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## TACCA starch citrate - A potential pharmaceutical excipient

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### ABSTRACT

Starch extracted from *tacca involucrata* was modified by preparing its citrate derivative by reacting the *tacca* starch with citric acid at elevated temperatures. The starch citrate was found to be a white, crystalline and non – hygroscopic powder with a percentage yield of 75.7 %. It was insoluble in water and had a swelling capacity of 11.9 at 85 °C in water. It did not gelatinize when heated at 100 °C in water for 30 min. It has a browning temperature of 223.5.0 – 231.2 °C, charring temperature of 251.7 – 258.9 °C, water absorption capacity of 83.25 %, pH of 4.7, foam and emulsion capacities of 4.2 % and 7.8 % respectively. The photomicrograph shows that the starch citrate granule is oval in shape, generally small sized with occasional large ones. Generally, the physicochemical properties of *tacca* starch citrate indicate that it might be a better disintegrant than native *tacca* starch in tablet formulations.

**Keywords:** *Tacca Involucrata*, physicochemical properties, starch citrate, swelling, disintegrant.

### INTRODUCTION

Starch is a natural biodegradable biopolymer which has wide industrial applications. Its high industrial demand requires that cheaper sources should continually be sought to meet global needs. Amura plant (*tacca involucrata*) is a crop found in the family of Taccaceae of the genus *tacca*, mostly found in the northern parts of Nigeria. Starch from the plant has shown outstanding physico-chemical properties and utilization as a disintegrant in tablet formulations (Kunle *et al*, 2003). The starch yield is about 30 % and the tuber is non – competitive as an edible crop (Zaku *et al*, 2009).

However, it has been shown that when native starch is modified, it generally shows better paste clarity, better stability, increased resistance to retrogradation and increased freeze – thaw stability (Zheng *et al*, 1999). Starch citrate has also been reported as a resistant starch in food industry, (Xueju *et al* 2004).

According to Chowdary and Enturi (2011), starch citrate derivatives exhibit good swelling in water and are a better promising disintegrant in tablet formulations than their parent starch.

The aim of the present study is to prepare and evaluate starch citrate, a chemically modified starch basically as a potential pharmaceutical excipient with an outstanding value addition compared with the native starch.

## MATERIALS AND METHODS

### Materials:

Tacca starch, citric acid and other analytical grade reagents were obtained from Chemistry Advanced Laboratory, Sheda Science and Technology Complex, Abuja Nigeria.

### Preparation of starch citrate

Preparation of tacca starch citrate was based on the method of Chowdary *et al* (2011) with some modifications. Citric acid (20 g) was dissolved in 20 ml of distilled water, the pH of the solution was adjusted to 3.5 with 10 M sodium hydroxide solution and finally the solution was made up to 50 ml by adding distilled water. The citric acid solution (50 ml) was mixed with 50 g of starch in a glass Petri-dish and conditioned for 16 h at room temperature (28 °C). The tray was then placed in a forced air oven and dried at 60 °C for 6 h. The mixture obtained was ground and further dried at 130 °C for 2 h. The dry mixture was repeatedly washed with water to remove unreacted citric acid. The washed starch citrate was further dried at 50 °C to remove the water / moisture completely. The product obtained was hence ground, weighed and stored in sample bottles for analysis.

### Determination of certain physicochemical properties

#### Swelling power

The method described by Daramola *et al* (2006) was used to determine the swelling power with slight modifications. The starch sample (0.1 g) was weighed into a test tube and 10 ml of distilled water was added. The mixture was heated in a water bath at a temperature of 50 °C for 30 min with continuous shaking. In the end, the test tube was centrifuged at 1500 rpm for 20 min in order to facilitate the removal of the supernatant which was carefully decanted and weight of the starch paste taken. The swelling power was calculated as follows:

$$\text{Swelling power} = \frac{\text{Weight of starch paste}}{\text{Weight of dry starch sample}}$$

This was carried out over a temperature range of 50 °C – 100 °C.

**Gelatinization temperature**

This was evaluated using the method of Attama *et al* (2003). 1 g of the starch sample was put in a 20 ml beaker and 10 ml of distilled water was added. The dispersion was heated on a hot plate. The gelatinization temperature was then read with a thermometer suspended in the starch slurry.

**Water holding capacity**

The method of Omojola *et al* (2010) was used with slight modifications. 5 % w/v of the starch sample was dispersed in a pre-weighed centrifuge tube. The tube was agitated in a vortex mixer for 2 min. The supernatant was then discarded and the weight of the tube and hydrated sample taken. The weight was calculated and expressed as the weight of water bound by 100 g dry starch.

