STUDIES ON DC CONDUCTIVITY AND LPG SENSING BEHAVIOR OF PANI/V2O5 NANO COMPOSITES

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ABSTRACT
Sensing of Liquefied Petroleum Gas (LPG) using polyaniline (PANI)/ Vanadium Pentoxide(V2O5) has been studied in the present work. The conducting polyaniline / Vanadium Pentoxide composites were prepared by in situ polymerization with 10, 20, 30, 40 and 50 wt% of V2O5 in polyaniline. The composites thus formed were characterized by Fourier infrared spectroscopy (FTIR), X-ray diffractometry (XRD) and scanning electron microscopy (SEM), which confirmed the presence of V2O5 in polyaniline matrix and the formation of the composite .DC conductivity studies show thermally activated behavior of all the composites. The conductivity was found to increase with the increase in temperature indicating the semiconducting behavior of all the compositions. Maximum conductivity was observed in 30 wt% of V2O5 in polyaniline. On exposure of the composites to LPG, increase in resistance was observed with the increase in gas concentration. Maximum sensitivity for gas sensing was observed in the composite of 30 wt% V2O5 in polyaniline

KEYWORDS: *Polyaniline, Vanadium Pentoxide, nano-crystalline, DC conductivity, LPG sensing.*

I. INTRODUCTION

Vanadium oxides are typical polyfunctional n-type semiconductor materials and have attracted strong interest over the past years for their electrical, optical, electrochemical properties: metal-to-insulator transitions, electrical switching of V_2O_5, high ion interaction capacity, high sensitivity of active elements for gas sensors, etc. Vanadium pentoxide in different forms depends on the conditions of preparation [1-7]. Polyaniline is one of the typical conductive polymers which are usually considered as p-type material used in making light weight battery electrode, electromagnetic shielding device, anti-corrosion coatings and sensors [8-11]. In the recent past, the conducting polymer-based nanocomposite have drawn attention in their application as gas sensing [12-17]. Therefore, Pani-V_2O_5 composites have been most intensively studied among various composites, because it could combine the merits of Pani and crystalline V_2O_5 within a single material and are expected to find applications in electro chromic devices, photo electrochemical devices, nonlinear optical system, and sensors.

In present work, attempts have been made to synthesize the V_2O_5 particles and PANI- V_2O_5 composite. The characterization has been carried out by X-ray diffraction, Fourier transform infrared spectroscopy and scanning electron microscopy. The dc conductivity measurements are done by using two probe set-up and the sensor studies of the sample using the laboratory set-up, has been discussed.

II. EXPERIMENTAL METHODS

Synthesis of V_2O_5 nano-particles by sol–gel method is given in the flow chart shown in Fig. 1(a). First, a sol solution consisting Vanadium nitrate hydrate (as inorganic basic reactant), citric acid (as complexing agent), double-distilled water, with specific weight percentages was prepared. As shown in Fig. 1(a), the resulting mixture was stirred and dissolved at 40 ^{0}C for 24 hours until a clear solution was obtained (pH ¼ 4.5). This solution was refluxed at T = 80 ^{0}C for 1h. During refluxing, the solution turned into a metal-citrate homogeneous complex with a slight colour change from milky white to clear solution. The completion of both the reactions gives rise to the development of the

complex and the evaporation of the solvent forms a gel. Further, the gel was slowly heated at T =100^{0}C for 3 h in a hot oven. During continued heating at this temperature, the chelating between metal cations and citric acid as complex- ing agent is developed [18]. This step helps in achieving a proper stoichiometry and control of the particle size without any need of a special atmosphere. In addition, this improves uniformity of the distribution of the metal cations in the solution. In the final step of the sol–gel process, the wet gel was fully dried by direct heating on the hot plate at T= 200^{0}C for 5 h. The resulting white powder (V_2O_5) was obtained.

The monomer aniline was distilled twice before use. Analytical reagent-grade Ammonium persulfate [$(NH_4)_2S_2O_8$], Hydrochloric acid (HCl) and Vanadium Pentoxide (V_2O_5) were used for synthesis. The polyaniline – Vanadium Pentoxide composites were synthesized by in situ polymerization .Aniline solution was formed by dissolving aniline (0.1 mol) in 1M HCl. Vanadium Pentoxide was added to the aniline solution with vigorous stirring to keep Vanadium Pentoxide suspended in the solution. 0.1M Ammonium persulfate, which acts as the oxidant, was added to this reaction mixture slowly with continuous stirring at 0–5°C. The reaction mixture was kept stirring for 24 hours. The polymer in the form of greenish precipitate was recovered by vacuum filtration and washed with deionized water. To achieve a constant weight the precipitate was dried for 24 hours in an oven. In this way polyaniline – Vanadium Pentoxide composites with 5 different wt % of V_2O_5 (10, 20, 30, 40, 50) in polyaniline were synthesized.

