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Synthesis and Structural Characterisation of Polyaniline (PANI)- Iron oxide (Fe2O3), nanocomposites for Optoelectronics application

[1]Okafor A.A., [2]Okafor L.U. and [3]Ifowodo H.F.
[1]Department of Mechanical Engineering, Nnamdi Azikiwe University, Awka, Anambra State, Nigeria
[2]Department of Physics and industrial physics, Nnamdi Azikiwe University, Awka Anambra State, Nigeria
[3]Department of Industrial/Production Engineering, Southern Delta University, Ozoro
Corresponding Author's e - mail: aa.okafor@unizik.edu.ng

Abstract
In this investigation, polyaniline (PANI), Iron oxide (Fe_2O_3) nanoparticles and polyaniline- Fe_2O_3 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/ iron oxide nanocomposite for optoelectronics application. The synthesized nanocomposite was characterized to ascertain possible applications of the nanocomposite. 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 with peak 2θ at 25.36°. X – ray diffraction pattern of iron oxide nanoparticles shows that the fabricated iron oxide nanoparticles are of rhombohedral phase of iron oxide with mineral name Hematite and corresponding to JCPDS file number (00 – 033 – 0664). Two theta angles of 33.15°, 35.70°, 54.10° and 64.00° were obtained corresponding to miller indices of [104], [110], [116] and [300] respectively. The average crystallite size of 20.68 nm was obtained for Fe_2O_3. PANI/Fe_2O_3 nanocomposites formed have structural phase of iron oxide hydroxide with mineral name Goethite belonging to Orthorhombic crystal structure with space group Pbnm and JCPDS file number (00 – 029 – 0713). Average crystallite sizes of 8.03 nm, 7.68 nm, 12.85 nm and 13.91 nm were obtained for different Fe_2O_3 compositions of polyaniline matrix.These results obtained suggest the possibility of using the material in optoelectronics devices like LEDs, solar cells, transducers, and photodetectors.
Keywords: Polyaniline–iron-oxide nanocomposites, nanoparticles, Structural properties, Diffraction Machine

1. Introduction

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 (Reddy et al., 2005). 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 (Rodriguez et al., 2002). 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. Several studies (Ayad et al., 2017; Mu et al., 2015; Parthibanab et al., 2020; Peymanfar et al., 2019; Predoi, 2007; Sriramprabha et al., 2020) reported the synthesis of polyaniline with iron oxide nanoparticles for the generation of nanocomposites with improved

magnetic and conducting properties. From literature, no work has been done on synthesis and characterization of of polyaniline (PANI)- Iron oxide (Fe_2O_3), nanocomposites for optoelectronics application. This present work concentrate on Synthesis and structural characterisation of polyaniline (PANI)- Iron oxide (Fe_2O_3), nanocomposites for optoelectronics application so as to fill up the literature gap.

2.0 Materials and methods

2.1 Materials

Materials used for the fabrication of polyaniline / iron oxides nanocomposite including reagents and laboratory apparatus are; (i) Iron (III) chloride hexahydrate ($FeCl_3 . 6H_2O$), (ii) Iron (II) tetraoxosulphate (VI) heptahydrate ($FeSO_4. 7H_2O$), (iii) Aniline ($C_6H_5NH_2$), (iv) Tin (II) chloride dihydrate ($SnCl_2 .2H_2O$), (v) Hydrochloric acid (HCl), (vi) Ammonium octaoxosulphate (VII) ($(NH_4)_2S_2O_8$), (vii) Ammonium hydroxide Solution (NH_4OH), (viii) Absolute ethanol (C_2H_6O), (ix) Whatman filter paper (110 mm), (x) Magnetic stirrer with heater, (xi) Digital weighing balance and (xii) 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 m^3 that was used in the experiment.

$$Reacting\ Mass = \frac{Molarity(mol) \times Molecular\ mass(g) \times Volume(ml)}{1000(ml)} \quad 1$$

$$Molar\ concentration\ of\ \ liquid\ reagent = \frac{density \times specfic\ gravity}{molar\ mass} \quad 2$$

