Structural and Film-Forming Properties of Millet Starches: A Comparative Study

Sneh Punia Bangar 1,* , Anil Kumar Siroha 2, Manju Nehra 2,* , Monica Trif 3, Vandana Ganwal 2 and Sumit Kumar 2

1 Department of Food, Nutrition and Packaging Sciences, Clemson University, Clemson, SC 29634, USA
2 Department Food Science and Technology, Chaudhary Devi Lal University, Sirsa 125055, Haryana, India; siroha01@gmail.com (A.K.S.); vandanaganwal12@gmail.com (V.G.); sumitkhoda1@gmail.com (S.K.)
3 Research and Development Department, CENCIRA Agrofood Research and Innovation Centre, Ion Meste 6, 40065 Cluj-Napoca, Romania; monica_trif@hotmail.com
* Correspondence: snehpunia69@gmail.com (S.P.B.); manju.vnehra@gmail.com (M.N.)

Abstract: Millets are an underutilized and important drought-resistant crop, which are mainly used for animal feed. The major constituent in millet is starch (70%); millet starch represents an alternative source of starches like maize, rice, potato, etc. This encouraged us to isolate and characterize the starches from different millet sources and to evaluate the application of these starches in edible film preparation. In the present study, the physicochemical, morphological, and film-forming characteristics of millet starches were studied. The amylose content, swelling power, and solubility of millet starches ranged from 11.01% to 16.61%, 14.43 to 18.83 g/g, and 15.2% to 25.9%, respectively. Significant differences (p < 0.05) were found with different pasting parameters, and the highest peak (2985 cP), breakdown (1618 cP), and final viscosity (3665 cP) were observed for barnyard, proso, and finger millet starch, respectively. Little millet starch achieved the highest pasting temperature. All starches showed A-type crystalline patterns, and relative crystallinity was observed at levels of 24.73% to 32.62%, with proso millet starch achieving the highest value. The light transmittance of starches varied from 3.3% to 5.2%, with proso millet starch showing the highest transparency. Significant differences (p < 0.05) were observed in the water solubility, thickness, opacity and mechanical characteristics of films. The results of the present study facilitate a better assessment of the functional characteristics of millet starches for their possible applications in the preparation of starch films.

Keywords: millets; starch; pasting; morphological; films

1. Introduction

Millets are underutilized crops whose potential is not fully explored as compared to other cereals, such as maize, wheat, and rice. According to the Food and Agriculture Organization (FAO), millet production in 2019 across the world was 28,371,792 tonnes, while in India it was 10,235,830 tonnes [1]. It is a highly valuable complementary nutrition source for human beings, due to the abundance of some of its constituents, such as starch, B vitamins, minerals, lysine, polysaccharides (antioxidants), and dietary fibers [2]. Many researchers have performed proximate analyses of millets, reporting on crude protein (4.48 to 12.49 g/100 g), crude fat (1.2 to 7.2 g/100 g), ash content (0.6 to 4.78 g/100g), crude fiber (0.49 to 8.72 g/100 g) and carbohydrate content (62.25 to 81.69 g/100g), respectively [3–6]. Starch, the major constituent of the fully ripened grains, is the main factor used for determining millets’ suitability for certain applications [7].

Starch is the most abundant polysaccharide in nature; it is cost-effective, a major source of energy in human food, and is widely utilized for various food and non-food purposes [8]. Starch is the main constituent of millet grain and is widely utilized in many food applications, such as in breakfast cereals, bakery products, and health foods. Millet starch exhibits a 0.38–28.40% amylose content, 7.64–11.48% moisture, 0.39–1.60% ash, and...
0.31–0.58% protein content [9]. Zhu [7] also reviewed millet starches, reporting starch yield of 52.0% to 93.7%, amylose contents (AC) of 6–38.6%, lipid contents of 0.16–2.9%, protein contents of 0.2–4.3%, and ash contents of 0.02–1.4%, respectively. Millet starch granules were reported to vary from small to large, with oval, spherical, and polygonal shapes [6,10]. Millet starch has an A-type X-ray diffraction pattern suggesting the crystalline structure of cereal starches, and the degree of crystallinity of the starches varied from 23.7% to 30.9% [9].

