

## Original Research Article

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

#### **ABSTRACT**

The study reports on the effects of acetylation on physicochemical properties of starch from *Caladium bicolor*. The starch obtained from *Caladium bicolor* was isolated and acetylated with different volumes of acetic acid and reaction times (30, 60, and 90 minutes). 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°C (unmodified), and 76–78 °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<sup>-1</sup>, 1018 cm<sup>-1</sup> and 1240 cm<sup>-1</sup> affirm the presence of OH, C-O, and CH<sub>3</sub> in the unmodified. Whereas, the prominent peak at 1647.28 cm<sup>-1</sup> 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*, Physico-chemical properties, Modified and Modified starch.

#### **1.0 Introduction**

Starch is an attractive raw material due to its abundant supply, low cost, biodegradability, biocompatibility and ease of chemical modification. Indeed, modified starches have been approved for food use, in which they act as thickeners, gelling agents, as sizing agents in textiles and as adhesives for paper and paper products. In recent years, several authors [1,2] have reported the preparation of modified starches with high degrees of substitution, using organic solvents. Reactions with starches, to prepare highly substituted derivatives, are not easy, mainly because of the almost impossible proposition of dissolving the granular starch in a suitable medium without significant degradation [3]. The introduction of an ester group into polysaccharides constitutes an important synthetic task, as it modifies their original hydrophilic nature and yields enhanced or new thermal and mechanical properties [1]

Starch modification, which implies the alteration of physical and chemical characteristics of native starch, is used to improve its functional characteristics and adapt it to specific industrial or domestic uses. A common and widely used modification in starch is acetylation that is the esterification reaction of starch with acetyl groups to form starch acetates [4].

Starch in its raw form has limited applications; therefore, it does not always have the desired properties for certain types of processing; but when modified, it increases its range of use. Chemically modified starch by acetylation has been widely studied. Acetylation may be performed to improve the physical, chemical and functional properties of starch [5]. In the acetylation process, the hydroxyl groups of the glucose monomers are converted to  $\text{CH}_3\text{COO}-$  groups; therefore the acetylation is an esterification of hydroxyl groups in the anhydroglucose unit of the starch molecule.

Acetylated starch is classified according to its degree of substitution (DS). Starches with low DS ( $<0.2$ ) are commonly used in the food industry, since they confer consistency, texture and stability, to baked goods, cake fillings, sauces and soups. Starches with medium DS ( $> 1.0$ ) are used as cellulose acetate substitutes [6]. Starches with high DS (2-3) are characterized by their thermoplasticity and their solubility in organic solvents such as acetone and chloroform have been used for non-food applications such as adhesives, coatings, biodegradable plastics and pharmaceutical applications[7,8].

*Caladium bicolor*(family Araceae) is an underutilized, inedible root tubers found abundantly in the wild in the south and south east regions of Nigeria. *C. bicolis* used as an ornamental plant in some areas, the leaves and rhizomes are used medicinally as topical application for boils, wounds and ulcers. It is used as purgatives and in the treatment of convulsion[9]. The potential of its flour for bio-ethanol production using indigenous fungal isolates has been reported[10].

Several researches on changes in the properties of acetylated starches in different range of degree of substitution, involving varied sources of starch have been carried out. However, few studies are based on effect of acetylation on the physicochemical properties of starch from *caladium bicolor*and with limited application of acetylation methodology. Singh et al., [4] studied the effect of acetylation on some properties of corn and potato starches in a range of DS 0.104 - 0.154. In other works, the effect of starch acetylation from different rice cultivars in a range of DS 0.107 - 0.144 were studied[11]. El Halal et al. [12] studied the effect of acetylation on the properties of low, medium and high amylose rice starch. Trela et al.,[11] studied the effect of different catalyst concentrations on the acetylation reaction of barley starch. Xu et al. [5] carried out the synthesis and characterization of acetylated maize starch with DS between 0.57. -2.59.

