INVESTIGATIONS THE EFFECT OF EOSIN B DYE ON X- RAY DIFFRACTION PATTERN OF SILVER NITRATE DOPED PVP FILMS

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ABSTRACT

In this research, X-ray diffraction of the powder (PVP polymer, Eosin B dye, and silver nitrate) and (EB/PVP, $AgNO_3$/PVP, EB/$AgNO_3$/PVP) films have been studied. Casting method is used to prepare homogeneous films on plastic petri dishes. All parameters accounted for the X-ray diffraction; full width half maximum (FWHM), Miller indices (hkl), size of crystalline (D), Specific Surface Area (S) and Dislocation Density (δ).The nature of the structural of materials and films will be investigated. The XRD pattern of PVP polymer was amorphous structure with two broader peaks and the Eosin B dye and silver nitrate have crystalline structure. While the mixture between these materials led to appearing some crystalline peaks into XRD pattern of PVP polymer.

Keywords: Eosin B dye, silver nitrate, X-ray diffraction, full width half maximum, PVP polymer.

الخلاصة

في هذا البحث، تم دراسة حيود الاشعة السينية لمساحيق (بوليمر PVP، صبغة الايوسين بي ونترات الفضة) واغشية (EB/PVP, $AgNO_3$/PVP, EB/$AgNO_3$/PVP). استخدمت طريقة الصب لتحضير اغشية متجانسة على أطباق بلاستيكية. تم حساب كل معاملات حيود الاشعة السينية ،أقصى منتصف لعرض الحزمة، معاملات ميلر، الحجم البلوري، المساحة السطحية المحددة وكثافة الانخلاع. وتم بحث الطبيعة التركيبية للمواد والاغشية. وبينت النتائج أن حيود الاشعة السينية لبوليمر PVP هو ذو تركيب عشوائي مع حزمتين عريضة وان صبغة الايوسين B ونترات الفضة لها تراكيب بلورية. بينما المزيج بين هذه المواد أدى الى ظهور بعض الحزم البلورية في نموذج حيود الاشعة السينية لبوليمر PVP .

INTRODUCTION

Among the many polymers, the polyvinylpyrrolidone (PVP) has good film - forming and adhesive behavior on many solid substrates and its formed films exhibit good optical quality (high transmission in visible range) and mechanical strength (easy processing) required for applications. The amorphous structure of PVP also provides a low scattering loss, which makes it as an ideal polymer for composite materials for different applications [1-3].

In order to examine the physico-chemical make-up of unknown materials, the mineralogists and solid state chemists use primarily the Powder X-ray Diffraction techniques which are the most important characterization tools used in solid state chemistry and materials science. The size, shape, lattice parameter determination and phase fraction analysis of the unit cell for any compound can be determined easily by XRD.The information of translational symmetry-size and shape of the unit cell are obtained from peak positions of diffraction pattern.

S. Bykkam*et al.* [4] illustrated technique for green synthesis of silver nanoparticles from silver nitrate solution by co-precipitation using the leaf extract of different species of ocimum which acts as reducing and capping agent. The important ingredients responsible for the

formation of silver nanoparticles present in the leaf extract are triterpenes, flavonoids and eugenol. Wide range of experimental conditions has been adopted in this process and its X-ray diffraction characterizations. Sh. U.D. Khan *et al.* [5] showed XRD patterns confirm the incorporation of Ag particles in PVP matrix and indicated the improvement in polycrystalline behavior by the appearance of more diffraction peaks with increasing $AgNO_3$ concentration in the composites. M. Mallik & R. K. Mandal [6] investigated the role of PVP as capping agent in the existence of PVA polymer, and the effect of weight ratio of PVP/ PVA to synthesize nanocrystalline FCC silver in different shapes and size range. A.Rawat *et al.* [7] prepared thin films of polyvinylpyrrolidone, polyacrylamide and their blends by using solution cast method and characterized using X-ray diffraction (XRD) technique. K. Sivaiah *et al.* [8] investigated structural, optical, thermal, electrical properties from measurement of XRD, FTIR, SEM, optical absorption spectra.

The aim of this work is to investigate the nature of the structural characteristics of PVP polymer, Eosin B dye, $AgNO_3$, EB/PVP film, $AgNO_3$ 0.008g/PVP film and EB/$AgNO_3$ (0.008g)/PVP film are done by X–ray diffraction measurement.

Crystalline size was calculated from Equation (1):

$$D = (L \lambda)/(\beta \cos \theta) \quad (1)$$

Where:

L is a constant related to crystallite shape, normally taken as 0.94.

β is the full width half max (FWHM) for peak of XRD.

λ is the wavelength of the target that used in XRD instrument.