Today, bioplastic substances offer a reasonable substitute to traditional plastics and their applications. Of the total level of global plastic production, i.e., 370 million tonnes, the bio-plastic market’s share is about 1% [11]. In spite of their mechanical characteristics, plastics derived from fossil fuels create environmental pollution due to their non-degradable nature [12]. Research on bioplastics has increased because of their biodegradable properties, which help to protect the environment by decreasing non-degradable plastic waste production. Researchers have shown their interest in biodegradable films to replace petroleum-based plastic materials [13]. Starch biodegradable films are currently popular, as starch is a sustainable resource, and it can be substitute for the synthetic materials that are used in packaging [14]. Dai et al. [15] found that starch has many beneficial properties, especially good film-forming characteristics, plentiful availability and its low cost, which can reduce the utilization of synthetic polymers.

Many researchers have conducted research on millet starches, but they have mainly focused on characterizing their various functional properties [9,10,16]. Millets are under-utilized, cost-effective crops, and they can be grown under adverse conditions, such as high salinity and low rainfall. More research is required into millet starches to make them a possible substitute for other traditional starch sources (corn, rice, potato), which are used directly as food. The preparation of biodegradable films from millet starches represents a new direction to the starch industry. The potential utility of millet starches for food and non-food purposes is limited. So, current study was undertaken to characterize the different millet starches, and to assess their utility for the formulation of films.

2. Materials and Methods

2.1. Materials

The millet cultivars finger (VL-Mandua) and barnyard (VL-181) were provided by ICAR-Vivekananda Parvatiya Krishi Anusandhan Sansthan, Almora, Uttarakhand (India), and foxtail millet (SiA-3222), kodo millet (JK-41), little millet (DHLM-36-3) and proso millet (TNAU-202) were provided by Indian Institute of Millets Research, Rajendranagar, Hyderabad (India). All chemicals used were of analytical grade.

2.2. Methods

2.2.1. Extraction and Characterization of Starch

Extraction of Starch

Sandhu and Singh’s [17] method was used for the extraction of the starch from millet grains. After soaking the grains in a 0.1% solution of Na$_2$S$_2$O$_5$ (18–20 h) and grinding them in a laboratory grinder, the slurry was sieved through different sizes, i.e., 0.250, 0.150, 0.100, 0.075, 0.045 mm. The slurry was allowed to settle for 4–5 h, and then the upper suspended portion was removed while the settled starch layer was centrifuged (Remi, New Delhi, India) at 2800 rpm for 5 min and the upper non-white layer was scrapped off to remove the non-starch portion. To remove the impurities, the white layer was re-suspended in water and centrifuged. This process was repeated 4–5 times. The starch was then collected and dried in a universal oven (NSW, New Delhi, India) at 45°C for 12 h.

Physicochemical Properties

The AC was measured by adopting the method of Williams et al. [18]. Starch (20 mg) was mixed with 10 mL of 0.5 mol/L KOH; then the content was transferred to a flask (100 mL) and water was added to the mark. The starch solution (10 mL) was then pipetted into a flask (50 mL) and 5 mL of 0.1 mol/L HCl was added. After that, 0.5 mL of iodine reagent
was added. Water was added to make the volume 50 mL and the absorbance was calculated at 625 nm in a spectrophotometer (Thermo Scientific, G10S UV-Vis, Shanghai, China). The method of Leach et al. [19] was followed to evaluate the swelling and solubility of the starch. Starch (1 g) was mixed with water (99 mL) and heated at 90 °C for 1 h, then cooled instantly in a water bath for 1 min, and then centrifuged at 3000 rpm for 10 min. Sediments were used for the calculation of swelling power, while the supernatant was transferred into pre-weighed petri dishes and dried in a hot air oven at 100 °C for 18–24 h and cooled to room temperature in a desiccator before re-weighing. The method described by Perera and Hoover [20] was applied for the determination of the transmittance of millet starches. To prepare the starch suspension (1%), it was heated at 90 °C for 60 min with continuous shaking. The transmittance of samples was analyzed at 640 nm at various storage intervals (0, 24, 48, 72, 96, and 120 h).

