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SYNTHESIS AND CHARACTERISATION OF B-CYCLODEXTRIN AND HYDROXYAPATITE COMPOSITE BY ULTRASONICATION METHOD

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ABSTRACT

Cyclodextrins (CDs) are one of the most versatile substances produced by nature, and it is in the aqueous biological environment where the multifaceted potential of CDs can be completely unveiled. Cyclodextrins form inclusion complexes with a variety of guest molecules, including polymers, producing very diverse biocompatible supramolecular structures. Among the polymeric biomaterials, hydroxyapatite plays an important role by possessing biomedical applications. In the present work, we have synthesized two component composite system incorporating β-cyclodextrin with hydroxyapatite by simple ultrasonication method in order to produce inclusion complexes of β-cyclodextrin. The binary composite has been characterised by using Fourier transform infrared spectroscopy (FT-IR) and Thermo gravimetric analysis (TGA) technique. Using this technique, we have concluded that intermolecular interactions between the two components and also thermal stability of the prepared sample.

KEYWORDS: β-cyclodextrin, biomaterial, hydroxyapatite, inclusion complex.

INTRODUCTION

Polysaccharides have become increasingly important materials over the past decade. Polysaccharides offer a green alternative to synthetic polymers.[1][2] Polysaccharide composites are composition material made from two or more constituents with significant physical or chemical properties with superior performance even at higher temperatures. The applications of polysaccharides used in preparing composites for biomedicine, for energy

production and storage, for electrical devices, in separation science, and in industrial and materials applications were established.[3-5] On accout of that β-Cyclodextrin and Hydroxyapatite composite has conquered major applications in biomedical field. The compounds nowadays called cyclodextrins were called cellulosine when first described by Villier[6] in 1891. Cyclodextrins (CDs), the water-soluble cyclic oligosaccharides, composed of α-D-glucopyranoside units linked in 1-4 manners, are synthetic substances obtained from the enzymatic degradation of starch by making use of a glycosyl transferase from *bacillus macerans.* CDs belong to the group of cage molecules having the core of their structure composed of a dimensionally stable hydrophobic cavity leading to a "host−guest" type relationship that can improve the chemical, physical and biological properties of the guest molecule and hence finding applications in the fields of pharmacy, chemistry, biotechnology, agriculture and medicine etc.[7][8]

The formation of the inclusion compounds greatly modifies the physical and chemical properties of the host molecule, mostly in terms of water solubility. This is the reason why cyclodextrins have attracted much interest in many fields, especially pharmaceutical applications[9] by making inclusion complexes with hydroxyapatite. Hydroxyapatite is the most suitable ceramic material for hard tissue replacement and most widely accepted biomaterial for the repair and reconstruction of bone tissue defects.[10] It has all the characteristic features of biomaterials such as biocompatible, bioactive, osteoconductive, non-toxic, non-inflammatory and non-immunogenic properties.[11]

In order to prepare the inclusion complex, it is also known that in addition to carbonyl groups, the alcohol functionalities present in the β-CDs structure accelerate the nucleation of hydroxyapatite through electrostatic attraction of calcium ions.[12] β-CDs can also bind other metal ions such as silver, to generate hydroxyapatite/β-CD composite.[13]

The present study was aimed to prepare the beta-cyclodextrin and HAp composite by ultrasonication method. The prepared composite was characterized using FT-IR, TGA and DTA analysis. The results were discussed below.

MATERIALS AND METHODS

Materials

β-Cyclodextrin (β-CD) and Hydroxyapatite were purchased from MPM Scientific chemicals india private Ltd. All chemicals were of reagent grade and were used as received, and distilled water was used in all solutions and reagents throughout the experiment.

