Effect of Acetylation on the Physicochemical Properties of Starch Extract from *Caladium Bicolor*

ABSTRACT

The study reports on the effect of acetylation on the physicochemical parameters of *Caladium bicolor* starch. The starch was extracted and acetylated using various acetic acid volumes and reaction periods (30, 60, and 90minutes). Both unmodified and acetylated starches were characterized with Fourier transform infrared spectrometry (FTIR) and some models. The findings estimated a low amylose content of 16.95%, and amylopectin at 83.05% in unmodified starch. The swelling power was 72.45 (unmodified), and 64.9–68.5 (modified); paste clarity was 2.013 (unmodified), and 1.73–1.91 (modified); gelatinization temperature was 75^{o}C (unmodified), and 76–78^{o}C (modified); water binding capacity was 68.6% (unmodified), and 68–92.7% (modified); oil binding capacity was 53.8% (unmodified), and 53.2–92.63% (modified); % acetyl group and degree of substitution were 0.73-3.63 and 0.03-0.13. The FTIR reveals the vibration frequencies of 3283.62 cm^{-1}, 1018 cm^{-1} and 1240 cm^{-1} affirm the presence of OH, C-O, and CH$_3$ in the unmodified. Whereas, the prominent peak at 1647.28 cm^{-1} assigned to the carbonyl C=O and its increase in intensity and height affirmed the increase in the acetylation of starch molecules. Starch obtained from *caladium bicolor* and the acetylated products could be used as glue, thickeners, gelling agents and adhesives for paper products and textile industry.

Key words: *Caladium bicolor*, Physioco-chemical properties, Modified and Modified starch.

1.0 Introduction

Starch is a desirable raw material because of its wide availability, low price, biodegradability, biocompatibility, and chemistry-friendly nature. It is now acceptable to use modified starches in food since they are useful as thickeners, gelling agents, textile sizing agents, and adhesives for paper and paper products. A large number of authors [1, 2] have lately described the effective utilisation of organic solvents in the production of highly substituted modified starches. However, it is difficult to dissolve the granular starch in a sufficient solution without significant degradation, making the preparation of highly modified derivatives via reactions with starches a demanding task [3]. Since esterification of polysaccharides changes their hydrophilic character and leads to enhanced or unique thermal and mechanical properties [1], it presents a considerable synthetic challenge.

Natural starch can have its functional qualities enhanced and adapted to various industrial and domestic uses by undergoing a process known as starch modification, which involves altering its physical and chemical properties. Starch acetates are produced via a common and commonly used modification, the esterification reaction of starch with acetyl groups[4]. Although starch has few applications in its raw form, its potential uses expand greatly when modified; yet, it may lack the ideal properties for some procedures. Improved physical, chemical, and functional properties of starch can be achieved through acetylation [5].

Natural starch can have its functional qualities enhanced and adapted to various industrial and domestic uses by undergoing a process known as starch modification, which involves altering its physical and chemical properties. The esterification of starch with acetyl groups to make starch acetates [4] is a common and widely used way to change starch.

In its unmodified form, starch has limited applications and may lack optimal characteristics for some processing methods. When it is modified, however, its utility increases. There has been a lot of study on the acetylation of starch. The acetylation of starch can result in enhanced physical, chemical, and functional characteristics [5]. Anhydroglucose units are acylated when their hydroxyl groups are esterified. This takes place as a result of the hydroxyl groups of the glucose monomers being reduced to CH3COO- groups.

The degree of substitution (DS) determines a category for acetylated starch. As a result of their capacity to add structure and hardness to baked goods, cake fillings, sauces, and soups, low DS (<0.2) starches receive widespread application in the culinary industry. Starches with a DS greater than 1.0 are used as cellulose acetate substitutes [6]. Starches with a DS of 2-3 are employed in non-food applications such as adhesives, coatings, biodegradable polymers, and pharmaceuticals because they are thermoplastic and soluble in organic solvents like acetone and chloroform [7, 8].

Caladium bicolor (family Araceae) is a common wildflower in the southeastern and southwestern parts of Nigeria, but its inedible root tubers are rarely harvested for human consumption. In addition to its decorative value, C. bicolor has been shown to have beneficial effects when applied topically to various skin conditions, including boils, wounds, and ulcers. It is used to treat convulsions and as a purgative [9]. Its flour's potential for use in the synthesis of bioethanol from local fungal isolates has been documented [10].

Numerous investigations into the differences in properties between acetylated starches of varying substitution degrees have been carried out. However, only limited research has been conducted on the physicochemical properties of unmodified Caladium bicolor starch. Singh et al. [4] evaluated the effects of acetylation on potato and maize starch characteristics in the DS range of 0.104-0.154. In previous papers [11], the influence of starch acetylation in different varieties of rice with DS values ranging from 0.107 to 0.144 was studied.

El Halal et al. [12] investigated how acetylation affected the characteristics of rice starches with low, medium, and high amylose levels. Trela et al. [11] studied how changing the concentration of a catalyst affected the acetylation of barley starch. Acetylated maize starch with a DS of 0.57 to 2.59 was produced and characterised by Xu et al. [5].

