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Development of Edible Films from Native and Modified Starches of Common Buckwheat

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Abstract: Buckwheat (*Fagopyrum esculentum Möench*) is an annual crop, it is a pseudocereal but chemical composition and utilisation is of its grains is similar to cereals. Starch is the major component of buckwheat grains. Starch was isolation from common buckwheat seeds and hydrothermally modified at 120°C for 6hr keeping the moisture content (30%) constant. Film were prepared from native and modified starches of buckwheat and evaluated for different parameters. All film samples were continuous, transparent and crack free. Heat-moisture treated starch film had higher tensile strength and moisture barrier capacity.

Keywords: Buckwheat, heat-moisture treatment, edible films, starch.

1. INTRODUCTION

Starch is the polysaccharide reserve of plants and has applications ranging from giving texture and consistency to foods and papers to adhesives and biodegradable packaging. Starch is widely used functional ingredient as thickener, stabilizer and gelling agent in food industry. The applications of starch in food industries are mainly governed by its characteristics including functional, pasting, gelatinization, and structural properties. Starch from each source is unique in nature having different characteristics determining its suitability for particular application. Irrespective of sources, native starches are undesirable for many applications due to their inability to withstand processing conditions. In order to meet the new technological requirements of specific industrial processes, starches are modified by physical, chemical, enzymatic treatments or genetic transformation. Modification of starches is done with a purpose of improving the functional characteristics of starches and tailoring them to specific applications. Heat moisture treatment of starch is a physical method of starch modification involving heat treatment (80 to 120°C) of starch at restricted moisture level (10 to 30%) for a time period ranging from 15 min to 16 hr. Growing demand of starch has created interest in searching the non-conventional starch sources. Buckwheat is an annual dicotyledonous crop which belongs to Polygonaceae family. Two types of buckwheat are used around the world: common buckwheat (*Fagopyrum esculentum Moench*) and tartary buckwheat (*Fagopyrum tataricum*) depending on the production zone. Starch is the major component of buckwheat endosperm, which plays a significant role in appearance, structure and quality of food products of buckwheat. Therefore, buckwheat could be a promising new starch source as it produces starch with diverse physico-chemical properties that give it a broad range of potential industrial applications. Starch as biopolymer suits all the major aspects like edibility, large availability, nutritional value, biodegradability, biocompatibility, diverse functional properties which make it as a potential material for preparation of edible coatings or films (Dang and Yoksan, 2015; Reis et al., 2015). Edible films prepared from starch are odourless, neutral in taste, colourless, free from toxic, and semi-permeable to moisture, carbon dioxide, oxygen, and lipid as well as flavour components (Shah et al., 2016). Therefore, keeping the above in view, present investigation was carried out with the objective of isolation and hydrothermal modification of buckwheat starch; and preparation of edible films using native and modified starches of buckwheat and their evaluation.

2. MATERIALS AND METHODS

Grains of common buckwheat of cultivar named VL-7 used in this study were procured from National Bureau of Plant Genetic Resources Regional Station, Shimla, India. The grains were screened to remove foreign matter and stored in sealed container at room temperature. Starch was isolated from buckwheat grains according to the alkaline steeping method (Choi et al., 2000). Grains were steeped in 0.25% aqueous NaOH solution for 18 hr at room temperature and stirred three times during this period. After steeping, the grains were washed with distilled water and ground in a blender at full speed for 2 min, and slurry was filtered step wise through 100 mash (150µm) and 270 mesh (53µm) sieves. The starch was isolated from the filtrate by centrifugation at 25000g for 20 min. The supernatant was discarded, and the top yellowish layer of protein was removed. This step was repeated to obtain a white starch layer. The starch layer was re-suspended in distilled water, shaken and centrifuged as described above. Thereafter, the isolated starch was dried in hot air oven at below 40°C for 8 to 10 hr and stored at room temperature in sealed container.

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The heat moisture treatment of buckwheat starch was carried out according to the method of Franco et al. (1995) with minor modifications. The moisture level of starch was adjusted to 30% by adding appropriate volume of distilled water (the moisture level of native starch was predetermined).The addition of distilled water was done slowly and simultaneously mixed for uniform distribution of water in starch powder. Sample was sealed in polyethylene pouches and equilibrated at 10°C overnight. After the incubation, starch was filled in air tight glass containers and heated for 6 hr 120°C. The sample was shaken occasionally for uniform distribution of heat. The starch was cooled to room temperature and dried at 40°C for 6 to 8 hr and equilibrated at room temperature for 4hr. The dried powder was sealed in polyethylene bags, labelled and stored at room temperature for further analysis.

