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Humidity Sensing Behavior of Polyaniline / Strontium Arsenate Composites

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Abstract: The response of conducting Polyaniline (PANI) / Ceramic ($Sr_3(AsO_4)_2$) composites system to air moisture environment is studied. The conducting PANI and its composites are prepared by in situ polymerization technique. These prepared samples were characterized by XRD, FTIR & SEM, which confirms crystallinity, composite formation and porosity of the samples. The temperature dependent conductivity measurement shows the thermally activated behavior, where the conductivity increases with increase in temperature. The decrease in electrical resistance with change in relative humidity (RH) over broad range (ranging between 20 to 95 %) is due to the increase in surface electrical conductivity resulting from moisture absorption and due to capillary condensation of water causing increase in conductivity within the sensing materials. *Copyright © 2009 IFSA.*

Keywords: Polyaniline, Ceramic, Strontium arsenate, Humidity sensor, Time response

1. Introduction

Humidity sensors are needed for humidity control, both for residential applications and for industrial processes. Sensing materials under investigation can be grouped into three main categories: organic polymers, electrolytes, and ceramics. Comprehensive reviews on humidity sensors can be found in the literature [1-4]. Numerous kinds of materials have been utilized for humidity sensing [5]. Out of them, metal oxides which are physically and chemically stable, have been widely investigated for humidity

detection at both elevated and room temperatures [6–13]. Many ceramic materials have been tested for humidity sensing, like $MgAlO_4$ [14], thick film titanate TiO_2-Cu_2O-Na_2O [15], linear behavior of $MnWO_4$ and Mn_3O_3 [16], Cr_2O_3/ WO_3 [17] and $ZnCr_2O_4$-$K_2Cr_2O_4$ ceramic system [18]. Conducting polymers have been widely studied for the past two decades because of their electrochromic properties for use in batteries, electrochromic devices, sensors, etc. [19-23]. Among the conducting polymers, polyaniline is one of the most intensively studied conducting polymer. Polyaniline has attracted considerable attention for the preparation of its composites with inorganic particles, the combination of organic / inorganic composites have been used for humidity sensing, such as conducting Polyaniline – Barium titanate composite [24], Polyaniline – lamellar bentonite / vanadium(V)oxide (BV) matrix, Interestingly, in small amounts, polyaniline dramatically increases the conductivity and charge-capacity of the BV matrix [25], conducting Polyaniline – inorganic salt composite [26], and Polyaniline / V_2O_5 composite [27].

In the present communication, authors reports organic / inorganic composites for humidity sensors, where Polyaniline as organic part and Strontium arsenate ($Sr_3(AsO_4)_2$) as inorganic part. The above composites have been studied for its response to change in humidity.

2. Experimental

All Chemicals used were analytical grade (AR). The monomer aniline was doubly distilled prior to use. Ammonium persulphate ($(NH_4)_2S_2O_8$), Hydrochloric acid (HCl), Strontium Arsenate (Sr_3 $(AsO_4)_2$), were procured and were used as received. Synthesis of Polyaniline – Strontium Arsenate (Sr_3 $(AsO_4)_2$) composites has been carried out by in situ polymerization. 0.1 mol of aniline was dissolved in 1 M HCl to form aniline hydrochloride. Strontium arsenate (Sr_3 $(AsO_4)_2$) is added in the weight percent to the above solution with vigorous stirring in order to keep the strontium arsenate suspended in the solution. To this reaction mixture, 0.1 M of ammonium persulphate [$(NH_4)_2S_2O_8$] which acts as the oxidant was added slowly with continuous stirring for 4 – 6 h at 0 – 5 ^{0} C. The precipitated powder recovered was vacuum filtered and washed with deionised water. Finally the resultant precipitate was dried in an oven for 24 hours to achieve a constant weight. In this way 5 different PANI / Sr_3 $(AsO_4)_2$ composites with wt % of Sr_3 $(AsO_4)_2$ (10, 20, 30, 40 and 50 %) in Polyaniline have been synthesized [28-29]. The pellets of 10 mm diameter are formed with thickness varying up to 2 mm by applying pressure of 10 Tons in a UTM – 40 (40 Ton Universal testing machine). For temperature dependent conductivity and sensor studies, the pellets are coated with silver paste on either side of the surfaces.