Mbougueng et al. [13] investigated the effects of acetylation duration on the physical-chemical, functional, and thermal aspects of potato and cassava starches and found a DS value of less than 0.26. Osundahunsi et al. [14] researched the effect of acetic anhydride on the acetylation response of two bread species with a DS ranging from 0.030 to 0.213. Biodegradable films were created by acetylating cassava starch in an acidic environment, as demonstrated by the work of Larotonda et al. [15]. The researchers obtained acetylated cassava starch with degrees of substitution ranging from 1.22 to 1.56.

The swelling power and solubility of acetylated cassava starches were shown to increase with the degree of substitution, as reported by Osundahunsi et al. [14]. According to Mbougueng et al. [13], the solubility of potato and cassava starch increased with increasing dispersity for DS values between 0 and 0.26. They also discovered that acetylated starch pastes, especially those made with cassava starch and DS, had better clarity than their natural counterparts.

Diop et al. found that the solubility of maize starch decreased with increasing DS between 1.83 and 2.9. Because of the intricate interplay between the physicochemical properties of acetylated starches and the degree of substitution, this research focuses on the isolation and characterization of *C. bicolor* acetylated and unmodified starch.

2.0 Materials and Methods

2.1 Extraction of starch

C. bicolor tubers were cleaned, skinned, and chopped before being blended with distilled water (3:1) for 1minute. The mixture was then filtered through two layers of cheesecloth. After leaving the filtrate in the bottom of the beaker for an entire day, the starch began to precipitate at the bottom. After drying the starch precipitate at 45°C until it reached a consistent weight, the supernatant was removed and the precipitate was washed three times with distilled water. After the dry starch had been pulverised with a grinder, it was put into polyethylene bags so that it could be used at a later time [14].

Equation 1 was used to determine the starch content.

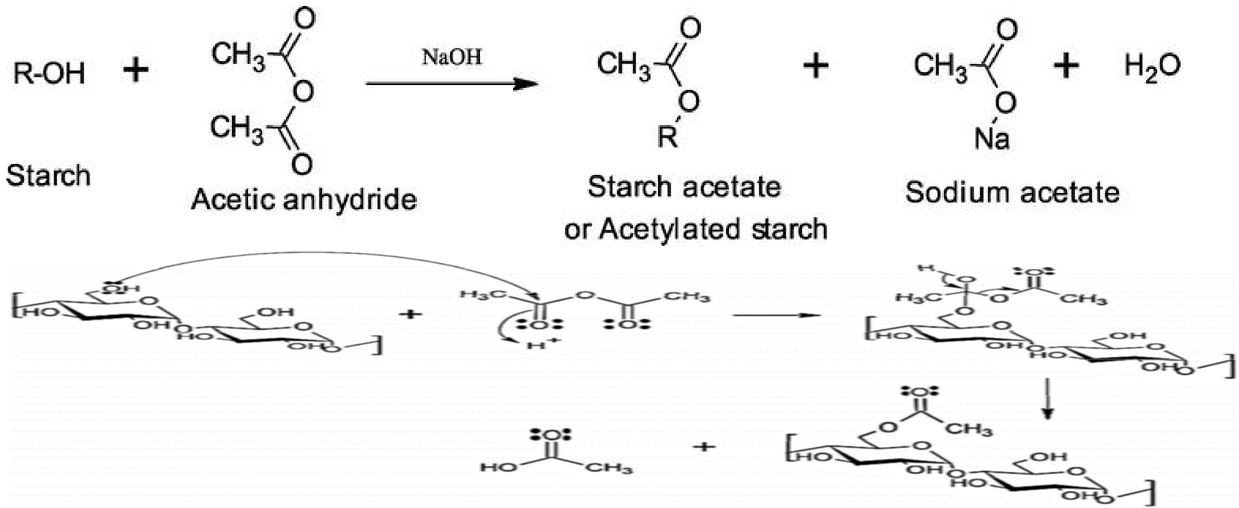
$$\textbf{Starch\%} = \frac{starch\ extracted(g)}{Fresh\ tuber(g)} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

2.2 Amylose and amyloectin content

The amylose and amylopectin content of the starches was determined using a colorimetric test and a modified published methodology [8]. After combining 5mg of starch with 1mL of 90% DMSO, the mixture was heated in an oven set to 95.0°C for an hour. The sample was then diluted 1:1 with iodine at 3g/L in 90% DMSO, and further diluted 10 times with water. An UV-Vis spectrophotometer was then used to determine the absorbance at 635nm. Amylose content reported as percentage.

2.3 Acetylation/esterification of starch

The production of acetylated starch required the utilisation of acetic anhydride as a reactant. After shaking the mixture constantly for one hour, 25g of starch were evenly distributed among 75mL of distilled water. To achieve a pH of 8.0, the suspension was treated with a solution containing 3% NaOH. After reacting the slurry with acetic anhydride at varied volume/volume ratios (10, 15, and 20%), the mixture was heated in a water bath at 45°C for varying amounts of time. (30, 60 and 90minutes). The product of the reaction was filtered, and the precipitate was washed until the pH of the water returned to normal. In an oven, the newly created precipitate was maintained at a temperature of sixty degrees Celsius throughout the process. In order to conduct further research, the produced starch acetate was evaporated, pulverized, and screened [17].



