Synthesis and Characterization of Selected Starch Nanoparticles as Matrix Reinforcements for Low Density Polyethylene

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Abstract
The non-degradability of conventional petroleum-based plastics and their subsequent hazardous accumulation in the environment has resulted in the vital need to produce biodegradable polymer replacements. The aim of this research was to convert locally sourced feedstock starches into nanoparticles for the purpose of modifying the matrix of Low-density polyethylene (LDPE), so as to enhance its biodegradability. The starches utilized are as follows; *Zea mays* (corn), *Ipomoea batatas* (potato), and *Manihot esculenta* (cassava). Conversion to nanoparticles was achieved by a process called acid hydrolysis. The synthesized nanoparticles were characterized by microstructural tests such as transmission electron microscopy (TEM), energy dispersive x-ray spectroscopy (EDX), fourier transform infrared (FTIR), x-ray diffraction (XRD), and scanning electron microscopy (SEM). The characterization results indicated that the acid hydrolysis process succeeded in the conversion of the starches to nanoparticles without affecting the chemical constituents and functional groups of the starches used.

Key words: Polymers, LDPE, Starch, Nanoparticles, Biodegradability

1 Introduction
Low Density Polyethylene is the most utilized petroleum-based synthetic polymer, due to its magnificent mechanical attributes, water holding capacity, lightness, cheapness and high energy effectiveness. LDPE owes its outstanding mechanical properties to its long and strong polymer chains or matrix. However, these features also result in the non-biodegradability of LDPE, which when combined with its omni present nature and improper disposal techniques becomes an exigent and overlying pollution issue [1, 2]. Starch on the other hand is under a class of natural biopolymer called polysaccharides extracted from the glucose found in plants, with much shorter and weaker polymer chains or matrix [3]. Its similarity to LDPE and biodegradability makes it a feasible matrix reinforcement material for LDPE when bolstering biodegradation is the aim, and it is projected that the use of starch nanoparticles would enhance the infusion of the starch molecules into the LDPE matrix [4, 5]. Nanoparticulate materials refer to materials whose particle size dimensions within 1 nm to 100 nm and can be categorized either as organic, inorganic and carbon based particles and are mostly biodegradable and not toxic [6]. Inorganic nanoparticulate materials are those which do not consist of carbon and can further be subdivided into metal-based nanoparticulate materials and metal oxide-based nanoparticulate materials. Carbon based nanoparticulate materials are materials which comprise completely of carbon.
Additional research has also shown that a reduction in the size of the starch particles leads to a consequent improvement of the biodegradability whilst maintaining competitive values of mechanical properties.

Synthesis is the first step in the production of nanomaterials, nanoparticle synthesis can be synthesized using various methods which include: coprecipitation, inert gas condensation, thermal decomposition, ion spluttering scattering, microemulsion, hydrothermal synthesis, pyrolysis, spinning, nanolithography, biosynthesis etc.

[7] processed ZnO nanoparticles using the coprecipitation method, hydrothermal autoclave at a low processing temperature and characterized them using X-ray diffraction and Transmission electron microscopy for application in optoelectronic devices for solar cells. The method proved viable and was seen to be suitable for mass production [7]. Thermal decomposition method was used to prepare carbon-coated wustite nanoparticles by [8] varying the reaction time between 1 hour and 3 hours to study the effect of the synthesis time on the nanoparticle stoichiometry. It was seen that the crystallinity degree increases with the synthesis time which was further supported by the XRD data that the magnetite phase disappeared with increased synthesis time [8].

Solid lipid nanoparticles of soy lecithin, cholesterol and stearic acid were produced by [9] using a supercritical fluid assisted process and the effect of the lipid concentration on particle size and morphology. The process was successful and the particles all fell within the range of nanomaterial i.e 1 to 100 nm [9].

Starch nanoparticles to be used as mucoadhesive carriers for trans-nasal insulin delivery were prepared by [10]. The method employed here was the gel crosslinking method and it was seen that the obtained materials were in the size range of 190 to 998 nm and were evenly distributed [10].

