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Annealed Effects of Poly-o-Toluidine (POT) Nanomaterial at Different Temperatures

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Abstract

Poly-O-Toluidine (POT) nanomaterials were prepared by using chemical oxidative polymerization method. The polymerization process was carried out using the monomer o- toluidine (1M), ammonium peroxydisulphate (APS) (0.5M) as oxidant and the dopant sulphuric acid (3M). The resultant polymer materials are heat treated at various temperatures such as 200˚C, and 400˚C. The prepared POT materials are characterized by using different Spectroscopic techniques, Fourier Transform Infrared Spectroscopy (FTIR), Ultraviolet Visible (UV-VIS) Spectrometry, Particle Size Analyser (PSA), and biological application like Anti-Bacterial activities. The FTIR study shows the various functional groups in POT. The optical properties of prepared polymer material band gap, electron transition are calculated by using UV-VIS techniques. The PSA studies are revealed that the measurement of the size distribution of individual particles in a POT nanomaterials. The antibacterial activity of the POT nanomaterials are indicates that the several microorganisms. These POT nanomaterials are used to examines that the Chemical, optical, size of the nanomaterials and antibacterial activity for different Bactria.

Keywords: POT (Poly O-Toluidine), Polymerization, ammonium peroxydisulphate, antibacterial activity, microorganisms

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1. Introduction

Toluidine (ortho-toluidine) is an organic compound. Among all conducting polymers, Polyaniline is one of the most promising conducting polymers, but this is inherently brittle and has poor processibility, due to the insolubility in common organic solvents.[1] This problem has been overcome to some extent by using substituted derivatives of anilines such as O-Toluidine, anisidine, N-methyl (or) N-ethyl aniline, etc. Poly (O-Toluidine) is a derivative of Polyaniline which contains methyl (-CH3) group in its ortho position of benzenoid ring. Poly O-Toluidine has been found to have additional advantage with respect to polyaniline (PANI) due to its faster switching time between the reduced and oxidized states[2-4]. O-Toluidine appears as a clear colourless or light yellow liquid. May it become reddish brown on exposure to air and light, O-Toluidine is used in the manufacture of various dyes, in printing textiles blue- black and as an intermediate in rubber chemicals, pesticides, and pharmaceuticals[6-8] POT was chosen as the conducting polymer owing to its higher processiability as well as solubility as compared with PANI. Basically the stages of polymerization of aniline and its derivatives (including O-Toluidine) can be described by a sequence of reaction involving oxidation process. The chemical properties of the toluidines are quite to those of aniline and toluidines have properties in common with other aromatic amines[5-6]

In present paper, we report the synthesis of POT nanomaterials by chemical oxidative polymerization method. The nanosized POT particles are characterized by PSA, FTIR, UV- VIS, and antibacterial studies have also been performed to study the structural changes.

2. Experimental Procedure

2.1. Synthesis of poly O-Toluidine:

Poly O-Toluidine was synthesized by chemical oxidative polymerization of O-Toluidine in the presence of sulphuric acid as dopant and Ammonium peroxy disulphate as oxidant. For the synthesis 10.62 ml of monomer O-Toluidine was

dissolved in 100 ml of distilled water into the conical flask and allowed to stir for 5 minutes. Then 16.07 ml of Sulphuric acid is added drop wise and the solution is kept under constant stirring at 500rpm for half an hour. Then 2.852 g of APS was dissolved in 25 ml of distilled water separating which was added drop by drop in the above solution with vigorous stirring within few minutes after the addition of APS, the solution becomes dark blackish blue colour indicating beginning of the polymerization of O-Toluidine. Further the solution was stirred for 24 hours at 600rpm and it completes the polymerization process. When the reaction was finished blackish blue precipitate of poly o-toluidine was obtained. Then the precipitate was filtered using whatmann filter paper and washed with 20 ml of Acetone and 25 ml of Ammonia solution respective in order to remove the excess acid and impurity. Poly O-Toluidine was dried under vacuum oven at 60 C for 3days. The synthesized poly O-Toluidine was finally grinded using mortar and the product is obtained in the form of fine powder and this powder was divided into 4 parts. The first part of the powder is pure at 60 C and then the other part of the powder was prepared by maintaining at different degree Celsius (200 °C, 300 °C, and 400 °C).

