Characterization Study of SnO₂ Blended with Polyaniline

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Abstract

Tin dioxide (SnO₂) has been widely used in many applications such as catalysts agent, hazardous gas sensors, heat reflecting mirrors, varistors, transparent conducting electrodes for solar cells and optoelectronic devices. SnO2 nanoparticles are synthesized and then characterized using FTIR. Polyaniline is synthesized from precursors as aniline and then characterized using FTIR. FTIR spectra of SnO₂ blended with polyaniline are discussed in this paper.

Key Words: Tin dioxide, Polyaniline, FTIR

Research in nanoscale materials is motivated by the possibility of processing and designing nanostructured materials with unique properties thereby offering new and important technological applications. Due to their finite small size and high surface-to-volume ratio, nanostructured materials often exhibit novel, and sometimes unusual properties. Tin dioxide (SnO2) is an important oxide semiconductor material, which has been widely used in many applications such as catalysts agent, hazardous gas sensors, heat reflecting mirrors, varistors, transparent conducting electrodes for solar cells, and optoelectronic devices. Tin dioxide-based gas sensor devices are very important in developing n-type semiconductor sensors, which can be utilized to detect various inflammable and harmful gases such as hydrogen (H2), carbon monoxide (CO), ethanol and methanol. Semiconductor sensors exhibit changes in electrical resistivity in the presence of small concentrations of certain gases. Sensors consisting of fine particles of metal oxides usually exhibit high sensitivity compared to their counterparts made of larger particles. The most important factor affecting the sensitivity of gas sensors is the actual crystallite size of the sensing materials. Tin dioxide powders used for gas sensors have been synthesized via various routes such as direct strike precipitation, two-step solid state synthesis etc. Among the above methods, conventionally accepted method is the synthesis of tin dioxide particles from precursor hydroxides precipitated by the direct addition of NH4OH to SnCl4 aqueous solutions. Although, this technique yields a large amount of powder instantaneously and at a reasonable cost, it is not easy to obtain powders with high surface area because of the irregular particle morphology, large particle size distribution and the high degree of agglomeration. In this article, we report the synthesis of tin oxide nanoparticles by a novel sol-gel combustion technique from tin

nitrate. The starting materials are very cheap and the synthesis procedure is simple, and the obtained SnO2 particles are in the nanometer scale and mono-dispersed with very high surface area. This should be a better choice instead of the sol-gel method from tin alkoxides. The particles have been characterized by means of XRD, SEM EDAX and TEM.

SnO2 is a very promising candidate for a number of applications like gas sensing, optoelectronic device applications and dilute magnetic semiconductors when doped with transition metals. CuO is also used for sensing applications, photovoltaic, antibacterial and biomedical applications. So, my future plan is divided in the following parts as given below:

(1) Development of gas sensor using pure and doped SnO2 nanoparticles. Variation of electrical conductivity on adsorption of gas molecules at materials surface is the basic phenomenon in gas sensors. On the basis of present research it has been observed that Ni doping reduces the band gap of SnO_2 and CuO along with the decrease in particle size. Particle size was reduced up to 2 nm in Ni doped SnO2 which is less than that of the critical size for sensing (6 nm) .So, Ni doped SnO2 nanoparticles can be used to achieve better sensing response. Mixed SnO2-CuO system has also been reported to 258 show good sensing properties by G. Zhang et al. X.H. Vu et al. have also shown the better LPG sensing response of CuO doped SnO_2.

(2) Transition metal doped SnO2 and CuO have been reported to behave as good dilute magnetic semiconductors. Fe and Co-doped SnO2 exhibit room temperature ferromagnetism while Fe doped CuO has also been found to exhibit room temperature ferromagnetism .Some authors have recently reported the development of magnetic behavior in Ni- doped SnO2 nanoparticles, which can be useful to use these materials in spintronic applications .So, I shall explore the magnetic behavior of both Ni and Mn doped SnO2 and CuO nanoparticles using VSM and SQUID.

(3) Tin oxide and Copper oxide, both are used in optoelectronic device application like photovoltaic. For photovoltaic application band gap should be in the range 1.2-1.8 eV. The results of our research indicate that Ni doping in SnO2 and CuO reduces the band gap, and in case of Ni doped CuO band gap reaches 1.7 eV, which makes this material important for solar cell applications. For this purpose, it is necessary to make thin films of these materials. So, we are planning to make the thin films of Ni doped SnO2 and CuO and analyze them.

