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SYNTHESIS AND CHARACTERIZATION OF HYDROXYAPATITE WITH TAMARIND KERNEL POWDER (BIO-POLYMER) FOR BIOMEDICAL APPLICATIONS

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ABSTRACT

Hydroxyapatite which composes inorganic phase is one of the biomaterials for artificial bone and reconstruction of broken or disordered bones. It has great biocompatibility. Polysaccharide based biomaterials are an emerging class in several biomedical field such as tissue regeneration, particularly in drug delivery device. Tamarind kernel powder is used as a source of carbohydrate for the adhesive or binding agent in many of the pharmaceutical products. There fore synthesis of Nano hydroxyapatite with tamarind kernel powder (nHAp/TKP) has been carried out to study its characterization in biomedical application. Chemical structures and functional groups of the synthesized composite have been examined by the Fourier transform infrared technique and x-ray diffraction. Composite morphology is determined using Transmission electron microscopy. Thermal stability was done by thermo gravimetric analysis(TGA). Crystallite size, fraction of crystallinity, surface area, microstrain, dislocation density are calculated using XRD data. The results confirms the presence of the nano particle in the composite. Nanosized composite have more number of properties such as higher specific surface area, improved strength , hardness and thermal stability. The lattice parameter and unit cell volume are matched with JCPDS card no.09-0432.

KEYWORDS: Hydroxyapatite, TKP, XRD, FTIR, TGA..

INTRODUCTION

Polysaccharide are a structurally diverse group of biological macromolecules of widespread occurrence in nature. They are composed of repetitive structural features that are polymers of monosaccharide residues joined to each other by glycosidic linkages. In this way they differ structurally from proteins and nucleic acids. Polysaccharides present the highest capacity for carrying biological information since they have the greatest potential for structural variability.[1] Polysaccharides are relatively complex carbohydrates. They provide good mechanical properties for application as fibers, films, adhesives, rheology modifiers, hydrogels, emulsifiers and drug delivery agents[2]. Tamarind kernel powder (TKP), Jellose is the major polysaccharide present in the kernel powder (60%). This is composed of D-glucose, D-xylose, D-galactose and L- arabinose in the ration 8:4:2:1. Tamarind xyloglucan has a (1□4)-β-D-glucan backbone that is partially substituted at the O-6 position of its glucopyranosyl residus with α-Dxylopyranose some of the xylose residues are β-D-galactosylated at O-2[3]. Jellose can be used as a excellent substitute for fruit pectins and it can be used as an effective remedy against diarrhea, dysentery and colitis. Other uses are in the cosmetics, pharmaceutical and insecticidal preparations.[4]

Hydroxyapatite has attracted much interest as a biomaterial for use in prosthetic application due to its similarity in crystallography and chemical composition to that of human hard tissue.[5] Its outstanding properties like biocompatibility, bioactivity, osteo conductivity, non-toxicity and non-inflammatory nature.[6] This bio ceramic has got a variety of applications which include: bone tissue engineering, restoration of periodontal defects,[7] edentulous ridge augmentation,[8] orthopedic and dental implant coating.[9] HAp is widely used for hard tissues repair, as a result, this inorganic phosphate has been studied extensively for medical application in the form of powders, composite or even coatings.[10]Although many routes have been explored for synthesis of hydroxyapatite powders such as hydrothermal methods[11,12], sol-gel process[13], reaction in solid state, microwave processing[14], the chemical precipitation route has proven to be popular because of its versatile and economical advantages, and thus has been extensively reported.[15]

The objective of the present work is to synthesis of nano HAp, HAp/TKP powder from modified wet chemical method at room temperature and carried out its characterization.

Synthesis of nano HAp

Nano HAp was synthesized by following a modified wet chemical method. At room temperature. $(Ca(OH)_2$ was first dissolved in a 100 ml volume of an ethanol-water mixture and was stirred for 3 hours. A solution of $(NH_4)H_2PO_4$ was dissolved in 100ml volume of water and then added to the $(Ca(OH)_2$ solution over a period of 24 hours.

