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EXTRACTION, SYNTHESIS, CHARACTERIZATION AND ANTIOXIDANT PROPERTIES OF OXIDIZED STARCH FROM AN ABUNDANT SOURCE IN NIGERIA

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ABSTRACT

Starch is a renewable and environmentally compatible polymer but due to some inherent drawbacks like its hydrophilic character, poor mechanical properties, and its inability to withstand processing conditions such as extreme temperatures, diverse pH, starch in its native form cannot be applied satisfactorily in industrial operations. Modification of starch however, enhances its applicability in both food and pharmaceutical industries. In this study, *Manihot esculentus* (*M. esculentus*) starch was modified chemically by oxidation. Foam capacity and emulsion capacity of the native and oxidized *M. esculentus* starch was determined. Fourier Transmittance Infra- Red (FTIR) and Raman spectroscopies was used to confirm starch modification while characterization was done using Scanning Electron Microscopy (SEM) and X- Ray Diffraction (XRD). Antioxidant activity of the oxidized *M. esculentus* starch was assessed using DPPH (2, 2-diphenyl-1-picryl-hydrazyl-hydrate) free radical assay. Our results show that modification had no significant effect on the foam and emulsion capacities. FTIR results revealed a new band at 3007 and 3283 cm^{-1} while SEM showed that oxidation did not alter the predominantly circular-shaped starch granules. The XRD patterns of both native and modified were found to be similar. Differential Scanning Calorimetry returned two new endothermic peaks in the oxidized *M. esculentus* starch with an improved gelation capacity and increased enthalpy of gelatinization. The IC_{50} of the oxidized starch was notably higher than that of the reference standard, ascorbic acid.

Keywords: *M. esculentus*, starch, oxidation, antioxidant activity, DPPH.
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INTRODUCTION

Manihot esculentus (cassava) is a perennial woody shrub, grown as an annual crop and is eaten in abundance by the populace in tropical Africa in particular [1]. Sequentially, Brazil, Thailand, Nigeria, Zaire and Indonesia were the highest in the production of cassava but today, Nigeria is the highest producer of cassava

Cultivation of *M. esculentus* is a simple root crop that produces in less than 12 months of warm weather. Freezing conditions are unfavorable to its growth; its productivity is highest in full sun and in soil with pH between 4.0 and 8.0 [1]. The dried root of *M. esculentus* contains 80 % of starch. No scientifically acceptable processing technology exists for processing cassava in Nigeria although it is the country with the highest production in the world. The pharmaceutical industries still import all of their starch, implying lack of value addition to starch-based sources like cassava in Nigeria [2]. In addition, industries rely heavily on starch gotten from cereal such as maize which is relatively expensive; consequently the cost of manufacturing products increases [3]. It is therefore pertinent to develop starch from locally abundant sources, like cassava, which is useful in its native and/or modified form.

Starch is predominantly becoming known as a renewable and environmentally compatible polymer due to increase in its use. It is a naturally occurring, non-soluble, biodegradable and environmentally compatible polymer extracted from tubers and cereals such as yam, potato, cassava, maize, rice and millet, etc. [4] as well as nuts, seeds and leaves. The botanical origin of starch is a major determining factor in selection of its method of development. Isolation of starch from root crops like potato and cassava is relatively simple because of the structure of their tissue while isolation from cereals is more tasking owing to the high Starch is used extensively in the textile, paper, wood, food, chemical and pharmaceutical industries. Starch molecule contains 300 - 1000 glucose monomers in repeated units and each starch molecule consists of two main polymers; amylose and amylopectin. Amylose forms the straight chain linked together by α 1-4 glycosidic bond while the amylopectin constitute the branched point linked together by α-(1 → 4) dglucopyranose units with α-(1 → 6)-linkages at the branching points [5, 6]. Starch is eaten in abundance in most part of the world and in recent years, is being processed into biodegradable plastics which are more eco-friendly than those derived from [7, 8].

