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Synthesis, Characterization and Aqueous-Release Properties of Zinc-Starch Nanoparticles Encapsulating Pendimethalin

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Abstract

To caution the effect of environmental pollution caused using pesticides for the increase in agricultural yield, the development of controlled and slow-release delivery systems is crucial, zinc-starch composite as a delivery agent due to its slow-release property is used in the preparation of controlled released formulation of pendimethalin using hydrothermal bio-template method. Pendimethalin was encapsulated in the synthesized zinc-starch nanoparticles and were characterized using scanning electron microscopy (SEM) and Fourier transformed infrared (FTIR) spectroscopy. The SEM images showed aggregate bud-shaped while the FT-IR spectra were observed at room temperature showing different peaks of functional groups. The aqueous release monitored for 96 hours in acidic (pH 4) basic medium (pH 9) and in neutral medium (pH 7) the cumulative percentage release was found to be 81 %, 77 %, 50% respectively due to the stability of pendimethalin in both acidic and basic media, Therefore, encapsulation of pendimethalin in zinc-starch nanoparticles is a good way of decreasing its rate of release and hence reducing pesticides hazard in the environment.

Keywords: Zinc, Starch, Pendimethalin, Aqueous Release

Introduction

Pesticides currently are widely used for disease and pest management in developing countries. Consistently environmental and human health issue due to pesticide residue as a result of pesticide handling are usually pronounced in these countries this is because agricultural workers and farmers may be unable to read and understand labels and do not have adequate safety instructions and personal protective equipment, however, with the teaming awareness of proofs that these chemicals contribute a long-term and serious risks to environment and human health, the resulting impact to plant and life do not conform to the united nations sustainable development goal [1]. Starch is a polysaccharide, composed of a great number of glucose monomers joined by glycosidic bonds. It is inexpensive, versatile, and well-known, polysaccharide which has attained great attention in delivery of drug as this is because they are biocompatible with cells and tissue, biodegradable, and hydrophilic. [2]. Starch nanoparticles having particle sizes within the range of 10– 1000 nm has been extensively studied as nano carriers for controlled release. example, propyl starch nanoparticles encapsulated with different types of drugs (testosterone, flufenamic acid, and caffeine) exhibit enhanced effectiveness upon permeation studies on the human skin 5-fluorouracil (5-Fu) captured in Dialdehyde starch nanoparticles (DASNPs) were found to have enhanced cancer cell in breast, free 5-Fu as compared to (MF-7) inhibition in vitro [3].

Pendimethalin is a pre mergence and post mergence herbicide belonging to the dinitroaniline class used to control certain broadleaf weeds and annual grasses. It inhibits cell elongation herbicide resistance action committee (HRAC) classification and is permitted in South America in Europe, North America, Asia, Africa, and Oceania for various plant, these include cereals (wheat, rye, barley, triticale), lawns and ornamental plants, corn, rice, soybeans, legumes, potato, nuts, fruits, as well as vegetables, [4]

Pendimethalin protects plants such as corn, wheat, soybeans, cabbage, potatoes, peas, asparagus and carrots. It is used to control certain broadleaf grasses and annual weeds which interfere with development, growth, quality and yield of horticultural and agricultural crops by competing on water, nutrients, and light [5]. In places where grass infestation is predominantly high, yield losses render the production of wheat economically unviable. In addition to wheat, a great number of plants are cultivated in Europe this is a relatively a small percentage of total output of agriculture. The options of Herbicide are limited for these minor plants having few available herbicides that are effective in the vegetable sector. [5] In Germany, Long-term field studies performed by advisory institutes and Governmental research together with agriculture workers ranked Pendimethalin as an herbicide that can efficiently be used to control black grass, with respect to crop yield, weed control efficacy, environmental impact and treatment costs. It has been shown that Pendimethalin exposure increases substantially the risk of developing cancer of pancreas [6] one of the most lethal forms of cancer. In the international journal of cancer, a study revealed threefold increase among applicators of this herbicide in the top-half of lifetime. [7].

