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# Improved Ammonia Sensing by Solution Processed Dodecyl Benzene Sulfonic Acid Doped Polyaniline Nanorod Networks

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**ABSTRACT** Dodecyl benzene sulphonic acid (DBSA) doped polyaniline (PANI) nanorods have been synthesized by a templateless route via in-situ emulsion polymerization of aniline in aqueous DBSA solution. XRD patterns, UV-Visible Spectroscopy, and FTIR analysis confirm the formation of PANI and PANI-DBSA, whereas FESEM images revealed their elongated rod shaped morphology. The chloroform dispersion of synthesized PANI-DBSA was spin coated over prefabricated interdigitated Pt patterned glass substrate to realize porous gas sensing active layer. The sensor displays good response in 1-1200 ppm ammonia concentration range and can repeatedly detect 300 ppm of ammonia with response time of 6 s, recovery time of 37 s and relative response value of 9.57. Besides, its response was found to be linear for 1-50 ppm ammonia concentration with relative response per ppm value of 0.01675.

**INDEX TERMS** Ammonia gas sensor, conducting polymer, dodecyl benzene sulfonic acid doped polyaniline.

## I. INTRODUCTION

The quality of air in the environment is one of the most important concerns in today's world. Gases like Carbon monoxide (CO), Oxides of nitrogen (NO<sub>x</sub>), and Ammonia (NH<sub>3</sub>) are detrimental to human health due to associated toxic effects. Particularly, Ammonia which is widely used chemical for refrigerant gas, water purification, manufacture of plastics, explosives, textiles, pesticides, dyes, and other chemicals etc., is known to be irritating and corrosive in nature [1]–[6]. Human beings can briefly tolerate the pungent odor of the Ammonia in the range 5 to 50 ppm while the irritation effect starts when the concentration approaches to ~25 to 50 ppm for 2 to 6 h. But its presence over 100 ppm in environment (due to nearby petrochemical plants, refrigeration leakage, pig farming etc.) can be of serious concerns for human health and safety [7]. Ammonia interacts immediately upon contact with available moisture in the skin, eyes, oral cavity,

respiratory tract and particularly mucous surfaces, thereby, causes cellular destruction and tissue necrosis [8]. Thus sensing and quantitative estimation of Ammonia has drawn worldwide attention. Accordingly, in the recent past, different gas sensor technologies have been used for Ammonia detection including gravimetric [9], catalytic [10], electrochemical [11]–[12], colorimetric [13], gas chromatographic [14], optical [15]–[17] and acoustic techniques [18]–[19]. Among other available detection technologies, chemi-resistor is frequently used in gas sensing technologies due to its simple configuration, easy fabrication, cost effectiveness, amenity for miniaturization, and its use in MEMS technology [20].

Different materialistic candidates viz. metal/metal oxide [19]–[21], carbonaceous materials [22]–[23], conducting polymer [7], [24]–[27], have been exploited as sensing element of chemi-resistor. However, metal oxide based gas sensors suffer from several drawbacks such as elevated operating temperature, high costs, complex fabrication steps, and high power consumption. Similarly, carbon materials based systems suffer from poor selectivity, high cost, film

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forming issues, and low sensitivity and selectivity. In this context, conducting polymers (CPs) present an attractive material option due to distinguished advantages such as low cost, ease in synthesis, environmental stability, solution processability, and tunable conductivity [28]–[32]. Moreover, CPs based sensors display fast response, high sensitivity, room temperature sensing/regeneration, and facile processing via solution route. Among various CPs, polyaniline (PANI) has attracted much attention due to facile synthesis, thermal/environmental stability, non-redox doping with various protonic acids, tunable conductivity, and counter-ion aided processing [25], [33]. It has been established that solution processability of PANI is crucial for realizing gas sensing thin film over patterned electrodes. However, the PANI salts based on conventional dopants e.g. hydrochloric acid and sulfuric acid, are not processable due to relative insolubility in organic/inorganic solvents [34]–[35]. To overcome this, use of surfactants like Camphor sulphonic acid [36]–[37] and Dodecyl benzene sulphonic acid [38]–[40] has been suggested as dopant. Besides, the designing and exploitation of PANI nanostructures e.g. nanofibers [38], nanoparticles [39] or nanorods [41] via suitable technique is also known to improve the sensor performance. The general routes for making processable PANI based nanostructured material involve either polymerization of aniline in presence of inorganic acids, followed by undoping with a base and redoping with bulky counter-ion dopants or direct polymerization in the presence of surfactant dopants [42]. However, the use of indirect routes [7], [39] to realize highly doped yet processable PANI nanostructures of desired shape add complexity to the system, whereas the direct route [41], [43] demand strict control over dopant concentration and residual dopant to regulate doping level, particle morphology, dispersibility etc. The mix dopant approach has also been used by researchers but the optimization of properties becomes extremely difficult [33], [38], [40]. The above in conjunction with processing to decide sensing film's morphology, particularly porosity adds to complexity of the system and often leads to moderate sensing response and sensor stability [7], [38]–[39].

