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Investigation of Physical Structure of Sudan III/PVK Film Composite Exposure to Acetone, and Alcohol Vapor by using XRD Method

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Abstract. Fabrication of Sudan III/PVK composite film for sensor technology application has been carried out by using casting method. The physical structure of Sudan III/PVK composite film exposure to acetone and alcohol vapor have been investigated by using XRD method. In this paper would be determined the interplanar distance of crystals plane, crystallinity index, the average crystallite size and the average polymer chain separation within the Sudan III/PVK composite film and also for pure Sudan III and PVK powder samples according to XRD experimental results.

1. Introduction

Development of a composite polymer material of Sudan III/poly (N-vinylcarbazole) as a sensing material for optical sensor technology application is an interesting subject research [1, 2].

The optical sensor based on polymer optical fibre is important because of resulting lightweight sensor device, and without electrical power at the sensing point and is easily produced and low cost [3].

Sudan III molecule possesses azobenzene group and so that this molecule is well known very sensitively to its environmental especially in presence of other molecules such as alcohol, ethanol and acetone vapor [4].

PVK material have been used as matrix polymer for fabrication of TiO₂-PVK composite film for gas sensor etahanol application. And aslo for development of composite material using inorganic nanocrystal such as PVK-LiNbO₃. The properties sensing material of course is also determined by the physical structure such as the crystallinity.

In this research we would like to determine the crystallinity content involving the crystal plane distance, the average crystal size, the average chain separation, and degree of crystallinity within the Sudan III/PVK composite film. In this paper the crystallinity of pristine Sudan III and PVK in form of powder also discussed.



2. EXPERIMENTAL

Fabrication of Sudan III/PVK composite film has been carried out using solution casting method. This experiment aims to produce a good quality composite film which are homogeneously film and a smoothly surfaces film which are strongly depend on a solvent used.

2.1. Materials

Sudan III molecule

Sudan III molecule is *red disazo dye* which possesses a hydroxyl group and two azo groups that are sensitively to interact with others molecule such as CO₂. The Sudan III molecule structure is shown in figure 2.1.

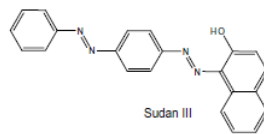


Figure 2.1. Chemical structure of Sudan III [6]

2.2. Poly (N-vinylcarbazole)

Poly(N-vinylcarbazole) or PVK is photoconductive polymer. The PVK possesses physical properties such as softening point above 175°C, glass transition 200°C, melting point : >320°C, Colors : transparent, slightly yellow, soluble in aromatic hydrocarbons, chloroform, chlorobenzene, methylene chloride, and tetrahydrofuran with refraction index $n_{0,20} = 1.696$.

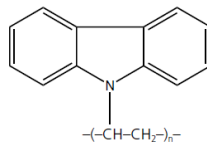


Figure 2.2. Poly (N-vinylcarbazole) or PVK structure [6].

a. Sample Preparation

Preparation of Solution Sample

The solvent used in this experiment was CHCl₃. Sudan III solution and PVK solution were prepared by dissolving Sudan III powder and PVK powder in CHCl₃ respectively and then stirred for 12 hours using magnetic stirrer in order to obtain the missible solution. Next, the stock of Sudan III solution and stock of PVK solution were combining to obtain the Sudan III/PVK composite solution. The composition of Sudan III/PVK solution were 20/80 % wight and 20/40 % weight.



Figure 2.3. Preparation of Sudan III/PVK composite solution.

Preparation of Sudan III/PVK composite film.

Preparation Sudan III/PVK composite film was carried out using casting solution method. The Sudan III/PVK solution was deposited on a clean glass substrate and then dried at room temperature. Next, it was covered to avoid contamination from unwanted material such as dust.

