

Effect of popping on physicochemical, technological, antioxidant, and microstructural properties of makhana seed

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Abstract

This study was carried out to investigate the effects of popping on properties of makhana (*Euryale ferox*) seed. The popping was done by initial roasting 270–280°C, tempering (48 hr), final roasting 260–270°C. Popping changed the matrix of makhana from loose crystalline to highly porous structure with increased ($p < .05$) water (376.24%) and oil absorption capacities (317.16%). A significant ($p < .05$) increase in the protein and dietary fiber content, whereas a decrease ($p < .05$) in the amylose, amylopectin content was found. The mineral profile of makhana was not affected during popping, however, a reduction ($p < .05$) in the iron content was noticed. Phytic acid and tannins were not detected in the popped makhana. The bran obtained during popping showed higher ($p < .05$) DPPH radical scavenging activity (76.3%). The bran showed higher dietary fiber (83.3%), phenolic (49.0 mg/g), and flavonoid (192.2 mg/100 g) contents.

Practical application: Popping of makhana enhanced protein content and dietary fiber content. Carbohydrate, starch, amylose, and amylopectin also decreased during popping. The majority of beneficial phytochemicals were concentrated in the bran which was a by-product of popping process. Therefore, the bran may be considered for its application in food products, whereas the raw kernel may be used for incorporation in food products to enhance mineral and amino acid profile.

1 | INTRODUCTION

Makhana (*Euryale ferox*) seed, commonly known as gorgon nut or fox nut, is an aquatic crop cultivated in stagnant water bodies like ponds, land depressions, lakes, swamps, and ditches. It belongs to water lily family, *Nymphaeaceae*, which has cultivation history exceeding more than 2000 years in Asia. This black seed is round in shape with a diameter ranging from 4.5 to 14.5 mm and having hard shell as an outer most structure (Jha & Prasad, 1993).

The edible part of the seed is the starchy kernel, which cannot be separated easily from its hard shell. Therefore, the raw makhana seeds are generally processed (popping) to obtain popped makhana for edible use. The consumption of popped makhana has been increased worldwide in the past few years due to its health benefits, lightness, and crispiness (APEDA, 2017). Popped makhana is highly nutritious

due to its negligible fat content, high quality protein, and presence of various health promoting bioactive compounds. The high amino acid index (89%–93%) and better arginine + lysine/proline ratio (4.7–7.6) make it superior in comparison to other cereals for achieving the amino acid requirements of the human body (Jha, Barat, & Jha, 1991).

The popping is done by initial roasting (270–280°C), tempering (48 hr), final roasting (260–270°C) and breaking of the hard shell for expansion of seed in hot condition (Jha & Prasad, 1996). In the initial roasting of raw seeds, the gelatinization, and retrogradation of starch molecules take place. The initial roasting brings characteristic changes and results in expansion of kernels (popping) during the second roasting. Popping process of makhana seeds is entirely different from rice and cocoa beans. The hull of paddy is removed after parboiling and then puffing is done through roasting (Mir, Bosco, Shah, & Mir, 2016). In cocoa beans, roasting and puffing is done in a single operation (Hu

et al., 2016). Contrary to this, the initial roasting of makhana is done along with hull, and popping is performed by mechanical breaking of the hull after completion of final roasting. Therefore, the roasting and subsequent popping operations of makhana seed are expected to affect the physicochemical properties in a different manner. Furthermore, the research related to makhana seed at present are mainly focused on isolation and characterization of chemical constituents present in the kernel and their starches (Song et al., 2011; Zhao et al., 2016). A little information is available about the changes in the structural, chemical constituents, and other properties during popping of makhana seed.

Therefore, this study was undertaken to investigate the changes in the various properties occurring during the popping of makhana seeds. Physicochemical (proximate analysis, mineral profile, amino acid profile, total starch, amylose content, amylopectin content, total phenolic content, total flavonoid content, tannins, and phytic acid content), technological (color attributes, water, and oil absorption capacities), antioxidants (DPPH radical scavenging activity), and microstructural (scanning electron microscopy) properties of raw kernel, popped makhana seed, and bran were analyzed during this study.

2 | MATERIALS AND METHODS

2.1 | Materials

Raw makhana seeds (variety *Swarn Vaidehi*) were procured from Krishi Vigyan Kendra, Dhamtari, Chhattisgarh, India. The seeds were cleaned and kept in water (25–30°C) under the submerged condition to prevent the seed germination. The seeds were taken out from the water prior to experiments.