Herein, we exploited DBSA for synthesis of DBSA doped PANI based elongated nanostructure, wherein due to its amphiphilic character and sulfonic acid groups, DBSA not only acts as soft template for formation of elongated rod type PANI nanostructures but also acts as dopant by protonating PANI backbone. The formation of PANI-DBSA and its doping status was checked by XRD pattern, UV-Visible Spectra, and FTIR analysis whereas its elongated rod shaped morphology was observed using Scanning Electron Microscopy. The chloroform solubility of PANI-DBSA and non-planar disposition between finite thickness PANI rods within active layer, also enabled realization of thin and porous gas sensing active layer. Such sensing platform based on DBSA doped PANI thin film over platinum (Pt) interdigitated electrodes is found to reliably detect ammonia vapors with low detection limit, broad detection range, fast response/recovery response,

high relative response, good repeatability, good selectivity and linearity.

## II. MATERIALS AND METHODS

### A. MATERIALS

Aniline (LobaChemie) was used as monomer and was freshly double distilled before use. Ammonium persulfate  $[(\text{NH}_4)_2\text{S}_2\text{O}_8]$ , Merck] was used as oxidant whereas dodecyl benzene sulfonic acid (DBSA, Merck) was used as surfactant dopant. Chloroform ( $\text{CHCl}_3$ , Merck) was used for making dispersion of synthesized polyaniline (PANI). De-ionized water having resistivity value  $>18 \text{ M}\Omega\text{-cm}$  was used for synthesis and washings.

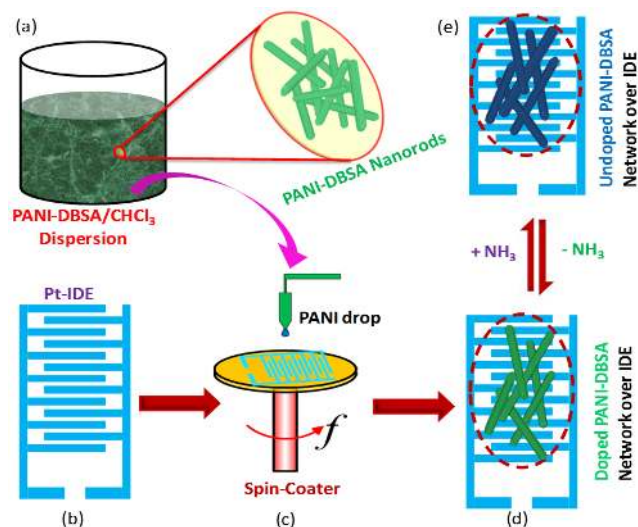
### B. SYNTHESIS

DBSA and water solution (0.3 M aqueous DBSA solution) was stirred to form an emulsion at  $0^\circ\text{C}$  followed by adding monomer (0.1 mol aniline) [41]. The polymerization was initiated by dropwise addition of aqueous solution of ammonium persulfate (0.1 mol in 100 mL de-ionized water) while maintaining reaction mixture at  $0^\circ\text{C}$  under continuous stirring. After completion of polymerization (8h), the formed dark green emulsion of DBSA doped polyaniline (PANI) i.e. PANI-DBSA was de-emulsified using propanol followed by filtration and repeated water washing till the pH of the filtrate became neutral. Subsequently, the PANI-DBSA was dissolved in chloroform followed by removal of residual water and DBSA using phase transfer method, to obtain PANI-DBSA dispersion in chloroform.

Despite of the usual steps of oxidative polymerization procedures, undertaken for the preparation of polyaniline, we have got nanorods morphology. To achieve this nanorods morphology, we have performed different synthesis experiments (with different concentrations, at different temperatures, for different time, etc.). The above mentioned optimized set of parameters (Dopant (DBSA) concentration of 0.3 M aqueous DBSA solution, Monomer (aniline) concentration of 0.1 mol, Oxidant (ammonium persulfate) concentration of 0.1 mol, Reaction temperature of  $0^\circ\text{C}$ , and Polymerization time of 8 hours) has resulted in nanorods morphology. This experiment was repeated five times and each time it has resulted morphology of nanorods.