(4) CuO nanoparticles have also been reported to show good antimicrobial properties against various microorganisms .We have started to explore the antimicrobial behavior of CuO nanoparticles. The antimicrobial activity of CuO nanoparticles was examined against four microorganisms which show that CuO nanoparticles exhibit good antimicrobial activity. The next step is to check their toxic behavior which is very important for biomedical applications.

Experimental:

In the process of preparation of SnO_2 nanoparticle, some instruments are needed some chemical and powder are needed.

Synthesis of nanoparticles of SnO_2. First wash the beaker magnetic needle, bath, measuring cylinder by the pure acetone by precaution of experiment. In the experimental set up heater, magnetic needle, water bath kept in particular space. First we kept 100 ml distilled water added $SnCl_2$. $2H_2O$ is more accurately is 2.2563 gm by waging machine. After this add this $SnCl_2$. $2H_2O$ in the water and stirred normally with glass rod. The solution of this is mixture is white. This solution heated with the help of heater at 60^oC to 63^oC. This solution stirring with the help of magnetic needle to maintain P_H of solution must be needed 1molar NaOH solution. The precipitation of 1molar NaOH solution 40gm solid NaOH that mix with 1000ml distilled water then the 1molar solution is create. To maintain P_H of solution add drop by drop NaOH solution. The P_H maintain of this solution is 8.9 is always constant. Addition of 1 molar NaOH solution processes up to changing the colour of solution. A solution after 15 to 20 min. white colour to grey colour the pure grey colour is obtained. After this stop the addition of NaOH solution and constant heating 60^oC and stirring 450 to 550 revolutions per minute. After 2 hours precipate layer is formed the water bath then stop heating and stirring. After this, solution is filtered by filter paper. Grey layer precipate from by filter paper. This filter paper kept in the behind of the IR lamp (Infera red). After some time powder is from this crush in the crusher. Then this powder is kept in small plastic bag. This process continues to form 6gm powder. After this, result is found by FTIR, UV, XRD. Following Procedure is used for the preparation of nanoparticle of SnO_2:

100 ml. D.W. kept in water bath at 60^oC
↓
$SnO_2 . 2H_2O$ (2.2563)gm
↓
Drop by drop addition of 1M NaOH to maintain PH 8.9
↓
Change in colour white to grey
↓
Stirring at 400^oC for 2hours
r.p.m. = 530 – 60
temp = 60 – 63^oC

Pellet preparation is one of the most in expensive techniques to make a sample. The powder is compacted in the form of pellet using press technique. The appropriate binder may be mixed with the materials. This is the simple technique; however large amount of material is required.

The samples in the form of pellets could be prepared using low cost equipment's like hydraulic press and a die set. These equipments are low cost equipment's compared with the equipment's required in vacuum evaporation technique. The sample preparation parameters can be easily optimized and the method is simple. Hence

the MPc samples were prepared in the form of pellets to study of the gas sensing performance.

Samples in the form of pellets were prepared for studying the gas response. The synthesized MPc material was compacted into the pellet form using press technique. Three parameters viz. Pressure, time of application of pressure and weight of MPc

material were optimized. Electronics contacts were made by coating silver paste on either surface of pellet. Synthesized powder material was compacted in the form of pellets of 13mm diameter under optimized pressure and giving optimized time to cure figure shows the photographs of the pellet making hydraulic press machine and die set.

Figure 1: Pellet making press machine and die set
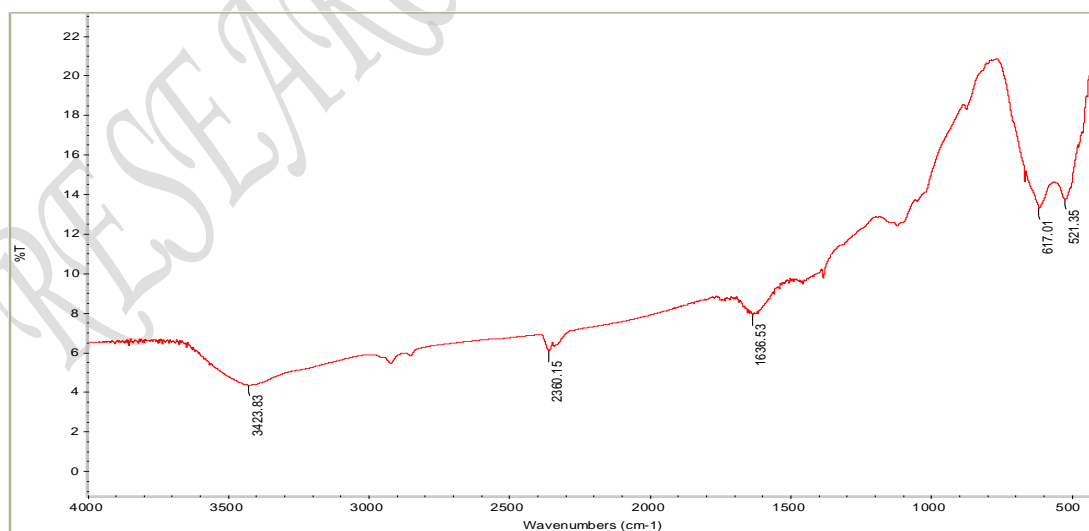
Results and Discussion:

Figure 2: FTIR Spectra of SnO$_2$

Table 1: Table showing peak values for SnO₂

Sr. No.	Peak No.	Frequency Range	Wavelength Range	Intensity	Types and Groups	Bond	Type of Vibration
1	1	433.60	ca.20.00	Strong , absorption	Iodides	C-I	Stretching
2	2	521.35	16.60-20.00	Strong , absorption	Bromides	C-Br	Stretching
3	3	617.01	ca.15.90	Strong , absorption	Alkynes	C-H	Deformation
4	4	1636.53	6.10-6.52	Strong , absorption	Ketones (-CO-CH2-CO)	C=O	Stretching
5	5	2360.15	4.00-4.30	Strong absorption	Charged amines(C=NH+)	S-H	Stretching
6	6	3423.83	2.91-2.94	Strong , absorption	Hadides	C-Cl	Stretching

Wave number of the peak is 433.60cm^{-1} and their wave length range is ca.20.00 and its intensity is Strong absorption. Its type of group is Iodides and bond is C-I and type of vibration is stretching. Wave number of the peak is 521.35cm^{-1} and their wave length range is ca.16.60-20.00 and its intensity is Strong absorption. Its type of group is Bromides and bond is C-Br and type of vibration is Stretching. Wave number of the peak three is 617.01cm^{-1} and their wave length range is ca.15.90 and its intensity is Strong absorption. Types and group is Alkyenes and bond is C-H.is type of vibration is Deformation. Wave number of the peak four is 1636.53cm^{-1} and their wave length range is 6.10-6.52 and its intensity is Variable intensity strong absorption Doublet. Types and group is Ketones(-CO-CH2-CO) and bond is C=O and type of vibration is Stretching. Wave number of the peak five is 2360.15cm^{-1} and their wave length range 4.00-4.30 and its intensity Variable intensity Strong absorption.. Types and group is Charged amines(C=NH+) and bond S-H .its type of vibration is Stretching. Wave number of the peak six is 3423.83 cm^{-1} and their wave length range is 2.90-2.94 and its intensity is Medium absorption. Types and group is Pyrroles and bond is N-H And type of vibration is stretching.
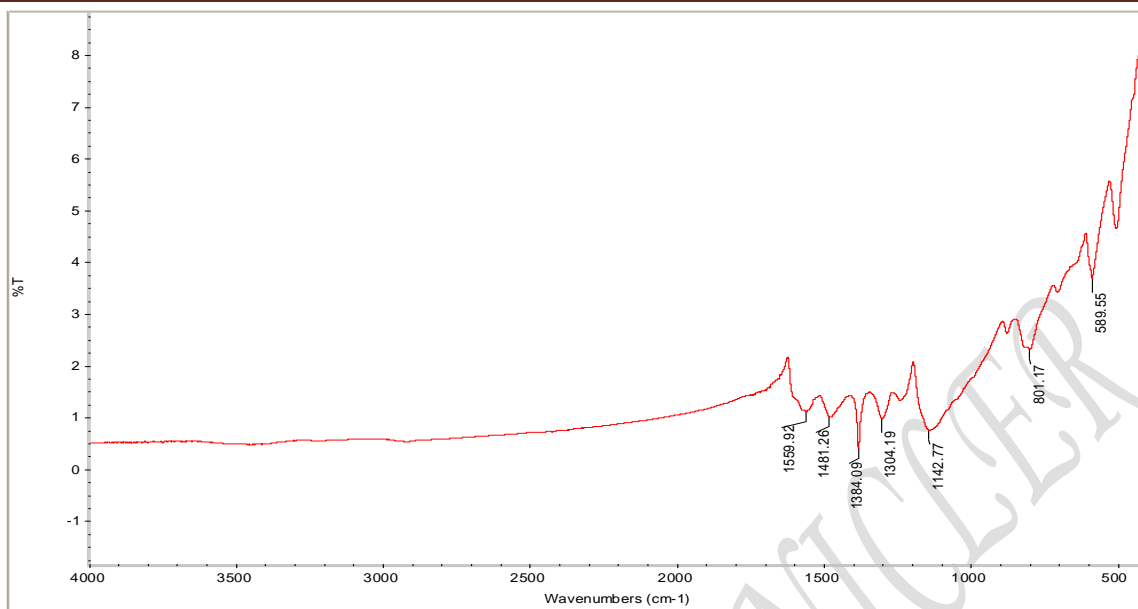
Figure 3: FTIR Spectra of Polyaniline

