

## Physico-chemical characterization of lesser known Palo (*Curcuma leucorrhiza*) starch

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### Article history

Received: 21 May 2014  
Received in revised form:  
12 December 2014  
Accepted: 17 December 2014

### Keywords

Palo  
Relative crystallinity  
Gelatinization  
X-ray diffraction  
Differential Scanning  
Calorimeter

### Abstract

Palo (*Curcuma leucorrhiza*) starch is isolated from palo tuber, which is better known as hidden lilly and distributed in the hilly areas of Orissa, West Bengal, Jharkhand and Chhatishgarh. Its rhizomes are good source of starch. The starch extracted from the rhizomes has good application as herbal medicines, different foods and food ingredients such as palo laddu which is used specially during fasting as a part of ritual. It is believed that the consumption of palo starch powder removes the extra heat from the body and develops a resistance against the diseases. The proximate studies showed that the palo contains moisture 15.45% (db), ash 1.10%, fibre 1.23%, starch 81.33% and negligible fat. The scanning electron microscopy showed that curcuma starch consist of large oval to small spherical granules with a diameter ranged between 30- 50  $\mu\text{m}$ . The surface of the granules appeared with evidence of certain fissure which might be due to rubbing action of occurred during starch isolation. X-ray diffraction studies of curcuma starch exhibited a B-type of crystal like other tuber starches and relative crystallinity was found to be 24.66%. Differential scanning calorimetric study showed that the curcuma starch had the gelatinization temperature of 59.5°C and enthalpy of gelatinization 5.565 J/g which might be due to the highest degree of crystallinity of its granules. The final viscosity of extracted starch observed higher than trough viscosity but lower than the peak viscosity which signifying the highest amylose retrogradation.

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### Introduction

Starch, the principal carbohydrate constituent of a majority of plant materials, merits a detailed investigation to understand better its biochemical and functional characteristics as well as variations (El faki *et al.*, 1983). Starches from different sources vary, particularly in their quantitative and qualitative make-up as well as in some of the physicochemical properties. Starch is considered of commercial importance due to its high industrial demand as an ingredient for a variety of processed foods (Whitaker and Tannenbaum, 1977., Wedlock and Williams, 1984). Starch is a mixture of two polymers, amylose, a linear (1 - 4) - linked- $\alpha$ -D-glucose and amylopectin a highly branched molecule which consist of short chains of (1 - 4) - linked- $\alpha$ -D-glucose with (1 - 6) -  $\alpha$ - linked branches. Chain lengths of amylose are commonly in excess of 6000 D-glucopyranose units with a molecular weight between 150000 and 600000 Da (Kennedy *et al.*, 1983). Although amylose was considered to be linear, studies using pullulanase suggest that a small number of branches exist in the molecule (Kennedy *et al.*, 1983). Amylopectin on the contrary, is very branched with an average of 17-26

D-glucosyl units separating the (1 - 6) - $\alpha$ - branch points (Kennedy *et al.*, 1983). The molecular size of amylopectin is almost too large to be determined accurately, but light scattering studies indicate a value of 106 D-glucosyl residues per molecule which makes amylopectin one of the largest naturally occurring macromolecules (Kennedy *et al.*, 1983). All starches are made up of these two polysaccharides. The ratio varies with the starch sources, but is typically 20:80 amylose to amylopectin (Orford *et al.*, 1987). Applications of starch in food systems are primarily governed by gelatinization, gelation, pasting, solubility, swelling and digestibility properties. Relatively a very few studies i.e. only chemical properties of *Curcuma leucorrhiza* locally called Palo starch have been reported (Asem and Laitonjam, 2014).

Palo (*Curcuma leucorrhiza*) starch is isolated from palo tuber, which is better known as hidden lilly and naturally distributed through the hilly areas of Orissa, West Bengal, Jharkhand and Chhatishgarh. Palo is a local name given to *Curcuma leucorrhiza* which belongs to Zingiberaceae family, Zingiberales order, Liliopsida class and Zingiberidae subclass.

