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Synthesis Characterization and Anti Microbial Activity of Polyacrylic Acid Kaaolin Hydrogel using Different Initiator

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Abstract: *The swelling kinetics of a series of carboxy methyl chitosan-g-poly(acrylic acid) hydrogels pretreated under acidic buffer media has been studied and Sigmoidal swelling curves are found to be exhibited in the buffer solutions of pH 6.0–7.4 and this phenomenon may be attributed to the disruption of a cooperative physical cross-linking (i.e. the hydrogen bonding and the ionic cross-linking) on the networks, which was proved by the change of FT-IR spectra of hydrogels during swelling. The buffer pH, the pretreating pH and the composition of hydrogels have an obvious influence on the sigmoidal effect. The profile of drug release at pH 7.0 from the hydrogel which was prepared in pH 2.2 buffer containing 5-aminosalicylic acid (5-ASA) exhibitsa sigmoidal release curve, namely, an initial slow release followed by a burst release. The swelling kinetics shows the potential in the design of the colon-specific drug delivery system.*

Keywords: Sigmoidal

I. INTRODUCTION

Hydrogels are extremely hydrated polymer gels with macromolecular three dimensional networks that swells but do not dissolve in water. Hydrogels are superabsorbent and they contain over 99% water in natural or in synthetic polymers. Hydrogels also possess a degree of flexibility very similar to natural tissue, due to their significant water content and they are cross- linked polymer networks that absorb substantial amounts of aqueous solutions. They havewide range of applications in agriculture, bio and chemical sensors, in water purification and removal of metal ions, oil spill removal, DNA separation, largely in the biomedical field such as contact lens, wound dressing, tissue engineering, drug delivery, and in food packaging etc.

II. EXPERIMENTAL TECHNIQUES

2.1 Preparation of PAA-Kaolin Hydrogel

The monomer acrylic acid (20ml), 0.4g of kaolin (clay) and 0.2g of initiator potassium per sulphate was mixed thoroughly using magnetic stirrer and the mixture was kept in an oil bathand maintained a temperature of 150˚C for 30 minutes with constant stirring. Poly acrylic acid - kaolin hydrogel was formed by free radical solution polymerization and the formed hydrogel waspreserved in desiccators for further studies.

The same procedure was adapted to synthesis PAA-kaolin hydrogel using ammonium persulphate as an initiator.

2.2 Materials and Methods

2.2.1 Chemical Used

The chemicals, Acrylic acid, Kaolin , Chitosan, potassium persulphate, ammonium persulphate, Sodium hydroxide were purchased as an Analar grade from Mumbai and used as received.

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III. RESULTS AND DISCUSSION

Characterization of synthesized PAA-kaolin hydrogel gives the more information about the type and nature of the hydrogels formed and these informations are used to predict the formation of the product, structure, nature of the product, size, thermal activity etc. The synthasised hydrogels were characterized using FTIR spectroscopy, SEM, TGA-DTA and DSC.

FT-IR SPECTRA OF PAA-KAOLIN HYDROGEL

The FT-IR spectra of kaolin is given in figure 1. It shows the absorption band at 3665– 3926 cm^{-1} due to the presence of –OH group present in the silicate structure and this hydrogen bond is intermolecular hydrogen bonding and other bands at 830 and 949 cm^{-1} are due to the presence of SiO stretching present in the kaolin surface

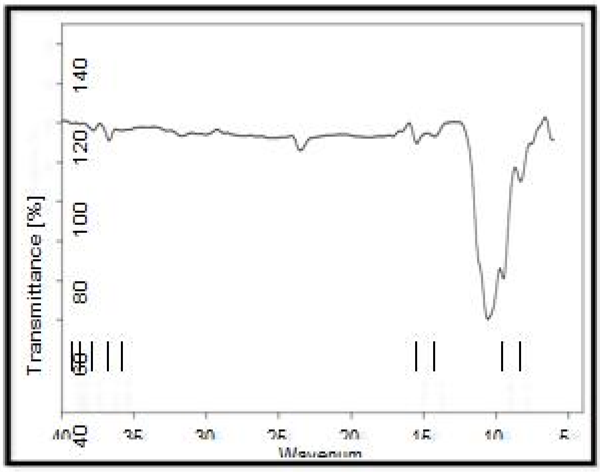
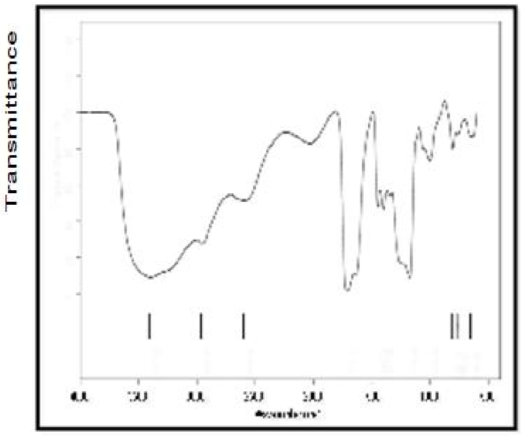


