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Guar gum (GG) / Methylcellulose (MC) Blends and their Composites with Maghemite Nanoparticles

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Abstract: ***Miscibility of polymers decides the applicability of the resulting blends and immiscible polymer blends can be made compatible with suitable compatibilizer. Miscibility of blends of guar gum (GG) and methyl cellulose (MC) in common solvent water were studied by refractive index, density, ultrasonic velocity, and dilute solution viscometry methods at 30°C and 40°C. Based on these measurements, it is found that the polymer blend of GG/MC is immiscible. The effect of compatibilization with 0.02 wt% maghemite nanoparticle was studied in aqueous solution and found that 10/90 GG/MC blend is miscible in the presence of maghemite. Solution cast technique was used to prepare thin films of GG/MC blend – maghemite composite and characterized by scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR) method.***

Keywords: **guar gum, maghemite, miscibility, methylcellulose, polymer blends.**

I. INTRODUCTION

Polymer blends are tailor-made materials for specific applications. The researches on polymer blends have gained considerable interest due to the reduction in the cost of production, improved processability, and improved properties [1]. The blending of polymers is significant to obtain a variety of physical and chemical properties from the constituent polymers [2]. The blend property depends on the polymer-polymer miscibility. Based on the miscibility, polymer blends are classified as miscible, semi-miscible, and immiscible blends [3]. Extensive studies on the miscibility of polymer blends by different techniques have been reported earlier by different researchers [4-8]. The miscibility of polymer blends is studied and confirmed by using density, refractive index, ultrasonic interferometry, viscometry, scanning electron microscopy (SEM) and Fourier transform infrared spectroscopic (FTIR) methods [9-17].

Guar gum (GG) is an extremely hydrophilic natural polymer with high viscosity. This property has allowed GG to be commercialized in fields such as textiles, foods, cosmetics, pharmaceuticals and oil recovery and drilling [13, 15]. Methylcellulose is a semi-synthetic water-soluble polymer derived from cellulose and is used as a thickener in the food industry, and as a matrix for the controlled release of drugs in the pharmaceutical industry [18].

Polymer blends of GG and MC will be having potential applications in drug release formulations. The miscibility can be enhanced by the incorporation of suitable compatible agents [11]. Maghemite is a biocompatible magnetic nanoparticle having potential applications in the biomedical field [19-22]. In this work miscibility of guar gum and methylcellulose at different compositions were studied in solution state at 30ºC and 40ºC by refractive index, density, ultrasonic velocity, and viscosity measurement techniques in aqueous solution and by SEM and FTIR in solid-state. The effect of biocompatible maghemite nanoparticles (Mag NPs) on the miscibility of GG/MC blends was studied in aqueous solution by density, ultrasonic velocity, dilute solution viscometry in aqueous solution and in the solid-state by SEM, and FTIR.

II. EXPERIMENTAL PROCEDURE

The polymers employed in the present study are guar gum (GG), methylcellulose (MC), and the nanoparticle used is maghemite. All were purchased from Merck, India. Schematic representation of guar gum and methylcellulose are shown in Fig. 1, and 2, respectively.

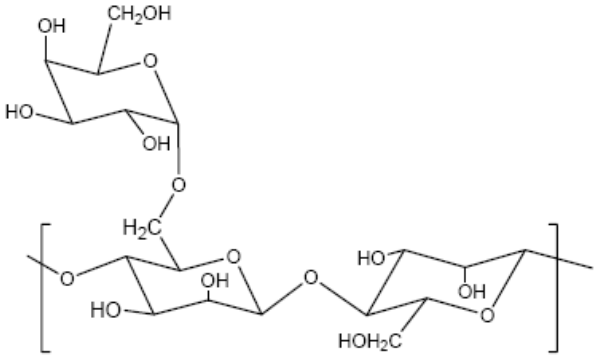

Fig. 1: Schematic representation of guar gum

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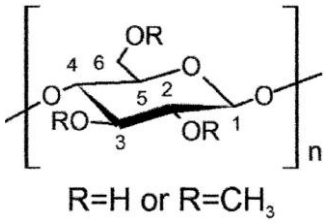