3. Results and Discussions

3.1 FTIR characteristics:

FTIR identified the function group of the POT nanomaterials. Fig. 1. shows the FTIR spectra of POT nanomaterials in the range of 400-4000cm-1 [9]. The spectrum of pure POT (Fig.1) acquires the main characteristics peaks at 3155. 54 cm-1, 2893.22 cm^{-1}, 1604.77 cm-1, 1402.25 cm-1, 1180.44 cm-1, 873.75 cm-1, 507.28cm^{-1}. The peak in the range ~3155.54cm-1 is corresponding to O-H stretching (Weak, Alcohol) vibration. The peak at 2893.22cm-1 is associated C-H stretching (Medium, Alkane) vibration. The peak at 1604.77cm-1 exhibits N- H bending (Medium, Amine). The peak at 1402.25cm-1 is indicates that O-H bending (Medium, Carboxylic Acid) vibration. The peak at 1180.44cm^{-1} is associated with C-O stretching (Strong, Tertiary Alcohol) vibration. The peak at 873.75cm^{-1} is exhibits that C=C bending (Strong, Alkane) vibration and the peak at 507.28cm^{-1} is indicates that C-Br stretching (Strong, Halo compound) vibration.[7]

The peaks at Fig.1 shows the different calcination temperature at 200°C and 400°C in the POT leads to small shifts of some peaks of pure POT. For 200°C, the presence of the vibration modes are 3037.89cm^{-1}, 2879.72cm^{-1}, 1593.20 cm^{-1}, 1490.97cm^{-1}, 1176.58cm^{-1}, 964.41cm^{-1} and 613.36cm-1. For 400°C, the presence of the vibration modes are 3022.45cm^{-1}, 2358.94cm^{-1}, 1695.43cm^{-1}, 1510.26 cm^{-1}, 1178.51cm^{-1}, 806.25^{cm-1}, 572.86 cm^{-1}. It is also evident from the Fig. 1 that the incorporation of nanoparticles to POT causes some observable changes in the spectrum. Fourier Transform infrared spectrograph confirms that the presence of functional groups. The bending & stretching along with corresponding wave number was shown in Table 1.[8]

3.2 UV –Vis analysis

UV-VIS spectra are used to find qualitative information and the optical properties about POT nanomaterials. Fig. 2

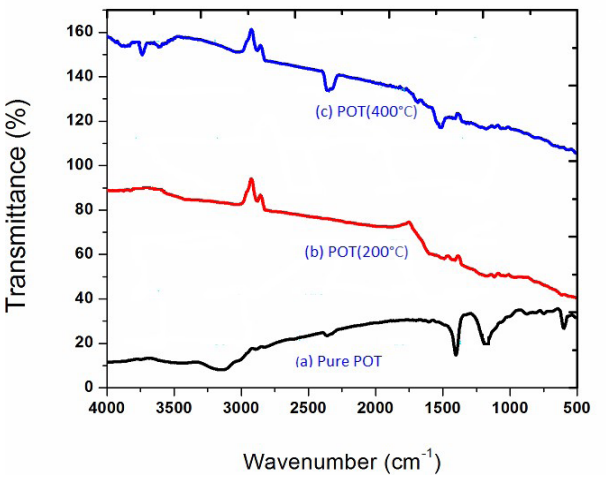


Fig. 1: FTIR Spectrum of (a) Pure POT, (b) POT (200°C), POT (200°C).