2.4 Determination of the acetyl percentage (Ac %) and degree of substitution (DS)

Both the degree of substitution (DS) and acetyl percentage of acetylated starch were determined via the titration of ethanol (50mL, 75%) in distilled water were combined with 1g of acetylated starch. The slurry-filled flask was covered with aluminium foil and placed in a water bath preheated to 50°C for 30minutes. After the samples were cooled to room temperature 40°C, 0.5M NaOH were added. Phenolphthalein was used as an indicator to determine whether the slurry's excess alkali had been neutralised by adding 0.5M HCl. An experiment was conducted using native starch as a comparison [17]. Equations (2) and (3) were used to determine the degree of substitution and the acetyl percentage (3).

$$\text{Acetyl \%} = \frac{(Vblank - Vsample) \times Molarityof\ HCl \times 0.043 \times 100}{Sample\ weight} \qquad (2)$$

$$\text{DS} = \frac{(162 \times Acetyl\%)}{(4300 - [42 - Acety\%])} \qquad (3)$$

2.5 Determination of pH

A beaker was used to dissolve the starch sample (1.5g) in 10ml of distilled water with careful stirring. After inserting the pH metre into the liquid, a reading was obtained. The procedure was repeated three times, and the average was recorded.

2.6 Determination of Gelatinization Temperature

The starch (1.50g) was dissolved in the water (10mL) in a beaker, which was then placed in a water bath and stirred. After persistent stirring, the solution finally thickened and took on a milky appearance. This temperature was later identified as the gelatinization temperature [18], marking the gel point. Three separate attempts at the identical procedure were recorded, and an average was calculated.

2.7 Determination of Swelling Volume, Swelling Power and Solubility

Hirsch and KoKini's [19] methodology was used to determine the swelling power and solubility. Carefully labelled and graduated centrifuge tubes were used to collect the samples. To prevent the starch granules from settling to the bottom of the water bath during gelatinization, they were gently shaken while the solution was swirled and heated to 95^{o}C. A water bath was used to keep the gelatinized samples at 95^{o}C for an hour. After being centrifuged at 1000rpm for 30minutes, the samples were cooled to room temperature by running water. When the tube containing the enlarged sediment was read after centrifugation, the swelling volume was directly determined. After separating the solution from the sediment, it was transferred to a metal dish, weighed, then dried at 105°C for 1 hour before being weighed a second time. The procedure was performed three times, and the mean was then calculated. Equations (4) and (5) were used to calculate the swelling power and solubility of starch (5).

$$\text{Solubility} = \frac{\text{Weight of dry supernatant}}{\text{Weight of starch sample}} \times 100 \qquad (4)$$

$$\text{Swelling power (\%)} = \frac{\textit{Weight of sediment paste}}{\textit{Weight of sample on dry basis}\times(100-\%\textit{Solubility})} \times 100 \qquad (5)$$

2.8 Water binding capacity (WBC)

The Osundahunsi et al. (2014) methods were slightly modified to determine the water binding capacity. Starch (5g) was dissolved in 75mL of distilled water, stirred for an hour, and then centrifuged at 3000rpm for 10minutes. The wet starch was dewatered and was then allowed to drain for 10minutes. The wet starch was then weighed. The same process was then carried out three times, with the average value recorded.

$$\text{Water binding capacity \%} = \frac{Wet\ starch(g)}{Dry\ starch(g)} \times 100 \qquad (6)$$

2.9 Oil-binding capacity (OBC)

The techniques used was in line with Osundahunsi et al. (2014) with slight modification. After centrifuging a 5g starch and 75mL distilled water solution at 3000 rpm for 10 minutes, the mixture was agitated for 1hour. The extracted wet starch was then drained of the remaining free water. After that, the weight of the wet starch was taken into consideration; this was repeated three times, and the mean value was recorded.

.

$$\text{Oil-binding capacity} = \frac{\text{Weight of residual starch}}{\text{Weight of sample}} \times 100 \qquad (7)$$

2.10 Paste clarity

The method of Zhang et al. (2017) was used in past clarity analysis. After putting a starch solution (1%w/v) in water and agitating it for 30minutes, it was heated in a pot of boiling water. The gel that was formed was then tested for its transmission (%T) at 650nm in duplicate using a unicospec UV-2150 spectrophotometer.

2.11 FTIR Analysis

The functional group variation during acetylation of obtained *C. bicolor* starch was studied using FTIR. For the identification of various functional groups added during acetylation, a PerkinElmer 1600 infrared spectrometer was employed. The spectra were acquired by the spectrometer using 32 running scans at a resolution of 4cm^{-1} for each sample across the range of 650-4000cm^{-1}. Nicolet Software's "find peak tool" functionality was utilized to locate significant transmittance peaks for a given wave number.

3.0 Results and Discussion

3.1 Amylose and amylopectin content

Amylose and amylopectin content of the unmodified and modified starch with acetic acid at different volumes (5mL, 10mL and 15mL) and at 90minutes are shown in Table 1. Generally, the starch of *C. bicolor* had low amylose content of 16.95%, and amylopectin 83.05%. The amylose content of *C. bicolor* was consistent with Sharlina et al. [20], who worked on *Dioscorea pyrifolia* tubers with starch contains; 44.47 ± 1.86% amylose, and the amylose concentration of maca starch is predicted to be between 21.0 to 21.3%, according to the research of Zhang et al. [21]. As a result of its low amylose content, *C. bicolor* starches may be useful in applications where soft gels or films are required, such as in the glue, detergent, plywood, and textile industries.

