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Synthesis and Structural Characterization of Polyaniline–Tin-Oxide Nanocomposite for Device Application

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ABSTRACT

In this research, polyaniline (PANI) and tin (iv) oxide (SnO_2) nanoparticles and polyaniline/SnO_2 nanocomposites were successfully synthesized by chemically oxidative polymerization of aniline in the presence of hydrochloric acid using ammonium persulfate as an oxidizing agent. The purpose of this study is to synthesize and investigate polyaniline/tin (iv) oxide nanocomposite for optoelectronics application. The synthesized nanocomposites were characterized to ascertain possible applications of the nanocomposites. The x- ray diffraction (XRD) analysis was carried out using x-ray diffraction machine. The results of the structural analysis showed that the formed polyaniline is a polymer, while SnO_2 showed tetragonal rutile phase. The average crystallite size of 8.40 nm was obtained for SnO_2. PANI/SnO_2 nanocomposites have structural pattern of tetragonal rutile phase and an average crystallite size range between 5.73 nm and 9.17nmwere obtained. The obtained results in this study revealed good results which are comparable with results obtained by other researchers in literature. These results obtained suggest the possibility of using the material in optoelectronics devices like LEDs, solar cells, transducers, and photodetectors.

1.0 Introduction

Nanotechnology is defined as the study and use of structures between 1 nanometer and 100 nanometers in size. Presently, various chemical and physical methods have been used for the synthesis of metallic oxides nanoparticles. Many years ago success has been made in every field of nanotechnology such as nanoparticles, nanolayers, and optoelectronics and in nanostructured materials [1]. Nanoparticles are particles which are 1 to 100 nanometers in size. In nanotechnology, a particle is defined as a small object that behaves as a whole unit with respect to its transport and properties. Ultrafine particles are the same as nanoparticles and are between 1 and 100 nanometers in size also [2]. Nanocomposites are nanomaterials that combine one or more separate components in order to obtain the best properties of each component (composite). In nanocomposite, nanoparticles (clay, metal, carbon nanotubes) act as fillers in a matrix, usually polymer matrix. The result of the addition of nanoparticles leads to a great improvement in properties such as mechanical strength, toughness, electrical and thermal conductivity of the formed composite [3]. Nanoparticles have high surface to volume ratio which greatly changes their properties when compared with their bulk sized equivalents. Some nanocomposite materials have been shown to be many times tougher than the bulk component materials [4].

In the last decade, nanocomposite and nanostructured materials, especially polymer-metal oxide nanocomposite and metal oxide nanoparticles have become an important class of new materials with several properties that make them very attractive for commercial development. In fact, they have been increasingly used for manufacturing diverse industrial items such as cosmetics or clothes and for infinite applications in electronics, aerospace and computer industry due to their novel special properties. Our manufacturing industries are highly in need of nanocomposite and nanostructured materials, especially polymer-metal oxide nanocomposite and metal oxide nanoparticles. From literature, many works have been done on synthesis and characterization of polyaniline-metal oxides nanocomposites but no work has been done on the synthesis and characterization of Polyaniline/SnO_2

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*Corresponding author: aa.okafor@unizik.edu.ng nanocomposite. The aim of this present study is to synthesize and investigate the structural properties of PANI–tin-oxide (SnO2) nanocomposites for optoelectronis applications. To achieve this, the following objectives must be accomplished; Synthesize of SnO_2 nanoparticles, fabrication of polyaniline/SnO_2 nanocomposites, and characterization of the synthesized nanoparticles. The significant of this study is to reveal the effects of mass variation of SnO_2 on the polyaniline/SnO_2 nanocomposite and to ascertain the possible industrial applications of the fabricated nanocomposite.

2.0 Materials and Methods

2.1. Materials

Materials used for the fabrication of polyaniline / tin oxide nanocomposite including reagents and laboratory apparatus are; Aniline ($C_6H_5NH_2$), Tin (II) chloride dehydrate ($SnCl_2.2H_2O$), Hydrochloric acid (HCl), Ammonium octaoxosulphate (VII) ($(NH_4)_2S_2O_8$), Ammonium hydroxide Solution (NH_4OH), Absolute ethanol (C_2H_6O), Whatman filter paper (110 mm), Magnetic stirrer with heater, Digital weighing balance and 250 ml beakers.

