Research Paper Engineering

X-ray Diffraction Studies of Conducting Macromolecules

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ABSTRACT

In the X-ray analysis of polymers the diffraction spots are board and diminish rapidly with increasing diffraction angle .X-ray spectroscopy, like optical spectroscopy is based upon measurement of emission absorption scattering fluorescence and diffraction of electromagnetic radiations. Such measurements provide much useful information about the composition and structure of matter [6,7,8] To study the structural properties of polymeric material X-ray diffraction measurements are performed through the interaction of X-ray the structure of the compound may be thoroughly investigated. The X-ray diffraction theory based on single crystals of low- molecular weight substances is not applicable in the explicit treatment of polymer samples. There are a few studies in this direction [9, 10, 11] X-ray diffraction formulas are sorted to kinds, A and B as follow –

(A) X-ray diffraction intensity by an infinitely large crystal.

(B) X-ray diffraction intensity by a finitely large crystal. X-ray studies of Polymers and their complexes have been done. On basis of X-ray diffractograms it is easy to explain the Composition and structure of polymeric samples. It is also possible to explain the increasing conductance of polymeric electrolytes.

X-ray diffraction method makes it possible to analyze the crystal structure of real crystallites which actually exists in synthetic fibers and plastics and thereby the structural property relation of polymer materials will be considered in more detail.

Keywords:

Introduction-

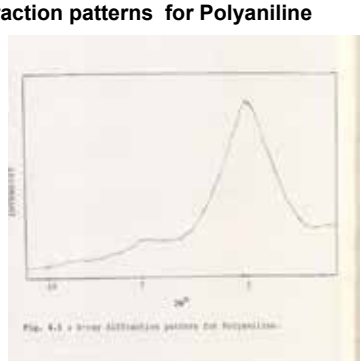
In the past seven decade's X-ray diffraction and other derived techniques viz- .DPP, cyclic voltammetry etc.have gained popularity among the analytical scientists for the qualitative and quantitative analysis of almost all types of organic substances. X-ray diffraction measurements are performed through the interaction of X-ray the structure of the compound may be thoroughly investigated. The X-ray diffraction theory based on single crystals of low- molecular weight substances is not applicable in the explicit treatment of polymer samplesThe polarographic parameter i.e. half wave potential $E_{1/2}$ plays a vital role in the polarographic techniques. Since the half wave potential is the characteristic of the electro active species which helps to analyze the electro active species qualitatively and quantitatively. The temperature dependence of the polyaniline film voltammetric response in aqueous and non aqueous by Q.Inselt[4] He observed that only a very slight shift into the direction of more negative potentials (Ca-10 mV) and a small increase in the temperature is increased by 30^0C

Preparation of polymer complex

An adequate quantity of the polyaniline host and the inorganic salts of Zn were separately dissolved in suitable solvent (e.g. acetonitrile) the two solutions were then mixed and after stirring the solvent were slowly evaporated to finally obtain powder form of polymeric complexes. Polyaniline sulphate polymer sulphate was prepared by chemical method applying oxidant (Potassium dichromate) the polymerization of 0.4 moles of aniline in 1lit. of 1M sulphuric acid was affected using 1g equivalent of the potassium dichromate a precipitated was separated, washed, dried and weighed as polyaniline sulphate

Polyaniline Chloride was prepared by equilibrating the polyaniline sulphate with1M HCl for about 10hrs. The mass so obtained was separated, washed and dried and weighed as polyaniline Chloride

X-ray Diffraction patterns for Polyaniline



X-Ray Diffraction signals Polyaniline and its complexes with Zink and Aluminium metals

Aluminium		Zink		Polyaniline		Zink-polyaniline		Aluminium-polyaniline	
2θ	d(A^0)	2θ	d(A^0)	2θ	d(A^0)	2θ	d(A^0)	2θ	d(A^0)
38.45	2.338	36.28	2.473	3	14.68	18.8	4.718	3	14.68
44.7	2.024	38.9	2.308			20.38	4.353	6.25	7.5
65.10	1.431	43.2	2.091			23.82	4.127	9	4.9
69.19	1.221	54.3	1.187			26.25	3.732	10.62	4.15
						29.32	3.392		

						31.99	3.043		
						35.1	2.795		
						35.77	2.554		
						42.97	2.508		

Experimental – The X- ray Diffraction
studies on the polymers/electrolytes /electrodes were carried out using a RIGAKU X-Ray Diffractometer (Japan) . The X-ray beam was Nickle filtered Cu KX Radiation from a seald tube operated at 40 KV and 50mA.

All the Chemicals used were of anala R/BDH grade.

0.01 M metal (Zn^{++}) solutions were prepared by dissolving the requisite quantity of their soluble salts in double distilled water 0.1 M PANI solutions were prepared in small amount of 0.02 0.03 hydrochloric acid diluted to required volume with distilled water.

Experimental sets of solutions

containing overall concentration of supporting electrolyte (KCl) and Metal ion fixed at 0.1 M and 1.0 mM respectively. Whereas in other sets in addition to the above supporting electrolyte and metal ion concentration of each polymer (ligand) was varied . Polarograms were recorded on an ELICO (Hyderabad) pulse polarograph ModelCL-90 having a dropping mercury electrode (DME) a saturated calomel electrode (SCE) a working electrode as a working electrode reference electrode respectively. The DME had a characteristics of m=2.33 mgs^{-1} t=3.03at 40cm effective height of mercury column ,$m^{2/3} t^{1/6}$=2.13$mg^{2/3\ s-1/2}$

Survey of literature –
W.John Albery[3], et.al have used electrode such as polyaniline, polypyrol and polythiophene. They showed that the behavior of the different polymers is similar and may be explained by a chemical model involving localized redox species with two possible conformations of the polymer.

The temperature dependence of the polyaniline film voltammetric response in aqueous and non aqueous media has been investigated by G.Inzelt[2] .He observed that only a very slight shift into the direction of more negative potentials in the peak potentials (Ca -10mv) and a small increase in the peak current as the temperature is increased by 30^0C.

Youn Chaol on Park Yong Woo studied behaviour Polyaniline and found that the electrons are moving in and out changing the polyaniline structure from one form to the another form

C. Herold 12 Yazmi, DBillaud attempted study of sodium doped polyparaphenylene film, John Alberry, et.al

Result and Discussion-
X-ray diffraction method makes it possible to analyze the crystal structure of real crystallites which actually exists in synthetic fibers and plastics and thereby the structural property relation of polymer materials will be considered in more detail. On the basis of X-ray diffractograms it is easy to explain the Composition and structure of polymeric samples. It is also possible to explain the increasing conductance of polymeric electrolytes. X-ray diffraction measurements are performed through the interaction of X-ray the structure of the compound may be thoroughly investigated. The X-ray diffraction theory based on single crystals of low- molecular weight substances is not applicable in the explicit treatment of polymer samples.

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