Preparation of β-Cyclodextrin and Hydroxyapatite (β-CD-HAp) composite

About 0.5 g of β-Cyclodextrin was dissolved in hot water and kept aside. Then, 0.5 g of Hydroxyapatite was weighed and dispersed in minimum amount of distilled water. This dispersed HAp was slowly added to β-Cyclodextrin in hot water with constant stirring. The mixture was agitated at room temperature using ultrasonicator for 45 minutes. The homogenous solution was poured into petridish and kept for air drying.

Characterization

Fourier Transform Infra Red Spectroscopy

The FT-IR spectra of β-CD/HAp composite was recorded by Fourier transform infra-red spectrophotometer (FT-IR) using the SHIMADZU FT-IR Spectrophotometer in the wavelength range of 400 – 4000cm^{-1}.

Thermogravimetric Analysis

Thermogravimetric analysis of the prepared samples was performed using SDT Q600 V8.0 Build 95 instrument. The range of temperature used is between 20ºC to 800ºC with a heating rate of 10ºC/min under nitrogen atmosphere.

RESULTS AND DISCUSSION

FT-IR spectral analysis

The FTIR spectral details of pure β-Cyclodextrin and β-CD/HAp composite was represented in Fig.1 and Fig.2. The FT-IR spectra shows all the characteristic bands of functional groups present in the pure β-Cyclodextrin and β-CD/HAp composite.

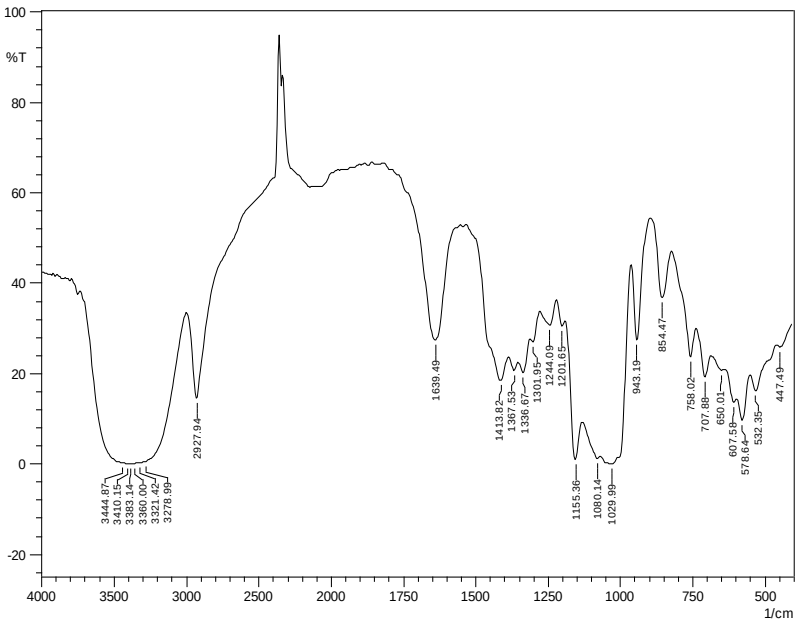

Figure-1: FT-IR spectrum of pure β-Cyclodextrin.