The characterization studies are employed on all the above synthesized PANI/ $Sr_3(AsO_4)_2$ composites. The characterization technique like X – Ray diffraction on PANI/ $Sr_3(AsO_4)_2$ composites was employed by using Phillips X – ray diffractometer (PW3710) with Cu Kα as source of radiation. FTIR spectra of these composites were recorded on a Perkin – Elmer 1600 spectrophotometer in KBr medium and SEM has been carried out on Phillips XL 30 ESEM.

Measurement of temperature dependence of electrical conductivity has been carried out using Keithely - 2000 multimeter, USA.

The samples in the pellet form were used for humidity sensing. The planar resistance of the sensor was recorded by controlling the humidity in a closed glass chamber at room temperature. The humidity was first lowered by keeping $CaCl_2$ in a chamber. Controlled water vapors at room temperature were then introduced steadily for increasing the humidity inside the chamber from 20 to 95 % RH. Relative humidity inside the chamber was monitored and measured by a standard pre-calibrated humidity meter Mextech-DT-615.

3. Results & Discussions

Fig. 1 shows the X-ray diffraction pattern of pure PANI, $Sr_3(AsO_4)_2$ & PANI/$Sr_3(AsO_4)_2$ composite with 50 wt % of Sr_3 $(AsO_4)_2$ in PANI. It is seen from this figure that the Rhombohedral peak of $Sr_3(AsO_4)_2$ indicates the crystalline nature of the composite. By comparing the XRD pattern of these composite with that of $Sr_3(AsO_4)_2$, the prominent peaks corresponding to $2\theta = 32.1^0$, 38.63^0, 42.89^0, 56.71^0 and 68.35^0 are due to (110), (202), (024), (300) and (220) planes (JCPDS 13 – 0261) of $Sr_3(AsO_4)_2$. The XRD diffraction studies performed on all the samples (10, 20, 30, 40 and 50 wt % $Sr_3(AsO_4)_2$) show that the same peaks in all the cases, this is possible because of the formation of oxide particles in PANI matrix. By comparing the XRD patterns of these composite and $Sr_3(AsO_4)_2$, it is confirmed that Strontium arsenate has retained its structure even though it is dispersed in PANI during polymerization reaction [28 - 29].

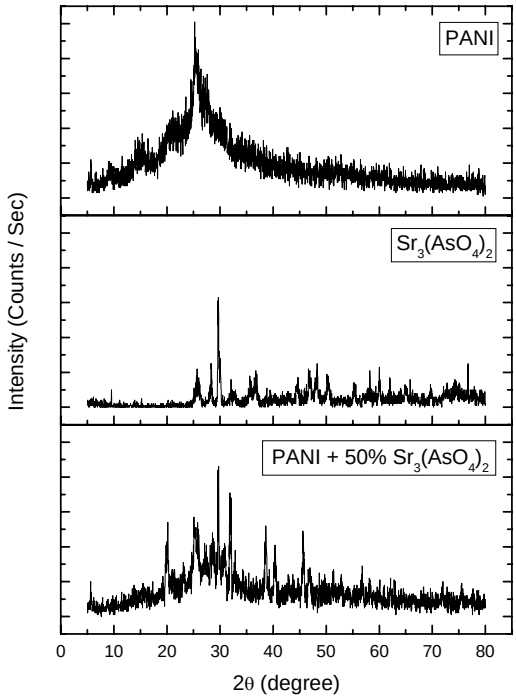


Fig. 1. X-ray diffraction of pure PANI, pure $Sr_3(AsO_4)_2$ & PANI/$Sr_3(AsO_4)_2$ composite.

The FTIR Spectra of pure PANI, $Sr_3(AsO_4)_2$ & PANI/$Sr_3(AsO_4)_2$ (50 wt %) composite is shown in Fig. 2. The important peaks observed in pure $Sr_3(AsO_4)_2$ are at 705 cm^{-1} and 800 cm^{-1} which are due to the presence of metal oxygen stretching frequencies. The characteristic stretching frequencies are observed at 3434 cm^{-1} for NH stretching, 1569 cm^{-1} for ring stretching, 1482 cm^{-1} for C=N stretching & C-C stretching, 1292 cm^{-1} for C-N stretching & CH bending, 1241 cm^{-1} for C-N stretching & C-C stretching, and 500 cm^{-1} & 797 for metal oxygen stretching. By comparing the IR spectra of PANI and PANI/$Sr_3(AsO_4)_2$ composite, it is observed that in the composite the characteristic stretching frequencies are shifted toward higher frequency side which may be attributed due to the Vander walls kind of interaction between $Sr_3(AsO_4)_2$ and PANI chain [30-31].

