

Short Communication

New Synthesis and Small Angle Neutron Scattering Studies of Polyaniline Nanofibers

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Abstract: Nano structured polyaniline (PANI) is synthesized for the first time by polymerization of aniline in the presence of hydrochloric acid as a catalyst and ammoniumperoxodisulphate as an oxidant by the chemical oxidative polymerization method. The product powder was pelletized with help of hydraulic machine to study the XRD, SEM and SANS. The XRD pattern indicates semi crystalline nature of PANI with interplanar distance 4.210 Å and 3.774 Å respectively. The SEM pictures shows fiber like nature of particles. SANS experiment is used to find different parameters of these crystalline polymer particles. Dynamic light Scattering (DLS) studies are performed to find the sizes of nano structured polyaniline. The length and radius of nano size polyaniline are identified as 617.6 nm and 75.05 nm respectively.

Keywords: NANO STRUCTURED PANI, XRD, SEM, SANS, DLS.

1. INTRODUCTION

Conducting polymers opened the way to progress in understanding the fundamental chemistry and physics of π -bonded macromolecules [1]. Among various types of π -electron systems, the electrical and optical properties of conjugated polymers have shown wide range of exciting features, leading towards applications like light emitting diodes, photodiodes, electronic circuits, lasers, sensors, solar cells, etc. Since polymeric materials like polyaniline have inherently partially crystalline and partially amorphous, the structure and morphology play significant roles in the electronic properties. Determination of size and morphology is an important subject in the nanoscience and nanotechnology. There are different experimental techniques (XRD, SEM, TEM and DLS) to find size and morphology of these polymers. Among these, the small angle neutron scattering (SANS) is a powerful and sophisticated experimental technique which can be used to find many physical parameters of Polymers [2-4].

2. EXPERIMENTAL SECTION

2.1. Synthesis of Polyaniline

Polyaniline was synthesized by *In-Situ* chemical polymerization of aniline in the presence of hydrochloric acid as a catalyst and ammoniumperoxodisulphate as an oxidant. For the synthesis, we took 50ml, 1M HCl, and 2 ml of aniline were added into a 250ml

beaker equipped with a Teflon coated magnetic stirrer at about 0°C temperature. Then 5 g of ammoniumperdisulphate ((NH₄)₂S₂O₈) aqueous solution in 50ml 1M HCl was drop wise added into the above solution. The polymerization temperature 0° C was maintained for 10 h to complete the reaction. Then the precipitate obtained was filtered. The product was washed successively by 1M HCl followed by double distilled water until the wash solution turned colorless. The product PANI was dried at 60°C for 24h to get powder form PANI. This powder was pelletized with help of hydraulic machine for characterization.

2.2. Characterization

The X-Ray diffraction studies of polyaniline were performed on Phillips XPERT diffractometer with Cu K_α X-ray ($\lambda = 1.54 \text{ \AA}$). Scanning electron microscope (SEM) experiments are performed by employing Hitachi SU 1510 microscope. Small angle neutron scattering (SANS) experiment was conducted at National facility for neutron beam research on Dhruva reactor at Bhabha Atomic Research Center (BARC), Mumbai, India. Malvern DLS instrument is employed to find the morphology and size distribution of polyaniline nanofibers by mixing the polymer in double distilled water.

3. RESULTS AND DISCUSSION

The X-ray diffraction pattern of polyaniline emeraldine salt form shows two sharp peaks at $2\theta = 21.10$ and 23.59 . The interplanar distances are calculated as 42.10 nm and 37.74 nm respec-

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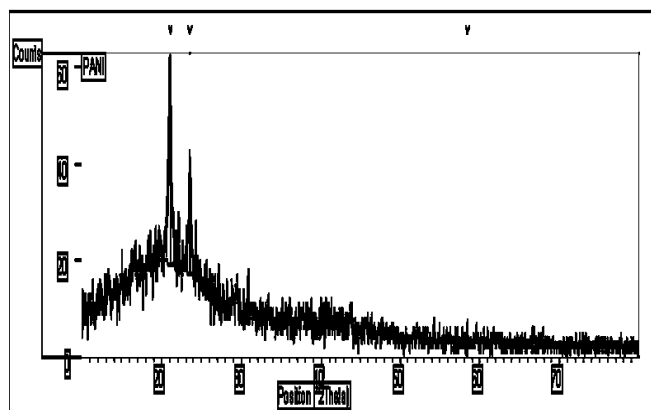


Figure 1. XRD Pattern of Polyaniline nanofibers

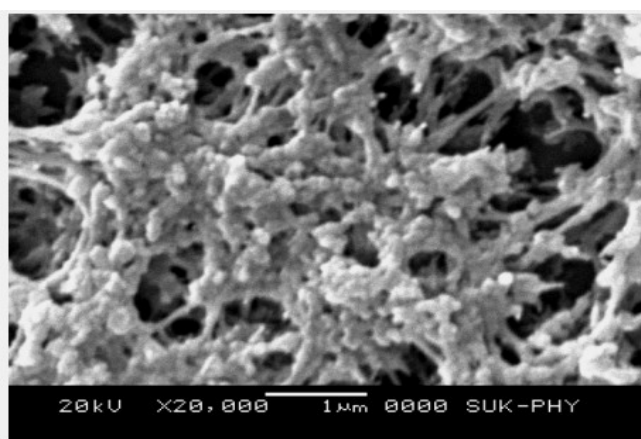


Figure 2. SEM Micrograph of Polyaniline

tively. The average crystallite size is calculated as 8.021 nm by Debye Scherer equation $D = [0.89 \lambda] / [\beta \cos \theta]$ respectively, where β is full width of half maxima (FWHM) [Fig.1].