Table 1: FTIR peaks and their assignments of POT nanomaterials

S.No	Characteristics vibration	Wave number (cm-1)		
		Pure POT	POT (200°C)	POT (400°C)
1.	O-H stretching	3155.54	3037.89	3022.45
2.	C-H stretching	2893.22	2879.72	2358.94
3.	N-H bending	1604.77	1593.20	1695.43
4.	O-H bending	1402.25	1490.97	1510.26
5.	C-O stretching	1180.44	1176.58	1178.51
6.	C=C bending	873.75	964.41	806.25
7.	C-Br stretching	507.25	613.36	572.86

gives UV-VIS absorption spectra of POT nanomaterials. The optical band gap energy (Eg) are obtained by using the fundamental law, Eg =hc/λmax, where λmax is the maximum absorption wavelength in nm [9]. UV-VIS spectra of POT nanomaterials are shown in Fig.2. The spectrum of pure POT show that there are two absorption bonds at 350 and 615nm, which are assigned to π-π* transition of the benzenoid ring and n-π* excitation of benzenoid to the quinoid ring in the polymer chain and the band gap energy are 3.55, 2.01 eV. The POT at 200°C are appeared two absorptions peaks and wavelength range at 301, 562nm and the band gap energy are 4.12, 2.20 eV. The POT at 400°C are appeared two absorptions peas and wavelength range at 338, 541nm and the band gap energy are 3.67, 2.29 eV respectively. The Eg value POT nanomaterials at different temperatures 200°C and 400°C are compared to the pure POT the band gap value is increased in the sample. The pure POT the peak intensity of the nanomaterials is increased with increasing concentration. In UV analysis the band gap energy of POT nanomaterials and their corresponding electron transition are tabulated in Table 2.

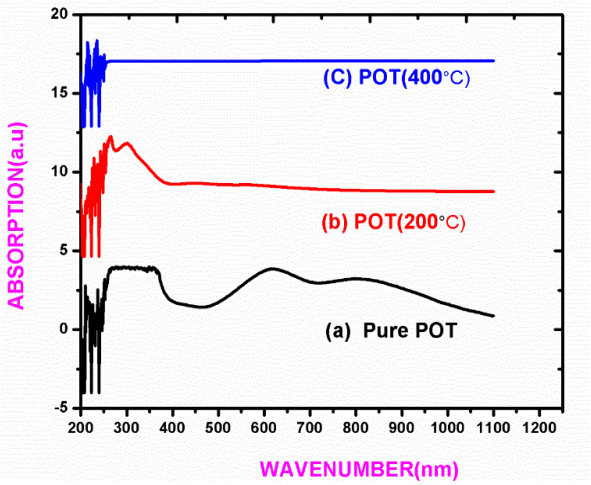


Fig. 2: UV spectrum of (a) pure POT, (b) POT (200°C), (c) POT (400 °C) nanomaterials

3.3 Particle size analyser:

A particle size analyser is used to find the particle size distribution. A particle size analyzer gives the particle size distribution for the synthesized POT nanomaterials at different temperature as 200°C and 400°C. In Fig.3. shows the particle size distribution peak for POT at different temperature and the graph drawn between the particle diameter (μm) and normalized particle amount (cum). In pure POT has normalized particle amount (cum) for 25% has the value of the particle size distribution is 17.600 μm, for 50% has the particle size distribution value is 27.089 μm, for 75% has the particle size distribution is 42.319 μm. The mean value of the Pure POT is 26.677.

In POT 200°C has normalized particle amount (cum) for 25% has the value of the particle size distribution is 23.541μm, for 50% has the particle size distribution value is 40.335 μm, for 75% has the particle size distribution is 62.344μm. The mean value of the Pure POT is 37.627. In POT 400°C has normalized particle amount (cum) for 25% has the value of the

Table 2: UV-VIS data and their assignments of POT nanomaterials

S. No	Sample name	Wavelength (nm)	Electron transition	Absorption	Band gap (eV)
1.	Pure POT	350 615	π-π* n-π*	3.996 3.879	3.55 2.01
2.	POT (200°C)	301 562	π-π* n-π*	3.208 0.561	4.12 2.20
3.	POT (400°C)	338 541	π-π* n-π*	0.158 0.160	3.67 2.29