Fig. 3 shows the Scanning Electron Micrograph (SEM) of pure PANI & PANI/$Sr_3(AsO_4)_2$ composite (50 wt % of $Sr_3(AsO_4)_2$ in polyaniline). The SEM studies performed on all the samples indicates branched chain structure (or a branched morphology) with uniform distribution of crystalline $Sr_3(AsO_4)_2$ [28]. SEM also reveals the presence of $Sr_3(AsO_4)_2$ in polyaniline which is homogeneously

distributed through out the polymer sample and presence of capillary pores facilitate the sensing behaviour of the composite.

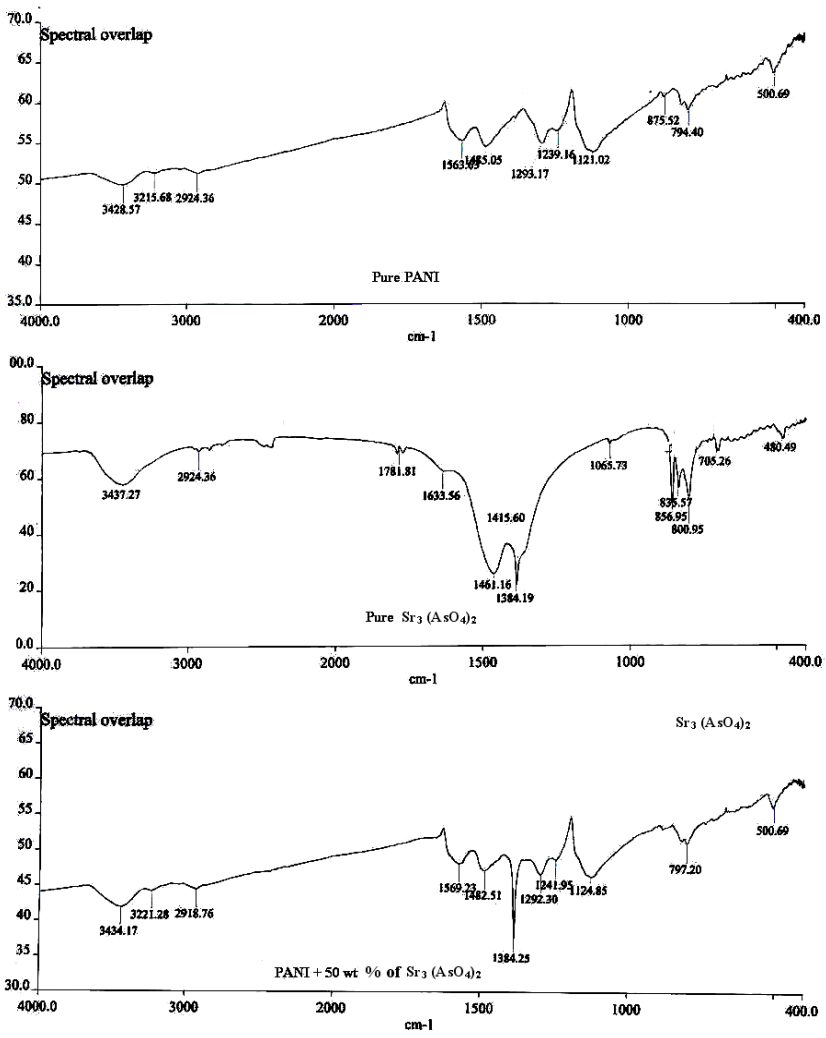
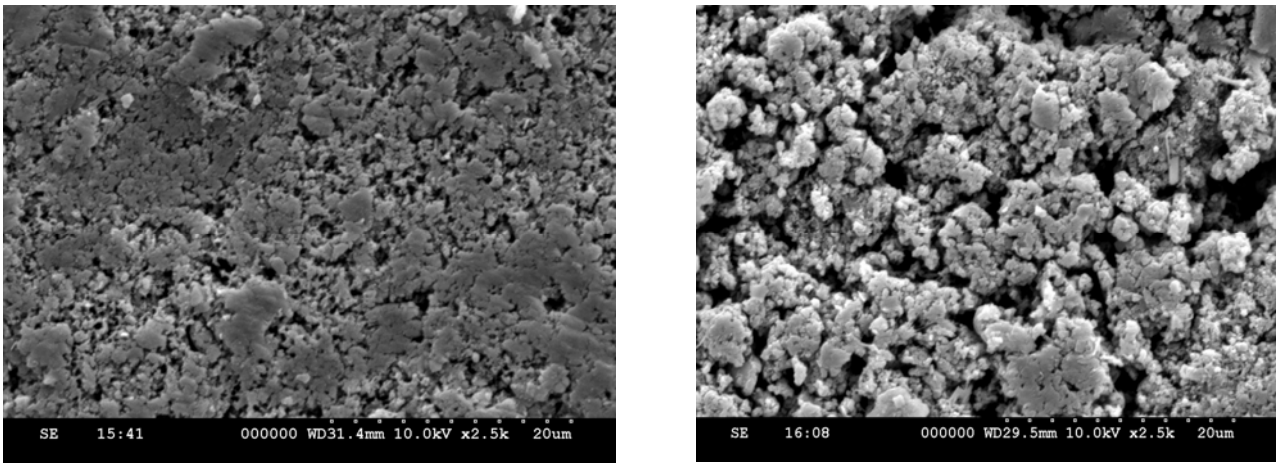