2.1 Measurements

The FTIR spectra of the samples were recorded on a Perkin Elmer 1600 spectrophotometer in KBr medium. X-ray diffraction studies were performed by using Philips X-ray diffractometer with Cu Kα as the radiation source. The morphology of the composites in the form of powder was investigated using scanning electron microscope (SEM) Model-EVO-18 Special Edison, Zeiss, Germany.

For temperature dependent DC conductivity studies and sensing studies, the test samples were prepared in the pellet form (10 mm diameter and thickness varying up to 2 mm) by applying pressure of 10 tons in a Universal testing machine. The pellets were coated with silver paste on either side. Temperature dependent electrical conductivity was measured from 30^{0}C to 180^{0}C using Keithley 6514 electrometer. For gas sensing, the pellets were kept in the gas sensing chamber. With the help of a regulator and a flow meter, LPG is allowed to enter the chamber at a constant rate of 20 ml/min. The variation in resistance of the composite pellets with increase in gas concentration is recorded at a regular interval of 20 seconds using a high accuracy dot-tech meter.

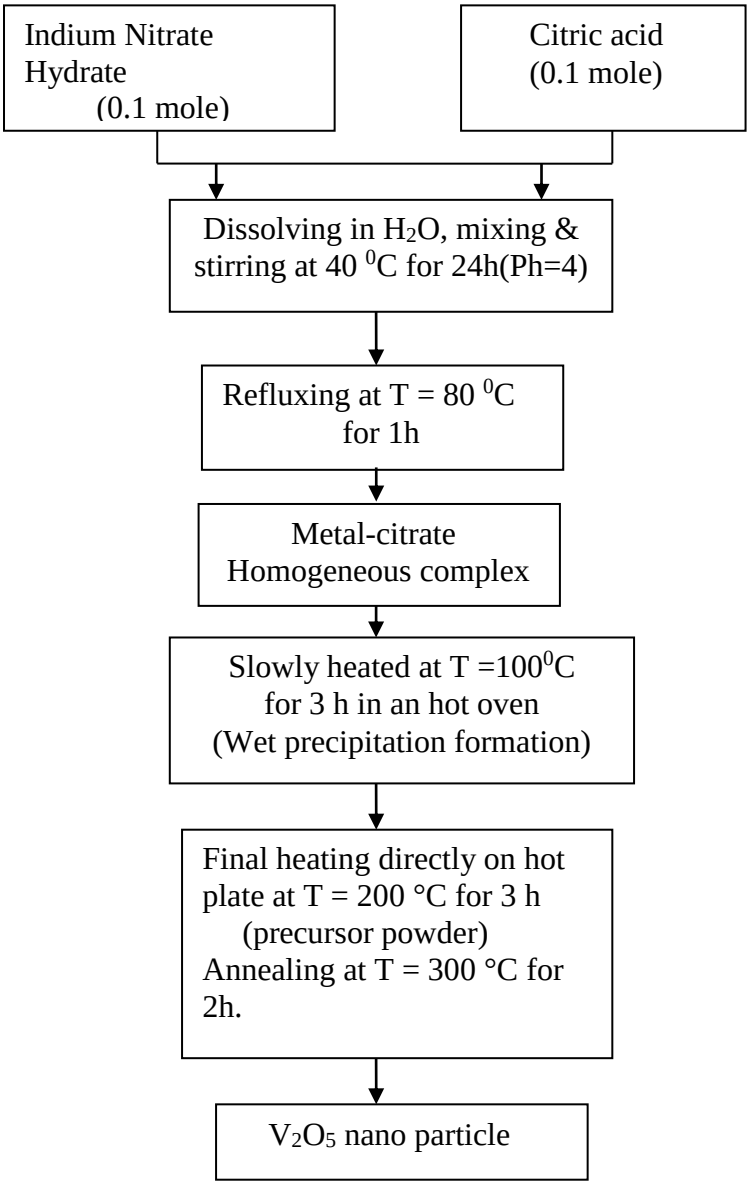


Fig.1 (a) The flow chart for preparation of V_2O_5 nano-particle by sol-gel method.