Generally polymers are considered to be amorphous, but here the synthesized polymer is showing crystalline structure due to their fiber nature and planar nature of Benzenoid and $\alpha\alpha$ Quinoid functional groups. The SEM micrographs reveal that they are actually agglomeration of nanofibers [Fig.2]. The diameter and length of nanofibers are measured as 80 nm and 550nm respectively.

SANS experiment is used to find different parameters of these crystalline polymer particles. In SANS experiment intensity of Scattered neutrons $I(Q)$ is measured as function of the scattering vector $Q = (4\pi/\lambda) \sin\theta$ [Fig.3]. The radius of gyration of PANI is obtained as $R_g = 52.09\text{nm}$. Length of the particles is calculated as 565.34nm. Similarly the surface fractal dimension (D_s) of these particles is calculated as 2.6 using the equation [5, 6].

$$\ln I(Q) = \ln I_0 - \alpha \ln Q$$

Where α is a constant and the surface fractal dimension $D_s = 6 - \alpha$. This surface fractal dimension indicates the surface morphology of polyaniline. The D_s values lies between 2 and 3. If $D_s = 2$ the surface is considered to be very smooth, if it is equal to 3 the sur-

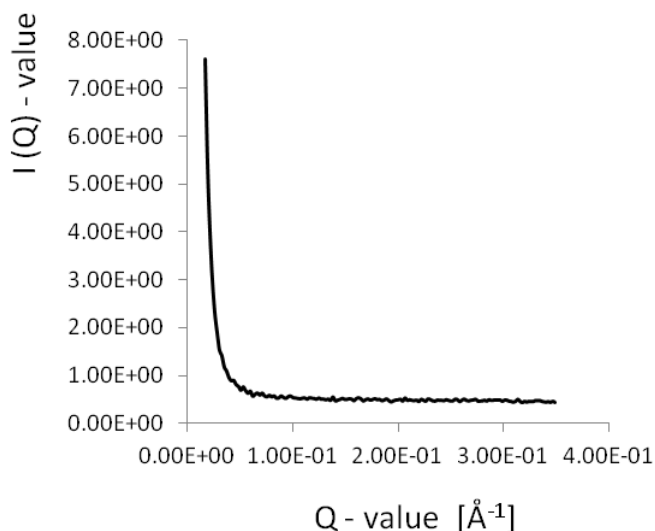


Figure 3. SANS Guinier plot of Polyaniline

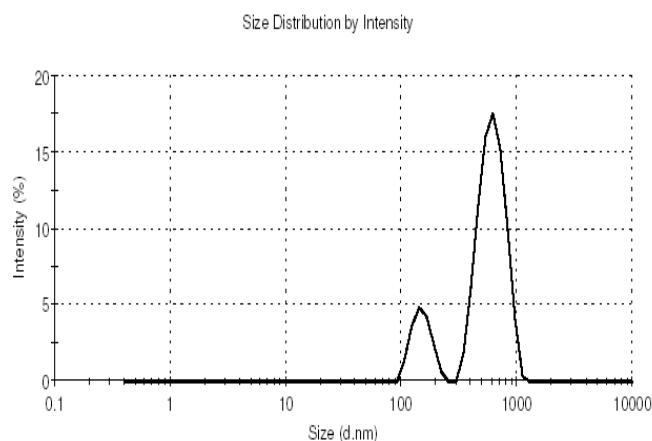


Figure 4. DLS size distribution curve of Polyaniline

face is considered as very rough [3-7]. The SANS results indicates that the Polyaniline fiber surface is rough, these results are quite similar to SEM results. In Dynamic light scattering (DLS) studies, the effective hydrodynamic diameter of polyaniline nanofiber particles size distribution curve shows two peaks at 150.1 nm and 617.6 nm [Fig.4]. They are attributed to diameter and lengths of PANI nano-fibers [8]. The DLS results are little bit more compare to the SEM and SANS studies, this may be due to swelling of the polymer nanofibers in double distilled water [9].

4. CONCLUSIONS

We successfully synthesized Polyaniline emeraldine salt form by chemical oxidation of aniline. The XRD studies reveal semi-crystalline nature, and SEM results are indicating fiber type morphology of polyaniline. SANS and DLS studies are used to find different physical parameters of polyaniline like radius of gyration, length and surface fractal dimensions.

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