### C. SENSOR DEVICE FABRICATION

The systematic representation of sensor fabrication via spin coating of polymer dispersion is schematically depicted in Fig. 1. First, a calculated amount (100 mg) of PANI-DBSA nanoparticles was suspended in chloroform (10 ml) under sonication for 3 hours followed by filtration through Whatman 41 filter paper. The obtained viscous solution (60 micro liter) of PANI-DBSA nanorods (a) was coated onto platinum (Pt) fingers patterned glass (b) interdigitated electrode (IDE), via spin coating at 1000 rpm (c) to realize thin film constituted of PANI-DBSA nanoparticle's network (d) covering IDE combs [25]. This highly doped green color



**FIGURE 1. Sensor Device Fabrication:** (a) Chloroform suspension of PANI-DBSA nanorods, (b) Platinum finger patterned glass, (c) Spin coater, (d) Doped PANI-DBSA nanorods, and (e) Undoped PANI-DBSA nanorods.

film upon exposure to ammonia undergoes undoping and changes its color to purple (e) which is accompanied by large increase in resistance.

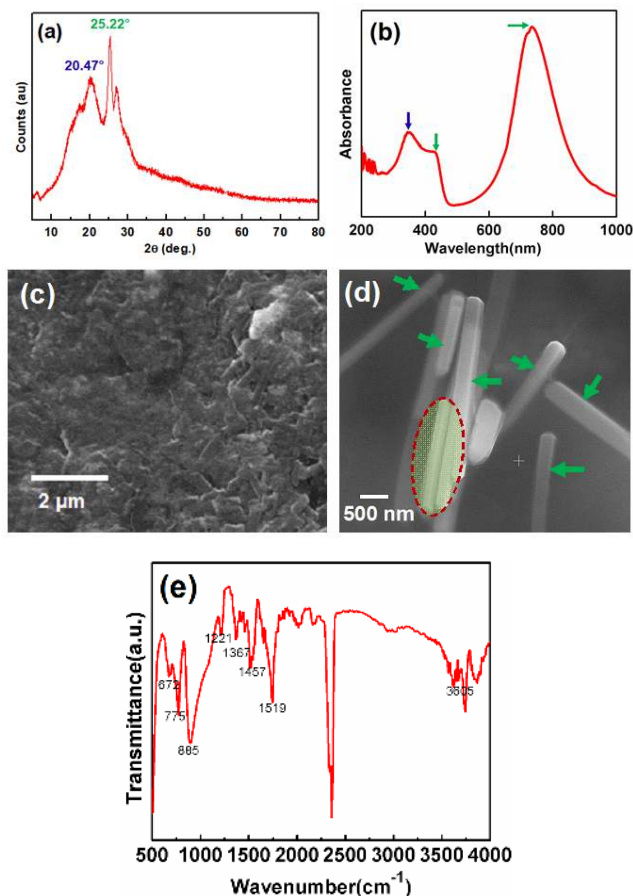
#### D. CHEMICAL CHARACTERIZATION

UV-Visible spectrophotometer (Perkin Elmer lambda 25) was used to record optical spectrum of the chloroform dispersion of PANI-DBSA in the wavelength range of 250–1000 nm. X-ray diffraction (XRD) pattern was recorded on Bruker Advanced D8 system in the diffraction ( $2\theta$ ) range of 10–80°, using  $\text{CuK}\alpha 1$  ( $\lambda = 1.540598\text{\AA}$ ) as radiation source.

The surface morphology of PANI-DBSA powder was investigated using the field emission scanning electron microscope (X-Max<sup>N</sup>, OXFORD Instrument) at accelerating potential 10.0 kV. The IRAffinity-1S FTIR spectrometer (Manufactured by Shimadzu) was used for recording of FTIR spectra of PANI-DBSA.

#### E. AMMONIA GAS SENSING PROCEDURE

The gas sensor characteristics were recorded using specially designed Gas Calibrator Test System (GCTS) having a glass test chamber of known volume (11.0 Liters). Initially, the test chamber was evacuated to  $\sim 10^{-3}$  torr to avoid any kind of interference from the residual gases present in glass chamber. The ammonia gas (from ammonia gas cylinder) at various ppm concentrations was introduced into the test chamber using calibrated leaks through fine needle valves along with clean (dry synthetic) air. The flow of ammonia gas in the test chamber was controlled by using pirani gauge with a rotary pump. Changes in resistance of PANI-DBSA thin film were recorded after every second using a data acquisition system (Computer interfaced digital multimeter, Keithley 2700). Sensor recovery period measurements were recorded by flushing out the ammonia gas from the chamber by



**FIGURE 2. PANI-DBSA Characterization:** (a) XRD pattern of PANI-DBSA powder, (b) UV-Visible spectra of PANI-DBSA dispersion in chloroform, (c) Low resolution, and (d) High resolution FESEM images of PANI-DBSA powder (e) FTIR spectra of PANI-DBSA.

introducing clean dry air in the chamber. All these experiments were performed at room temperature (27°C) with 50% relative humidity.