It is also cultivated as medicinal crop in many

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parts of these states. Its rhizomes are good source of starch. The natives prepare many herbal dishes using the starch extracted from the rhizomes. Palo laddu is most popular among dishes. It is used specially during observing fast as a part of ritual. It is believed that the Palo removes extra heat from the body and develops resistance against common diseases. The people of the area generally consume the palo for its taste. In tribal belts of Chattishgarh and Orissa Palo is naturally and abundantly available and people prefer its dishes in hot summer. In different parts of the state, many similar methods with slight variations of preparing Palo laddu are adopted. The dry fruits may also be added to make the preparation more delicious and nutritive. The habitant of remote forest areas collect the root from the fields and make starch powder at home but in urban areas the Palo (*Curcuma leucorrhiza*) powder is easily available in shops. The rhizomes of *Curcuma* have been extensively used as traditional medicines in Manipur, northeast India (Sinha, 1996). It is used as an anti-inflammatory and anti-asthmatic in Ayurvedic medicine and rhizomes are used in curing for enlarged liver and spleen, and ulcer in stomach in Shingbhum, Bihar, India (Pal and Srivastava, 1976). It has got numerous pendulous tubers, oblong, inwardly pearl white. Leaves are broad, lanceolate, uniformly green, about two feet long and height of the plant ranges from 3 to 4 feet. The literature available on Palo (*Curcuma leucorrhiza*) is very scarce. After looking these diversified traditional applications, an effort was made to study physico-chemical, morphological and thermal properties of Palo as this information have not been reported so far.

## Materials and Methods

### Starch isolation from Palo

Raw palo tubers were collected from a hamlet situated in Similipal National Park, Odisha, India for starch isolation. Palo starch is generally extracted by traditional way which includes washing, de-rooting, wet grinding, filtration, sedimentation and drying. The hairy roots of the rhizomes are manually removed, washed and ground to paste like consistency. The paste is diluted with water and filtered through fine cloth to separate the skin and other solid materials. The filtrate is kept in an earthen pot overnight below 20°C (to avoid the fermentation) to facilitate sedimentation of suspended particles. The starch particles are settled down, the water is decanted and deposits are dried under the sun (Srivastav *et al.*, 2004). About 10-12 kg of tuber is required to produce 1 kg of Palo (*Curcuma leucorrhiza*) powder by traditional method.

### Proximate analysis of Palo (*Curcuma*) starch

Standard AOAC (1990) methods were used for the measurement of the moisture, ash, fiber, fat, protein, pH and % of total starch. pH values were determined electrometrically on a suspension of 2 g dry solids in 65 ml of distilled water. Fat was determined by extracting oil from 5 g of sample in a thimble using Soxhlet apparatus. Crude protein content was determined by kjeldhal method, taking 5.7 as protein factor.

Total starch was determined by estimating reducing sugar content of the sample. Reducing sugar was determined by Shaffer Sumoygi Method (Ranganna, 2003). Total starch was calculated by following formula

$$\text{Total starch, (\%)} = \text{reducing sugar (\%)} * 0.9 \quad (1)$$

### Physical properties of curcuma starch

#### Density

Physical properties such as bulk density and true density were determined by multivolume pycnometer 1305. This instrument accomplishes the measurement of skeletal volumes by observing the variation of gas capacity in the sample chamber caused by the presence of the sample. Since, helium or most other suitable gases penetrates even the smallest pores and surface irregularities, the volume obtained permits computation of the ultimate theoretical density of the solid comprising the sample if there are no closed pores. The general equation for computing the sample volume ( $V_s$ ) is

$$V_s = \frac{V_c - V_e}{\left(\frac{P_1}{P_2} - 1\right)} \quad (2)$$

Where,  $V_c$ ,  $V_e$ ,  $P_1$ ,  $P_2$ , are volume of the sample cell, expansion volume added when the PREP test valve is rotated to TEST, charge pressure recorded and pressure after expansion respectively. Then the sample density is calculated by

$$\rho_s = \frac{W_s}{V_s} \quad (3)$$

Where  $P_s$ ,  $W_s$ ,  $V_s$  are density, weight and volume of the sample respectively. Specific volume is defined as the volume per unit weight and is computed as

$$U_s = \frac{1}{\rho_s} \quad (4)$$

Where,  $U_s$  and  $P_s$  are specific volume and density respectively

### Granule morphology

Microstructural characterization was accomplished

using scanning electron microscope (JEOL JSM-5800, JEOL Japan) coupled with an energy dispersive X-ray micro-analytical system (OXFORD ISIS300). Scanning Electron Microscopic (SEM) analysis was carried out to determine morphology of the curcuma starch. Starch samples were suspended in ethanol to obtain a 1% suspension. One drop of the starch ethanol suspension was applied on an aluminum stub using double-sided adhesive tape and the starch was coated with gold sputter. An accelerating potential of 20 kV was used during micrography.