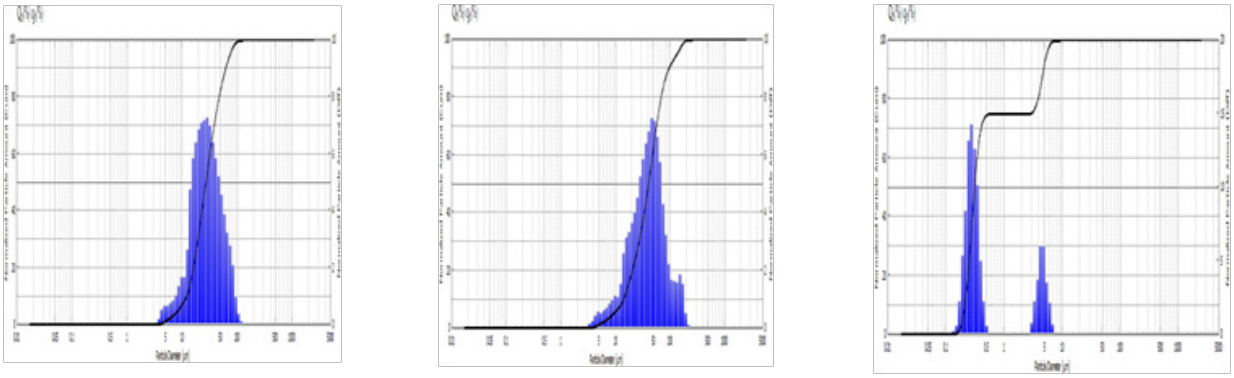


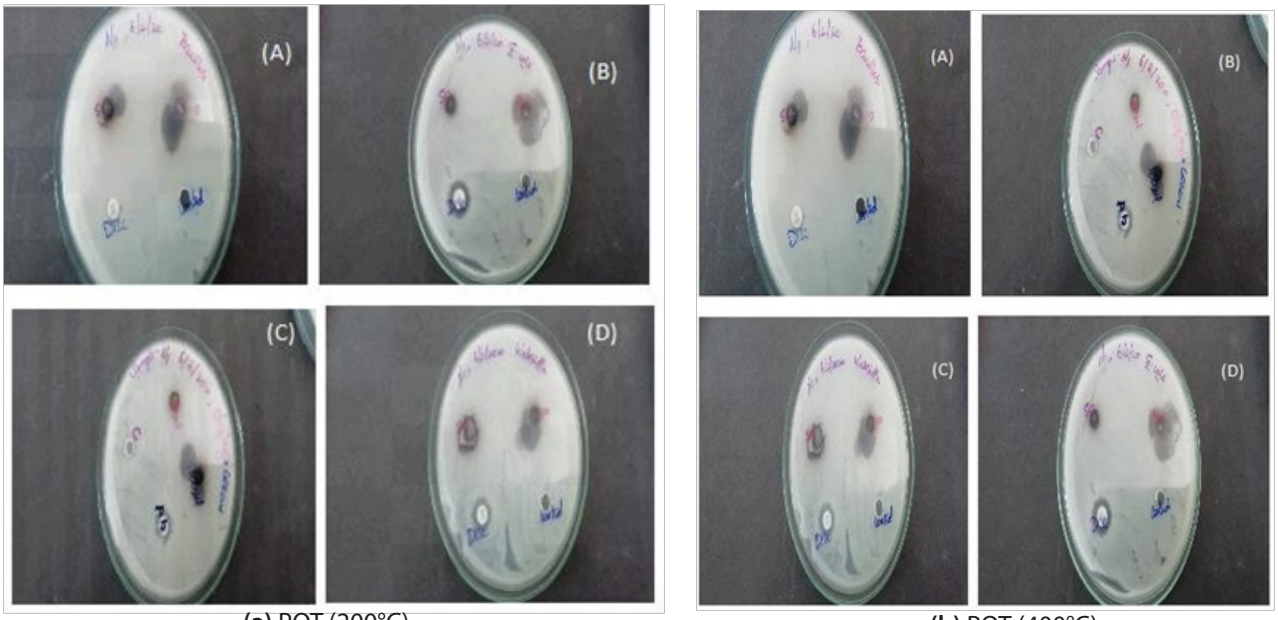
Fig.3: Particle size distribution peak for (a) Pure POT, (b) POT (200°C), (c) POT (400°C).