2.2 Preparation of reagents used.

All the salt form reagents used for this work were prepared in solution form. Molar solutions of the desired salts were prepared by dissolving the mass (g) of the equivalent salt in 1000 cm^3 of distilled water at room temperature. The equation (1) was used to determine the exact amount of the solid reagent in gram that can dissolve in 1000 ml (cm^3) of distilled water for solid salts while equation 2 was used to determine the exact amount of liquid reagent in dm^3 that was used in the experiment.

$$ReactingMass = \frac{Molarity(mol) \times Molecularmass(g) \times Volume(ml)}{1000(ml)} \qquad 1$$

$$Molar\ concentration\ of\ \ liquid\ reagent = \frac{density \times specficgravity}{molarmass}$$

$$V_1 = \frac{V_0 C_0}{C_1} \qquad 2$$

where C_0 is the desired molar concentration of the liquid reagent (mol/dm^3), C_1 is the molar concentration of the liquid reagent (mol/dm^3), V_0 is the volume of deionized water used to dissolved the liquid reagent (dm^3) and V_1 is the volume of the reagent to be measured (dm^3).

2.3. Preparation of aniline ($C_6H_5NH_2$) solution

In this experiment, 100ml of aniline solution was dissolved in 500ml of 0.1M of HCl to form 0.5M of aniline – hydrochloride solution. This was calculated using equation (2).

V_1 =volume of diluted HCl used $= 500\ ml$

C_1 =concentration of HCl used $= 0.1\ m$

V_2 =Volume of aniline used $= 100\ ml$

C_2 = Concentration of aniline used $= \frac{V_1 C_1}{V_2} = \frac{0.1 \times 500}{100} = 0.5\ m$

2.4 Preparation of tin (II) chloride dehydrate ($SnCl_2.2H_2O$) solution

$SnCl_2$ has a molar mass of 189.60 g / mole. Tin (II) chloride serves as precursor for Sn^{2+} ion. In this experiment, 0.2 molar solution of $SnCl_2$ was prepared by dissolving 18.96 g in 500Ml of deionized water. The solution was magnetically stirred for 10 minutes and at a temperature of 40℃ to have a homogenous solution of the reagent.

$$Reacting\ mass = \frac{0.2 \times 189.60 \times 500}{1000} = 18.96\ g$$

2.5 Preparation of diluted hydrogen chloride (HCl) solution

It has a molar mass of 36.46 g/mol. HCl is an important chemical reagent and industrial chemical. 0.1M of HCl was prepared by adding 8.18ml of 37.5% concentrated HCl to 1000ml of distilled water. The 8.17ml of HCl was obtained by using the percentage concentration of the HCl (37.5%), molar mass of HCl (36.46 g/mol) and specific density of (1.19 g/ml).

$$1\ mole\ of\ HCl = \frac{36.46}{1.19} \times \frac{100}{37.5} = 81.7\ ml$$

$$\therefore 0.1\ mole = 0.1 \times 81.7\ mL = 8.17\ ml$$

2.6 Preparation of ammonium octaoxosulphate (VII) solution

It has a molar mass of 228.10 g/mol. In this work, 0.80M of the salt was prepared by dissolving 18.25g in 100ml of hydrochloric acid. The solution was magnetically stirred for 10 minutes and at a temperature of 40 ℃ to have a homogenous solution of the reagent.

$$Reacting\ mass = \frac{0.80\ \times 228.10 \times 100}{1000} = 18.25\ g$$

2.7 Preparation of ammonium hydroxide (NH_4OH) solution

It was used in the experiment to regulate the pH of the baths. 36.5% concentration of ammonium hydroxide solution was used in this work. Absolute ethanol was also used without further dissolution