Pasting Properties

The pasting properties of millet starches were analyzed using RVA (Starch Master TM, Newport Scientific, Warriewood, Australia). The starch suspension was held at 50 °C for 1 min, then heated to 95 °C for 4 min and 30 s, and after holding at 95 °C (2 min 30 s) it was cooled to 50 °C for 4 min and held at 50 °C for 1 min. Different pasting parameters were calculated from the pasting curves.

X-ray Diffraction

X-ray diffraction patterns were measured using a diffractometer (Rigaku Miniflex, Tokyo, Japan). X-ray diffractograms were recorded at 25 °C in the 2θ angle range of 4° to 40° with a step size of 0.02 and 10 s scan speed. Origin Pro 8E (Origin Lab, Northampton, MA, USA) software was used for plotting the graph. The relative crystallinity (RC) of the starch samples was analyzed using following formula:

\[
RC(\%) = \frac{Ac}{(Ac + Aa)} \times 100
\]

where \(Ac\): crystalline area, and \(Aa\): amorphous area.

Morphological Properties

Morphological properties were analyzed using SEM (Model EVOLS10 ZEISS, Oberkochen, Germany). Ethanol was used to form a 1% starch solution to record the micrographs. The starch solution was transferred onto a metal stub and coated with Au-Pd (60:40). The samples were assessed at an accelerating potential of 10 kV, and a magnification power of 2500× was used during micrography.

Particle Size Distribution (PSD)

PSD was calculated with a particle size analyzer (Malvern Instruments Ltd., Malvern, UK). Prior to the calculation of particle size, sonication treatment was performed for three minutes to mix the starch suspension thoroughly. To analyze the starch sample, the refractive indexes were 1.33 (distilled water) and 1.53 (starch).

2.2.2. Film Preparation and Characterization

Preparation of Films

To prepare the starch films, the method given by da Rosa Zavareze et al. [21] was used with slight modification. A starch solution (4%) was prepared by dissolving the starch in distilled water and heating it at 90 °C for 10 min. The starch solution was cooled and 1% glycerol \((w/w)\) was added; after that, it was stirred at 150 rpm for 20 min. The starch solution was then strained through a muslin cloth. Solutions were loaded on baking trays and dried in the oven (50 °C; 16–20 h). After the required time, the films were removed from the trays and stored at 25 °C/53% relative humidity by using Mg(NO₃)₂ solution over 48 h for further determinations.
Properties of Film

The method of Mei et al. [22] was followed to measure the moisture content of the starch films using a moisture analyzer (Mettler Toledo, Greifensee, Switzerland). Film thickness was analyzed using the method of Fan et al. [23]. Thicknesses were analyzed at 10 random locations on the films, and their mean values in mm were calculated. The solubility of films was determined by dipping them in water for 24 h [24]. The films were collected from the water and put in a desiccator to obtain the final dry weight, and the difference between the two weights of the films was recognized as their solubility (%). The opacity of films was evaluated using a spectrophotometer (Thermo Scientific, G10S UV-Vis, Shanghai, China), and values were calculated at 600 nm [25].

\[ \text{Opacity} = \frac{A_{600}}{X} \text{ or } \left( -\log T_{600} X \right) \]

where \( A_{600} \) and \( T_{600} \) are respectively the absorbance and transmittance at 600 nm, and \( X \) is the thickness of films (mm).

Tensile Strength (TS) and Elongation at Break Point (EAB)

TS and EAB were measured with a texture analyzer (TA XT Plus Connect, Stable Micro Systems, Godalming, UK). Filmstrips of 1 cm × 7 cm were affixed to a pair of grips on the AT/G probe. The initial grip separation and cross-head speed were set at 50 mm and 1 mm/s, respectively.

2.2.3. Statistical Analysis

Statistical analyses of the data were carried out using Minitab Statistical Software version 14 (Minitab Inc, State College, PA, USA). The data shown in the tables are the mean of triplicate values. The data were subjected to one way analysis of variance (ANOVA).