The industrial selection of any starch is made by considering its accessibility and its physico-chemical characteristics which vary depending on its source [9]. Native starches like *M. esculentus* starch, have good gelling and thickening property but possess poor mechanical properties and is unable to withstand processing conditions like extreme temperature, diverse pH, high shear rate, freeze thaw variation and dimensional stability [2] .These inherent characteristics of native starch are congruent on the molecular size, crystallinity degree, amylose content and its rheological properties [7, 9] .Therefore, in order to circumvent the inherent drawbacks, starch can be modified to impart new properties that meet the requirements for particular applications as well as improve their inherent characteristics. These modifications could be physical, chemical, enzymatic or genetic [10] with cross-linking and substitution being the most common types of modification. Cross-linking improves the shear, acid and heat stability of starch while substitution reduces starch retrogadation [11]. However, modifications *via* cationisation and copolymerization are undertaken for applicability in non-food industries. Generally, modification of starch *via* any of the aforementioned methods is useful as fillers, emulsion stabilizers, adhesives, binders and disintegrants [2].

Chemical modification like oxidation reduces the amylose content of native starch thereby improving paste clarity, freeze thaw stability, film-forming and binding ability while reducing the viscosity of starch; this improves their applicability in the paper, textile and pharmaceutical industries [12, 13]. Oxidation of starch can be prepared by using hydrogen peroxide, ozone, ambient oxygen, bromine, chromic acid, nitrogen dioxide, ultraviolet radiation and hypochlorite [13]. Oxidation *via* the use of hydrogen peroxide is not very common although, its decomposition, which is basically oxygen and water, is less harmful than when other reagents like sodium hypochlorite, are used. This process usually occurs in moderately alkaline environment to foster the formation of carboxyl group which during the reaction stabilizes the linear amylose fraction and consequently reduces retrogradation [12]. Oxidation substitutes the hydroxyl groups on starch with carboxyl and carbonyl groups and the amount of substituted groups shows the level of oxidation with positions C_2, C_3 and C_6 of the hydroxyl group showing where oxidation occurs [14].

In this study, *Manihot esculentus* (cassava) starch was chemically modified *via* oxidation

and the effect of this modification on the structural property, and functionality indices of *Manihot esculentus* starch was evaluated.

MATERIALS AND METHOD

Extraction

Manihot esculentus tubers devoid of defects were purchased from a local market in FCT, Abuja, Nigeria. The skin of the tubers were peeled off to expose the inner layer, then the tubers were cut into small pieces and washed using a solution of 0.1 %w/v sodium metabisulphite. The cut tubers were wet milled, filtered using a muslin cloth and the filtrate was then allowed to stand for 12 h. Afterwards, the supernatant was decanted and the starch sediment was collected, washed severally in sodium metabisulphite solution until the starch was white in color. The starch was air-dried for 72 h, triturated, sieved using a sieve mesh size of 250 µm and then stored in a desiccator until further use [15].

Synthesis

A catalyst (FeSO4 solution; 0.01 %w/v) was added to hydrogen peroxide solution and the pH of the mixture was adjusted to 3.0. *Manihot esculentus* starch (100 g) was added to the hydrogen peroxide-catalyst mixture, stirred mechanically at 20 ºC while hydrogen peroxide was continuously added until a concentration of 1.25 %v/v was achieved. The total reaction time was 15 min after which the synthesized starch (OCS) was collected, washed with distilled water and dried in the oven at 45 ºC for 24 h [12].

Evaluation of the physicochemical properties of oxidized *Manihot esculentus* starch (OCS)

Foam capacity:

Manihot esculentus starch (2 g) was homogenized in 100 mL of distilled water using a vortex mixer (Vortex-2 Genie set at shake 8) for 5 min. The homogenate was poured into a measuring cylinder (250 mL) and the volume occupied after 30 sec was recorded. The foam capacity was expressed as a percentage the volume increase [16].