2.2 | Chemicals

Sodium hydroxide, aluminum chloride, sodium nitrite, sodium carbonate, and Folin-Ciocalteu reagent were purchased from Sisco Research Laboratory Pvt. Ltd. (India). Toluene, methanol, hydrochloric acid, and isopropyl alcohol were supplied by Thermo Fisher Scientific India Pvt. Ltd. (India). Gallic acid and rutin were procured from SD Fine-chem Limited (India). 2, 2-diphenyl-1-picrylhydrazyl (DPPH) was procured from Sigma-Aldrich Co. LLC., India. All the chemicals used in this study for quality analysis were of AR grade.

2.3 | Sample preparation and makhana popping process

About 20 kg raw makhana seeds were taken out from the water and spread in a single layer for drying under sun till the moisture

content of seeds reached to about $30 \pm 1\%$. The seed was decorticated manually and kernel powder was collected. The powder was packed into 75 μm thickness low density polyethylene (LDPE) bags, sealed, and stored in a refrigerator at 4°C. For popping, the seeds were graded into seven sizes according to their diameter (<7, 7, 8, 9, 10, 11, and 12 mm diameter). The seeds from each grade were roasted separately in 1 kg batches onto mild steel pan by the traditional roasting method (270–280°C) (Jha & Prasad, 1996). After completion of roasting, the seeds were transferred into jute sacks and kept under shade for 48 hr. Thereafter, the seeds were popped using a makhana popping machine developed by ICAR-Central Institute of Post-Harvest Engineering and Technology, Ludhiana (India). The machine was operated at 265°C (260–270°C) and roasting was done for 3–5 min. The decorticator part of machine broke the seed coat of roasted seeds to form the popped makhana. The popped makhana (≥ 13 mm diameter) and bran were separated and packed into 75 μm thickness LDPE bags for further studies.

2.4 | Proximate analysis

Moisture, crude protein, fat, total minerals, soluble and insoluble dietary fibers, and total fiber contents of the samples were determined using the AOAC methods (AOAC, 2016). Total carbohydrate content was determined using difference method.

2.5 | Color attributes

The measurement of CIE (*International Commission on Illumination*) color values (lightness, L^* ; redness, a^* ; yellowness, b^*) of makhana samples was done using hunter colorimeter model 45/0-L mini scan XE PLUS (Hunter Associates Labs, Reston, VA, USA). Hue angle (h_{ab}) and chroma (C^*) values were determined using Equations 1 and 2.

$$\text{Hue angle: } h_{ab} = \tan^{-1} \left(\frac{b^*}{a^*} \right) \quad (1)$$

$$\text{Chroma: } C^* = \sqrt{(a^*)^2 + (b^*)^2} \quad (2)$$

2.6 | Water absorption capacity

Sample (1 g) was dispersed in 15 ml distilled water with continuous stirring for 30 s which was allowed to stand for 30 min in a water bath at 30°C. It was centrifuged at $1107 \times g$ for 15 min and the supernatant was decanted. The weight of tubes with the pellet was taken. The results were expressed as g water/g dry matter.

$$\text{Water absorption capacity (g/g)} = \frac{(\text{weight of sample with tube after removal of water} - \text{weight of tube}) - \text{sample weight}}{\text{sample weight}} \quad (3)$$

2.7 | Oil absorption capacity

Oil absorption capacity (OAC) was measured according to the method reported by Jogihalli, Singh, and Sharanagat (2017) with slight modifications. Sample (1 g) was mixed with 15 ml refined soybean oil and allowed to stand for 30 min. This was centrifuged at $1107 \times g$ for 25 min. The oil, which separated due to centrifugation, was drained by holding the tubes in the inverted position for 25 min and then weighted. The results were expressed as g oil/g dry matter.

2.8 | Minerals profile

The mineral profile (P, K, Ca, Mg, Na, Mn, Zn, Cu, Fe, Mo, Co, Se, I, S, and Cl) of samples was determined using the AOAC methods (AOAC, 2016). In brief, the samples were digested with $\text{HNO}_3/\text{H}_2\text{O}_2$ and the extract were re-dissolved in double distilled water and subsequently analysed by ICP-MS (Agilent 7,700 series ICP-MS, Agilent Technologies Inc., USA). The contents were calculated from the standard curve for each element and expressed as mg/100 g dry matter.

2.9 | Amino acid profile

The amino acid composition was determined using the acid hydrolysis method (Mattila, Salo-Väänänen, Könkö, Aro, & Jalava, 2002). In brief, 1 g of sample was hydrolyzed with 6 N HCl at 110°C for 24 hr. Afterward, dilution was made by addition of 6 N NaOH to adjust pH to 2.2. Derivatization was done by taking extract in injection vial containing borate buffer to make up volume to 200 ml and vortexed briefly. AccQ Tag Ultra reagent was added, vortexed for a few seconds and incubated in water bath for 10 min at 55°C . All the samples were transferred to high-performance liquid chromatography (Agilent Technologies, Inc., USA, 1,200 series) for determination of amino acid profile. The tryptophan amino acid was not determined in this study. The concentration of different amino acids was calculated using Equation 4:

$$\text{Concentration (mg/100g)} = \frac{\text{concentration of standard} \times \text{area of sample} \times \text{dilution factor}}{\text{area of standard} \times \text{weight of sample}} \quad (4)$$