3. Results and Discussion

3.1. Physicochemical Properties

The physicochemical properties of millet starches are tabulated in Table 1. AC was shown to play a vital role in the characteristics and applications of starch [26]. AC can be calculated in many ways, but iodine-binding spectrophotometry is widely used [27]. The AC of millet starches ranged between 11.01% to 16.61%; foxtail millet starch showed the highest value. AC is the most important characteristic of starch, as it affects the physicochemical characteristics and determines the utility of the starches in various applications [28]. The swelling power (SP) and solubility of millet starches ranged from 14.43 to 18.83 g/g and 14.9% to 25.8%, respectively; the largest values were observed for barnyard and little millet starch, respectively. The SP of starch granules is associated with amylopectin, and a higher SP correlated with a high water-holding capacity of amylopectin [29]. The solubility power of starch refers to the amylose leached during heating, and it is affected by starch constituents and granule size [30,31]. Xia et al. [32] explained that solubility is influenced by crystalline arrangement, granule size, the magnitude of gelatinization, and the morphology of the starch granule. Siroha et al. [16] reported the AC, SP and solubility of millet starches varying from 11.57% to 21.93%, 11.11 to 17.91 g/g, and 12.20% to 15.20%, respectively.

The transparency of gelatinized starch paste indicates the degree of starch chains in the paste [33]. It was calculated at 640 nm, using water as a blank, for a storage period of 120 h at 4 °C (Figure 1 and Table 1). The light transmittance of the starches ranged between 3.35% to 5.24%; the largest value was found for proso millet starch, and this value decreased with an increase in the storage time for all starches. Wani et al. [34] explained that this decrease may be due to starch retrogradation, and consequently the re-association of broken bonds in the organized starch. Transmittance is an essential property of starch pastes, and is vital for foods such as jellies and fruit pastes [35].
Table 1. Physicochemical properties of millet starches.

| Sample            | Amylose Content (%) | Swelling Power (g/g) | Solubility (%) | Transmittance (%) |
|-------------------|---------------------|----------------------|----------------|-------------------|
| Barnyard millet   | 14.42 ± 0.2 d       | 18.83 ± 0.3 f        | 20.1 ± 0.1 e   | 5.14 ± 0.01 c     |
| Finger millet     | 11.63 ± 0.4 b       | 14.43 ± 0.2 a        | 18.7 ± 0.2 d   | 5.06 ± 0.03 c     |
| Foxtail millet    | 16.61 ± 0.2 e       | 16.42 ± 0.4 c        | 18.1 ± 0.1 c   | 3.46 ± 0.02 a     |
| Kodo millet       | 13.99 ± 0.3 c       | 15.37 ± 0.3 c        | 14.9 ± 0.3 a   | 4.23 ± 0.01 b     |
| Little millet     | 11.01 ± 0.4 a       | 15.83 ± 0.3 d        | 25.8 ± 0.2 f   | 3.35 ± 0.01 a     |
| Proso millet      | 11.12 ± 0.2 a       | 14.72 ± 0.2 b        | 15.9 ± 0.2 b   | 5.24 ± 0.02 d     |

Data are means of triplicate observations. Values within the same column followed by the same superscript are not significantly different (p < 0.05).

Figure 1. Transmittance of millet starches: A: barnyard millet; B: finger millet; C: foxtail millet; D: kodo millet; E: little millet; F: proso millet.

3.2. Pasting Properties

The application of starch as a thickening agent is determined by its pasting characteristics. Significant differences (p < 0.05) were observed in the pasting properties of millet starches (Table 2). The difference between the pasting characteristics may be due to the variance between the SP and the solubility of starches. The peak viscosity (PV) of starches varied from 1518 to 2985 cP; the highest PV was found for barnyard millet starch. The PV of starch granules is affected by aspects such as RC, SP, friction between swollen granules, and granular strength [36]. Granule swelling and leaching occur in pasting, which result in high PV. During the period of holding at 95 °C, the swollen granules broke under shear stress, and the viscosity is then reduced to trough viscosity (TV) by way of breakdown viscosity (BV). The BV and TV of starches varied from 200 to 1436 cP and 1202 to 2332 cP, respectively; the largest values were found for barnyard and finger millet. Little millet starch showed the lowest BV, indicating that this starch is more stable during shearing and cooking. The setback viscosity (SV) of millet starches ranged between 810 and 1906 cP; the lowest value was found for little millet starch. Bangar et al. [37] explained that the low value of SV demanded by frozen food requires a lower syneresis value. The highest final viscosity (FV) value was achieved by finger millet starch. FV represents the capacity of starch to form a viscous paste or gel after cooking and cooling; it is considered an essential parameter for starch processing, such as canning [38]. At the minimum pasting temperature (PT), starch granules starts to swell. The PT of millet starches ranged between 79.1 and 84.1 °C; the largest and the smallest values were found for finger and little millet starches, respectively. Bangar et al. [31] explained that a high PT in starch might be due to the amylose–lipid complex, which enhances granular integrity and delay swelling.
Siroha et al. [16] reported PV, BV, SV and PT values of 1291–1853 mPa·s, 146–518 mPa·s, 506–961 mPa·s and 74.0–85.4 °C, respectively, for millet starches.