Emulsion capacity:

Manihot esculentus starch (2 g) was dispersed in 25 mL of distilled water using a vortex mixer for 30 sec, after complete dispersion, vegetable oil (25 mL) was added gradually and mixed continuously for another 30 sec. The suspension was then centrifuged at 1600 rpm for 5 min and the volume of oil above the sample was noted. The emulsion capacity was expressed as the percentage of the quantity of oil per gram of the sample [16].

Scanning Electron Microscopy (SEM):

M. esculentus starch micrographs were obtained from the Hitachi S5200 field emission scanning electron microscope (Hitachi High- Technologies Canada, Inc., Ontario, Canada). The imaging was done at 1.0 kV accelerating voltage in order to obtain platinum-coated samples.

X-Ray Diffraction:

Structural characterization of *M. esculentus* starch molecules was done using the Siemens D5000 X-ray diffractometer (Siemens, Munich, Germany). Starch powder samples, packed in rectangular aluminium cells, were illuminated using CuKa radiation (l = 1.54056 Å) at 45 kV and 40 mA. The samples were scanned between diffraction angles of 5 and 80 ^{o} using scan step 0.1 and dwell time of 15 s; a nickel filter was used to reduce the size (kb) contribution to the X-ray signal. Three (3) measurements were taken at ambient temperature.

Differential Scanning Calorimetry (DSC):

Using the DSC (Model DSC 204 F1Netzsch, Germany), thermograms of starch samples were obtained. The temperature axis and cell constant of the DSC cell were calibrated using indium (10 mg, 99.999 % pure, melting point 156.60 ^{o}C, heat of fusion 28.40 J/g). Starch samples were weighed into aluminum pans and approximately 9 mL of distilled water was added to each pan; the samples were allowed to equilibrate for 2 h and then scanned between 26 ^{o}C and 180 ^{o}C at a heating rate of 10^{o}C/min under continuous nitrogen flow. Gelatinization parameters were characterized by onset temperature (*To*; ^{o}C), peak temperature (*Tp*; ^{o}C), conclusion temperature (*T*c; ^{o}C), and gelatinization enthalpy (D*H*gel; J/g).

Raman Spectroscopy:

The Bruker FRA-106/S FT-Raman spectrometer was used to obtain the Raman spectra of the starch samples. The exciting source was Nd+YAG laser functioning at 1064 nm with power of about 400 mW; the scattered light was collected at an angle of 180 ° (back-scattering) at a typical spectral resolution of 1 cm^{-1}.

Fourier transform infrared spectroscopy (FT-IR):

The synthesized and native starches were triturated with potassium bromide and the pellets placed in the Nicolet FTIR spectrometer. Infra-Red (IR) spectra of the samples were obtained between frequency ranges of 4000 and 500 cm^{-1}.

Antioxidant Activity

DPPH radical scavenging activity:

The oxidized *M. esculentus* starch samples were mixed with 1 mL of methanol solution containing DPPH radicals to obtain concentrations of 0.1mg/mL and 0.2mg/mL; DPPH (0.2 mM) was used as the resultant final concentration. The mixture was vortexed, allowed to stand for 30 min and the absorbance was read at 517 nm. Ascorbic acid was used as the reference [17]. The percentage of inhibition in DPPH radical scavenging activity was calculated as;

$$Absorbance\ of\ control - Absorbance\ of\ sample / Absorbance\ of\ control \times 100 \quad \ldots\ldots\ldots (1)$$

RESULTS AND DISCUSSION

Foam capacity and emulsion capacity:

Emulsion capacity informs the formulation scientist on the emulsification characteristics of a material. High emulsion capacity indicates that the material may be a good emulsifying agent [16]. Foam capacity of the native and oxidized starch were low and similar, this could be attributed to low protein contents in the starch samples. Modification of *Manihot esculentus* starch had no significant effect on the foam capacity as well as the emulsion capacity as presented in Table 1.

