

# Micro Structural Parameters Studies on Sodium Sulphate Doped HPMC Polymers

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**ABSTRACT:** The changes in microstructural parameters in Hydropropyl methylcellulose (HPMC) polymer films have been studied using X-ray data. Exponential functions for the column length distribution have been used for the determination of microstructural parameters. The crystal imperfection parameters like crystal size  $\langle N \rangle$ , lattice strain (g in %) and enthalpy ( $\alpha^*$ ) have been determined by Whole pattern fitting method and the AC conductivity of the film, which is supported by the results obtained and they are discussed briefly in this paper.

**KEYWORDS:** XRD, crystallite size, **PACS:** 61.05.cp 61.10.Nz;

## I. INTRODUCTION

When a polymer is subjected to irradiation by ionizing radiation such as gamma rays, X-ray, or accelerated electrons various effects can be expected from the ionizations that occur. The ratio of resultant recombination, cross-linking, and chain scission will vary from polymer to polymer and some degree from part to part based on the chemical composition and morphology of the polymer, we have synthesized Hydroxypropylmethylcellulose(HPMC) is a polysaccharide, prepared from cellulose, with methyl and hydroxyl groups that render the cellulose molecule hydrophobic and hence it acquires surface-active properties. HPMC is water soluble tasteless non-ionic derivative of cellulose, which has been accepted as food additives in Europe, inactive ophthalmic preparations, oral capsules, suspension syrups, and eye drops [1]. HPMC blend with Sodium sulfate, Sodium sulfate is an inorganic salt with a melting point of approximately 88°C, a relative density of 2.7 g/cm<sup>3</sup> at 20°C and a water solubility of 161 g/l at 20°C . In the dry state it exists a white powder or white crystalline solid of formula Na<sub>2</sub>SO<sub>4</sub> , it has a bitter saline taste. Hence in this paper we have carried out X-ray profile analysis computed here using Whole Pattern Power Fitting (WPPF). The accuracy of a this Fourier method has been established in the recently included IUCr Round Robin test[2]

## II. LITERATURE SURVEY

Microstructural parameters such as crystal size( $\langle N \rangle$ ) and lattice strain (g in %) are usually determined by employing Fourier method of Warren and Averbach and Warren[1]. The intensity of a profile in the direction joining the origin to centre of the reflection can be expanded in terms of Fourier cosine series:

$$I(s) = \sum A(n) \cos\{2\pi n d(s - s_0)\} \quad (1)$$

Where the coefficients of the harmonics A(n) are function of the size of the crystallite and disorder of the lattice. Here, s is  $\sin(\theta)/\lambda$ , n is the harmonic number and d is the lattice spacing. The Fourier coefficients can be expressed as;

$$A(n) = A_s(n).A_d(n) \quad (2)$$

An analytical expression for the size distribution yields an expression for crystallite size distribution as

$$A_s(n) = A(0) \exp\{-\alpha(n-p)\} / (\alpha N) \quad (3)$$

and for lattice strain, the expression turns out to be;

$$A_d(n) = \exp\{-2\pi^2 n^2 m g^2\} \quad (4)$$

Where m is the order of reflection, Here the unknown quantities are N, g,  $\alpha$  and background which can be computed using multidimensional minimization SIMPLEX program[2]. The experimental profile is matched with simulated profile using the above parameters till a good fit is observed. The goodness of the fit is determined by computing,

$$\Delta^2 = [I_{cal} - (I_{exp} + BG)]^2 / (npt) \quad (5)$$

### III. METHODOLOGY

#### X-ray diffraction measurements:

The X-ray diffractograms (XRD) of the polymer samples were recorded using RigakuMiniflex-II X-ray diffractometer with Ni filtered, CuK $\alpha$  radiation of wavelength  $\lambda = 1.5406 \text{ \AA}$ , with a graphite monochromator. The specifications used for the recordings were 30Kv, 15mA. The samples were scanned in the  $2\theta$  range  $10-60^\circ$  with a scanning speed and step size of  $1^\circ/\text{min}$  and  $0.02$  respectively.