Synthesis of TKP/HAp nano composites

The Hydroxyapatite/ Tamarind Kernel Powder were coded as HAp/TKP. Water was used as the solvent to prepare the polymer solution. TKP was dissolved by using magnetic stirrer for 3 hours. The suitable amount of HAp was added the solution. The homogeneously mixed solution is immediately taken heat process, the sample were dried.

FTIR

The Fourier transform infrared (FT-IR) Spectra were recorded on a Perkin elemer Spectrometer, in the range of 400 cm^{-1} to 4000 cm^{-1}. The spectra shows the absorbance bands at 3425.82 cm^{-1} and 3609.39 cm^{-1}correspond to the presence of hydroxyl group in the composite. Absorbance bands at 1669.95 cm^{-1},1600.51 cm^{-1} and 1414.83 cm^{-1},1417.25 cm^{-1} reveals the presence of C=O and CH_2 asymmetric bending and bands at 1036.51 cm^{-1},1035.53 cm^{-1} and 564.98 cm^{-1},565.74 cm^{-1} shows the presence of phosphate group in the HAp/TKP nano composite. In an isolated state the compound of HAp shows a broad band at 3425 cm^{-1}, indicates the presence of intermolecular H-bonding. The spectrum of HAp/TKP. The sharp peak occurs in the region of 3609 cm^{-1} indicates the presence of free O-H, and there is no inter molecular H-bonding between the molecules of HAp.

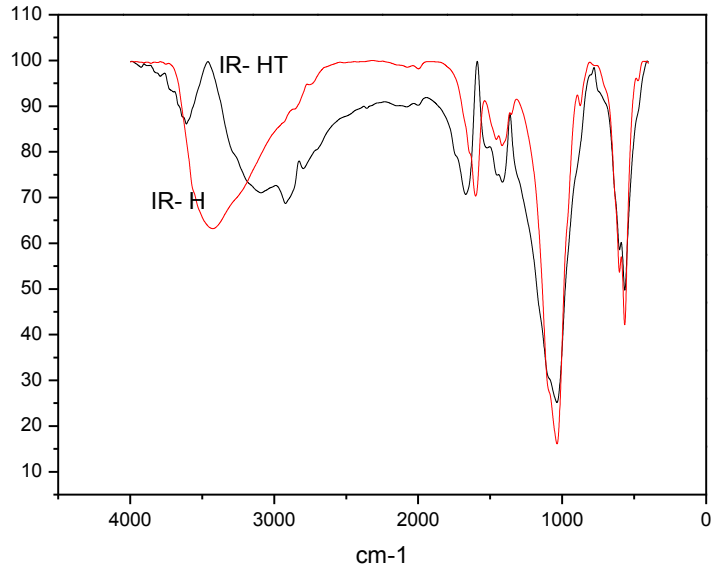

Fig : 1.FTIR spectrum for Hap, Hap/TKP composite.

XRD

The XRD patterns of nHAp and nHAp/TKP composite were taken. The pattern indicates the presence of crystalline nature of HAp. The reflection planes corresponding to the characteristic XRD spectral peaks of pure nHAp and nHAp/TKP are shown in figure 2. The observed diffraction peaks were identified by standard joint committee on powder Diffraction standard (jcpds) file no 09-0432. The diffraction peaks , particularly in the planes 002, 300, 202 are high narrow implying that the HAp crystallizes well.

The intensity diffraction peaks namely.(002),(300),(202) were chosen to estimate the following parameters. The crystallite size of the pure HAp and HAp/TKP composite was calculated using Scherrer's formula[16]. The Fraction crystallinity, Specific surface Area[16], Micro strain, Dislocation density[17] of HAp and HAp/TKP composite was

calculated in Table 1 and 2. The lattice parameter and unit cell volume[18] were and compared with JCPDS card data in Table 3.