Mbougoung et al.[13] studied the influence of acetylation time on physico-chemical, functional and thermal properties of cassava and potato starches in a DS range lower than 0.26. Osundahunsi et al. [14] studied the effect of acetic anhydride on the acetylation reaction of two bread species in the DS range of 0.03 and 0.213. Larotonda et al.[15] used acetylation reaction in acid medium to obtain acetylated cassava starch with degrees of substitution in the range of 1.22 and 1.56 to form biodegradable films.

Osundahunsi et al. [14] working with acetylated cassava starches, observed that the swelling power and solubility increased with increase in the degree of substitution. Mbougoung et al. [13] studying potato and cassava starch found that for DS between 0 and 0.26, the solubility increased as a function of DS. They also found that the clarity of acetylated starch pastes increased with respect to the native one and in the cassava starch, it increased with the DS. Diop et al. (2011) observed that the solubility decreases as a function of DS in a range (1.83-

2.9) in corn starch. This complex relationship of the physicochemical properties of acetylated starches with degree of substitution makes necessary a systematic study of the characteristics of acetylated cassava starch covering a wide range of degree of substitution, as it is intended in this study. The research is aimed at the isolation and physicochemical analysis of unmodified and acetylated starch obtained from *C. bicolor*.

## 2.0 Material and Methods

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### 2.1 Extraction of starch

This extraction method used here was reported by Osundahunsi et al. [14]. Fresh tubers of *Caladium bicolor* were washed, peeled, cut into small pieces and blended with distilled water (3:1) for 1 min using a blender (Sharp SM110, Japan). Then, it was filtered using a double fold cheesecloth. The filtrate was transferred into a beaker and left overnight so that the starch precipitated on the bottom of the beaker. The supernatant was discarded, and the starch precipitate was washed three times with distilled water; then, it was dried in an oven at 45°C until a constant weight was achieved. The dried starch was subsequently pulverized using a grinder and then stored in polyethylene bags until the next step of the experiment. The starch content was calculated using equation 1

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

### 2.2 Amylose and amyloectin content

The starches were analysed for their amylase and amylopectin content using a colorimetric assay following a modified published protocol [8]. 5 mg of starch were mixed with 1 mL of 90% DMSO followed by incubation at 95.0°C for 1 h. Afterwards, the sample was diluted with 3 g/L iodine in 90% DMSO at a ratio of 1:1, and then 10 times diluted with water. Finally, absorbance at 635 nm was measured using a UV-Vis spectrophotometer (Genesys 10S, Thermo Scientific, Loughborough, UK). Amylose content reported as percentage.

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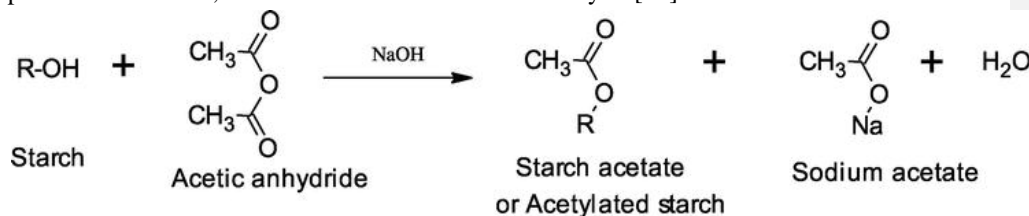
### 2.3 Acetylation/esterification of starch