3.2 Swelling Power and Water Solubility

Tables 1-3 display the swelling power and water solubility of unmodified and modified starch with acetic acid at various volume and time intervals, respectively. The swelling power of starch obtained from unmodified *C. bicolor* was 72.45, whereas, the modified starch was estimated at 67.5-68.5. The swelling power of natural starch was slightly higher than the acetylated starch of *C. bicolor*. This implies that some component of the raw starch should have been lost during acetylation process, since the hydrogen of the OH groups were involve in esterification. The results in Tables 1-3 also indicate that increase in the volume of acetic acid slightly increased the swelling power of acetylated starch of *C. bicolor*. The water solubility analysis in Tables 1-3 shows that at 30^{0}C, the modified starch has a higher solubility than the unmodified starch of *C. bicolor*. This is associated with the increase in the polarity as a result of functionalization of the starch moiety. Similar values in solubility could be attributed to the same volume of acetic acid and amylose of component of unmodified starch at different time considered. This is in agreement with the findings of Tang et al. [22], who investigated the relationship between functionality and structure in barley starches, and the research of Sindhu and Khatkar [23], who investigated the physicochemical and functional properties of starch and flour from tartary buckwheat (Fagopyrum tataricum) grains.

Indicators of the degree of interaction between starch chains in the amorphous and crystalline domains and a measure of starch hydration, the swelling power and water solubility index, were introduced by Sindhu and Khatkar [23]. As discussed by Sharlina et al. [20], starch swelling and solubility profiles are affected by factors such as the presence of lipids or phosphates and the degree of cross-bonding inside the granules [20]. Considering that high amylose content and strong intermolecular interactions limit swelling, it is possible that the starches' high swelling power and solubility index at varied acetylation volumes are due to their low amylose content and low lipid content [21].

3.3 Water and Oil Binding Capacity

Tables 1-3 display the water and oil binding capacities of *C. bicolor* starch produced using varying volumes of acetic acid and times of acetylation, respectively. Acetylated starches have a higher water-binding capacity than their unmodified counterparts. Acetylated starch's high water binding ability is due to both the substantial hydrogen bonding between water molecules and starch hydroxyl groups and the loose contact between amylose and amylopectin molecules in starch granules. This is further confirmed by the findings of Jiang et al. [24], who characterised starches extracted from five species of *Dioscorea L.* Values of starch acetylation, water binding capacity, and solubility all rise in tandem with acetic acid concentration. Uwem and Ita [25], who studied the physicochemical, functional, and antinutritional aspects of *Dioscorea dumentorum* starches, came to similar conclusions. Variation in the amount of water-binding surface available within the granule may account for the observed range in staches' water binding capacities [12].

Tables 1–3 display the oil binding capacity of *C. bicolor* starch with varying acetylation volumes of modified starch. The results show that acetylated starch outperforms unmodified starch in terms of its binding capacity for both vegetable oil and crude oil. There was a positive correlation between acetic acid concentration and oil-binding capacity. This is because of the structural re-orientation of the starch molecules, which could have been triggered by the high acetyl content [26]. This would have resulted in the maximum oil binding capacity. One possible explanation for starch's poor water-binding properties is that the hydroxyl groups tend to generate hydrogen and covalent bonds between the starch chains rather than with water [27].

Table 1: Physicochemical Properties of Acetylated Starch from *C. bicolor at* 45^{o}C for 90minutes, at Different Acetic Acid Values

Parameters	**unmodified**	**Modified**		
Volume acetic acid		5mL	10mL	15mL
pH	4.49			
Gelatinization temp.	75	76	76	77
Amylose %	16.95	NA	NA	NA
Amylose pectin %	83.05	NA	NA	NA
WBC	68.6	88.9	89.1	92.7
OBC(Vegetable oil)%	53.8	60	62	63
OBC(Crude oil)%	42.2	58.1	60.9	60.2
% acetyl group	NA	1.35	2.43	2.15
DS	NA	0.052	0.092	0.082
Past clarity	2.013A, 0.97%T	1.789A, 1.59%T	1.74A,1.60%T	1.73A,1.57%T

Solubility at 30^{0}C(%)	38.5	54.2	54.2	54.6
Swelling power	72.45	67.5	67.8	68.5

WBC=water binding capacity, OBC= oil binding capacity, DS=degree of substitution

Table 2: Physicochemical Properties of Acetylated Starch from *C. bicolor at* 45^{o}C for 60minutes, at Different Acetic Acid Values

Parameters	**Unmodified**	**Modified**		
Volume acetic acid		5mL	10mL	15mL
pH	4.49			
Gelatinization temp.	75	75	76	76
Amylose %	16.95	NA	NA	NA
Amylose pectin %	83.05%	NA	NA	NA
WBC	68.6	84.1	85.9	88.4
OBC(Vegetable oil)%	58.5	58.5	58	59.2
OBC(Crude oil)%	42.1	57.5	58.2	58.8
% acetyl group	NA	0.71	1.14	1.78
DS	NA	0.04	0.03	0.07
Past clarity	2.013A, 0.97%T	1.65A, 1.53%T	1.62A,1.53% T	1.69A, 1.60T
Solubility at 30^{0}C(%)	38.5	53.1	53.1	53.4
Swelling power	72.45	66.3	66.5	66.7