Table 1: Table showing peak values for PANI

Sr. No.	Peak No.	Frequency Range	Wavelength Range	Intensity	Type of Group	Bond
1	1	589.55	16.60-20.00	Strong Absorption	Bromide	C-Br Stretching
2	2	801.17	12.50-16.67	Strong Absorption	Halide	C-Cl Stretching
3	3	1142.77	8.62-8.77	Strong Absorption	Sulfones	S=O
4	4	1304.29	7.70-8.00	Strong Absorption	Nitrites	$O-NO_2$ Vibrating
5	5	1384.09	7.20-7.35	Medium, Doublet	Alkane, Geminal dimethyle, isopropyl CH_3	CH_3 Deformation
6	6	1481.26	6.74-6.92	Medium Absorption	Alkanes (-CH_2-)	CH Deformation
7	7	1559.92	6.39-6.47	Strong absorption	Primary and Secondary Nitro Compound	NO_2 stretching

Wave number of the peak is 589.55cm^{-1} and their wave length range is 16.60-20.00 and its intensity is Strong absorption. Its type of group is Bromides and bond is C-Br and type of vibration is stretching.   Wave number of the peak is 801.17cm^{-1} and their wave length range is 12.50-16.67 and its intensity is Strong absorption. Its type of group is Halide and bond is C-Cl and type of vibration is Stretching. Wave number of the peak three is 1142.77cm^{-1} and their wave length range is 8.62-8.77 and its intensity is Strong absorption . Types and group is Sulfonic acid and bond is S=O. Its type of vibration is Stretching. Wave number of the peak four is 1304.29cm-1 and their wave length range is 7.70-8.00 and its intensity is Variable Strong absorption. Types and

group is nitriles and bond is O-NO2 vibration. Wave number of the peak five is 1384.09cm-1 and their wave length range 7.20-7.35   and   its   intensity   Medium absorption.. Types and group is alkanes, Geminal, dimethyl , isopropyl CH3. and bond CH3 .its type of vibration is Deformation. Wave number of the peak six is 1481.26cm-1 and their wave length range is 6.74-6.92 and its intensity is medium absorption. Types and group is Alkanes and bond is (-CH$_2$-). And   type of vibration is Deformation. Wave number of the peak seven is 1559.92cm^{-1} and their wave length range is 6.39-6.47 and its intensity is strong absorption . Types and group is Primart And Secondary Nitro Compound   and bond is NO$_2$ .and type of vibration is Stretching.

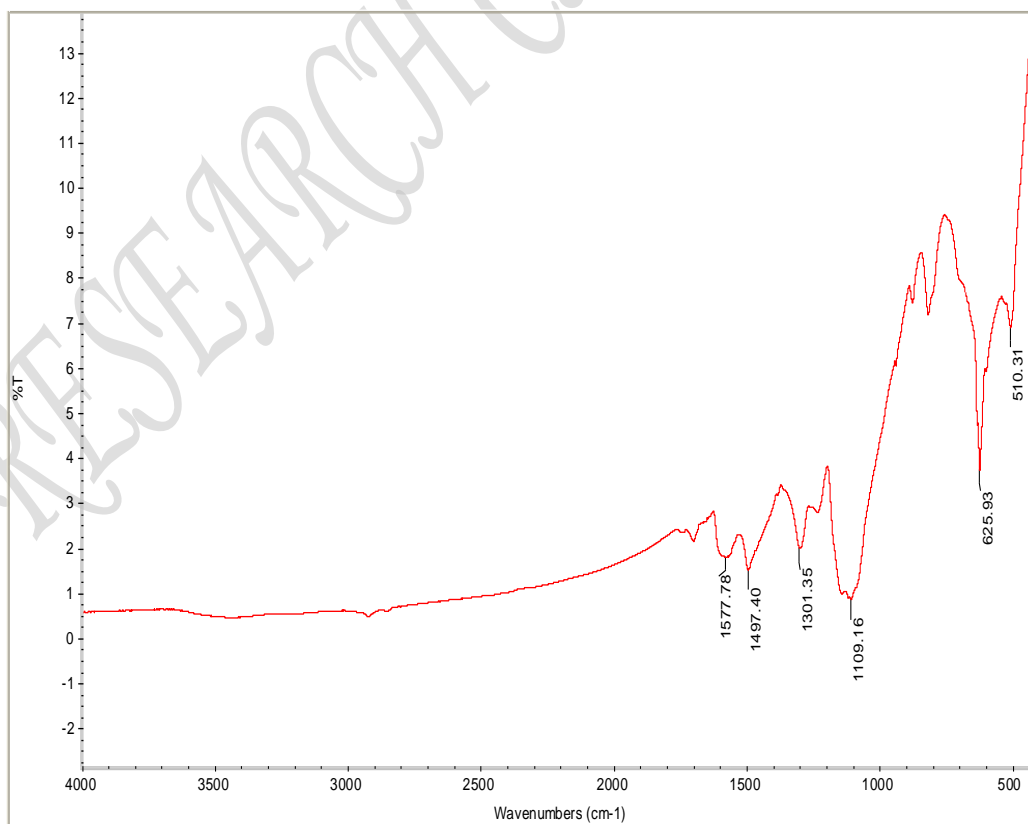FTIR spectra of SnO$_2$ blended with water are shown in figure 4.