FIGURE 1 FT-IR SPECTRA OF KAOLIN

The FT-IR spectra of PAA-kaolin synthesized using potassium persulphate as an initiator is given in the fig 2 The band at 1714 cm^{-1} show the presence of ester group present in the PAA-kaolin fig 1 and this confirms the formation PAA-kaolin hydrogel. The ester groups are formed by the reaction between the acid groups present in poly acrylicacid and carboxyl groups present in kaolin clay material.



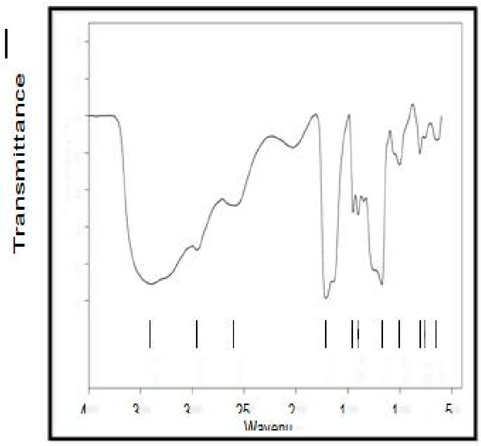


FIG 2 spectra of PAA-kaolin synthesized using Potassium persulphate as an initiator

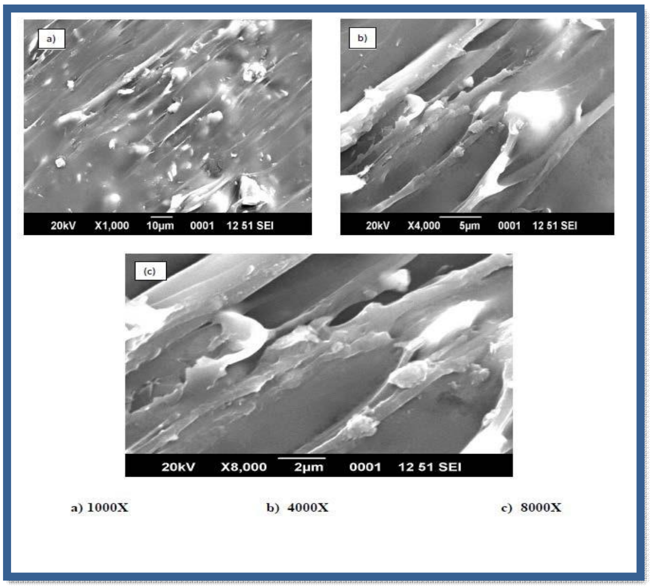
FIG 3 spectra of PAA-kaolin synthesized using Pammonium persulphate as an initiator

The FT-IR spectra of PAA-kaolin synthesized using ammonium persulphate as an initiator are given in the figure 2. The band appeared at 1711 cm^{-1} show the presence ofester group present in the PAA-kaolin (fig 2) and this confirms the formation PAA-kaolin hydrogel The ester groups are formed by the reaction between the acid groups present in poly acrylic acid and carboxyl groups present in kaolin clay material.

TABLE. 1 Comparative FT-IR Spectral Data of PAA-KaolinHydrogels Using Different Initiator

Band assignment		**$K_2S_2O_8$ (cm^{-1})**	**$(NH_4)_2S_2O_8$ (cm^{-1})**
	Pure kaolin (cm^{-1})	**PAA-kaolin**	**PAA-kaolin**
-OH stretching	3665-3926	3397	3421
SiO stretching	830-949	648-806	624-806
Skeletalvibration in carboxylic stretching	-	-	-
Ester group	-	1714	1711
-CH2 bending in PAA	1406	1448-1401	1443-1402
-CH,-CH2 stretching	-	2969	2960
-CN Stretching	-	-	-
C=O stretching	-	-	-
Combination of COOH and –OH stretching	-	2694-3397	2544-3421
C-O stretching	-	-	-