WBC=water binding capacity, OBC= oil binding capacity, DS=degree of substitution

Table 3: Physicochemical Properties of Acetylated Starch Obtained from *C. Bicolor at* 45^{o}C for 30minutes, at Different Acetic Acid Values

Parameters	**Unmodified**	**Modified**		
Volume acetic acid		5mL	10mL	15mL
pH	4.49			
Geletinization temp.	75	77	77	78
Amylose %	16.95	NA	NA	NA
Amylose pectin %	83.05%	NA	NA	NA
WBC	68.6	76	78.3	84
OBC(Vegetable oil)%	58.5	52.3	52.4	62.3
OBC(Cruid oil)%	42.1	51.2	50.2	60.4
% acetyl group	NA	3.63	3.08	3.50
DS	NA	0.14	0.12	0.13
Past clarity	2.013A, 0.97%T	1.82A, 0.91T	1.79A, 1.58%T	1.91A, 1.60%T
Solubility at 30^{0}C(%)	38.50	52.6	52.6	52.8
Swelling power	72.45	64.9	65.3	65.7

WBC=water binding capacity, OBC= oil binding capacity, DS=degree of substitution

3.4 Percentage of Acetyl Group and Degree of Substitution (DS)

Tables 1–3 show several characteristics of the modified and acetylated starch of *C. bicolor*. The ratio of hydroxyl (OH) groups to D-glucopyranosyl (Glc) groups in a single D-glucopyranosyl (Glc) unit is used as an indication for the degree of substitution in starch. Considering that each glucose molecule contains three hydroxyl groups, the maximum DS value is 3. This is because the primary OH group on C-6 is more accessible and reactive than the secondary OH groups on C-2 and C-3, which are sterically hindered. Time, stoichiometric amounts, and amylose and amylopectin concentrations all have a role in determining the DS.

Based on their DS values, Mark and Mehltretter [28] divide the acetated starches into three categories: those with a low DS (<0.1), those with a medium DS (0.1-1.0), and those with a high DS (>1.0). The film-forming, adhesive, thickening, stabilizing, and texturizing capabilities of starches in the DS range of 0.01-0.20 are of commercial importance [29].

Native starch can be esterified with acetic anhydride in an aqueous solution using an alkaline catalyst to create acetylated starch with a low DS. Between 0.12 and 0.14, the DS shows some variation throughout the duration of the 30minutes. The degree of subtraction at the half-hour mark is regarded as moderate. The acetylated product improves film formation, adhesion, thickening, stabilization, and texturizing. On the basis of ratings between 0.03 and 0.094(<0.1), we classify the DS range of 60–90minutes as low. More acetic acid was found to be related to a higher substitution degree throughout the periods studied [4, 29].

When the degree of substitution (DS) is high enough, starch esterified with organic acids produces thermoplastic and hydrophobic polymers [27, 29]. Acetic acid with a larger volume caused a greater collision rate between molecules and led to more acetic acid molecules being present in the region of the starch [7]. Increases in DS make starch more hydrophobic, which makes it easier to mix with other polymers that are also hydrophobic [7, 30]. These results concluded that C. bicolor starch has potential applications in the thermoplastic and polymer sectors.

3.5 Paste Clarity (PC)

The transparency of a gel can be measured by its paste clarity (PC). As can be seen in Tables 1-3, the acetylation of C. bicolor starch results in varying degrees of paste clarity depending on the amount of acetic acid used and the length of time it is left to react with the starch. The paste made from unmodified starch was clearer than the paste made from acetylated starch. A possible cause for this is the presence of acetyl groups in the modified starch and the degree of substitution. As the amount of acetic anhydride and the amount of time increased, there were variations in the paste's clarity. This is in line with the findings of Singh et al. [7], who investigated how acetylation affected various characteristics of corn and potato starches. Therefore, amylose molecules with a high propensity for retrogradation may be the cause of the low clarity of cocoyam starch pastes.

3.6 Gelatinization Temperature (GT)

As shown in Tables 1–3, at various times and acetic acid volumes, the gelatinization temperature of the acetylated starch was higher than that of the unmodified starch. The increase in the volume of acetic acid increases the gelatinization of acetylated starch. This implies that acetylated starch contains more amylose, which is strongly bound and requires higher energy to dissociate from their inner embedded core than the low amylose starches [29-32]. The lower gelatinization implies that the unmodified starch has less tightly bound amylase, and on heating releases this less bound amylose out of its inner core structure more easily, faster and with less energy. This is in line with reports that starch with high amylose or packed together need a high temperature to fully gelatinize [33].

3.7 FTIR Analysis

The FT-IR spectra of unmodified and acetylated *C. bicolor* starches are shown in Figures 1–3. A number of peaks at 1018cm^{-1}, 1022.31cm^{-1}, and 1153.74cm^{-1} may be observed in the IR spectra of unmodified starch, all of which are the result of the C-O bond stretching. At 1639.55cm^{-1}, corresponding to the firmly bound water (H_2O) in the starch, a very broad band at 3367.11cm^{-1} was found due to the vibration of the hydroxyl group (O-H), and at around 3367.11cm^{-1} [34, 35].

