

# Synthesis of Nanostructured Polyaniline in Dodecyl Sulphuric Acid (DSA) mediated Micellar Medium with Isopropyl Alcohol as Cosurfactant

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## Abstract

*Intrinsically conducting polymers including polyaniline (PANI) have received considerable attention owing to their wide ranging applications. An emerging area of research in conducting polymers is the nanostructured conducting polymers. Nanostructured polyaniline<sup>1</sup> has been synthesized by the use of hard templates<sup>2,3</sup>, soft templates<sup>4</sup>, electro-spinning<sup>5,6</sup>, interfacial polymerization<sup>1,7</sup>, emulsion polymerization<sup>8,9</sup> etc. Here in, we report the synthesis of nanostructured polyaniline in a micellar medium based on dodecyl sulphuric acid (DSA), acting as surfactant as well as dopant and cosurfactant isopropyl alcohol at varying surfactant molar concentration and ammonium peroxydisulfate (APS) to aniline molar ratios. The nanostructured polyaniline was characterized by UV-Vis spectroscopy, Fourier transformed infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The distribution of particle size was determined by dynamic light scattering (DLS) studies. The conductivity was measured by the standard Four-Probe technique.*

**Keywords:** Conducting polymer, polyaniline, nanostructured, transmission electron microscopy.

## Introduction

Nanostructured materials have created interest among scientists of different hues primarily due to their unusual properties and potential applications, besides the interdisciplinary nature of the subject. A considerable contribution to the advancement of this field has been made by chemists working primarily on the goal to design and control nanostructures and thereby to functionalize them using both room-temperature solution-based pathways and high-temperature (thermodynamic) methods. The micellar medium method with cosurfactant is one among the room temperature routes to synthesize nanoparticles.

Polyaniline (PANI) is one of the most important and widely studied intrinsically conducting polymers<sup>10-12</sup> due to its good environmental stability, easy method of preparation (e.g. by the oxidation of aniline with APS in acidic aqueous medium), interesting electrical and optical properties compared to the other conducting polymers such as polypyrrole, polythiophene, polyacetylene and

polyphenylenevinylene. Typically, conducting polyaniline is synthesized chemically or electrochemically in acidic solutions and is not soluble in common organic solvents. The insolubility in common organic solvents creates difficulty in the processability of polyaniline. Polyaniline doped with HCl or H<sub>2</sub>SO<sub>4</sub> acid is difficult to process because it cannot be dissolved or melted below the decomposition temperature in the conducting state. The protonation of PANI with organic acids having a bulky hydrocarbon component has been used to increase the solubility of PANI in organic solvents and the plasticity. The use of dodecylbenzenesulfonic acid<sup>13-15</sup>, dinonylnaphthalenesulfonic acid<sup>16</sup> or diesters of sulfosuccinic acid<sup>17,18</sup> may serve as examples.

Various surfactants have also been used as a component of the reaction mixture for the same purpose.<sup>19,20</sup> Alternative processing strategies consist in coating of the surfaces of various substrates with a conducting PANI film and in the preparation of PANI colloids<sup>21,22</sup>. The preparation of polyaniline in colloidal form containing nanoparticles of polyaniline is an important way to improve the processability of this polymer.<sup>23</sup> Kuramoto et al<sup>8</sup> have successfully synthesized polyaniline nanoparticles in sodium dodecyl sulphate (SDS) and dodecylbenzene sulphonic acid (DBSA) based micellar medium using ammonium peroxydisulphate (APS) as initiator. Similarly, Kim et al<sup>19,23</sup> synthesised colloidal dispersions of PANI particles via dispersion polymerization using SDS as surfactant in acidic medium and a strong oxidizing agent, mainly APS or potassium persulphate (KPS). However, the morphology control was rather poor. Though SDS was used extensively by many chemists, there is no report of using the corresponding acid as the surfactant in the synthesis of nanostructured polyaniline.