Starch films using native and heat moisture treated starches of buckwheat were prepared by following the method described by Chandla et al. (2017) with minor modifications. Filmogenic solutions were prepared by dispersion of 5g starch in 100 ml distilled water with continuous stirring at magnetic stirrer for 15min. Glycerol at rate of 3g/100g starch was added as plasticizer and mixed thoroughly. The solution was magnetically stirred for 15min at 85°C. The resulting solution was cooled at room temperature to avoid air bubbles during pouring. Casting technique was used to prepare films. The prepared solution was poured onto the polypropylene round trays of diameter 12.5cm and dried at 40°C for 16hr in hot air oven with circulating air in chamber.

The thickness of starch films was determined using Digital micrometer with an accuracy of ±0.001mm. The average value of 10 thickness measurement at different locations on each film was used in all calculations. Moisture content of starch films was determined by drying the pre-weighed pieces of films at 110°C for 6 to 8hr or till the weight comes constant. Water solubility of starch films was determined by following the method of Gontard et al. (1994). Pre-weight piece of starch film was immersed in water at room temperature for 24hr. The immersed film piece was removed from water and dried in oven at 110°C for 4 to 5 hr, cooled and weighed. The water solubility of starch film was measured as the difference in weight of dried piece of film before and after immersion in water. Color of native and modified starch films was measured using CR-300 Chroma meter (Minolta, Japan). Water vapour permeability of starch films was determined by following the E96-95 ASTM standard method (ASTM, 1995). Each film sample was sealed over the circular opening of a permeation cell containing anhydrous $CaCl_2$ (0% RH) and weighed. These cells were placed on desiccators with a saturated NaCl solution (75% RH) at 25°C. The weight of each permeation cell was recorded after 24hr and water vapour permeability of films was calculated using following formula-

$$\mathrm{WVP} = \frac{\Delta W \times X}{t \times A \times \Delta P}$$

Where WAP is the water vapour permeability (g.mm/m^2.day.kPa);ΔW is the weight gain by descant (g); X is the film thickness (mm); t is the incubation period (days); A is the area of the exposed film surface (m^2); and ΔP is the difference of partial pressure (kPa). Tensile strength of films was determined using texture analyser (TA-XT 2i Stable Micro Systems, UK). The films were cut in strips (20mm × 50mm) and thickness of strips was measured at eight points. The strip was gripped from both the edges of width on 'tensile grip' probe and initial grip separation was set at 30mm. The force and distance were recorded during extension of strips at 0.8mm/s up to break. The tensile strength of films was calculated using following formula-

$$\mathrm{TS} = \frac{F}{A}$$

Where TS is the tensile strength (MPa); F is the maximum force (N); A is the area of film cross-section (thickness × width; m^2). Analytical determinations were done in triplicate.

3. RESULTS AND DISCUSSION

Films formed of native and heat moisture treated starches of buckwheat were continuous and easily peel able from polypropylene surface after drying. All the film samples were transparent and free of cracks and pores. Results of moisture content, thickness, solubility, water permeability and tensile strength of films of buckwheat starch are represented in Table 1. Various factors such as drying time and temperature, relative humidity of drying chamber, film thickness and starch properties affects the moisture content of starch films. Higher moisture content of heat moisture treated starch films than native starch film could be attributed to the disruption of hydrogen bonds between amorphous and crystalline regions resulting in slight expansion of amorphous region during hydrothermal treatment, consequently increased water retention capacity of film. Film thickness is an important factor as it influences the transparency and water vapour permeation rate of film. Even film thickness is required to attain good and uniform mechanical strength. The film thickness of native starch film was 0.154mm while it was 0.186mm in film samples of hydrothermally modified starch. Zavareze et al. (2012) reported that the film thickness of potato starch films (native, oxidised and heat moisture treated starches) ranged from 0.073 to 0.168mm. Films of native and modified starches of buckwheat were intact after immersion in water for 24hr. The solubility of native buckwheat starch films was 33.68% and which decreased to 28.57% in heat moisture treated starch film. Higher solubility of film could result in disintegration of film

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with in short time while lower solubility could decrease the rate of degradation of film. Thus, water solubility of film is favourable when film is consumed with product, while in other cases; lower solubility is desired to increase product integrity. The water solubility of native buckwheat starch film was in range from 33.64 to 37.56% solubility of films of amaranth starches reported by Chandla et al. (2017). Zavareze et al. (2012) observed lower water solubility of starch films formed of hydrothermally modified starches of potato than solubility of native potato starch films. The reduced water solubility of the hydrothermally modified buckwheat starch films might be attributed to the increased interactions between amylose-amylose, amylose-amylopectin chains and strengthened intermolecular bonds promoted during modification treatments.