The absorption bands at 1647.28cm^{-1} became prominent after acetylation by acetic anhydride (5ml), while the absorption bands at 3387.11cm^{-1} became weaker. With the addition of 15ml of acetic anhydride, the intensity of the peak at 1647.28cm^{-1} increased while the peak at 3387.11cm^{-1} declined. The carbonyl C=O of an ester explains the 1647.28cm^{-1} signal, while the hydroxyl group explains the 3387.11cm-1 peak. Ethanol esters are generated during the starch esterification process, as evidenced by the appearance of two additional absorption bands at 1647.28cm^{-1} and 1720.18cm^{-1} [9, 30].

These peaks' magnitude and altitude are particularly sensitive to the substitution ratio. The acetylation peaks at 3387.62cm^{-1} and 1647.28cm^{-1}(Figure 2) are related to 5ml of acetic anhydride and indicate a modest degree of substitution in the acetylated starch. Figure 3 shows peaks at 1647.28cm^{-1} and 3387.11cm^{-1}; the addition of 15ml of acetic anhydride increased the intensity and height of these peaks, indicating a higher degree of substitution. This agrees with the findings of Biswas et al. [6], who found that the peaks, regions, and heights about 3450 cm-1, especially those connected to the OH vibration, vanish or diminish in intensity as substitution increases.

