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Changes Induced by Modification on the Rheological Properties of *Icacina trichantha* Starch

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Abstract

This research aimed at investigating the potentials of a non-food source of starch for use in food and industrial application. It will reduce competition for sources of food which are also used as sources of starch for the industry. In this work starch from *Icacina trichantha* tubers (under-utilized crop) were isolated and the purity level determined. The native starch was chemically modified using acid (HCl), alkaline (NaOH) and oxidized using H_2O_2. Functional properties, pasting properties were determined and photomicrographs of the starch samples were obtained. Bulk density of native and modified starches ranged from 0.56 to 0.78; pH 3.50 to 8.86; Gelatinization temperature 60 to 80°C; water holding capacity 64.0 to 72.0; swelling power 6.71 to 10.71; wettability 07.47 to 18.94 and solubility 14 to 37. The peak viscosity ranged from 143.0 to 2965.0 RVU. The native starch had the highest peak viscosity (at a temperature of 84.10°C in 4.53 min) and acid treated had the lowest (at 85.70°C in 4.33 min). The breakdown viscosity ranged from 120.0 to 1291.0 RVU with the alkaline treated starch having the highest while acid treated starch had the lowest. The final viscosity ranged from 81.0 to 2066.0 RVU. Alkaline treated starch had the highest value while acid treated starch had the lowest value. The setback viscosity ranged between 58.0 and 737.0 RVU with the alkaline treated starch having the highest value and acid treated starch having the lowest value. The pasting temperature ranged from 84.10 to 85.90°C. The photomicrographs showed that the starch granules in all the samples had irregular shape, generally small to medium sized with sizes ranging from 30 to 110nm and granular frequency ranging from 45 to 183. *Icacina trichantha* starch resembled conventional food starches, and will compete favourably with them in food systems as an industrial starch.

1. Introduction

The increasing use of native and modified polysaccharides in food processes and other industrial applications has resulted in a growing interest in starch as a renewable and environmentally compatible polymer source. Starch has a wide range of food applications [1] and can be modified for improved use in the food industry [2, 3] However, native starch may not be satisfactorily applied in industrial processes [1]. The physicochemical properties of starch will determine its industrial application [5]. The biological origin determines the morphology and physicochemical characteristics of starch [6]. Through modification processes, native starch properties are altered and their

industrial applications are diversified [7]. Also, the wide applications of starch nowadays in food processing and other industrial purposes is seriously competing with the basic conventional food products in the globe thereby reducing drastically the amount of starch to be consumed by the large teaming population of the world.

For this reason it is necessary to seek for alternative sources of starch. *Icacina trichantha* is an underutilized crop that presently grows in the wild and contains significant amounts of starch in its tubers. Its potentials as a source of industrial starch can be explored.

The objectives of the study were; to extract and determine the purity of starch from *Icacina trichantha* tuber; chemically modify the native starch using acid, alkaline, and oxidation treatments; evaluate the rheological and functional properties of the native and the modified starches and to investigate the effect of modification on native *Icacina trichantha* starch using microscopy.

2. Materials and Methods

2.1. Material

Icancina trichantha is an underutilized plant shown in figure 1.

Figure 1. *Icacina trichantha plant.*

2.2. Preparation of Native *Icacina trichantha* Starch

Icacina trichantha starch was prepared following the method of Moorthy *et al.* [8] with slight modification. About 20kg of the *Icacina trichantha* tubers were washed, peeled, washed in tape water and disintegrated in a grating machine (wet milling). The resulting *Icacina trichantha* mash was mixed with water in the ratio 1:5 (w/v %). The mash was filtered through sieve 44, and 80µm mesh size respectively to obtain pure starch solution. The starch was carefully separated from the water by sedimentation and decantation. The process of sedimentation and decantation was done repeatedly until a clean starch solution was obtained. The starch was spread thinly on an aluminium foil covered tray of a hot air oven to dry at 40°C for 10 hour and was further dried at 60°C for another 6hours. The native starch was pulverized, weighed and stored in sample container for analysis. The percentage yield was calculated as:

$$\%\ \text{Percentage yield} = \frac{\text{Weight of starch isolated}}{\text{Weight of mashed tuber}} \times \frac{100}{0}$$

2.3. Test for Starch Purity

Test for starch purity was carried out by determining the ash, protein and fat according to AOAC methods [9].

2.4. Starch Modificaion

2.4.1. Preparation of Acid-modified Starch

The method described by Atichokudomchai and Varavinit [5] was employed with slight modification. Three hundred grams (dry basis) of native *Icacina trichantha* starch were hydrolyzed by incubating the starch in 600 mL 6% HCl at 28°C for 24 hours without stirring. The suspension was neutralized with 10% (w/v) NaOH solution. The starch slurry was washed five times with distilled water and dried in a hot air oven at 60°C for 8 hours. The starch was pulverized and passed through a sieve of 80µm mesh size.