Specific Surface Area (S) from Equation (2) and Dislocation Density (δ) from Equation (3) were calculated in Table (3).

$$S = (6\times10^3)/(D. \rho) \quad (2)$$

Where S is the specific surface area and ρ is the density of silver nitrate 4.35 g/cm^3.

$$\delta = (15 \beta \cos\theta)/(4a D) \quad (3)$$

Where δ is dislocation density. It is calculated from broadening of diffraction line measured at half of its maximum intensity (radian), θ Bragg's diffraction angle (degree) and a lattice constant (nm).

EXPERIMENTAL WORK

The chemical structure of PVP (PolyVinylPyrrolidone) is $(C_6H_9ON)_n$, with average molecular weight ($\overline{M_W}$ = 40,000 g/mole),Silver Nitrate ($AgNO_3$) with molecular weight 169.8731 g/mole, Eosin B dye ($C_{20}H_6N_2O_9Br_2Na_2$) with molecular weight 624.06 g/mole. The solvent used for all materials was Ethanol with purity 99.99 %.

Preparation of pure PVP and Eosin B- PVP film are achieved by casting method [9]. The solution of the PVP polymer is prepared by dissolving the required amount (0.5g) of polymer in (10 ml) ethanol solvent .Then PVP solution is stirred very well at magnetic stirrer for (25) min. and put onto a plastic petri dish with diameter (10cm) .Homogeneous pure PVP films obtained after drying at room temperature 30 °C for (24 hours).To prepare Eosin B- PVP film, volume ratio of Eosin B solution (20ml) added to PVP solution and mixed very well at magnetic stirrer. Then the mixture poured into plastic petri dish and left for (24 hours) to obtain homogeneous Eosin B/PVP film. The concentration of Eosin B is 1×10^{-5} mole/liter.

In the same method, silver nitrate $AgNO_3$ will be added to pure PVP and Eosin B/PVP and left quarter hours on magnetic stirrer to get $AgNO_3$/PVP and Eosin B/$AgNO_3$/PVP film, the amount of mass of $AgNO_3$ are taken (0.008g).

X–ray diffraction measurement by using XRD Spectrophotometer type (Shimadzu -6000) made in Japan with characteristics (Target (Cu), Current (30 mA), Voltage (40 KV), Wavelength (1.54056 °A)).

RESULTS AND DISCUSSIONS

The XRD pattern for pure PVP film exhibited broad peak with 2θ = 20.9402°, intensity is 62 and peak with 2θ = 11.4695°, intensity 36 this means the amorphous nature of pure PVP film,

as shown in Figure 1. Table 1 illustrated the interplanar spacing between the atoms (d), the magnitude of (2θ) for peaks with their intensities. These results matched with A. Rawat et.al [7].

Table 1: XRD Parameters of PVP powder

2θ (deg)	d (°A)	Intensity (counts)
20.9402	4.23888	62
20.3418	4.36221	54
20.0826	4.41792	52
11.4695	7.70892	36

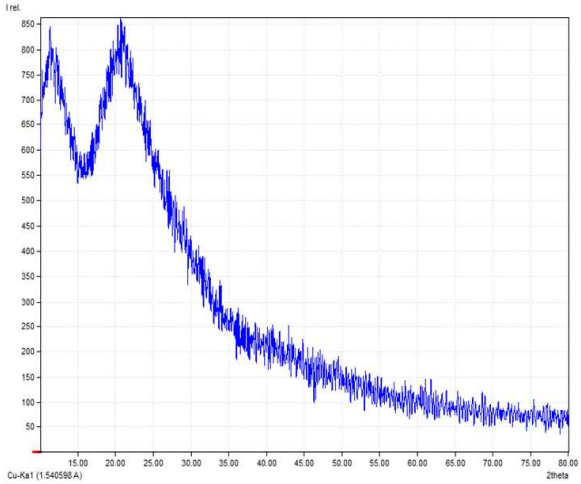

Figure 1: The X-ray diffraction for PVP Powder

Figure 2 illustrated the X-ray diffraction for pure Eosin B dye powder, and Table 2 illustrated the magnitude of (2θ) for peaks with their intensities and FWHM. There are three strongest peaks appeared at 2θ equal to (22.7906, 27.2073, 24.5519) deg.