Materials and Methods

Materials

The materials procured for this research includes: Pendimethalin ($C_{13}H_{19}N_3O_4$), analytical grade zinc chloride ($ZnCl_2.2H_2O$) as precursor cassava starch as stabilizer and capping agent, sodium hydroxide (NaOH) as pH moderator, methanol, ethanol and distilled water. All chemicals used for this research work were obtained from Emole Chemicals Nigeria. Limited, high level Makurdi and used without further purification. All solutions were prepared using distilled water. Equipment includes Centrifuge (Fisher Scientific model 228), analytical weighing balance, dropping pipette, pH meter, mortar/pestle, sieves, thermometer, stirrer, spatula, wash bottle, furnace, syringes, aluminum foil and filter paper. Regular laboratory glass wares like volumetric flasks measuring cylinder breakers test tubes funnels conical flasks and pipettes were also employed; these were properly washed with nitric acid and dried. UV-Vis spectrophotometers, FT-IR spectrometer and Scanning Electron Microscopy

Methods

In preparing 1000mL ppm Pendimethalin stock 0.3mL (0.15g) of the herbicide formulation was drawn into 1000mL volumetric flask and was made up to mark using distilled water. MeOH: H_2O (80:20 v/v) solution: Concentrated methanol of 80 ml measured using 100 ml measuring cylinder, it was transferred into a 100ml volumetric flask and was made to mark with distilled water. H_2O (80:20 v/v) solution: Concentrated methanol of 80 ml measured using 100 ml measuring cylinder, it was transferred into a 100ml volumetric flask and was made to mark with distilled water.

Cassava Starch Processing

Cassava roots were harvested peeled and washed. These were grated, mixed with water and filtered, the filtrate was allowed to settle and the water removed by pressing in a clean muslin cloth. They were dried and milled to get cassava starch.

Synthesis of Zinc Starch Nanoparticles

The method of [8] was employed with slight modification. 1g of cassava starch was completely dissolved in 40ml of distilled water, and then heated at 60^{0}C to obtain a clear gel. 4.5g of $ZnCl_2$ was dissolved in 10ml of distilled water with constant stirring for 30 minutes. Meanwhile, 1.5g of NaOH is dissolved in 10ml of distilled water for about 30 minutes with constant stirring the aqueous solution of $ZnCl_2$ is mixed with the clear starch solution and stirred. NaOH solution was added drop-wise to the starchzinc chloride aqueous solution with constant manual stirring to achieve a final pH value of 8.4. The composite was aged for 30 minutes with constant stirring at 80 °C, the Zn-starch nanocomposite was obtained. The Zn-starch nanoparticles were easily obtained by calcining the dried Zn starch nanocomposite at 600°C for one hour.

Loading of Pendimethalin onto Zinc-Starch Nanoparticles

The method by [9] was adopted for the loading of Pendimethalin. Pendimethalin (0.05g) i.e 0.1ml of Pendimethalin was added to 20ml of methanol: water solution to form pendimethalin solution. Zinc-starch nanoparticles (1.0g) were dissolved in 10ml of distilled water. The pendimethalin solution was added to Zinc Starch Nanoparticles aqueous solution. The resultant mixture was stirred manually and left standing overnight (24 hrs) to remove the solvent. The pendimethalin encapsulated Zinc starch NPs was harvested by centrifugation at 3400 rpm for 30 min. washing was done thrice with distilled water. After final washing, Pendimethalin Zinc Starch Nanoparticles obtained were dried in an oven at 40^{o}C for 6 hours. The dried loaded NPs were properly stored in vacuum desiccators for further use.

Characterization of the Synthesized Zinc-Starch Nanoparticles Scanning electron microscopy (SEM) analysis

The scanning electron microscopy (SEM) was used for particle size distribution and morphology of the synthesized Zinc Nanoparticles and the pendimethalin loaded nanoparticles. This is carried out using the scanning electron microscopy. Samples will be dropped on stainless steel plates dried at room temperature, and coated with a layer of platinum using auto fine coater. The SEM images of the platinum coated nanoparticles will then be obtained; images of blank zinc-starch nanoparticles will also be obtained. This will also be repeated for the pendimethalin zinc-starch nanoparticle

Fourier transform infra-red Spectroscopy (FT-IR)

Fourier transforms infra-red spectroscopy (FT-IR) analysis was conducted to determine the possible molecular interaction(s) between the plain synthesized zinc-starch nanoparticles and the pendimethalin loaded nanoparticles. About 2 mg of sample and 200 mg KBr will be dried and ground the particle size will be unified and less than two micrometers then, the mixture is squeezed to form transparent pellets which can be measured directly to obtain the FTIR of sample. This method will also be employed for the pendimethalin zinc-starch nanoparticles.