SCANNING ELECTRON MICROSCOPY



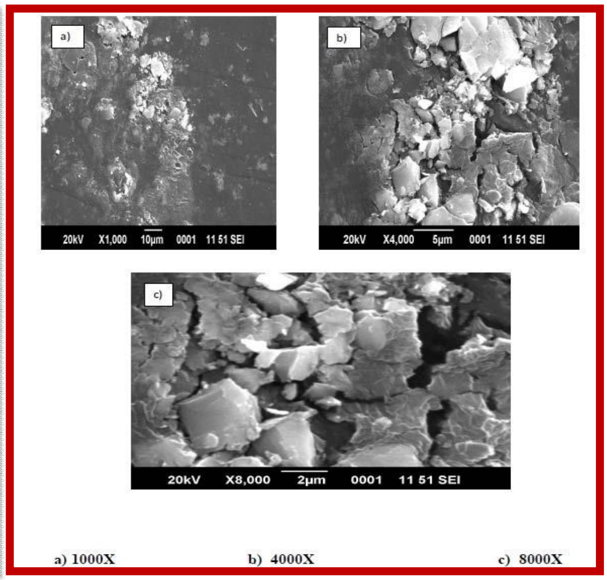
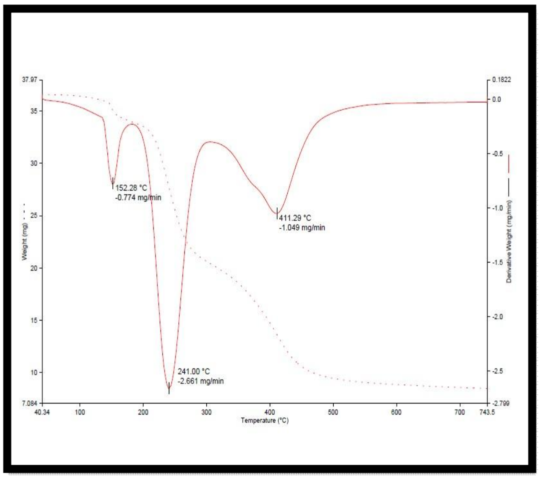


FIG 4 SEM analysis of PAA-kaoline using potassium persulphate as an initiator

FIG 5 SEM analysis of PAA-kaoline using ammonium persulphate as an initiator

The SEM analysis of PAA-kaoline using different oxidants like potassium persulphate and ammonium persulphate are given in figures 4 and 5 with different magnification. The morphology of PAA-kaolin using potassium persulphate is found to have soft porous structure as it is evident from the figure 4.The SEM analysis recorded for PAA- kaolin hydrogel synthesized using ammonium persulphate as the initiator are is found to have a flakes like structure which contains voids (Fig 4).

THERMAL ANALYSIS OF HYDROGELS



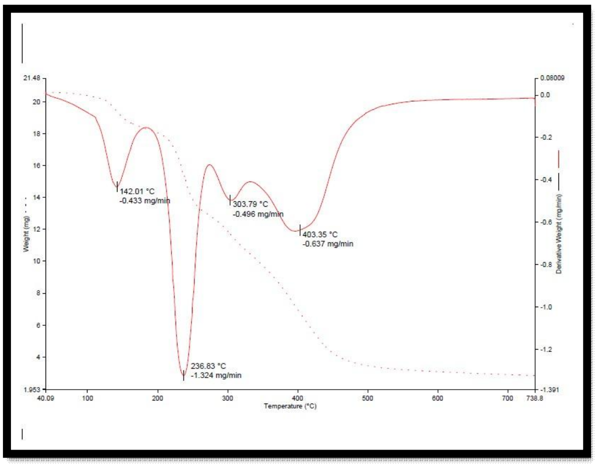
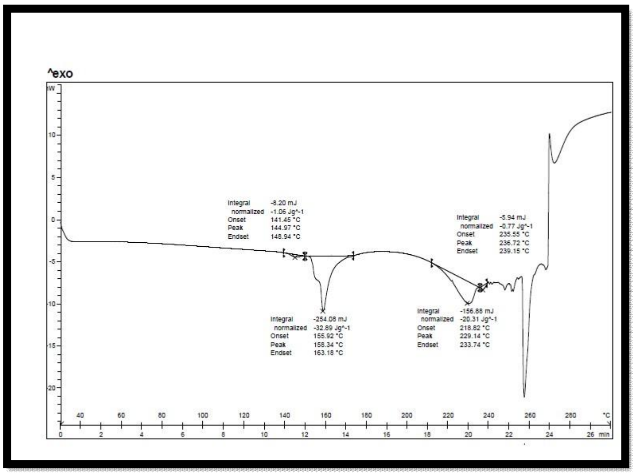


