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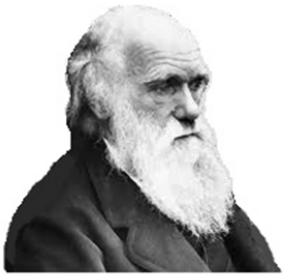

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Isolation of starch from acha grain (*Digitaria exilis* (Kippist) Stapf), modification, characterization and application of benzoylated starch in stabilization of oil in water emulsion

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ABSTRACT

Starch and its derivatives are biocompatible, biodegradable, non-toxic with applications in food, pharmaceutical and allied industries. In this study, starch was isolated from acha grain (*Digitaria exilis*

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(Kippist) Stapf). Esterification of starch was carried out using benzoyl chloride at room temperature (30 °C ± 2 °C). Characterization of native and benzoylated starch were achieved by FT-IR and XRD analyses. Emulsifying properties of native and benzoylated starch were reported. FT-IR spectra of native modified revealed the broad absorption bands at 3375.70 cm^{-1} was due to stretching hydrogen bonded O-H groups in the starch. However, for the modified starch, the major new absorption bands at 1600.92 cm^{-1} which is associated with C=O esters indicating the modification of the native starch confirms that esterification took place in the starch molecules and the band in the region 1716.65 cm^{-1} confirms the water adsorption. The X-Ray diffraction pattern of native and acetylated starch samples prominent peaks (2) at 18.08 and 19.02 respectively. After acetylation, slightly reduction in crystallinity was observed. The loss of crystallinity would mean enhanced ability for acetylation starch or other polymer products made from it to absorb water. The native and benzylated starch significantly enhanced the stability of water of oil water emulsion.

***Keywords*:** Starch, biodegrable, modification, acetylation, *Digitaria exilis*

1. INTRODUCTION

Starch is a commonly used biodegradable polymer which is increasingly used in many branches of industry because of its respective physio-chemical properties (Haq et al, 2019). In addition, it is a naturally occurring, biodegradable, inexpensive and abundantly available polysaccharide molecule. It is widely distributed in the form of tiny granules as the major reserve carbohydrate in stems roots, grains, and fruits of all forms of green leafed plants. This section reviewed related materials and studies on Acha, chemical modification, physiochemical and functionality, characterization and application of starch.

Starch is a mixture of linear amylose (poly-α-1,4-D-glucopyranoside) (molecular weight of 10^4-10^6 g mol^{-1}) and branched amylopectin (poly-α-1,4-D-glucopyranoside and α-1,6-D-glucopyranoside) (molecular weight of 10^6-10^8 g mol^{-1}), where it is regenerated from carbon dioxide and water via photosynthesis in plants (Averous & Pollet, 2012). About 70% of the mass of a starch granule is amorphous, which consist mainly of amylose, while ~ 30% is crystalline, which contain primarily of amylopectin (Sajilata, Singhal, & Kulkarni, 2006; Perez, 2009; Ochubiojo & Rodrigues, 2012). The chemical structures of amylose and amylopectin are depicted in Figure 1. Structure of (a) amylose and (b) amylopectin.

Acha (Fonio) is a cereal crop of West African origin belonging to the family Graminaea with scanty knowledge about its evolution, origin, distribution and genetic diversity even within West Africa itself, this is despite its ancient heritage and widespread importance as reported by Adoukonou, (2006). The crop is grown in various parts of Nigeria, Sierra Leone, Ghana, Guinea Bissau, Senegal, Togo, Mali, Benin Republic and Cote d'Ivoire (Jideani, 1999, Gyang and Wuyep, 2005) goes by various names, such as fundi, findi, acha or "hungry rice". The crop has been so neglected that it is called the lost crop of Africa, having received but a fraction of the attention accorded to sorghum, pearl millet, and maize.

Abdul & Jideani, (2019) recently reviewed there is a significant amount of the new knowledge published on the knowledge of the nutritional properties and food applications of Acha (Fonio), which is essential to support the development of the breeding programs. The process of altering native starch's usual characteristics through enzymatic, chemical, or physical techniques is known as modification. Starch is commonly converted or modified into useful

derivatives which has made the demand of cellulose high in recent years and has found its usefulness in so many industrial application, on industrial scale, chemical modifications are still the most commonly used They also offer a number of desirable properties such as high viscosity, better thickening power, low gelatinization and retrogradation. Chemical modification is generally achieved through derivatization such as etherification, esterification, cross-linking and acid hydrolysis (Zia-ud-Din *et al.*, 2017). In order to improve starch pasting properties and shelf life extension, modification of starch by using chemical is the most frequently used method granular molecular re-arrangement of potato starch granules which resulted into changes relate different physicochemical properties of treated starch inducing some new characteristics and functions.