### Table 2. Pasting characteristics of millet starches.

| Sample        | PV (cP)        | BV (cP)        | TV (cP)        | SV (cP)        | FV (cP)        | PT (°C)        |
|---------------|----------------|----------------|----------------|----------------|----------------|---------------|
| Barnyard millet | 2985 ± 21 f    | 1436 ± 15 f    | 1549 ± 14 c    | 1904 ± 20 e    | 3453 ± 23 e    | 82.4 ± 0.1 d  |
| Finger millet  | 2956 ± 25 e    | 624 ± 11 b     | 2332 ± 25 e    | 1333 ± 11 c    | 3665 ± 26 f    | 79.1 ± 0.1 a  |
| Foxtail millet | 2178 ± 19 b    | 976 ± 14 c     | 1202 ± 12 a    | 1906 ± 18 e    | 3108 ± 21 c    | 80.0 ± 0.2 b  |
| Kodo millet    | 2734 ± 26 c    | 1187 ± 16 d    | 1547 ± 16 c    | 1691 ± 20 d    | 3238 ± 28 d    | 81.5 ± 0.1 c  |
| Little millet  | 1518 ± 20 a    | 200 ± 9 a      | 1318 ± 11 b    | 810 ± 11 a     | 2128 ± 21 a    | 84.1 ± 0.2 e  |
| Proso millet   | 2934 ± 27 d    | 1316 ± 12 e    | 1618 ± 15 d    | 888 ± 9 b      | 2204 ± 23 b    | 79.9 ± 0.2 b  |

Data are means of triplicate observations. Values within the same column followed by the same superscript are not significantly different (p < 0.05).

### 3.3. Particle Size Distribution (PSD)

The PSD of starches is tabulated in Table 3. In this investigation, d (4,3) represents the volume mean diameter (VMD); d (3,2) characterizes the area mean diameter (AMD), and d (10), d (50), and d (90) denotes the numbers of starch granules that are smaller (i.e., 10%, 50%, and 90%, respectively) than the average starch granules in the sample. The AMD and VMD of starches varied from 4.67 to 5.98 μm and 7.16 to 9.48 μm, and the largest and smallest values were found for barnyard and little millet starch, respectively. The d (10), d (50), and d (90) values of millet starch varied from 3.62 to 4.70 μm and 9.03 μm and 11.5 to 15.5 μm, respectively; the highest values were achieved by foxtail and barnyard millet starches. The physicochemical characteristics of starch granules, such as PSD, amylose/amylpectin ratio, and mineral content, influence the pasting properties of starch in aqueous systems [39]. Starch’s particle size is important for handling, packing, and various food and miscellaneous applications [40]. Wu et al. [9] reported that the particle size of millet starches varied from 3 to 15 μm. The PSD and granule size are crucial for the applicability of starches, and are associated with their WAC, swelling, and pasting properties [41].