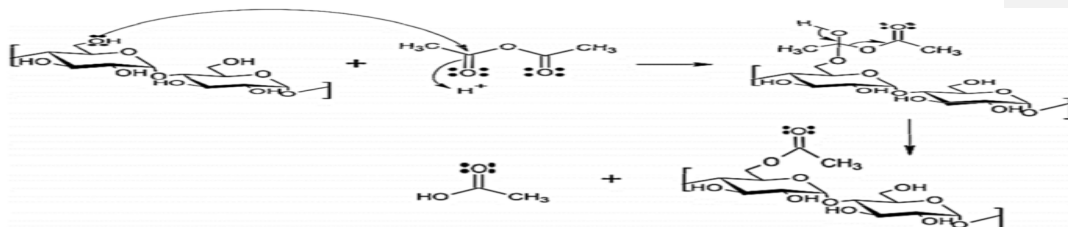
Acetylated starch was prepared using acetic anhydride as reactant. 25 g starch was dispersed in 75 ml of distilled water with constant shaking for 1 hour. The pH of the suspension was adjusted to 8.0 with 3% NaOH solution. Slurry was reacted with acetic anhydride v/v (10, 15 and 20%) and heated with a water bath at 45°C with a soaking time (30, 60, 90 minutes). The reaction product was filtered; the precipitate was washed to a neutral pH. The precipitate obtained was kept in an oven at a temperature of 60°C to constant levels. Starch acetate produced was dried, mashed and sieved for further analysis [17].

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

The acetyl percentage (Ac%) and degree of substitution (DS) of acetylated starch were determined using titration method. Acetylated starch (1 g) was mixed with 50 ml 75% ethanol in distilled water. The flask containing the slurry was covered with aluminium foil and heated in water bath at 50°C 30 min. The samples were cooled and 40 ml of 0.5 M NaOH was added. The excess alkali in the slurry was titrated with 0.5 M HCl using phenolphthalein as an indicator until pink color disappeared. A blank using native starch was also carried out. The acetyl % and degree of substitution calculated according to equations (2) and (3) respectively [17].

$$\text{Acetyl \%} = \frac{(V_{\text{blank}} - V_{\text{sample}}) \times \text{Molarity of HCl} \times 0.043 \times 100}{\text{Sample weight}} \quad (2)$$

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

## 2.5 Determination of pH

This was done using a pH meter (model HI 8424 with pH buffer 7). 1.5g of starch sample was dissolved in beaker with 10ml of distilled water and stirred properly. The pH meter was inserted into the solution and the reading taken, the same procedure was repeated in triplicate and average value recorded.

## 2.6 Determination of Gelatinization Temperature

1.5g of starch sample was dissolved in a beaker with 10ml of distilled water, mixture is stirred, a thermometer inserted and beaker placed in a water bath. The solution was stirred continuously until its colour became milky and thickened. This is the gel point and the temperature at this point was read off as the gelatinization temperature [18] the same procedure was repeated in triplicate and average value recorded.

## 2.7 Determination of Swelling Volume, Swelling Power and Solubility

The swelling power and solubility were determined in accordance with the methods described by Hirsch and KoKini, [19]. The samples were poured into graduated centrifuge tubes that were appropriately labelled. The solution was stirred, placed in a water bath heated to 95°C while shaking the sample gently to ensure that the starch granules remained in suspension until gelatinization occurs. The gelatinized samples were held at 95°C in the water bath for 1 hour. The samples were cooled to room temperature under running water and centrifuged for 30 minutes at 1000rpm. After centrifuging, the swelling volume was obtained directly by reading the volume of swollen sediment in the tube. The solution was removed from the sediment, placed in a metal dish, weighed, dried at 105°C for 1 hour, weighed, and dried

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again. This technique was repeated three times, and the average value was recorded. The starch swelling power and solubility were determined according to equations (4) and (5).

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

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

## 2.8 Water binding capacity (WBC)

Water binding capacity was determined using methods by Das et al. (2010) with slight modification. A suspension, 5 g of starch in 75 ml distilled water was agitated for 1 hour and centrifuged at 3000 rpm for 10 minutes. The free water was removed from the wet starch, which was then drained for 10 minutes. The wet starch was then weighed; the same procedure was repeated in triplicate and average value recorded.

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

## 2.9 Oil-binding capacity (OBC)

OBC of the starches was determined using methods of Das et al. (2010). A suspension, 5 g of starch in 75 ml oil was agitated for 1 hour and centrifuged 3000 rpm for 10 minutes. The free oil was removed from the wet starch, which was then drained for 10 minutes. The residue was then weighed the same procedure was repeated in triplicate and average value recorded.