2.10 | Total starch

The total starch content was determined by the method reported by Jagadeesh et al. (2007). The alcohol-insoluble matters of the samples were hydrolyzed by 52% perchloric acid, centrifuged at 3,000 rpm for 10, 15, 20, 25, and 30 min and the supernatant was collected and pooled. This process was repeated thrice. The supernatant containing the hydrolyzed starch was made up to a known volume. The transmittance of the D-glucose standard solutions and

sample against reagent blank was read at 540 nm using UV-VIS spectrophotometer (M/s LabIndia Analytical Instruments Pvt. Ltd., India, model UV 3,200) This method gives reducing sugar volume and the total starch percentage was calculated by multiplying the reducing sugar values by a factor 0.9.

2.11 | Amylose and amylopectin content

Amylose content was determined using the method described by Juliano (1985). The sample (100 mg) was mixed with 1 ml of 95% alcohol and 10 ml of 1 N NaOH followed by heating in a water bath at 85°C till the sample was dispersed. The dispersion was allowed to stand overnight at room temperature and then the dispersion was diluted to 100 ml and shaken for 5 min. Thereafter, phenolphthalein indicator and 50 ml distilled water was added in 5 ml diluted sample. Then 2 ml of iodine solution was added and volume was adjusted to 100 ml with boiled distilled water, mixed, and allowed to stand for 20 min. The absorbance was recorded at 620 nm using UV-VIS spectrophotometer. The amylose content was calculated using the standard curve of potato amylose (20–100 $\mu\text{g/ml}$). Amylopectin content of the samples was calculated after subtracting amylose content from total starch.

2.12 | Total phenolic content

Phenolic extraction of the samples was performed using the method reported by Mir et al. (2016) with slight modification. The powdered sample (500 mg) was extracted with 25 ml of 80% acetone followed by shaking for 2 hr at 30°C . The mixture was centrifuged at $3074 \times g$ for 30 min at 4°C in a refrigerated centrifuge. The supernatant was stored at -4°C for analysis of total phenolic content using Folin-Ciocalteu method (Dudonné, Vitrac, Coutière, Woillez, & Mérillon, 2009). The results were reported as gallic acid equivalents GAE/100 g dry matter using gallic acid as a standard.

2.13 | Total flavonoid content

Total flavonoid content was measured according to the method used by Lin and Tang (2007) with slight modifications. The sample (1 g) was extracted with methanol (50 ml) by refluxing for 6 hr. The extract was filtered and concentrated to about 10 ml under reduced pressure using a rotary evaporator (M/s Eppendorf AG, Concentrator Plus, Germany, Model 5,305). Then, 0.5 ml of extract was mixed with 1.5 ml of methanol, 0.1 ml of aluminum chloride solution, 0.1 ml of potassium acetate solution, 2.8 ml of distilled water, shaken on vortex shaker for 30 s, and incubated for 30 min at room temperature. The absorbance was recorded at 415 nm using UV-VIS spectrophotometer against blank without extract. The total flavonoid content was calculated using the

standard curve of rutin and expressed as mg rutin equivalent per 100 g dry matter.

2.14 | Tannins and phytic acid

Tannins were estimated using the method reported by Burns (1971). Tannin content was calculated using a calibration curve of catechin (0.2–0.6 mg) and expressed as mg equivalent of catechin per 100 g dry matter. The phytic acid was determined as per the method reported by Davies and Reid (2007). The phytic acid content was calculated using a calibration curve of sodium phytate (0.05–0.2 mg) and expressed as mg phytate per 100 g dry matter.

2.15 | DPPH radical scavenging activity

Free radical scavenging activity was measured using the 2,2-diphenyl-2-picryl-hydrazyl (DPPH) method (Dudonné et al., 2009). 0.1 ml of phenolic extract was mixed with 2.9 ml of DPPH solution (25 µg/ml of methanol). The solution was placed in the dark at 25°C for 30 min and reduction in the absorbance was recorded at 517 nm using UV-VIS spectrophotometer using methanol as blank. DPPH radical scavenging activity was calculated as:

$$\text{DPPH scavenging activity (\%)} = \frac{(\text{absorbance blank} - \text{absorbance sample})}{\text{absorbance blank}} \times 100 \quad (5)$$

2.16 | Microstructure

The structure of raw kernel, roasted kernel, and popped makhana was analyzed by scanning electron microscopy. The powdered samples were placed on SEM aluminum stubs using adhesive tape and a coating of palladium was applied. Samples were scanned by SEM (Model EVO 18, Carle Zeiss Microscopy GmbH, Germany) at 15 kV. Micrographs from different locations were taken at different magnifications and representative images were selected for further analysis.