2.4.2. Preparation of Alkaline-Modified Starch

The method described by Wang and Wang [6], was used with slight modification. Three hundred grams (300g) of the native *Icacina trichantha* starch was soaked in 600ml of 0.1% sodium hydroxide (NaOH) solution for 24h at room temperature (28°C), and was neutralized with 0.1M of hydrochloric acid (HCl) to pH 6.5, and was allowed to settle. The starch was then washed with distilled water for five times and was dried in a hot air oven at 60°C for 8h. The starch was pulverized and passed through sieve 80µm mesh size.

2.4.3. Preparation of Oxidized Starch

Hydrogen peroxide oxidation of starch was carried out as described by Parovuori *et al*. [7] with slight modifications. The native *Icacina trichantha* starch was slurred in distilled water containing 40% dry solid and the pH was adjusted to 10 with NaOH solution. The temperature of the slurry was maintained at 28°C. Copper sulphate (0.1% based on starch) was added as a catalyst and hydrogen peroxide solution was added drop-wise over a period of 15 minutes to the reaction mixture to reach a final concentration of 3% (based on starch). During the addition of reagent and the course of reaction, pH of the slurry was maintained at 10 with NaOH solution. The mixture was stirred for 30 minutes (at a stir rate of about 5 stirs per minute) and the reaction was terminated by addition of sodium bisulphite. Subsequently, the pH was adjusted to 6.5–7.0 using HCl. The sample was thoroughly washed with distilled water for five times and was dried in a hot air oven at 60°C for 8h. The starch was pulverized to pass through sieve of 80µm mesh size.

2.5. Determination of Functional Properties

Bulk Density, wettability and gelatinization temperature were determined according to the method of Attama *et al*. [8].

Water holding capacity and pH were determined according to the method of Omojola *et al*. [9]. Swelling power was determined by the method of Daramola and Osanyinlusi [10]. The swelling power was calculated as follows:

$$\text{Swelling power} = \frac{\text{Weight of swollen sediment}}{\text{Weight of dry starch}}$$

Solubility was determined in accordance with the method described by Adebowale *et al.* [11]. Solubility of the starch samples was calculated as follows:

$$\text{Solubility} = \frac{\text{Weight of dry supernatant}}{\text{Weight of starch sample}} \times 100$$

2.6. Rheological Properties of Starch

The rheological or pasting characteristics were determined with a Rapid Visco Analyzer (RVA), (Model RVA3D+, Network Scientific, Australia). First, flour samples (2.5g) were weighed into a dried empty canister; then 25ml of distilled water was dispensed into the canister containing the sample. The solution was thoroughly mixed and the canister was fitted properly into the RVA. The slurry was heated from 50°C to 95°C with a holding time of two minutes followed by cooling to 50°C with 2 minutes holding time. The rate of heating and cooling was at a constant rate of 11.25°C per min. Peak viscosity, trough, breakdown, final viscosity, set back, peak time, and pasting temperature were read from the pasting profile with the aid of thermocline for windows software connected to a computer [12].

2.7. Starch Granule Morphology

Starch granule morphology of native starch from *Icacina trichantha* was studied using scanning electron microscopy (SEM). One gram of dry starch was suspended in 10ml of water at 25°C and gently stirred for 5 min. Starch samples were stained with crystal eosine solution, mounted on circular aluminium stubs using adhesive and then coated with thin layer of gold using Bio-Rad Sputter coating System. The samples were examined and photographed in a Joel scanning microscope (JSM-6400, TOKYO, Japan) at an accelerated voltage of 5kv and a magnification of X100 and X400. Twenty (20) granules were selected randomly and their size measured using ruler.

Statistical analysis

All data were subjected to Analysis of Variance (ANOVA) using SPSS version 16.00 and means were separated using Duncan's Multiple Range Test (DMRT).

3. Results and Discussions

3.1. Starch Purity

The dry matter content of prepared starch was 96.8%. Impurity detected as crude protein was 0.20% while ash was detected as 1%. The purified starch contained 2% moisture. No fat was detected in the sample.