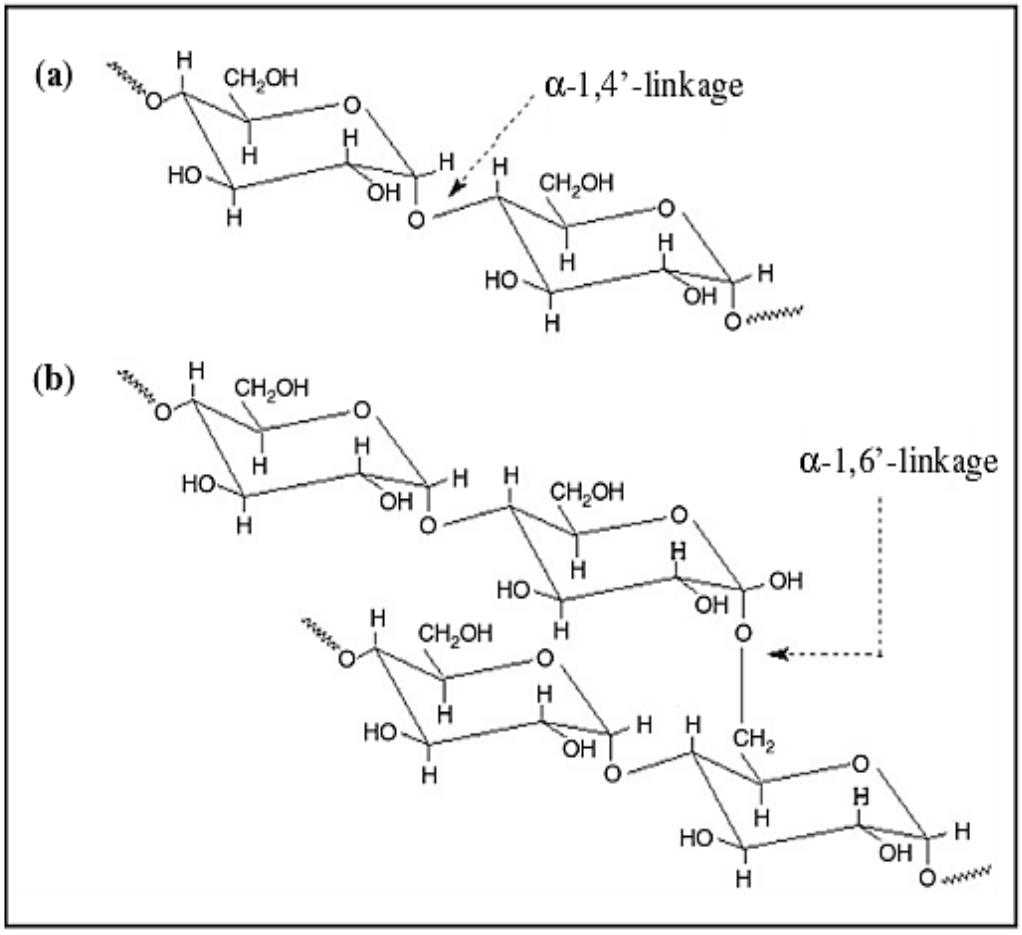

Figure 1. The structure of amylose and amylopectin.

Among them are three basic reactions: oxidation, esterification, and etherification (Matheus 2022). Esterifications as a form of chemical modification involve hydroxyl groups which are converted into hydrophobic ester groups. Another prominent method of chemical modification involves etherification through acetylation (Adebowale et al, 2005; Lawal et al, 2005). The several methods that produce modified starches for use in pharmaceuticals and other industrial applications are covered in this article.

2. METHODS

2. 1. Materials

White acha (*Digitaria exilis*) grain was purchased at a local market in Kastina, Kastina State, Nigeria.

2. 1. 1. Chemicals/Reagent

Sodium hydroxide (NaOH, Loba Chemie pvb. Ltd), benzoyl chloride (C_7H_5ClO, Sigma Adrich), Ethanol (C_2H_5OH, Analar). All reagent used were analytical grade.

2. 2. Isolation of Starch

The grains were screened, after being screened, the grains were cleaned with distilled water to remove any remaining pigment. Then the acha grain was soaked in distilled water (15L) for 6 hours and it was blend. The slurry obtained after blending was re-suspended in distilled water (40 mL.) and the pH was adjusted to 8.0, using NaOH solution (0.5M). It was then manually stirred for 2 minutes. The slurry was filtered using cheese cloth. The filtrate was thoroughly washed with tap water to separate the starch granules. The filtrate was decanted after being left to settle after 24 h. The suspension obtained was screened through mesh using the cheesecloth for 30 minutes before air-drying for 48 hours at 30 °C # 2 °C (Lawal, 2009).

2. 2. 2. Modification of Native Starch using Benzoyl Chloride

Soduim hydroxide (NaOH) was measured into a beaker (50 mL; 0.5M) the sample was added into it (1.000g). The stirrer was set to stir the solution for 30 minutes. After stirring vigourously for 30 minutes, benzoyl chloride was added drop-wise into the solution (5 mL), making use of the PH to maintain an alkaline meduim. It was then stirring for another 30 minutes. The solution was iter, The starch was filtered, and washed with distilled water and finally with 95% ethanol (100 mL). The starch residue was then dried overnight in a 40 °C convention oven to have the modified starch.

