

Production and Characterization of Hydroxyapatite Prepared From Periwinkleshell

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Abstract— *The application of biogenic wastes for the production of hydroxyapatite for the adsorption of Contaminants from wastewater has become attractive in recent time. Hydroxyapatite was produced from periwinkle shell in this study. The periwinkle shell was washed, sun-dried for 48 h and later soaked in 50% hydrogen peroxide for 24 h to oxidize all organic matters present. The periwinkleshell was ground and dried in an oven at 105°C; thereafter, it was calcined at 400°C for 2 h and ground to granules before soaked with Di-ammonium hydrogen phosphate solution for 24 h. The calcined hydroxyapatite (PSHAP) was oven-dried at 105 °C for 24 h and sieved to 2 mm mesh sizes. The Fourier Transform Infrared (FTIR) analysis of the hydroxyapatite was characterized by broad band at 3437 cm⁻¹, representing bonded -OH groups. The band observed at about 2913–2847 cm⁻¹ was assigned to the aliphatic C - H group. The peak around 1786 cm⁻¹ corresponds to the C=O stretch. The peaks observed at 1682, 1548 and 1513 cm⁻¹ correspond to (NH) the secondary amine group. Similarly, the Scanning Electron Microscopy (SEM) analysis shows that the average pore size on the hydroxyapatite (PSHAP) is 50 μm. This indicates that the hydroxyapatite (PSHAP) produced from periwinkle shell is a potential adsorbent for the adsorption of pollutants found in wastewater.*

Keywords— *Adsorption, periwinkle shell, Production, Characterization, hydroxyapatite.*

I. INTRODUCTION

In recent years, there is greater environmental awareness on the need to treat wastewater effluents before discharging into receiving water bodies. This has necessitated a great deal of research into finding cost-effective methods for the removal of contaminants from wastewater [Zahra, (2012)]. Hydroxyapatite (HAP) is a phosphate crystal of calcium which has a replaceable hydroxyl ion. The empirical formula of HAP is Ca₅(PO₄)₃(OH). The OH⁻ ion in the

crystal may be replaced by metals, halides or carbonates. Most natural bone material contains about 70% of hydroxyapatite (Wikipedia). Therefore, bones and teeth are very good sources of hydroxyapatite [Thomson et al., 2003]. HAP interacts very well with lanthanides and actinides [Jerden and Sinha]. Actinides form surface complexes, and rare earth elements substitute calcium in the crystal lattice of HAP [Jones et al., 1996]. The sorbed metals react and form stable metal-phosphate minerals with apatites. Uranyl phosphate mineral, which is formed when uranium interacts with HAP, is so stable that temperatures as high as 800-1200°C may be required to decompose the mineral. Stability under a wide range of geological conditions is a very attractive property of apatite [Nriagu, 1974]. The metals adsorbed by apatite cannot be desorbed, leached or even exchanged, at a broad range of pH and temperature [Conca and Wright, 2006].

Hydroxyapatite (HA) Ca₁₀(PO₄)₆(OH)₂ is a bioactive ceramic that is a major mineral component of bones and hard tissues. It mimics the mineral composition of natural bone and has been found to provide a stimulating effect on bone formation, which is known as Osseo induction that results in the formation of strong bonds with bone [Kweh et al., 1999]. HA is employed in forms such as powders, porous blocks or beads to fill bone defects or voids. Apart from these applications, it can also be used as coatings on implant materials. Implant materials such as titanium alloys which are used inside the human body generally fail due to the occurrence of significant localized corrosion by interactions with corrosive body fluids. Implants also undergo loosening and detachment from the bone due to poor biocompatibility of the implant material. In order to overcome these difficulties, nanohydroxyapatite coated titanium implants are suggested [Birtwistle et al., 1996]. Hydroxyapatite [Ca₁₀(PO₄)₆(OH)₂; HAp] is the main mineral constituent of teeth, bones and phosphate mineral rocks. It was shown that HAp is efficient in removal of many toxic metal ions [Mobasherpour et al., 2012]. Among

the different sources of phosphate, both natural and synthetic Hydroxyapatite (HAp) has been supposed as a reactive material to remove metals [Oliva et al., 2011]. Natural sources are more cost-effective and safer over cross-reaction and other immunological reactions for the synthesis of HAp.