### III. RESULTS AND DISCUSSIONS

#### A. CHARACTERIZATION OF PANI-DBSA

The XRD pattern of PANI-DBSA powder (Fig. 2(a)) displays three distinguished peaks at  $2\theta$  values of  $20.47^\circ$ ,  $25.22^\circ$  and  $26.87^\circ$ . Particularly, the peaks at  $2\theta$  values of  $\sim 20^\circ$  and  $\sim 25^\circ$  represent the periodicity in parallel and perpendicular to chain axis respectively [43]. Here, the relative prominence of  $2\theta \sim 25^\circ$  peak compared to  $\sim 20^\circ$  peak confirms the doping of PANI by DBSA to yield most conductive emeraldine salt form of PANI.

UV-Visible absorption spectra of PANI-DBSA dispersion in chloroform (Fig. 2(b)) displays three distinguished peaks positioned at  $\sim 345$  nm, 430 nm, and 730 nm, corresponding to doping induced bandgap and polaronic states [43]. Again, sufficient doping level present in the polymer is confirmed by the prominence of 730 nm peak compare to 345 nm feature. The FESEM image (Fig. 2(c)) shows the elongated morphology of PANI-DBSA particles whose high resolution version (Fig. 2(d)) clearly displays clusters of PANI

rods (green arrow). The typical dimensions of individual rod (shaded encircled region) is  $\sim 300$  nm in diameter and  $\sim 2$   $\mu$ m in length, which is considered advantageous due to increase in surface to volume ratio, thereby extending more sites for interactions with target gas molecules. Thus, better transduction and sensor response is expected in terms of relative response value. In existing literature [39], [7], the reported particle type is nanoparticles of diameter 100 nm [39] and 500 nm [7]. Nanofibers synthesis (100 nm length and 60 to 90 nm diameter) is reported in [38].

The FTIR spectra of PANI-DBSA is shown in Fig. 2(e). The PANI-DBSA nanorods exhibited a well-defined absorption band at about  $3605$   $\text{cm}^{-1}$  that is attributed to the N-H stretching vibration mode. The characteristic peaks of C=C stretching at about  $1519$   $\text{cm}^{-1}$  and  $1457$   $\text{cm}^{-1}$  are assigned to the presence of quinoid and benzenoid rings of emeraldine salt in PANI-DBSA indicating the proper conjugation in the backbone of PANI polymer as a result of doping of DBSA [44]–[45]. The absorption bands observed at  $1367$   $\text{cm}^{-1}$  and  $1221$   $\text{cm}^{-1}$  corresponds to the C-N stretching vibration modes in secondary aromatic amines and C-N<sup>+</sup> stretching in the polaron form of PANI-DBSA respectively [44], [46]–[47]. The broad absorption band at about  $885$   $\text{cm}^{-1}$  is due to C-H out-of-plane deformation vibration. The band at  $775$   $\text{cm}^{-1}$  is assigned to the aromatic C-H bending vibration mode due to out of plane for the 1,4-disubstituted aromatic rings which arises due to the presence of DBSA dopant in the polymeric chain [46]. The absorption peak observed at  $673$   $\text{cm}^{-1}$  is assigned to S = O stretching shows the presence of DBSA in the doped PANI nanorods.

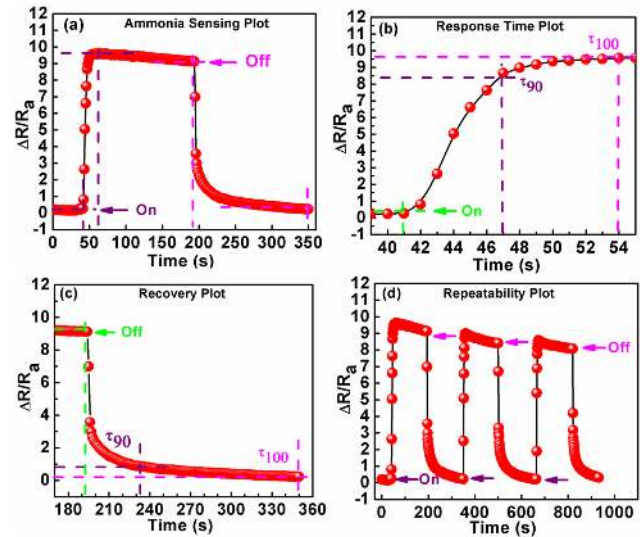
The conductivity values of the deposited PANI-DBSA thin films were measured by using Signatone QuadMap system. The measured average conductivity value on a set of five samples was  $2.5 \times 10^{-3}$  S/cm when APS/An is 1.0. Reported conductivity of PANI-DBSA in the literature is  $2.2 \times 10^{-3}$  S/cm when APS/An is 1.0 [41], [48].

