Archives of Pharmaceutical Sciences and Research
E-ISSN0975-2633|PRINTISSN0975-5284
Vol 15 Issue 2 073–079 June 2025
Research Article

A COMPARATIVE STUDY ON ISOLATION AND CHARACTERIZATION OF MILLETS VARIETIES

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ABSTRACT

The present study investigates the isolation and physicochemical characterization of starch from three varieties of millet: foxtail millet, little millet, and Kodo millet. Starch was isolated using an alkaline extraction method, and the yield was determined for each millet variety. Kodo millet demonstrated the highest starch yield (74.50%), followed by foxtail millet (68.54%) and little millet (45.35%). The isolated starch was subjected to iodine testing, confirming its identity with a characteristic blue-black coloration. Physicochemical properties of the starches were evaluated, including swelling power, angle of repose, bulk density, tapped density, Hausner's ratio, and Carr's index. Foxtail millet exhibited the highest swelling power (16.42%), while little millet had the highest angle of repose (35.30°), indicating poorer flow ability. Kodo millet showed superior bulk and tapped densities (0.81 g/ml and 0.96 g/ml, respectively), along with the lowest Hausner's ratio (1.18) and Carr's index (15.62%), suggesting better flowability and packing efficiency. These characteristics indicate that Kodo millet starch is more suitable for applications requiring higher packing efficiency and good flowability.Viscosity measurements were conducted using a Brookfield viscometer, where Kodo millet starch exhibited the highest viscosity among the three varieties. At a 4% starch concentration, Kodo millet starch displayed a viscosity of 21.31 centipoise, surpassing foxtail (18.76 centipoise) and little millet (13.28 centipoise).These findings highlight the distinct physicochemical properties of millet starches and provide insights into their potential applications in food processing, packaging, and industrial uses. Kodo millet, with its superior packing and flow properties, shows promise for commercial starch production, while foxtail and little millet can be considered for specialized applications depending on their functional characteristics.

Keywords: Millets, Starch, Nutraceuticals and Isolation of Starch

1 INTRODUCTION

Cereals are staple foods globally, with millets being among the most economically important, especially in semi-arid and tropical regions of Asia and Africa. Millets are drought-resistant, have a short growing season, and thrive in poor soils, making them vital in food security[1]. The four major types of millets include **pearl millet (Pennisetum glaucum)**, **foxtail millet (Setaria italica)**, **proso millet (Panicum miliaceum)**, and **finger millet (Eleusine coracana)**, with India being the leading producer.Nutritionally, millets are comparable to other cereals, rich in protein, dietary fiber, micronutrients, and resistant starch, which contributes to slower digestion and better glycemic control[2]. They are traditionally used in foods such as **injera**, **porridge**, **flatbreads**, and **local beverages** like tela and areki. Environmental factors, post-harvest handling, and processing significantly influence the nutritional quality of millets[3]. Studies have shown wide variations in nutrient composition across millet varieties due to genetic and environmental factors[4]. Millets also serve as raw materials for value-added products including **snacks, beverages, bakery items**, and **fermented and non-fermented foods**.Among the components of millets, **starch** plays a crucial functional and nutritional role[5]. It typically comprises **65–85%** of millet flour and is used as a **thickener, stabilizer, and gelling agent**[6]. Millet starch contains both **amylose (20–30%)** and **amylopectin (70–80%)**, with physicochemical properties that influence its application[7].

Starch and Its Extraction

Starch is a renewable carbohydrate polymer widely used in food and non-food industries due to its availability, low cost, and biodegradability[8]. In millets, starch shows diverse granule shapes and crystalline structures. Starch extraction typically uses **alkaline methods**, which involve sodium hydroxide (NaOH) to release starch granules by breaking protein-starch complexes, followed by ethanol precipitation and separation by centrifugation[9]. This method yields **high-purity starch** suitable for industrial applications[10].

Functional Modification and Analysis

Native starch often requires **modification** to improve its functional properties such as gel strength and stability[11]. This can be achieved through **physical, chemical, enzymatic, or genetic** methods that alter the structure of **amylose and amylopectin**.To analyze starch properties, a **Brookfield viscometer** is commonly used. It measures viscosity based on the torque required to rotate a spindle in the starch sample. The torque is directly proportional to the fluid's viscosity, providing critical data for product formulation in the food industry[12-20].

2. Materials and Methods

2.1 Materials

The foxtail millet, little millet, and Kodo millet used in this study were sourced from Cuddalore. All other chemicals and reagents were procured from local markets.

2.2 Isolation of Starch (Alkaline Extraction Method)

Starch was isolated from the millets using the alkaline extraction method as follows:

1. Initial Extraction

- 100 g of millet grains were dispersed in 1500 mL of distilled water.
- The pH was adjusted to 10 using 1N NaOH and stirred moderately for 1 hour.
- The mixture was centrifuged at 5000 rpm for 30 minutes. The supernatant was discarded.