Fig. 2. FTIR Spectra of pure PANI, pure $Sr_3(AsO_4)_2$ & PANI/$Sr_3(AsO_4)_2$ composite.



(a) (b)

Fig. 3. (a). SEM image of Pure PANI, (b). SEM image of PANI / $Sr_3(AsO_4)_2$ composite.

Fig. 4 shows the variation of log of dc conductivity as a function of temperature which suggests that as temperature increases conductivity also increases, therefore thermally activated behavior of conductivity has been confirmed. The increase in conductivity is due to the increase of efficiency of charge transfer between $Sr_3(AsO_4)_2$ and polymer chains with increase in temperature [32, 33]. It is also suggested that the thermal curling affects the chain alignment of the polymer, which leads to the increase of conjugation length and which in turn brings about the increase of conductivity. Also, there will be molecular rearrangement on heating, which make the molecules favourable for electron delocalization [34]. The temperature dependence of the conductivity for conducting polymers is expressed by a variable range hopping (VRH) model proposed by Mott [35]. According to this model, the behavior of electronic conduction in disordered and non-metallic materials is controlled by the thermally assisted hopping of electrons between localized states and is given by,

$$\sigma\,(T) \propto \exp\,[-\,T_0 / T^{\,1/(n\,-\,1)}\,] \tag{1}$$

$$k\,T_0 = \lambda\,\alpha^3/\rho_0\,, \tag{2}$$

where α is the coefficient of exponential decay of the localized states, ρ_0 is the density of states at the Fermi level and λ is a dimensional constant. However, many models have predicted as $\sigma \propto T^{-1/2}$.

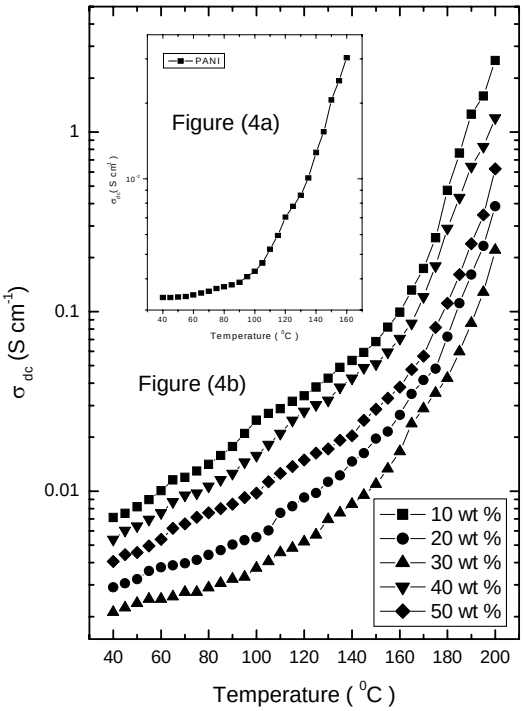


Fig. 4. (a) Variation of σ_{dc} as a function of temperature of pure PANI, (b) Variation of σ_{dc} as a function of temperature of PANI / $Sr_3(AsO_4)_2$ composites.