### B. AMMONIA GAS SENSING RESPONSE

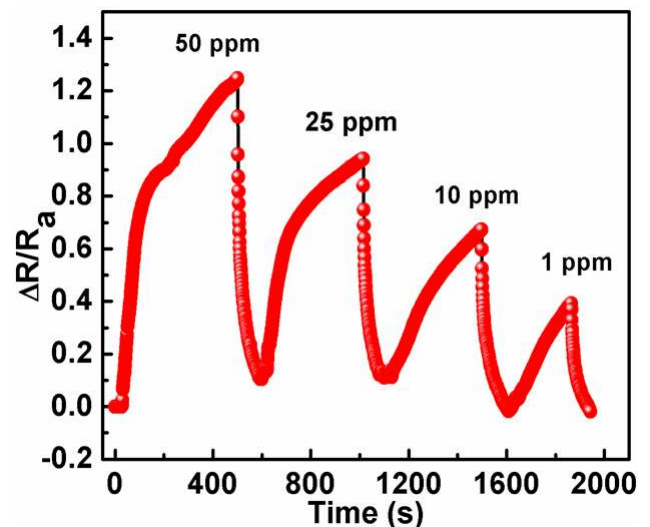
Fig. 3 shows the reversible sensing characteristics and shape of response curve of PANI-DBSA thin film based sensors for 300 ppm concentration of ammonia. The relative response of the sensor towards ammonia gas has been expressed in terms of normalized resistance change values in the presence and absence of ammonia gas as given in (1).  $R_g$  and  $R_a$  are sensor's resistance in the presence of ammonia gas and air, respectively

$$\text{Relative Response} = (R_g - R_a)/R_a \quad (1)$$

Fig. 3(a) shows that when ammonia is supplied, the resistance sharply increases and attain saturation. Subsequently, when ammonia is removed and dry air is injected in the chamber, the resistance shows initial sharp decrease followed by exponential recovery. The Fig. 3(b) and Fig. 3(c) show the expanded version of response curve to highlight the response time and recovery time respectively. In our case,



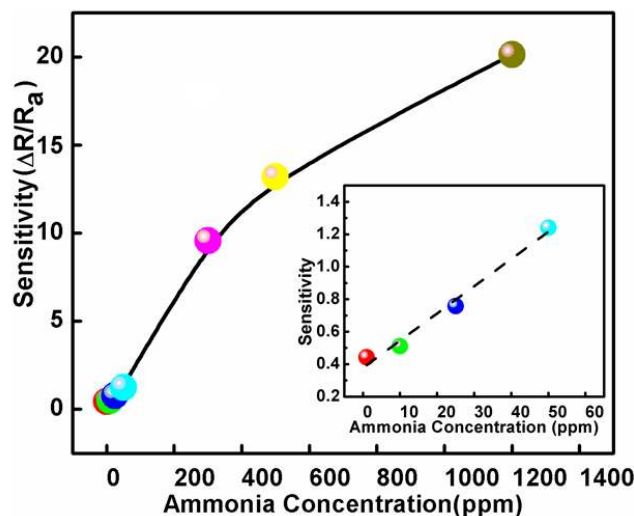
**FIGURE 3.** (a) Steady state response and its signal shape, (b) Response time plot, (c) Recovery time plot, and (d) Repeatability plot, of the sensor towards 300 ppm ammonia concentration at room temperature (27°C) with 50% relative humidity.



**FIGURE 4.** Room temperature (27°C) sensing responses of sensor at different ammonia concentrations viz. 1, 10, 25 and 50 ppm (with 50% relative humidity).

the maximum relative response, response time and recovery time values were found to be 9.57, 13 s and 156 s respectively. For the sake of comparison, response time and recovery time values were also measured at 90% of maximum response and 90% fall of response, with corresponding values of 6 s and 37 s respectively. Fig. 3(d) shows that sensor can repeatedly sense ammonia without loss in performance.

In order to evaluate the ammonia concentration dependence of sensor, response is also measured in the range of 1 to 50 ppm. It can be seen that (Fig. 4) the relative response value increases with gas concentration, which can be attributed to the increase in deprotonation and/or localization of polarons in the PANI-DBSA sensing layer

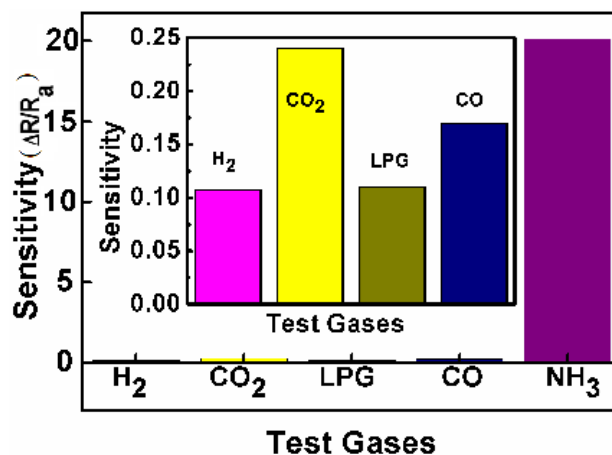


**FIGURE 5.** Relative Response versus concentration curve of PANI-DBSA thin film sensor (at room temperature (27°C) with 50% relative humidity). Inset shows the linear response region in 1-50 ppm range.

caused by increasing adsorptions of ammonia molecules on PANI-DBSA rod's surface. Further, the sensor was found to regain the initial value of sensor resistance, after removal of ammonia except for a small drifting in the base line.

