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Estimation of Radius of Gyration, Correlation Length and Invariant for HPMC with Fe_2O_4 using SAXS Method

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Abstract: *SAXS patterns have been recorded for different composition of Fe_2O_4 with host polymer of HPMC (Hydroxypropyl Methyl Cellulose). The SAXS data has been used for the estimation of radius of gyration (R_g), correlation length (L_{cor}) and invariant (Q). The variations of these parameters were explained in terms of different percentage weight of Fe_2O_4 in HPMC contents. It is observed that 0.02% wt and 0.03% wt composition of Fe_2O_4 with higher radius of gyration value along the drawn axis due to localized breaking of polymer network.*

Keywords: SAXS, HPMC, Radius of gyration, Correlation length, Invariant.

1. Introduction

Conducting polymers are a class of materials which conducts without being heated. The era of conducting polymers began with the invention of ionic conduction in Polyethylene based electrolyte in 1973 by Wright [1]. In recent years, there is an increased interest in the research activity in the conducting polymers to bring out new materials which are suitable for electronics, opto-electronics, and electrical devices [2, 3]. Use of polymers as conducting materials solves many problems such as cost, processability, leakage, power loss and fabrication, weight of the device, good mechanical properties [4]. Barring a few conducting polymers, majority of the polymers is non-conductors or having remarkably low conductivity. Blending two or three polymers to get a binary or ternary system or doping an inorganic salts or use of plasticizer, in the polymers and their blend matrix do change the conducting aspect of a polymer. This procedure inhibits more number of charge carriers and also provides a more flexible backbone for polymer network. HPMC is chosen in our study as a host polymer in the preparation of HPMC/Fe_2O4 films, since not many reports of investigations are available. The results of the investigation do correlate stability and microstructure details of these blends do suggest that there is indeed interesting results with regard to conducting polymers in keeping view of other social obligations like polymer doped with in organic salts are degradable and environmental friendly.

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2. Materials and methods

HPMC were purchased from Loba chem Mumbai and Fe_2O_4 samples were synthesized by solution combustion method. Pure and doped films were prepared by using solvent cast method [5, 6]. HPMC (5%wt) dissolved in 100 ml of distilled water with continuous stirring. After complete dissolution the solution was filtered using filter paper to remove undissolved particles. Fe_2O_4 of different (%wt) 0.01 g-0.05 g dissolved in distilled water, and added to 100ml of 5% HPMC solution and stirred continuously using magnetic stirrer for 20 min to ensure uniform mixing of the solutions in the polymer matrix. Solution was allowed for a while and then it is poured on to the clean glass plate and allowed to dry for a week. After drying, the films were peeled out of the glass plate and stored in desiccators to avoid moisture.

3. SAXS Recording

SAXS recordings of the samples were carried out using diffractometer with Ni filtered Cukα radiation of wavelength 1.5406°A, with graphite monochromatic. Samples were scanned in the 2θ range 0°- 2.5° with step of 0.005°. Specifications used for recording are 30 kV and 15 mA. SAXS pattern of blend films contain both amorphous and crystalline region in the form of band and these SAXS profiles were used to estimated Radius of gyration, Correlation length and Invariant.

4. Theory

Radius of gyration (R_g) is defined as the root mean square of the distances of all the electrons (particles) from the electronic center of gravity of the representative molecule [7] and can be estimated using Gunier´s plot of $\log_{10}I(s)$ versus S^2. From the slope, one can compute the radius of gyration using the relation

$$R_g = \frac{1}{2\pi}\sqrt{3 * 2.303 * slope} \qquad (1)$$

This procedure was repeated for different composition of Fe_2O_4 with host polymer of HPMC.

Invariant (Q) is defined as the second moment of the SAXS curve. The invariant can be computed using expression

$$Q = \int_0^{\infty} I(q)\, q^2 \, dq \qquad (2)$$

The Q values obtained for different composition of Fe_2O_4 with host polymer of HPMC.