2.8. Preparation of tin oxide nanoparticles

SnO_2 nano - powders were prepared by means of mixing 150 ml of 0.2M stannous chloride dihydrate ($SnCl_2.2H_2O$) with aqueous ammonia solution drop wise under vigorous stirring. The rate of drop – wise addition of ammonium solution was maintained at 1ml per minute for 30 minutes which amount to 30ml of aqueous ammonium solution. The final solution was allowed for another 30 minutes under continuous stirring at 60℃. The resulting precipitate was filtered with filter paper, washed with distilled water for several times and then with ethanol to remove unreacted salts and excess ammonia. The nanoparticles obtained were dried at 80ºC for 12 hours in order to remove water molecules. Finally, tin oxide nano-powder was calcined at 400ºC for 2hrs. The standard sample obtained was labeledA_2. The overall reaction can be written as

$$2Sn^{2+} + 8OH^{-} \rightarrow 2SnO_2 + 4H_2O \qquad 3.$$

2.9. Preparation of polyaniline nanoparticles

The polymerization of aniline to form the desired polyaniline was based on mixing aqueous solutions of aniline hydrochloride (100ml of aniline solution was dissolved in 500ml of 0.1M of HCl) and ammonium persulfate – hydrochloride (18.25 g of ammonium persulphate mixed with 100 ml of 1.0M of HCl) at room temperature). Firstly, 100 ml of aniline hydrochloride solution was stirred for 30 minutes in an ice bath followed by a drop – wise addition of 20ml of ammonium persulphate – hydrochloride at drop rate of 1 drop per minute. The solution was subjected to further magnetic stirring in the ice bath for another 30 minutes. The final solution was removed from the ice bath and allowed to age for 24 hours to polymerize completely. The separation of PANI hydrochloride precipitate was done by filtration using whatman filter paper. The precipitate was washed several times with 1.0ml HCl, similarly with acetone and finally with distilled water. Polyaniline hydrochloride powder was dried in air and then in vacuum at 60℃ for 24hours. Polyaniline prepared under these reaction and processing conditions are further referred to as “standard” samples.

2.10 Preparation of polyaniline – tin oxide (PANI/SnO_2) nanocomposites

Synthesis of the Polyaniline– tin oxide nanocomposites were carried out by in-situ polymerization method. 100ml of aniline – hydrochloride was stirred with a magnetic stirrer for 30 minutes in an ice bath. 0.5 g of tin oxide nanoparticles fabricated were dispersed in the above solution with vigorous stirring for 30 minutes in order to keep the tin oxide homogeneously suspended in the solution. To this solution, 20ml of ammonium persulphate hydrochloride was added drop – wise with drop rate of 1 drop per minute. The final solution was removed from the ice bath and allowed to age for 24 hours to completely polymerize. The precipitate was filtered then washed with 1.0ml hydrochloric acid, acetone, water for many times and finally dried in a hot air oven at 60℃ for 24 hours. In this way, Polyaniline – tin oxide nanocomposites containing various weights of 1.0 g, 1.5 g, and 2.0 g SnO_2 in PANI were synthesized using the same procedures

3.0 Results and Discussion

The structural properties of the polyaniline and tin-oxide nanoparticles as well as of their nanocomposites, produced, were investigated using x-ray diffraction machine.