3.2. Functional Properties

Table 1. *Functional properties of Icacina trichantha starch samples.*

Sample	Bulk density (g/ml)	pH	Gel. Temp. (°C)	Water holding capacity (ml)	Swelling power	Wettability (s)	Solubility (%)
NIS	0.65^{a}	6.60^{b}	60^{d}	64.0^{b}	8.41^{b}	15.46^{b}	17^{c}
AIS	0.56^{a}	3.50^{d}	65^{c}	70.4^{a}	6.71^{c}	10.02^{c}	27^{b}
BIS	0.66^{a}	8.86^{a}	75^{b}	64.0^{b}	10.71^{a}	07.47^{d}	14^{c}
OIS	0.78^{a}	4.99^{c}	80^{a}	72.0^{a}	4.95^{d}	18.94^{a}	37^{a}

Means followed by the same superscript in a column are not significantly different at $p<0.05$

Where NIS = unmodified (native) *Icacina trichantha* starch; AIS = acid modified *Icacina trichantha* starch; BIS = alkaline modified *Icacina trichantha* starch; OIS = oxidized *Icacina trichantha* starch

The functional properties of *I trichantha* starch are shown in Table 1. pH ranged from pH 3.50 in acid modified starch to pH 8.86 in alkaline modified starch. The pH of acid modified, oxidized and native starches were in the acidic pH region. Modified starches had higher gelation temperatures than native *I trichantha* starch. There were no significant differences ($p>0.05$) in bulk density of native and modified starches. Values ranged from 0.56 (AIS) to 0.78 (OIS). Oxidized starch had the highest gelation temperature. Water holding capacity of native starch was comparable to that of alkaline modified starch, while the water holding capacity of oxidized starch was the highest, but not significantly different from the water holding capacity of acid modified starch. Alkali modified starch (BIS) had the highest swelling power (10.71) which was about thrice the swelling power of oxidized *I trichantha* starch (4.95). The swelling power of native starch (8.41) was significantly higher ($p < 0.05$) than the swelling power of acid modified starch (6.71). Wettability value was highest for oxidized starch (18.94) and lowest for alkaline modified starch (7.47). Native starch had higher wettability (15.46) than acid modified starch. There were significant differences ($p<0.05$) in the wettability of all starch samples. Solubility of the oxidized starch was highest (37) and significantly different ($p < 0.05$) from the other samples. Acid modified starch was more soluble ($p< 0.05$) than native and alkaline modified starches. The values obtained for swelling of *I trichantha* native and modified starches were higher than those observed for legume starches possibly because legumes are naturally different and contain more proteins than tubers [2]. Functional properties of cereal starches were also found to be lower than those observed for tuber starches [3].

3.3. Pasting Properties of *I. trichantha* Starch

The pasting properties of the starch samples are shown in table 2.

Table 2. *Rheological properties of native and modified Icacina trichantha starch samples.*

Sample	Peak 1 (RVU)	Trough (RVU)	Break down (RVU)	Final viscosity(RVU)	Setback (RVU)	Peak time (min)	Pasting temp (°C)
NIS	2965.0[a]	1280.0[b]	1685.0[a]	1919.0[a]	637.0[a]	4.53[a]	84.10[a]
AIS	143.0[d]	23.0[d]	120.0[c]	81.0[b]	58.0[b]	4.33[a]	85.70[a]
BIS	2620.0[b]	1329.0[a]	1291.0[b]	2066.0[a]	737.0[a]	4.67[a]	84.85[a]
OIS	262.0[c]	66.0[c]	148.0[c]	150.0[b]	84.0[b]	4.20[a]	84.90[a]

Means followed by the same superscript in a column are not significantly different at $p<0.05$
Where RVU= Rapid viscosity unit; NIS = Unmodified (native) *Icacina trichantha* starch; AIS = Acid modified *Icacina trichantha* starch; BIS = Alkaline modified *Icacina trichantha* starch; OIS = Oxidized *Icacina trichantha* starch.

3.4. Pasting Properties of *Icancina trichantha* Starch

The rheological properties of native and modified *I trichantha* starch are shown in Table 2. The pasting temperatures and the periods for attaining peak viscosities were not statistically different for the native and modified starches at 95% confidence interval. Peak viscosity was highest ($p<0.5$) for native *I trichantha* starch ($p<0.05$) followed by the alkaline modified starch. The acid modified starch had the lowest peak viscosity value. The modification process profoundly influenced the peak viscosity values of samples. Oxidation and acid modification markedly reduced the peak viscosity of I *trichantha* starch. Acid modified *I trichantha* starch was the most stable followed by the oxidized starch. Breakdown values were significantly higher ($p<0.05$) for native {NIS) and alkaline (BIS) modified starches. This implies that NIS and BIS have low resistance to heat treatment. It also suggests that the freeze-thaw stability of *I trichantha* starch modified using acid and oxidation treatments will be better than that of other modified starches [18] [19]. Rheological properties of starch showcase their areas of optimized utilization in food systems [20]. NIS and BIS could serve as good humectants. Chemical modification of starches may be achieved by mild reactions between the hydroxyl group of the native starch and the modification agent. It may involve mild or moderate oxidation, mono or poly functional esterification, alkaline gelatinization or combinations of these treatments [21]. In the process, physical and functional properties of starch are modified to produce desirable properties. Weak internal bonding is indicated by high swelling as observed in NIS and BIS. Acid modified starches generally possess lower hot paste viscosity as observed in AIS. Chemicals like sodium sulphate and urea increase the gelatinization temperature of starch. Amylose retrogrades more rapidly than amylopectin. During retrogradation new hydrogen bonds are formed and the starch starts to develop a new crystalline structure, quite different from the crystalline structure that was lost during birefringence.