### Table 3. Particle size distribution and relative crystallinity of millet starches.

| Sample        | D(3,2) (μm) | D(4,3) (μm) | Dv (10) (μm) | Dv (50) (μm) | Dv (90) (μm) | RC (%)       |
|---------------|-------------|-------------|--------------|--------------|--------------|--------------|
| Barnyard millet | 5.98 ± 0.1 d | 9.48 ± 0.5 d | 4.60 ± 0.2 d | 8.98 ± 0.4 d | 15.5 ± 0.6 d | 28.21 ± 2 b  |
| Finger millet  | 5.49 ± 0.3 c | 8.80 ± 0.3 c | 4.16 ± 0.3 c | 8.37 ± 0.5 c | 14.4 ± 0.3 c | 30.12 ± 1 d  |
| Foxtail millet | 5.93 ± 0.2 d | 9.46 ± 0.4 d | 4.70 ± 0.2 c | 9.03 ± 0.4 d | 15.3 ± 0.4 d | 24.73 ± 2 a  |
| Kodo millet    | 5.96 ± 0.4 d | 9.43 ± 0.5 d | 4.66 ± 0.3 c | 8.92 ± 0.3 d | 15.4 ± 0.5 d | 29.88 ± 2 c  |
| Little millet  | 4.67 ± 0.3 a | 7.16 ± 0.4 a | 3.62 ± 0.1 a | 6.82 ± 0.3 a | 11.5 ± 0.3 a | 31.06 ± 3 e  |
| Proso millet   | 5.17 ± 0.1 b | 8.10 ± 0.2 b | 4.10 ± 0.2 b | 7.71 ± 0.5 b | 13.0 ± 0.4 b | 31.62 ± 2 f  |

Data are means of triplicate observations. Values within the same column followed by the same superscript are not significantly different (p < 0.05).

### 3.4. XRD Pattern

The crystalline pattern of starches as analyzed by XRD is shown in Figure 2. It is well documented that the major component of the crystalline part of starch is amylpectin, while the amorphous region is made up of amylose [42]. Parallel double helices arrange these crystalline structures in a hexagonal structure with glucose residues [43]. Amylopectin’s structure regulates the morphology, size, and crystallinity of the starch [44]. Starch can be categorized into A-, B-, C-, and V-type, based on its peak distribution patterns, as shown by XRD diffractograms. Millet starches show an A-type crystalline pattern with peaks at 15°, 17°, 18° and 23.1° (2θ). This pattern is also demonstrated by cereal starches. The RC of millet starch varied from 24.73% to 31.62%; the largest value was observed for proso millet starch. Hoover and Ratnayake [45] explained that variations in the RC (%)
of different starches could be ascribed to crystal size, the quantity of the crystalline part, and the degree of interaction among double helices. In 2014, Annor et al. [46] reported that the RC (%) ranged from 27% to 30% in millet starches from four genotypes of four different species.

3.5. Morphological Properties

The micrographs of millet starches are presented in Figure 3. The granules of millet starches ranged from small to large, and were spherical and polygonal in shape. Small pores and cavities were also found on the surfaces of millet starch granules. Annor et al. [46] observed that the shapes of millet starch granules are spherical and polygonal, with a peak size of ~7.6 µm. In 2021, Wang et al. [47] explained that the morphology of starch granules is an essential feature while analyzing the association between the structure and the characteristics of starch. It plays a major part in the processing of starches. Sandhu and Siroha [10] reported a negative correlation between AC and starch granule size. The physicochemical, functional, and nutritional characteristics of starches are influenced by the shape and size of the starch granules; larger granules are responsible for high paste viscosity, while small granules correlate with higher digestibility [48].

3.6. Characteristics of Starch Films

The characteristics of millet starch films are shown in Table 4. Moisture content was determined to evaluate the moisture in the films, and it was found to vary from 10.1% to 14.6%; the highest value was found for the foxtail millet starch film. In 2020, Ballesteros-Martínez et al. [49] explained that water insolubility might be necessary to maintain product integrity and water resistance in packaging materials. However, high water solubility may be valuable for the coatings of fresh and minimally processed foods. The thickness of the film is a significant parameter that relates to the mechanical strength and permeability of the film to gases and water vapors. Evaluating the thickness is also related to assessing the uniformity of the films [50]. The thickness of the films ranged between 0.11 and 0.15 mm; the largest value was found for little millet, while the smallest was observed for finger millet starch films. Opacity is an important characteristic of films that indirectly relates to their transparency. The lowest opacity (1.75%) was found for little
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 opacity is an important property for films used on products that must be visible to consumers, especially foodstuffs [24]. Solubility is an essential factor for food packaging materials. The choice between packaging materials with different solubility characteristics is made on the basis of the food that is to be packed. The solubility of the films varied from 25.6% to 31.5%; the highest value was observed for the film prepared from little millet starch. Similar results for millet starch films were observed by Bangar et al. [51], who reported values for thickness, water solubility and opacity of 0.105 mm, 34.04%, and 1.744%, respectively. Thakur et al. [52] concluded that the thickness of films depends on the granule shape and size, and the amylose/amylopectin ratio. These film characteristics are interrelated, with film thickness often used to calculate the permeability, optical and mechanical properties of films [53, 54].