Table 2: XRD Parameters of Eosin B Dye

2θ (deg)	d (°A)	FWHM (deg)	Intensity (counts)
22.7906	3.89873	0.27000	107
24.5519	3.62289	0.34000	84
27.2073	3.27503	0.24000	92

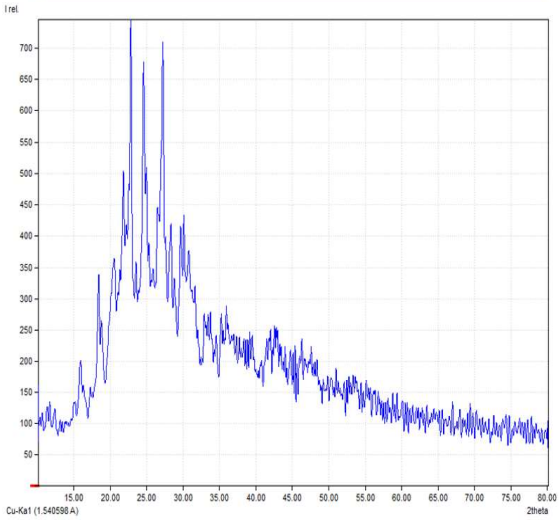

Figure 2: The X-ray diffraction for Eosin B dye powder

The X-ray diffraction pattern for pure $AgNO_3$ powder that shows the structure is a crystalline, as illustrated in Figure -3-. The crystal structure of $AgNO_3$ is orthorhombic with cell parameter a = 10.125°A, b = 7.3350°A, c = 6.9920°A and the Table 3 demonstrated the magnitude of (2θ) for peaks with their intensities, FWHM, Miller indices (hkl), size of crystalline (D).

Table 3: XRD Parameters of $AgNO_3$ powder

2θ (deg)	d (°A)	FWHM (deg)	Intensity (counts)	Miller indices (hkl)	D (nm)	$S\times10^6$ m^2/g	$\delta\times10^{14}$ m^{-2} at a=1.0125 $\times10^{-9}$ m	$\delta\times10^{14}$ m^{-2} at b=0.73350 $\times10^{-9}$ m	$\delta\times10^{14}$ m^{-2} at c=0.69920 $\times10^{-9}$m
19.7835	4.48403	0.14610	124	111	55	0.02487	1.67	2.30	2.41
24.3754	3.64872	0.22640	144	020	36	0.03830	3.96	5.46	5.73
29.7234	3.00327	0.19890	364	211	42	0.03322	2.98	4.11	4.32

32.8755	2.72216	0.20740	134	122	40	0.03442	3.19	4.41	4.63
40.1715	2.24299	0.15140	152	131	56	0.02457	1.63	2.25	2.36
49.7524	1.83117	0.13180	186	322	67	0.02059	1.14	1.58	1.66

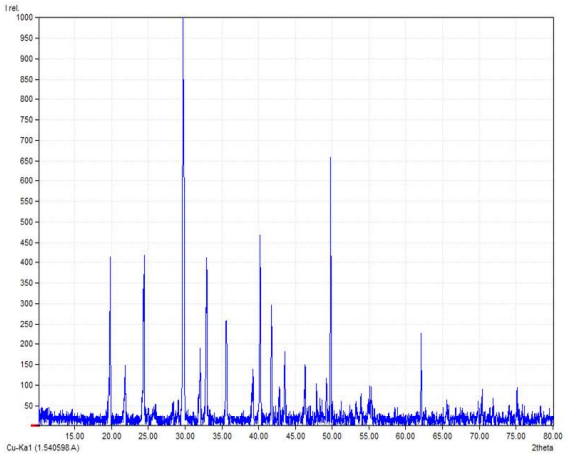

Figure 3: The X-ray diffraction for $AgNO_3$ powder

When Eosin B dye was added to PVP polymer, the amorphous structure of PVP still constant but there are some peaks of Eosin B will appeared in spectrum; as shown in Figure -4- .The peaks (66,93,83) counts for Eosin B clearly showed. This make the film be amorphous structure. Table -4- illustrated the characteristics for this structure.

Table 4: XRD Parameters of Eosin B/PVP Film

2θ (deg)	d (˚A)	FWHM (deg)	Intensity (counts)	D (nm)
11.3701	7.77609	0	53	-
18.7717	4.72338	0.54000	38	15
19.5093	4.54643	0.51000	49	16
22.2967	3.98397	0	38	-
24.6737	3.60528	0.48400	66	17
25.1665	3.53579	0.31170	93	26
29.3120	3.04448	0.36220	83	23

When adding $AgNO_3$ (0.008g) to PVP polymer; the amorphous structure still for PVP film and some peaks of $AgNO_3$ clearly appeared in XRD spectrum as showed in Figure 5. Table 5 illustrated the information of peaks such 2θ, FWHM, Intensity.