Fig. 5 shows the conductivity versus wt% $Sr_3(AsO_4)_2$ in PANI at three fixed temperatures viz., at 50, 100 and 150^{0}C. From the Fig. 5, it has been observed that the values of dc conductivity of these composites decreases up to 30 wt % of $Sr_3(AsO_4)_2$ in PANI and later it increases up to 40 wt %, further increase in wt% of $Sr_3(AsO_4)_2$ in PANI it decreases. The conductivity at 10 wt% and 40 wt % is due to the increase of efficiency of charge transfer between $Sr_3(AsO_4)_2$ and polymer chains. The decrease in conductivity of 20 wt %, 30 wt % and 50 w t% is due to blocking of charge transfer between $Sr_3(AsO_4)_2$ and polymer chains.

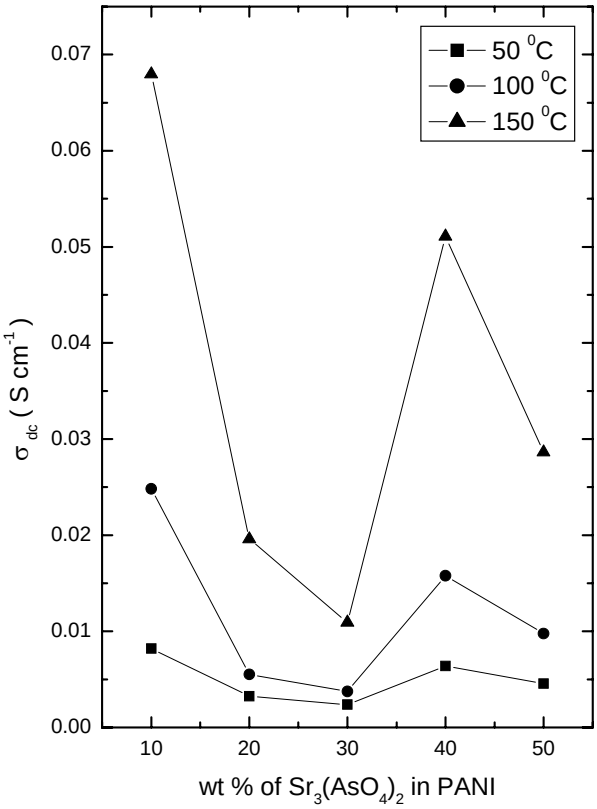


Fig. 5. Variation of σ_{dc} as a function of wt % of $Sr_3(AsO_4)_2$ in PANI at three fixed temperature.

Fig. 6 shows the variations in resistance as a function of relative humidity (RH) value for PANI/ $Sr_3(AsO4)_2$ composites in the form of pellets for five different wt% of $Sr_3(AsO_4)_2$ (10, 20, 30, 40 and 50 wt%) in PANI. The decrease in the resistance or increase in the conductivity with increasing relative humidity is shown in Fig. (6). This increase in conductivity may be attributed to the mobility of the $Sr_3(AsO_4)_2$ ion which is loosely attached to the polymer chain by weak Vanderwalls forces of attraction. The conductivity depends on capillary condensation of water causing change in conductivity within the sensing materials [36]. The resultant surface conductivity increases with increase of humidity due to combined effect of the proton type hopping and the ionic conduction [37]. At low humidity, the mobility of the $Sr_3(AsO_4)_2$ ion is restricted because under dry conditions the polymer chains would tend to curl up into compact coil form. On the contrary, at high humidity, the polymer absorbs water molecules and followed by the uncurling of the compact coil for into straight chains that are aligned with respect to each other. This geometry of the polymer is favourable for enhanced mobility of the $Sr_3(AsO_4)_2$ ion or the charge transfer across the polymer chains and hence the conductivity increases[28-29]. Fig. 7 shows the variation of resistance with weight percent of Sr_3 $(AsO_4)_2$ in polyaniline for three fixed relative humidity of 20, 50 & 90 % RH, where it was observed that as weight percent of $Sr_3(AsO_4)_2$ in PANI increases the resistance increases, hence conductivity decreases. This could be due to an increase in the disorderliness of the composite with increasing amount of $Sr_3(AsO_4)_2$ which is also seen from SEM data. $Sr_3(AsO_4)_2$ particles could possibly induce conformational changes in PANI, leading to a reduction in the order and a consequent reduction in the delocalization length, which is reflected with a decrease in conductivity with increase in wt%.

