



# Comparative sensing of aldehyde and ammonia vapours on synthetic polypyrrole-Sn(IV)arsenotungstate nanocomposite cation exchange material



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## ABSTRACT

Polypyrrole-Sn(IV)arsenotungstate (PPy-SnAT) conductive nanocomposite cation exchange have been synthesized by in-situ chemical oxidative polymerization of polypyrrole with Sn(IV)arsenotungstate (SnAT). PPy-SnAT nanocomposite was characterized by Fourier transform infra-red spectroscopy (FTIR), X-Ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy-dispersive x-ray (EDX) and thermogravimetric analysis (TGA). The ion exchange capacity (IEC) and DC electrical conductivity of nanocomposite was found to be 2.50 meq/g and  $5.05 \times 10^{-1}$  S/cm respectively. The nanocomposite showed appreciable isothermal stability in terms of DC electrical conductivity retention under ambient condition up to 130 °C. The nanocomposite cation exchange based sensor for detection of formaldehyde and ammonia vapours were fabricated at room temperature. It was revealed that the resistivity of the nanocomposite increases on exposure to higher percent concentration of ammonia and lower concentration of formaldehyde at room temperature (25 °C).

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## 1. Introduction

Hazardous waste is discharged in the environment in many forms, like liquids, solids, gases, and sludge [1]. It can have an immediate as well as long-term devastating impact on ecosystem, comprising of plants and animals, making fish unfit for human consumption. Hazardous waste remains in the environment for a long time and in some cases for thousands of years. It may affect the environment in the form of hazardous gases like; ammonia, aldehyde and amines etc. [2–4].

Aldehydes are known to be highly toxic and corrosive, and even short-term exposure has major health risks. Consuming liquid aldehyde causes severe intestinal irritation and sometimes

becomes fatal. Even at low levels, aldehyde gas causes eye, throat and skin irritation, nausea, wheezing and asthma attacks. Aldehydes are now classified as a known carcinogens, or cancer-causing agents, by the International Agency for Research on Cancer (IARC) [5]. The Environmental Protection Agency (EPA), for example, places a limit of 16 ppb (parts per billion) of aldehyde in the environment [6].

Poly(pyrrole) and its derivatives have received a great deal of attention due to their high electrical conductivity, good environmental stability, relatively easy synthesis and good redox reversibility. These properties are favourable for its applications in the area of secondary batteries [7], electro catalysis, electro chromic display devices, light-emitting devices, chemical sensors and biosensors [8–10]. However, the structure of poly(pyrrole) needs to be modified in order to be used in chemical sensor and biosensor fields since it has no functional group [11]. The conducting polymers also permit a charge transfer to produce electrochemical signals between the electrode and the immobilized biomolecules [12,13]. Thus for biosensor applications, the conducting polymers have been functionalized with carboxyl, amino, formyl or succinimidyl carbonate groups, and conjugated directly with various electronic mediators or bio-recognizable molecules to facilitate

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immobilization.

However, conducting polymer based ion exchangers with polyvalent sites have been poorly reported in the field of gas sensing although some researchers have explored the electrical conductivity and sensing activity on the polymer based cation exchanger. In the present work, we have reported polypyrrole-Sn(IV)arsenotungstate (Ppy-SnAT) nanocomposite cation exchanger with better ion exchange capacity (IEC) than other Sn(IV) based cation exchangers reported earlier [14]. Formaldehyde, ammonia and mixed (formaldehyde + ammonia) solution vapour sensing behaviour on Ppy-SnAT nanocomposite cation exchanger is the sufficient part of our studies.

## 2. Experimental

### 2.1. Chemicals, reagents and instruments

Pyrrole monomer from Spectrochem, iron(III)chloride (Merck, 98%), HCl (35%) from E-Merck (India Ltd.) were used. Sodium arsenate (Finar reagent), sodium tungstate (CDH), stannic chloride ( $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ ) (CDH), methanol from CDH (India Ltd. 98%), formaldehyde from Fisher scientific, ammonia from Merck (India Ltd. 25%) and demineralised water (DMW) were used in the experimental work. All other reagents and chemicals were of analytical grade.

Fourier Transform Infra-Red (FTIR) spectrophotometry was performed using Perkin-Elmer, USA, model Spectrum-BX, range 4000–400 nm. Scanning Electron Microscopy (SEM), Energy Dispersive X-ray diffraction (EDX) and Transmission Electron Microscopy (TEM) was done using LEO 435–VF Electron Microscope. Thermo-Gravimetric Analysis (TGA) was done using Thermal Analyzer-V2.2A DuPont 9900 with nitrogen atmosphere. X-Ray Diffraction (XRD) was performed using Rigaku X-Ray powder diffractometer with Cu anode ( $K\alpha \lambda = 1.54186 \text{ \AA}$ ) using a PW, 1148/89 based diffractometer with Cu  $K\alpha$  radiations. Four in-line probe conductivity meter (scientific equipment Roorkee) was used for conductivity and sensing measurements.