Correlation length (L_{cor}) is the mean width of the correlation function which is computed by taking the Fourier transform of the given SAXS curve. For the scattering along the meridional direction is given by

$$I(s) = \frac{KL_{cor}^2}{(1+4\pi^2 L_{cor}^2 S^2)} \qquad (3)$$

The correlation length is determined from the slope and intercept (K) of a plot of $I(s)^{-1}$ verses s^2 for obtained values for different composition of Fe_2O_4 with host polymer of HPMC.

5. Results and Discussion

Figure 1(a-f) shows the meridional SAXS profile (Intensity (in a.u) against 2θ in degrees) of different composition of Fe_2O_4 with host polymer of HPMC. These SAXS profiles have been used for the analysis in order to compute the radius of gyration (R_g), the invariant (Q) [8, 9] and correlation length (L_{cor}) [10].

We observe that the values of radius of gyration varies from 166 to 237 Å for different composition of Fe_2O_4 with host polymer of HPMC is given in Table-1 and the change in radius of gyration value along the drawn axis due to localized breaking of polymer network [11]. Kratky [12] has reported a value of 75 Å for a dried silk sample which is very low compared to our values.

The correlation length for different composition of Fe_2O_4 with host polymer of HPMC varies from 41.18 to 79.38 Å and it is given in Table 1. Also, the invariant is of the order of 11 in silk fiber as reported by Kratky in his paper. These results of Kratky are obtained with a small x-ray recording which occurs in the range 65 to 75 Å and they do mention that there are maxima observed at higher resolution of the order 500 to 600 Å. Here we have carried out our analysis in the range of 1500 Å region and hence we observe higher values for radius of gyration and almost same range of values for the invariant.

On the other hand, correlation length different composition of Fe_2O_4 with host polymer of HPMC varies from 41.18 to 79.38 Å these values are pretty low compared to the approximate estimation of correlation length of 200 nm by Miller et al [10] in fibers along axial direction. They also mention that there are very large uncertainties in the determination of correlation lengths. But direct Atomic Force Microscopy (AFM) measurements show that the crystals are within 100 Å or less in fibers and hence are in agreement with our results here. In fact these are believed to give rise to the power law scattering in the SAXS experiments.

Figures1 (a-f) indicates that with same value in ´Q´ the invariant, there is a increase in the values of radius of gyration up to Fe_2O_4(0.02%wt) and decrease in value up to Fe_2O_4(0.05%wt). This is due to change in volume fraction of the crystalline lamellae and also the change in electron density contrast between the crystalline and amorphous regions [13] for different composition of Fe_2O_4 with host polymer of HPMC. This is in agreement with the observed increase in lamellar value for nylon6, 6 [14].