Fig. 2: Schematic representation of methylcellulose

0.01, 0.02, 0.03, 0.04, and 0.05 wt% maghemite nanocomposites were prepared with guar gum (0.5% w/v) and with methylcellulose (0.5% w/v) separately in distilled water as solvent. The mixtures were mechanically stirred for 6 hours in a polymer mixer and were further kept for ultrasonication for 4 hours to ensure uniform mixing. The prepared samples were taken in test tubes and kept in contact with a strong magnet for 30 minutes to check the stability of the solution. All the maghemite-MC composite solutions showed stability towards the magnetic field, whereas the solutions of guar gum containing 0.05, 0.04, and 0.03 wt% of maghemite, the maghemite got settled along the sides of the test tube attracted by the strong magnet. Hence the experiments were continued with 0.02 wt% of maghemite.

Blends of GG/MC of different compositions (10/90, 30/70, 50/50, 70/30, and 90/10) were prepared in distilled water. The refractive index, density, and ultrasonic velocity of the GG, MC, GG/MC blend solutions (0.5%, w/v) with and without maghemite nanoparticles (0.02 wt%) were measured at 30°C and 40ºC using specific gravity bottle, Abbe's refractometer, and ultrasonic interferometer (Model F-81, Mittal Enterprises, India), respectively. Different temperatures were maintained using a thermostat bath with a thermal stability of ± 0.05ºC.

Stock solutions of GG, GG – maghemite composite, MC, and MC – maghemite composite were prepared (0.1% w/v of polymer). The maghemite composition was maintained at 0.02 wt%. The blend stock solutions (10/90, 30/70, 50/50, 70/30 and 90/10) and blend – maghemite composite stock solutions (10/90, 30/70, 50/50, 70/30 and 90/10) were prepared by stirring the mixtures at room temperature for about 45 minutes. With the above pure and blend stock solutions, different dilute blend solutions (0.01, 0.02, 0.03, 0.04, 0.05, 0.06, 0.07, 0.08, 0.09 and 0.1 w/v concentrations) were prepared. Dilute solution viscosity measurements were done at 30°C and 40ºC using an Ubbelohde suspended level viscometer. Different temperatures were maintained using a thermostat bath with a thermal stability of ± 0.05ºC.

Thin films of polymer/polymer blend - maghemite composites were prepared by solution casting technique for characterization purposes. SEM photographs were recorded using a ZEISS Sigma FESEM 300 analyzer and FTIR spectra were recorded using NICOLET AVATAR 530 spectrophotometer.

III. RESULTS AND DISCUSSIONS

A. Refractive index measurements

The refractive index (RI) values of pure polymers and their blends were measured. The RI value for MC is 1.3420 and 1.3410, and for GG is 1.3402 and 1.3385 at 30°C and 40°C, respectively. A slight linearity is observed for the 10/90 GG/MC blend compared with RI values of pure GG and MC both at 30°C and 40°C (Fig. 3). It has been well established that linearity and non-linearity of the plot indicate the miscible and immiscible nature of the blends [9, 10] respectively.

With the addition of 0.02 wt% maghemite showed an RI value of about 1.46 for pure GG, MC and their blend compositions at 30°C and of about 1.44 at 40°C. The higher RI value after the maghemite addition may be due to the brownish-red color of the maghemite nanoparticle and hence the RI measurement technique may not be considered as a suitable proof to assess the miscibility of the blend - maghemite composites with GG and MC.