Herbicide loading determination

Method presented by [10] was adopted. The pendimethalin loaded Zinc-starch nanoparticles were separated from the aqueous suspension medium by centrifugation at 3400 rpm and 25^{o}C for 30 minutes. The amount of free pendimethalin was measured in the clear supernatant by UV-Vis spectrophotometer at a wavelength of 250nm (Wavelength obtained from calibration curve for pendimethalin). The encapsulation efficiency (EE) or loading efficiency (LE) of the nanoparticles was calculated.

Pendimethalin release study

1g of pendimethalin loaded zinc-starch nanoparticle was placed in 15ml of Buffer solution at pH 7at 37±0. 5 5ml aliquot of the solution was taken every 5 minutes and 5ml of the buffer solution replaced to maintain a sink condition [8] the amount of pendimethalin released in the supernatant was determined using a UV/VIS spectrophotometer. The concentration of pendimethalin released is calculated with reference to the regression equation generated from constructed calibration curve of pendimethalin. The percentage released at a specific time will be determined based on the following equation.

$$\% \, Released \, of \, pendimethalin = \frac{(pendimethalin \, Released) \, X \, 100}{(pendimethalin \, loaded)}$$

Results

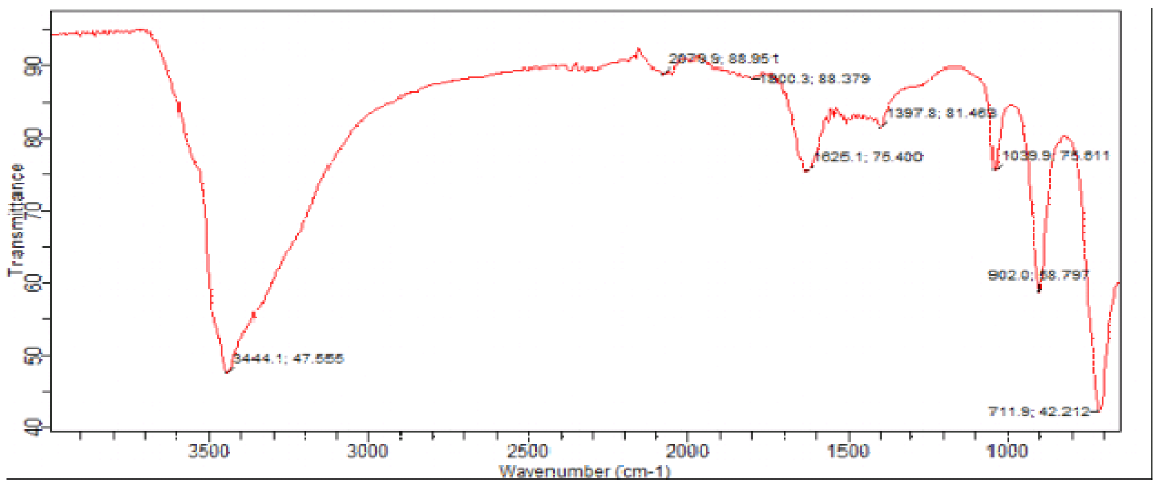

Figure 1: FT-IR of zinc-starch nanoparticles

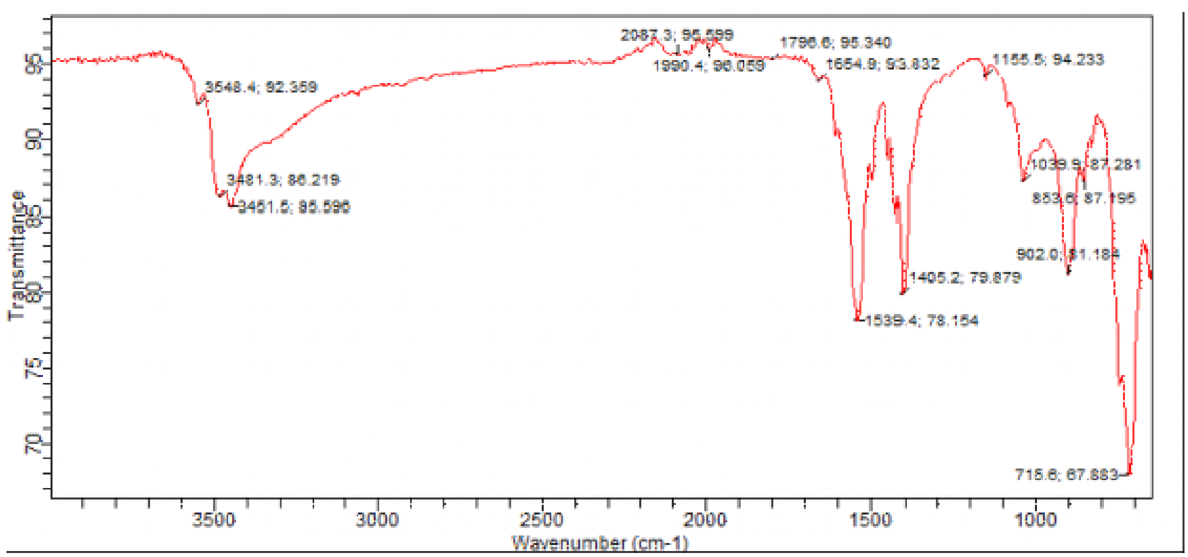

Figure 2: FT-IR spectrum for pendimethalin zinc-starch nanoparticles

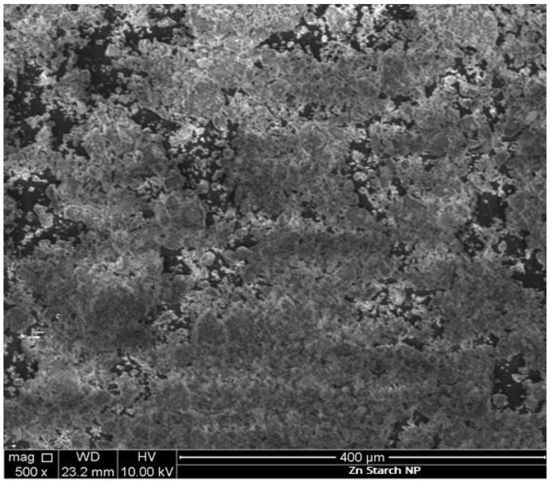

Plate 1: SEM image of zinc-starch nanoparticles(mag: 500x)

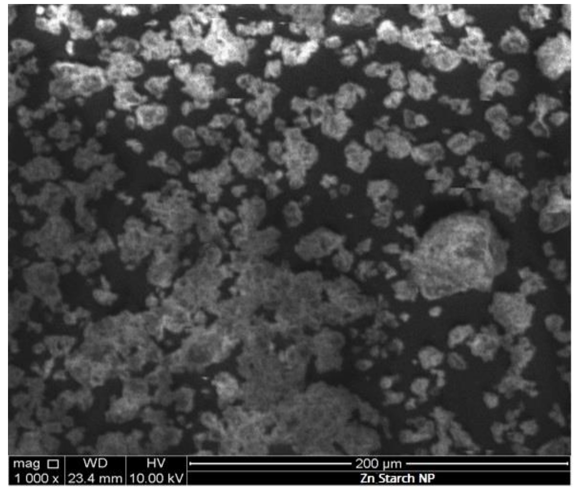

Plate 2: SEM image zinc-starch nanoparticles (mag: 1000 x)