The relative response versus ammonia concentration plot of our PANI-DBSA thin film sensor is shown in Fig. 5. In the range of 1-50 ppm gas concentration relative response value increases linearly from 0.443 to 1.241 (inset Fig. 5) with gas concentration relative response value of 0.01675 per ppm. As linearity is desirable in the device operation, the prepared PANI-DBSA sensing film shows considerable promise in the concentration regime of 1-500 ppm. Beyond 500 ppm, device has to be conveniently calibrated to find the relative response as a function of analyte concentration.

To analyze the selectivity of the fabricated PANI-DBSA thin film based sensor, its sensing response for five different gases viz. carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>), ammonia (NH<sub>3</sub>), hydrogen (H<sub>2</sub>) and liquefied petroleum gas (LPG) has been recorded at gas concentration of 1200 ppm (Fig. 6). The sensing responses towards these five interference gases is found to be orders of magnitude lower in comparison to ammonia, which clearly demonstrate relatively higher selectivity of prepared sensor towards the ammonia. This is crucial in avoiding false positive or negative alarms due to cross selectivity issues, and brings reliability into sensing systems. These selectivity experimental results are in agreement with [49] where Density Functional Theory (DFT) studies were carried out to evaluate the ability of the PANI as sensing film for different gases and it was concluded that PANI has greater response selectivity towards NH<sub>3</sub> compared to CO and CO<sub>2</sub>. The higher sensitivity for NH<sub>3</sub> as compared to other gases is due to pure redox reaction taking place between ammonia molecules with surface of PANI-DBSA nanorods which leads to deprotonation of the PANI [7]. This undoping leads to reduction of charge carriers and increases resistance. For other mentioned gases the reaction is not pure



**FIGURE 6.** Response of PANI-DBSA based sensor towards different interfering gases at 1200 ppm ammonia concentration. Inset shows the enlarged view of the response for gases other than ammonia, clearly showing their response and direction of resistance change.

redox reaction at room temperature and under mild condition. Therefore, it is difficult to detect them by their chemical reaction with PANI-DBSA. However, they may have weak physical interactions with PANI-DBSA (like absorbing or swelling of the polymer matrix) and partial charge transferring [50]. These phenomena do not change the doping levels of the PANI-DBSA, but influence the properties of the PANI-DBSA thin film, thereby, making them detectable by PANI-DBSA thin film but with very low sensitivity as compared to NH<sub>3</sub>.

All these experiments were performed at room temperature (27°C) with 50% relative humidity. Polymers like PANI and its variants are sensitive to humidity, but PANI-DBSA directly tends to interact less with moisture compared to HCl doped PANI due to its hydrophobic nature (due to long hydrocarbon tail of dopant). Also, it is well reported in the literature that PANI-DBSA sensors have been found to display only a slight and negligible change in response for variation in relative humidity compared to effect of dopant [51]. To verify this literature's claim for our fabricated sensor, it's important to perform experiments to check the influence of humidity on the prepared sensor. To analyze the effect of humidity on fabricated sensor device, the change in resistance value is recorded by varying the relative humidity from 20% to 80% in steps of 10% (Fig. 7). For each step change a time of 40 minutes is given for stabilization of the recorded resistance value. The environmental temperature is kept at 30°. EPSEC environmental chamber is used for controlling humidity. It is observed that as the value of relative humidity increases (from 20% to 80%), the resistance decreases (from 0.20 MΩ to 0.03 MΩ). The average relative change in resistance for single step of 10% humidity variations is 0.156, which is small and negligible compared to sensor's relative response (9.57) due to effect of dopants.

