



## Effect of presence of sulphurdioxide on acetylation and sorption isotherm of acetylated starches from cultivars of cassava



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### ABSTRACT

Starches from cultivars of cassava were modified with acetic anhydride. Treatment with sulphurdioxide was compared with native. The starches were evaluated for functional properties and moisture isotherms were calculated.

Addition of 3.5% acetic anhydride resulted in starches with DS of 1.66% and 3.25% in sweet and bitter cultivars. Sweet starch alone will be applicable for food. Least gelation concentrations for the native were 14% and 10% against 6% and 8% acetylated samples, respectively. Degree of substitution (DS) was reduced with SO<sub>2</sub> by 45% and 39% in sweet and bitter cultivar with 150 mg/kg starch, respectively. Swelling power and solubility increased with DS. Exudates from samples varied. Monolayer values of the starches were between 1.05% and 9.16% under 18 °C and 30 °C that simulated distribution and storage. R<sup>2</sup> value of water adsorbed and water activity ranged from 50% to 97%. X-ray patterns were not disrupted.

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### 1. Introduction

Cassava (*Manihot esculentum* Crantz) is among the world's cheapest and most popular staple crops in many countries including Nigeria, Brazil and Thailand (Osundahunsi, Seidu, & Mueller, 2011). World production of cassava in the year 2000 amounted to 165 million metric tonnes with Africa producing over 91 million metric tonnes of cassava annually and Nigeria has been reported to be the highest world producer of cassava with the output of over 45 million metric tonnes in 2004 (FAOSTAT, 2009; Sanni, Oyewole, & Olowogbade, 1998; Sriroth, Piyachomkwan, Wanlapatit, & Oates, 2000). However, there is the need to source value-added products such as sweeteners, dextrin, amino acid and starch from cassava for economic reasons rather than subsistence (Sriroth et al., 2000). Research activity has led to the development of low cyanide (sweet), high and early yielding cassava tubers. One prominent method of chemical modification of starches is esterification through acetylation, since the tendency of an aqueous starch dispersion to increase in viscosity on cooling and finally to gel, depend on the association of amylose molecules (Tester & Morrison, 1990). Acetylation, which retards or eliminates the recrystallisation or retrogradation phenomenon, will affect stability of the starch

(Adebowale, Afolabi, & Lawal, 2002). Acetylation involves esterification of the hydroxyl functional groups of the starch and provides sol-stability and functional properties such as hydrophilic, cationic or anionic character at relatively low cost (Adebowale & Lawal, 2003). Acetylated starches with low degree of substitution (DS) have been widely used in food industries for many years because of the unique characteristics, such as low gelatinisation temperature, high swelling and solubility, and cooking and storage stability (Wang & Wang, 2001).

Addition of sulphur-dioxide (SO<sub>2</sub>) usually during centrifugal or extraction stage of starch processing is expected to increase separation of starch from other bound impurities (Cosbishley, 1984). In addition, sulphur-dioxide also possesses good bleaching and antimicrobial properties (Cosbishley, 1984). The influence of sulphur-dioxide on the physical properties of starch granules or macromolecular compounds has in recent years, been a source of interest. It is suggested that low levels of sulphur-dioxide can influence the functional properties of starch, lower paste viscosity, reduce swelling and increase the amounts of soluble material (Sriroth, Wanlapatit, & Piyachomkwan, 1998).

The knowledge of the sorption characteristics of food is important in the design of drying processes/equipment, studies of shelf-life and packaging requirement of dehydrated products (Crapiste & Rotstein, 1982; Johnson & Brennan, 2000). The moisture level at which some dehydrated products have good storage stability has been found to agree closely with the moisture level calculated from the moisture sorption isotherm (Mazza, 1982). Using the Brunauer, Emmett and Teller (BET) theory of molecular adsorption, the

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optimum moisture levels for stability of complementary products were established to be 6.5 to 8.0 (Osundahunsi & Aworh, 2000). Presently, there is scanty information on the moisture sorption properties of acetylated starches. Acetylated starches at low degree of substitution were prepared from the sweet and bitter cultivars of cassava. Treatment with SO<sub>2</sub> was compared with the native counterparts. The starches were evaluated for swelling power and solubility, freeze-thaw stability, absorption, least gelation concentration, X-ray diffraction and moisture sorption isotherm for storage stability.

## 2. Materials and methods

### 2.1. Materials

The cassava tubers, TMS 30572-low cyanide (sweet) and TMS 50395-high cyanide (bitter) cultivars were collected from the International Institute for Tropical Agriculture (IITA) Ibadan, Nigeria. In the following text only the abbreviation SAC and BAC will be used for sweet and bitter respectively. All chemicals used were of Analytical grade.

### 2.2. Isolation of starch

The starches were extracted using established procedures. Briefly, peeled tubers were washed thoroughly and grated. The grated pulp was mixed with sufficient amount of water to form slurry with subsequent filtration. The resulting starch was allowed to sediment and the supernatant decanted. The starch was spread in a thin layer on a tray and dried in air oven at temperature of 35 ± 2 °C, the dried starch was pulverised and screened through standard 100 mesh opening (Retsch Analysensieb DIN 4188). Analytical sieve shaker (Model 03-502, Fritsch GmbH & Co., Haan, Germany) was used.