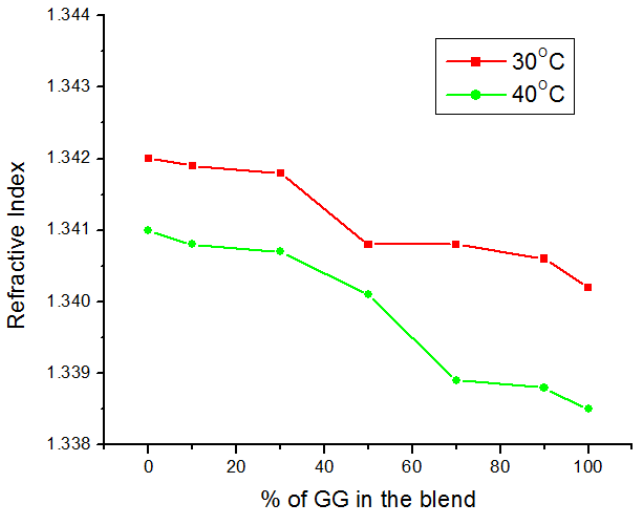

Fig. 3: Variation of refractive index of GG/MC blends in aqueous solution at 30°C and 40°C

B. Density measurements

The measured density values (ρ) of GG were $0.9972x10^3$ Kg/m^3, and $0.99376x10^3$ Kg/m^3, and for MC were $0.9985x10^3$ Kg/m^3, and $0.99525x10^3$ Kg/m^3, at 30°C and 40°C, respectively. The density values of blend compositions were in between that of pure GG and MC. The ρ values were higher for MC compared to GG attributed to their molecular structure. The graph showed only non-linear regions indicating the immiscible nature of the GG/MC blend at both 30°C and 40°C (Fig. 4). The variation is linear for miscible and non-linear for immiscible blends [11, 12].

The recorded density values for maghemite composites were higher compared to that of pure polymers without maghemite. Density values for 0.02 wt% maghemite-GG composites were $1.007x10^3$ Kg/m^3, and $0.9985x10^3$ Kg/m^3, and for 0.02 wt% maghemite-MC composites were $1.031x10^3$ Kg/m^3, and $1.012x10^3$ Kg/m^3, at 30°C and 40°C, respectively (Fig. 5). Only 10/90 GG/MC blend – maghemite composite showed linearity compared with the maghemite composites of GG and MC, other compositions got deviated from linearity. Hence it can be concluded that the addition of maghemite improved the miscibility nature of GG/MC blends.

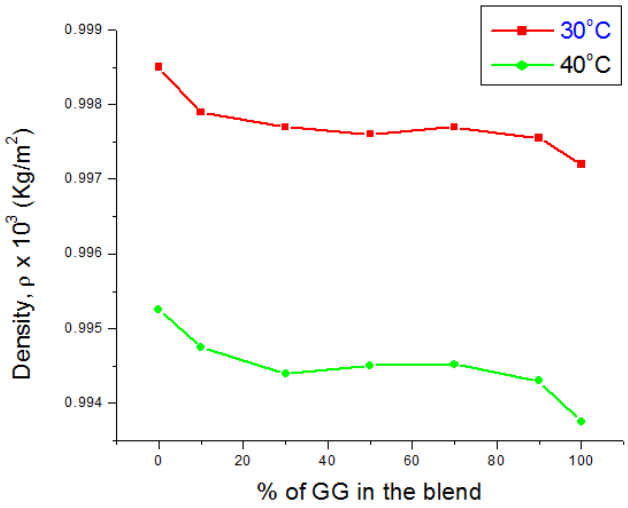

Fig. 4: Variation of density with the composition of GG/MC blend in aqueous solution at 30°C and 40°C

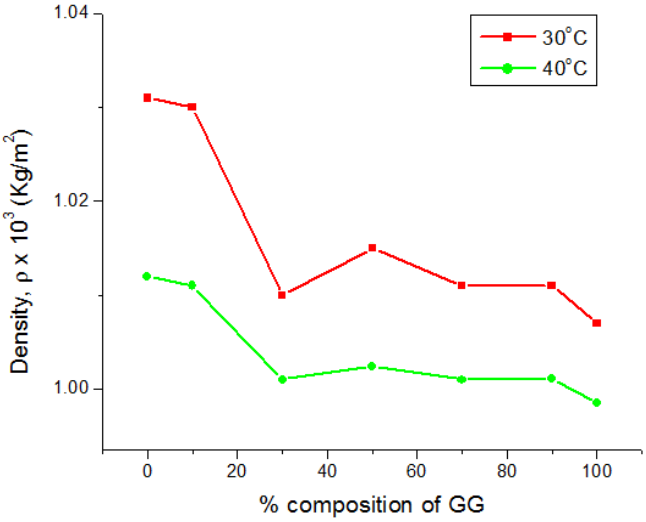

Fig. 5: Variation of density with the composition of GG/MC blend – maghemite composites in aqueous solution at 30°C and 40°C