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

## 2.10 Paste clarity

Paste clarity was measured using the method of Zhang et al., (2017). Starch (1% w/v) was suspended in water and thereafter heated in a boiling water bath for 30 minutes with shaking. The resulting gel was then analyzed in duplicate for its transmission (%T) at 650 nm using a unicospes UV-2150 spectrophotometer.

## 2.11 FTIR Analysis

FTIR was carried out to understand the functional group variation during acetylation of starch of obtained *C. bicolor*. PerkinElmer 1600 Infrared spectrometer was used for the detection of various functional groups introduced during the acetylation. The spectra were collected by the spectrometer with 32 running scans at a resolution of 4 cm<sup>-1</sup> for each sample within 650–4000 cm<sup>-1</sup> range. The “find peak tool” functionality of Nicolet software was used to determine the positions of significant transmittance peaks at a particular wave number.

## 3.0 Results and Discussion

### 3.1 Amylose and amylopectin content

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Amylose and amylopectin content of the unmodified and modified starch with acetic acid at different concentration and at 90 minutes 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 pycnostachya* tubers with starch contains;  $44.47 \pm 1.86\%$  amylose, and Zhang et al. [21] who worked on physicochemical properties of maca starch apparent and estimated amylose contents ranged from 21.0 to 21.3%. The low amylose content observed in *C. bicolor* starches suggested its use in situations where soft gels/films are needed such as in the glue, detergent, plywood and textile industry.

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### 3.2 Swelling Power and Water Solubility

Swelling power and water solubility of the unmodified and modified starch with acetic acid at different volumes and at different times are shown in Table 1-3. 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  $30^{\circ}\text{C}$ , 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-line with Tang et al. [22], who studied the relationship between functionality and structure in barley starches, and Sindhu and Khatkar [23], who worked on the physicochemical and functional properties of starch and flour of tartary buckwheat (*F. tataricum*) grains.

Sindhu and Khatkar [23] postulated that swelling power and water solubility index provides evidence of the magnitude of interaction between starch chains with both the amorphous and crystalline domains, and gives a measure of the hydration status of starch molecules. In the same vein, Sharlina et al. [20] opined that the extent of cross-bonding within the granules and the presence of lipids or phosphates affect the swelling and solubility profiles of starches. The high swelling power and solubility index exhibited by the starches at different volumes of acetylation may be attributed to its low amylose content and low lipid content as high amylose content and strong intermolecular bonds reduce swelling. These suggest that these starches may find application in adhesives, pastes and glues in the non-food industries [21].

### 3.3 Water and Oil Binding Capacity

Water and oil binding capacity of starch obtained from *C. bicolor* at different volume and time of acetylation is shown in Tables 1-3. Water binding capacity of the acetylated starches was greater than unmodified starch. The high water binding capacity of acetylated starch is attributed to the extent of hydrogen bonding between water molecules and starch

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hydroxyl groups as well as loose association between amylose and amylopectin molecules in starch granules. This is supported by Jiang et al. [24], who researched on the characterizations of starches isolated from five different *Dioscorea L. species*. The values of starch acetylation, water binding capacity increases as the volume of acetic acid increases, and this could also be attributed to the increased solubility. This is in-line with the findings of Uwem and Ita[25] who worked on physicochemical, functional and antinutritional properties of starches from *Dioscorea adumentorum*. The diversity in starches' Water Binding Capacity (WBC) may be influenced by the dissimilarity in a degree of water binding area availability inside the granule [12].