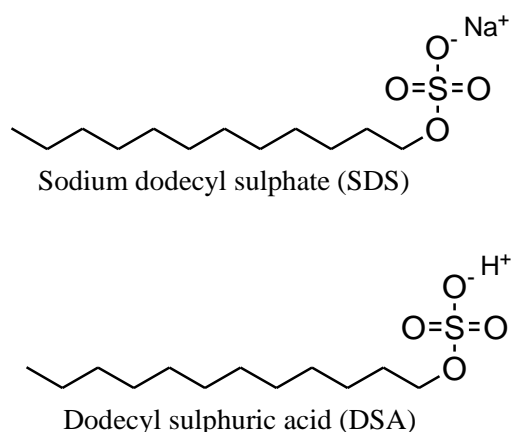
It was further observed that for the appropriate packing of amphiphiles (surfactant molecules) to form stable micelles, surface active substances, in addition to surfactants are often used. These are generally short-chain alcohols or amines commonly referred to as cosurfactants. The role of cosurfactant is to lower the interfacial tension between surfactant and water for the spontaneous formation of surfactant aggregates. Marchand et al<sup>24</sup> carried out in detailed investigation on the effect of cosurfactant on the micellar size using NP-5 as surfactant and short chain amines as cosurfactant. They observed a substantial decrease in average micellar size. This was attributed to the higher fluidity of the interfacial film and a higher mean

curvature of radius, which in turn influences the intermicellar exchange.

A high exchange rate implies the higher consumption of reactants at the nucleation stage, thus reducing the effective concentration for further growth and results in smaller nanoparticles. Curri et al<sup>25</sup> have studied the effect of pentanol as a co-surfactant on the synthesis of cadmium sulphide nanoparticles using the CTAB/hexane/ water system. A pronounced effect was found on the size, size distribution and stability of crystallites on addition of co-surfactant. It can be summarized that the presence of cosurfactant promotes the micelle formation even below critical micellar concentration, stabilizes the micelle formed and controls the size of particle size.

## Material and Methods

**Materials:** Aniline monomers (99.9%) were purchased from Merck, distilled twice at low pressure and stored in dark in dark coloured bottle before use. SDS (99%, Sigma), Amberlite IR 120 resin from Sigma Aldrich, Isopropanol and APS from Merck are all used as received. As dodecyl sulphuric acid (Figure 1) is not commercially available, we prepared it from the commercially available SDS by passing a 6% aqueous solution (W/V) of it through column containing a cation exchange resin Amberlite IR 120.



**Figure 1: Structure of SDS and its corresponding acid (DS acid)**

Stock solution of DSA was standardized by using standard  $\text{Na}_2\text{CO}_3$  solution and methyl orange as indicator. The Critical Micelle Concentration of DSA was determined by the conductivity measurements of the acid. The CMC value was found at  $7.2 \times 10^{-3}\text{M}$  of dodecyl sulphuric acid at room temperature.

**Synthesis of PANI dispersion:** To a 50mL dodecyl sulphuric acid solution (concentration ranging from 0.001M to 0.2M), calculated amount of (in mole) aniline was added and stirred for 30minutes to dissolve at room temperature. To it 25mL of isopropyl alcohol (cosurfactant) was added. Then calculated amount of (in mole) APS dissolved in 25mL of varying concentration of dodecyl sulphuric acid

(i.e. from 0.001M to 0.2M) was added under constant stirring to the above solution. The resulting solution was kept undisturbed overnight. Figure 2 shows the schematic representation of preparation of green coloured PANI dispersions. The whole procedure was also repeated for varying aniline to APS molar ratios at a constant concentration of dodecyl sulphuric acid. Stable dispersions were formed at or above critical micellar concentrations. Detailed synthesis of nanostructured PANI is presented in tables 1 and 2.

**Characterization of PANI nanoparticles:** The stable green coloured nanoparticles of PANI so formed were characterized in detail by standard techniques. Scanning electron micrographs (SEM) were obtained from a SEM instrument LEO 1430 VP, operated at 10.00 kV. The magnification was from 7.00 KX to 17.41 KX. For scanning electron microscopic investigation, slides were prepared on thin glass plates of the size of  $1\text{ cm}^2$ , wrapped with the shining face of aluminium foil and smoothed as much as possible. A minute drop of the dispersion from a capillary tube was placed on the slide and dried in a vacuum desiccator. Transmission electron microscopic (TEM) studies were carried out using a JEOL-JEM 100C X II instrument operated at an accelerating voltage of 100 kV. TEM specimens were prepared by placing a small drop of the diluted (by adding cyclohexane) sample on a carbon coated copper grid and then dried carefully.