C. Ultrasonic velocity measurements

The measured ultrasonic velocity (v) value for methylcellulose were 1525 m/s and 1545 m/s, and for GG were 1479 m/s and 1519 m/s, at 30°C and 40°C, respectively. The v for the entire compositions were found to be in between that of pure GG and MC, but the graph showed non-linear regions (Fig. 6). This indicates that the GG/MC blend is immiscible at all compositions [12, 13].

The incorporation of maghemite nanoparticles decreased the v of GG, MC, and GG/MC blends. The measured values were 1510 m/s, and 1524 m/s for MC-maghemite composite, and 1464 m/s, and 1488 m/s for GG-maghemite composites at 30°C and 40°C, respectively (Fig. 7). The observation with ultrasonic velocity measurement studies was similar to that of density studies. The presence of maghemite might have influenced the miscibility of the GG/MC blend for the 10/90 blend composition.

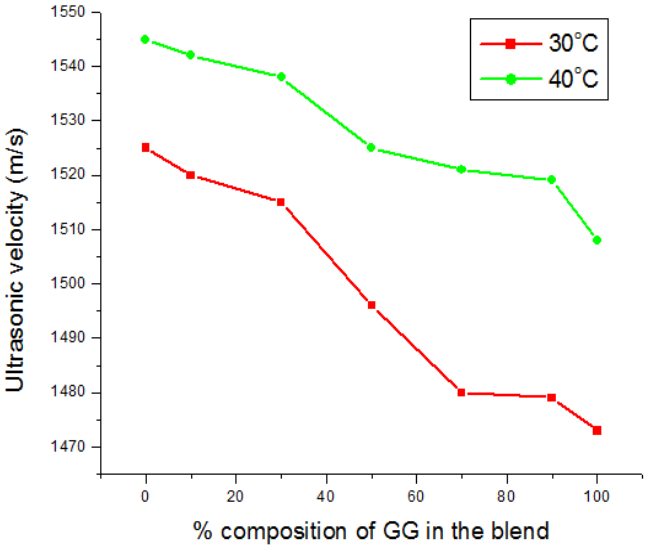

Fig. 6: Variation of ultrasonic sound velocity of GG/MC blend in aqueous solution at 30°C and 40°C

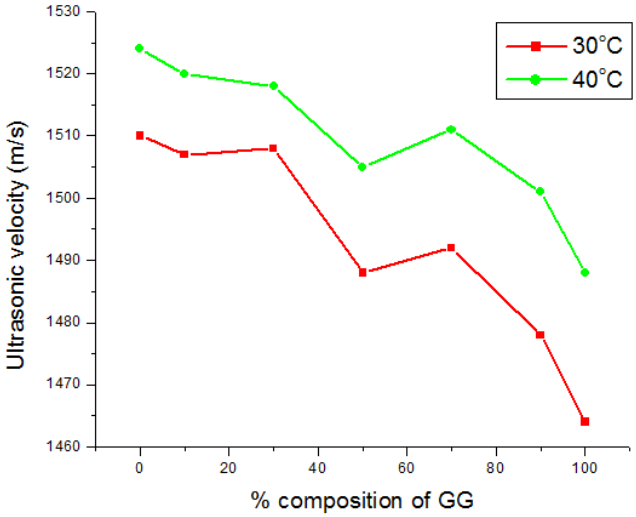

Fig. 7: Variation of ultrasonic sound velocity of GG/MC blend – maghemite composites in aqueous solution at 30°C and 40°C

D. Reduced viscosity measurements

Reduced viscosities (ηsp/C) of homopolymers GG, MC and 10/90 GG/MC, 30/70 GG/MC, 50/50 GG/MC, 70/30 GG/MC, and 90/10 GG/MC were measured at 30°C and 40ºC. The Huggin's plots of η_{sp}/C against concentration (C) are shown in Fig. 8 and Fig. 9, respectively.