Table 1: Foam capacity and Emulsion capacity

Parameters	OCS	NCS
Foam capacity (%)	1	1.3
Emulsion capacity (%)	60	62.74

Scanning Electron Microscopy (SEM):

The granule shape, size and morphology of a pharmaceutical material is largely determined by the biological and botanical source [22]. These internal characteristics do not only help distinguish between varieties of materials but also gives an insight into the processing technique to be used. Figures 1A and 1B shows the micrographs of the native *M. esculentus* (NCS) and modified starch (OCS) respectively; the starch granules were predominantly circular in shape and approximately 30 µm in size. It is common knowledge that different varieties of starches have varying morphologies; they could be polygonal, hexagonal, circular, oval or irregular in shape [18, 19, 20, 21]. However, our result is in tandem with that of a previous report on the morphology of native cassava starch [11].

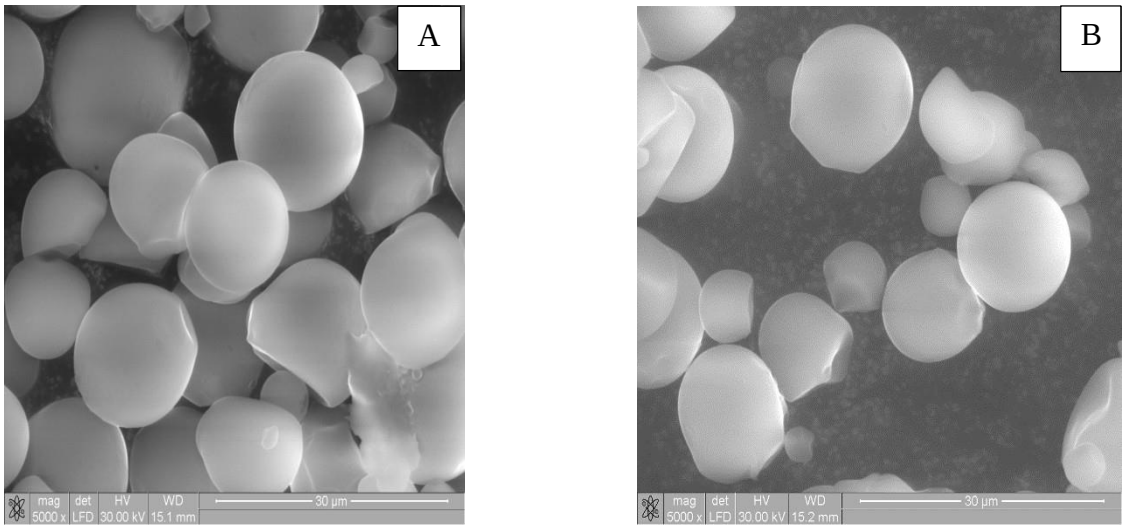

Figure 1: SEM of native *M. esculentus* starch (A) and oxidized *M. esculentus* starch (B)

Our also results show no observable difference in the micrographs of NCS and OCS thus, implying that oxidation had no significant damaged on the starch morphology, appearance and arrangement. In a similar report [14], corn, potato and rice starch were modified with hypochlorite. In yet another study [22], significant effect on the structure of *M. esculentus* starch granule after modification with acidified ethanol was reported. The result of this study however, shows a green approach to polymer/biomaterial development involving minimal quantity of chemicals while securing the critical components of the biomaterial (starch) [22].

X-ray diffractometry (XRD):

The granules of starch exhibit diffraction pattern classified as A, B, C dependent upon the double helical arrangement of the branched chain amylopectin. The A pattern has densely aligned molecules of water between each double helix while the B pattern has a more hexagonal packing and the central cavity has molecules of water [23]. The C and A patterns are similar except that a peak is observed between 5°2Θ and in addition, the C pattern is a crystalline polymorph which is a hybrid of the A and B polymorph [24]. The biologic origin of starch, largely determines its x-ray diffraction pattern, however, other factors like the length of amylopectin and amylose chain and moisture content are also known to affect its diffraction pattern [2].