The oil binding capacity of *C. bicolor* starch is showed in Tables 1-3, with different acetylation volumes of modified starch. From the findings, the acetylated starch has high binding capacity for both vegetable and crude oil, which is higher than the unmodified starch. The oil binding capacity increased as the volumes of acetic acid increased. This is attributed to higher value of acetyl content in the starch molecules which might have caused maximal oil binding capacity due to structural re-orientation of the starch molecules [26]. Low water binding capacity of starches could be attributed to the involvement of a larger proportion of the hydroxyl groups in forming hydrogen and covalent bonds between the starch chains than with water [27].

**Table 1: Physicochemical Properties of Acetylated Starch from *Caladium Bicolor* at 45°C for 90minutes, at Different Acetic Acid Values**

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Parameters	unmodified	Modified		
		5ml	10ml	15ml
Volume		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°C(%)	38.5	54.2	54.2	54.6
Swelling power	72.45	67.5	67.8	68.5

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

**Table 2: Physicochemical Properties of Acetylated Starch from *Caladium Bicolor* at 45°C for 60minutes, at Different Acetic Acid Values**

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Parameters	Unmodified	Modified		
		5ml	10ml	15ml
Volume		5ml	10ml	15ml

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<b>pH</b>	4.49			
<b>Gelatinization temp.</b>	75	75	76	76
<b>Amylose %</b>	16.95	NA	NA	NA
<b>Amylose pectin %</b>	83.05%	NA	NA	NA
<b>WBC</b>	68.6	84.1	85.9	88.4
<b>OBC(Vegetable oil)%</b>	58.5	58.5	58	59.2
<b>OBC(Crude oil)%</b>	42.1	57.5	58.2	58.8
<b>% acetyl group</b>	NA	0.71	1.14	1.78
<b>DS</b>	NA	0.04	0.03	0.07
<b>Past clarity</b>	2.013A, 0.97%T	1.65A, 1.53%T	1.62A,1.53%T	1.69A, 1.60T
<b>Solubility at 30<sup>0</sup>C(%)</b>	38.5	53.1	53.1	53.4
<b>Swelling power</b>	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 *Caladium Bicolor* at 45°C for 30minutes, at Different Acetic Acid Values**

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

Percentage of acetyl group and degree of substitution of acetylated starch obtained from *C. bicoloris* shown in Tables 1-3. The degree of substitution of a starch is the number of hydroxyl (OH) groups substituted per D-glucopyranosyl structural unit of the starch polymer. Since each glucose unit possesses three reactive hydroxyl groups, the maximum possible DS

value is 3. The primary OH group on C-6 is more reactive and is esterified more readily than the secondary ones on C-2 and C-3 due to steric hindrance. The DS varies with the source of starch, amylose and amylopectin fractions, stoichiometric amounts and reaction time.

According to Mark and Mehlretter[28], the starch acetates, in accordance with DS, are classified as low DS (<0.1), medium DS (0.1–1.0) and high DS (>1.0) acetated. There is a commercial interest in starches with a DS of 0.01–0.20 because of their use based on properties for film formation, adhesion, thickening, stabilizing and texturizing [29].

The acetylated starch with a low DS is commonly obtained by the esterification of native starch with acetic anhydride, in an aqueous medium, and in the presence of an alkaline catalyst.

The DS within the time limit of 30 minutes is on the range of 0.12-0.14. The degree of substitution at 30 minutes is said to be at medium. The acetylated product is best suited for film formation, adhesion, thickening, stabilizing and texturizing. The DS range of 60-90 minutes is said to be low based on the values within the range of 0.03-0.094(<0.1). The findings also indicate that increase in the volume of acetic acid in each of the time considered is associated with increase in the degree of substitution [4, 29]

The esterification of starch with organic acids results in thermoplastic and hydrophobic materials when the DS is high enough [27, 29]. The DS of the starch acetate increased with higher ratios of acid chloride to starch. Higher volume acetic acid resulted in a higher rate of molecular collision and a greater availability of acetic acid molecules in the vicinity of starch [7]. The hydrophobicity of starch increases with the increasing DS, which in turn increases the miscibility of starch with other hydrophobic polymers [7, 30]. These observations were consistent with the finding of the research, and as such, *C. bicolor* starch can be used as thermoplastic and polymer industries.