2.17 | Statistical analysis

The samples were prepared in triplicate and all the analyses were done in triplicate. The mineral and amino acid profile analyses were conducted in duplicate. All the data obtained from different experiments were analyzed statistically using SPSS software version 20. The results were expressed as mean ± standard deviation. Statistical significance between the samples was determined by ANOVA followed by Duncan multiple range test.

3 | RESULTS AND DISCUSSION

3.1 | Proximate analysis

Proximate composition of the raw kernel, popped makhana, and bran on dry matter basis is presented in Table 1. The popped makhana contained significantly ($p < .05$) higher protein content than the raw kernel. The complex protein molecules present in the raw kernel might be converted into simpler form during heat treatment, therefore, the protein content increased in the popped makhana. Similar observation was reported for popping of kiwicha grain by Paucar-Menacho, Dueñas, Peñas, Frias, and Martínez-Villaluenga (2018).

The crude fat content was slightly higher in the raw kernel in comparison to the popped makhana though the difference was not significant ($p > .05$). Since, the popping was done by breaking the hull of roasted seeds. Therefore, the possibility of free fatty acids formation and/or oxidation of fat might be negligible. Moreover, the fat was mainly concentrated in the bran layer, which detached during popping process, and therefore, the fat content of popped makhana reduced slightly.

The total mineral content of popped makhana was slightly less than the raw seed, though the difference was not significant ($p > .05$). However, the total mineral content of bran was significantly ($p < .05$) higher than the raw and popped makhana. High mineral content of the bran in comparison to the endosperm is reported in the literature for other grains (Javed et al., 2012).

| Item (dry matter basis) | Raw kernel | Popped makhana | Bran |
|-----------------------------|---------------------------|---------------------------|---------------------------|
| Protein (%) | 9.15 ± 0.10 ^b | 11.03 ± 0.45 ^a | 11.25 ± 0.38 ^a |
| Fat (%) | 0.46 ± 0.04 ^b | 0.33 ± 0.12 ^b | 2.72 ± 0.24 ^a |
| Total minerals (%) | 0.45 ± 0.03 ^b | 0.38 ± 0.03 ^b | 2.43 ± 0.03 ^a |
| Total dietary fiber (%) | 2.55 ± 0.04 ^c | 3.26 ± 0.06 ^b | 77.70 ± 0.22 ^a |
| Soluble dietary fiber (%) | 1.36 ± 0.03 ^b | 2.83 ± 0.08 ^a | 2.68 ± 0.06 ^a |
| Insoluble dietary fiber (%) | 1.19 ± 0.02 ^b | 0.43 ± 0.04 ^c | 75.01 ± 0.10 ^a |
| Carbohydrates (%) | 87.52 ± 0.15 ^a | 84.87 ± 0.60 ^b | 5.90 ± 0.24 ^c |
| Starch (%) | 85.71 ± 0.33 ^a | 78.23 ± 0.34 ^b | 8.11 ± 0.13 ^c |
| Amylose (%) | 28.99 ± 0.13 ^a | 25.49 ± 0.37 ^b | 4.44 ± 0.63 ^c |
| Amylopectin (%) | 56.72 ± 0.34 ^a | 52.74 ± 0.37 ^b | 3.67 ± 0.69 ^c |

Note: Means ± SD in a row with different superscripts are significantly different ($p < .05$).

TABLE 1 Effect of popping on proximate composition and other chemical constituents of raw kernel, popped makhana, and bran

Popping resulted in significantly ($p < .05$) higher total dietary fiber (TDF) and soluble dietary fiber (SDF) content, whereas the insoluble dietary fiber (IDF) decreased significantly ($p < .05$) during popping. Furthermore, the bran contained very high amount of TDF (77.7%) out of which the IDF was 75%. It indicated that the bran is mainly composed of IDF and, therefore, it may be used as a rich source of fiber. Increase in the SDF and decrease in the IDF after roasting was also reported for barley (Bader UI Ain et al., 2019).

The carbohydrate content of popped makhana was significantly ($p < .05$) lower than the raw kernel. This decrease might be due to the increase in the protein and TDF content. Paucar-Menacho et al. (2018) also reported decreased carbohydrate content after the popping of kiwicha. The bran had very less carbohydrate content than the raw kernel, which might be due to a higher amount of the IDF content in the bran.