2. 3. Proximate Analysis

2. 3. 1. Determination of moisture content

This was done by the gravimetric method described by Lawal et al, (2005). A measured weight of the sample (5.0 g) was weighed into a previously weighed petri dish. The sample in the dish was dried in the oven for 3 h at 105 °C. It was allowed to ool in a dessicator and weighed. Then returned to the oven for further drying. Drying, cooling and weighing were done repeatedly at hourly interval until there were no further diminutions in the weight (that is, constant weight was obtained). The weight of moisture lost was calculated and expressed as a percentage of the weight of sample analyzed. It was given by the expression below;

$$\text{Moisture Content (\%)} = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

where:

W_1 = weight of empty moisture can

W_2 = weight of empty petri dish sample before drying

W_3 = weight of petri dish sample dried to constant weight

2. 3. 2. Determination of Ash content

The dried material from moisture content above was transferred to a muffle furnace. The porcelain dish in the muffle furnace was maintained at a temperature of 550 °C for 24 hours.

The dish now transferred to a dessicator and cooled. Ash content (total mineral constituents) is calculated using the equation below;

$$\text{Ash Content} = \frac{W_2 - W_1}{\text{Weight of Sample}} \times 100$$

where:

W_1 = Weight of empty crucible (g)
W_2 = Weight of crucible + Ash (g)

2. 3. 3. Determination of crude protein

This was done by Kjeldahl method described by Chang (2003). The total nitrogen was dermined and multiplied with factor 6.25 to obtain protein content. Sample (0.5 g) was mixed with concentrated 2504 (10 ml.) in digestion flask. A tablet of selenium eatalyst was added to it digest was diluted into a volumetric flask (100 ml.) and used for the analysis. The 10 ml. of the digest was mixed with equal volume of NaOH solution (45%) in a Kjeldahl distillation aparatus. The mixture was distilled into 40% boric acid (10 mL.) containing 3 drops of mixed indicator (bromocressol green/methyl red). Total distillates (50 mL) was collected and titrated apaint EDTA (0.02 N) from green to a deep red end point. A reagent blank was also digested, distilled and titrated. The nitrogen content and hence the protein content was calculated using the formula below:

1 mL of 1N H_2SO_4 = 14 mg

$$\text{Nitrogen } (N_2)\ (\%) = \frac{100}{W} \times \frac{N \times 14}{1000} \times \frac{V_t}{V_a} \times T.B$$

$$\text{Protein } (\%) = N_2(\%) \times 6.25$$

W = Weight of sample
N = Normality of titrant
Vt = Total digest volume
Va = Volume of digest analysed
T = sample titre value
B = Blank titre value

2. 3. 4. Determination of Crude Fibre

Crude fibre was determined by the method of James (1995). Sample (5.0 g) processed sample was boiled in solution for 30 min under reflux (150 mL; 1.25% H_2SO_4). The boiled sample was washed in several portions of hot water using a two-fold cloth to trap the particles.

It was returned to the flask and boiled again in 150 mL of 1.25% NaOH for another 30 min under (150 mL; 1.25% NaOH). After washing in several portion of hot water the sample was allowed to drain dry before being transferred quantitatively to a weighed crucible where it was dried in the oven at 105 °C to a constant weight. It was thereafter taken to a muffle furnace where it was burnt, only ash was left of it. The weight of the fibre was determined by difference and calculated as a percentage of the weight of sample analyzed thus:

$$\text{Fibre Content} = \frac{W_2 - W_3}{\text{Weight of Sample}} \times 100$$

where:

W_2 = Weight of empty crucible (g) + sample after washing, boiling and drying
W_3 = Weight of crucible + sample of ash

2. 3. 5. Determination of Fat

This was determined by solvent extraction gravimetric method described by Kirk and Sawyer (1980). Five gram of sample was wrapped in a porous paper (whatman filter paper) and put in a thimble. The thimble was put in a soxlet reflux fask and mounted into a weighted extraction flask containing of petroleum ether (200 mL). The upper of the reflux flask was connected to a water condenser. The solvent (petroleum ether) was heated, boiled vaporized and condensed into the reflux flask filled. Soon the sample in the thimble was covered with the solvent until the reflux flask filled up and siphoned over, carrying its oil extract down to the boiling flask. This process was allowed to go on repeatedly for 4 h before the defatted sample was removed, the solvent recovered and the oil extract was left in the flask. The flask (containing the oil extract) was dried in the oven at 60 °C for 30 min to remove any residual solvent. It was cooled in desiccator and weighed. The weight of oil (fat) extract was determined by difference and calculated as a percentage of the weight of sample analyzed thus;

$$\text{Fat Content (\%)} = \frac{W_2 - W_1}{\text{Weight of Sample}} \times 100$$

where;

W_1 = weight (g) of empty extraction flask
W_2 = weight of flask + oil (fat) extract

2. 3. 6. Determination of Carbohydrate

The carbohydrate is made up of starch and sugar. This value was derived by deducting the total of crude protein, crude fat, crude fiber, total ash and moisture content from 100.

2. 4. Modification of Acha Starch

The starch esterification was carried out in two steps. In the first step, native starch was dispersed in an alkali reaction medium, and in the second step, it was treated for esterification. Finally, hydrophobic starch esters were obtained. Starch (1g) was added to NaOH solution (50 ml, 0.5M) at room temperature with mechanical stirrer, until the starch granules getalinised fully. After 30 min, the benzoyl chloride was added dropwise (5 ml) and the reaction was

performed for 45 mins. Upon completion the mixture was neutralized to pH 10 with NaOH. The product was collected by filtration washed with water and it was recollected. Thereafter, it was washed twice with ethanol. Residual ethanol was removed by evaporation in air and the benzoylated esterified starch was dried at 50 °C overnight.