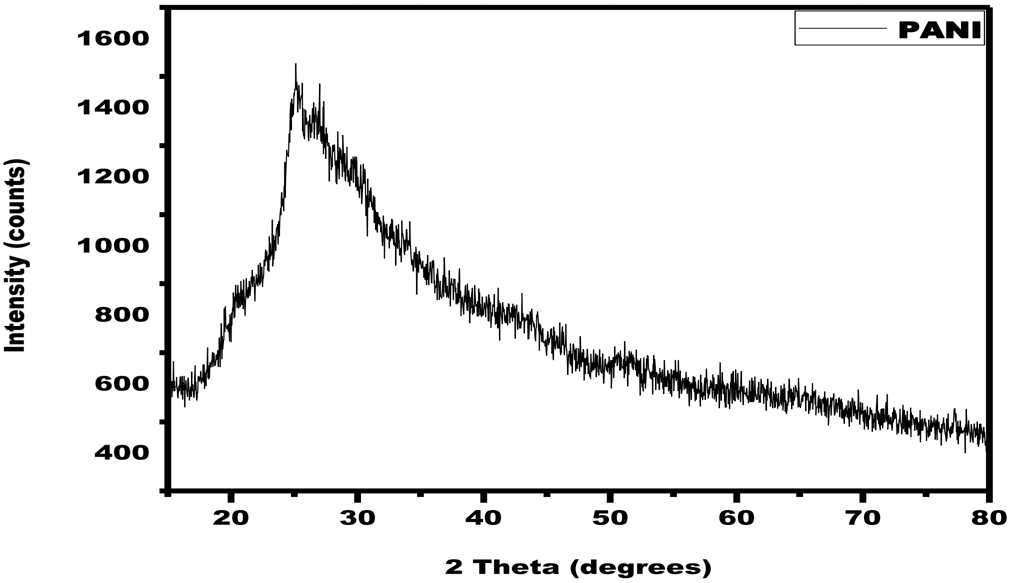

Figure 1: XRD pattern of Polyaniline (PANI) nanoparticles

Figure 1 shows the x – ray diffraction pattern of polyaniline nanoparticles. The result shows that there is a dome shape in the crystal pattern of the fabricated polyaniline suggesting the polymeric nature of the nanoparticles. Peak angle of 25.36° was observed. The PANI structural pattern obtained in this work is similar to broad amorphous scattering with 2θ angle of 25°

obtained by [5]. Also, the result corresponds to preferential orientation peak of 2θ angle of $25.19°$ for polyaniline obtained by [6].

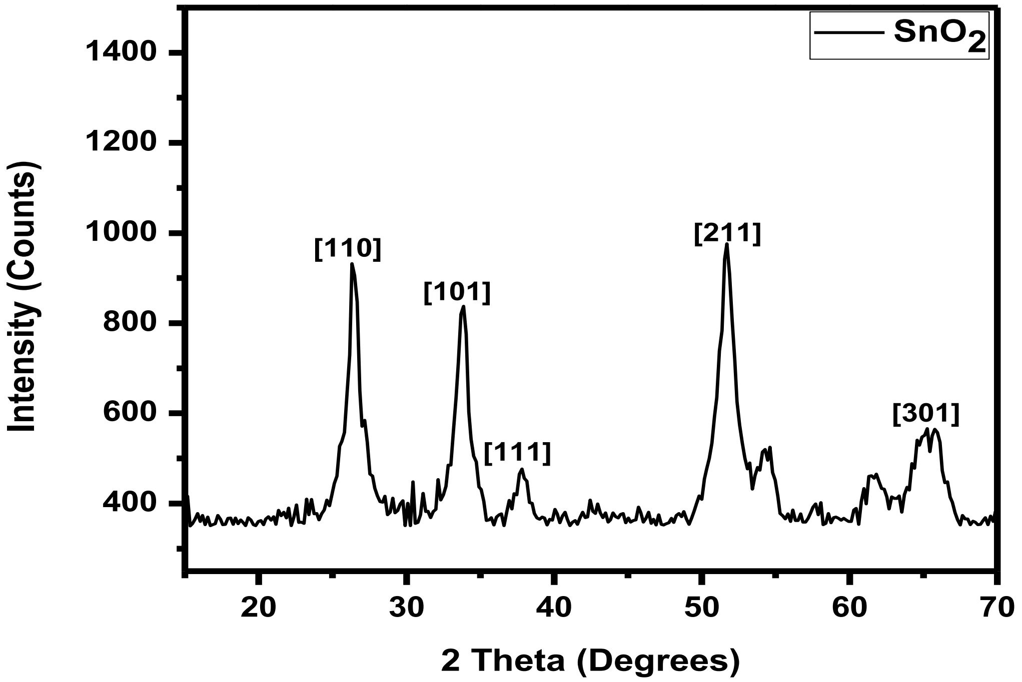

Figure 2: XRD pattern of Tin oxide (SnO_2)nanoparticles

Figure 2 shows the x – ray diffraction pattern of tin oxide nanoparticles. The result shows that the peaks correspond to standard x – ray diffraction pattern of tetragonal rutile phase of tin oxide crystal structure with mineral name cassiterite and JCPDS file number (00 – 014 – 1445). Two theta angles of $26.60°$, $34.79°$, $38.82°$, $52.70°$ and $66.29°$ were obtained which correspond to miller indices of [110], [101], [111], [211] and [301]. The structural parameters such as full width half maximum (FWHM), miller indices (hkl), standard and observed 2 theta angles, standard and observed d – spacings, and crystallite sizes of the iron oxide nanoparticles were presented in Table 1. From Table 1, the average crystallite size of $8.40\,nm$ was obtained for SnO_2. Corresponding structural phase has been obtained by [7]; [8]; [9],[10] and [11] obtained average crystallite sizes of $8\,nm - 10\,nm$ and $8\,nm - 43\,nm$ respectively which are in line with the average crystallite size of $8.40\,nm$ obtained in this work