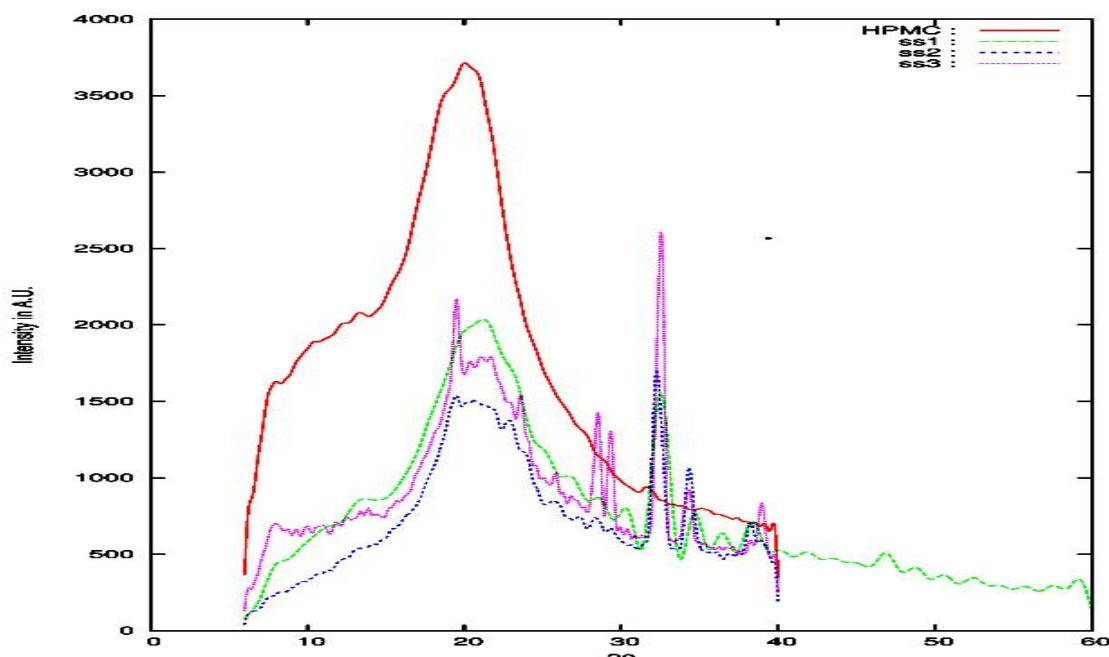


Figure 1. XRD pattern of HPMC/ NaSO<sub>4</sub> polymer films.