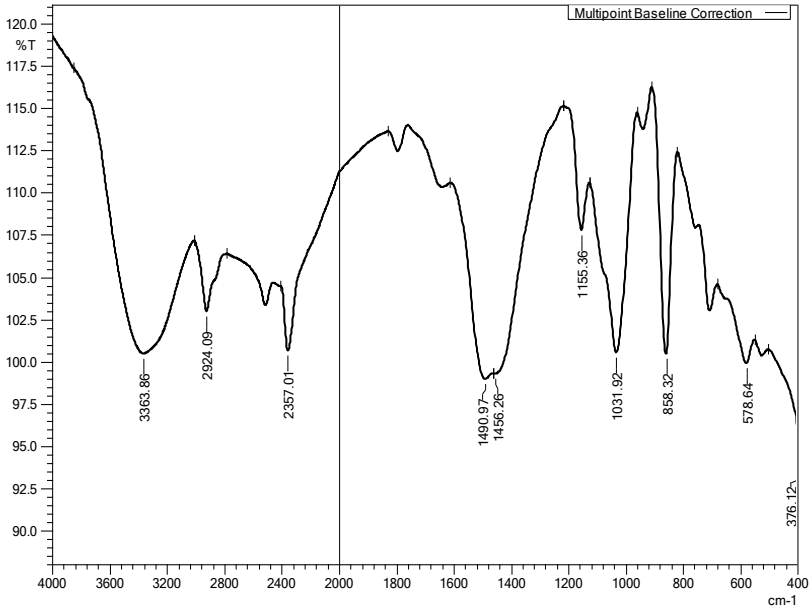

Figure-2: FT-IR spectrum of β-CD/HAp composite

The absorption peaks observed at 3444.87 cm^{-1} and 2927.24 cm^{-1} in case of pure β-CD (Fig.1) corresponds to the intermolecular hydrogen bonded -OH stretching frequency of hydroxyl group[14] and aliphatic methylenic-CH stretching. The sharp peak observed at wavenumber such as 1639.49 cm^{-1} corresponds to the presence of C=O stretching, the absorption bands at 1155.36 cm^{-1} and 1029.99 cm^{-1} revealed the presence of C-O-C stretching and O-H bending vibrations respectively.

The FT-IR spectrum of the β-CD/HAp composite has shown in the Fig. 2. The figure shows that the broad peak appeared in the range of 3363 cm^{-1} may be due to the overlapping of OH stretching band of HAp with the OH stretching band of β-CD. The shifting of bands to lower values indicates the strong interaction between β-CD and HA during ultrasonication and also with composite formation. The appearance of new bands in the range of 2357 cm^{-1} and 2924 cm^{-1} in case of β-CD/HAp composite may be attributed to the coordinative interactions between -OH of β-CD and Ca^{2+} of HAp that becomes the basis of nucleation and growth point of apatite crystals.[15] Furthermore, the less intense peaks observed at about 1490 cm^{-1} and 1456 cm^{-1} should be attributed to the absorption bands of CO_3^{2-}, indicating the presence of carbonate ions. The intense peaks located at 1031 cm^{-1} and 578 cm^{-1} in case of β-CD/HAp composite can be attributed to the PO_4^{3-}.[16] Then, intensive absorption band in 858 cm^{-1} corresponds to a characteristic band to HPO_4^{2-}. The appearance of these new peaks in case of β-CD/HAp composite confirms that the strong interaction had taken place effectively between β-Cyclodextrin and HAp.

Thermo gravimetric analysis (TGA)

Thermogravimetric analysis is a method of thermal analysis which deals with the measurement of changes in physical and chemical properties of materials as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss).[17] The TGA and DTA curves of β-CD/HAp composite has shown in Fig.3 and Fig.4.

