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# Process Standardization for Isolation of Starch from Buckwheat *(Fagopyrum esculentum) flour*

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**Abstract—***Buckwheat (Fagopyrum spp) commonly known as kuttu is a non-glutinous pseudo cereal belonging to the family Polygonaceae. The gross chemical composition of dehulled buckwheat grains is similar to the cereals. It is a rich source of starch, proteins, antioxidants, and dietary fibre. The starch content of whole buckwheat grains varies from 59%-70%. The alkali treatment is normally employed to dissociate proteins from the starch. The published literature shows that varying alkali concentrations were used to isolate the starch from buckwheat. So the present investigation was undertaken to study the effect of varying alkali concentrations (0.1 - 0.3%) on the starch yields and to standardize a process for the isolation of buckwheat starch based on maximum starch yield with minimum residual components. It was observed that alkali concentration significantly affected the starch yield and amylose content. The maximum starch yield (45.3%) was obtained using alkali concentration of 0.2%. The amylose content of the isolated starch had a negative correlation with alkali concentration.*

**Keywords:** *Buckwheat, starch, physiochemical properties, pasting properties, SEM*

## 1. INTRODUCTION

Buckwheat (*Fagopyrum spp)* is a non-glutinous pseudo cereal belonging to the family Polygonaceae. It is a cool climate crop and cultivated throughout the world but the main producers of buckwheat are China, Russian Federation, Ukraine, and Kazakhstan [3,7]. Buckwheat seed is dicotyledonous with a tetrahedron shape. The dehulled buckwheat seed, called the groat, resembles the cereal kernel in its gross chemical composition. The groats showed a total carbohydrate percentage of 67.8–70.1% [7,14], of which 54.5% was found to be starch [14]. Buckwheat seeds exhibited a higher carbohydrate percentage of 73.3% due to the presence of the pericarp but had a similar starch percentage (55.8%). Buckwheat starch granule sizes are 2.9–9.3 μm with a mean size of 5.8 μm and are round or polygonical shaped [11]. The water-binding capacity of buckwheat starch is higher than that of wheat and corn starch due to the small size (and hence a bigger surface area) of buckwheat starch granules [13].

In general, buckwheat starch exhibited higher peak viscosities than cereal starches and more resembled pasting behaviour of root and tuber starches as buckwheat starches exhibited a higher granule swelling and gelling tendency than cereal starches. [12-13, 17].

In the recent years the starch consumption is increasing both in food and industrial applications. Being a rich source of starch with good functional properties buckwheat can be one of the important non conventional starch sources. While going through the published literature, it was observed that different procedures for starch isolation from buckwheat were adopted worldwide [11,13] and[18]. Most of these researchers used alkali treatment to dissociate proteins at varying levels. So the present investigation was planned to standardise a process of starch isolation from Indian buckwheat with the objective to maximise starch yield with minimum impurities, and to characterize the extracted starch for its physiochemical and functional properties.

## 2. MATERIALS AND METHODS:

### 2.1 Procurement of materials

Buckwheat (*Fagopyrum esculentum*) grains were obtained from local market. The grains were cleaned for impurities, immature, shriveled and damaged grains. Cleaned grains were stored under refrigerator (5±2°C) for further use. All chemicals used in this study were of analytical grade.

### 2.2 Isolation of Starch

Preliminary studies find out that alkali concentration effect the starch isolation yield. Buckwheat starch was isolated from the grains through modifications of some reported steps [11,13and18] as per the following procedure:

The buckwheat grains were ground in the plate mill. The flour obtained was screened through 60 mesh sieve. The flour was steeped in hexane (1:5w/v) for 2 hr while stirring continuously. The mixture was filtered to get defatted flour.

The protein was removed using a centrifugation technique. Defatted flour was steeped in 0.1%, 0.15%, 0.2% and 0.25% and 0.3 % NaOH (1:6 w/v) solution overnight. The mixture was then blended and sieved through US no 70 mesh (0.208mm). The mixture was then centrifuged at 1000*g* for 15 min. The supernatant was discarded and brown-yellow protein layer at the top of white starch was removed. The white starch layer was re-suspended in distilled water, centrifuged, decanted, and cleaned of the top brown-yellow protein layer. The process was repeated till no brown layer was visible. The remaining starch cake was neutralized by repeated washing with distilled water. It was then dried overnight at 40±1$^0$C, grinded, weighed and analysed.

### 2.3 Physico-chemical properties of extracted starch

Moisture content, crude protein, fat, and fibre content of extracted starches were determined according to AOAC method [2].