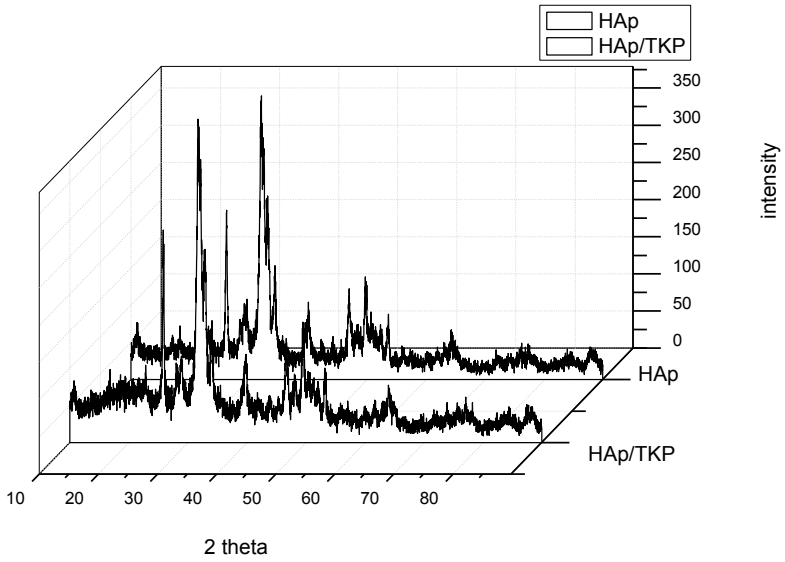

Fig: 2 XRD pattern for HAp and nHAp/ TKP composite

Peak	2 theta	FWHM	Crystallite size (nm)	Fraction of crystallinity	Surface area	Micro strain	Dislocation density
0 0 2	26.107	0.315	26	0.4422	420.35	0.0767	0.0014
3 0 0	33.09	0.50	16.20	0.1105	656.54	0.1198	0.0038
2 0 2	34.32	0.52	16.30	0.0983	680.59	0.1242	0.0037

Table 1: pure HAp spectrum data

Peak	2 theta	FWHM	Crysallite size (nm)	Fraction of crystallinity	Surface area	Micro strain	Dislocation density
0 0 2	25.793	0.28	30	0.6297	373.88	0.0682	0.0011
3 0 0	32.79	0.46	18.23	0.1420	604.53	0.1103	0.0030
2 0 2	33.98	0.54	16.11	0.8870	707.45	0.1291	0.0038

Table 2; nano HAp/TKP spectrum datas

Sample	Lattice parameter		Unit cell volume
	A	C	
Jcpds 09-0432	9.4180	6.8840	528.80
HAp	9.4515	6.8377	528.35
HAp/TKP	9.4472	6.8988	532.59

Table 3: lattice parameter and unit cell volume compared with JCPDS 09-0432.

TEM

Transmission electron microscope experiments were performed on a Tecnai T20 electron microscope with an acceleration voltage of 200kV. TEM Image shows that particles exhibits nano rod like type morphology. 100nm TEM image clearly exhibit HAp and TKP. These nanocomposite had good dispersive properties and displayed a relatively uniform morphology. The inorganic phase was further identified nHAp/TKP from the SAED pattern of the composites, where in the polycrystalline rings are detected. This is agreed with XRD result and confirmed the Nano size components of HAp/TKP nano composite.

THERMAL ANALYSIS

In order to evaluate the thermal stability of the composite materials the TGA analysis was performed in the range in the range 0 to 800˚C for the weight rate of HAp/TKP sample(Figure:). As can be seen from the TGA curve, the

initial weight loss from 90˚C to 190˚C is about 6% loss, which may be due to the evaporation of surface adsorbed water molecules and hydroxylation of HAp. The main weight loss is observed about 24% from 270 to 500˚C. the weight loss is possibly due to decomposing of organic phase in the composite material when the temperature was higher than 600˚C, there was no obviously weight loss for the composite material exhibit good thermal stability and meet the requirements of tissue engineering materials.

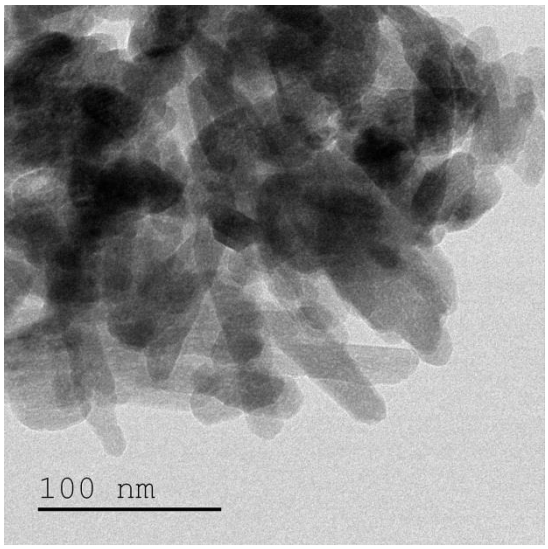

Fig:3a TEM image for nHAp.