Table 1 Moisture content, thickness, solubility, water vapour permeability and tensile strength of films of native and modified starches of buckwheat

Treatments	Moisture content (%)	Thickness (mm)	Solubility (%)	WVPR (g.mm/m^2.day.kPa)	Tensile strength (MPa)
NS	11.44±0.30	0.186±0.00	33.68±1.74	6.31±0.00	1.84±0.00
HMT	11.89±0.12	0.154±0.00	28.57±0.49	5.21±0.30	2.69±0.00

All values are mean of triplicate determinations ± standard deviation mean. Values within same column with different letters are significantly different ($p \leq 0.05$). NS: native starch; HMTS: heat moisture treated starch; mm: millimetres; kPa: kilopascal; MPa: megapascal

The tensile strength of film is the maximum force applied at the break point of film sample. The native buckwheat starch film had 1.84MPa tensile strength which was consistent with the finding of Biduski et al. (2017) observed tensile strength ranged from 1.2 to 1.6MPa for native starch films of sorghum. The variation in mechanical strength of starch films in present study and previous records might be due to differences in source of starches, level of starch used to form film, thickness, film formation conditions like drying temperature and rate. Noticeably increased tensile strength was noticed for films of hydrothermally modified starch as compared with films of native starch. It has been reported by Zavareze et al. (2012) that heat moisture treatment of potato starch increased the tensile strength of the films from 3.53 to 6.07MPa. However, Majzoobi et al. (2015) reported reduced tensile strength of films from 2.22 to 1.84MPa after hydrothermal treatment of rice starch. The mechanical properties of the starch films depend on various factors such as molecular chain interactions, film thickness, quantity and type of the plasticizer used, and drying conditions of film. Additional interaction among amylose and amylopectin molecules caused by hydrothermal treatment could be the reason for increased tensile strength of film.

Suitable moisture barrier property is essential to maintain quality of food products during storage as moisture content immensely affects the quality of food. The water vapour permeability of native buckwheat starch films was 6.31g.mm/m^2.day.kPa and heat moisture treated starch film showed decreased water vapour permeability of films. Retrogradation in starch gel takes place due to interaction in amorphous region followed by interaction in crystalline domain. Thus retrogradation in amorphous region of starch during heat moisture treatment leaded to dense packing of starch chains resulted in decreased water vapour permeability of film of heat moisture treated starches of buckwheat. Thickness of film is an another important factor affecting water vapour permeability and lower thickness of films of heat moisture treated starch could be the grounds for lower water vapour permeability of starch films than native buckwheat starch film. Correspondingly increasing water vapour permeability with increasing thickness and hydrophilicity of starch films was recorded for different starches in previous studies (Cuq et al., 1996; Mali et al., 2004; Zavareze et al., 2012).

Table 2 Color properties of films of native and modified starches of buckwheat

Treatments	L*	a*	b*
NS	83.71±0.13	-0.27±0.02	3.19±0.10
HMT	82.88±0.58	-0.28±0.01	4.23±0.05

All values are mean of triplicate determination ± standard deviation mean. Values within same column with different letters are significantly different ($p \leq 0.05$).NS: native starch; HMTS: heat moisture treated starch; L*: black to white; a*: green to red; b*: blue to yellow

Appearance of products is important for consumers and greatly affected by color parameter of applied film. Table 2 represents the color parameters of films prepared from native and modified starch of buckwheat. Heat moisture treatment of buckwheat starch affected the color of starch, consequently altered color values (L*, a* and b* parameter) of produced starch films. The L value indicating the lightness of films was decreased following hydrothermal

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modification of buckwheat starch. Negative values of a* indicated slight greenish shade in buckwheat starch films. Yellowness in starch films represented by positive b* values and heat moisture treated starch film showed higher value than that of native starch. Change in color of heat moisture treated starch films could be attributed to the occurrence of Millard reaction during modification treatment of starch.

4. CONCLUSION

Buckwheat starch can be successfully isolated and modified by heat-moisture treatment. Buckwheat starch was found suitable for edible film formation. Films prepared from native and modified starch were continuous and transparent. Heat moisture treatment of buckwheat starch increased the tensile strength and moisture barrier capacity of film. Water solubility of film was decreased following hydrothermal modification of buckwheat starch.

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