Table 1: Structural parameters of SnO_2 nanoparticles

2θ (°)		**D – spacing (Å)**		**[hkl]**	**FWHM (°)**	**Crystallite Size (nm)**
Observed	**Standard**	**Observed**	**Standard**			
26.44	26.62	3.34	3.34	110	1.079	7.90
33.79	33.89	2.64	2.64	101	0.976	8.88
37.82	37.95	2.38	2.37	111	0.690	12.71
51.70	51.86	1.77	1.76	211	1.330	6.93
65.29	65.89	1.43	1.42	301	1.770	5.57

It could also be seen from Table 1 above that the results we obtained for tin oxide nanoparticles compared favorably with the standard results

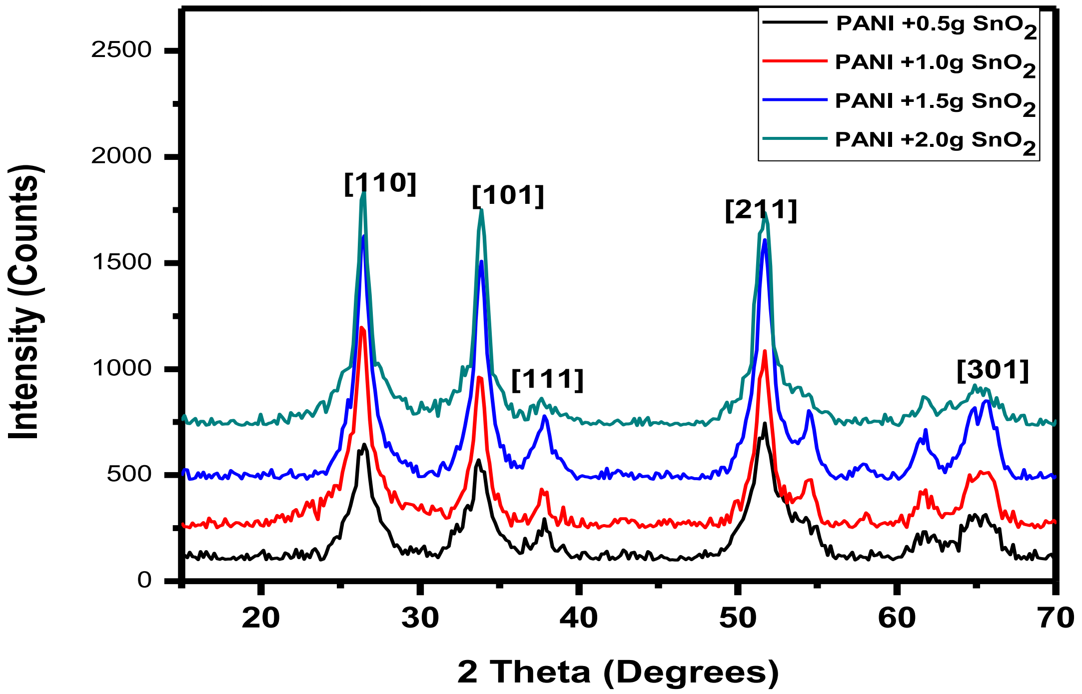

Figure 3: XRD pattern of polyaniline / tin oxide (PANI / SnO_2) nanocomposites

Figure 3 is the x – ray diffraction pattern of PANI/SnO_2 nanocomposites fabricated with different weights of SnO_2 nanoparticles ranging from 0.5 g to 2.0 g. The x – ray diffraction pattern reveals that the PANI/SnO_2 nanocomposites formed have the same structural phase with the pure SnO_2 shown in Figure 2.