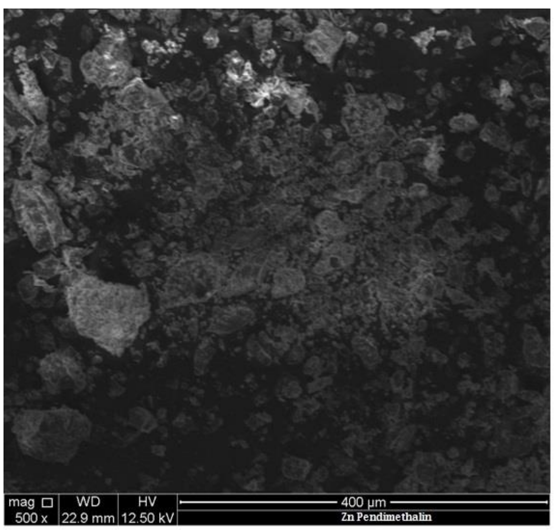

Plate 3: SEM image of pend-Zn-St. nanoparticles (mag: 500)x

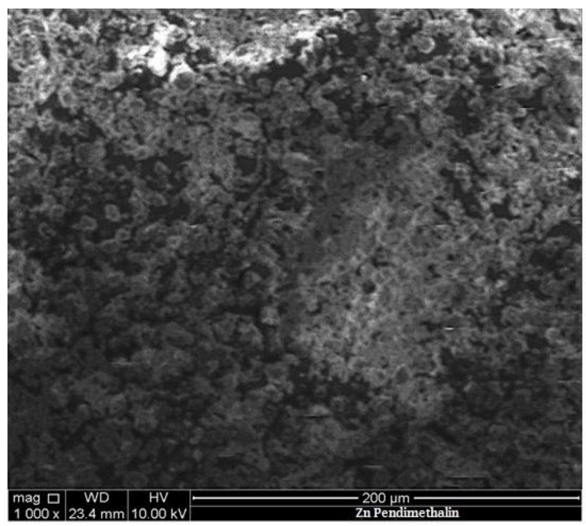

Plate 4: SEM image of pend-zn-starch nanoparticles (mag: 1000 x)

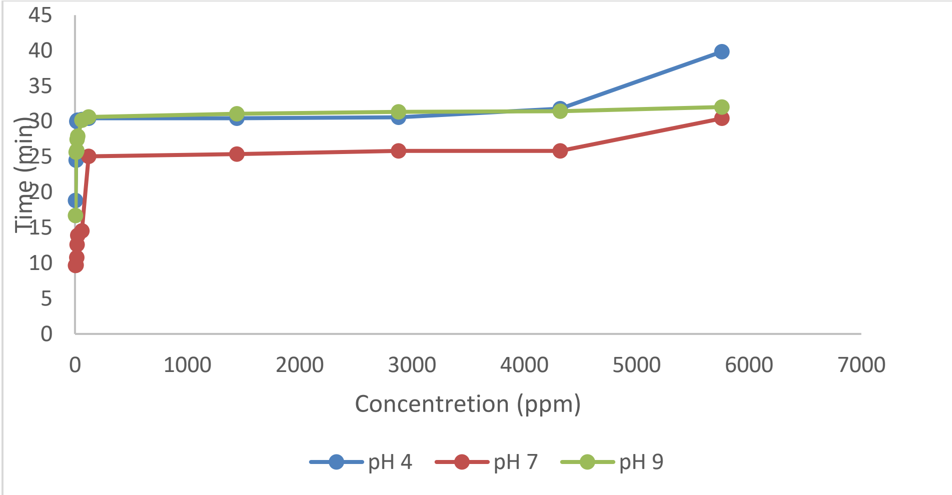

Figure 3: Concentration (ppm) of Pendimethalin released from Zinc-Starch Nanoparticles in Buffer Solutions of pH 4, 7 and 9

Discussion

Loading efficiency

With the method earlier employed for the loading of the herbicide unto the zinc-starch nanoparticles, it was observed that the loading efficiency of the herbicide onto the synthesized zinc-starch nanoparticles was found to be 80%.