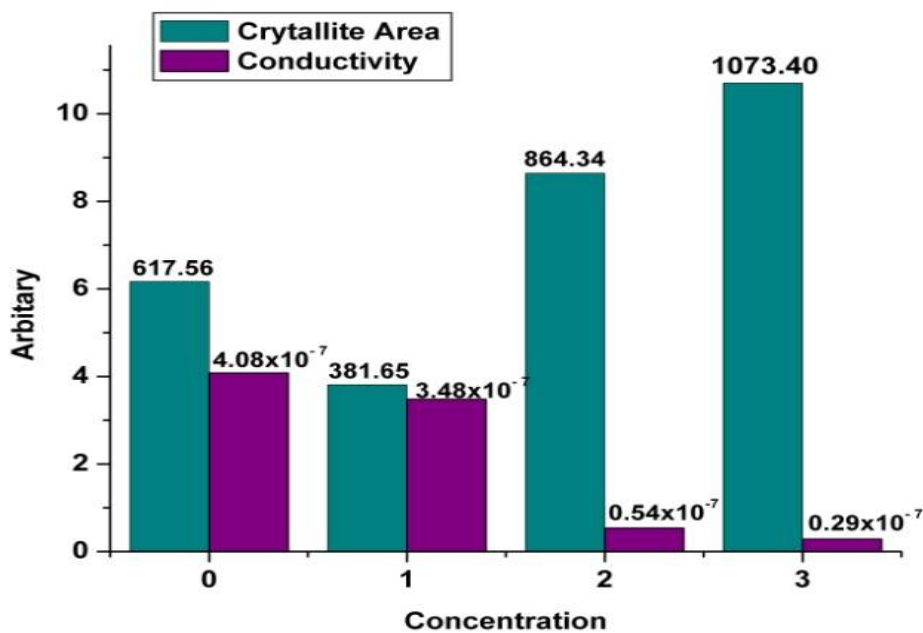
**Conductivity Test:**

TABLE 1: **Micro structural parameters of HPMC/ NaSO<sub>4</sub> polymer films** The conductivity measurements of samples were determined by using HIOKI LCR-Hi tester 3532-50 model available. Our samples were taken in the sample holder provided in the instrument. This instrument gives the transconductance (G) of the sample which is dependent of its thickness and the area. The conductivity ( $\sigma$ ) of the sample can be obtained by the relation,

$$\sigma = \frac{t \times G}{A}$$

Where 't' is the thickness and 'A' is the area of the sample.

This instrument gives the transconductance values for different frequencies from 50Hz to 5MHz. The obtained transconductance values for various frequencies were evaluated to get the conductivity results for the same corresponding frequencies.



**Figure 2:** Pictorial Representation of Conductivity and Crystallite Behavior

**IV. SIMULATION RESULTS**

X-ray diffractograms for HPMC polymer blend films with NaSO<sub>4</sub> are given figure 1. Whole pattern fitting method is quite useful for relative comparison of crystallite size and strain in a series of polymer blend films of various concentration of NaSO<sub>4</sub> in HPMC. The computed micro structural parameters of HPMC and HPMC:NaSO<sub>4</sub> polymer blend are given in Table 1. The computed microcrystalline parameters like crystallite size <N>, lattice strain g in % and

the standard deviation of these parameters while computing are also shown. Using these parameters ( $\langle N \rangle$ ,  $g$ ) one can estimate the minimum enthalpy ( $\alpha^*$ ) and the crystallite area of the sample. From the obtained results of these polymer composites it is clear that the crystallite area of the polymer composites increases with the increase in the concentration of Sodium Sulphate salt. The result of conductivity gives a contradictory response by showing low conductance at higher concentrations. That is the conductivity of the polymer composites decreases with the concentration. In other terms, as crystallite area of the polymer composites increases the conductivity decreases. The figure 2 describes this behavior of the polymer composites thoroughly and conductivity study.

**TABLE 1:** Micro structural parameters of HPMC/ NaSO<sub>4</sub> polymer films and conductivity study.

Sample	$2\theta$	$d$ in Å	$\langle N \rangle$	$D_s$ in Å	$g$ in %	$\alpha^*$	Conductivity $10^{-7}$ s/cm	Crystallite Area
HPMC	7.72	11.44	4.74	54.22	0.5	1.29	4.08	617.56
	20.09	4.415	2.58	11.39	0.5	1.19		
HPMC+ 1%NaSO <sub>4</sub>	20.97	4.23	2.61	11.04	0.0	1.43	3.48	381.65
	32.48	2.75	12.56	34.57	0.0	0.14		
HPMC+ 2%NaSO <sub>4</sub>	21.16	4.80	2.64	12.67	0.5	1.33	0.54	864.34
	32.48	2.76	22.05	61.05	0.0	0.07		
	34.48	2.61	26.14	68.22	0.0	0.07		
HPMC+ 3%NaSO <sub>4</sub>	7.09	11.09	3.17	35.18	0.5	0.77	0.29	1073.40
	20.75	4.27	2.65	11.33	0.5	1.30		
	28.50	3.12	30.28	94.74	0.5	0.07		
	29.30	3.04	27.07	82.42	0.5	0.07		
	32.53	2.75	29.31	80.60	0.5	0.06		

## V. CONCLUSION

Sulphate group acts an hindrance to free flow of conduction electron a polymer dielectric medium. We expected a contrary result when we started this investigation. The presence of sulphate group do increase the crystallite size, which may enhance the mechanical property of the films, which needs further investigations.

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