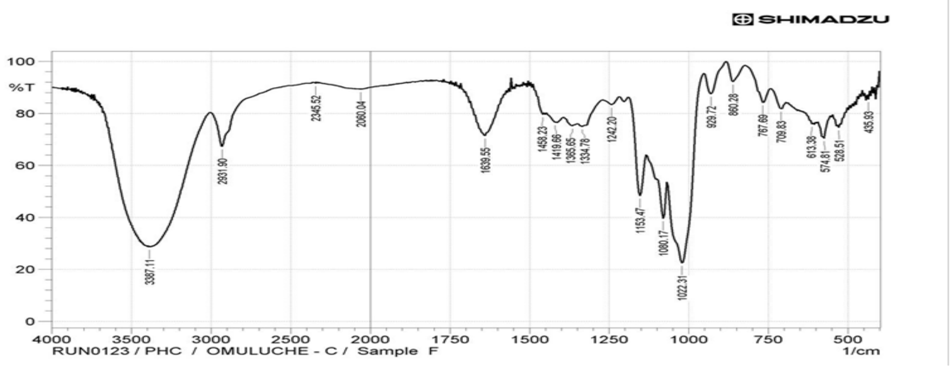


Figure 1: FTIR of Unmodified *C. bicolor* Starch

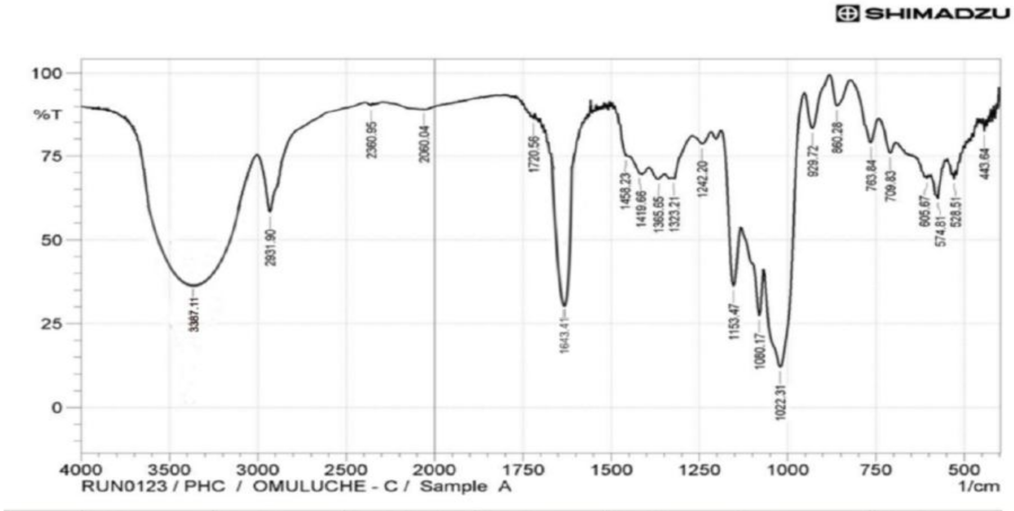


Figure 2: FTIR of Acetylated *C. bicolor* Starch at 5ml of Acetic Anhydride

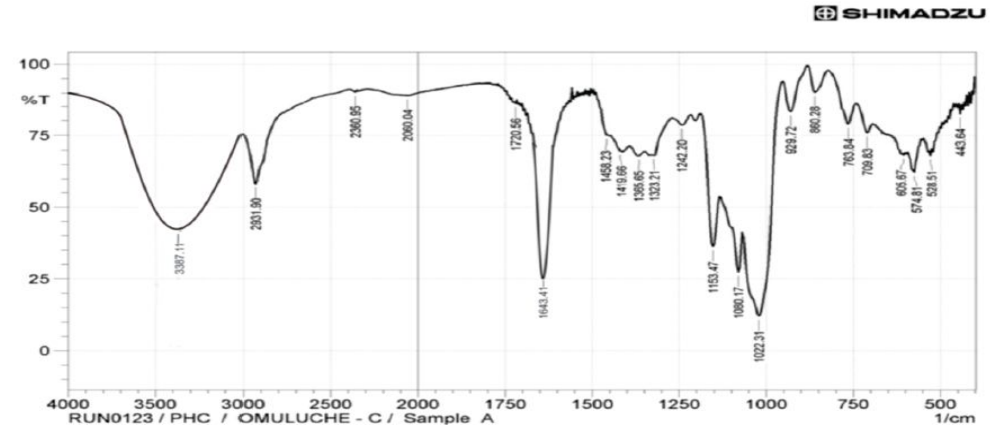


Figure 3: FTIR of Acetylated *C. bicolor* Starch at 15ml of Acetic Anhydride

CONCLUSION

The research considered how acetylation altered the physicochemical characteristics of *C. bicolor* starch. Current evidence suggests that *C. bicolor* starch extracted in this way can be acetylated using acetic anhydride. FTIR data indicated several functional groups linked with starch and also validated its acetylation, whereas the results of the other analyses showed that acetylated starch had a lower degree of substitution, a higher percentage of acetyl groups, and superior paste clarity.

In comparison to unmodified starch, acetylated starch performed better in tests of gelatinization temperature, water binding capacity, oil binding capacity, and solubility at varying amounts (5mL, 10mL, 15mL) and periods (30minutes, 60minutes, and 90minutes). Because of the hydrophilic group that holds water and the fact that hydrogen bonds can be made, acetylation usually makes starch better at holding both water and oil. The results of this study suggest that *C. bicolor* starch could be used in the non-food and textile industries to make adhesives, detergents, plywood, pastes, and glues.

Competing Interests

The authors declare that they have "no competing interests" in this article.

Funding

All the funds used in each stage of the research were personal funds from the authors.

Acknowledgements

I wish to thank the department of pure and industrial chemistry at the University of Port Harcourt for making the laboratory and the departmental library available during the period of this work.

Authors' Contributions

Omuluche, C.O., conducted the experiment on the effect of acetylation on the physicochemical properties of starch from *Caladium bicolor*. The study was designed by Prof. Abayeh, O.J., and the experiment was supervised by Dr. Achugasim, O., and Dr. Duru, R.U. The spectra were interpreted by all the authors. The manuscript was read by all the authors before submission.

Reference

1. Aburto J, Alric I, Thiebaud S, Borredon E, Bikiaris D, Prinos J, Panayiotou C. Synthesis, characterization, and biodegradability of fatty-acid esters of amylose and starch. *Journal of Applied Polymer Science*, 1999;*74*(6): 1440-1451
2. Fang JM, Fowler PA, Tomkinson J, Hill CAS. The preparation and characterisation of a series of chemically modified potato starches. *Carbohydrate Polymers*, 2002; *47*(3), 245-252.
3. Mathew S, Abraham TE. Physico-chemical characterization of starch ferulates of different degrees of substitution. *Food Chemistry*, 2007;*105*(2): 579-589.
4. Singh J, Kaur L, & McCarthy OJ. Factors influencing the physico-chemical, morphological, thermal and rheological properties of some chemically modified starches for food applications—A review. Food hydrocolloids. 2007; 21(1), 1-22
5. Xu, Y., Miladinov, V., & Hanna, M. A. (2004). Synthesis and characterization of starch acetates with high substitution. *Cereal chemistry*, *81*(6), 735-740.
6. Biswas, A., Shogren, R. L., Selling, G., Salch, J., Willett, J. L., & Buchanan, C. M.. Rapid and environmentally friendly preparation of starch esters. *Carbohydrate Polymers*, 2008; *74*(1), 137-141.
7. Singh J, Kaur L, & Singh N. Effect of acetylation on some properties of corn and potato starches. Starch-Stärke. 2004; 56(12), 586-601
8. Nsude OP, Agboeze E, Ezeh EC, Ike OC, Omuluche OC, Orie KJ., & Ogbobe O. Isolation and characterization of cellulose from Pentaclethra macrophylla Benth pod biomass wastes for polymer reinforcement composite. Journal of Chemical Society of

Nigeria.2022; 47(3). https://journals.chemsociety.org.ng/index.php/jcsn/article/view/765