III. RESULTS AND DISCUSSION

3.1 FTIR Spectra

The Figure 2(a) shows FTIR spectra for pure Polyaniline. The absorption peaks are found to be at 2924.12 cm^{-1},1603 cm^{-1},1574.72cm^{-1}, 1494.11cm^{-1}, 1302.17cm^{-1}, 1146cm^{-1}, 734cm^{-1}, and 502 cm^{-1}, The formation of polyaniline is confirmed by noticing the predominate peak at the wave number of 1603 cm^{-1} .The peak at 2924.12 cm^{-1} is due to CH_2 asymmetric stretching. The intense peaks at 1574.72 cm^{-1} and 1494.11cm^{-1}may be attributed due to the presence of quinoid (N=Q=N) and benzenoid (N=B=N) ring stretching , 1302.11cm^{-1} is due to N-H deformation,1146 cm^{-1} due to C-O-C stretching of excess oxidant, 734 cm^{-1} for C-H vibration of Para coupling benzenoid ring and 502 cm^{-1} bond corresponding to aromatic ring [19].

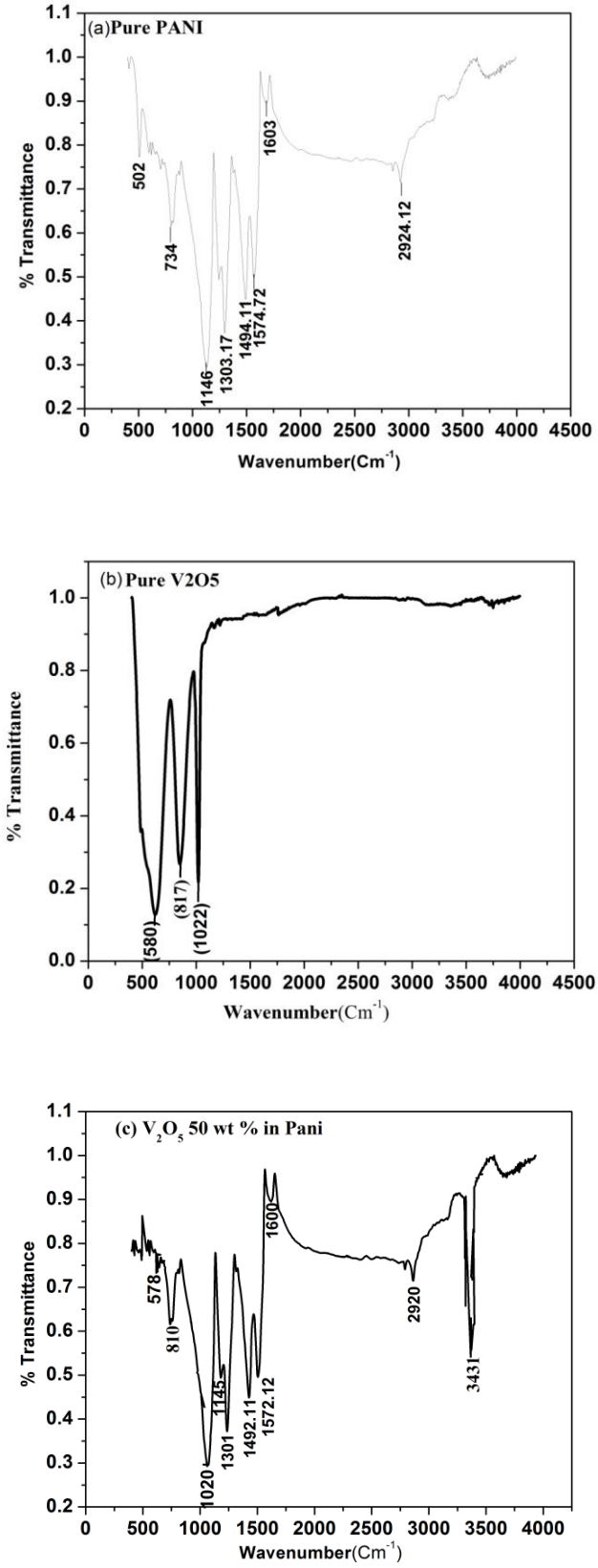
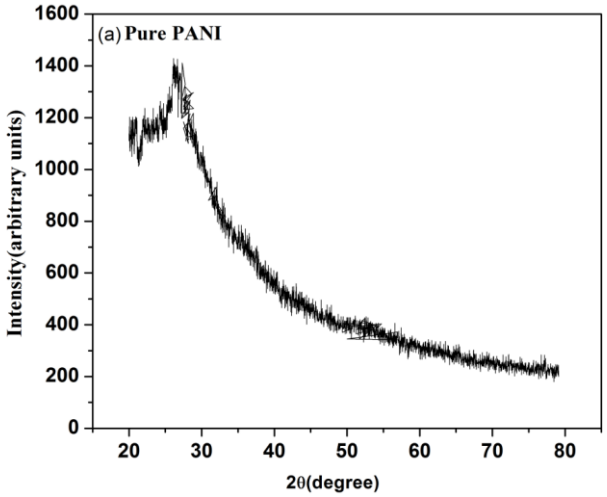
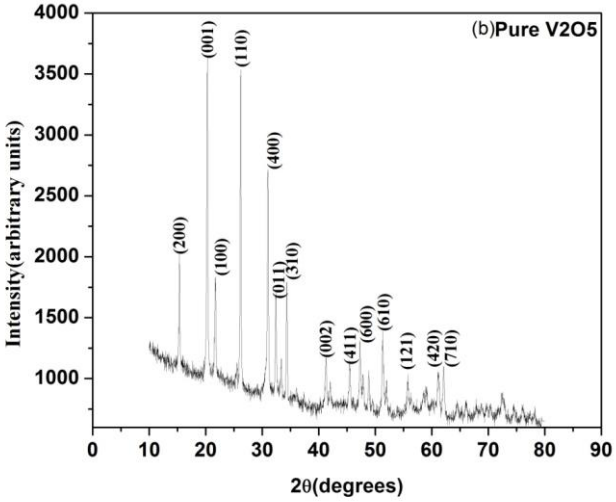