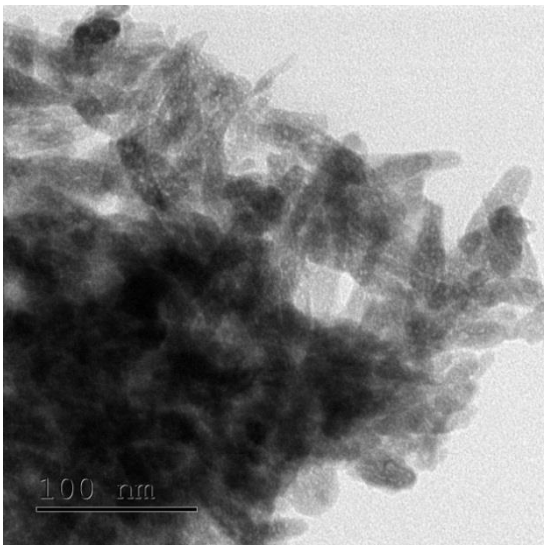

Fig:3b TEM image for nHAp/TKP

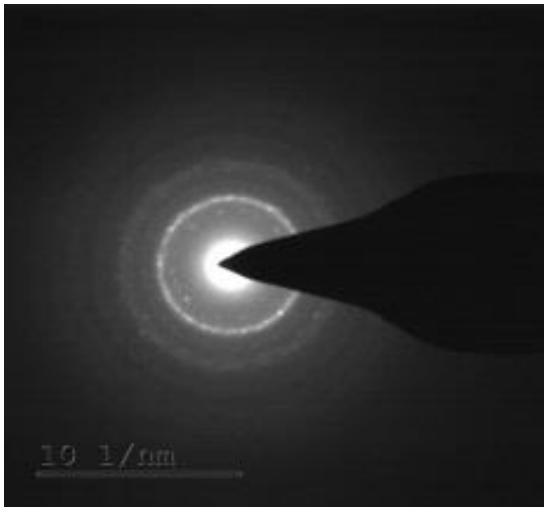

Fig : 3c SAED for nHAp/TKP composite.

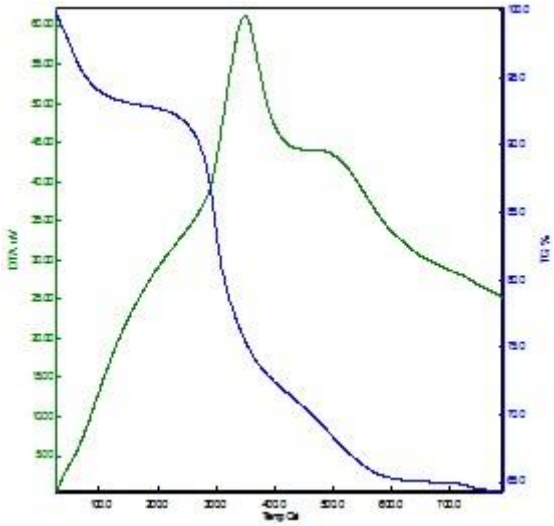

Fig: 4 Thermal analysis for nHAp/TKP composite

CONCLUSION

Hydroxyapatite with Tamarin kernel powder nano composite was successfuly synthesized by modified wet chemical method. TEM and XRD results exhibits the nano level composits of NHAp/TKP. FTIR spectrum confirm the formation of Hydroxyapatite. TGA studies inticate a therml stability of Nano composite. SAED pattern confirm the crystalline nature of the composite. Lattice parameters and cell volume are matched with JCPD card no 04-0932. Crystallite size, Fraction of crystallinity, surface area, micro strain and dislocation density are calculated by using XRD data. Particle size decreased surface area is increased. The result are agreed with previous reports. The synthezied nanocomposite could be utilized for the pharmaceuticals and biomedical applications.

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