3.2 | Starch, amylose, and amylopectin content

Starch, amylose, and amylopectin contents of the raw kernel, popped makhana, and bran are presented in Table 1. Starch is the major chemical component of makhana kernel and popping resulted in a significant ($p < .05$) decrease in the starch content. This decrease might be due to the degradation of starch into smaller molecules, such as glucose, maltose, etc., which also resulted in an increase of total sugar content (Huang et al., 2018). Amylose and amylopectin content also decreased significantly ($p < .05$) during popping. A similar observation during the popping of sorghum was reported by Nathakattur Saravanabavan, Manchanahally Shivanna, and Bhattacharya (2013). Amylose content plays an important role in puffing because of more pressure build up during the thermal treatment, which results in more expansion of the endosperm. This phenomenon was reported for rice having amylose content of more than 25% (Kamaraddi & Prakash, 2015).

3.3 | Color attributes

The color attributes of makhana kernel, popped makhana, and bran are presented in Table 2. The lightness (L^*) value of popped makhana decreased significantly ($p < .05$) as compared to the raw kernel. Moreover, the redness (a^*), yellowness (b^*), hue, and chroma increased significantly ($p < .05$) after popping. Various studies reported the change in the color during roasting and popping of grains, nuts, beans, seeds, and kernels (Chung, Kim, Moon, & Youn, 2014; Devi & Das, 2017; Kahyaoglu & Kaya, 2006; Nizamlioglu & Nas, 2016; Özdemir & Devres, 2000; Shi, Sandeep, Davis, Sanders, & Dean, 2017). The consumer acceptability of final product is highly correlated with the color attributes which are specific for different types of food products (Spence, 2015). However, in most of the grains and seeds, a high temperature during roasting and popping process negatively affects the color and appearance of final product (Pittia, Dalla Rosa, & Lerici, 2001; Saklar, Ungan, & Katnas, 1999). The nonenzymatic browning

TABLE 2 Effect of popping on color attributes of raw kernel, popped makhana, and bran

| Attributes | Raw kernel | Popped makhana | Bran |
|------------|---------------------------|---------------------------|---------------------------|
| L^* | 92.53 ± 0.08 ^a | 83.21 ± 0.09 ^b | 31.23 ± 0.13 ^c |
| a^* | 0.14 ± 0.19 ^c | 1.26 ± 0.14 ^b | 5.58 ± 0.73 ^a |
| b^* | 5.86 ± 0.35 ^b | 6.92 ± 0.07 ^a | 6.77 ± 0.73 ^a |
| h_{ab} | 0.76 ± 1.54 ^c | 1.39 ± 0.02 ^a | 0.88 ± 0.10 ^b |
| C^* | 5.86 ± 0.35 ^c | 7.03 ± 0.05 ^b | 8.81 ± 0.50 ^a |

Note: Means ± SD in a row with different superscripts are significantly different ($p < .05$).

h_{ab} -hue angle values are in radian.

C^* -chroma values.

associated with degradation, oxidation, Maillard reaction, and caramelization of sugars is mainly responsible for color changes during roasting and high-temperature processing of these food products (Friedman, 1996).

3.4 | Water absorption capacity (WAC) and oil absorption capacity (OAC)

The water and oil absorption capacities of raw makhana, popped makhana, and bran are presented in Table 3. The popped makhana showed ($p < .05$) five times higher WAC in comparison to the raw kernel. The gelatinization of starch and formation of porous structure during popping resulted into an increase in the surface area (Figure 1) which might be the reason behind higher WAC of popped makhana. Similar increase in the WAC was observed during popping of other grains (Jogihalli et al., 2017; Mishra, Joshi, & Mohapatra, 2015). The bran had significantly ($p < .05$) lower WAC than popped makhana and significantly ($p < .05$) higher WAC than the raw kernel, which might be attributed to the higher insoluble fiber content of bran (Table 1).

OAC of the raw kernel was 0.84 g oil/g dry matter and popping of makhana increased the OAC by 4 times. Similar findings were also reported in the literature studying the popping of various horse gram (Sreerama, Sasikala, & Pratape, 2008) and chickpea (Jogihalli et al., 2017). The porous structure of popped makhana and physical entrapment of oil to the nonpolar side chain of the protein might be the reason behind higher OAC of popped makhana. The higher protein content, hydrophobic amino acid, and conformational structures also contributed to the higher OAC of popped makhana (Sreerama et al., 2008). The OAC of bran was two times to that of the raw kernel, which might be attributed to the higher protein content and porous structure.

3.5 | Mineral profile

The mineral profile (Ca, Na, K, Mg, P, Fe, Mn, Mo, Zn, Cu, and Co) of raw makhana, popped makhana, and bran is presented in Table 4.

| Properties | Raw kernel | Popped makhana | Bran |
|---------------------------------|--------------------------|--------------------------|--------------------------|
| Water absorption capacity (g/g) | 1.03 ± 0.01 ^c | 4.91 ± 0.08 ^a | 2.49 ± 0.08 ^b |
| Oil absorption capacity (g/g) | 0.84 ± 0.03 ^c | 3.50 ± 0.09 ^a | 1.78 ± 0.01 ^b |

Note: Means ± SD in a row with different superscripts are significantly different ($p < .05$).