In order to study the thermal stability of the sensor, the effect of the temperature on sensor's performance is analysed. It is found that the relative response of the sensor for

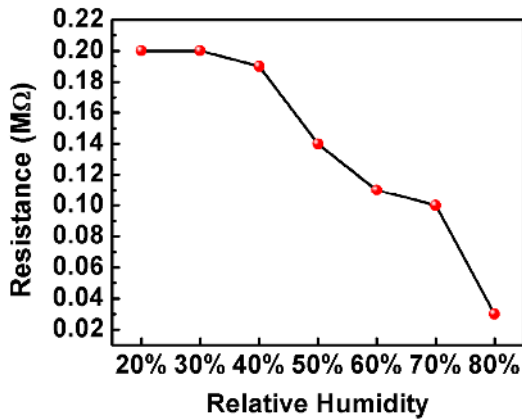


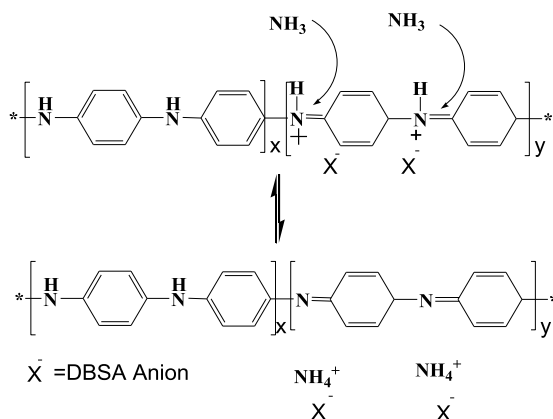
FIGURE 7. Change in resistance value of the sensor thin film with variations in relative humidity from 20% to 80% in steps of 10%.

TABLE 1. Effect of Temperature on Sensing Response of the Sensor.

Temperature (°C)	Relative Response (at 50 ppm NH <sub>3</sub> Concentration)
27	1.240
45	0.162
65	0.116
80	0.078
95	0.068

ammonia decreases with increase in temperature (Table 1). This is due to shift of the adsorption-desorption equilibrium in the desorption direction [52]. This experiment is performed at 50 ppm NH<sub>3</sub> concentration.

To better understand the performance of our sensor and compare it with literature, the general sensing mechanism is revisited. As the ammonia molecules comes in contact with surface of PANI-DBSA nanorods, it interacts with the same and tends to deprotonate the PANI [7]. Such undoping leads to reduction of charge carriers and increases resistance. The chemical reaction taking place between ammonia molecules and PANI-DBSA layer is pure redox reaction and is shown below.



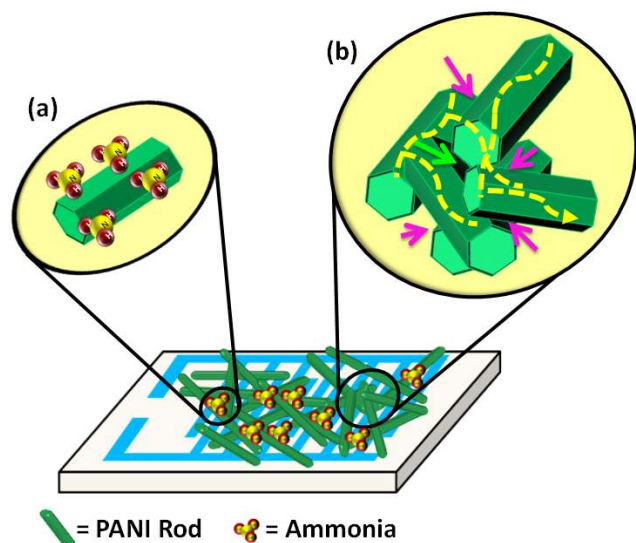
The ammonia (NH<sub>3</sub>) molecules take up protons from PANI and form ammonium ions (NH<sub>4</sub><sup>+</sup>) while PANI itself turned into its base form. This process is reversible and when

TABLE 2. Materials, Relative Response, Response time and Recovery time of the various Ammonia gas sensors.

Ref.	Materials Used	Concentration (ppm)	Relative Response	Response time (s)	Recovery time (s)
[55]	PANI-CSA-SWCNT	400	0.50	-	-
[53]	SnO <sub>2</sub> -PANI	300	0.16	12-15	80
[54]	PANI-HCl	300	0.1	-	-
[54]	PANI-HCl/graphene	300	0.4	-	-
[56]	PANI-PSSM	250	0.95	360	1440
[57]	TiO <sub>2</sub> -PANI/PA6	250	1.4	-	-
[58]	PANI	140	1.3	-	47
[58]	PANI-TiO <sub>2</sub>	140	9.2	-	20
[51]	Inkjet Printed PANI	100	0.0024	90	90
[56]	PANI-PSSM	100	0.5	231	458
[59]	CSA doped PANI-SnO <sub>2</sub>	100	0.9	45	3300
[7]	PANI-DBSA	100	3.3	27	58
[38]	PANI-DBSA	100	0.64	72	227
[39]	PANI-DBSA	20	0.145	300	900
<b>Our Work</b>	<b>PANI-DBSA</b>	<b>300</b>	<b>9.57</b>	<b>6</b>	<b>37</b>
<b>Our Work</b>	<b>PANI-DBSA</b>	<b>50</b>	<b>1.24</b>	<b>346</b>	<b>94</b>

ammonia atmosphere is removed the ammonium ion (NH<sub>4</sub><sup>+</sup>) decomposes to ammonia (NH<sub>3</sub>) gas and proton [50]. The initial fast rise in resistance is due to surface adsorption whereas the following slow resistance rise is due to ammonia diffusion across film's thickness (Surface adsorption phenomenon is faster than diffusion). Similarly, the sensor's resistance recovery is due to gas desorption and its diffusion back to pure carrier gas.