**2.3.1Amylose Content** Amylose content of the samples was determined by the method of Morrison and Laignelet [9]. 70mg of starch was mixed with 10 ml of urea and DMSO (Di-methyl-sulphoxide) solution in 1:9 ratio and heated for 10 min at 100°C while continuous stirring. The mixed sample was incubated at 100°C for 1 h and then cooled to room temperature. Addition of 0.5 ml solution of above mixed incubated sample was taken with subsequent addition of 25 ml distilled water and 1 ml solution of Iodine (I) and potassium Iodide (KI). The 1 ml solution was made by addition of Iodine (I) (2mg) and potassium Iodide (KI) (20mg) and volume was made up to 1 ml by distilled water. Blank sample was also prepared without addition of starch sample and absorbance was taken at 635nm.

$$\text{Blue Value (\%)} = \frac{\text{Absorbance} \times 100}{2 \times \text{g of solution} \times \text{weight of sample}}$$

$$\text{Amylose Content (\%)} = 28.414 \times \text{Blue Value}$$

**2.3.2 Water binding (WBC) and oil binding (OBC) capacity (% w/w):** Water binding and oil binding capacity of the extracted starch was determined using the method described by Medcalf and Gilles [8]. A suspension of 5g (dry mass) starch in 75ml distilled water and oil respectively was agitated for 1 hour and centrifuged at 3000 rpm for 10 minutes. The free water/oil was removed from the starch, which was then drained for 10 minutes and weighed. Water binding and oil binding capacity the starch was calculated as follows:

$$WBC/OBC(\%) = \frac{Weight\ of\ resudual\ starch \times 100}{Weight of\ sample}$$

**2.3.3 Solubility and Swelling power:** Solubility(%) and Swelling power (SP) of extracted starch was determined as per method adopted by Adebooye and Singh [1] with slight modification. Starch samples (500 mg) each were cooked for 30 min with 20 ml distilled water at different temperatures of 95 °C. The cooked samples were cooled to room temperature and centrifuged at 3000 rpm for 15 min. The supernatant was poured into pre-weighed glass dish, oven dried at 105 °C and weighed for solubility determination while the residue was weighed for swelling power estimation. Percent solubility and swelling power of extracted starch was calculated as follows:

$$\%SOL = \frac{A}{S} \times 100$$

$$Swelling\ Power = \frac{(B \times 100)}{S(100 - \%SOL)}$$

Where % SOL=Percent solubility; A= weight of dissolved solids in supernatant; B=weight of sediment paste; S=weight of sample.

**2.3.4 Pasting properties:** A Rapid Visco-Analyzer (RVA) (model 3D, RVA; Newport Scientific Pvt. Ltd., Warriewood, Australia) was used to determine the pasting properties of the buckwheat starch samples. A suspension of 3g starch (14% moisture basis) in 25 ml of distilled water underwent a controlled heating and cooling cycle under constant shear. It was held at 50°C for 1 minute; and then heated from 50°C to 95°C with 12°C /minute. Again held at 95°C for 3 minutes and then cooled with 12°C / minute upto 50°C. Peak viscosity (PV), temperature at which peak viscosity was reached ($P_{temp}$), viscosity at the end of hold time at 95°C or hot paste viscosity (HPV), viscosity at the end of the hold time at 50°C or cold paste viscosity (CPV).

**2.3.5 Morphological characteristics** The granule shape as a major morphological characteristic of the sample was observed at a moisture content of 5-6%. Starch sample was analyzed by scanning electron microscope (SEM), JEOL, Tokyo, Japan, Model No. JSM 6610-LV at magnifications of 2500X. The samples were mounted on aluminium stub using a double backed cellophane tape, coated in auto fine coater, JEOL-JFC-1600, with gold palladium (60:40 w/w).

## 3. RESULTS AND DISCUSSIONS

### 3.1 Starch yield

Starch yield varied between 28.9 to 45.3%. There was significant difference ($p \leq 0.05$) in starch yield between samples isolated by different alkali concentrations. Yield increased from 28.9 to 45.3% when alkali concentration increased from 0.1-0.2 %, but decreased to 34.4% at higher alkali concentration. At low alkali concentrations there might be incomplete dissolution of proteins which lead to decrease in yield similarly higher alkali concentrations lead to the formation of mucilaginous starch layer on the surface during centrifugation and which made difficult to separate the starch that lead to reduced starch yield.

### 3.2 Proximate composition of isolated starches

The proximate composition of isolated starch is shown in **Table 1.** There was no significant difference ($p \leq 0.05$) in moisture content within starch samples isolated by different alkali concentrations.