Starch nanoparticles have most commonly been synthesized using acid hydrolysis method and enzymatic treatment [11]. Nanoparticles prepared through acid hydrolysis were seen to be more stable at high temperatures and as such has made them very suitable for applications in food processing as well as drug delivery for medical applications.

The acid hydrolysis method for preparing nanoparticles/nanocrystals has been in existence since early stages of the year 1996 [12], it depends on several parameters which include starch botanic source, starch granule structure, acid in use and its corresponding concentration, temperature of the hydrolysis and length of time of the process [13]. The acid hydrolysis method of preparing nanocrystals has been to significantly reduce the synthesis time while simultaneously increasing the yield [14].

This research outlines the process of the synthesis and characterization of starch nanoparticles from three different starch sources.

2 MATERIALS AND METHODS
2.1 Starch Extraction process
The Manihot esculenta and Ipomoea batatas tubers were washed thoroughly and cleaned to remove dirt. The peeled tubers were sliced and afterwards ground with a Winkworth Z Blade Sigma industrial blender to produce a smooth mash. The resulting mash was then introduced into a starch extractor. The supernatant liquid was drained away and the paste was dewatered to decrease the moisture content. The 200 μm wire mesh sieve was used to separate starch milk from the solid fibrous residue. The starch milk was then heated in an oven to obtain the dry powdered starch which was further sifted to obtain micro sized particles using the 80 μm wire mesh sieve. At the end of the process, approximately 25 % starch by weight was obtained from the Manihot esculenta and Ipomoea batatas tubers. The procedure is shown
in Figure 1. The process followed for extracting starch from *Zea mays* is as shown in Figure 2 [15, 16, 17].

![Flow Chart for Ipomoea batatas and Manihot esculenta starch production.](image)

Figure 1: Flow Chart for *Ipomoea batatas* and *Manihot esculenta* starch production.
Soaking & preliminary grinding of *Zea mays* (Steeping)
- Soaking in water at about 50 °C for 4 to 5 hours.
- Grinding to free the germ.

Separation and washing
Germ is separated from the hull and bran and then washed.

Drying of germ
Washed germ is dried in oven to about 8 % moisture content

Fine grinding, separation and final dehydration
- Germ is finely ground
- Gluten separation
- Final dehydration of pasty starch
- Sieving of dry starch to remove unwanted particles

Figure 2: Flow Chart for *Zea mays* Starch Production

### 2.2 Synthesis of the starch nanoparticles

2 Litre/2 M solution of dilute sulphuric acid was prepared; the following calculations were made to determine the dilution factor:

\[
\text{Molar Mass of } H_2SO_4 = (2 \times 1) + (32) + (16 \times 4) = 98 \text{ g/mol}
\]

\[
\text{Hence for a 2M of the solution, we should have: } 2 \times 98 \text{ g/mol} = 196 \text{ g/mol in one(1) litre of distilled water.}
\]

\[
\text{Density of sulphuric acid} = 1.84 \text{ g/mol}
\]
Therefore 196 g/mol of H₂SO₄ is equivalent to \( \frac{196}{1.84} = 106.52 \text{ ml of acid.} \)

Considering purity of the acid = 98%, the volume to be used = \( \frac{106.52}{0.98} = 108.69 \text{ ml} \)

Hence, for a 2L solution, the volume of conc. H₂SO₄ to be used = \( 2 \times 108.69 = 217.387 \text{ ml} \)

Volume of distilled water to be used = 2000 ml – (217 ml of acid) = 1783 ml

Hence, 217 ml of conc. H₂SO₄ was measured using measuring cylinders and was transferred into a flat bottom flask using a funnel. 1783 ml of distilled water was also measured using measuring cylinders and transferred into an amber reagent bottle after which the concentrated acid was added slowly into the reagent bottle containing water after which it was allowed to cool due to the fact that it was an exothermic reaction.