The light scattering experiments were performed in a dynamic light scattering particle size analyser instrument – Zetasizer 3000 HS<sub>A</sub>. The UV – Vis spectra of the samples were recorded in a Perkin Elmer spectrophotometer in the range 200–900 nm. Partially full cuvette of polyaniline dispersion was directly taken on the photometer to record the UV–Vis spectra. The FTIR spectra of the samples were performed on a Perkin Elmer spectrophotometer using KBr disk in the range  $4000\text{--}400\text{cm}^{-1}$  with a scan rate of 16 and resolution of  $4.0\text{cm}^{-1}$ . The conductivity of polyaniline nanoparticles was measured by the standard four-probe method. A thin film of the polyaniline dispersion was deposited on a glass plate, whose conductivity was then measured. At least four readings were taken at different points of the thin film, the average of which was taken to measure the conductivity value.

## Results and Discussion

**Polymerization of Aniline:** Polymerization of aniline was done in a micellar medium. Aniline was dissolved in dodecyl sulphuric acid solution to which the co-surfactant isopropanol was added. To this, ammonium peroxydisulphate dissolved in dodecyl sulphuric acid was added under constant stirring. The formation of stable colloidal dispersion of PANI-nanoparticles was found to be dependent on two factors, viz. the CMC of dodecyl sulphuric acid and the APS to aniline molar ratio (table 1 and 2). The CMC value of dodecyl sulphuric acid is an important criteria and below this value no stable dispersion was

formed. Further presence of co-surfactant strengthens the micelle formed and facilitates the formation of stable dispersions at such dodecyl sulphuric acid concentration (bold entries table 1) which was not possible in absence of co-surfactant (bold entries table 2). It was also observed that the formation of stable dispersion was also dependent on the APS to aniline molar ratio and the most suitable ratio was found to be 1.0 and 1.5. When formed the dispersions were stable even under centrifugation at 12 000 rpm.

**Particle Size and Morphology:** The morphology and particle size were investigated by SEM, TEM and DLS methods. The scanning electron micrograph (SEM) of the polyaniline colloidal dispersion is shown in figure 3. It shows the spherical morphology of the polyaniline particles with the size ranging from 37nm to 48nm. SEM images A and B are for PANI dispersions obtained by using cosurfactant with dodecyl sulphuric acid conc. 0.05 M at APS to aniline molar ratio 1.5 and 1.0 respectively and C & D are those in the absence of cosurfactant. The average particle sizes are 48, 37, 59 and 98nm for A, B, C and D respectively. SEM images clearly reveals that addition of cosurfactant reduces the particle size of PANI nanoparticles.

Figure 4 shows the TEM images of PANI nanoparticles in the colloidal dispersions. The spherical polymeric nanoparticles were produced and the sizes are 20 to 35nm. It is observed that the rise in APS to aniline molar ratio value resulted in the rise in the size of PANI particles. TEM images A and B are for PANI dispersions obtained by using isopropanol as cosurfactant for DSA conc. 0.05M at APS to aniline molar ratio 1.0 and 1.5 respectively and C and D are those in the absence of cosurfactant. The average particle sizes are 18, 25, 50 and 30nm for A, B, C and D respectively.

TEM micrographs clearly indicate that addition of cosurfactant reduces the particle size of PANI nanoparticles. The particle sizes and their distribution were also investigated by the dynamic light scattering (DLS) method. The results of DLS studies are presented in figure 5. Figure shows that majority of the PANI nanoparticles were within 50nm (figure 5A) in presence of cosurfactant and in the absence of cosurfactant these are within 100nm (figure 5B). The particle sizes observed by dynamic light scattering are in line with the values observed by the TEM. However, a slight increase in size was detected in dynamic light scattering method as it reflects the hydrodynamic volume rather than the actual particle size.