### 2.3. Starch acetylation

Starch acetylation was carried out using the method described by Wurzburg (1964) as modified by Golachowski (2003). Two hundred grams of starch (db) was dispersed in 200 ml of distilled water and then made up to 560 ml with distilled water, the pH was adjusted to 8–9 using 3% sodium hydroxide solution, predetermined volume of acetic anhydride was added at a constant rate of 1 ml per minute while maintaining a pH of 8–9 using 3% NaOH with pH metre (Ino Lab level 2 Model). The reaction proceeded for 5 min after the addition of acetic anhydride. The pH was finally adjusted to 5.2–5.6 with 10% Hydrochloric acid solution. The acetylated slurry was centrifuged (Beckman Coulter, Avanti J-25, USA) at G = 885 for 30 min, the residue obtained was washed three times with distilled water and air dried at 30 ± 2 °C, the dried starch was pulverised through 100 mesh sieve and packaged in plastic container and stored for further analyses. The degree of acetylation was varied by the quantity of acetic anhydride that was added, which was obtained by calculation.

### 2.4. Determination of the degree of acetylation of the modified starches

The degree of acetylation was determined by the procedure of Golachowski (2003). Ten gram of acetylated starch (db) and 65 ml of distilled water was added and neutralised by the addition of few drops of 0.1 M NaOH to obtain a faint pink colour using few drops of phenolphthalein indicator. Measured volume of 0.5 M NaOH was added and the mixture was mixed for 35 min, the resultant mixture was titrated in a bottle-top burette Titrette with an electronic digital display (Digital Titrette – A) against 0.5 M HCl un-

til the pink colour disappeared. The percentage acetylation was calculated according to Wurzburg (1964).

$$\% \text{ Acetylation} = \frac{(bb - t)0.043 \times 0.5 \times 100}{w} \quad (1)$$

- *bb* = Blank titre.
- *t* = Volume of 0.5 M HCl used for titration of sample.
- *w* = Weight of starch (db).

### 2.5. Least gelation concentration

Samples of starch, 2–18% (w/v) were prepared in test-tubes with 5 ml of distilled water. The starch suspension was mixed with properly for 5 min. The test tubes were heated for 30 min at 80 °C in a water-bath, followed by rapid cooling under running cold tap water. The test-tubes were further cooled at 4 °C for 2 h. Least gelation concentration was determined as that concentration when the sample from the inverted test-tube did not slip.

### 2.6. Treatment with sulphurdioxide

Starch milk suspension was prepared as described earlier for acetylated starch. Treatment with sulphurdioxide was carried out according to Golachowski (2003). Briefly, liquid NaHSO<sub>3</sub> was added to the total weight of suspension i.e. (Starch + Water + NaHSO<sub>3</sub> liquid) and brought up to 560 g with water. The concentration of NaHSO<sub>3</sub> expressed as mg SO<sub>2</sub> per 1000 g of starch were as follows: 0, 75, 150, 600 and 1200. The solution was blended and kept for about 30 min. The degree of acetylation was determined later.

### 2.7. Determination of swelling power and solubility

Method of Schoch (1964) as described with minor modifications (Osundahunsi, Fagbemi, Kesselman, & Shimoni, 2003) was used. Suspension 1% (w/v) of starch was weighed into graduated centrifuge tube with distilled water. These tubes were immersed in water bath at a temperature range from 45 to 95 °C at 10 °C interval for 30 min, with shaking every 5 min during heating. The tubes were removed, cooled to room temperature and centrifuged (Megafuge 1.0R Heraeus Thermo Scientific, USA.) at G = 221 for 30 min after which the supernatant was carefully sucked into already weighed Petri dishes and dried in the air oven at 110 °C for 4 h. The weight of the pastes was determined and used to calculate the swelling power as gram of sediment paste per gram starch. The difference in weight of the Petri dish after drying the supernatant was taken as the weight of soluble starch percentage; solubility was calculated as percentage gram of soluble starch per gram starch. All measurements were done in triplicate.

### 2.8. Freeze-thaw stability

Freeze-thaw stability of gelatinized starch was measured by using the method of Bhandari and Singhal (2002) with some modifications. Aqueous suspension of starch (5 g/100 g) was heated at 95 °C under constant agitation for 1 h. The paste was weighed (exactly 20 g each) into previously weighed polypropylene centrifuge tubes and capped tightly. Alternate freezing and thawing was performed by freezing at –20 °C for 18 h and thawing at 25 °C for 6 h. Samples were centrifuged at 5000 rpm for 10 min and the percentage of water separated after each cycle was measured and expressed as percentage

$$\text{Syneresis (\%)} = \frac{\text{Water separated (g)}}{\text{Total weight of sample (g)}} \times 100 \quad (2)$$

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