73.45 g of dry Zea Mays, Manihot esculenta and Ipomoea batatas powders were each measured using Adam™ PGW digital weighing balance into a 250 ml flat bottom flask, 16ml of hydrogen peroxide (H₂O₂) was added to the starch powders and the mixture was stirred vigorously using a glass rod.

426.5 ml of the prepared dilute acid was then added to the above mixture after which the mixture was again stirred vigorously for even dispersion of all the constituents. A magnetic stirrer and a Stuart™ US152 Hotplate stirrer were set to 2000 rpm and a temperature of 150 °C respectively, the mixture was stirred for a period of 72 hours within which the heater was turned off every 12 hours.

The slurries obtained from the hydrolysis process were transferred into 10 ml centrifuge tubes while being topped with distilled water and were then subjected to centrifugation using a Surgifield SM-8-2 Centrifuge Machine at 4000 rpm, for 10 minutes for each set of 12 centrifuge tubes, to attain neutrality of the resulting agglomerates. The slurries were centrifuged until there was a visible settling of the solid layer at the bottom of the centrifuge tube and the liquid above was decanted.

The centrifuged samples were again dispersed in distilled water and subjected to ultrasonic treatment using the Branson M2800H Ultrasonicator for 90 minutes so as to attain even dispersion of the particles. The sonicated samples were finally oven dried at a temperature slightly below 100 °C. The dried samples obtained from the oven were then carefully declutter from the clusters using a crucible and a hand pestle into the fine powders. The mass of each produced starch nanoparticles were measured using Adam™ PGW digital weighing balance to determine the yield.

### 2.3 Characterization of the starch nanoparticles

The composition, size and morphology of the produced nanoparticles were investigated using Energy-dispersive X-ray Spectroscopy (EDX), Transmission Electron Microscopy, X-Ray Diffraction, Fourier Transform Infrared (FTIR) and UV-Vis Spectroscopy using ASTM E1508–12a, ASTM E986-04, ASTM E168/E1252 and ASTM E169–16 standards respectively. FTIR analyses were carried out with a Nicolet iS10 spectrometer with a collected spectrum from 350 cm⁻¹ to 4400 cm⁻¹. X-ray diffraction analyses were operated at 40 kV and 100 mA with Cu \( Kα \) radiation detector. The starch particles were scanned through the \( 2θ \) range of 0 to 30 °.

ImageJ was used to determine the average nanoparticle size from the TEM micrographs as shown in Table 1.
3 Results and Discussion
The characterization results of the produced starch nanoparticles are shown in Figures 3 to 14.

3.1 EDX Characterization
Figure 3 shows that the *Zea Mays* nanoparticles starch have composition of Carbon 65.24\%, Sulphur 3.91\% and Oxygen 30.85\%. The percentage composition of *Ipomoea batatas* is 61.55\% Carbon, 38.35\% Oxygen and 0\% Sulphur while the composition of *Manihot esculenta* is 65.27\% Carbon, 27.95\% Oxygen and 6.78\% Sulphur as shown in Figures 4 and 5 respectively.

![Figure 3: EDX Spectrum of *Zea may*](image1)

![Figure 4: EDX Spectrum of *Ipomoea batatas*](image2)

![Figure 5: EDX Spectrum of *Manihot esculenta*](image3)

From the EDX results (Figures 3 to 5) *Manihot esculenta* has the highest percentage carbon composition when compared to *Zea mays* with 65.24\% Carbon and 61.55\% composition in *Ipomoea batatas* nanoparticle starch. It was observed that there was no presence of sulphur in *Ipomoea batatas* nanoparticle starch unlike other synthesized nanoparticle starches.

The produced nanoparticles of *Zea mays* are rounded in nature with some level of agglomeration as shown in Figure 6. From the ImageJ analysis shown in Table 1, the particle length is within the range of 100 nm as indicated by the image scale with an average particle length of about 1.850 nm, confirming that the synthesized particles are truly in the nano scale and further validating the acid hydrolysis process. From Figure 7, the produced nanoparticles of *Ipomoea batatas* starch appear to be dense, flaky, crystalline and more clustered in nature, the particles fall in the size range of 200 nm with an average particle length of ~1.500 nm as
described in Table 1. The crystalline nature of *Ipomoea batatas* nanoparticles could be attributed to the absence of sulphur in its composition as seen in Figure 4.