#### *X-ray diffraction (XRD) analysis.*

X-ray diffraction analysis was carried out to confirm the phase identification of the sample as well as for percent crystallinity of the sample. Powder patterns were measured on a RIGAKU miniflex machine (Rigaku-USA) fitted with X-ray generator and a diffractometer control unit using Ni-filtered CuK  $\alpha$  radiation. Scanning was done in the  $2\theta$  range of 3 to  $40^\circ$  at a rate of  $2^\circ/\text{min}$ . Per cent crystallinity was calculated by measuring the crystalline area and amorphous area i.e.

$$X_c = \frac{A_c \times 100}{A_c + A_a} \quad (4)$$

Where,  $X_c$ ,  $A_c$ ,  $A_a$ , percent crystallinity, crystallinity area, and amorphous area respectively.

#### *Thermal analysis*

Thermal characteristics of curcuma starch was studied by using a differential scanning calorimeter-DSC204, HP (NETZSCH, Germany) equipped with a thermal analysis station. Curcuma starch (3.5 mg, dry weight) was loaded into a 40  $\mu\text{l}$ , capacity aluminum pan and distilled water was added to achieve a starch-water suspension containing 80% water. Samples were hermetically sealed and allowed to stand for 1 h at room temperature before heating in the DSC. Sample pans were heated at a rate of  $5^\circ\text{C}/\text{min}$  from 25 to  $100^\circ\text{C}$ . Onset temperature ( $T_o$ ), peak temperature ( $T_p$ ), conclusion temperature ( $T_c$ ) and enthalpy of gelatinization ( $\Delta H_{\text{gel}}$ ) were calculated automatically. The gelatinization temperature range (R) was computed as ( $T_c - T_o$ ) as described by Vasanthan and Bhatti (1996) and gelatinization parameters for native and retrograded starch were also evaluated and described by Chavan (1999) and Hoover (2001)

#### *Viscosity of extracted Curcuma starch*

Viscosity of palo was studied by Rapid Visco Analyser (RVA 4500, Perten, Sweden) to analyse the change in viscosity with temperature. About 3.0 g of

traditionally extracted curcuma starch was suspended in 25 ml of distilled water and equilibrated at  $40^\circ\text{C}$  for 1 min. This sample was heated to  $90^\circ\text{C}$  at a heating rate of  $5^\circ\text{C}/\text{min}$ . From the pasting curve, Peak viscosity, Trough viscosity, Breakdown viscosity and Final viscosity were found out.

## Results and Discussion

#### *Proximate composition.*

Some basic properties namely moisture, ash, crude fiber, crude protein and of total starch content of the curcuma starch was determined and numerically found to be 15.45% (db), 1.10%, 0.83%, 0.40% and 81.33%. It was also found that fat content is almost negligible.

#### *Physical Properties*

Physical properties like sample density, sample volume, specific volume and morphological characteristic by SEM, X-ray diffraction pattern of the starch and relative crystallinity of the sample. Sample density, sample volume and specific volume of the curcuma starch- Sample density, sample volume and specific volume of the curcuma starch were determined by multivolume pycnometer 1305 and found to be  $1551.6 \text{ kg}/\text{m}^3$ ,  $25.65 \text{ m}^3$  and  $0.6444 \text{ m}^3/\text{kg}$  respectively.

#### *Morphological study of Palo (curcuma) starch*

Scanning electron microscopic (SEM) study was conducted to characterize morphological pattern of the curcuma starch. SEM showed the presence of large ovule to small spherical shape granules with diameter ranging between 30 – 50  $\mu\text{m}$ . The surface of the granules appeared with evidence of certain fissure which might be due to rubbing action during the starch isolation. Figure 1 shows a typical SEM of curcuma starch.