### 3.5 Paste Clarity (PC)

Paste clarity (PC) is an important property that reflects the transparency of a gel. Differences in paste clarity were observed between the times and different volume of acetic acid in the acetylation of starch obtained from *C. bicolor* as shown in Tables 1-3. The paste clarity of the unmodified starch was greater than that of the acetylated starch. This could be attributed to the degree of substitution and the percentage of acetyl group in the modified starch. There was variation in the paste clarity as the volume of acetic anhydride increases, and also as the time increases. This is consistent with Singh et al. [7], who researched on the effect of acetylation on some properties of corn and potato starches. The low clarity of cocoyam starch pastes could therefore be due to the presence of amylose molecules of high susceptibility to retrogradation

### 3.6 Gelatinization Temperature (GT)

The gelatinization temperature of the acetylated starch was greater than that of the unmodified starch as indicated in Tables 1-3, at different time and volume of acetic acid. 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 amylose, 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 Figure 1-3. In the IR spectrum of unmodified starch, there are several discernible peaks at  $1018\text{cm}^{-1}$ ,  $1022.31\text{cm}^{-1}$ , and  $1153.74\text{cm}^{-1}$ , which were attributed to the C–O bond stretching. Other characteristic absorption bands were observed at around  $1639.55\text{cm}^{-1}$ , corresponding to the tightly bound water ( $\text{H}_2\text{O}$ ) in the starch, an extremely broad band at around  $3367.11\text{cm}^{-1}$ , resulting from the vibration of the hydroxyl group (O–H); and at around  $2930.90\text{cm}^{-1}$ , attributed to the C–H vibration stretch[34, 35].

After acetylation by acetic anhydride (5 ml), the absorption bands at  $1647.28\text{cm}^{-1}$  became prominent in intensity, with a corresponding decrease in the absorption bands at  $3387.11\text{cm}^{-1}$ . The peak at  $1647.28\text{cm}^{-1}$  increased in intensity, while the peak at  $3387.11\text{cm}^{-1}$  decreased in intensity with the use of 15ml of acetic anhydride. The peak at  $1647.28\text{cm}^{-1}$  was assigned to the carbonyl C=O of an ester, while the peak at  $3387.11\text{cm}^{-1}$  was assigned to the hydroxyl group. These new absorption bands at  $1647.28\text{cm}^{-1}$  and  $1720.18\text{cm}^{-1}$  suggest that the ester carbonyl groups were formed during the esterification process in starch[9, 30].

The intensity and height of these peaks are highly dependent on the degree of substitution. A low degree of substitution of acetylated starch is shown at the peaks of  $3387.62\text{cm}^{-1}$  and  $1647.28\text{cm}^{-1}$  (Figure 2), which were associated with 5 ml of acetic anhydride acetylation. Figure 3 also has peaks at  $1647.28\text{cm}^{-1}$  and  $3387.11\text{cm}^{-1}$ , and its relative increase in intensity and height of the peaks was attributed to 15 ml of acetic anhydride, and also implies a better degree of substitution. This is consistent with Biswas et al. [6], who opined that an increased degree of substitution leads to the disappearance or decrease in the intensities of the peaks, areas, and heights at approximately  $3450\text{cm}^{-1}$ , particularly those related to the OH vibration.