Figure 4: FTIR Spectra of Polyaniline

Sr. No.	Peak No.	Frequency Range	Wavelength Range	Intensity	Types and Groups	Bond	Type of Vibration
1	1	510.31	16.60-20.00	Strong , absorption	Bromides	C-Br	Stretching
2	2	625.93	ca15.90	Strong , absorption	Alkynes	C-H	Deformation
3	3	1109.16	8.93-9.09	Strong , absorption	Secondary Alcohols	C-OH	Stretching
4	4	1301.35	7.64-7.72	Medium absorption	Alkene (R1CH=CHR2) trans	C-H	Deformation
5	5	1497.40	6.67-6.94	Strong absorption	Nitroamines (RNN=O)	N=O	Stretching
6	6	1577.78	6.33-6.58	Medium absorption	Pyrimidines And Pyrines Comdination c=c And c=n	C=C+ C=N	Stretching

Wave number of the peak is 510.31cm^{-1} and their wave length range is ca.16.60-20.00 and its intensity is Strong absorption. Wave number of the peak is 625.93cm^{-1} and their wave length range is ca.15.90 and its intensity is Strong absorption. Its type of group is Alkyens and bond is C-H and type of vibration is Deformation. Wave number of the peak three is 1109.16cm^{-1} and their wave length range is 8.93-9.09 and its intensity is Strong absorption. Types and group is Secondary Alcohol and bond is C-OH.is type of vibration is Stretching . Wave number of the peak four is 1301.35cm^{-1} and their wave length range is 7.64-7.72 and its intensity is Variable intensity medium absorption. Types and group is Alkene and bond is C-H and type of vibration is stretching. Wave number of the peak five is 1497.40cm^{-1} and their wave length range 6.67-6.94 and its intensity Variable intensity Strong absorption. Types and group is Nitroamines. and bond N=O .its type of vibration is stretching. Wave number of the peak six is 1577.78cm^{-1} and their wave length range is 6.33-6.58 and its intensity is Medium absorption. Types and group is Pyrimidines and Pyrines Combination and bond is C=C+C=N And type of vibration is stretching.

Conclusion:

Wave number of the peak is 433.60cm^{-1} and their wave length range is ca.20.00 and its intensity is Strong absorption. its type of group is Iodides and bond is C-I and type of vibration is stretching. Wave number of the peak is 521.35cm^{-1} and their wave length range is ca.16.60-20.00 and its intensity is Strong absorption. its type of group is Bromides and bond is C-Br and type of vibration is Stretching.

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Wave number of the peak is 589.55cm^{-1} and their wave length range is 16.60-20.00 and its intensity is Strong absorption. its type of group is Bromides and bond is C-Br and type of vibration is stretching. Wave number of the peak is 801.17cm^{-1} and their wave length range is 12.50-16.67 and its intensity is Strong absorption. its type of group is Halide and bond is C-Cl and type of vibration is Stretching. Wave number of the peak three is 1142.77cm^{-1} and their wave length range is 8.62-8.77 and its intensity is Strong absorption . Types and group is Sulfonic acid and bond is S=O. Its type of vibration is Stretching .Wave number of the peak four is 1304.29cm-1 and their wave length range is 7.70-8.00 and its intensity is Variable Strong absorption. Types and

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