**UV – Visible Absorption Spectra:** The UV – Vis Spectra of polyaniline dispersions synthesized from using isopropanol as cosurfactant in micellar medium are shown in figure 6. The UV-Vis spectra of all the samples show similar features with very little variations. The absorption peaks in the region 300–350nm are due to  $\pi \rightarrow \pi^*$  electron

transition within the benzenoid segment. The peak at 400–440nm and 800nm are due to polaron  $-\pi^*$  and  $\pi$ - polaron band transition which indicates the doped status of PANI.<sup>26, 27</sup>

**FTIR–Spectra:** The FTIR spectrum of polyaniline nanoparticles is shown in figure 7. It shows all the characteristic bands of polyaniline. The N–H stretching band is observed at  $3418\text{cm}^{-1}$ . The bands near  $1456$  and  $1540\text{cm}^{-1}$  are assigned to C = C stretching of benzenoid and quinoid rings respectively. These bands appear at lower wave number for undoped PANI than were observed near  $1500$  and  $1600\text{cm}^{-1}$  for undoped PANI. This was usual for well doped PANI. The peak at  $1234\text{cm}^{-1}$ , which is characteristic of the conducting PANI–ES form, is also seen<sup>14,28</sup>. The peaks ranging from  $2800$  to  $3000\text{cm}^{-1}$  are assigned to aliphatic C–H stretching mode.

**Conductivity:** The conductivity of PANI depends on the degree of doping, oxidation state, particle morphology, crystallinity, inter- or intra-chain interactions, molecular weight etc. The particle size has an effect on the conductivity of PANI<sup>19,23</sup>. A decrease in the particle size facilitates more effective doping resulting in an increase in the conductivity value. In the present case also the conductivity is marginally increased with lowering of particle size of PANI. The conductivity values of PANI dispersions in  $\text{S cm}^{-1}$  were shown in the table 3. From the table it is evident that conductivities marginally increased for the PANI dispersions prepared by using isopropanol cosurfactant and probably it is due to the decrease in particle size due to the addition of cosurfactant. Conductivity values are in good agreement with the values reported by other researchers<sup>29,30</sup>. The variation of conductivity with DSA concentration at APS to aniline molar ratio 1.5 is shown in figure 8(a) for the PANI dispersions prepared in presence of isopropanol cosurfactant and those in the absence of cosurfactant is shown in figure 8(b).

## Conclusion

Polyaniline nanoparticles were successfully synthesized in a dodecyl sulphuric acid based micellar medium in which dodecyl sulphuric acid functioned both as a surfactant and a dopant. As the DSA content increased, the polymerization rate increased due to lower pH value and enhanced micelle formation. The green coloured colloidal dispersion has good long-term stability and exists as stable dispersion without precipitation over one year. The PANI nanoparticles synthesized in this way are spherical in nature and the majority particles are in the range of 20 to 30nm. Further the sizes of nanoparticles were found to be dependent on the CMC value of the surfactant acid and APS to aniline molar ratio. The conductivity value is also dependent on the size of the PANI nanoparticles.

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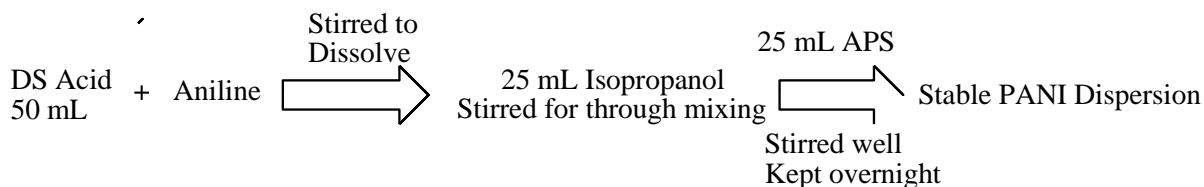


