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PREPARATION OF NANOPARTICLES AND ALOEVERA LOADED STARCH/GUM GUAR COMPOSITE FILM

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ABSTRACT

Polymers are used to great extent in pharmaceutical industry for drug delivery because of their surface and bulk properties. They are also used in formulations of drug and devices for drug delivery. Polymeric Drug Delivery systems are used for controlled drug delivery ensuring patient compliance. Metallic Nanoparticles are proven to have antibacterial properties against both gram positive bacteria and gram negative bacteria. Some substances obtained naturally such as Honey, Aloe vera, Tulsi, Turmeric, Garlic, Clove, Nutmeg, etc also contain antibacterial properties. This work demonstrates fabrication of polymeric composite of Gum guar (GG) and Starch (St). Then this polymeric composite was loaded with Aloe vera and Lead (Pb) nanoparticles simultaneously. This polymeric composite was also characterized using several methodologies including X-ray diffraction (XRD), Fourier Transform Infrared spectroscopy (FTIR), Scanning electron microscopy (SEM). Chemical properties were also determined using Swelling and Expansion experiments. Ultraviolet-Visible spectroscopy (UV-Vis) were also carried out for Lead Nanoparticles along with characterization of Aloe vera by Ultraviolet-Visible spectroscopy (UV-Vis). Swelling test using pf solution was also carried out to evaluate swelling parameters. Also, expansion study was done using gelatin.

Keywords: Gum Guar, Starch, Lead Nanoparticles, Aloe vera, Gelatin

INTRODUCTION:

System of Drug Delivery using polymer is defined as formulation used to introduce therapeutic compound in our body. Not only it improves safety but also improves efficacy with control over of place and time of releasing drugs in our body [1]. Polymeric blends combine two or more polymers improved and functionality which can result in great advantages in drug delivery applications [2]. Starch can be thermally processed when a plasticiser like water added to it. When various materials are mixed with starch, their performance changes due to hydrophilicity of starch [3]. Starch is a cheap and abundant material in raw form which is used in biomaterial field [4]. Gum guar is high molecular weight and water soluble compound. It has backbone of linear chain of β -1, 4-linked mannose residues linked with 1,6 mannose every second of galactose residues, which results in formation of short sidebranches. As guar gum contains properties like hydrophilicity and biocompatibility, it is used in field of wound healing as bio-absorbable material [5]. Aloe vera has therapeutic and curative effects due to which it has been used from many centuries [6]. Lead nanoparticles have unique properties and some important uses such as luminescent compound, gas sensing devices, devices for

storage purpose, UV blocking devices and as modifiers, etc. [7].

MATERIALS AND METHODS

MATERIALS:

All chemicals used in this work were of high analytical grade. Potato starch, Gum Guar, Glutaraldehyde (25% aqueous solution), Lead acetate trihydrate and Sodium Hydroxide were all purchased from Loba Chemie Pvt. Ltd. Mumbai (India). Aloe vera Gel was extracted naturally from plant. Distilled water was used in all experiments.

METHODS:

1. Preparation of Gum Guar/Starch composite film:

The films were prepared using solvent casting method. In this process, 3 gm Starch was dissolved in 50ml distilled water and 0.3 gm Gum guar was dissolved in 50 ml distilled water using magnetic stirrer with gentle heating at around 40°C. These two solutions were then mixed with addition of 2 ml glutaraldehyde (cross-linking agent) and stirred until it attains homogenization. The solution was cooled at room temperature to remove air bubbles and then poured into a petri-dish and dried at 40°C in hot air oven for 24 hours. The film was peeled off from the petri-dishes and used for further analysis [8].

Similarly, another two films with different ratios were prepared by taking Starch 1 gm and 2 gm respectively without changing amount of Gum Guar in both compositions.

2. Preparation of Lead nanoparticles:

During this synthesis, 1M Lead acetate aqueous solution was prepared by dissolving 37.9 gm Lead acetate trihydrate in 100 ml distilled water and heated upto 90°C. In different beaker, 19M Sodium hydroxide aqueous solution was prepared by dissolving 38gm NaOH in 50 ml distilled water. Both of this solution were mixed and stirred vigorously. A cloudy solution appears and then turned peach and finally deep orange red on further mixing. At this moment, the reaction was stopped and formation of precipitates was observed. The supernatant was decanted, filtered, washed with distilled water and dried in oven at 90°C. The sample was then removed and lightly crushed using a mortar and pestle [9].