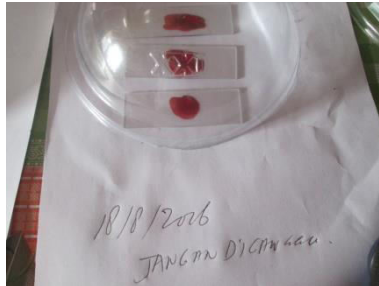


Figure 2.4. Fabrication of SudanIII/PVK composite by casting solution method.

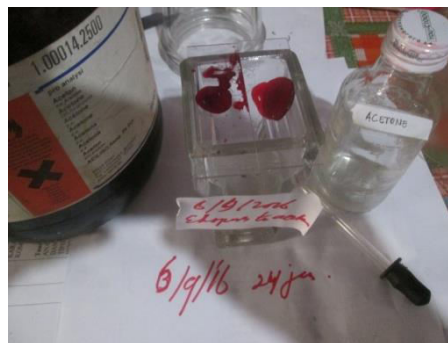


Figure 2.5. Experimental of Sudan III/PVK composite film exposure to acetone vapor

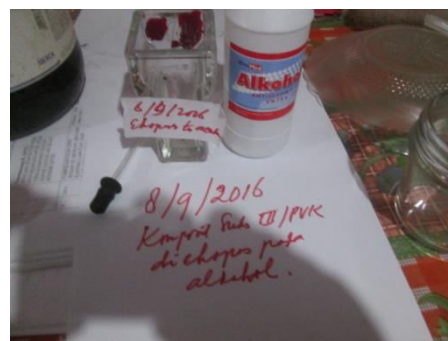


Figure 2.6. Experimental of sudan III/PVK composite film exposure to alcohol vapor

The experimental of Sudan III/PVK composite film exposure to acetone vapor and alcohol vapor were worked out as shown in figure 2.5 and 2.6. The composite film was placed at about 10 cm above the jar which is containing of 5 ml acetone and alcohol liquids. And then it was vaporized at 23°C for 12 hours.

Sample Characterization

Characterization of the samples namely Sudan III molecule and PVK and Sudan III/PVK composite film were carried out by using XRD measurement.

The XRD (*X-Ray Diffraction*) measurement is intended to investigate the structural physics of the samples. From the XRD spectra can be then determined the crystal plane distance, degree of crystallinity, the average polymer chain/molecule separation, the average crystal size. The XRD measurement using Philips Diffractometer. In this experiment XRD diffraction pattern is recorded with

scan of 2θ at 30 kV, 30 mA with radiation source of $\text{CuK}\alpha$ ($\alpha = 1.5406$ Angstrom). The XRD measurement was in the range of diffraction angle $2\theta : 3.5\text{-}75^\circ$. For sudan III/PVK composite film exposure to acetone vapor and alcohol vapor were measured using Rigaku Miniflex 600 (600 W, 30 kV, 10mA with X-Ray tube Cu).

3. RESULTS AND DISCUSSION

The result of XRD measurement of PVK powder is shown in figure 3.1.

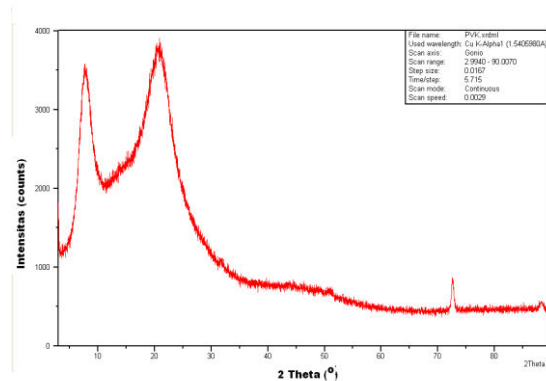


Figure 3.1. XRD spectra of PVK powder [1]

From XRD spectra can be calculated the crystal plane distance according to the Bragg formula : [7] .

$$d_{hkl} = \frac{\lambda}{2 \sin \theta} \quad 3.1.$$

In Angstrom. The result is shown in table (3.1).