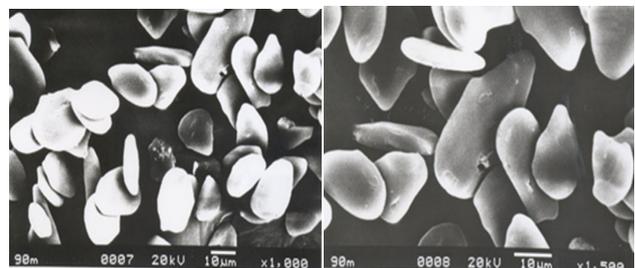


Figure 1. SEM image of the curcuma starch (a) at 1000 times resolution and (b) at 1500 times resolution

#### *X-ray diffraction analysis*

XRD was conducted to characterize the crystal types of curcuma starch to which it belong. Generally starch is categorized into three, i.e. A-type, B-type

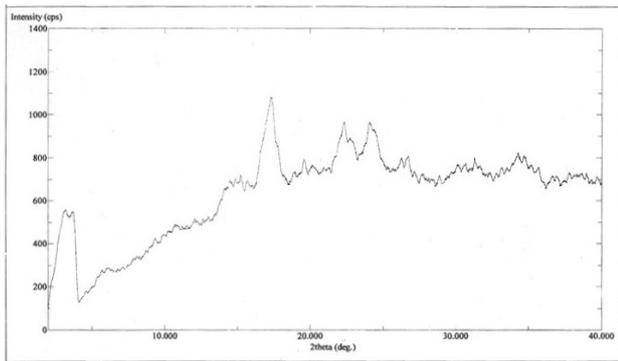


Figure 2. Diffractogram of curcuma starch

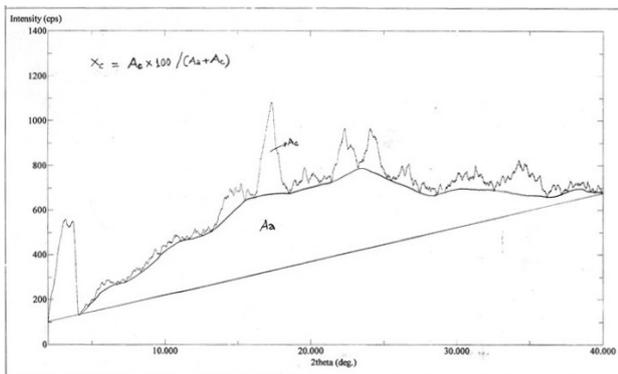


Figure 3. Diffractogram showing relative crystallinity

and C-type depending upon the arrangement of crystals. The X-ray diffractogram of curcuma starch is given in Figure 2 and 3 which show that there is a strong peak at  $2\theta$  value of  $17.34^\circ$  and a few small peaks at  $22.3$  and  $24^\circ$  of diffraction angle  $2\theta$ . Curcuma starch is showing strong peak at  $17.34^\circ$  have typical B type crystal like other tuber starches. Relative crystallinity was quantitatively estimated by drawing a smooth curve, which connected the peak baseline, was plotted on the diffractograms, The area above the smooth curve was taken to the crystalline portion, and the lower area between the smooth curve and a liner base line, which connected the two points of intensity at  $2\theta$  of  $3.5^\circ$  and  $40^\circ$ , was taken as the amorphous section. The upper diffraction peak area and total diffraction area over the diffraction angle  $3.5^\circ - 40^\circ$  is plotted on log paper. The ratio of the upper area to the total diffraction area was taken as the relative crystallinity. Relative crytallinity of curcuma starch is found to be 24.66% (Figure 3).

#### Thermal properties of Palo (Curcuma) starch

DSC was conducted to know the gelatinization temperature and enthalpy of gelatinization of the curcuma starch. Thermal characteristics of isolated starch were studied by using a differential scanning calorimeter. The Figure 4 and 5 revealed that gelatinization temperature of curcuma starch was found to be  $59.5^\circ\text{C}$ , which is somewhat similar to