3. Preparation of Pb NPs and Aloe vera loaded St/GG composite films:

The film was prepared using above mentioned method by dissolving 3 gm Starch and 0.3 gm Gum Guar in 50 ml water each with gentle heating and stirring using magnetic stirrer. After this, both the solutions were mixed together by stirring and 1 ml of Glutaraldehyde was mixed. Along with this,

Aloe vera and Lead Nanoparticles with appropriate weighing were mixed in this solution. The blend was poured into a petri dish and dried for 24 hours at 40°C in hot air oven. This film was peeled off to obtain the loaded film.

CHARACTERIZATION:

FTIR: FTIR spectrum of 1000 cm⁻¹ was used to analyze the polymeric composite of Starch-GG using Fourier-transform infrared spectroscopy at Chemistry Research for Development, Parul University, Vadodara. The scan resolution was 4 cm⁻¹ and range of 3500-1000 cm⁻¹.

XRD: Analysis of X-ray diffraction was performed at MS University, Vadodara. The 2θ was ranged from 5° to 60°. The step size was 0.0080° 2θ and scan step time was 8.2550 s. The wavelength of Copper K-α radiation was 1.54060 Å.

SEM: The Scanning electron microscopy analysis was performed at PNP Analytical Solutions, Vadodara to determine the morphological properties of the composite film. The accelerating voltage of electron beam (HV) 5.00 kV was used and a working distance (WD) of 6.6 mm was maintained.

UV-Visible of Aloe vera: The Ultraviolet Visible spectroscopy of Aloe vera was performed at Chemistry Research for

Development, Parul University, Vadodara. The wavelength was set between 200-800 nm.

UV-Visible of Pb-NPs: The UV-Visible analysis performed for the identification of Lead Nano-particles at Chemistry Research for Development, Parul University, Vadodara. The wavelength for this analysis was set between 200-800 nm.

Swelling Study: The study of swelling of composite films was carried out employing physiological fluid (PF). This fluid was made by dissolving 8.307 g sodium chloride (NaCl) in one liter distilled water and 0.367 g Calcium Chloride (CaCl₂) in one liter distilled water. Then, both were mixed to make the PF solution. Now, small portions of all ratio films were cut and weighed separately. The films were immersed into the PF solution and at different time intervals, they were removed, dried using filter paper and weighed. This process was continued several times and at 24 hours.

Expansion Study: The wound dressing film expanding on surface of injury was studied by measuring the diameter change of round films cutting in a 10% solution containing gelatin. In brief, 10 g of gelatin powder in 100 ml preheated distilled water by constant agitating till getting a clear solution. This solution then was poured into a petri-dish and all ratio films of studied diameter were put in solution of

gelatin and the difference in diameter of film was measured and noted till the sample attains a constant diameter. Then the Ratio of Expansion denoted by ER was calculated using the following expression:

$$ER = D_t / D_o$$

where D_t = t time diameter and D_o = diameter at initial time

RESULTS AND DISCUSSION:

FTIR: The FTIR spectra was recorded using FTIR spectrophotometer with KBr. The samples were run in wavelength range of 3500-1000 cm⁻¹. The results are shown below:

1. (1: 0.3) Starch/Gum Guar FTIR analysis:

The broad peaks from 3000 to 3900 cm⁻¹ shows presence vibrational stretching of hydroxyl groups of starch. The sharp peak at 2935 cm⁻¹ shows presence of C-H stretching. Peaks around 1849 cm⁻¹ refers to strongly bound water in starch while the band at 1415 cm⁻¹ shows C-H bending of CH₂. Peak at 1148 cm⁻¹ were attributed to OH bending because of primary and secondary alcohols. Peaks in range of 900 to 1200 cm⁻¹ shows variations in crystallinity. The bands at 936 cm⁻¹ and 1148 cm⁻¹ refers to C-O bond. Peak at 1006 cm⁻¹ corresponds to presence of C-O-H bond. Peaks around 700 cm⁻¹ refers to CH bending vibrations.