Table3.1. Bragg angle (2θ) in degree and crystal plane distance in $d_{(hkl)}$ in Angstrom.

PVK powder sample			
2θ	7.7	19.3	72.5
$d_{(hkl)}$ (Å)	11.4	4.6	1.3

From the XRD spectra (figure 3.1) the average polymer chain separation for PVK sample in powder form was calculated by choosing peak with maximum intensity $2\theta = 19.3$ ($d_{hkl} = 4.6$ Angstrom). By using the formula

$$S = 5\lambda / 8 \sin \theta. [8] \quad 3.2.$$

where S is separation polymer chain, λ is X-ray wavelength (1.5406 Angstrom) and θ is diffraction angle at maximum intensity from *halo* amorf . So we get $S = 5.744$ Angstrom atau 57.44 nm.

The average polymer chain separation of PVK sample film (figure 3.3) was calculated from the amorf peak with a maximum intensity. By using the formula 3.2 is obtained $S = 49.48$ nm.

Next, the average polymer chain separation of Sudan III/PVK (1/2) composite film is calculated from XRD spectra as shown in figure 3.4. From the amorf peak with a maximum intensity of at $2\theta = 22,44^\circ$ so $S = 49.48$ nm and for Sudan III/PVK (1/4) composite film (figure 3.5) from the amorf peak with a maximum intensity of at $2\theta = 22,42^\circ$ we get $S = 49.18$ nm.

Finally, for Sudan III/PVK composite film exposure to acetone and alcohol can be obtained that the value of S for those sample are the same namely $S = 51.86$ nm.

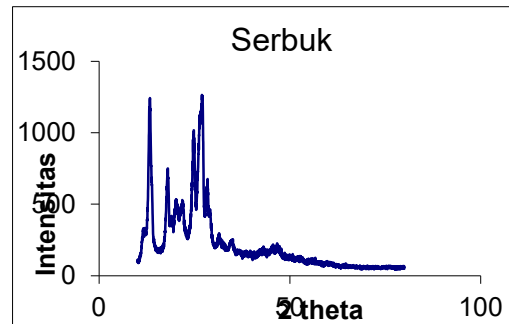


Figure 3.2. The XRD spectra of Sudan III pristine powder [2]

Identification of XRD spectra of Sudan III pristine powder is shown in table (3.2).

The results of XRD measurement for PVK film and for Sudan III/PVK composite film without exposure to the vapor is shown in figure 3.3 and 3.4.

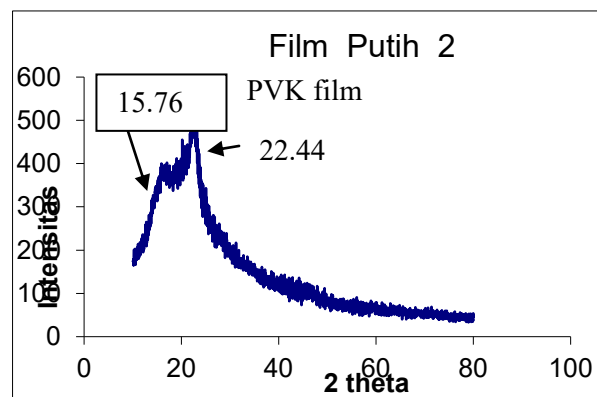


Figure 3.3. The XRD spectra of PVK film resulting from casting solution

Tabel 3.2. Bragg angle (2θ) in degree and crystal plane distance in $d_{(hkl)}$ in Angstrom of Sample PVK film.

Sample PVK film		
2θ	15.76	22.44
$d_{(hkl)}$ (Å)	5.13	3.96

Table 3.3. Bragg angle (2θ) in degree and crystal plane distance in $d_{(hkl)}$ in Angstrom of Sudan III pristine powder.