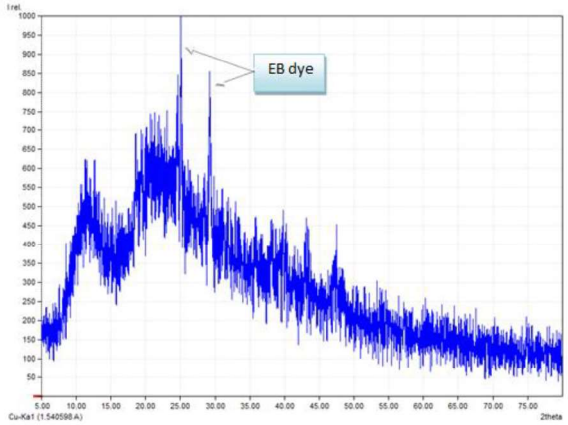

Figure 4: The X-ray diffraction for Eosin B/PVP film

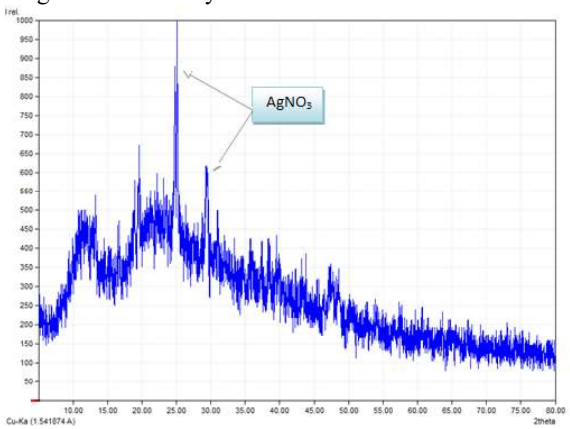

Figure 5: The X-ray diffraction for $AgNO_3$/PVP film

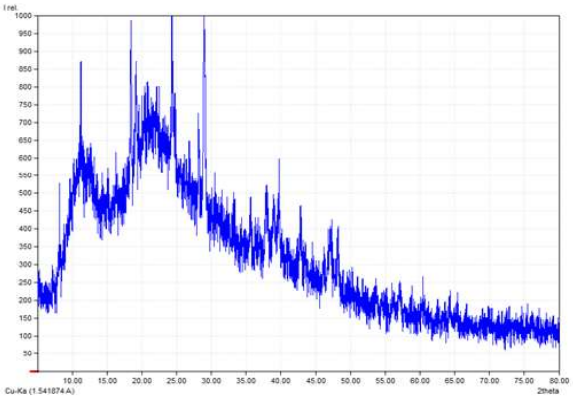

Figure 6: The X-ray diffraction for Eosin B/$AgNO_3$/PVP film

Table 5: XRD Parameters of $AgNO_3$/PVP Film

2θ (deg)	d (˚A)	FWHM (deg)	Intensity (counts)	D (nm)
11.4894	7.69561	0	33	-
12.8426	6.88762	0	31	-
13.3327	6.63552	0.21080	41	38
18.9295	4.68436	0.34000	31	24
19.6439	4.51558	0.22400	50	36
20.1823	4.39632	0.32000	15	25
22.7024	3.91368	0.13330	21	61
23.5875	3.76879	0.22750	27	36
24.8488	3.58027	0.19500	97	42
25.1624	3.53636	0.15940	101	51
29.2044	3.05545	0.20000	25	41
29.4465	3.03088	0.35500	49	23
31.2618	2.85890	0.08000	9	104
32.0209	2.79284	0.12800	18	65

Table 6: XRD Parameters of Eosin B/ $AgNO_3$ /PVP Film

2θ (deg)	d (˚A)	FWHM (deg)	Intensity (counts)	D (nm)
11.3304	7.80324	0.72000	67	11
18.5040	4.79111	0.28670	69	28
19.2128	4.61592	0.33500	51	24
22.2169	3.99810	0.27200	37	30
24.4182	3.64242	0.36400	59	22
24.7914	3.58843	0.36000	32	23
28.2457	3.15694	0.28000	35	29
29.0123	3.07525	0.33110	102	25
36.4416	2.46354	0.27000	15	31
38.5382	2.33420	0.11330	9	75
39.7191	2.26748	0.35000	38	24
42.8270	2.10986	0.33000	28	26
46.7498	1.94155	0.14660	20	59
47.5094	1.91226	0.24000	10	36
48.1524	1.88822	0.38000	28	23

CONCLUSIONS

In this research PVP, Eosin B, $AgNO_3$, Eosin B/PVP film, $AgNO_3$/PVP film, Eosin B/$AgNO_3$/PVP film were dissolved in ethanol and prepared in casting method in plastic petri dishes. XRD parameters of materials and films calculated and have known the nature of the structural of materials and films. In spite of crystalline materials Eosin B and $AgNO_3$, the polymer still amorphous behavior with appearing some crystalline peak from these materials in it.

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