2. (2: 0.3) Starch/Gum Guar FTIR analysis:

The broad band having various peaks in range 3000 to 3900 cm⁻¹ shows presence vibrational stretching of hydroxyl groups of starch. The sharp peak at 2921 cm⁻¹ shows presence of C-H stretching. Peaks around 1849 cm⁻¹ refers to strongly bound water in starch. The peaks around 1600 cm⁻¹ shows C-H bending of CH₂. Peak at 1149 cm⁻¹ were attributed to OH bending because of primary and secondary alcohols. Peaks in range of 900 to 1200 cm⁻¹ shows variations in crystallinity. The bands at 993 cm⁻¹, 929 cm⁻¹ and 1149 cm⁻¹ shows C-O bond stretching. Some peaks around 700 cm⁻¹ is because of CH bending (Figure 2).

3. (3: 0.3) Starch/Gum Guar FTIR analysis:

The broad peak around 3000 to 3900 cm⁻¹ shows presence vibrational OH stretching of starch. The sharp peak at 2937 cm⁻¹ shows presence of C-H stretching. Peaks around 1849 cm⁻¹ refers to strongly bound water in starch. The peak at 1415 cm⁻¹ shows C-H bending of CH₂. Peak at 1149 cm⁻¹ were attributed to OH bending because of primary and secondary alcohols. Peaks between of 900 to 1200 cm⁻¹ shows changes in crystal structure of the sample. The bands at 933 cm⁻¹ and 1149 cm⁻¹ shows C-O bond. Peak at

1009 cm⁻¹ shows C-O-H bond. Peaks around 900 shows strong C=C bending (Figure 3).

4. XRD:

X-ray diffraction analysis is a material science technique for determining the material's crystallographic structure. This method shows irradiating the sample with X-ray and measurement of intensities and scattering of X-rays exiting sample. Figure below shows XRD spectrum of Starch/GG composite. As seen in the spectrum, a hump is observed at 17.34 Å ($2\theta = 5.0919$) corresponding to amorphous nature (Figure 4).

5. SEM:

It is an analytical method to produce an enlarged image of the sample using electronic beam. Figure below shows the micrographs of the surface of polymeric film containing Starch and Gum Guar. The size of particles was shown to be between 250-500 nm (Figure 5).

6. UV-Visible of Aloe-Vera:

The qualitative UV-Visible profile of this sample was taken at the wavelength of 200-800 nm wavelength. The absorption peak of aloe vera was determined using UV-Visible spectrophotometer. The absorbance peak of aloe vera was found at 280 nm (Figure 6).

7. UV-Visible of Lead Nanoparticles:

The absorbance of nanoparticles of lead was evaluated using UV-Visible

spectrophotometer. Pb-NPs were dissolved in a solvent and UV analysis was taken. The absorption peak was seen at 258 nm (Figure 7).

8. Swelling Study

Swelling study was performed according to the process mentioned in characterization section above (Table 1, Figure 8). The ratio of swelling denoted by SR was determined by this formula:

$SR = (Mt-Mo)/Mo \text{ g/g}$ where Mo =mass at initial and Mt = mass at interval t.

9. Expansion Study:

Expansion study is carried out to determine the expanding diameter of the polymeric composite (Table 2, Figure 9). The ratio of expansion denoted using ER was determined by the formula:

$ER= Dt/Do$ where Dt = diameter measured at time t and Do =diameter at initial

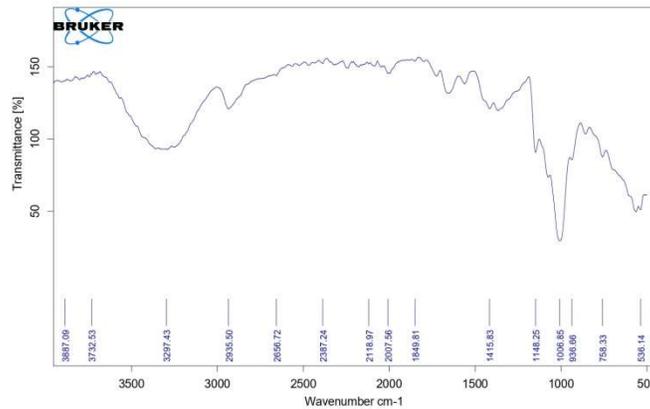


Figure 1: (1: 0.3) Starch/Gum Guar FTIR spectra

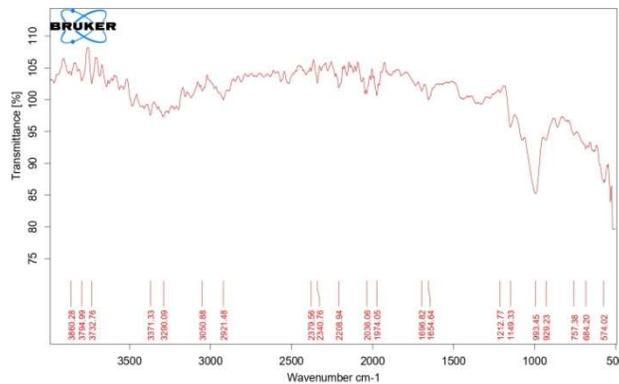


Figure 2: (2: 0.3) Starch/Gum Guar FTIR spectra

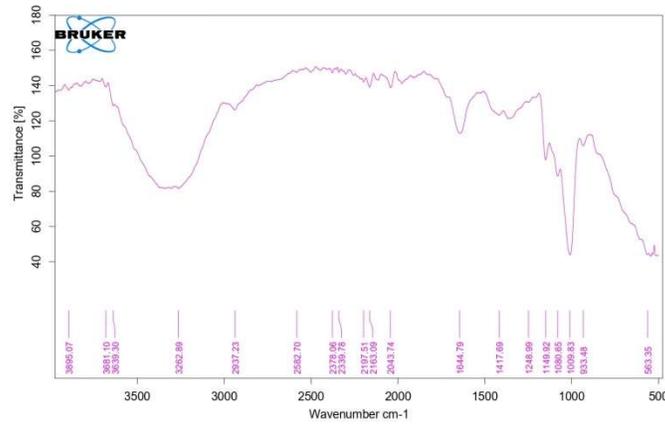


Figure 3: (3: 0.3) Starch/Gum Guar FTIR spectra

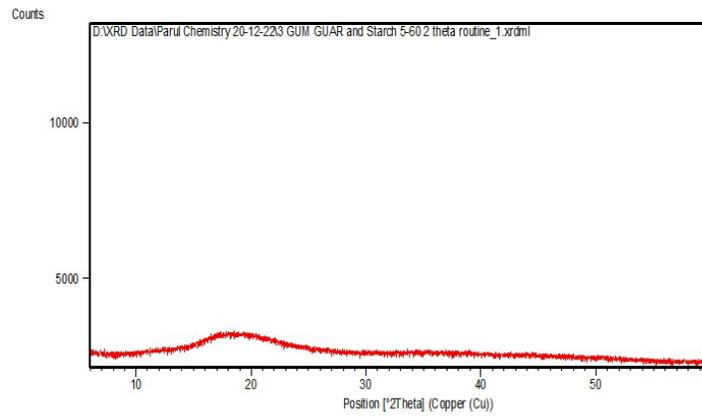


Figure 4: XRD spectra of Starch/Gum Guar composite film

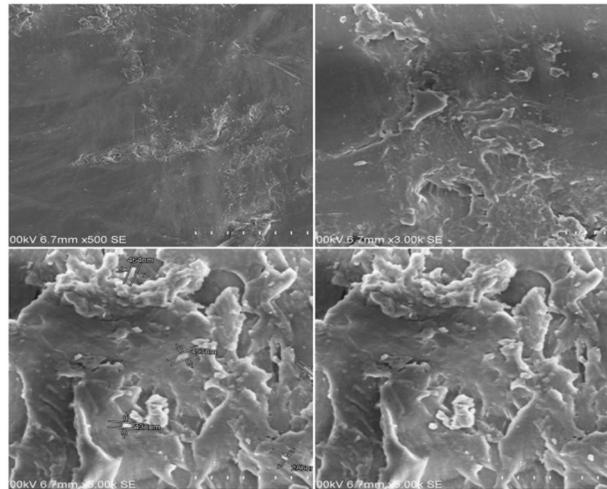


Figure 5: SEM of Starch/Gum Guar

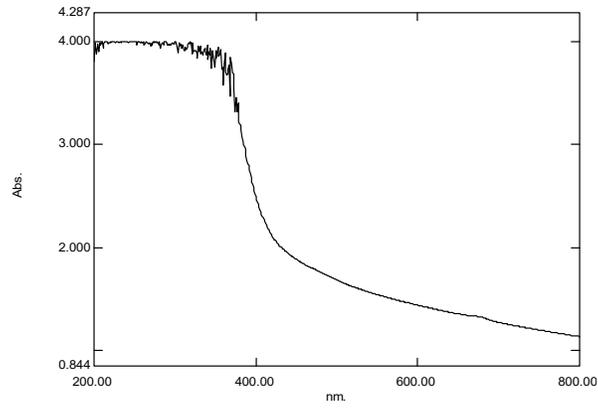


Figure 6: UV-Visible spectra of Aloe vera

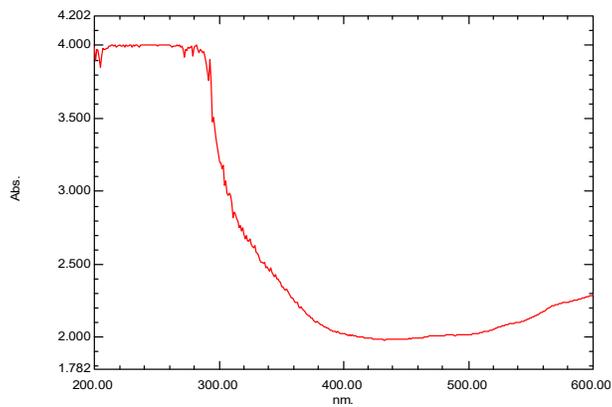


Figure 7: UV-Visible spectra of lead nanoparticles

Table 1: Swelling ratio

Time	Swelling Ratio (g/g)		
	Starch:GG (1:0.3)	Starch:GG (2:0.3)	Starch:GG (3:0.3)
0 min	0	0	0
30 min	1.218	1.852	0.54
60 min	1.96	2.94	1.01
120 min	2.02	3.00	1.10
180 min	2.19	3.04	1.13