2. 5. Characterization

2. 5. 1. FT-IR Spectroscopy Analysis

FTIR spectra of native and modified starch were run as KBr pellets on Perkin Elmer Spectrum Two TM spectrometer in the frequency range 4000–400 cm^{-1} with resolutions of 2 cm^{-1} taking four scan for each sample.

2. 5. 2. X-Ray Diffraction (XRD)

Wide angle X-Ray diffraction patterns of powdered CMC and CMC-CH nanocomposites samples were obtained using Empyrean XRD diffractometer at 40 mA and 45 kV with Cu Kα (1.5418 Å) radiation at an angular incidence of 10-75°.

2. 5. 3. SEM Analysis

Scanning electron microscopy (SEM) was used for granule morphology studies. Using SEM, a thin layer of starch granule was mounted on aluminum specimen holder by double sided tape. The specimen holder was loaded in a polaron Sc 7610 sputter coater (fison instrument, England). i was coated with gold palladium, to a thickness of about 30mm. The specimen holder was then transferred to XI-20 series. Scanning electron microscope of cellulose and its derivatives were examined at 10K V (Lawal et al, 2005).

2. 6. Preparation of Water in Oil Emulsion

The powdered particle method was employed for the preparation of emulsion using equal volumes of water and oil (Øw = 0.5). 5 mL each of water and oil (1:1 v/v) were measured into 25 mL vial having an internal diameter of 20 mm and height of 100 mm. The denser liquid (water) was first measured and followed by the less dense liquid (vegetable oil). 0.1 g of native and modified acha was subsequently measured into separate 25 cm^3 screw-capped vials containing the water-vegetable oil mixture. The particle-water-vegetable oil mixture was homogenized at 12,000 rpm for 3 min. The drop test was carried out to determine emulsion type.

3. RESULTS AND DISCUSSIONS

3. 1. Proximate Analysis Result

Table 1. Proximate analysis result of *Digitaria exilis*.

Sample	Moisture%	Protein%	Fat%	Fibre %	Ash%	CHO%
Acha starch	14.50	4.86	0.93	0.12	0.11	79.48

The chemical composition of the native starch is presented in Table 1. All parameters studied include the moisture content, protein, fat, crude fiber, ash content and carbohydrate with the composition 14.50%, 4.86%, 0.93%, 0.12%, 0.11% and 79.48 respectively.