Table 2: Structural parameters of PANI / SnO_2 nanocomposites

PANI/SnO$_2$ (g)	**2θ (°)**		**D – spacing (Å)**		**[hkl]**	**FWHM (°)**	**Crystallite Size (nm)**
	Observed	**Standard**	**Observed**	**Standard**			
0.5	26.43	26.62	3.37	3.34	110	1.578	5.40
	33.73	33.89	2.65	2.64	101	1.623	5.34
	37.76	37.95	2.38	2.37	111	0.933	9.40
	51.82	51.86	1.76	1.76	211	2.337	3.95
1.0	65.09 26.33	65.89 26.62	1.43 3.34	1.42 3.34	301 110	2.151 1.441	4.57 5.91
	33.75	33.89	2.65	2.64	101	0.990	8.75
	37.75	37.95	2.38	2.37	111	0.521	16.82
	51.63	51.86	1.77	1.76	211	1.120	8.23
1.5	65.27 26.42	65.89 26.62	1.43 3.37	1.42 3.34	301 110	1.609 1.155	6.12 7.38
	33.83	33.89	2.65	2.64	101	1.057	8.20
	37.75	37.95	2.38	2.37	111	1.111	7.89
	51.68	51.86	1.77	1.76	211	1.249	7.38
2.0	65.28 26.42	65.89 26.62	1.43 3.37	1.42 3.34	301 110	1.641 1.071	6.00 7.95
	33.86	33.89	2.64	2.64	101	0.921	9.41
	37.71	37.95	2.38	2.37	111	0.970	9.04
	51.62	51.86	1.77	1.76	211	1.132	8.14
	65.06	65.89	1.43	1.42	301	1.933	5.09

It could be seen from figure 3 that the intensity of the diffraction peaks increases as the mass of SnO_2 increases in the nanocomposites. The structural parameters such as full width half maximum (FWHM), miller indices (hkl), standard and observed 2 theta angles, standard and observed d – spacings, and crystallite sizes of the PANI/tin oxide nanocomposites were presented in Table 2. Table 2 revealed that the average crystallite sizes of $5.73\,nm$, $9.17nm$, $7.37nm$ and $7.93nm$ were obtained for different SnO_2 compositions of polyaniline matrix. These results show that the polyaniline – tin oxide particles are in nanoscale. The results correspond to structural phases obtained by [12]; [13];[14].and [15] for polyaniline – tin oxide nanocomposites. It could also be seen from table 2 that the results obtained for PANI/SnO_2 nanocomposites were in line with the standard results. In comparison therefore, the results of the XRD of the fabricated polyaniline nanoparticles shows that there is a dome shape in the crystal pattern of the polyaniline nanoparticles suggesting the polymeric nature of the nanoparticles, for tin oxide nanoparticles the result shows that it has tetragonal rutile phase. The x – ray diffraction pattern reveals that the PANI/SnO_2 nanocomposites formed have structural phase same with the pure SnO_2 and average crystallite sizes of $5.73\,nm$, $9.17nm$, $7.37nm$ and $7.93nm$ were obtained for different SnO_2 compositions of polyaniline matrix.

Conclusion

Polyaniline nanoparticles, tin oxide nanoparticles, and polyaniline – tin oxide nanocomposites were successfully synthesized using chemical processes. The samples formed were subjected to structural characterization. The results of the structural analysis showed that the formed polyaniline is a polymer with peak 2θ at $25.36°$. That of tin oxide shows that the peaks correspond to standard x – ray diffraction pattern of tetragonal rutile phase of tin oxide crystal structure with mineral name cassiterite and JCPDS file number (00 – 014 – 1445). Two theta angles of$26.44°$, $33.79°$, $37.82°$, $51.70°$ and $65.29°$ were obtained which correspond to miller indices of 110, 101, 111, 211 and 301. The average crystallite size of $8.40\,nm$ was obtained for SnO_2. The x – ray diffraction pattern reveals that the PANI/SnO_2 nanocomposites formed have structural phase same with the pure SnO_2 and average crystallite sizes of $5.73\,nm$, $9.17nm$, $7.37nm$ and $7.93nm$ were obtained for different SnO_2 compositions of polyaniline matrix. These results obtained suggest the possibility of using the material in optoelectronics devices like LEDs, solar cells, transducers, and photo detectors.

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