TABLE 4 Effect of popping on mineral content of raw kernel, popped makhana, and bran

| Minerals (mg/100 g, dry matter basis) | Raw kernel | Popped makhana | Bran |
|---------------------------------------|------------|----------------|--------|
| Calcium | 14.50 | 20.94 | 133.00 |
| Sodium | 2.92 | 4.06 | 10.74 |
| Potassium | 50.25 | 48.39 | 85.60 |
| Magnesium | 13.83 | 12.71 | 79.96 |
| Phosphorus | 132.53 | 124.01 | 213.84 |
| Iron | 5.73 | 2.67 | 114.06 |
| Manganese | 1.30 | 1.24 | 4.76 |
| Molybdenum | 0.03 | 0.04 | 0.08 |
| Zinc | 1.50 | 1.04 | 8.42 |
| Copper | 0.71 | 0.76 | 6.42 |
| Cobalt | ND | ND | 0.07 |

ND, not detected.

The mineral contents were higher ($p < .05$) in bran in comparison to raw and popped makhana. The mineral profile of raw makhana and popped makhana was similar except for Ca and Fe contents. Higher Ca content in popped makhana indicated that the Ca might be evenly distributed in the kernel. The lower Fe content in popped makhana indicated that the Fe was mainly present in the bran. Similar mineral distribution was reported during puffing of amaranth seed (Murakami, Yutani, Yamano, Iyota, & Konishi, 2014). Micro minerals such as selenium, iodine, sulfur, and chloride were not detected in makhana except a very small amount of cobalt was found in the bran (Table 4).

3.6 | Amino acid profile

The amino acid content of the raw and popped makhana is presented in Table 5. The popping of makhana resulted in a decrease in the essential and nonessential amino acids with an exception of lysine content. Lysine increased significantly ($p < .05$) from 2.19 mg/g to 6.34 mg/g during popping. Among all the amino acids in raw kernels, histidine content (301 mg/g) was highest followed by threonine (122 mg/g), leucine (49.2 mg/g), and phenylalanine (31 mg/g). Similar amino acid content was also observed in the popped makhana (Table 5) though the contents were significantly ($p < .05$) lower in comparison to the raw kernel. The reduction in the amino acid contents during popping might be due to the thermally induced chemical modifications of protein residues, such as glycoxidation,

TABLE 3 Effect of popping on functional properties of raw kernel, popped makhana, and bran

TABLE 5 Effect of popping on amino acid profile of raw kernel, popped makhana, and bran

| Amino acid (mg/g of protein) | Raw kernel | Popped makhana |
|------------------------------|------------|----------------|
| Lysine | 2.46 | 6.77 |
| Histidine | 300.99 | 204.14 |
| Arginine | 17.2 | 1.93 |
| Isoleucine | 14.74 | 2.91 |
| Leucine | 49.15 | 8.71 |
| Methionine | 15.97 | 3.87 |
| Phenylalanine | 31.95 | 4.84 |
| Threonine | 121.63 | 29.02 |
| Aspartic acid | 57.75 | 11.61 |
| Serine | 93.37 | 24.18 |
| Glutamic acid | 82.31 | 17.41 |
| Proline | 70.03 | 15.48 |
| Glycine | 58.97 | 51.28 |
| Alanine | 17.2 | 3.87 |
| Valine | 17.2 | 3.87 |
| Cysteine | 11.06 | 1.93 |
| Tyrosine | 22.11 | 2.91 |

deamidation, glycation, and oxidation (Arena, Renzone, D'Ambrosio, Salzano, & Scaloni, 2017). The decrease in the amino acid contents during dry heat puffing was also reported for kiwicha and quinoa grain (Paucar-Menacho et al., 2018). Higher histidine content in raw and popped makhana shows that the makhana may be a good ingredient for infants' foods in view of importance of histidine in the growth of children (Cho, Anderson, Wixom, Hanson, & Krause, 1984).

3.7 | Total phenolic content

Total phenolic content (TPC) of the raw kernel, popped makhana, and bran is presented in Table 6. The popping process reduced the TPC by 49.6%. The decrease in the TPC might be due to the thermal degradation of heat-labile phenolic compounds during the roasting (Randhir, Kwon, & Shetty, 2008). A similar decrease in the TPC was reported for puffing of horse gram (Sreerama et al., 2008). Bran contained significantly ($p < .05$) higher phenolic content than the kernel and popped makhana. It indicated that the phenolic compounds were mainly present in the bran and separation of bran during popping resulted in the significant decrease of TPC in popped makhana. Phenolic compounds chelate metals, inhibit lipoxigenase,