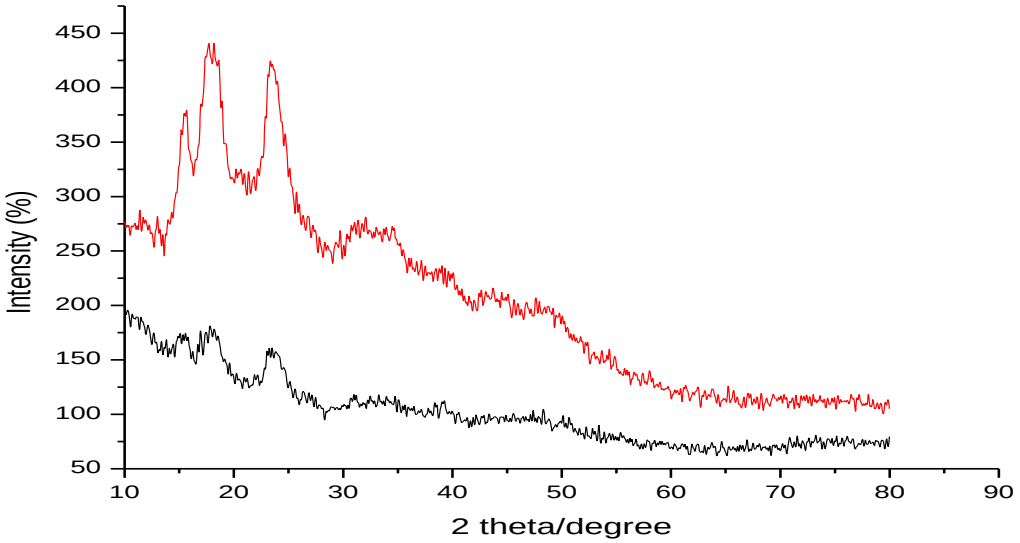

Figure 2: XRD of native *M. esculentus* starch (black) and oxidized *M. esculentus* starch (red)

The X-ray patterns of NCS and OCS as shown in Figure 2 are observed to be similar although the diffractogram of OCS showed stronger and higher peaks indicating that oxidation increased the crystalline portions and conferred thermal and structural stability on the modified starch.

Differential Scanning Calorimetry (DSC):

Starch granules undergo physicochemical transformation on application of heat and in the presence of organic or inorganic solvents; this modification is referred to as gelatinization [25]. Although thermodynamically, starch gelatinization refers to the enthalphic transitions involving the granular starch treated as a semi-crystalline entity (spherulite), the simplest explanation of gelatinization is that, it describes an array of thermally associated events which converts an aqueous dispersion of starch into paste. The temperature at which this gelatinization occurs is largely dependent on the crystallinity of the starch molecules [11]. Previous studies [2, 20] have reported exhaustively the process of gelatinization which is contingent on the starch spherulites theory. The differential scanning calorimeter (DSC) is mostly used to evaluate the phase transitions of the gelatinization process due to its accurate precision and sensitivity.