Fig. 2: (a) FTIR Spectra of pure polyaniline. (b) FTIR Spectra of pure $V2O_5$ (c) FTIR Spectra of 50 wt % of V_2O_5 in polyaniline.

Figure 2(b) shows the FTIR spectrum of the vanadium pentoxide which is characterized by three absorption bands centred at 1022 cm^{-1}, 817 cm^{-1} and 580 cm^{-1}. The first band at 1022 cm^{-1} is assigned to the V=O stretching, the last two at 817 cm^{-1} and 580 cm^{-1} are due to V-O-V deformation modes[20]. Figure 2(c) shows the FTIR spectrum of polyaniline / V_2O_5 composite with 50 wt % of V_2O_5 in polyaniline. The absorption peaks are found to be at 3431cm^{-1} , 2920cm^{-1}, 1600cm^{-1}, 1572.12 cm^{-1}, 1492.11 cm^{-1}, 1302 cm^{-1}, 1145 cm^{-1},1020 cm^{-1}, 810 cm^{-1}, 578 cm^{-1} which confirm the V_2O_5 presence in polyaniline. The spectrum shows that the absorption frequencies are slightly shifted towards lower side due to the weak Vander Waals force.

3.2 X – Ray diffraction





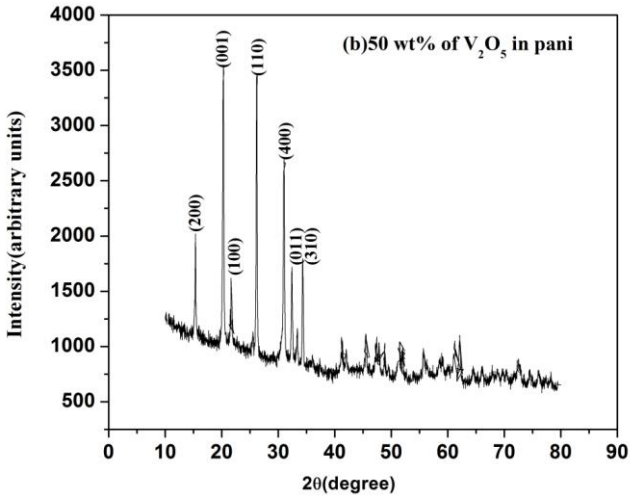


Figure 3: X-ray diffraction pattern of (a) pure polyaniline, (b) pure V_2O_5 (c) 50 wt % of V_2O_5 in polyaniline.