TABLE 6 Effect of popping on total phenolic content, flavonoid content, phytic acid, tannins, and antioxidant activity of raw kernel, popped makhana, and bran

| Item | Raw kernel | Popped makhana | Bran |
|---|---------------------------|--------------------------|----------------------------|
| Total phenolic content (mg GAE/g) | 2.22 ± 0.06 ^b | 1.12 ± 0.06 ^c | 45.42 ± 0.79 ^a |
| Total flavonoid content (mg RE/100 g) | 3.23 ± 0.28 ^b | 1.26 ± 0.18 ^c | 177.94 ± 1.30 ^a |
| Phytic acid (mg/100 g) | 87.36 ± 7.7 ^b | ND | 665.05 ± 7.08 ^a |
| Tannins (mg CE/g) | 0.88 ± 0.12 ^b | ND | 35.82 ± 0.57 ^a |
| Antioxidant activity (% inhibition of DPPH) | 27.57 ± 2.13 ^b | 17.48 ± 1.2 ^c | 95.49 ± 0.75 ^a |

Note: Means ± SD in a row with different superscripts are significantly different ($p < .05$).

Abbreviations: GAE, gallic acid equivalent; RE, rutin equivalent; CE, catechin equivalent; ND, not detected.

and scavenge free radicals, thus improve the antioxidant activity (Kumar & Langoo, 2016). Thus, the bran which showed higher phenolic content and higher antioxidant activity may be used to stabilize oxidation susceptible food products.

3.8 | Total flavonoid content (TFC)

A significant ($p < .05$) reduction of 34.7% in TFC was observed due to the popping of makhana (Table 6). The flavonoids are heat susceptible compounds which might have reduced during roasting and puffing as also observed by Mir et al. (2016) during rice puffing. However, in this study the bran contained very high TFC of 178 mg/100 g. Ti et al. (2014) also reported 10.4 times higher TFC in rice bran in comparison to the rice endosperm. It showed that the TFC was mainly present in the bran and the separation of bran during popping resulted in the significant decrease of TFC.

3.9 | Tannins and phytic acid

Tannins were not present in the popped makhana, whereas the bran contained 35.8 mg/100 g tannins (Table 6). It indicated that the tannins were concentrated in the bran only. The phytic acid was not present in the popped makhana, whereas 87.4 mg/100 g phytic acid was found in the raw kernel. Furthermore, the phytic acid content of bran was 665 mg/100 g. It indicated that the phytic acid was present only in the bran. The bran was detached from the kernel when popping took place, and therefore the phytic acid was not observed in the popped makhana. It may, therefore, be inferred that popping resulted in the complete removal of phytic acid and tannins due to separation of bran from popped makhana.

3.10 | DPPH radical scavenging activity

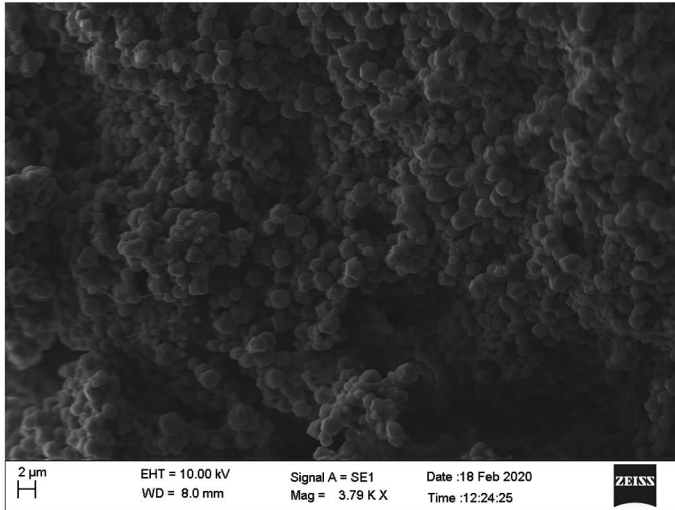
The DPPH radical scavenging activity of the samples ranged from 17.5% in popped makhana to 95.5% in bran (Table 6). The bran had significantly ($p < .05$) higher activity than those of raw kernel and popped makhana. Popping resulted in a significant ($p < .05$) decrease in DPPH activity in popped makhana, which might be

due to the decomposition of phytochemicals (Mir et al., 2016) and separation of bran during popping. It was also reported in earlier studies that higher concentration of phenolics and flavonoids was positively correlated with DPPH radical scavenging activity (Yogesh, Jha, & Ahmad, 2014). It may, therefore, be inferred that the phenolics, flavonoids, and phytic acids were mainly concentrated in the bran as discussed in earlier sections and hence the antioxidant activity of bran was more. As discussed in earlier section, this bran may be used to improve the oxidative stability of oxidation susceptible food products containing high amount of polyunsaturated fatty acids (Kumar, Tyagi, Vishwakarma, & Kalia, 2017).