The comparative analysis of our PANI-DBSA nanorods based ammonia sensor with previously reported ammonia sensors is shown in Table 2. Among previously reported systems, Deshpande et. al [53] and Z. Wu et. al [54] presented their sensors' performance at 300 ppm ammonia gas concentration. Our PANI-DBSA nanorods based ammonia sensor shows much better performance (relative response, response time, and recovery time) as compared to these two sensors. The performance of all other ammonia sensors reported in the literature is for concentrations different than 300 ppm and 50 ppm. As sensor performance parameters change with ammonia concentration, therefore, apples to apples comparison of our sensor is not possible with other reported sensors mentioned in Table 2. However, it can be concluded from Table 2 that our PANI-DBSA nanorods based active layer show relatively better ammonia sensing performance than previously reported systems. This is attributed to the formation of PANI-DBSA nanorods, that facilitates the availability of their exterior surface for adsorption of ammonia molecules (Fig. 8(a)) leading to immediate undoping and resistance rise with high relative response value. Further, the active layer consisting of nanorods network over IDEs (Fig. 8) actually forms porous system (one such porous channel is pointed by green arrow in Fig. 8(b)) due to non-planar



**FIGURE 8.** Active layer consisting nanorods over IDEs and their enlarged version showing (a) Ammonia adsorption on exterior surface of nanorod, (b) Inter- and intra-rod carrier transport via overlapping individual rods.

**TABLE 3.** Effect of Thin Film Thickness on Sensing Response of the Sensor.

Thickness of Thin Film (nm)	Spin Coating RPM (Spin Time of 30 Seconds)	Relative Response (For $\text{NH}_3$ at 50 ppm)
350	2000	1.24
740	1000	1.14
980	500	0.97

configuration assumed by finite thickness overlapping rods (Fig. 8(b), marked by magenta arrows) compared to nanofibrous [38], nanobelts, or nanoparticulate systems [39], [7]. This in turn enables fast diffusion of ammonia across entire film thickness, resulting in faster response/recovery. Besides, such networks facilitate the charge transport across the active layer through improved inter- and intra-rod carrier transport, which again contribute to improvement of response. Overall, the above advantages, collectively contribute toward improved transduction in terms of relative response and response time. We believe that this strategy may be adopted to synthesize large area sensor arrays for realization of reliable, reusable and viable ammonia detectors and sensing devices.

To study the effect of PANI-DBSA film thickness on sensor's sensing response for ammonia ( $\text{NH}_3$ ), we fabricated three sensors of different thickness values by using spin coating method. We used spin coating at 2000 RPM (Rotations Per Minute), 1000 RPM, and 500 RPM. The resulted PANI-DBSA thin film thickness values were 350 nm, 740 nm, and 980 nm (variation within  $\pm 15\%$  for 350 nm and within  $\pm 10\%$  for 740 nm and 980 nm) corresponding to 2000 RPM, 1000 RPM, and 500 RPM, respectively. From this experiment it was observed that sensitivity (i.e. relative response) of the sensor for ammonia ( $\text{NH}_3$ ) decreases with increasing thickness of the DBSA doped PANI thin

film (Table 3). This trend is in agreement with what is reported in the literature [60]. To improve the sensitivity (i.e. relative response) of the sensor, the sensing PANI-DBSA film must be thin. However, very thin film breaks, therefore, we have used thin film layer of thickness about 350 nm and all the gas sensing results reported in this research paper are for PANI-DBSA thin film layer of thickness about 350 nm.

#### IV. CONCLUSION

Chemi-resistive ammonia sensor consisting of emulsion polymerized PANI-DBSA rods network based active layer has been fabricated via spin coating of chloroform dispersion of PANI-DBSA onto the Pt-IDE substrate. Our sensor can efficiently detect upto 1 ppm ammonia with good linearity in the 1-50 ppm range. Further, it can also detect higher ammonia concentration upto 1200 ppm while maintaining good selectivity. Particularly, being benefitted from high surface area and porous networks of highly doped PANI rods, formed sensing platform displays relative response value of 9.57, response time of 6 s, and recovery time of 37 s along with good reversibility and repeatability even at 300 ppm of ammonia. Overall, the room temperature operation and good sensor linearity with relative response per ppm value of 0.01675 demonstrates the good sensing characteristics and reflect its high potential for realizing low cost, simple, and efficient ammonia sensors.

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