The Figure 3(a) shows X-ray diffraction pattern of Polyaniline. Careful analysis of X-ray diffraction of polyaniline suggests that it has amorphous nature with a broad peak centered on $2\theta \approx 25.53^0$ which corresponds to (200) diffraction planes of pure PANI [21].

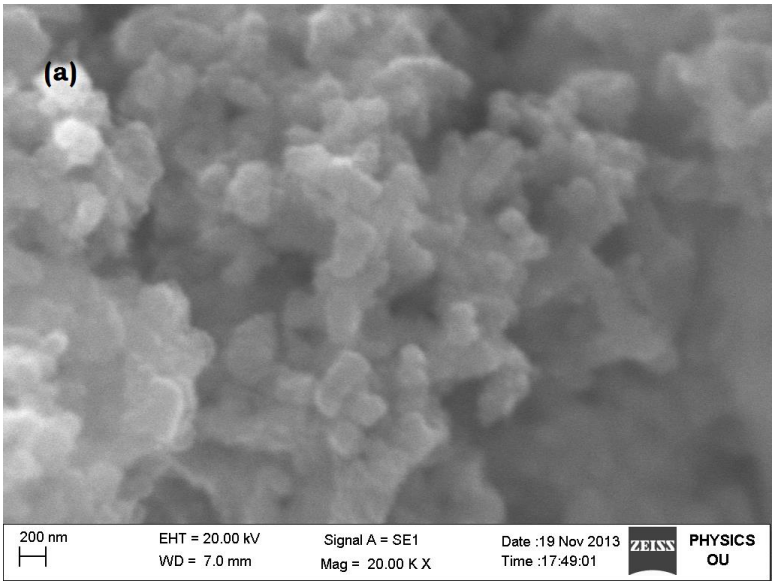
The XRD patterns of V_2O_5 nanoparticles are shown in Figure 3 (b) V_2O_5 synthesized by sol-gel method. These nanoparticles have shown good crystallinity because of the existence of sharp peaks in the XRD pattern. The crystallite size of the synthesized V_2O_5 nanoparticles was calculated using Scherer's formula given by $D=0.9\lambda/\beta\cos\theta$; where, D is the average crystallite size, λ is the X-ray wavelength (1.5405Å) and β is full width at half maximum in radian. The average crystallite size is found to be ~11 nm. In the XRD pattern, different lines are attributed to the (200), (001), (110), (400), (011), (310), (002), (411), (600), (601), (121), (420) and (710) planes are in good agreement with the data of V_2O_5 powder file (JCPDS number 09-0387) which corresponds to the orthorhombic crystalline structure. The XRD spectrum exhibits an intense (001) peak indicating preferential orientation in the <001> direction. This result reveals that the structure is such that the crystallographic c-axis is perpendicular to the substrate surface and the crystalline orientation is favoured [22]. Figure 3(c) shows the X-ray diffraction pattern of Polyaniline – V_2O_5 composite with 50 wt % of V_2O_5 in Polyaniline. In the XRD pattern, different lines are attributed to the (200), (001), (110), (400), (011), (310), (002), and (310) planes, by comparing the XRD pattern of the composite and V_2O_5, it is confirmed that V_2O_5 has retained its structure even though it is dispersed in PANI during polymerization reaction.

3.3 Scanning electron micrographs

Figure 4(a) shows that Scanning Electronic Micrograph (SEM) image of pure polyaniline. It is found from the image that polyaniline contains a highly elongated chain like structure. Polyaniline grains have well interaction with each other. The average grain size was calculated by using the linear intercept formula and it is found to be 243nm to 250nm.

Figure 4(b) shows the higher resolution SEM image of pure V_2O_5 and it is seen to be chain like structure. The average grain size was found to be 190 nm to 210 nm. The grains are found to be well interconnected with each other which indicate that they have enough binding energy to combine with neighbour grains or molecules.

Figure 4(c) shows the SEM image of V_2O_5/PANI composites. It is found to have highly agglomerated chain like structure. The crystallinity of the V_2O_5 is seen to decrease with the addition of PANI in it. The average grain size is found to be 74 nm to 79 nm.