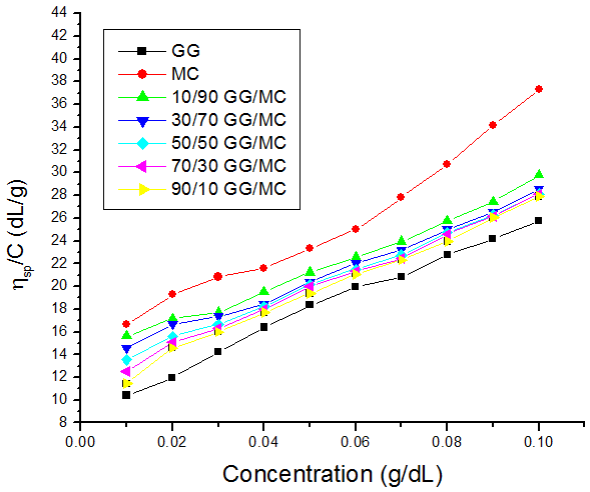

Fig. 8: Huggins's plot for GG/MC blend at 30°C

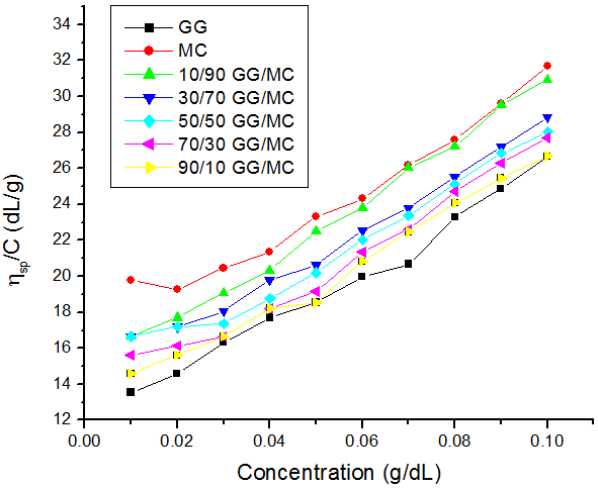

Fig. 10: Huggins's plot for GG/MC blend – maghemite composite at 30°C

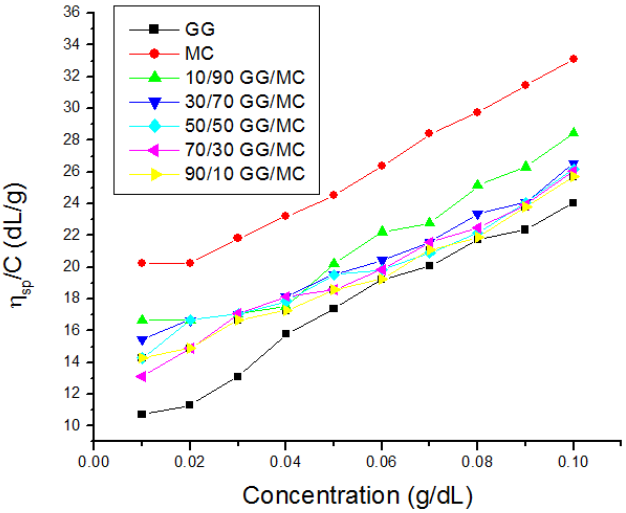

Fig. 9: Huggins's plot for GG/MC blend at 40°C

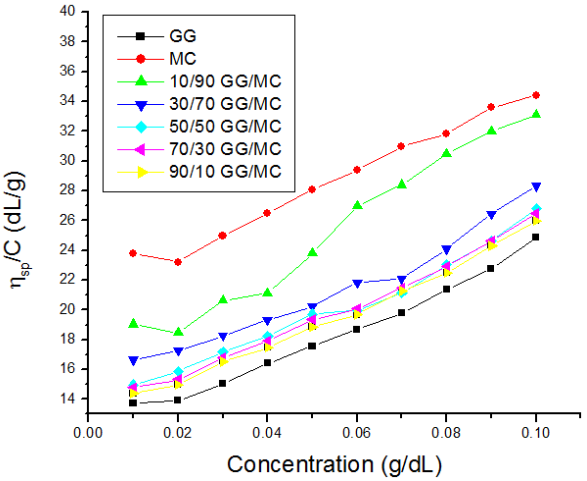

Fig. 11: Huggins's plot for GG/MC blend – maghemite composite at 40°C

Reduced viscosities of GG-maghemite composite, MC-maghemite composite, and the blend-maghemite composite compositions (10/90, 30/70, 50/50, 70/30, and 90/10) were measured at 30°C and 40ºC. η_{sp}/C against concentration plots of maghemite composites are shown in Fig. 10, and Fig. 11, respectively.

From the Huggins plots the slope values were calculated and tabulated in Table I. The slope values of the blends are lower than that of pure GG and MC. This confirms phase separation of polymers in the blend. Higher slope variations for 10/90 GG/MC blend composition with maghemite nanoparticles may be attributed to the mutual attraction of macromolecules in solution which in turn leads to the increase of hydrodynamic volume [14]. The 90/10, 70/30, 50/50, and 30/70 GG/MC blend – maghemite composite compositions showed a lower slope, which may be due to the phase