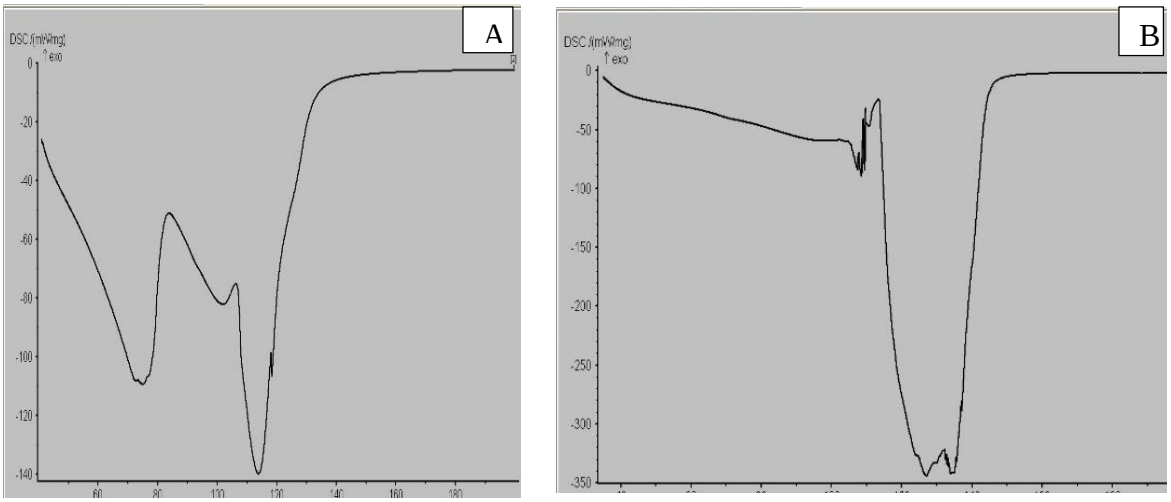

Figure 3: DSC thermogram of native *M. esculentus* starch (A) and oxidized *M. esculentus* starch (B)

The thermograms for native and oxidized *M. esculentus* are shown in Figures 3A and 3B respectively; the native *M. esculentus* starch is observed to be highly amorphous since no sharp endothermic peak is evident while the oxidized starch is observed to have crystalline portions as evidenced by the two continuous sharp endothermic peaks. These results suggest that oxidation increased the crystallinity of *M. esculentus* starch and this is in correlation with a previous report where the transition temperature of potato starch was observed to reduce when bromine was used for the oxidation process, this reduction was relative to the intensity of oxidation [12].

The corresponding parameters for the thermograms of NCS and OCS are presented in Table 2 and the endothermic transition observed in both the native and modified starch indicates swelling of granules and melting of crystallites occurring at different gelatinization [2, 20]. The onset, peak, and conclusion temperatures of gelatinization of OCS were observed to be higher than that of NCS. There are inconclusive reports of oxidative effect on gelatinization temperature which is said to be dependent on the plant source and method of modification.

Table 2: Thermal properties of NCS and OCS starches

Parameters	NCS	OCS
Onset Temperature (T_0)(˚C)	61.0	131.5
Peak Temperature(Tp)(˚C)	75.1	127.2
Conclusion Temperature(Tc)(˚C)	72.7	145.7
Enthalpy of gelatinization (J/g*K)	131.17	2618.05
$\Delta T(Tc-T_0)$ ˚C	11.7	14.2
Peak Height Index(PHI) $J^{g-1}K^{-1}$	9.3	-608.85

In a similar report [24] oxidized bean starch was also found to have lower onset and peak temperature however in another study, where hypochlorite and hydrogen peroxide where used in modification of cassava starch, it was posited that the carboxyl group in starch inhibits its ability to absorb water thereby weakening the granules [26].

The enthalpy of gelatinization is a measure of the quantity and quality of crystallinity and also indicates the loss of order in the molecular network of granules due to hydrogen bond breakage [24]. The enthalpy of gelatinization of OCS was observed to be significantly higher than that of the native NCS. This finding differs from that of an earlier study where the enthalpy of gelatinization of oxidized starch was observed to be lower than that of the native starch [27]. Increase in the gelatinization temperature is known to increase the amylose content leached out of starch molecule [3], thus, OCS may have more amylose leached out than NCS thereby increasing its viscosity. Therefore, oxidation is seen here to reduce the rate of starch gelatinization while conferring thermal and structural stability on the starch.