The produced *Manihot esculenta* starch nanoparticles showed the greatest reduction in size as indicated by the micrograph scale in Figure 8, the nanoparticles are more dispersed and the particles have an average length of 2.920 nm as shown in Table 1. The particles are more rounded than those of *Zea mays*.

The dense, flaky and clustered morphology of the *Ipomoea batatas* starch nanoparticles may be as a result of lack of sulphur in its composition. The nanoparticle starches for *Manihot esculenta* and *Zea mays* compose of sulphur which may be responsible for roundedness of the nanoparticles. It was also observed during centrifugation that the slurries of *Ipomoea batatas* did not settle or coagulate as the other starches did which could be recognized to be the cause of the low yield of the starch. The yield of *Manihot esculenta* nanoparticle starch from the hydrolysis process was moderate, as it was more than that of *Ipomoea batatas* but still less than *Zea mays*.

Figure 6: TEM micrograph of *Zea mays* Figure 7: TEM micrograph of *Ipomoea batatas*

Figure 8: TEM micrograph of *Manihot esculenta*

| Starch         | Average particle size(nm) |
|----------------|---------------------------|
| *Ipomoea batatas* |                           |
| *Manihot esculenta* |                           |
| *Zea mays*     |                           |
From Figures 1 to 3, it could be observed that there was very significant reduction in the average nanoparticle size of the starch; this signifies that acid hydrolysis process as a valid method for preparing nanoparticles.

However, the hydrolysis process from these results is seen to have been most efficient on *Manihot esculenta* in terms of particle size reduction, and in terms of yield on *Zea mays*.

### 3.2 FTIR Analysis

FTIR analysis was carried out to determine the functional groups present in the starch nanoparticles and are presented in Figures 9 to 11.

From Beer’s law, it is known that the amount of absorption from the spectra is proportional to the amount of the constituent present in the composition of each of the samples.

The spectra for the various samples are presented and the functional group assignments for each starch are presented in Tables 2 to 4.

Generally, the starches are seen to have a uniform spectral shape with characteristic peaks occurring within the same regions.

However, a proper analysis of the transmittance peaks revealed some differences as explained: *Zea mays* and *Manihot esculenta* are seen to have sulphone functional groups which corroborates the results obtained in the EDX compositional analysis whereas *Ipomoea batatas* did not show the presence of the sulphone group.

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| Starch            | Reduction |
|-------------------|-----------|
| *Zea mays*        | 1.85      |
| *Manihot esculenta* | 2.92     |
| *Ipomoea batatas* | 1.57      |

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Figure 9: Infrared spectrum of *Manihot esculenta*
Table 2: FTIR band assignment for *Zea mays*

| S.No | Wave Number (cm\(^{-1}\)) | Functional Group                                    |
|------|-----------------------------|----------------------------------------------------|
| 1    | 381.49                      | Sulphones (S=O stretching)                          |
| 2    | 416.25                      | Sulphones (S=O stretching)                          |
| 3    | 581                         | Alkylbromides(C-Br)                                 |
| 4    | 880.2                       | Carbohydrates (C-O-H ring mode vibration)          |
| 5    | 1050                        | Alcohol (C-O-H stretching)                         |
| 6    | 1199.82                     | Ethers (C-O-C antisymmetric stretching)            |
| 7    | 1644                        | Carbonyl (C=O stretching)                          |
| 8    | 2256                        | Nitrile (C≡N triple bond)                          |
| 9    | 3426                        | Water of crystallization (O-H stretching)          |
Common absorption peaks were noticed in the three samples around the 3300 to 3600 cm\(^{-1}\) range which was attributed to the O-H group stretching, 1630 to 1750 cm\(^{-1}\) which was attributed to the C=O stretch due to the carbonyl group which was also observed by [18] in his study of natural and acetylated thermoplastic starch where the characteristic carbonyl group was identified in the 1740 cm\(^{-1}\) region for all his samples [18].