(a)
200 nm
EHT = 20.00 kV
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Signal A = SE1
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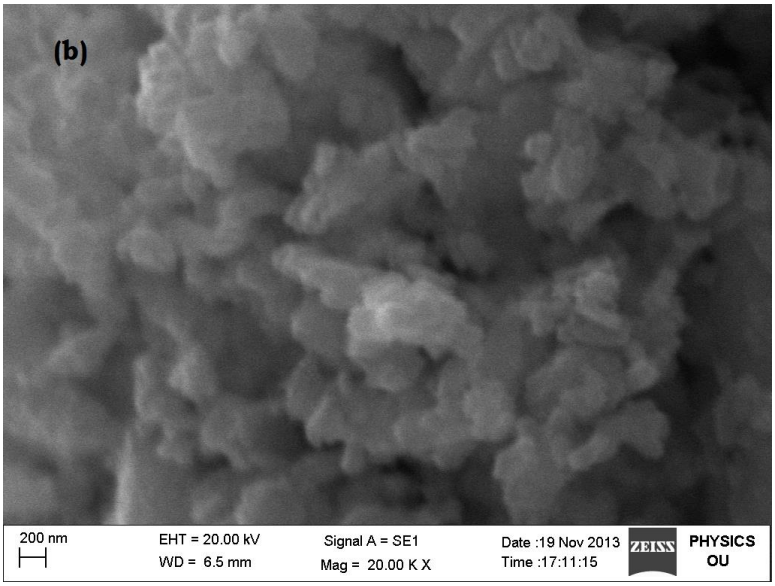
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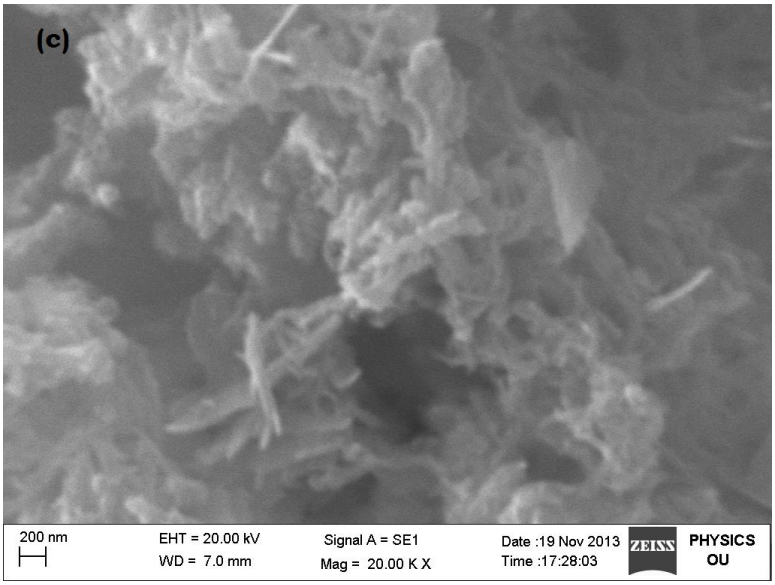

Figure 4: (a) SEM macrograph pure Polyaniline, (b) SEM macrograph pure V_2O_5, (c) SEM micrograph of polyaniline – V_2O_5 composite with 50 wt % of V_2O_5 in polyaniline.

3.4 DC Conductivity Studies

Figure 5(a) shows the σ_{dc} conductivity as a function of temperature for V_2O_5/PANI composites at various weight percentages. It is observed that the conductivity of the composites increases with increase in temperature ranging from 30^0C to 180^0C. Among all the V_2O_5 /PANI composites 30 wt% shows higher conductivity. This clearly indicates that the conductivity is not only the motion of ions (V_2O_5) but also hopping of charge carriers like polarons and bipolarons from one island to another.

It is also suggested here that the thermal curling effects of the chain alignment of the polyaniline leads to the increase in conjugation length and that brings about the increase of conductivity. Also, there will be molecular rearrangement on heating which makes the molecules favourable for electron delocalization. The conductivity varies directly with the temperature obeying an expression of the following form.

$$\sigma (T) = \sigma_0 \exp [- (T_0/T)^{¼}] \quad (1)$$

Where σ is the conductivity, T is the temperature, and σ_0 is the conductivity at characteristic temperature T_0. Conductivity varying with various values of the exponent (e.g., $T^{-1/4}$, $T^{-1/3}$and $T^{-1/2}$) has been reported and different models have been used to interpret this data.