Table 3: The cum% and particle diameter (µm) for Pure POT, POT (200°C), and POT (400°C).

S. No	Particle diameter (µm)			
	Cum%	Pure POT	POT (200°C)	POT (400°C)
1.	For 25%	17.600	23.541	0.250
2.	For 50%	27.089	40.335	0.314
3.	For 75%	42.319	62.344	2.572

Table 4: Photographs of antimicrobial results of POT nanomaterials

S.NO	Sample name	Zone of inhibition in diameter (mm)							
		Gram positive				Gram negative			
		Bacillus cereus		Staphylococcus aureus		Escherichia coli		Klebsiella pneumonia	
		50 µl	100 µl	50 µl	100 µl	50 µl	100 µl	50 µl	100 µl
1.	POT (200°C)	5	15	14	18	5	10	12	18
2.	POT (400°C)	12	14	15	18	0	0	0	0



(a) POT (200°C) **(b)** POT (400°C)

Fig. 4: Photographs of antimicrobial results of (A) POT (200°C), (b) POT (400°C) for gram positive (A) Bacteria Bacillus cereus, (B) Staphylococcus aureus and gram negative (C) Bacteria Escherichia coli, (D) Klebsiella pneumonia

particle size distribution is 0.250µm, for 50% has the particle size distribution value is 0.314µm, for 75% has the particle size distribution is 2.572µm. The mean value of the Pure POT is 0.561. In Table 3, it shows the cum% and particle diameter of the prepared sample at different temperatures.[10]

3.4 Antibacterial activities:

The POT nanomaterial was tested for antimicrobial activity by well diffusion method. Liquid Mueller Hinton agar media and the Petri plates were sterilized by autoclaving at 121°C for about 30 minutes at 15 lbs pressure. Under aseptic conditions in the laminar airflow chamber, about 20ml of the agar medium was dispensed into each Petri plate to yield a uniform depth of 4mm. After solidification of the media, 18 hrs culture of Gram positive microorganisms such as Bacillus cereus(MTCC 430), Staphylococcus aureus (MTCC 3160), Gram negative microorganisms such as E.coli (MTCC 1698) and Klebsiella pneumoniae (MTCC10309) obtained from

IMTECH, Chandigarh were swabbed on the surface of the agar plates. Well was prepared by using cork borer followed with loading of 50 µl and 100 µl of each sample to the distinct well with sterile distilled water as negative control and ampicillin (30mcg/disc) as positive control. The sample loaded plates were then incubated at 37°C for 24 hours to observe the zone of inhibition.

4. Conclusion:

The POT nanomaterial was successfully prepared by insitu chemical oxidative polymerization method. Ammonium peroxy disulphate (APS) which act as oxidant and sulphuric acid which act as dopant and the samples were name as a pure POT, POT (200°C), and POT (400°C). From the characterization of prepared samples the following conclusions were arrived. The FTIR spectrum confirmed the presence of POT nanomaterials and changes the functional group during each reaction. The functional groups are O-H, C-H, C-O, C-Br, stretching and N-H, C=C, O-H bending are present in POT nanomaterials. The UV spectra of POT nanomaterials are assigned to π-π* and n-π* transition. The maximum absorption peaks are observed and the corresponding band gap energy is found. Particle size analyzer (PSA) used to analyze the size distribution of the individual particles in POT nanomaterials. The POT nanomaterial samples shown antibacterial activity against both gram positive as well as gram negative organisms. Conducting POT nanomaterials are believed to be useful as an antibacterial agent, self- clean as well as multifunctional material for improving the human health and living environment.

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