Figure 3. Morphological properties of millet starches—(A): barnyard millet; (B): finger millet; (C): foxtail millet; (D): kodo millet; (E): little millet; (F): proso millet.

Table 4. Properties of starch films.

| Sample            | Moisture Content (%) | Thickness (mm) | Water Solubility (%) | Opacity (%) | Tensile Strength (MPa) | Elongation at Break (%) |
|-------------------|----------------------|----------------|----------------------|-------------|------------------------|-------------------------|
| Barnyard millet   | 12.1 ± 1.5 c         | 0.13 ± 0.001   | 28.1 ± 0.3 d         | 2.41 ± 0.00 c | 4.77 ± 0.30 c         | 53.4 ± 1.5 a            |
| Finger millet     | 11.9 ± 1.1 c         | 0.11 ± 0.002   | 27.2 ± 0.4 c         | 3.21 ± 0.00 d | 6.87 ± 0.09 e         | 64.4 ± 2.1 c            |
| Foxtail millet    | 14.6 ± 1.3 c         | 0.12 ± 0.003   | 27.6 ± 0.3 c         | 2.45 ± 0.00 c | 6.95 ± 0.11 f         | 73.2 ± 1.8 e            |
| Kodo millet       | 13.5 ± 1.2 d         | 0.13 ± 0.002   | 25.6 ± 0.2 e         | 2.14 ± 0.00 b | 4.21 ± 0.08 b         | 63.6 ± 2.2 b            |
| Little millet     | 10.1 ± 1.1 a         | 0.15 ± 0.001   | 31.5 ± 0.4 e         | 1.75 ± 0.00 a | 3.79 ± 0.23 a         | 64.1 ± 1.5 c            |
| Proso millet      | 10.6 ± 1.0 b         | 0.12 ± 0.001   | 26.8 ± 0.2 b         | 2.42 ± 0.00 c | 5.91 ± 0.31 d         | 65.4 ± 2.4 d            |

Data are means of triplicate observations. Values within the same column followed by the same superscript are not significantly different (p < 0.05).
The primary mechanical properties of films are determined mainly via TS and EAB. TS refers to the highest tensile stress endured by the sample during the tension test. The TS of the films varied from 3.79 to 6.95 MPa, and the highest value was observed for films prepared from foxtail millet starch. EAB is an indication of the film’s flexibility and extensibility, which is determined by the point at which the film breaks under tensile testing, and is shown as the percentage of change in the original length of the sample [55]. The EAB of films varied from 53.4% to 73.2%, and foxtail millet starch films showed the highest value. Al-Hashimi et al. [56] reported the TS of millet films to be 10.52 MPa.

4. Conclusions

In the present study, millet starches were isolated and characterized for their physico-chemical, pasting and morphological properties, as well as their film-forming characteristics. Significant ($p < 0.05$) differences between amylose, swelling, and solubility were determined; the highest values for amylose, swelling power, and solubility were displayed by foxtail, barnyard, and little millet starch, respectively. Millet starches showed a range of peak viscosities, which implies their utility in various food applications. Millet starches contain granules varying in size and shape; small to large starch granules were detected, and their shapes were spherical and polygonal. The highest values for $d$ (10), $d$ (50), and $d$ (90) were shown by foxtail and barnyard millet starches. All starches displayed an A-type crystalline arrangement, which is the same pattern as cereal starches. Mechanical properties were evaluated mainly in terms of TS and EAB. Films were prepared and characterized from millet starches, showing that millet starch might be used to prepare biodegradable and edible films. More research is needed to make millet starches useful as raw materials for packaging films.

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