2. Second Extraction

- The residue was extracted with 1 L of distilled water at 40°C for 24 hours.
- The mixture was centrifuged at 10000 rpm for 30 minutes. The supernatant was discarded.

3. Third Extraction

- The residue was further extracted with 1 L of 2% NaCl solution at 40°C for 24 hours.
- The mixture was again centrifuged at 10000 rpm for 30 minutes. The supernatant was discarded.

4. Fourth Extraction

- The residue was extracted twice with 300 mL of 0.1N NaOH for 48 hours at 40°C.
- The mixture was centrifuged at 10000 rpm for 30 minutes, and the supernatant was discarded. A white starch layer was collected.

5. Purification

- The starch was suspended in 80% ethanol and blended for 1 minute using a stirrer.
- The suspension was heated in a water bath at 80°C for 1 hour.
- After 4 hours of settling at 40°C, the supernatant was discarded.

6. Final Step

- The residue was freeze-dried to obtain starch powder.

2.3 Confirmatory Tests

2.3.1 Iodine Test

To confirm the presence of starch, a few drops of iodine solution were added to the starch powder. The formation of a blue-black complex indicated the presence of starch.

2.4 Physicochemical Characterization

2.4.1 Swelling Power

The swelling power of the starch was determined to assess its water-holding capacity. A 5% w/v starch suspension was prepared at room temperature and shaken for 5 minutes. The sedimentation volume was recorded, and the dispersion was allowed to stand for 24 hours. The swelling capacity was calculated as follows:

$$\textbf{Swelling capacity} = \frac{v2}{v1} \times 100$$

2.4.2 Angle of Repose (θ)

The angle of repose was measured to determine the flow behavior and inter-particulate friction of the starch powder. A glass funnel was clamped 3 cm above a flat surface. 25 g of starch powder was transferred into the funnel, and the orifice was blocked with a thumb. Upon removing the thumb, the powder was allowed to flow and form a pile. The height (h) and radius (r) of the pile were measured, and the angle of repose was calculated using the following formula:

$$\theta = \tan{-1}^{h}/r$$

2.4.3 Bulk Density

The bulk density was determined by introducing 40 g of starch powder into a 100 mL graduated cylinder and recording the volume occupied by the powder. Bulk density (ρ) was calculated as:

$$Bulk\ density(\rho) = \frac{Mass\ of\ Powder}{bulk\ volume}$$

2.4.4 Tapped Density

Tapped density was determined after mechanically tapping the graduated cylinder containing 40 g of starch powder. The cylinder was subjected to 100 tappings, and the final volume was noted. The tapped density was calculated as:

$$Tapped\ density = \frac{Mass\ of\ Powder}{Tap\ Volume}$$

2.4.5 Hausner's Ratio

Hausner's ratio was calculated to evaluate the compressibility of the powder. It is the ratio of tapped density to bulk density:

$$Hausner's\ ratio = \frac{Tapped\ density}{Bulk\ density}$$

2.4.6 Carr's Index

Carr's Index (CI) was used to assess the flowability and compressibility of the starch powder. It was calculated as the difference between tapped and bulk densities, expressed as a percentage of the tapped density:

$$Carr's\ Index = \frac{Tapped\ density - Bulk\ density}{Tapped\ density} X100$$

2.4.7 Viscosity Measurement by Brookfield Viscometer

The viscosity of starch solutions from foxtail millet, little millet, and Kodo millet was measured using a Brookfield viscometer. A 5% w/v starch solution was prepared by mixing the millet starch with distilled water. The solution was boiled for 20 minutes and then cooled to room temperature. The viscosity was measured at 100 rpm using the viscometer, and the readings were recorded in centipoise (cP).

3. Results and Discussion

3.1 Isolation of Starch

The three millet varieties foxtail millet, little millet, and Kodo millet—were processed to isolate starch. The percentage yield of starch obtained from each millet variety was compared, and the results are presented in Table 1.

Table 1 : Percentage Yield of Starch from Different Millet Varieties

S.No	VARIETY OF MILLET	YIELD OF STARCH (%)
1.	Foxtail Millet	68.54
2.	Little Millet	45.35
3.	Kodo millet	74.50

As shown in Table 1, Kodo millet exhibited the highest starch yield (74.50%), followed by foxtail millet (68.54%) and little millet (45.35%).

3.2 Iodine Test

The iodine test was conducted to confirm the presence of starch. A blue-black coloration was observed, which is a positive indication for starch. The test results were consistent with expectations, confirming the successful isolation of starch from the millet varieties.

3.3 Physiochemical Characterization

The physicochemical properties of the starch from the three millet varieties were evaluated, and the results are presented in **Table 2**.