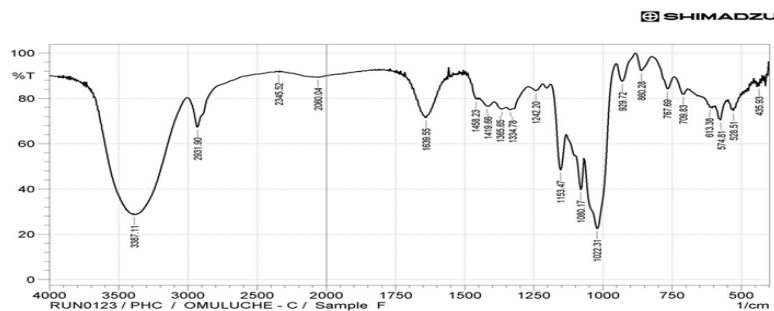


Figure 1: FTIR of Unmodified *C. bicolor* Starch

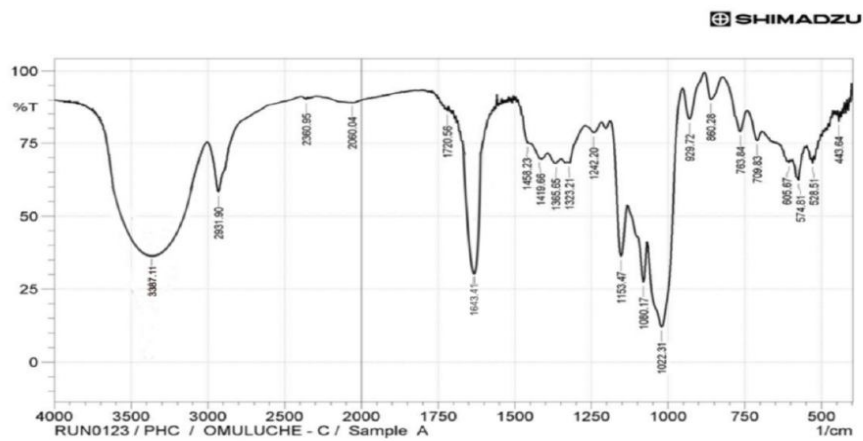


Figure 2: FTIR of Acetylated *C. bicolor* Starch at 5ml of Acetic Anhydride

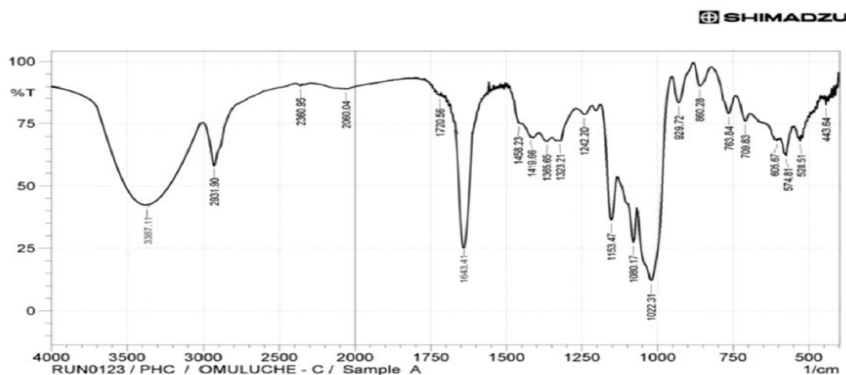


Figure 3: FTIR of Acetylated *C. bicolor* Starch at 15ml of Acetic Anhydride

### CONCLUSION

The effect of acetylation on the physicochemical properties of starch from *Caladium bicolor* was considered in the research. The analysis so far has affirmed that isolated starch of *C. bicolor* can be acetylated with acetic anhydride. The findings showed that acetylated starch has a lower degree of substitution, a higher percentage of acetyl groups, and greater paste clarity, while the FTIR results revealed some functional groups associated with starch and also confirmed its acetylation.

The gelatinization temperature, water binding capacity, oil binding capacity, and solubility of the acetylated starch across different volumes (5 mL, 10 mL, 15 mL) and times (30, 60, 90 minutes) were greater than those of the unmodified starch. In general, acetylation

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increased the water and oil binding capacity of starch due to the existence of the hydrophilic group that holds water and the capability to establish hydrogen bonds.

The findings from this study suggest that starch from *Caladium bicolor* may be a viable source of adhesives, detergents, plywood, pastes, and glues in the non-food and textile industries.

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### Competing Interests

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

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### 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.

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