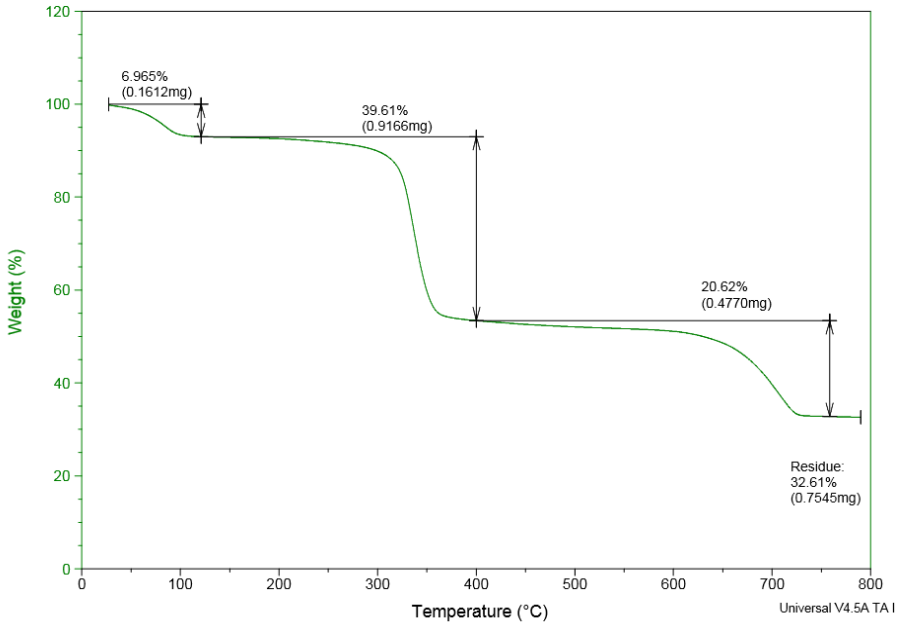

Figure-3: TGA thermogram of β-CD/HAp composite.

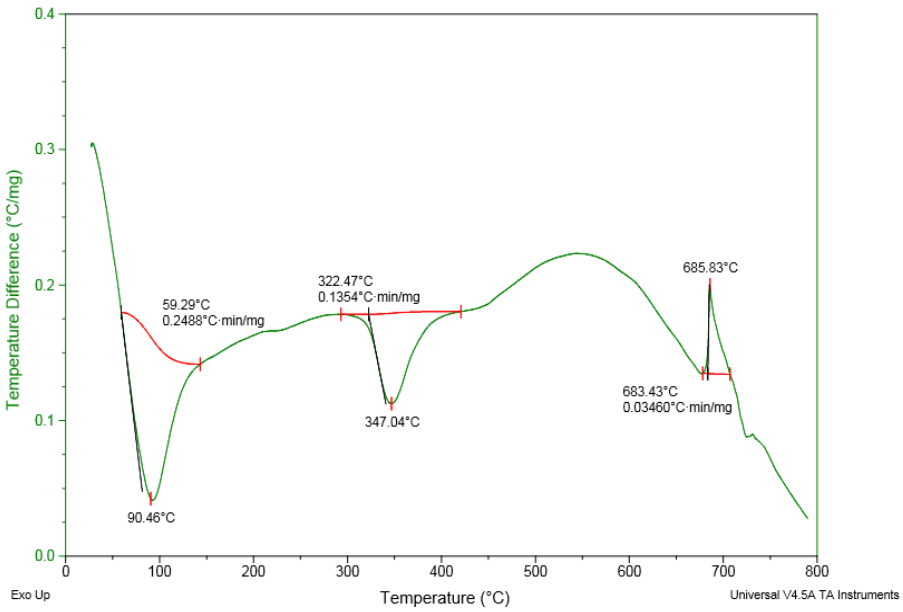

Figure-4: DTA Curve of β-CD/HAp composite.

TGA thermogram of prepared β-CD/HAp composite (Fig. 3) showed the various stages of decomposition. The weight loss occurred below 100 °C can be assigned for desorption of adsorbed water molecules and weight loss occurred at this temperature is very less (6.9%), in that stage decomposition of the composite has taken place. The TGA graph of β-CD/HAp composite involves two step weight loss in the range of 300-400 °C and 600-700 °C in consistency with DTA curve with total weight loss of 68% at the end of the experiment, which indicated that HAp raised the thermal stability of β-CD reflecting the interaction between β-CD and HAp. The higher decomposition temperature 322 °C in the first step involves the breaking of -OH group present in the β-CD which requires higher temperature due to the presence of interaction between the hydroxyl groups present in the cyclic structure.[18] The second step involves that decomposition temperature 685°C which is due to the incorporation of HAp further improved the thermal stability of β-CD/HAp composite which may reasonably be explained in terms of additional OH groups leading to increased hydrogen bonding between the components β-CD and Hap.[19] The TGA analysis confirms the presence of β-CD and HAp and their decomposition temperatures and it was concluded to be thermally more stable due to the higher decomposition temperature.

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