Scanning electron microscopy (SEM)

SEM was one of the instruments used in the characterization of the zincstarch nanoparticles, and the pendimethalin zinc-starch nanoparticles , this is to determine the morphology, agglomerates rather than single particles were observed, the morphology obtained shows that the appearance of the particles are bud-shaped they do not appear as discrete particles but form larger ones, this observation of such larger nanoparticles are a clear indication that the particles are composed of van der Waal clusters of smaller entities, also there are magnetic interactions among the particles this conform very well to that observed by [11]

Aqueous release of synthesized pendimethalin zinc-starch nanoparticles

This was carried out in pH 4, pH 9 and pH 7, acidic neutral and basic media respectively, the aim of this work is to study the release of pendimethalin from the formulated zinc-starch nanoparticles, the mode of release in the different media show a steady pattern as time increases. At all the pH, there was a burst release, presumably from the particle surfaces , followed by sustained release by diffusion of the herbicide through the nanoparticles wall and nano erosion [11] this can be of advantage since the sprouting of weed seeds and still exist to kill and inhibit new infestation [12] in this work it shows that the release process can still continue for a longer period beyond 96 hours-time frame used in the work this may ensure 100% release of the encapsulated herbicide.

The cumulative percentage release of pendimethalin as observed showed that the highest release was observed in the acidic medium, pH4, with 81% this great difference in the release rate can be due to the enhanced solubility and stability of pendimethalin under acidic condition. [12] and [13]. The neutral medium was observed to have the least release percentage, (51%), in the alkaline medium, cumulative release was found to be (77%), lower than the acidic medium and higher than the neutral medium, this release also proved that pendimethalin is stable under alkaline condition [14]. The statistical treatment using ANNOVA shows that there is no significant difference between pH4 and pH9 as mean difference is non-significant ($p>0.05$).

Fourier transform –infra red (FT-IR)

This was employed to investigate the effect of the bio template (cassava starch) on the resulting chemical properties of zinc-starch nanoparticles. FT-IR spectra were observed at room temperature in the range of 4000280cm^{-1} figures 1 and 2 show different absorption at various frequencies, cassava contains mainly starch (carbohydrates) and water with minor components such as proteins, vitamins lipids etc this composition mostly contain alkene, esters , aromatic and alcohols with different combination of functional group as shown in the figure 1 the zinc-starch nanoparticles involves characteristic peaks such as a strong peak at 3441cm^{-1} is attributed to O-H stretching vibration possibly including H_2O, alcohols OH phenolic OH and/or carboxylic OH [15], a band 2845cm^{-1} is assigned to stretching of N-H amine, however the absorption band at 2079cm^{-1} represent stretching vibration of aliphatic C=C=C, the observed strong peak at 1800cm^{-1} indicates C=O peak at 1625 cm^{-1} is assigned to C=C bending in aromatic compound [20]. Absorption bands at C-O in aromatic ether and tertiary alcohols respectively. The strong break at 902 cm^{-1}indicates C=C bending in alkene, 711 cm^{-1} shows a derivative of benzene. [16]. In figure 2, new peak was also observed at 3481cm^{-1} which indicates N-H in primary amine and medium peak at 3451cm^{-1} in aliphatic primary amine, 2087 cm^{-1} C=C=C another new weak peak at 1990 cm^{-1}for aromatic bending in aromatic compound there is a decrease in peak from 1800 cm^{-1} strong peak to 1790 cm^{-1} C=O, and an increase in peak from 1625 cm^{-1} to 1654 cm^{-1} for C=C stretching in alkene and angular deformation in alkane, while that of 1155 cm^{-1} indicates the C-O stretching of C-O-C in group of anhydrous glucose ring of starch [17]. Comparing the both spectra, peaks for C-O in tertiary alcohol and the peak for C=C bending in Alkane, it was observed that there was an insignificant difference in their peaks at 1039 cm^{-1} and 902 cm^{-1}respectively.

Conclusion

In conclusion, zinc-starch nanoparticles were successfully synthesized encapsulating pendimethalin, the method of formulation was found to be ecofriendly, the loading efficiency was also observed to be 80%, the morphology of the synthesized zinc-starch nanoparticles as observed showed agglomerated particles rather than single discrete particles. The aqueous release carried out showed burst release followed by a steadily sustained release, pH4, acid medium has the highest cumulative release of the herbicide, this is followed by the cumulative release at the alkaline medium and the neutral showed least cumulative release of the herbicide. The FT-IR functional groups recorded for pendimethalin zinc-starch nanoparticles showed that the stabilizing ability of starch in the formulation for control release study is important since peaks showed that the herbicides was successfully encapsulated on the zinc-starch nanoparticles.

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