separation of polymers. According to the reduced viscosity-concentration [9-13] criteria, it may be concluded that this polymer blend is miscible for 10/90 compositions and immiscible for 90/10, 70/30, 50/50, and 30/70 GG/MC blend compositions.

Table- I: Slope values from Huggins plots

Blends Comp.	30°C		40°C	
	Without Mag NPs	With Mag NPs	Without Mag NPs	With Mag NPs
0/100	217.884	139.618	152.496	131.478
10/90	154.090	163.739	140.490	179.218
30/70	150.630	140.115	117.715	126.351
50/50	158.218	136.030	117.612	124.842
70/30	166.218	142.630	132.654	128.915
90/10	172.006	139.236	123.327	128.266
100/0	170.260	142.721	154.666	124.333

E. Morphological studies

Morphology studies were carried out to identify the interaction between maghemite and the polymers and the influence of maghemite on the miscibility of GG/MC blend. SEM images recorded for GG, MC, and maghemite incorporated GG, MC, and 10/90 GG/MC blend (Fig. 12). The SEM images confirm that guar gum form weak interaction with maghemite, whereas the maghemite is well dispersed in methylcellulose. The 10/90 GG/MC blend with maghemite does not show any phase separation.

The presence of maghemite might have stabilized the structure of guar gum to form a miscible blend with MC at 10/90 blend composition.