Figure 2: Schematic representation of synthesis of PANI dispersion

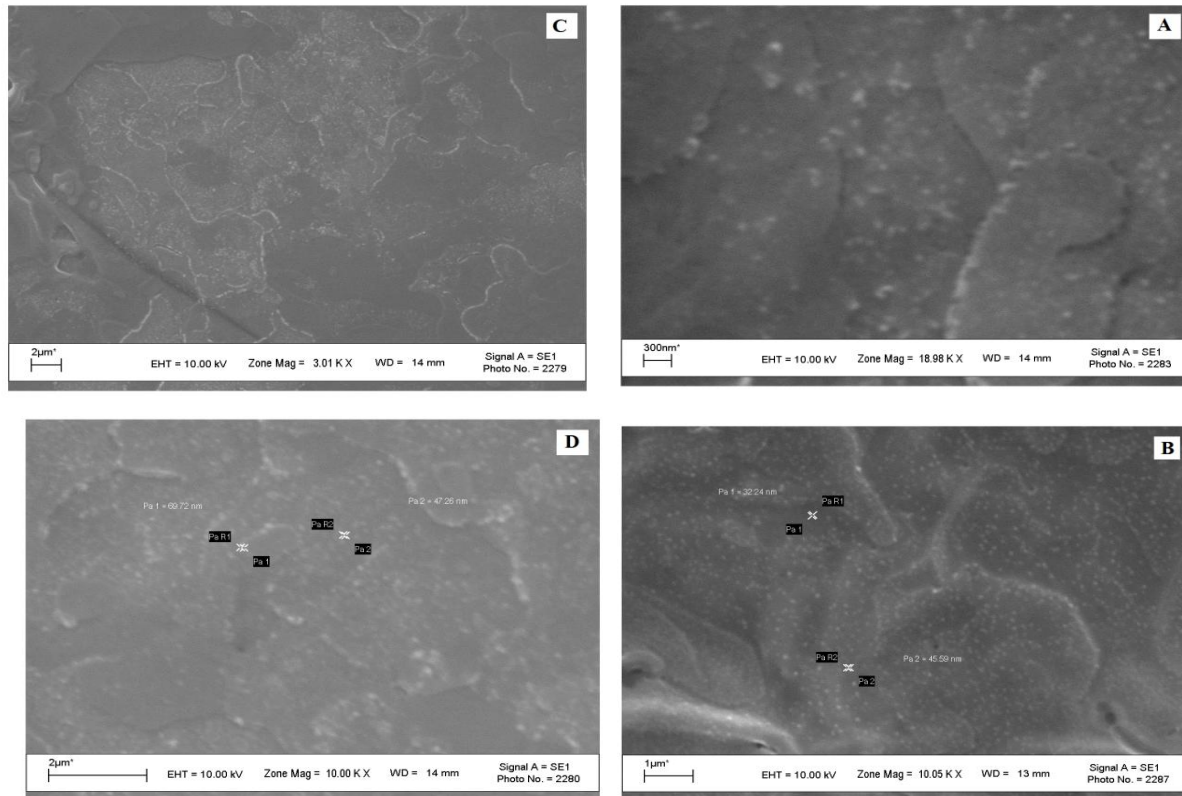


Figure 3: SEM images of Nanostructured PANI

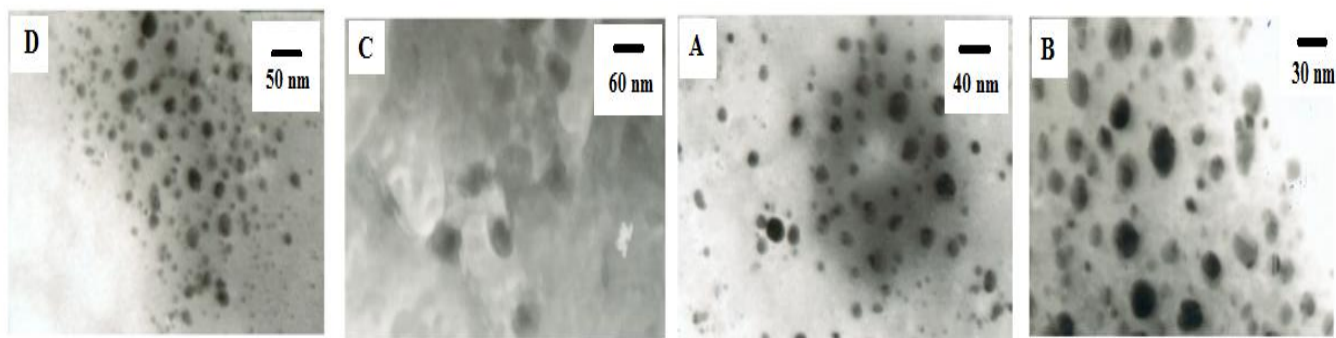


Figure 4: TEM of Nanostructured PANI

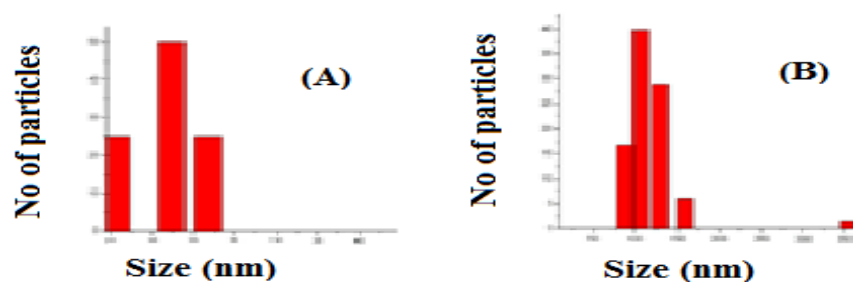