FIGURE. 6 TGA and DTA of PAA-kaolin hydrogel using potassium persulphate as an initiator
FIGURE.7 TGA and DTA of PAA-kaolin hydrogel using ammonium persulphate as an initiator



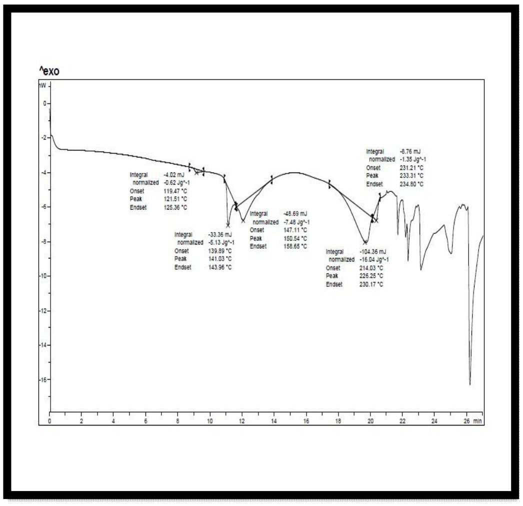


FIGURE.8.1DSC curve of PAA-kaolin hydrogel using potassium persulphate as a initiator
FIGURE.9 DSC curve of PAA-kaolin hydrogel using ammonium persulphate as a initiator

TABLE 2: Comparative TGA/DTA Dates of PAA-Kaolin Hydrogel Using Different Initiator

No.of transition	$K_2S_2O_8$ (˚C)		$(NH_4)_2S_2O_8$ (˚C)	
	PAA-Kaolin(˚C)	Weight loss(%)	PAA-Kaolin(˚C)	Weight loss(%)
1	102-180	95	110-190	90
2	180-300	55	190-290	65
3	300-580	30	290-340	58
4	-	-	340-510	20
5	-	-	-	-
6	-	-	-	-

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ANTIMICROBIAL STUDIES OF HYDROGELS

PAA-kaolin hydrogels are synthesized by using different initiator like potassium persulphate and ammonium persulphate. The synthesized hydrogels were investigated for Antimicrobial study by using different bacteria and fungi.

ANTIBACTERIAL ACTIVITIES OF PAA-KAOLIN HYDROGELS

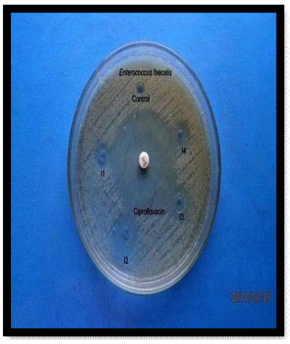
The antimicrobial activities of synthesized hydrogels were studied using well cut diffusion method against two gram positive bacteria like *Enterococcous faeslis, Staphylococcus aureus* and one gram negative bacteria like *Proteus mirabils.* The antibacterial activities were carried out at the hydrogel concentration of 0.5µg/ml and Ciprofloxacin was used as the standard[99], and their result are given in the table 2. The results from the table value shows that the inhibition takes place depending on the type of species used and the nature of the hydrogel.

The zones of inhibitions were measured including the well in millimeter. The antibacterial can be classified into the following types:

>12mm zone of inhibition-high sensitive, 9-12mm zone of inhibition-moderate sensitive, 6-9mmzone of inhibition-less sensitive and < 6mm zone of inhibition-bacteria resistant[100].

The antibacterial activities of PAA-kaolin hydrogels synthesized using potassium persulphate as an initiator is given in the (Table.3). From the result it is evident that the zones of inhibition vary depending on the type of bacteria and the nature of the hydrogel. The antibacterial activities of PAA-kaolin hydrogels studied against *Enterococcous faeslis* and *Staphylococcus aureus* are found to have 9mm and 8mm and zones of inhibition gives better result.The zone of inhibition for PAA-kaolin hydrogels tested against gram negative bacteria like*Proteus mirabilis* exhibited 6 mm and 5mm zone of inhibition and this confirms that the hydrogels are not so active against studied gram negative bacteria. The antibacterial activity of PAA-kaolin hydrogel synthesized using ammonium persulphate as a initiator are given in table.3 and the values shows that the zones of inhibition of PAA-kaolin hydrogel tested against gram positive bacteria *Enterococcous faeslis* shows highest inhibition efficiency with the zones of inhibition 16mm and the values are closer to the standarded (24mm) and *Staphylococcus aureus* shows the activity of 6mm. The comparative antibacterial activities are given in figure 9 and the sample plates are given in figures. 9,a) ***Enterococcus faecalis b) Staphylococcus aureus c) Proteus mirabilis.***