Periwinkle shell contains 95-99% by weight of CaCO_3 which has enabled it to be applied for a number of purposes. Via thermal decomposition process known as calcinations, CaCO_3 can be converted into CaO which is used in the Synthesis of HA. There are many methods that can be used to produce HA powder, such as precipitation, solid-state synthesis, hydrolysis, hydrothermal and sol-gel method [Webster et al., 2000]. However, precipitation method is the most often method used to produce HA powder either by the reaction between diammonium hydrogen phosphate with calcium nitrate or the reaction between orthophosphoric acid and calcium hydroxide [Asaoka et al., 1995, Zhang and Gonsalves, 1997].

II. METHODOLOGY

2.1 Materials

Fresh periwinkle shell was obtained from Oyingbo market, Ebutemetta, Lagos State. The shell was washed thoroughly with tap water and later with distilled water, to reduce the odour, sun-dried for 48 hours and later crushed into pellet. It was soaked in 50% hydrogen peroxide for 24 hours to

oxidize all organic matters present and it was then rinsed repeatedly with distilled water, and air dried for 24h on laboratory bench, it was oven dried at 105° for 24 h to remove moisture content.

2.2 Production of Hydroxyapatite from periwinkle shell.

The dried periwinkle shell (PS) was Calcine using an electric furnace at 400°C for 2 h. The calcined sample was ground into granular form and sieved to a particle size of 2 mm; soaked with Di-ammonium hydrogen phosphate solution for 24 h. The liquid part was decanted and the sediment was washed repeatedly with distilled water and drained using Buchner funnel and suction pump, this was then oven dried at 105°C for 2 h to remove moisture content completely. Prepared PSHAP store in tight container and kept in a desiccator for further use.

III. CHARACTERIZATION OF ADSORBENT

3.1 FTIR Characterization of HAP: Infrared analysis was conducted on the PSHAP to investigate the functional groups present on its surface in the range of $500\text{--}4500\text{ cm}^{-1}$ using Fourier Transform Infra-Red spectrometer (Perkin-Elmer Spectrum GX, Kuala Lumpur, Malaysia). The pore sizes were investigated with scanning electron microscope (SEM, JEOL JSM-6480 LV).

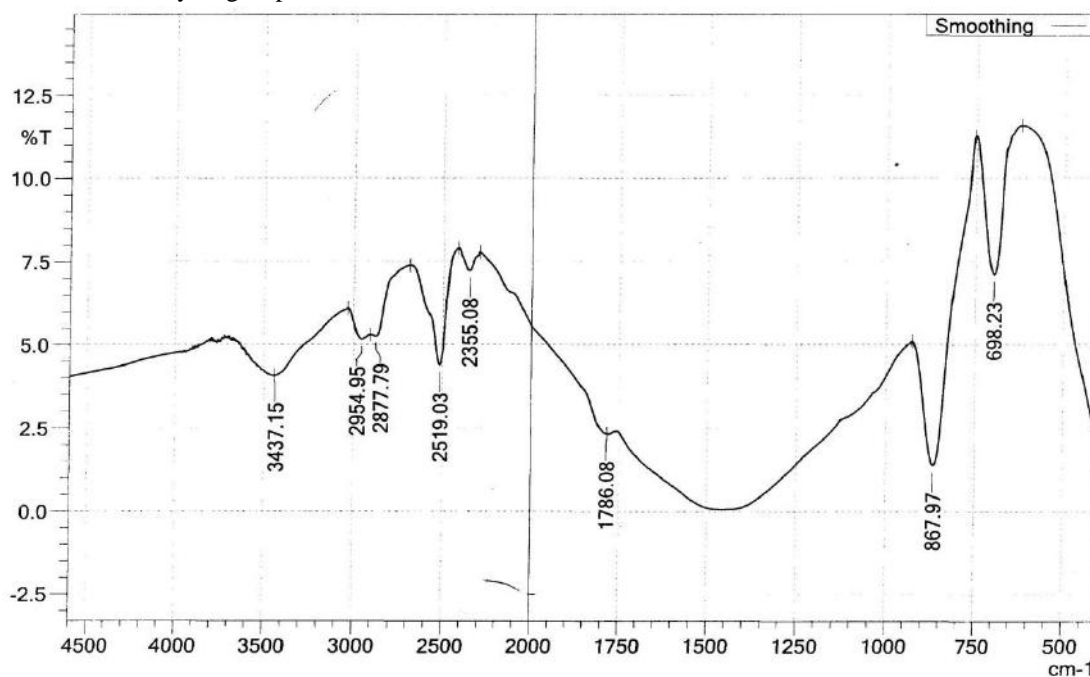


Fig.1: FTIR characterization of PSHAP.