3. 2. Characterization Results

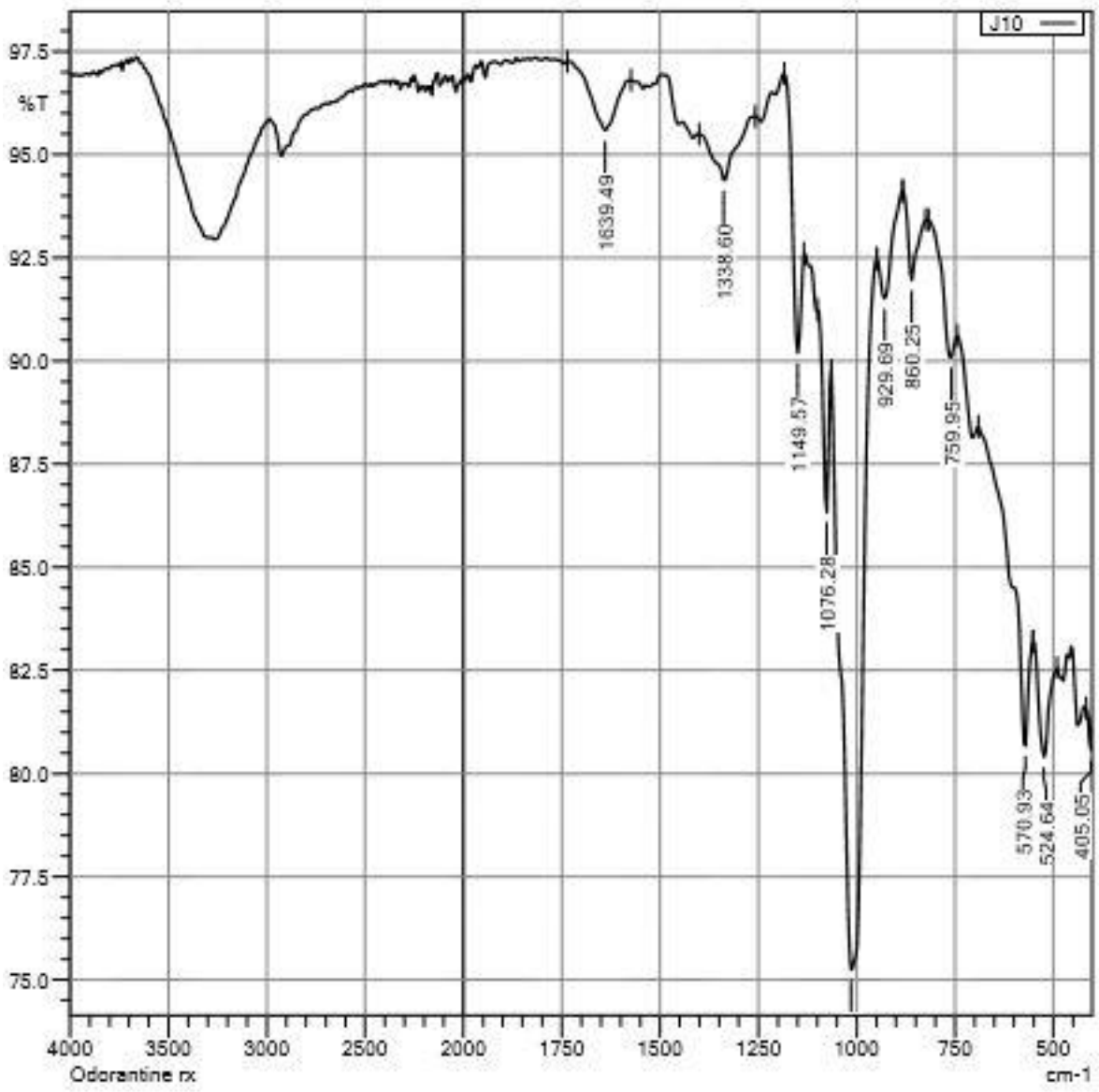

Figure 2. The Infrared Spectroscopy of the Acha Native Starch

The Discussion on the Acha Native Starch as shown in Figure 2

The FT-IR spectra of native sample distinctive peaks in both spectra are described as follows: The Fourier Transform Infrared (FTIR) spectra of the native acha starch is showed in

Figure 2. A typical absorption bands of a starch backbone, the broad absorption bands at 3375.70 cm^{-1} is due to stretching vibration of hydrogen bonded O-H groups in crystalline starch. This might be as a result of intermolecular hydrogen bonds in the glycosidic ring weakening the O–H bond, thereby shifting the band absorption region to a lower frequency between 3400 and 3200 cm^{-1}. The absorption bands at 2903.74cm^{-1} may be due to C-H (methyl; asymmetry) stretching. The absorption band at 1639,49 cm^{-1} is characteristic bands of the water bounded to the starch. The detection of bands at 1338.50 cm^{-1} and 1149.57 cm^{-1} may be attributed to δCH_2 and C-O-C asymmetric stretch vibration respectively and the C-O-C also stretching affirms to the linkages (Cerna et al., 2003).

The absence of the C=O band at 1700 – 1762 cm^{-1} in the FTIR of the native starch indicates that there is no any hemicellulose or lignin residue in the extracted starch sample and as well as none of the hydroxyl groups was oxidized during isolation process. In addition broad and weak bands which occurred in the range of 929.69 - 570.93 cm^{-1} probably arose from out of plane bonded O–H deformation and C–H deformation frequencies.