**Foam capacity**

The method of Omojola *et al* (2010) was used with slight modifications. 1 g of starch sample was homogenized in 50 ml distilled water using a vortex mixer (vortex 2 Genie set at shake 8) for 5 min. The homogenate was poured into a 100 ml measuring cylinder and the volume recorded after 30 s. The foam capacity was expressed as the percent increase in volume.

**Emulsion capacity**

The method of Omojola *et al* (2010) was again used also with slight modifications. 1 g sample was dispersed in 5 ml distilled water using a vortex mixer for 30 s. After complete dispersion, 5 ml vegetable oil (groundnut oil) was added gradually and the mixing continued for another 30 s. The suspension was centrifuged at 1600 rpm for 5 min. The volume of oil separated from the sample was read directly from the tube. Emulsion capacity is the amount of oil emulsified and held per gram of sample.

**Browning and charring temperature**

The method of Builders *et al* (2001) was used. Some of the starch sample was put into a capillary tube, the browning and charring temperatures were determined using a melting point apparatus with model name Electrothermal 9100.

**Proximate analysis:**

Moisture, protein, fat, ash, crude fibre and carbohydrates was determined according to AOAC (1990).

**pH**

A 20 % w/v dispersion of the sample was shaken in water for 5 min and the pH was determined using a pH meter.

All the above parameters were determined in triplicates and the mean and standard deviations were recorded.

**Scanning electron microscopy**

A scanning electron microscope (SEM) model EVO/ MA 10 was used to determine the particle size at 500X, 1KX and 2KX magnifications. The sample was imaged with the secondary

electron detector at an accelerating voltage of 20 kV, probe current of 200 PA and variable pressure of 50 Pa.

#### **Phytochemical screening:**

Preliminary phytochemical screening of the starch extracted was done according to the procedures described by Sofowora (1993). Cardiac glycosides screening was done using the Liberman's test. In this method, a small quantity of the extract was dissolved in 2 ml of acetic anhydride and cooled well in ice. Concentrated sulphuric acid was then carefully added. A violet colour which changes to blue and then to green indicates the presence of a steroidal nucleus.

### **RESULTS AND DISCUSSION**

Table I below shows some physicochemical properties of the starch citrate. The photomicrographs of unmodified (native) starch and modified (citrate) starch at various magnifications are depicted in Plates I and II respectively, while the swelling pattern for the sample is given in Figure I.

Phytochemical screening revealed that both samples contain carbohydrates and terpenoids but only the native tacca starch contains cardiac glycosides.

#### **Chemical Composition**

Tacca starch citrate was found to be a white, crystalline, non- hygroscopic powder with a yield of about 75.7 %. It exhibited no pasting or gelling properties when heated at 100 °C for 30 min. This is in accordance with an earlier work on potato starch (Chowdary and Enturi, 2011)

The pH value of 4.68 for tacca starch citrate was found to be slightly lower than that of the native starch obviously because of the reaction with citric acid but it still falls within the pH range of 3 – 9 obtained for most starches used in the pharmaceutical, cosmetics and food industries (Coursey and Rasper, 1967).

The phytochemical screening of both starches (that is native and citrate) shows that they have the same essential active chemicals, carbohydrates and terpenoids (from the parent material) although cardiac glycosides found in the native starch was absent in the modified starch. Proximate analysis of both starch samples indicates that they still contain the same constituents and the quantity was comparable to each other.

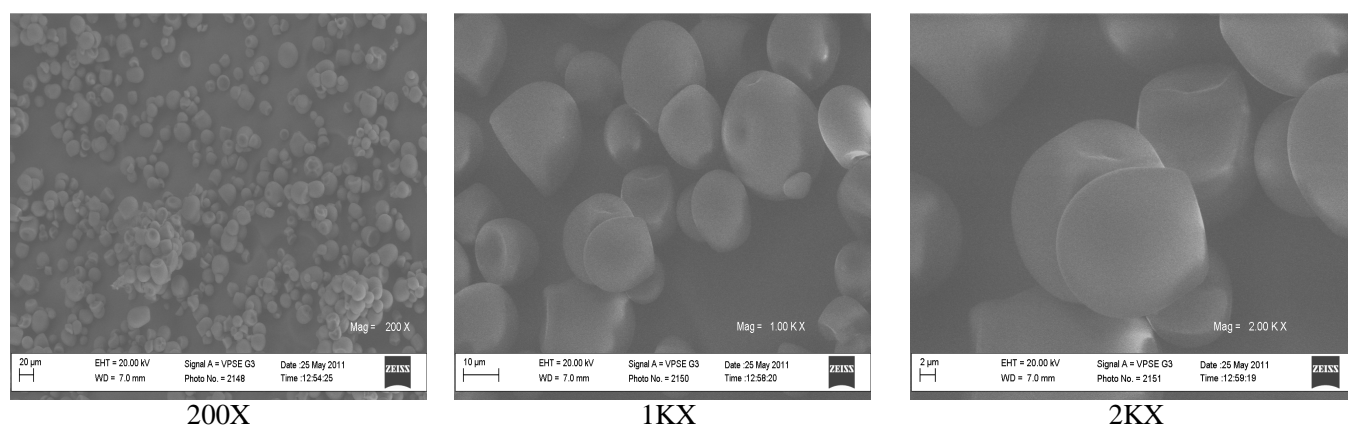
From Table I, it can be seen that the water absorption capacity of tacca starch citrate was quite higher than that of the native starch. This might be a reflection of the desirability of the modified starch in the pharmaceutical industry (Chowdary and Enturi, 2011). Foam and emulsion capacities of both starches were comparable with each other. The browning and charring temperatures of the starch citrate was observed to be higher than that of the unmodified. This shows that the starch citrate can even be heated to a higher temperature without changing colour or charring. This quality will make it a preferable starch in industries that use starch at higher temperatures.