Raman Spectrum of NCS:

Figure 4 represents the Raman spectra of NCS; the band at 478 cm^{-1} can be attributed to strong aliphatic chain C-C, while the band at 1129 - 1386 cm^{-1} can be due to strong aromatic ring chain vibrations; the band at

about 2909 cm^{-1} is attributed to strong C-H bond and that at 944 cm^{-1} is attributed to C-O-C bond.

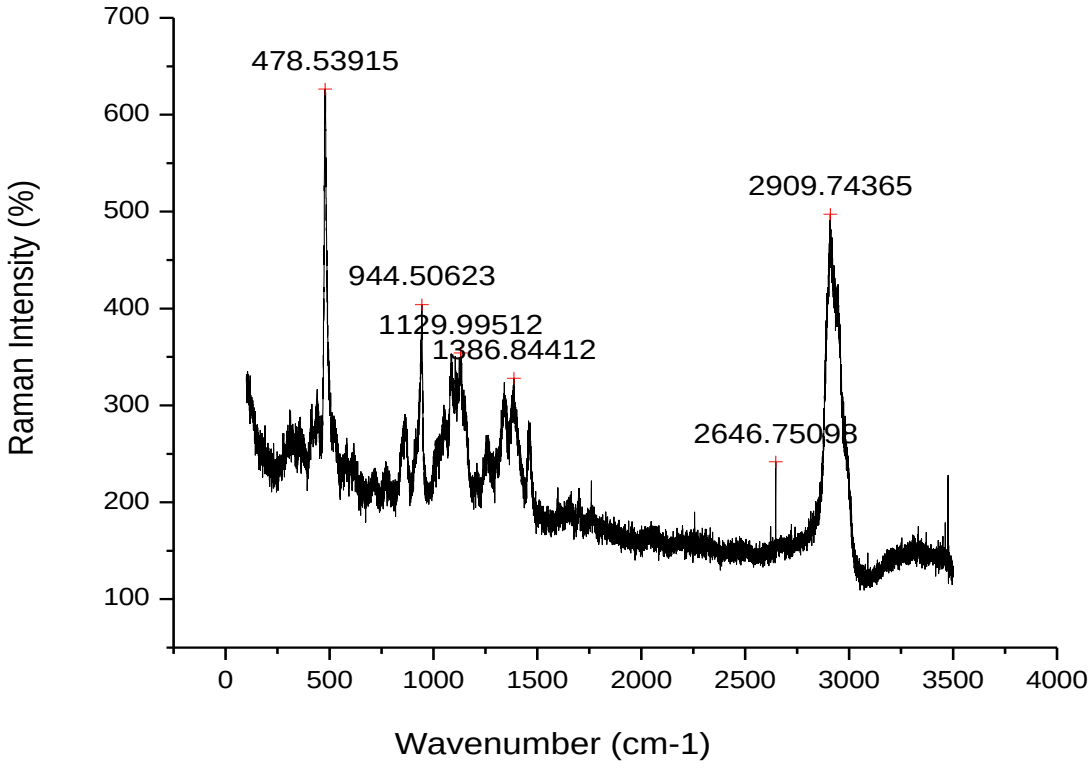

Figure 4: Raman spectrum of Native *M. esculentus* starch

FTIR of Native and Oxidized M. esculentus starch:

The infrared spectra of NCS and OCS are shown in Figures 5A and 5B respectively. The band at 651- 994 cm^{-1} is attributed to moderate absorption of carboxylic acid, that at 1050 - 1260 cm^{-1} is attributed to C-O stretch while the band observed between 2800 - 3300 cm^{-1} is due to C-H stretching vibration. The band between 3000- 3100 cm^{-1} is due to C-H bond, the sharp band displayed about 1648 cm^{-1} could be attributed to the stretching vibration of the carbonyl group. A new broad band around 3007 cm^{-1} indicates OH stretch due to carboxylic acid thus, showing modification of the starch molecules *via* oxidation. The result of the FTIR confirms our position that, the desired modification was achieved without destroying the structural integrity of the

starch molecules.