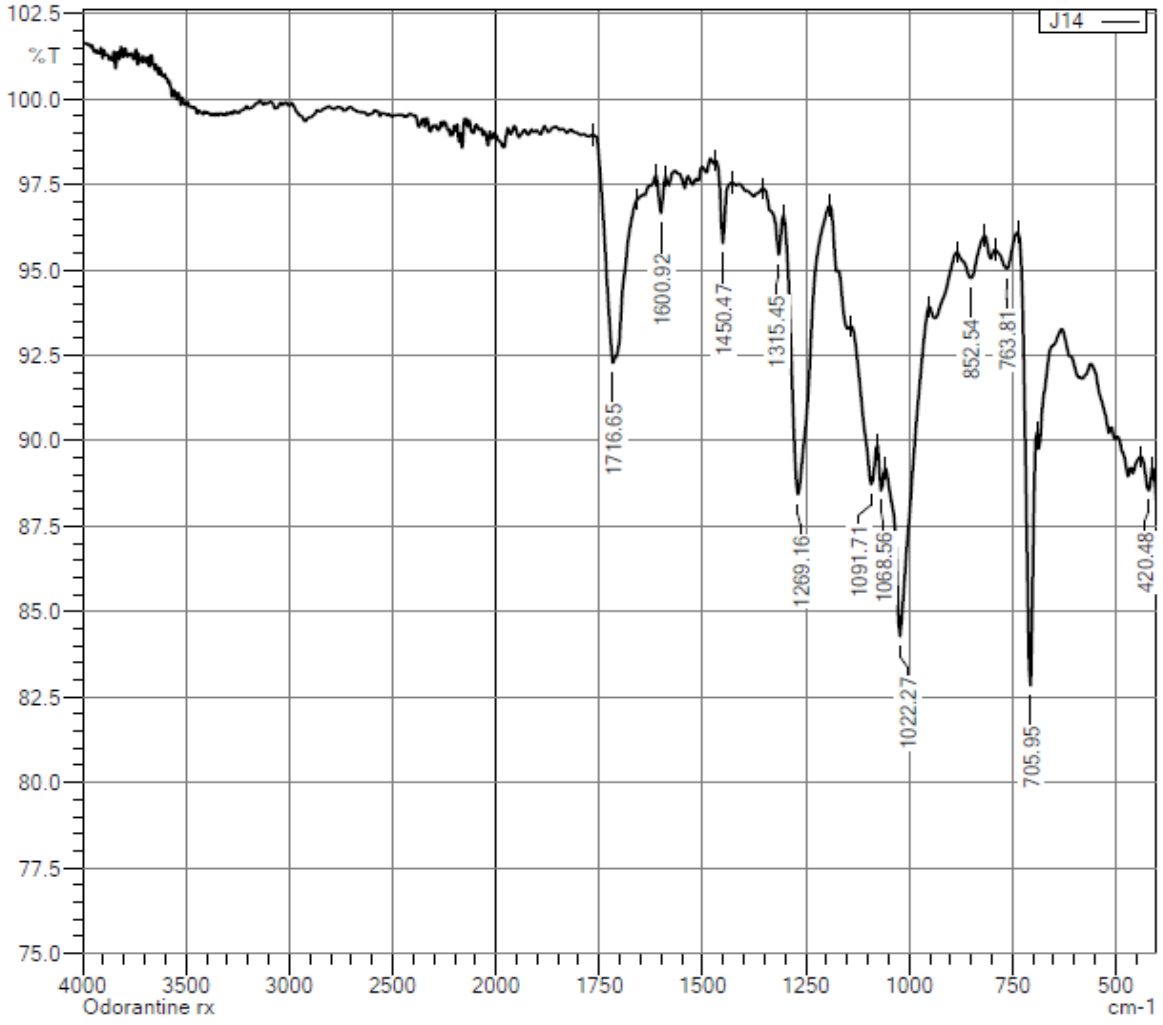

Figure 3. The Infrared Spectroscopy of the Acha Modified Starch

The discussion on the Acha Modified Starch as shown in Figure 3

However, for the modified starch showed at (Figure 3) the major new absorption band are at 1600.92 cm^{-1} which is associated with C=C streching aromatic indicating the modification of the naive starch. The FTIR spectra of benzoylated starches, it showed a stronger peak band at 1600.92 cm^{-1} than the native and the starch. The band in the go 1716.65 cm^{-1} corresponds to the C=O streching vibration of the ester group confirming the water adsorption and successful modification. The band at 705.95 cm^{-1} is characteristic peak of C-H out-of-plane bending mono-substituted aromatic ring. The absorption bands 450.47 cm^{-1} further supported presence of aromatic ring. The absorption bands at 1269.16 cm^{-1} can be attributed to C -0 - C vibration of the modified starch molecule. The weak absorption peaks 1091.71 cm^{-1} and 1068.56 cm^{-1} are probably due to the stretching of the C-OH bond in the modified starch. The absence of an absorption band at 1800 cm^{-1} base in the spectra of the modified samples indicated that the products was isolated free of an unreacted acyl chloride (benzoyl chloride). The strong broad absorption band at 3375.70 cm^{-1} due to O-H stretching in the starch sample decreased significantly following the esterification reaction, as the relatively moderate DS-value of benzoylation meant that only few quantity of unreacted hydroxyl groups as still present which has characteristic absorption band at 3450.90 cm^{-1} in the modified sample. Theses FTIR results indicate the structural changes where the benzoylation makes the starch more hydrophobic, which can enhance its stablity in oil- water interfaces for emulsion applications.

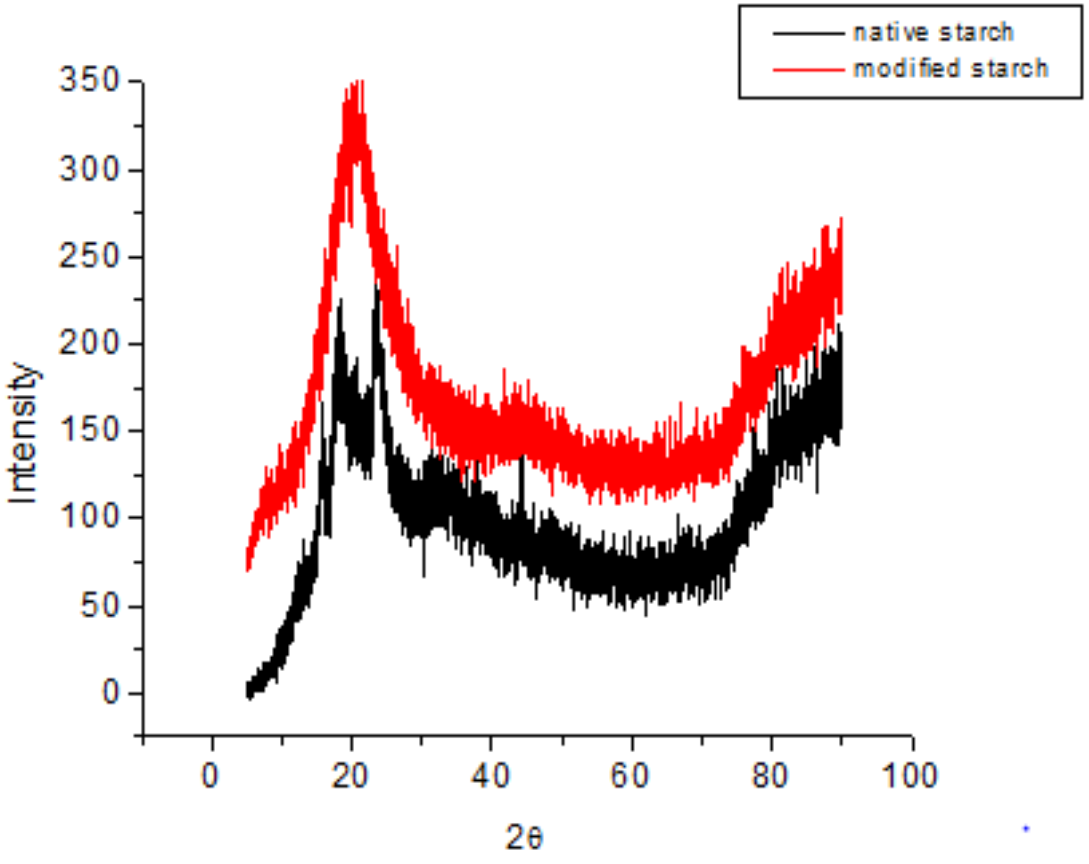

Figure 4. XRD profile of native and modified acha starch

The degree of crystallinity was calculated using Scherrer formula which states that:

$$D = \frac{k\Lambda}{\beta cos\theta}$$

where: k = the Scherrer constant which is 0.68 to 2.0 to 0.94
Λ = X-ray wavelength which is 0.154 nm
β = the line broadening at FWHM in radians
θ = the Bragg's angle in degrees.

This is used to determine the particle size which is calculated thus;
For native starch:

$$2\theta = 18.08$$

For modified starch;

$$2\theta = 19.02^{\circ}$$

From the above as shown in (Figure 4), there is reduction in the particle size of the modified acha compared to the particle size of native acha which results in a larger surface area, making it a stable emulsifying agent.

The percentage of crystallinity is calculated using the formula

$$\% \text{ of crystalinity} = \frac{I_{cr} \times I_{am}}{I_{cr}} \times 100$$

where: I_{cr} = intensity of crystallinity
I_{am} = intensity of amorphous

X-Ray diffraction measurements in Figure 4 were performed to check if chemical modification altered the crystallinity of starch as shown in Figure 4. The X-Ray diffraction spectra of native and modified starch showed the sample peaks (2θ) peak at 18.08° and 19.02° respectively. After acetylation, slightly reduction in crystallinity was observed. The loss of crystallinity would mean enhanced ability for acetylation starch or polymer products made from it to absorb water. The native acha starch shows a A-type pattern with peaks typically at 18.08° which indicate a well organized crystalline structure resulting from the amylopectin chains in its granules The crystallinity in the native starch gives rigidity to its granules and affects the solubility, swelling and thermal stability properties. However, in the benzoylated starch there is a shift in the position to 19° 2θ which indicates alteration in the starch's crystalline structure due to benzoylation, the benzoylation introduces the bulkiness of benzoyl groups, which interfere with the molecular packing of amylopectin and amylose unit. The benzoylated starch shift at 19° 2θ decreases in peak intensity, indicating a reduction in crystallinity. This shift is indicative of structural changes , making the modified starch more suitable for applications requiring high interfacial activity, such as emulsion in pickering emulsion.

Pickering Emulsion

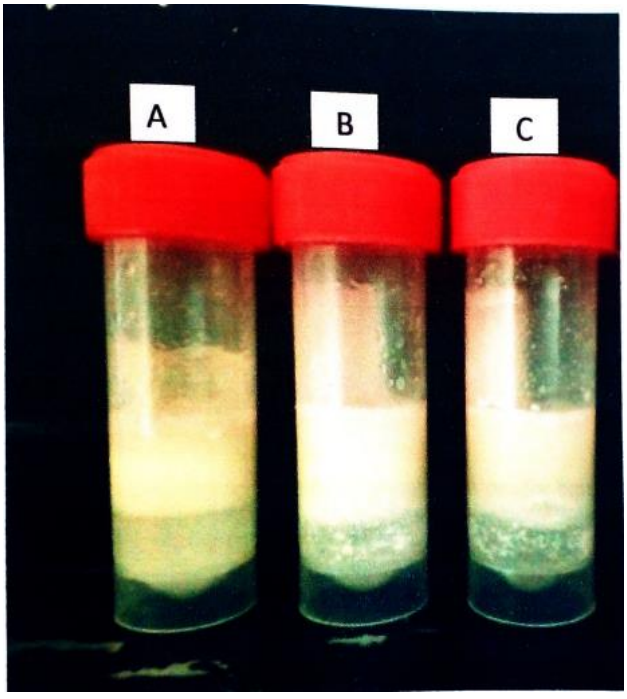

Figure 5. Preparation of O/W emulsion of A, B and C.
A- ACHA NATIVE STARCH (0.01g)
B- BENZYLATED STARCH (0.01g)
C- BENZYLATED STARCH (0.01g)