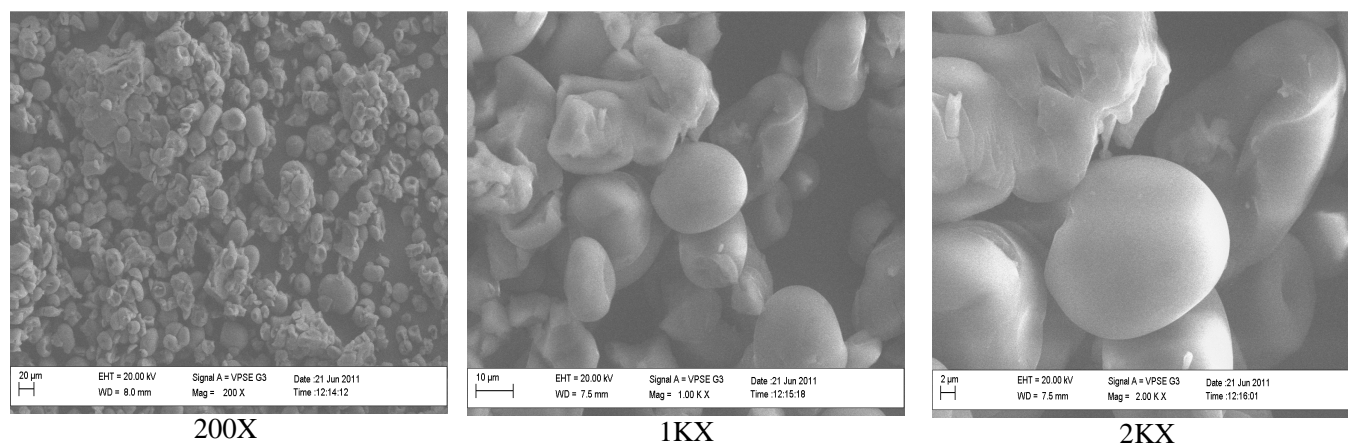
**Table I: Physicochemical properties of tacca starch and tacca starch citrate**

PARAMETERS	TACCA STARCH	TACCA STARCH CITRATE
pH	5.02 ± 0.0	4.68 ± 0.0
Fat (%)	1.5 ± 0.1	1.25 ± 0.15
Moisture content (%)	10.5 ± 0.3	11.75 ± 0.22
Protein (%)	0.44 ± 0.0	0.44 ± 0.0
Ash (%)	2.5 ± 0.1	2.5 ± 0.1
Carbohydrate (%)	82.96 ± 0.03	84.06 ± 0.02
Crude fibre (%)	2.10 ± 0.1	-
Water absorption capacity (ml)	78.0 ± 0.1	83.25 ± 0.13
Foam capacity (%)	4.0 ± 0.1	4.2 ± 0.2
Emulsion capacity (%)	10.63 ± 0.24	7.8 ± 0.2
Gelatinization temperature (°C)	62 ± 0.0	-
Browning temperature (°C)	243.7 – 251.3 ± 0.0	223.5 – 231.2 ± 0.0
Charring temperature (°C)	280.5 – 290.1 ± 0.0	251.7 – 258.9 ± 0.0