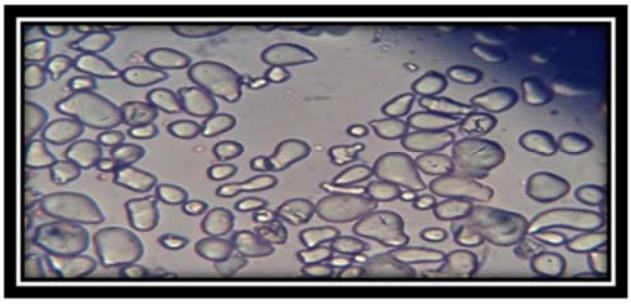

Figure 2. *Native Icancina trichantha starch.*

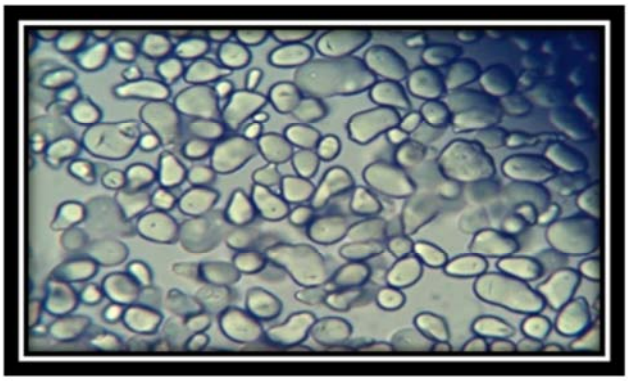

Figure 3. *Acid modified Icancina trichantha starch.*

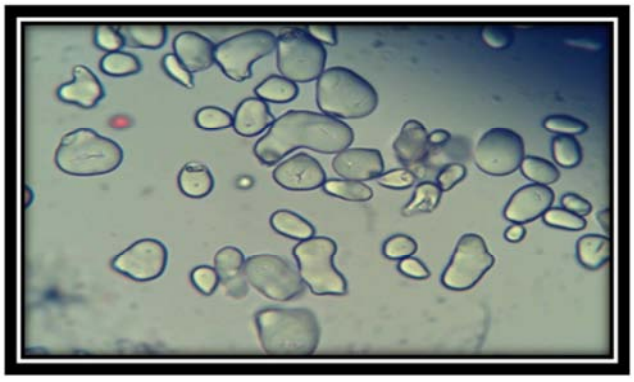

Figure 4. *Alkaline modified Icancina trichantha starch.*

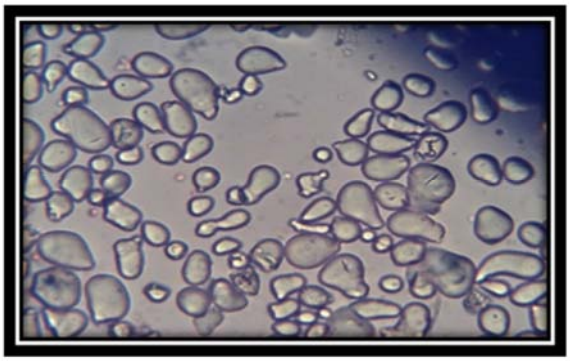

Figure 5. *Oxidized Icancina trichantha starch.*

Photomicrographs of native and modified starch granules are shown in Figures 2, 3, 4 and 5.

Table 3 shows the microscopic features of native and modified *I trachantha* starches.