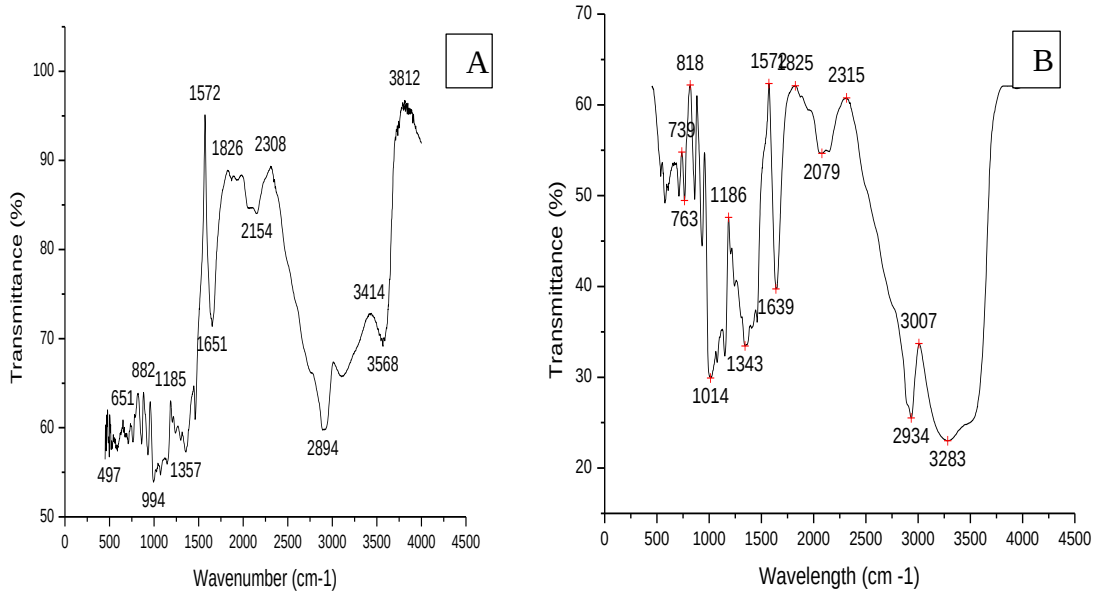

Figure 5: FTIR Spectrum of native *M. esculentus* starch (A) and oxidized *M. esculentus* starch (B)

Antioxidant; DPPH scavenging activity:

The antioxidant activities of OCS were assessed by using 2,2-diphenylpicryl-1-picrylhydrazyl (DPPH) and the results are presented in Table 3.

Table 3: Percentage inhibition of DPPH scavenging activity

	Oxidized starch	Ascorbic acid
0.1 mg/mL	57.6 %	78.3 %
0.2 mg/mL	60.1 %	75.7 %
IC_{50}	1.8 mg/mL	1.58 mg/mL

The percentage inhibition of OCS at 0.1 mg/mL was observed to be lower (57.6 %) than that of the standard ascorbic acid at same concentration (78.3 %) and also lower (60.1

%) at a higher concentration (0.2 mg/mL) when compared to that of the standard (75.7 %) at the same concentration. The DPPH scavenging activity of OCS however was observed to have similar dose dependent inhibition of DPPH activity with 50 % of inhibition (IC50) of 1.8 mg to that of ascorbic acid (1.58 mg). These results show the potential of OCS as an antioxidant and a moderate free radical scavenger.

CONCLUSION

Starch is an abundant, eco-friendly and renewable polysaccharide. Indigenous starch, irrespective of its biological origin, possesses inherent properties that limit their applicability. Modification of native starch however, confers new properties and improves its applicability. This study shows that modification of *Manihot esculentus* starch *via* oxidation improved its thermal and structural stability and also impacted an antioxidant property thus making it more desirable for use in both food and pharmaceutical industries.

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