Figure 5(b) shows the variation of dc conductivity as a function of different weight percentages of Pani in V_2O_5 matrix's at three different temperatures (50, 100 and 150 ^{0}C). It is observed that the conductivity for 10 wt%, 20 wt% and 40 wt% of V_2O_5 /PANI Composites decreases. In 30 wt% and 50 wt% of V_2O_5 in polyaniline conductivity increases which is due to the variation in distribution of PANI which may be supporting for more number of charge carriers to hopp between favorable localized sites causing increase in conductivity. The decrease in conductivity may be attributed due to the trapping of charge carriers. This can be well supported by VRH model.

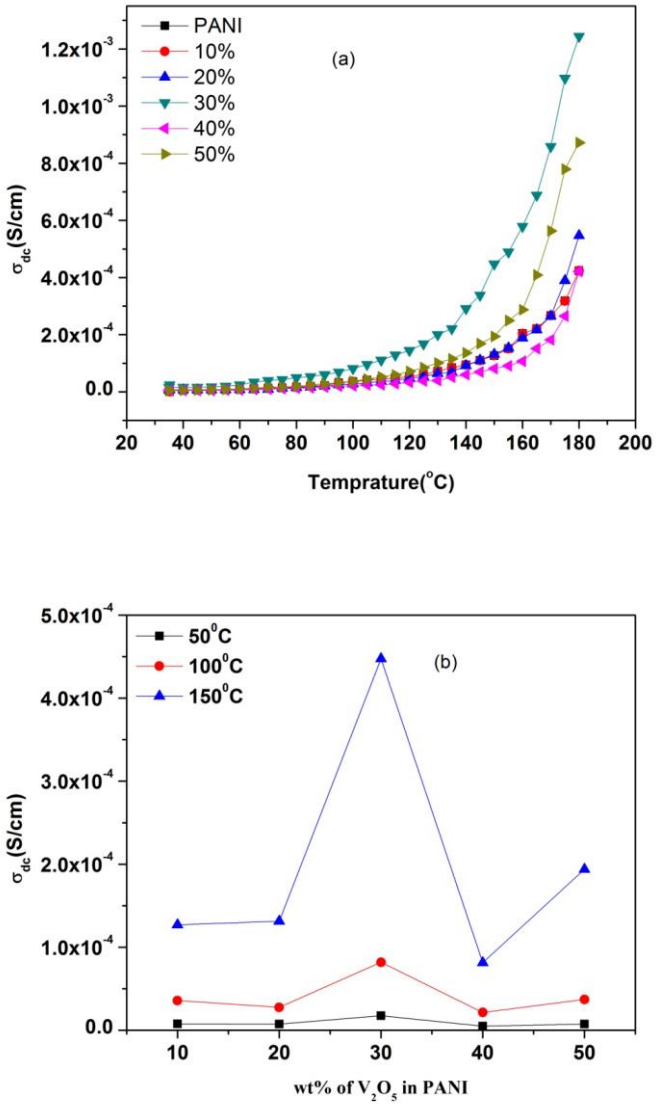

Figure 5: (a) Variation of σdc as a function of temperature of PANI/ V_2O_5composites (b) Variation of σdc as a function of weight % of V_2O_5 in PANI at three different temperatures.

3.5 Sensor studies

The sensitivity for LPG with time for PANI and PANI/ V_2O_5 composites is shown in figure 6(a) & figure 6(b). Maximum sensitivity is observed in composite of 30 wt% of V_2O_5 in polyaniline.
The change in resistance with gas concentration for pure PANI and PANI / V_2O_5 composites is shown in figure 6(c) & figure 6(d). With increase in gas concentration the resistance of all the samples was found to increase. Maximum change in resistance was observed in composites of 50 wt% of V_2O_5 in Pani. The change in resistance in the samples could be due to two reasons.

1) Swelling of polymer on exposure to gas
2) Reaction between gas molecules and metal oxide V_2O_5

On exposure to LPG, polymer matrix swells due to the absorption of gas which leads to disruption of conducting paths through the composites. This results in increased resistance of composites. After removal of gas, the polymer returns to original size, restoring the conducting path.