Sample	2θ	$d_{(hkl)}$ (Å)
Sudan III pristine powder	13.3	6.64
	17.94	4.94
	18.36	4.81
	20.12	4.40
	21.7	4.05
	24.84	3.58
	26.96	3.31
	27.4	3.25
	31.36	2.85
	34.26	2.62
	42.08	2.14
	45.2	2.01
	45.38	2.00

Figure 3.4 shows a phenomenon a decreasing of XRD intensity at $2\theta = 28.6$ as increasing of PVK concentration.

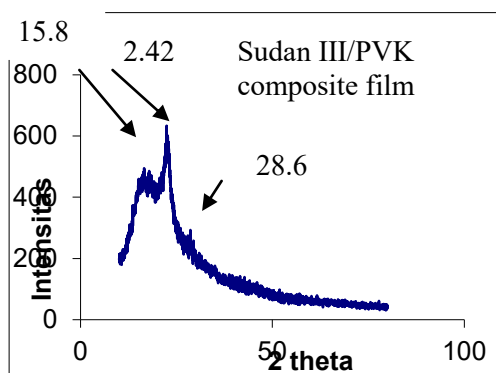


Figure 3.4. The XRD spectra of Sudan III/PVK (% weight 1:2) composite film.

Table 3.4. Bragg angle (2θ) in degree and crystal plane distance in $d_{(hkl)}$ in Angstrom of Sudan III/PVK (% weight 1:2) composite film.

	Sudan III/PVK (% weight 1:2) composite film		
2θ	15.8	22.42	28.6
$d_{(hkl)}$ (Å)	5.60	3.96	3.12

XRD spectra of Sudan III/PVK (% weight 1:4) composite film is shown in figure 3.5.

The crystallinity index (CI) is calculated according to Segal peak height [9] with intensity method calculation is:

$$CI = (I_c - I_{am}) / I_c \quad 3.3.$$

From figure 3.4 can be calculated the crystallinity index [9] that is $CI = (I_c - I_{am}) / I_c = (634 - 420) / 634 = 0.337$.

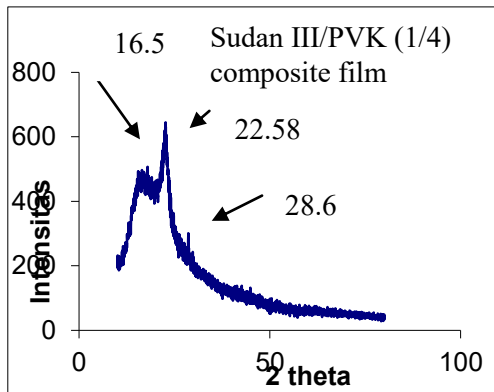


Figure 3.5. The XRD spectra of Sudan III/PVK (% weight 1:4) composite film.

Table 3.5. Bragg angle (2θ) in degree and crystal plane distance in $d_{(hkl)}$ in Angstrom of Sudan III/PVK (% weight (1:4) composite film.

	Sudan III/PVK (% weight (1:4) composite film		
2θ	16,5	22,58	28,6
$d_{(hkl)}$ (Å)	5,37	3,95	3,12

From figure 3.5, so the CI for Sudan III/PVK (% weight 1:4) composite film is obtained $CI = (I_c - I_{am}) / I_c = (635 - 423) / 635 = 0.334$.

The XRD spectra of Sudan III/PVK composite film exposure to acetone vapor is shown in figure 3.6.

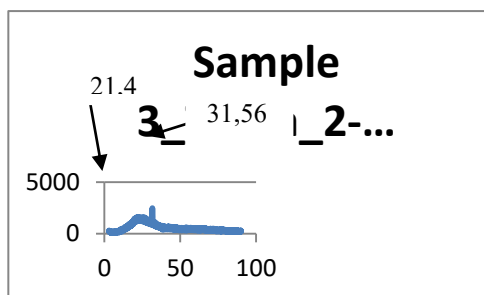


Figure 3.6. XRD spectra of Sudan III/PVK composite film exposure to acetone vapor.