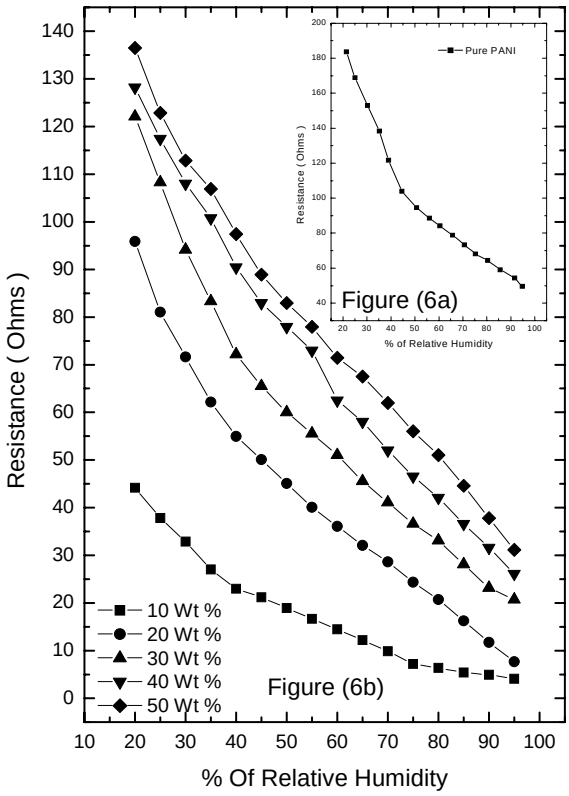


Fig. 6. (a) Variation of resistance as a function of humidity (% RH)of pure PANI, (b) Variation of resistance as a function of humidity (% RH) of PANI/$Sr_3(AsO_4)_2$ composites.

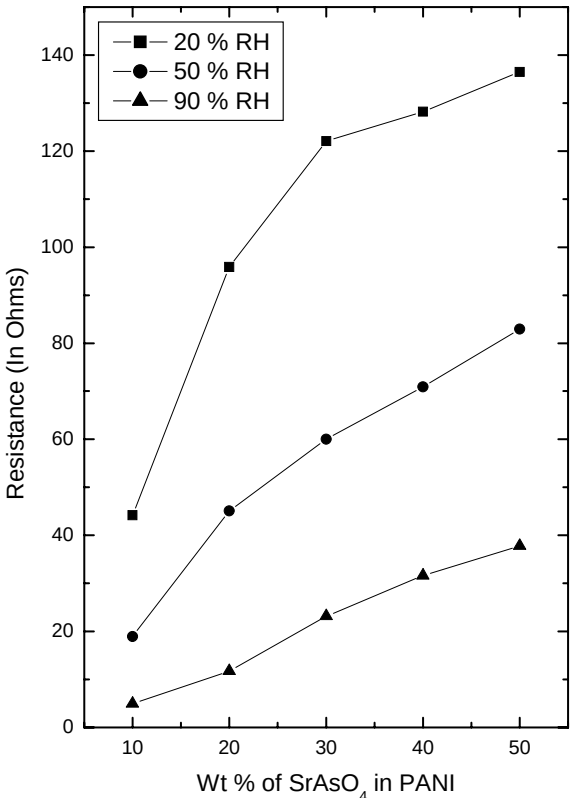


Fig. 7. Variation of resistance as a function of wt % of $Sr_3(AsO_4)_2$ in PANI at three fixed humidity (%RH).

4. Conclusions

PANI / $Sr_3(AsO_4)_2$ composites are synthesized by in situ polymerization method. Characterization using XRD confirms the structure retention of $Sr_3(AsO_4)_2$ in PANI matrix. The presence of amine, rings, metal oxide groups and formation of polyaniline / $Sr_3(AsO_4)_2$ composites as been observed from FTIR studies. It was shown from SEM micrograph that there is highly branched structure with uniform distributed grains in polyaniline. The temperature dependence conductivity is found to be thermally activated behavior of the PANI/$Sr_3(AsO_4)_2$ composites, where the conductivity increases with increase in temperature for all the PANI/$Sr_3(AsO_4)_2$ composites. The decrease in the resistance or increase in the conductivity with increasing relative humidity can be attributed to the mobility of the $Sr_3(AsO_4)_2$ ion, which is loosely attached to the polymer chain by weak Vanderwalls forces of attraction, and also due to capillary condensation of water causing change in conductivity within the sensing materials. As wt% of $Sr_3(AsO_4)_2$ increases in PANI, the disorderliness with in the sensing material increases and porasivity decreases, hence there is increase in resistance. These composites are sensitive to broad humidity range from 20 to 95 % of RH. Hence PANI/$Sr_3(AsO_4)_2$ composites are found to be promising sensing materials for broad humidity range.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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