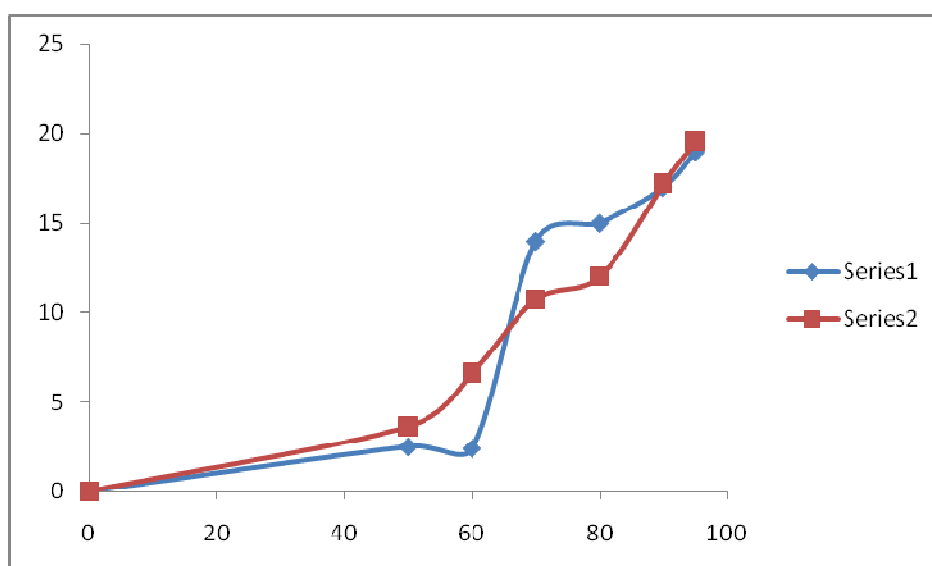
### Morphology

The photomicrographs of *Tacca involucrata* starch and the modified one at various magnifications are shown in plates I and II below. The photomicrograph of both starches indicates that the starch granules are generally small sized and oval in shape with slight spacing between each unit of granule. The morphology of both starches look alike but some granules in the starch citrate appear ruptured. This might be attributed to the chemical reaction that occurred at the elevated temperatures employed. When compared with other starch standards it falls in agreement with the granule sizes that have been observed and reported (Bandhari and Singhal, 2002). Generally, small and medium sized starch granules have been reported to have varied utilization in the food and pharmaceutical industries (Omojola *et al*, 2010) which makes both the native and modified starch desirable both in the pharmaceutical, food and other industrial applications.

**Plate 1: Photomicrograph of tacca involucrata starch sample at 200 X, 1 KX and 2 KX respectively**



**Plate 2: Photomicrograph of tacca starch citrate at 200 X, 1 KX and 2 KX respectively**



**Figure I: Swelling profile for native tacca starch and tacca starch citrate**

\*1 = Native tacca starch

\*2 = Tacca starch citrate

### Swelling

The swelling profile of tacca starch compared with that of tacca starch citrate over a temperature range of 50 – 100 °C is shown in Figure I below. The swelling profile shows a general trend of increase with increase in temperature which is more uniform in the modified starch than in the unmodified starch. This is an indication of the water absorption characteristic of the granules during heating. The modified starch at lower temperatures of 50 – 70 °C was observed to have higher swelling capacity than the unmodified starch, while the swelling power of the unmodified starch experienced an increase in swelling power at higher temperatures. Since increase in swelling power is indicative of suitability of a starch being used as a disintegrant in the pharmaceutical industry (Chowdary *et al*, 2011) hence it appears that tacca starch citrate might be a better choice as a disintegrant in the formulation of tablets especially since tablets are

compounded and used at the temperature range where it has a better swelling. Also high swelling power results into high digestibility and ability to use starch in solution suggesting improved dietary properties and the use of starch in a range of dietary applications (Nuwamanya *et al*, 2010), this confirms the applicability of tacca starch citrate in other industries.

### **Gelatinization Properties**

Gelatinization is the process whereby starches undergo an irreversible change under heat and absorb water with swelling thereby making the granules swell more and become a paste rather than a dispersion which it forms in cold water. The native starch sample was observed to have a gelatinization temperature of 62 °C which falls within the range of gelatinization temperatures commonly observed for starches (Muazu *et al*, 2011). The modified starch (citrate) does not gelatinize at even higher temperature, this indicates that its absorption of water at all temperatures is reversible and also industries that require the use of starch in the gel form might not find this modified starch very useful

### **CONCLUSION**

The physicochemical properties of *tacca involucrata* starch citrate have been examined and these properties shows better swelling and water absorption properties over the native starch indicating that *tacca involucrata* is a potential source of industrial starch. The good swelling power makes it a promising pharmaceutical excipient. This type of starch from a non conventional source will reduce the cost of producing starch and eliminate or minimize competition on stable food crops like cassava or potatoes.

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