24 hours	4.37	5.11	4.40

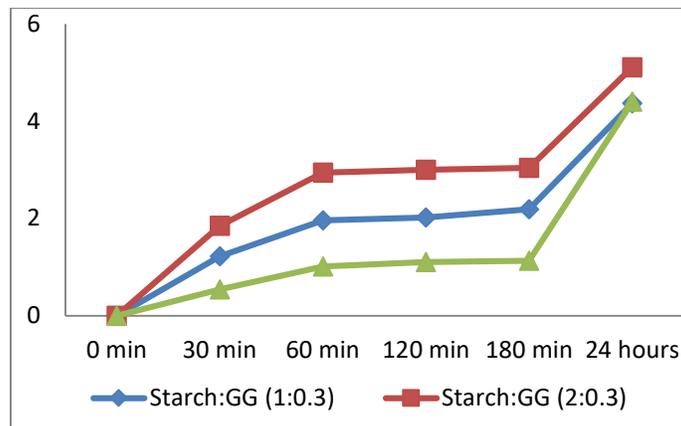


Figure 8: Swelling graph

Table 2: Expansion ratio

Time	Expansion ratio Dt/Do		
	Starch:GG (1:0.3)	Starch:GG (2:0.3)	Starch:GG (3:0.3)
0 min	1	1	1
15 min	1.03	1.04	1.04
30 min	1.07	1.08	1.08
45 min	1.15	1.17	1.16
60 min	1.15	1.17	1.17

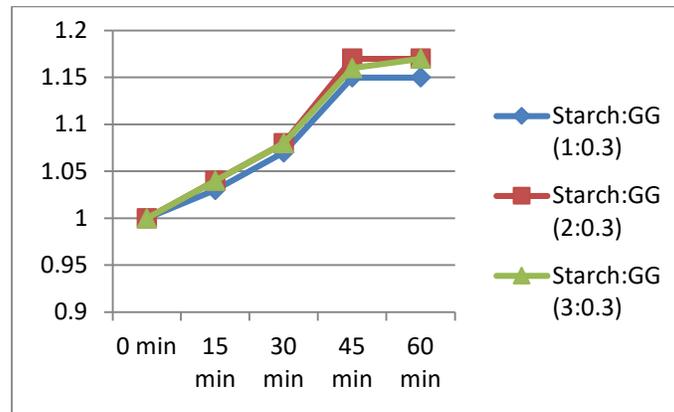


Figure 9: Expansion graph

CONCLUSION:

This paper demonstrates the fabrication of Starch/Gum guar composite using solvent casting method. This polymeric composite contains absorption properties as shown by swelling study and expansion study. Furthermore, the shape, functional groups and structure were found out by UV-Visible spectroscopy, FTIR, XRD and surface morphology was determined using SEM analysis. This paper also contains the preparation of Aloe vera and Lead nanoparticles incorporated polymeric composite of Starch and Gum guar.

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