FTIR Spectra of periwinkle shell Hydroxyapatite (PSHAP) Surface

The FTIR spectra of PSHAP were taken to ascertain the possible involvement of the functional groups on the surface of PSHAP. The broad band at 3437 cm^{-1} , representing bonded $-\text{OH}$ groups. The band observed at about $2913\text{--}2847\text{ cm}^{-1}$ was assigned to the aliphatic $\text{C}-\text{H}$ group. The peak around 1786 cm^{-1} corresponds to the $\text{C}=\text{O}$ stretch. The peaks observed at 1682 , 1548 and 1513 cm^{-1} correspond to $(\text{N}-\text{H})$ the secondary amine group. The peaks at $1709.05 - 1682\text{ cm}^{-1}$ may be attributed to $\text{C}=\text{O}$ stretching,

$-\text{CH}$ bending vibrations of Carbonyl compound, respectively.

3.2 SEM

Plate: 1 show that SEM images of the PSHAP exhibits a honeycomb, caves-like, uneven and rough surface morphology shape. This shows that the PSHAP was effective in creating well-developed pores on the surface of the precursor, leading to PSHAP with a large surface area and porous structure [Ricou-Hoeffler et al., 2001, Hameed and Daud, 2008].

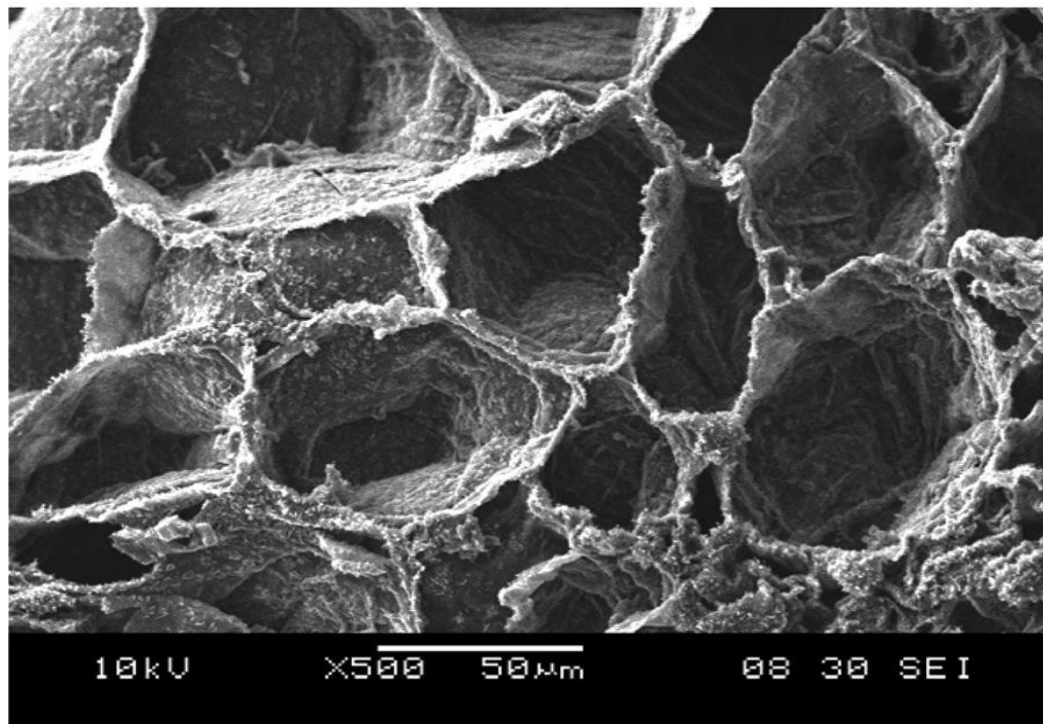


Plate.1: The SEM image of the raw PSHAP

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