Table 3. *Microscopic features of the native and modified Icacina trichantha starch samples.*

Sample	Size range (nm)	Mode (nm)	Granular frequency	Mean (nm)
NIS	30-110	80	105	62.50
AIS	30-110	50	132	67.00
BIS	30-100	100	45	70.00
OIS	50-110	60	183	71.50

Where NIS = Unmodified (native) *Icacina trichantha* starch; AIS = Acid modified *Icacina trichantha* starch; BIS = Alkaline modified *Icacina trichantha* starch; OIS = oxidized *Icacina trichantha* starch

3.5. Microscopy of *Icacina trichantha* Starch

The size ranges for native and modified starches were the same (30 to 110 mm) except for alkaline modified *I trachantha* starch (BIS) that had smaller dimensions (30 to 100mm). BIS also had the lowest granular frequency of 45, the highest mode and one of the highest means. Granule size has a profound effect on the swelling and gelatinization characteristics of starch. The size ranges denote variable granule sizes and non- uniform gelatinization temperatures for both native and modified starches. Observed results also indicate that alkaline treatments (BIS) restricts swelling in *I trichantha* starch. Oxidized and alkaline modified starch granules were more aggregated than acid modified and native starch granules.

3.6. Viscosity Profiles for Native and Modified Starches

Figures 6, 7, 8 and 9 show the viscosity profiles of natïve and modified *I trichantha* starch. There was a broad range of gelatinization temperature for all the starches indicating that the starch granules were not of uniform sizes.

The gelatinization temperature range of native starch granules was however slightly higher than that of modified starches.

The viscosity profile of alkaline modified starch was more similar to that of native *I trichantha* starch in terms of the lowest viscosity attained, while oxidized and acid modified starches showed similar trend in their trough

Viscosity patterns were howbeit different for all the starch samples indicating that each modification process conferred peculiar qualities on the starches and that the starches will possess different functional properties in food systems. Omojolola *et al.* [14] had observed that citrate modification of *I trichantha* starch improve its swelling and water absorption capacities making the starch more useful for the pharmaceutical industry. High swelling of oxidized and alkaline modified starches suggest increased digestibility and solubility. It also suggests that starches modified using these methods will have improved dietary properties.

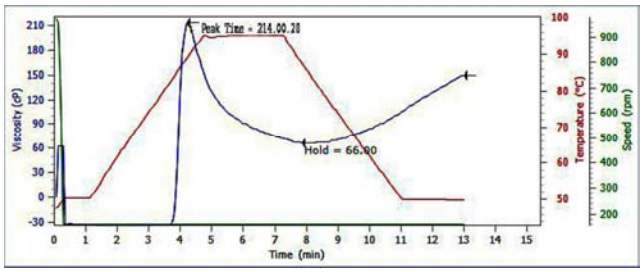

Figure 6. *Viscosity profile of oxidized I trichantha starch.*

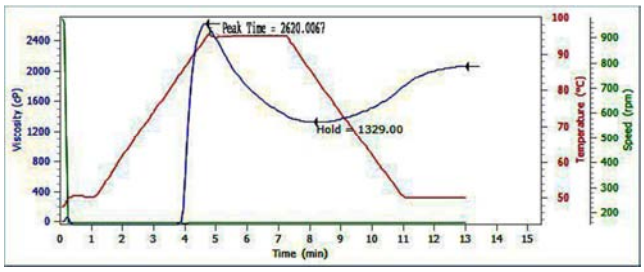

Figure 7 *Viscosity profile of Alkaline modified I trichantha starch.*

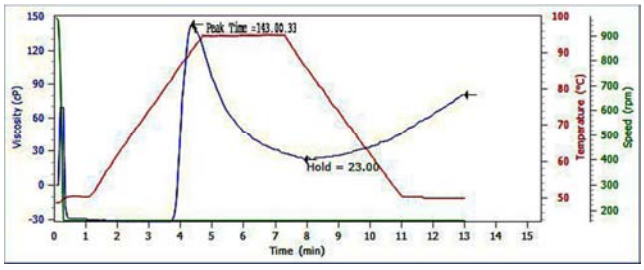

Figure 8. *Viscosity profile of Acid modified I trichantha starch.*

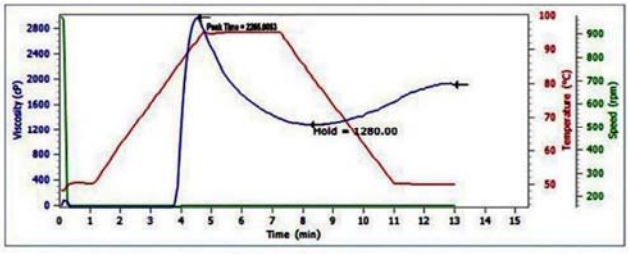

Figure 9. *Viscosity profile of native I trichantha starch.*

4. Conclusion

Findings from this research indicate that *Icacina*

trichantha is a potential source of starch which can be modified for use in food and other industrial applications. Alkaline modification process makes the starch useful as humectants.

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