SAMPLE P SAMPLE PLATES OF ANTIBACTERIAL ACTIVITIES OF PAA- KAOLIN HYDROGELS



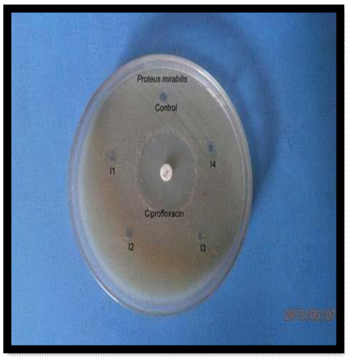


TABLE 3: Ntibacterial Activities of PAA-Kaolin Hydrogel

Initiator	$K_2S_2O_8$(mm)	$(NH_4)_2S_2O_8$ (mm)	Ciprofloxacin (mm)
Microorganism	**PAA-Kaolin**	**PAA-kaolin**	
Enterococcus faecalis	9	16	24
Staphylococcus aureus	8	6	30
Proteus mirabilis	6	4	30

ANTIFUNGAL ACTIVITIES OF PAA-KAOLIN HYDROGELS

The antifungal activities of synthesized hydrogels were studied using well cut diffusionmethod against three fungi like *Aspergillus niger, Mucor sps* and *Aspergillus flavus* and the results are given in table 4. The antifungal activity were carried out with the hydrogel concentration of 0.5µg/ml and Amphotericin–B was used as a standard[101]. Depending on the measured values with the zone of inhibitions, the antifungal study can be classified into the following catogory: >12mm zone of inhibition-high sensitive, 9-12mm zone of inhibition- moderate sensitive, 6-9mm zone of inhibition-less sensitive and < 6mm zone of inhibition-fungi resistant[102].

The antifungal activities of PAA-kaolin hydrogel synthesized using potassium persulphate and ammonium persulphate as an initiator shows good result. The activity is same as that of the standard and PAA-kaolin synthesized using ammonium persulphate shows the activity if 11mm against *Aspergillus flavus* which is little higher than the standard (10mm). All the other synthesized hydrogels were found to be inactive against other tested fungi. The sample plates of fungi studies are given in fig.,**a) Aspergillus niger b) Mucor sps c) Aspergillus flavus.**

SAMPLE PLATES OF ANTIFUNGAL ACTIVITIES OF PAA- KAOLINHYDROGELS

TABLE 4: Antifungal Activities of PAA-Kaolin Hydrogel

Initiator	$K_2S_2O_8$ (mm)	$(NH_4)_2S_2O_8$ (mm)	Amphotericin-B(mm)
Microorganism	**PAA-Kaolin**	**PAA-kaolin**	
Aspergillus niger		-	24
Mucor sps	-	- 11	30
Aspergillus flavus	-5		10

II. CONCLUSION

The poly acrylic acid and kaolin hydrogels were synthesized using different initiator like potassium persulphate and ammonium persulphate and the PAA-kaolin hydrogel formed by free radical solution polymerization method.

The synthesized PAA-kaolin hydrogels were characterized using FT-IR, SEM, TG-DTA, DSC. The FT-IR spectra recorded shows the formation of hydrogel from the ester linkage whichshows the peak at 1714 cm^{-1} for PAA-kaolin hydrogel.

The SEM analysis gives the information about the morphology of the hydrogel. PAA- kaolin using potassium persulphate as an initiator gives the image of soft porous like structure, and PAA-kaolin synthesized by using ammonium persulphate gives the image of flakes with void like structure.

The TG-DTA analysis of given hydrogels shows different decomposition temperature depending the nature of the hydrogel and the types of initiator used and the DSC analysis gives the information about the glass transition temperature, crystallization temperature and melting temperature of the hydrogel and they changes depending on the nature of the hydrogel and the type of the initiator used.

The antibacterial activity of the PAA-kaolin shows varied activities depending on the nature of the species used and the type of the hydrogel. The antifungal study shows that the PAA-kaolin synthesiszed using ammonium persulphate was found to be highly active against *Staphylicoccus aureus* with the zone of inhibition 11mm and the activity is higher than the standard Amphotericin-B. The synthesized hydrogels can be used in future in different biomedical applications like drug delivery, DNA separation, agriculture, bio and chemical sensors , water purification and removal of metal ions, oil spill removal, contact lens, wound dressing, tissue engineering, and in food packaging. It can also be a prominent candidate for future applications in different fields.