### 2.2. Preparation and ion exchange capacity (IEC) of polypyrrole-Sn(IV)arsenotungstate (Ppy-SnAT) composite ion-exchanger

#### 2.2.1. Synthesis of Sn(IV)arsenotungstate inorganic precipitate

Sn(IV)arsenotungstate was prepared at room temperature ( $25 \pm 2 \text{ }^\circ\text{C}$ ) by mixing 0.1 M stannic chloride (prepared in 1 M HCl), 0.1 M sodium arsenate and 0.1 M sodium tungstate (in DMW) in 1:1:1 vol ratio respectively. The solution was constantly stirred and the pH was maintained at 1. As a result, white colored gel of Sn(IV)arsenotungstate was obtained as precipitate. Then, the precipitate was allowed to settle down for 24 h, filtered and washed under suction with DMW until the filtrate showed neutral pH. Finally it was dried in an oven at  $80 \text{ }^\circ\text{C}$ . The different ratios of inorganic ion exchanger are given in Table 1. On the basis of high ion exchange capacity (IEC) sample **S-2** was selected for further studies.

**Table 1**  
Synthesis and IEC of Sn(IV)arsenotungstate ion exchange material.

S. No.	0.1 M Stannic chloride (1 M HCl) (ml)	0.1 M Sodium tungstate (DMW)(ml)	0.1 M Sodium arsenate (DMW)(ml)	pH	IEC $\text{Meqg}^{-1}$
S-1	50	50	50	1	2.0
<b>S-2</b>	<b>100</b>	<b>50</b>	<b>50</b>	<b>1</b>	<b>2.1</b>
S-3	50	100	50	1	1.4
S-4	50	50	100	1	0.8

#### 2.2.2. Preparation of polypyrrole-Sn(IV)arsenotungstate (Ppy-SnAT) composite ion-exchange material

Ppy-SnAT was synthesized by in-situ chemical oxidative polymerization of Polypyrrole in the presence of Sn(IV)arsenotungstate (**S-2**) particles. A certain amount of Sn(IV)arsenotungstate was dispersed in 200 ml DMW, taken in three necked round bottom flask, and stirred for 30 min with the help of magnetic stirrer. A definite amount of pyrrole monomer was then added and the mixture was stirred for 45 min for the adsorption of pyrrole on the surface of Sn(IV)arsenotungstate (**S-2**) particles. After that, a fixed amount of aqueous  $\text{FeCl}_3$  solution (prepared by dissolving 2 g  $\text{FeCl}_3$  in 50 ml DMW) was added to the mixture and constantly stirred for another 24 h. The resultant mixture slowly turned into black colored slurry indicating the formation of Ppy-SnAT. It was then filtered off, washed thoroughly with DMW followed by fixed amount of methanol to remove excess acid and any adhering trace of  $\text{FeCl}_3$ . The washed material was finally dried in oven at  $50 \text{ }^\circ\text{C}$  to obtain Ppy-SnAT composite ion exchanger.

### 2.3. Ion Exchange Capacity (IEC)

IEC expresses the amount of  $\text{H}^+$  ions released by cation exchanger to the flow of neutral salt through it. To compute IEC, Ppy-SnAT composite was converted into  $\text{H}^+$  form by keeping it in 1 M  $\text{HNO}_3$  solution for 24 h with occasional shaking, and intermittently replacing the supernatant liquid. The excess acid was removed by washing it several times with DMW and dried it in the oven. The glass column (i.d.-1 cm) was packed with 1 g dry sample in  $\text{H}^+$  form, 1.0 M sodium nitrate ( $\text{NaNO}_3$ ) as eluent was used to elute the  $\text{H}^+$  ions completely from the cation-exchange column keeping a flow rate of  $\sim 0.5 \text{ ml min}^{-1}$ . The effluent was titrated against a standard 0.1 M NaOH solution using phenolphthalein as indicator [15].

On the basis of high IEC and better electrical conductivity sample Ppy-SnAT (**P-2**) was selected for further studies, results are shown in Table 2.

**Table 2**  
Synthesis, IEC and conductivity of Ppy-Sn(IV)AT nanocomposite cation exchanger.

S.No.	Sn(IV)arsenotungstate Inorganic ion exchanger (gm)	Pyrrole monomer in ml	$\text{FeCl}_3$ (Water)(gm)	IEC $\text{Meqg}^{-1}$	Electrical Conductivity ( $\text{Scm}^{-1}$ )
P-1	2	1	2	1.50	$4.63 \times 10^{-1}$
<b>P-2</b>	<b>2</b>	<b>3</b>	<b>2</b>	<b>2.50</b>	<b><math>5.05 \times 10^{-1}</math></b>
P-3	2	5	2	1.20	$3.99 \times 10^{-1}$
P-4	2	7	2	1.00	$4.82 \times 10^{-1}$
P-5	2	9	2	0.80	$4.92 \times 10^{-1}$
<b>P-6</b>	-	<b>3</b>	<b>2</b>	<b>0.60</b>	<b>1.78</b>

**Table 3**  
Percent composition of Pure Polypyrrole, Sn(IV)arsenotungstate and Ppy-SnAT nanocomposite cation exchanger.

S. No.	Element	Percentage (%)		
		Pure Polypyrrole	Sn(IV)arsenotungstate	Ppy-SnAT composite
1	C	32.36	–	53.05
2	N	30.16	–	2.03
3	Cl	7.45	–	4.90
4	Fe	0.06	–	–
5	O	–	34.51	17.55
6	Sn	–	23.85	9.98
7	W	–	22.97	8.08
8	As	–	18.50	4.40
9	Na	–	0.17	–

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