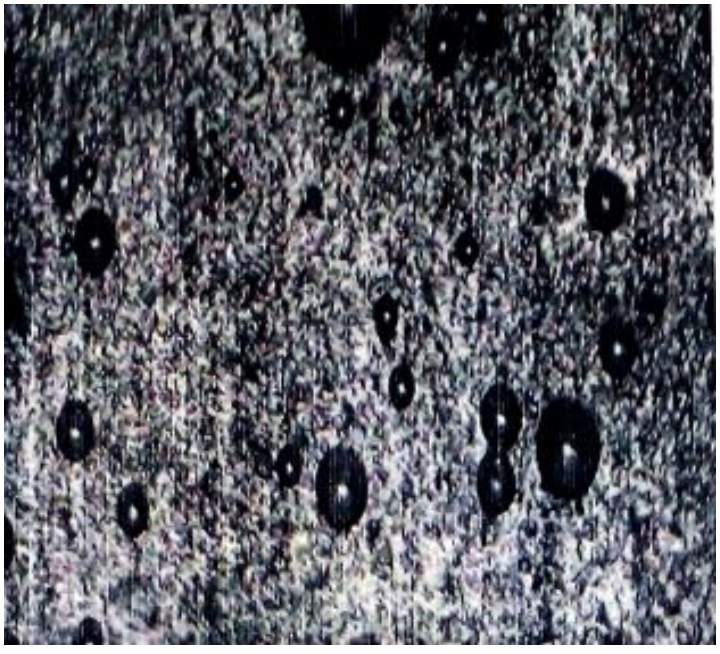

Figure 6. Optical microscope image of emulsion of native starch

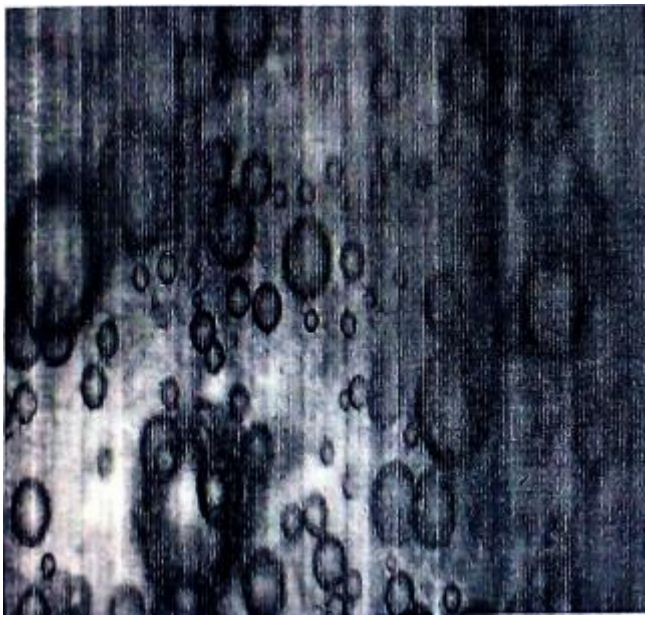

Figure 7. Optical microscope images of modified starch.

The emulsion stability of the native and bezoylated starch obtained from *Digitaria exilis* (Kippist) Stapf), was investigated. The picture of emulsions formulated for native and benzoylated starch were shown in Figure 5. The optical micrographs of emulsions prepared were shown in Figure 6 and Figure 7. The emulsions formed droplets which are spherical in geometry. The droplets are poly-dispers and the microstructure of both the native starch and benzoylated starch. The native starch exhibit the hydrophilic nature and struggles to stabiliize oil-in-water (O/W) emulsions effectively due to its limited ability to adsorb at the oil water interface. However, the benzoylated starch increased in water absorption capacities (WAC) than the native starch, indicating enhanced hydrophilic tendency and a slight expansion of the amorphous region. This may also be linked to the introduction of bulky functional groups and their electrostatic repulsion facilitating percolation and absorption of water within the starch matrices. Similar water absorption capacities following esterification has also been reported (Emeje *et al.*, 2012). Increase in the level of incorporation of these starches into food formulation like dough would beneficially improve handling characteristics and also maintain freshness of product. The modification enhances the stability of O/W emulsions by creating a steric and electrostatic barrier that is resistant to environmental changes like pH and ionic strength. The modified starch also creates smaller and more uniformly distributed droplets, resulting in a more stable emulsion with potential applications in food, pharmaceuticals and cosmetics.

4. CONCLUSIONS

Starch has been successfully isolated from acha grain (*Digitaria exilis* (Kippist) Stapf), Acha grain was utilized as source of starch for producing benzoylated starch and starch

derivatives, various reaction parameters were investigated such as proximate analysis, isolation and modification (benzoylation) of starch from Acha grains. Therefore, modification was carried out to improve the functional properties of the native starch and characterizations were carried out. The emulsion properties of the native starch showed considerable limited stability under the prevailing reaction condition. For technical applications, such as the preparation of biopolymer based flocculants, drag-reduction biomaterials, drug-release and other applications where bio-based polymers are relevant, benzoylated starch could be strategic because the source material is reasonably cheaper than other conventional sources of starch and it has a reasonable wide distribution globally. Having known using starch in its native form is often limited by certain undesirable characteristics such as poor solubility, low mechanical properties and instability at high temperature, pH and shear during processing. Hence, it is recommended that more chemical modifications should be carried out to suit specific industrial purposes. Also, it is recommended that starch from other plant sources should be isolated and they should be considered for relevant applications such as encapsulation of vitamins, essentials oil, flavors, drugs and microorganisms, so as to influence the controlled release of food ingredients in food and nutritional application and improve the properties of starches.

List of Abbreviation

FTIR - Fourier Transformed Infrared spectroscopy
SEM - Scanning emission microscopy
XRD - X-Ray diffraction
NAOH - Soduim Hydroxide
O/W - Oil in water
WAC - Water Absorption capacity

Authors' contributions

MA and OL participated in all paper sections. EB prepared the abstract and OO and CO conducted the modification and characterization, PI prepared the introduction, SP and DM carried out the isolation of starch. All the authors have read and approved the final manuscripts.

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