**Table 1: Effect of alkali concentration on proximate analysis isolated starch samples of buckwheat**

| Parameters | Alkali concentration (%) | | | | |
|---|---|---|---|---|---|
| | **0.10** | **0.15** | **0.20** | **0.25** | **0.30** |
| Moisture content (%) | 10.63 ±0.45a | 10.45 ±0.50a | 10.35 ±0.34a | 10.19 ±0.65a | 10.30 ±0.55a |
| Starch Yield (%) | 28.9 ±0.5a | 34.8 ±0.6b | 45.3 ±0.8c | 40.6 ±0.4c | 34.4 ±0.5d |
| Amylose content (%) | 29.6 ±0.3a | 28.9 ±0.3a | 28.3 ±0.2c | 28.1 ±0.3c | 27.2 ±0.4d |
| Fibre (%) | 0.21 ±0.01a | 0.19 ±0.02a | 0.16 ±0.03 | 0.16 ±0.03 | 0.17±0.02 |
| Protein (%) | 0.83 ±0.02a | 0.76 ±0.01b | 0.52 ±0.04c | 0.54 ±0.02c | 0.64 ±0.04d |

* Results are expressed as mean values±standard deviations. Means in a row with different superscripts are significantly different (P<0.05)

There was significant difference between proteins in samples isolated by different alkali concentrations. As the alkali concentration increased from 0.1-0.25%, the residual protein decreased from 0.83-0.52%. However, the residual protein retention increased to 0.64% upon further increasing the alkali concentration to 0.3%. This might be due to isoelectric pH shift. Similar results were observed by Zheng et al (1998) and Quian et al (1998).

There was no significant difference ($p \leq 0.05$) in fibre content of starch samples isolated by different alkali concentrations.

The amylose content decreased from 29.6-27.2% with increase in alkali concentration 0.1-0.3%**.** According to Karim et al. [4] the reduction in amylose content of alkali-treated starches could be attributed to the disruption of the amorphous region that contains amylose chains. Additionly, the alkali probably affects the amylose rather than the amylopectin molecules and/or regions of the granules. The ions in alkali solution diffuse into the amylose-rich amorphous regions of the granules, break intermolecular bonds, and cause the granules to swell to a higher degree, with a concomitantly higher exudation of amylase [5].

Therefore, on the basis of maximum starch yield and minimum impurities like residual protein and fibre content, 0.2% NaOH concentration of was found optimum for isolation of starch fro buckwheat flour.

**3.2.1 Water binding and Oil binding capacity:** Water binding capacity of starch granule is the tendency to absorb water and the degree of association of water molecules within starch granule [6]. WBC and OBC depend on the availability of hydrophilic and hydrophobic sites in starch molecules. WBC and OBC of native buckwheat starch were 110.36±0.23 % and 110.76±0.22 %, respectively. The isolated starches had higher values for WBC and OBC in comparison to some other nonconventional starches.

**Table 2: Physiochemical analysis of isolated starch from buckwheat**

| Water Binding Capacity (%) | Oil binding Capacity (%) | Swelling Power (%) | Solubility (%) |
|---|---|---|---|
| 110.36±0.45 | 110.76±0.85 | 12.84±0.80 | 19.89±0.42 |

**3.2.2 Swelling power and solubility:** The swelling and solubility represents the integrity and rigidity of the starch granules. Swelling is primarily a property of amylopectin and amylose, whereas lipids can inhibit swelling [15]. Swelling power of buckwheat starch was found to be 12.84% whereas the solubility was 19.89%.

**3.3.4 Pasting properties:** The pasting behavior is usually studied by observing changes in the viscosity of a starch system based on rheological principles. From the pasting curve, several parameters can be observed that indicate the extent of disintegration and whether there is retrogradation. Viscosity of starch, as a food component is a vital factor for applicability to food systems. The result of pasting properties of the buckwheat starch is presented in Table 3. Various parameters observed were peak (PV), trough (TV), breakdown (BDV), final (FV) and setback viscosity (SV).

**Table 3: Pasting properties of isolated buckwheat starch**

| PV (cP) | TV (cP) | BDV (cP) | FV (cP) | SBV (cP) |
|---|---|---|---|---|
| 6062 | 2970.22 | 3476.24 | 3414.66 | 2359.50 |

### 3.3.4 Scanning electron microscopy (SEM)

The SEM micrograph of isolated buckwheat starch is shown in Fig. 1. The buckwheat starch granules were polygonal in shape with smooth surface. The size of granules varied from 3-9 µm.

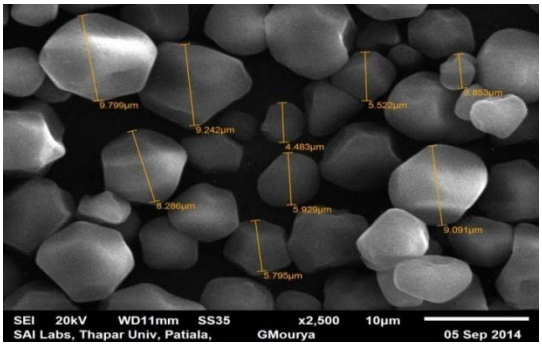


**Fig. 1: Scanning electron micrographs of native buckwheat starch**

## 4. CONCLUSIONS

Buckwheat flour was extracted to produce clean starch with acceptable yield by alkaline extraction. The optimum conditions for alkaline extraction of the buckwheat starch was steeping overnight at 0.2% NaOH concentration.

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