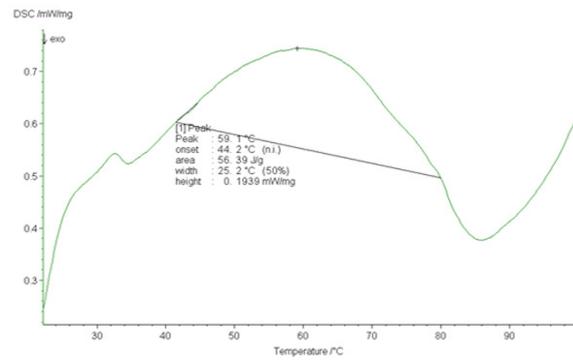


Figure 4. Thermograph of curcuma starch at 80% water (w/w)

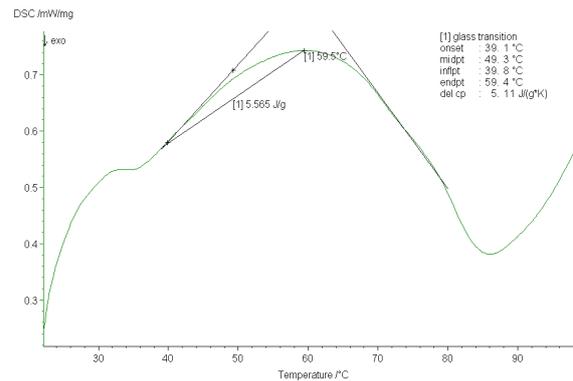


Figure 5 Thermograph of curcuma starch at 80% water (w/w) showing enthalpy of gelatinization

the gelatinization temperature of potato starch and low compared to starch from sweet potato, tania and yam (Tian *et al.*, 1991; Veletudieet *et al.*, 1995), The enthalpy of gelatinization 5.565 J/g and occurrence time of 6.8 min. This may be due to the highest degree of crystallinity of its granules. The gelatinization temperature and enthalpy of the starches depends on the microstructure and degree of crystallinity within the granule and also on granule size and the amylose to amylopectin ratio. Normally the smaller the granule the higher will be the gelatinization temperature (Cowburn, 1989., Fasihuddin *et al.*, 1999).

#### Viscosity of the palo starch

The pasting properties of starch are highly dependent on the size, form, coherence, and degree of swelling of starch granules. It is also related to the heat and shear stability of the swelled granules as well as the structural difference in amylose and amylopectin (Lee *et al.*, 2001). Viscosity of Palo sample was studied by Advanced Rheometer 1000 and curve showed in Figure 6.

Peak viscosity indicates water-holding capacity of the starch, which exhibits the resistance of the swollen granules against the shear force and swelling degree of starch granules (Dengate, 1984). It has been reported that starch granules with high swelling power occupied more volume within water suspension thus the space among the granules became narrower, and

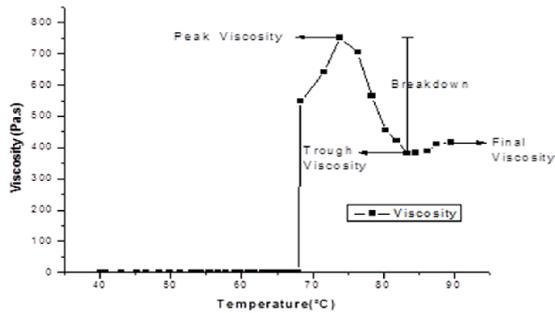


Figure 6. Viscograph of *palo* starch frictions among granules likely to occur (Kim *et al.*, 1999). The breakdown is regarded as the measure of degree of disintegration of granules or paste stability (Dengate, 1984). At breakdown, the swollen granules disrupt further and amylose molecules will generally leach out into the solution. The final viscosity of extracted starch observed higher than trough viscosity but lower than the peak viscosity which signifying the highest amylose retro-gradation. However, the numerical values of peak, trough, breakdown and final viscosity were found to be 750.5, 380.7, 369.8 and 415.6 Pa.s respectively.

## Conclusion

The Proximate analysis of *Curcuma leucorrhiza* showed that it has high amount of starch and negligible fat. Ash content and fibre content are somewhat similar to starch material obtained from other tubers. The average granule size of curcuma starch was from small to large and round or oval. Its diameter was around 30 - 50µm. Curcuma starch showed strong peak at 17.34° have typical B type crystal like other tuber starches. Relative crytallinity of curcuma starch was found to be 24.66%. The curcuma starch had the gelatinization temperature of 59.5°C and enthalpy of gelatinization 5.565J/g which might be due to the highest degree of crystallinity of its granules.

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