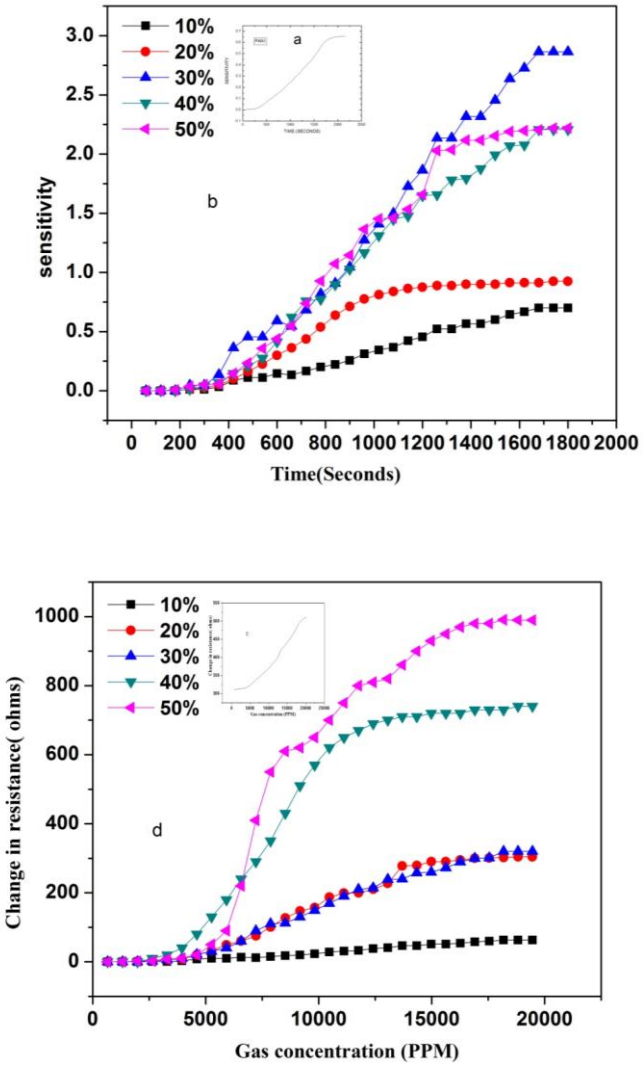

Figure 6 (a) Sensitivity of PANI, (b) Sensitivity of PANI / V_2O_5 composites for LPG, (c) Variation of resistance in PANI, (d) Variation of resistance in PANI / V_2O_5 composites with gas concentration

V_2O_5 is intrinsically n-type semi-conducting material. The adsorption of atmospheric oxygen atoms on the semiconductor surface and at grain boundaries of polycrystalline semiconductor traps the free electrons and creates an electrical double layer which acts as scattering centers for conducting electrons. When adsorbed oxygen atoms react with the reducible gases an increase in conductivity is observed because of generation of free electrons and decrease in scattering centers. The overall conduction in a sensor element is determined by the surface reactions, the resulting charge transfer processes with the underlying semi conducting material and the transport mechanism through the sensing material and morphology of sensing layer [23].

Both the mechanisms mentioned above are responsible for variation of resistance in PANI/ V_2O_5 composites. On exposure to LPG, N-type V_2O_5/P-type polyaniline composite forms hetero-junctions which are discreetly distributed in bulk part of sample. The composite samples are in emeraldine base form of polyaniline. On absorption of gas by emeraldine base polyaniline, it could react with imine nitrogen atom to generate protonated form of polyaniline resulting in significant changes in electrical conductivity [24-25]. There is strong interaction between polyaniline macromolecule and V_2O_5. Presence of V_2O_5 helps to activate this reaction. When absorbed gas react with imine nitrogen atom in polyaniline, the coordination bond between V_2O_5 and nitrogen atom in polyaniline may break down and more charges are available for conduction resulting in significant change in electrical conductivity.

IV. CONCLUSIONS

Polyaniline/Vanadium Pentoxide(Pani/V_2O_5) composites of various weight percentages were synthesized by in situ polymerization method. Studies on Fourier infrared spectroscopy (FTIR) confirms the formation of polyaniline/V_2O_5 composites, X-ray diffraction studies shows the retention of V_2O_5in polymer matrix and The SEM image reveals the presence of V_2O_5 particles which are uniformly distributed throughout the composite sample. Temperature dependent conductivity shows thermally activated behaviour of PANI/V_2O_5 composites. Maximum conductivity was observed in the composite of 30 wt% of V_2O_5 in polyaniline. On exposure to LPG, change in resistance was observed in all the composites with increase in gas concentration. Maximum sensitivity for gas sensing was observed in the composite of 30 wt% V_2O_5 in polyaniline.

V. FUTURE WORK

The author would like to investigate in the future work the various transport properties and also studies on sensing properties such as response time, recovery time etc.

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