The C-O functional group was also observed in the 1000 to 1100 cm\(^{-1}\) range for all the starches as a result of the alcohol functional group.

In addition, the characteristic peak at the 800 to 880 cm\(^{-1}\) appeared for all the starches signifying the carbohydrates functional group of which all the starches fall into by virtue of their primary food group.

Finally, the results of this analysis prove that the synthesized nanoparticle starches possess significantly the same functional groups as the raw macro-particle starches as seen in the research by [19, 20, 21] who characterized potato, corn and cassava starches to identify similarities in their chemical properties and it can be seen that the results obtained in this research match closely those obtained by the researchers [19, 20, 21].

Table 3: FTIR band assignment for *Manihot esculenta*

| S.No | Wave Number (cm\(^{-1}\)) | Functional Group                           |
|------|----------------------------|-------------------------------------------|
| 1    | 442.18                     | Sulphones (S=O stretching)                |
| 2    | 877.56                     | Aldehydes (C-H out-of-plane deformation)  |
| 3    | 1048.33                    | Alcohols (C-O stretching)                 |
| 4    | 1197.78                    | Ethers (C-O-C antisymmetric stretching)   |
| 5    | 1642.33                    | Ketones (C=O stretch)                     |
| 6    | 3426                       | Water of crystallization (O-H stretching) |

Table 4: FTIR band assignment for *Ipomoea batatas*

| S.No | Wave Number (cm\(^{-1}\)) | Functional Group                           |
|------|----------------------------|-------------------------------------------|
| 1    | 875.85                     | Carbohydrates (C-O-H ring mode vibration) |
| 2    | 1047.95                    | C-O stretching                            |
| 3    | 1195.85                    | Ethers (C-O-C antisymmetric stretching)   |
| 4    | 1641                       | Carbonyl (C=O vibration)                  |
| 5    | 3424                       | Water of crystallization (O-H stretching)  |

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3.3 X-Ray Diffraction Characterization

Figure 12: a: X-ray diffractogram of *Zea mays* b: X-ray diffractogram of *Ipomoea batatas* c: X-ray diffractogram of *Manihot esculenta.*
Starch naturally comprises of semi-crystalline granules which could be in various polymorphic structures which include A-type, B-type and C-type. The effect of recrystallization on the crystalline structure of starch nanoparticles was studied by using X-ray diffraction. The diffractograms shown in Figure 12 reveals strong diffraction peaks at around 18°, 20°, 23°, 25° and 28° (2θ) for Zea mays which is similar to the results obtained by [22] amongst other authors where a characterization of several starch nanoparticles was carried out revealing similar peak patterns. The diffractograms of Zea mays and Manihot esculenta (Figures 12a and 12b) indicate typical diffraction behaviour of cereals which is of type A X-ray pattern while that of Ipomoea batatas indicate typical B-type pattern with characteristic peaks at 17° which is particular to tuber starches as suggested by [23]. The loss of the crystalline amylopectin accompanied by increase in amylose fractions is evident in the disappearance of the peaks at 18°, 20° and 23° which can be linked to the structure of the particles as seen in the TEM micrographs.

4 Conclusion
The aim of this study was to produce and characterize nanoparticulate starches of Manihot esculenta, Zea mays and Ipomoea batatas using the acid hydrolysis process. From the above results, it is evident and can be concluded that despite the hydrolysis process carried out on the starches to convert them to nanoparticles, the chemical constituents and functional groups of the nanoparticulate starches still remained intact as shown from the FTIR analyses comparing the commercial starch and those of the produced nanoparticles. X-ray diffraction for this study also corresponded with research of previous authors corroborating the validity of this research. Finally, it can be evidently concluded that the acid hydrolysis is a suitable method for synthesizing nanoparticulate substances whilst still maintaining the characteristic properties of the original substance.

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