Figure 5: Particle size analysis by DLS

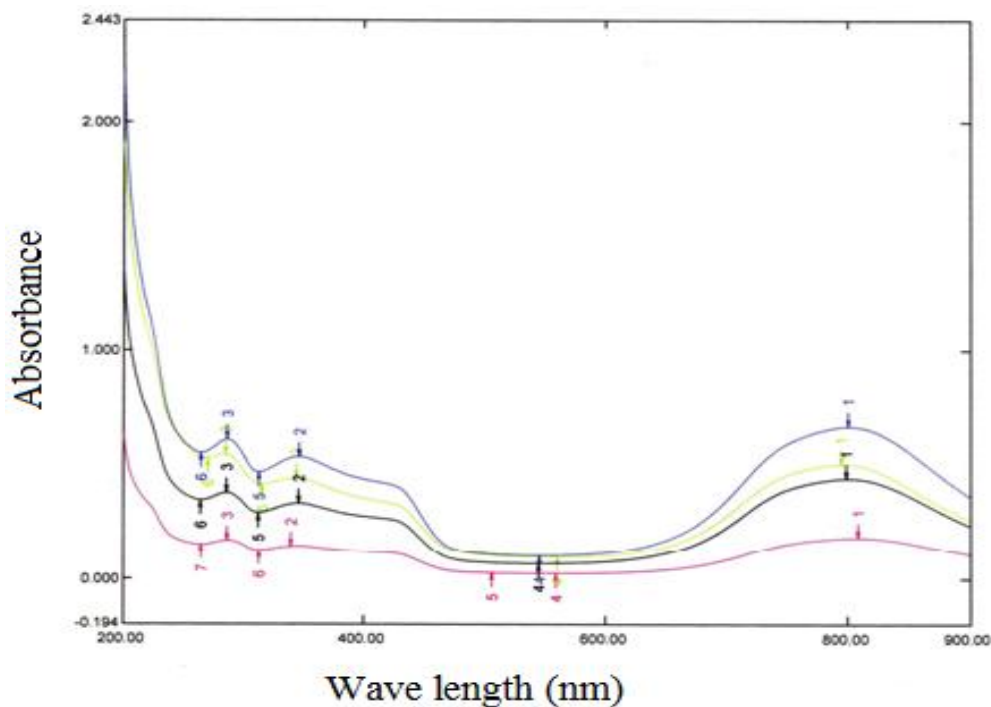


Figure 6: UV-VIS spectra of Nanostructured PANI

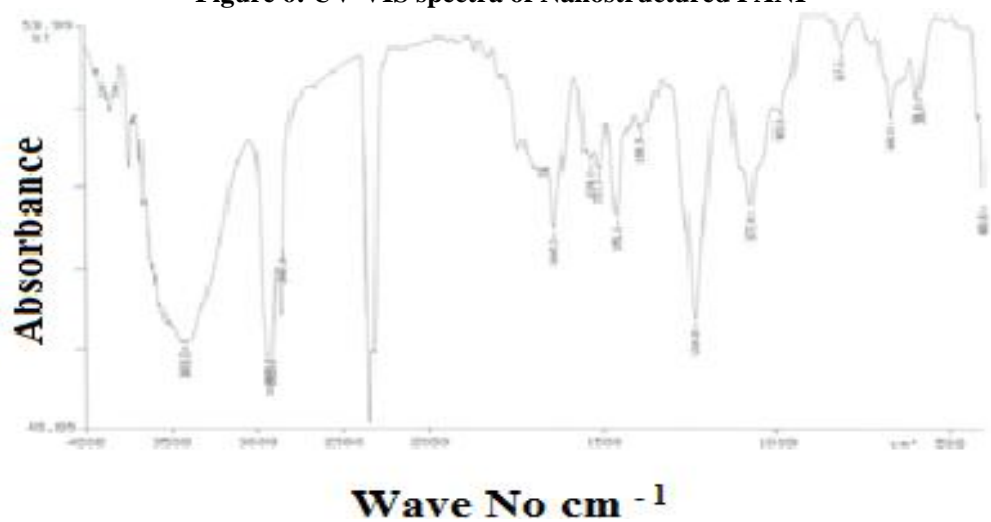


Figure 7: FTIR spectra of Nanostructured PANI

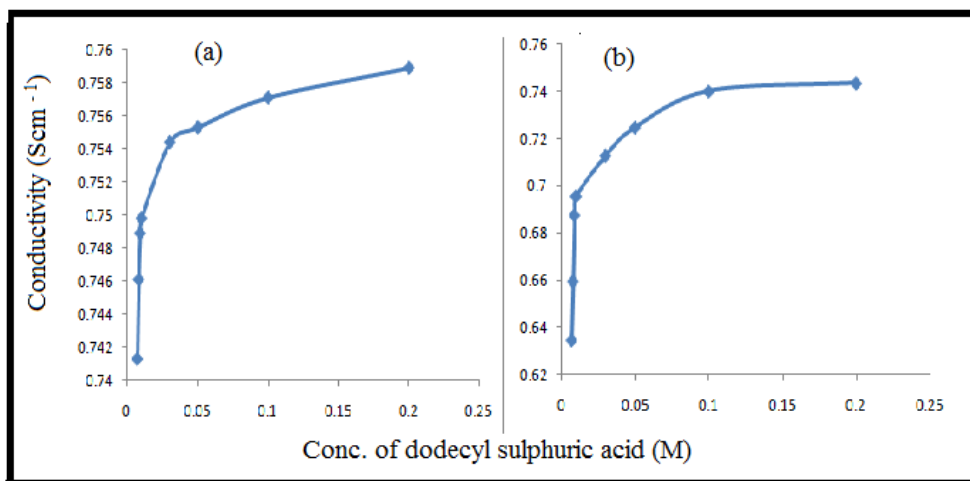


Figure 8: Variation of conductivity with dodecyl sulphuric acid concentration (M) at APS to aniline molar ratio 1.5

**Table 1**  
**Synthesis of PANI dispersions at varying conc. of dodecyl sulphuric acid in presence of 25mL isopropanol cosurfactant**