9. Odugbemi T. (Ed.). Outlines and pictures of medicinal plants from Nigeria. 2008; University of Lagos Press, p, 283. https://searchworks.stanford.edu/view/7516604
10. Amadi O. C., Onyema, N., Nwagu, T. N., Moneke, A. N., Okolo, B. N., & Agu, R. C. Evaluating the potential of wild cocoyam "caladium bicolor" for ethanol production using indigenous fungal isolates. *Procedia Environmental Sciences*, 2016; *35*, 809-817.
11. Trela VD, Ramallo AL, & Albani OA. Synthesis and characterization of acetylated cassava starch with different degrees of substitution. Brazilian Archives of Biology and Technology. 2020; 63.
12. El Halal, SLM, Colussi R, Pinto VZ, Bartz J, Radunz M, Carreño NLV, & da Rosa Zavareze E. Structure, morphology and functionality of acetylated and oxidised barley starches. Food chemistry. 2015; 168, 247-256.
13. Mbougueng PD, Tenin D, Scher J, Tchiégang C. Influence of acetylation on physicochemical, functional and thermal properties of potato and cassava starches. Journal of Food Engineering. 2012; 108(2), 320-326
14. Osundahunsi OF, Seidu KT, & Mueller R. Effect of presence of sulphurdioxide on acetylation and sorption isotherm of acetylated starches from cultivars of cassava. Food Chemistry. 2014; 151, 168-174.
15. Larotonda FD, Matsui KN, Soldi V, & Laurindo JB. Biodegradable films made from raw and acetylated cassava starch. Brazilian Archives of Biology and Technology. 2004; 47, 477-484.
16. Montague ET, & Airul H. Identification and Modeling of Inter-Reservoir Cross-flow Using a Novel Material Balance Method. Paper presented at the SPE Asia Pacific Oil & Gas Conference and Exhibition, Adelaide, Australia, October 2014. doi: https://doi.org/10.2118/171488-MS
17. Zuhra, S., Khan, N. S., & Islam, S. (2018). Magnetohydrodynamic second-grade nanofluid flow containing nanoparticles and gyrotactic microorganisms. *Computational and Applied Mathematics*, *37*, 6332-6358.
18. Amadi OC, Onyema N, Nwagu TN, Moneke AN, Okolo BN, & Agu RC. Evaluating the potential of wild cocoyam "caladium bicolor" for ethanol production using indigenous fungal isolates. Procedia Environmental Sciences. 2016; 35, 809-817.
19. Hirsch, J. B., & Kokini, J. L. (2002). Understanding the mechanism of cross-linking agents (POCl3, STMP, and EPI) through swelling behavior and pasting properties of cross-linked waxy maize starches. *Cereal chemistry*, *79*(1), 102-107.
20. Sharlina ME, Yaacob WA, Lazim AM, Fazry S, Lim SJ, Abdullah S, & Kumaran M. Physicochemical properties of starch from Dioscorea pyrifolia tubers. Food Chemistry. 2017; 220, 225-232.
21. Zhang L, Li G, Wang S, Yao W, & Zhu F. Physicochemical properties of maca starch. Food chemistry. 2017; 218, 56-63.
22. Tang H, Mitsunaga T, & Kawamura Y. Relationship between functionality and structure in barley starches. Carbohydrate polymers. 2004; 57(2), 145-152.

23. Sindhu R, & Khatkar BS. Physicochemical and functional properties of starch and flour of tartary buckwheat (F. tataricum) grains. Int J Eng Res Tech. 2016; 5(6), 315-320.
24. Jiang Q, Gao W, Li X, Xia Y, Wang H, WuS, Xiao P. Characterizations of starches isolated from five different Dioscorea L. species. *Food Hydrocolloids*, 2012; *29*(1), 35-41.
25. Uwem IN, & Ita BN. Physicochemical, functional and antinutritional properties of starches from Caladium bicolor and Dioscorea dumentorum. European Journal of Chemistry. 2019; 10(3), 228-233
26. Hoover R, Sosulski F. Effect of cross-linking on functional properties of legume starches. *Starch‐Stärke*, 1986; *38*(5), 149-155.
27. Mark AM, Mehltretter CL. Facile preparation of starch triacetates. *Starch‐Stärke*, 1972; *24*(3), 73-76.
28. Boutboul A, Giampaoli P, Feigenbaum A, Ducruet V. Influence of the nature and treatment of starch on aroma retention. *Carbohydrate polymers*, 2002; *47*(1), 73-82.
29. Otache MA, Duru RU, Achugasim O, & Abayeh OJ. Advances in the modification of starch via esterification for enhanced properties. Journal of Polymers and the Environment, 2021; *29*, 1365-1379.
30. Nsude OP, & Orie KJ. Microcrystalline Cellulose of Oil Bean Pod: Extraction, Physico-chemical, Brunauer–Emmett–Teller (BET), and Flow-ability Analysis. Asian Journal of Applied Chemistry Research, 2022; *12*(4), 1-12.
31. Rudnik E, Matuschek G, Milanov N, & Kettrup A. Thermal properties of starch succinates. Thermochimica Acta. 2005; 427(1-2), 163-166
32. Thiebaud S, Aburto J, Alric I, Borredon E, Bikiaris D, Prinos J, & Panayiotou C. Properties of fatty‐acid esters of starch and their blends with LDPE. Journal of Applied Polymer Science. 1997; 65(4), 705-721.
33. Atichokudomchai N, & Varavinit S. Characterization and utilization of acid-modified cross-linked Tapioca starch in pharmaceutical tablets. Carbohydrate polymers. 2003; 53(3), 263-270.
34. Mishra S, & Rai T. Morphology and functional properties of corn, potato and tapioca starches. Food hydrocolloids. 2006; 20(5), 557-566.
35. Diop CIK, Li HL, Xie BJ, & Shi J. Effects of acetic acid/acetic anhydride ratios on the properties of corn starch acetates. Food chemistry. 2011; 126(4), 1662-1669.