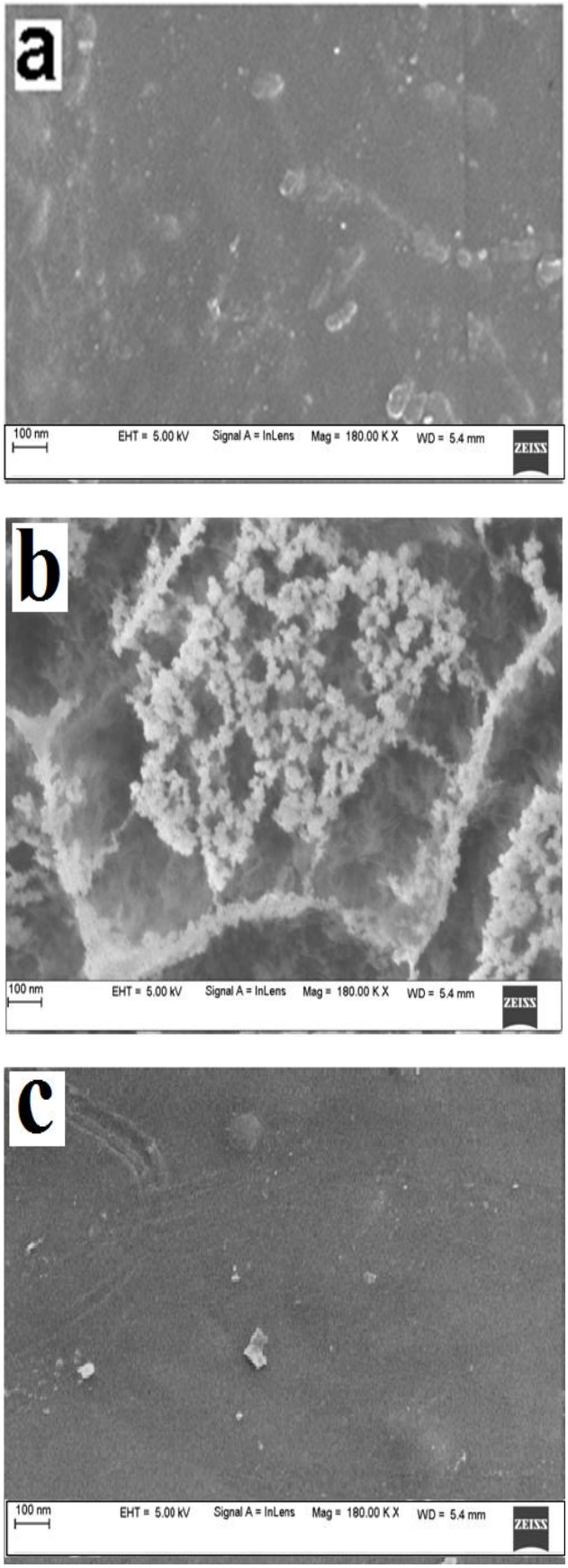

Fig. 12: SEM images of a) GG, b) GG-maghemite composite, c) MC, d) MC-maghemite composite, and e) 10/90 GG/MC blend-maghemite composite

F. FTIR spectroscopic measurements

FTIR spectra of GG, GG – maghemite composite, MC, MC – maghemite composite, and 10/90 GG/MC blend-maghemite composite were recorded in the wavelength range of 4000-500 cm^{-1} (Fig. 13). Guar gum showed (Fig. 13a) the broad and strong absorption band at 3347.3 cm^{-1}, the absorption band at 2918.2 cm^{-1}, 1638.3 cm^{-1} is due to the to –OH bond stretching, –CH group stretching, and –OH bond belonging to water molecules, respectively. The absorption band at 1370.9 cm^{-1} and 1012.7 cm^{-1} is the bending of $–CH_2–O–CH_2–$ groups [23].

The FTIR spectra of MC (Fig. 13b) showed a –OH stretching at 3350.3 cm^{-1}, and 1593.7 cm^{-1} is due the –OH bond belonging to water molecules. C-H stretching of the $–CH_2$ groups is observed at 2916.7 cm^{-1}, a $–CH_2$ scissoring around 1412 cm^{-1} and -O- stretching at 1026 cm^{-1} [24, 25].