S. N.	DSA conc (M)	Dispersion type with APS to Aniline molar ratio				
		2.5	2.0	1.5	1.0	0.5
1	0.2	Dispersion	Dispersion	Dispersion	Dispersion	Dispersion
2	0.1	Dispersion	Dispersion	Dispersion	Dispersion	Dispersion
3	0.05	Dispersion	Dispersion	Dispersion	Dispersion	Unstable Dispersion
4	0.03	Unstable Dispersion	Unstable Dispersion	Dispersion	Dispersion	Unstable Dispersion
5	0.01	Unstable Dispersion	Unstable Dispersion	Dispersion	Dispersion	Unstable Dispersion
6	0.009	Unstable Dispersion	Precipitation	Dispersion	Dispersion	Unstable Dispersion
7	0.008	Unstable Dispersion	Precipitation	Dispersion	Dispersion	Unstable Dispersion
8	0.007	Precipitation	Precipitation	Dispersion	Unstable Dispersion	Unstable Dispersion
9	0.006	Precipitation	Precipitation	Unstable Dispersion	Unstable Dispersion	Precipitation
10	0.005	Precipitation	Precipitation	Unstable Dispersion	Unstable Dispersion	Precipitation
11	0.004	Precipitation	Precipitation	Unstable Dispersion	Unstable Dispersion	Precipitation
12	0.003	Cloudy Solution	Precipitation	Precipitation	Precipitation	Precipitation
13	0.002	Cloudy Solution	Cloudy Solution	Precipitation	Precipitation	Precipitation
14	0.001	Cloudy Solution	Cloudy Solution	Precipitation	Precipitation	Precipitation

**Table 2**  
**Synthesis of PANI dispersions at varying conc of dodecyl sulphuric acid in absence of cosurfactant**

S.N.	DSA conc (M)	Dispersion type with APS to Aniline molar ratio				
		2.5	2.0	1.5	1.0	0.5
1	0.2	Unstable Dispersion	Dispersion	Dispersion	Dispersion	Dispersion
2	0.1	Unstable Dispersion	Dispersion	Dispersion	Dispersion	Dispersion
3	0.05	Unstable Dispersion	Unstable Dispersion	Dispersion	Dispersion	Unstable Dispersion
4	0.03	Unstable Dispersion	Unstable Dispersion	Dispersion	Dispersion	Unstable Dispersion
5	0.01	Unstable Dispersion	Unstable Dispersion	Dispersion	Unstable Dispersion	Unstable Dispersion
6	0.009	Unstable Dispersion	Unstable Dispersion	Dispersion	Unstable Dispersion	Unstable Dispersion
7	0.008	Unstable Dispersion	Unstable Dispersion	Dispersion	Unstable Dispersion	Unstable Dispersion
8	0.007	Precipitation	Precipitation	Dispersion	Unstable Dispersion	Unstable Dispersion
9	0.006	Precipitation	Precipitation	Unstable Dispersion	Unstable Dispersion	Precipitation
10	0.005	Precipitation	Precipitation	Unstable Dispersion	Unstable Dispersion	Precipitation
11	0.004	Precipitation	Precipitation	Unstable Dispersion	Unstable Dispersion	Precipitation
12	0.003	Cloudy Solution	Precipitation	Precipitation	Precipitation	Precipitation
13	0.002	Cloudy Solution	Cloudy Solution	Precipitation	Precipitation	Precipitation
14	0.001	Cloudy Solution	Cloudy Solution	Precipitation	Precipitation	Precipitation

**Table 3**  
**Conductivities of PANI dispersions at various DSA conc and APS to aniline molar ratio**

DSA conc (M) ↓ APS/Aniline molar ratio →	Conductivity of PANI dispersion (Scm <sup>-1</sup> ) in thin film form									
	With cosurfactant					Without cosurfactant				
	2.5	2	1.5	1	0.5	2.5	2	1.5	1	0.5
0.2	0.4013	0.4881	0.7589	0.7411	0.5083	-	0.4873	0.7435	0.7411	0.5013
0.1	0.4109	0.4923	0.7571	0.7381	0.4995	-	0.4901	0.7401	0.7356	0.4965
0.05	0.4154	0.4945	0.7553	0.7358	-	-	-	0.7247	0.7218	-
0.03	-	-	0.7544	0.7313	-	-	-	0.7128	0.7013	-
0.01	-	-	0.7498	-	-	-	-	0.6955	-	-
0.009	-	-	0.7489	-	-	-	-	0.6875	-	-
0.008	-	-	0.7461	-	-	-	-	0.6594	-	-
0.007	-	-	0.7413	-	-	-	-	0.6344	-	-

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