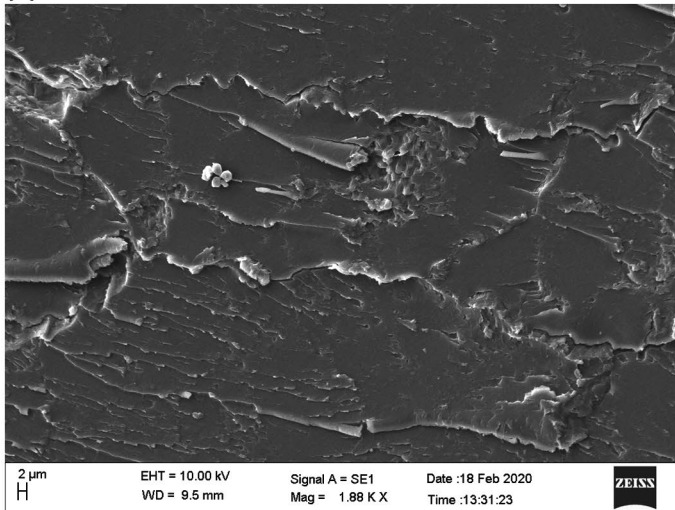
3.11 | Microstructure

The scanning electron micrographs of makhana samples during popping are shown in Figure 1. The raw makhana kernel had crystalline particles, which were loosely attached with clearly visible voids (Figure 1a). The initial roasting of raw makhana resulted in the swelling and gelatinization of starch, and therefore the voids were filled. The paste-like surface of roasted kernel showed that the crystalline structure of the amylopectin molecules converted into amorphous form (Figure 1b). Some cracks on the surface indicated the development of fissures due to shrinkage induced by moisture loss during storage of makhana seeds for 48 hr after initial roasting. The gelatinized starch with denatured protein in the kernel gave hardness to the makhana kernel. In the expansion process, the compact structure further converted into a highly porous matrix with many big cavities, which were formed due to sudden expansion upon breaking of hard seed coat. The compact structure of the gelatinized starch granules and uniform vapor pressure release during breaking the seed coat allowed expansion in all directions and thin films of starch were formed (Figure 1c). These films were disrupted from several places to release the water vapors and the porous structure was generated in the endosperm. The high temperature roasting of initially roasted makhana did not reduce the moisture because of the presence of seed coat, and therefore very high vapor pressure built up inside the kernel. This led to the formation of numerous voids inside the seed that forced the retrograded starch to expand upon breaking of the seed coat.

(a)



(b)



(c)

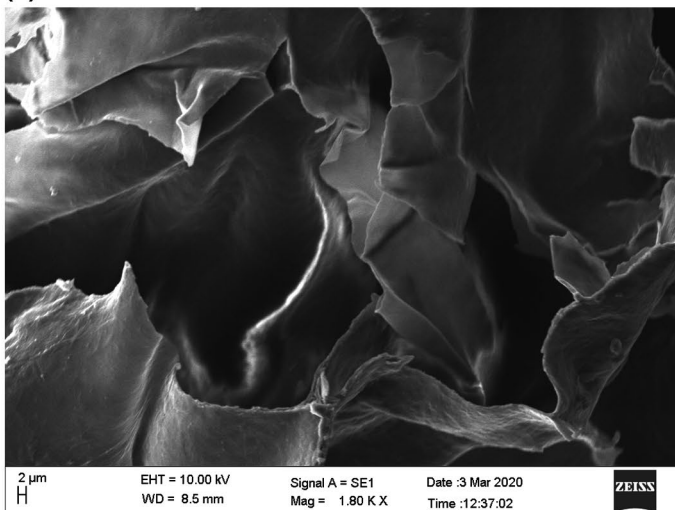


FIGURE 1 Scanning electron microscopic images of (a) raw makhana at 3.79 KX (b) roasted makhana at 1.88 KX (c) popped makhana at 1.80 KX

4 | CONCLUSION

The present study was conducted to understand the changes taking place in various properties of makhana during popping. The

popping changed the starch characteristics and microstructure, whereas the expansion of kernel improved water and oil absorption capacities. Popping of makhana enhanced protein content and dietary fiber while total phenolics, total flavonoids, phytic

acid, tannins, and antioxidant activity reduced significantly. Amino acids, carbohydrate, starch, amylose, and amylopectin also decreased during popping. The majority of beneficial phytochemicals were concentrated in the bran which was a by-product of popping process. Therefore, the bran may be considered for its application in food products, whereas the raw kernel may be used for incorporation in food products to enhance mineral and amino acid profile. Popped makhana may be used as mineral and phytochemical-rich food product.

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CONFLICT OF INTEREST

There is no conflict of interest.

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