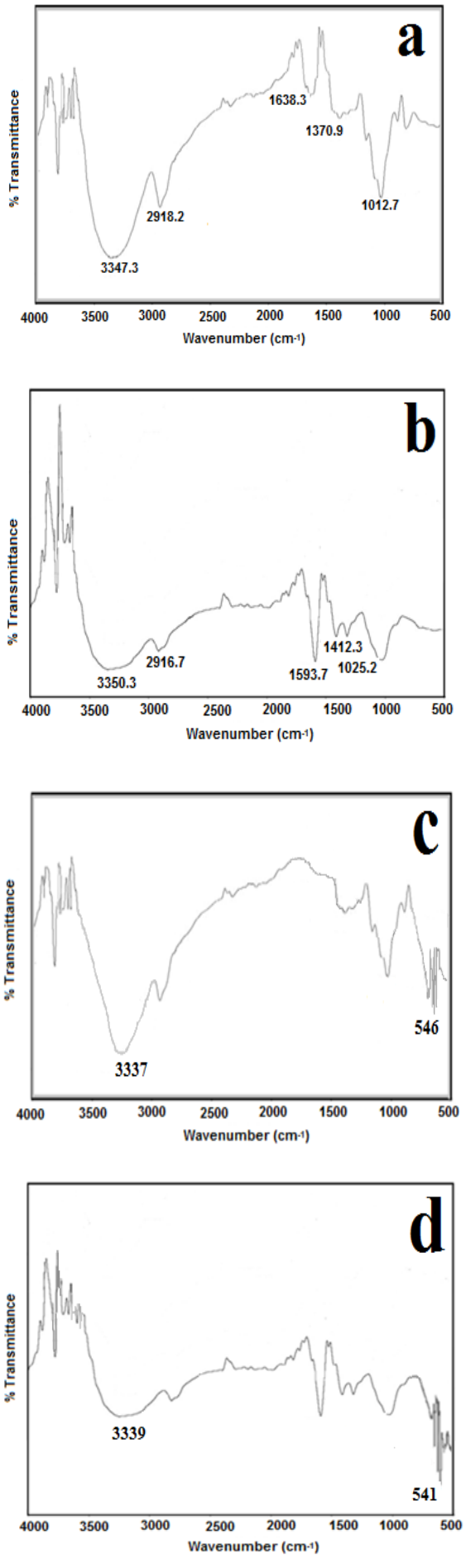

Fig. 13: FTIR spectra of a) GG, b) MC, c) GG-maghemite composite, d) MC-maghemite composite, and e) 10/90 GG/MC blend-maghemite composite

FTIR images (Fig. 13c, and Fig. 13d) showed the characteristic maghemite peaks in the GG - maghemite composite, and MC - maghemite composite. FTIR spectroscopy of 10/90 GG/MC blend - maghemite composite is given in Fig. 13e. The major peaks were associated with alcohol (υ-OH) group observed at 3310 cm^{-1}. Alkyl stretching, carbonyl group stretching, and –CH bending, -C-O stretch were observed at 2941 cm^{-1}, 1735 cm^{-1}, 1245 cm^{-1}, 1088 cm^{-1}, respectively. The peak at 537 cm^{-1} corresponds to the vibration of Fe-O, and the other peaks of pure maghemite are 454 cm^{-1}, and 632 cm^{-1} [26-27]. The instrumental limitation did not allow these two peaks to be clearly shown. The hydroxyl characteristic bands for 10/90 GG/MC blend - maghemite composite got shifted to lower wavelength compared to GG and MC, which confirms the formation of H-bonding between guar gum and methylcellulose in the presence of maghemite. Hence, the FTIR spectroscopic measurement confirms that the incorporation of maghemite influenced the miscibility of GG/MC blend, and 10/90 GG/MC blend is miscible in the presence of 0.02 wt% maghemite.

IV. CONCLUSIONS

Based on the refractive index, density, ultrasonic velocity, adiabatic compressibility, and dilute solution viscometry studies it is confirmed that guar gum and methylcellulose form immiscible blends. The influence of biocompatible magnetic nanoparticles on the miscibility of GG/MC was studied using density, ultrasonic velocity, adiabatic compressibility, and dilute solution viscometry in aqueous solution and with solution cast thin films by scanning electron microscopy and Fourier transform infrared spectroscopy. The results confirms that 10/90 GG/MC blend composition is miscible with the presence of maghemite nanoparticles. The formation of intermolecular hydrogen bonding between GG and MC in the presence of maghemite nanoparticles were confirmed with SEM and FTIR studies. The maghemite compatible blend composition of GG/MC will be having applications in drug release formulations.

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