Table 3.6. Bragg angle (2θ) in degree and crystal plane distance in $d_{(hkl)}$ in Angstrom of Sudan III/PVK (% weight (1:4) composite film exposure to acetone vapor).

	Sudan III/PVK (% weight 1:2) exposure to acetone vapor	
2θ	21.4	31.56
$d_{(hkl)}$ (Å)	4.15	2.83
S (nm)		56.6
CI		0.52
D (Å)	0.40	

The XRD spectra of Sudan III/PVK composite film exposure to alcohol vapor is shown in figure 3.7.

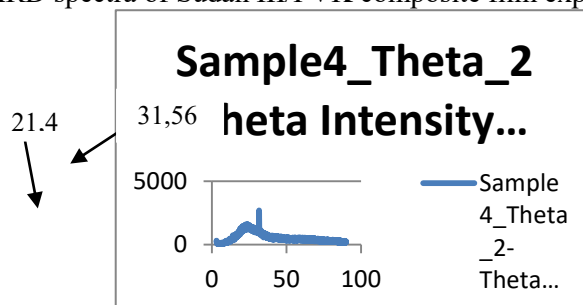


Figure 3.7. XRD spectra of Sudan III/PVK composite film exposure to alcohol vapor.

Table 3.7. Bragg angle (2θ) in degree and crystal plane distance in $d_{(hkl)}$ in Angstrom of Sudan III/PVK (% weight (1:4) composite film exposure to alcohol vapor).

	Sudan III/PVK composite film (% weight 1:2) exposure to alcohol vapor	
2θ	21.4	31.56
$d_{(hkl)}$ (Å)	4.15	2.83
S (nm)		56.6
CI		0.64
D (Å)	0.40	

Figure 3.7 shows wide peak at around $2\theta=21.4^\circ$, $2\theta = 31.56^\circ$. This X-ray diffraction peak with height intensity is attributed to Sudan III contribution.

The average polymer chain separation (S) for Sudan III/PVK composite film exposure to alcohol vapor was found to be 56.6 nm. And this is the same value of S for Sudan III/PVK composite film exposure to acetone vapor.

Crystallinity index (CI) for Sudan III/PVK composite film exposure to alcohol vapor was found to be 0.64. Meanwhile for Sudan III/PVK composite film exposure to acetone vapor is 0.52. So CI for Sudan III/PVK composite film exposure to alcohol vapor is higher around 19% c.f CI for Sudan III/PVK composite film exposure to acetone vapor.

The average crystal size can be estimated from a sharp peak at $2\theta = 22.42^\circ$ for Sudan III/PVK composite film (% weight 1:2 (figure 3.4). By using Scherrer's formula [10,11] :

$$D = K\lambda / \beta \cos \theta \quad 3.4.$$

With D is crystal size, K is shape factor, $K = 0,89$. θ is diffraction angle at maximum intensity, and β is FWHM diffraction angle in radian (1 degree = 0.0174 radian). From this formula can be obtained the average crystal size for Sudan III/PVK (% weight 1:2) is 40 nm. And also for Sudan III/PVK (% weight 1:4) composite film is 40 nm.

4. Conclusions

From the XRD spectra can be obtained the average chain separation for PVK powder and in form of film are 57.44 nm and 49.48 nm respectively. And for Sudan III/PVK (1/2) composite film and Sudan III/PVK (1/4) composite film are 49.53 nm and 49.18 nm respectively.

From the XRD spectra can be also obtained that degree of crystallinity or crystallinity index for Sudan III/PVK (1/2) composite film and Sudan III/PVK (1/4) composite film are 0.337 nm dan 0.334 nm respectively.

From the XRD spectra can be obtained that the crystallite size for Sudan III/PVK (1/2) composite film exposure to alcohol vapor and for Sudan III/PVK (1/4) composite film exposure to acetone vapor are the same that are 40 nm (0.4 Angstrom).

5. Acknowledgements

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