Table 2: Physiochemical Characterization of Starch from Different Millet Varieties

S.No	PARAMETERS	KODO MILLET	FOXTAIL MILLET	LITTLE MILLET
1.	Swellingpower	15.37	16.42	15.83
2.	Angleofrepose **(º)**	25.95	33.75	35.30
3.	Bulk density(g/ml)	0.81	0.60	0.61
4.	Tapped density(g/ml)	0.96	0.73	0.81
5.	Hausner‘sratio	1.18	1.21	1.32
6.	Carr‘sindex	15.62	17.80	24.69

Figure 4: Iodine Test for Starch Presence

3.3.1 Swelling Power

Foxtail millet exhibited the highest swelling power (16.42%), followed by little millet (15.83%) and Kodo millet (15.37%). Swelling power is indicative of starch granule size and strength; higher swelling power typically correlates with larger granules and stronger starch structures. The differences observed can be attributed to variations in starch granule size, shape, and composition among the three millet varieties. Foxtail millet’s larger starch granules likely contribute to its higher swelling power.

3.3.2 Angle of Repose

Little millet exhibited the highest angle of repose (35.30°), indicating poorer flowability. Foxtail millet displayed a moderate angle of repose (33.75°), while Kodo millet had the lowest angle of repose (25.95°), suggesting better flowability. The angle of repose is crucial in assessing powder flowability, where a higher angle implies poor flowability. These results suggest that little millet may need additional processing to enhance flowability for various applications.

3.3.3 Bulk Density

Kodo millet exhibited the highest bulk density

(0.81 g/ml), signifying better packing efficiency. Foxtail millet (0.60 g/ml) and little millet (0.61 g/ml) had lower bulk densities, suggesting poorer packing efficiency. Bulk density is important in powder handling and processing, as it influences flowability and compressibility. Kodo millet's higher bulk density suggests its suitability for applications requiring better packing efficiency, such as food processing.

3.3.4 Tapped Density

Kodo millet also had the highest tapped density (0.96 g/ml), indicating more efficient packing and lower void space. Little millet (0.81 g/ml) and foxtail millet (0.73 g/ml) exhibited lower tapped densities, implying poorer packing efficiency. These findings are aligned with the bulk density data and further suggest Kodo millet's advantage in applications requiring high packing efficiency.

3.3.5 Hausner's Ratio and Carr's Index

Little millet had the highest Hausner's ratio (1.32) and Carr's index (24.69%), suggesting poor flowability and high compressibility. Foxtail millet exhibited a moderate Hausner's ratio (1.21) and Carr's index (17.80%). Kodo millet had the lowest Hausner's ratio (1.18) and Carr's index (15.62%), indicating better flowability and lower compressibility. Both Hausner's ratio and Carr's index are critical parameters for powder handling, and these results suggest that Kodo millet is more suitable for applications requiring better flowability and lower compressibility, such as in food processing.

3.5 Viscosity of Starch

The viscosity of starch solutions from the three millet varieties was measured using a Brookfield viscometer at various concentrations (1%, 2%, 3%, and 4%). The results, as shown in **Table 7**, demonstrate that Kodo millet starch solution exhibited the highest viscosity, followed by little millet and oxtail millet.

Table 7: Viscosity of Starch Solutions by Brookfield Viscometer

S.No	VARIETY OF MILLET STARCH	VISCOSITY(CENTIPOSE)			
		1% SOLUTION	2% SOLUTION	3% SOLUTION	4% SOLUTION
1.	Foxtail	2.5	6.82	12.61	18.76
2.	Little	1.2	4.53	10.25	13.28
3.	Kodo	3.8	8.53	14.92	21.31

The results indicate that Kodo millet starch has the highest viscosity across all concentrations, followed by little millet and foxtail millet. The higher viscosity of Kodo millet starch suggests stronger thickening properties, which could make it suitable for food applications requiring thicker pastes or gels.

CONCLUSION

This study provides a comprehensive analysis of the starch extraction and characterization from three varieties of millet— Kodo millet, Foxtail millet, and Little millet. The results highlighted distinct differences in starch yield, physical properties, and potential industrial applications among the millet varieties. Kodo millet exhibited the highest starch yield and viscosity, making it a promising candidate for use as a thickening agent in various industries.

Additionally, Kodo millet demonstrated superior flowability and packing efficiency, making it an efficient material for manufacturing processes. Foxtail millet, while having a moderate starch yield, exhibited the highest swelling power, indicating its potential in applications requiring rapid water absorption. Little millet, although having a lower starch yield, showed the highest compressibility and poorer flowability, suggesting that it may have applications in areas requiring higher density formulations

. Overall, the study underscores the valuable nutritional and functional properties of millet starches, particularly in the food, pharmaceutical, and cosmetic industries.The findings provide a foundation for further exploration into optimizing extraction processes and understanding the effects of processing conditions on millet starch properties. Future research should aim at improving extraction techniques to enhance yield and explore new applications based on the diverse characteristics of these millets.

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