functional properties of composite flours and sensorial attributes of composite flour biscuits. J Food Sci Technol 52(6):3681–3688
Chen YY, McPherson AE, Radosavljevic M, Lee V, Wong KS, Jane J (1998) Effects of starch chemical structures on gelatinization and pasting properties. Żywność Technologia Jakość Suplement 5(4):63–71
Coffman AW, Garcia VV (1977) Functional properties and amino acid contents of protein isolate from mung bean flour. J Food Technol 12:473–484
Coral DF, Pineda-Gómez P, Rosales-Rivera A, Rodriguez-Garcia ME (2009) Determination of the gelatinization temperature of starch presented in maize flours. In Journal of Physics: Conference Series. 167(1): 012057, IOP Publishing.
Falade KO, Okafor CA (2015) Physical, functional, and pasting properties of flours from corms of two Cocoyam (Colocasia esculenta and Xanthosoma sagittifolium) cultivars. J Food Sci Technol 52(6):3440–3448
Gani A, Haq SS, Masoodi FA, Broadway AA, Gani A (2010) Physico-chemical, morphological and pasting properties of starches extracted from water chestnuts (Trapa natans) from three lakes of Kashmir. India Braz Arch Biol Technol 53(3):731–740
Goni I, Garcia AA, Saura CF (1997) A Starch Hydrolysis Procedure to Estimate Glycemic Index. Nutr Res 17:427–437
Hamid S, Muzzafar S, Wani IA, Masoodi FA (2015) Physicochemical and functional properties of two cowpea cultivars grown in temperate Indian climate. Cogent Food Agric 1(1):1099418
Hasjim J, Li E, Dhital S (2012) Milling of rice grains: The roles of starch structures in the solubility and swelling properties of rice flour. Starch-Stärke 64(8):631–645
Hussain SZ, Beigh MA, Naseer B, Amin T, Naik HR (2019) Characteristics of resistant starch in water chestnut flour as improved by preconditioning process. Int J Food Prop 22(1):449–461
Hussain SZ, Singh B, Naik HR (2014) Viscous and thermal behaviour of vitamin A and iron-fortified reconstituted rice. Int J Food Sci Technol 49(5):1324–1329
Kaushal P, Kumar V, Sharma HK (2012) Comparative study of physicochemical, functional, anti-nutritional and pasting properties of taro (Colocasia Esculenta), rice (Oryza sativa), pegion pea (Cajanus Cajan) flour and their blends. LWT-Food Sci Technol 48:59–68
Lan H, Hoover R, Jayakody L, Liu Q, Donner E, Baga M, Asare EK, Hucl P, Chibbar RN (2008) Impact of annealing on the molecular structure and physicochemical properties of normal, waxy and high amylose bread wheat starches. Food Chem 111:663–675
León AE, Barrera GN, Pérez GT, Ribotta PD, Rosell CM (2006) Effect of damaged starch levels on flour-thermal behaviour and bread staling. Eur Food Res Technol 224(2):187–192
Liu YF, Laohasongkram K, Chaiwanichsiri S (2016) Effects of Heat-Moisture Treatment on Molecular Interactions and Physico-chemical Properties of Tapioca Starch. MOJ Food process Technol 3(3):00072
Mir NA, Gul K, Riar CS (2014) Physicochemical, pasting and thermal properties of water chestnut flours: A comparative analysis of two geographic sources. J Food Process Preserv 39(6):1407–1413
Narayana K, Narasinga RMS (1982) Functional properties of raw and heat processed winged bean flour. J Food Sci 47:1534–1538
Olu-Owolab BI, Afolabi TA, Adebowale KO (2011) Pasting, thermal, hydration, and functional properties of annealed and heat-moisture treated starch of sword bean (Canavalia gladiata). Int J Food Prop 14(1):157–174
Rafiq SI, Jan K, Singh S, Saxena DC (2015) Physicochemical, pasting, rheological, thermal and morphological properties of horse chestnut starch. J Food Sci Technol 52(9):5651–5660
Rani B, Verma D, Bhati I, Chharang H, Maheshwari RK (2016) Health Benefits of Scrumptious Water Chestnuts/Water Caltrop (Trapa natans L.). Int Arch App Sci Technol 7(3): 32–36.
Sakhare SD, Inamdar AA, Soumya C, Indrani D, Rao GV (2014) Effect of flour particle size on microstructural, rheological and physico-sensory characteristics of bread and south Indian parotta. J Food Sci Technol 51(12):4108–4113
Salmeron J, Manson JE, Stampfer MJ, Colditz GA, Wing AL, Willett WC (1997) Dietary fiber, glycemic load, and risk of non—insulin-dependent diabetes mellitus in women. JAMA 277(6):472–477
Shad MA, Nawaz H, Hussain M, Yousuf B (2011) Proximate composition and functional properties of rhizomes of lotus (Nelumbo nucifera) from Punjab. Pakistan Pak J Bot 43(2):895–904
Shafi M, Baba WN, Masoodi FA, Bazaz R (2016) Wheat-water chestnut flour blends: effect of baking on antioxidant properties of cookies. J Food Sci Technol 53(12):4278–4288
Shah A, Masoodi FA, Gani A, Ashwar BA (2016) Geometrical, functional, thermal, and structural properties of oat varieties from temperate region of India. J Food Sci Technol 53(4):1856–1866
Soest VJJ, Tournois H, de Wit D, Vliegenthart JF (1995) Short-range structure in (partially) crystalline potato starch determined with attenuated total reflectance Fourier-transform IR spectroscopy. Carbohydr Res 279:201–214
Sosulski FW, Garatt MO, Slinkard AE (1976) Functional properties of ten legume flours. Int J Food Sci Technol 9:66–69
Timgren A, Rayner M, Dejmek P, Marku D, Sjöö M (2013) Emulsion stabilizing capacity of intact starch granules modified by heat treatment or octenyl succinic anhydride. Food Sci Nutr 1(2):157–171
Wani IA, Hamid H, Hamdani AM, Gani A, Ashwar BA (2017) Physico-chemical, rheological and antioxidant properties of sweet chestnut (Castanea sativa Mill.) as affected by pan and microwave roasting. J Adv Res 8(4): 399–405.
Yadav DN, Kaur J, Anand T, Singh AK (2012) Storage stability and pasting properties of hydrothermally treated pearl millet flour. Int J Food Sci Technol 47(12):2532–2537
Yasumatsu K, Sawada K, Maritaka S, Toda J, Wada T, Ishi K (1972) Whipping and emulsifying properties of soy bean products. Agric Biol Chemi 36:719–727