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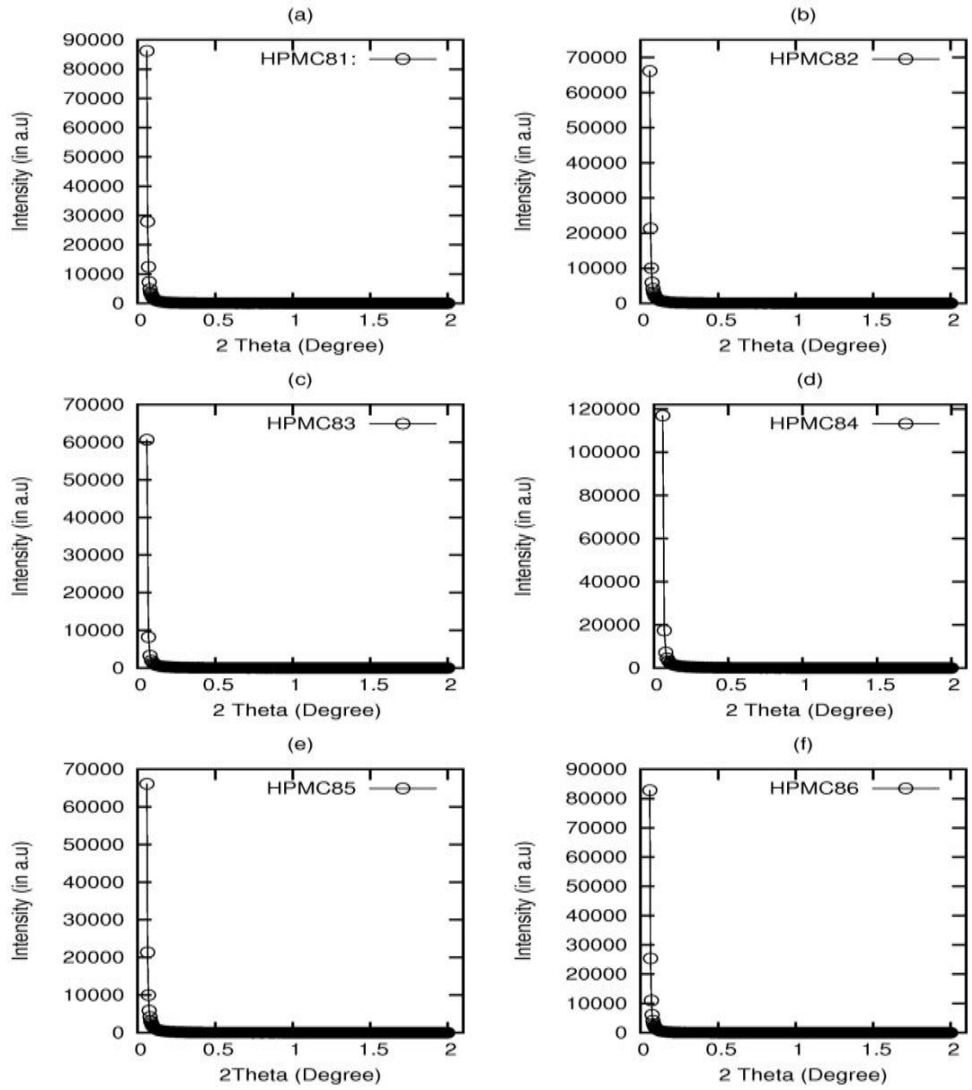

Fig.1: The SAXS profiles a) HPMC (5%wt), b) HPMC (5%)+Fe_2O_4 (0.01%wt) c) HPMC (5%)+Fe_2O_4 (0.02%wt) d) HPMC (5%)+Fe_2O_4 (0.03%wt) e) HPMC (5%)+Fe_2O_4 (0.04%wt) f) HPMC (5%)+Fe_2O_4 (0.05%wt).

Sample	R_g in Å	L_{cor} in Å	Q in $Å^{-2}$
HPMC (5%wt)	169.36 ± 4.5%	58.58	6.791×10^{-4}
HPMC(5%wt)+Fe2O4(0.01%wt)	167.20 ± 4.6%	44.92	6.791×10^{-4}
HPMC(5%wt)+Fe2O4(0.02%wt)	237.17 ± 5.2%	41.18	6.791×10^{-4}
HPMC(5%wt)+Fe2O4(0.03%wt)	229.80 ± 4.3%	79.38	6.791×10^{-4}
HPMC(5%wt)+Fe2O4(0.04%wt)	166.70 ± 4.3%	44.92	6.791×10^{-4}
HPMC(5%wt)+Fe2O4(0.05%wt)	166.81 ± 0.62%	56.26	6.791×10^{-4}

Table 1. Values of radius of gyration (R_g), correlation length (L_{cor}) and invariant (Q) for different composition of Fe_2O_4 with host polymer of HPMC.

6. Conclusion.

Small angle X-ray scattering pattern for different composition of Fe_2O_4 with host polymer of HPMC. The variation of radius of gyration and correlation length along the drawn axis due to localized breaking of polymer network.

The changes observed in amorphous and crystalline lengths within HPMC with different composition of Fe_2O_4 arises not only due to organizational changes of the structures along the length of the meridonial direction, but also due to unfolding of the molecular chains [15].

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