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

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

2.2.1 Preparation of Iron (lll) chloride ($FeCl_3. 6H_2O$) Solution

The hexahydrate salt has a molar mass of 270.30 g. In this experiment, 0.4M of the salt was prepared by dissolving 54.06 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. The final solution served as precursor for the co – precipitation of Fe_2O_3 nanoparticles.

$$Reacting\ mass = \frac{0.4\ \times 270.30\ \times 500}{1000} = 54.06\ g$$

2.2.2. Preparation of Iron (ll) tetraoxosulphate (VI) solution

In this experiment, 0.2M of the salt was prepared by dissolving 27.80 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. The final solution served as precursor for the co – precipitation of Fe_2O_3 nanoparticles.

$$Reacting\ mass = \frac{0.2 \times 278.02 \times 500}{1000} = 27.80\ g$$

2.2.3. 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 (3.2).

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 $= \frac{V_1 C_1}{V_2} = \frac{0.1 \times 500}{100} = 0.5\ m$

2.2.4 Preparation of tin (II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) 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.

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

2.2.5 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).

$$1\ mole\ of\ HCl = \frac{36.46}{1.19} \times \frac{100}{37.5} = 81.7\ ml$$
$$\therefore 0.1\ mole = 0.1 \times 81.7\ mL = 8.17\ ml$$

2.2.6 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.

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

2.2.7 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.

2.2.8. Preparation of absolute ethanol (C_2H_6O) solution

In this experiment, absolute ethanol was used without further dissolution.

2.2.9. Preparation of nanoparticles and nanocomposites

Procedures for the synthesis of polyaniline nanoparticles, iron oxides nanoparticles and polyaniline/iron oxide nancomposites are described in the next subsections.

2.3. Preparation of iron oxide nanoparticles

Co – precipitation of iron oxide nanoparticles was obtained by mixture of iron (III) chloride and iron (II) tetraoxosulphate (VI) in the 2:1 molar ratio. The 100ml of ferric chloride and 50ml of ferrous sulfate solutions were transferred to a 250ml beaker, the mixture was then heated up to 60℃ for 10 minutes. After heating, the solution was precipitated by drop – wise addition of ammonia solution with the rate of 1 mLper minute for 1 hour. The final solution was allowed to stay under continuous stirring on the magnetic stirrer at 60℃ for another 1hour. At end of the 2 hours, the beaker containing the solution was allowed to cool. Brown colored particles of iron oxides were precipitated. The brown colour suggests that iron (III) oxide (Fe_2O_3) was formed. These particles were then separated from the solution by using a strong magnet and then were washed many times with distilled water. The powder was then dried in hot air oven at 100℃ for 24 hours. The standard sample obtained was labelled A_1. The overall reaction can be written as

$$Fe^{2+} + Fe^{3+} + 12OH^- \rightarrow 2Fe_2O_3 + 6H_2O \qquad 3$$

2.3.1. 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.

2.3.2. Preparation of polyaniline – iron oxide (PANI/Fe_2O_3) nanocomposites

Synthesis of the Polyaniline–iron 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 iron oxide nanoparticles fabricated were dispersed in the above solution with vigorous stirring for 30 minutes in order to keep the iron 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 1.0m hydrochloric acid, acetone, water for many times and finally dried in a hot air oven at 60℃ for 24 hours. In this way, Polyaniline – iron oxide nanocomposites containing various masses of 1.0g, 1.5 g, and 2.0 g Fe_2O_3 in PANI were synthesized using the same procedures.

Table 1: Optimization of polyaniline / iron oxide nanoparticles

Bath name	Aniline hydrochloride (ml)	Ammonium hydrochloride (ml)	Fe_2O_3 (g)	Aging (hrs)
A_1	100.00	20.00	–	24.00
A_4	100.00	20.00	0.50	24.00
A_5	100.00	20.00	1.00	24.00
A_6	100.00	20.00	1.50	24.00
A_7	100.00	20.00	2.00	24.00

2.4. Characterization of synthesized nanoparticles

The synthesized iron oxide nanoparticles and polyaniline/iron oxide nanocomposites were characterized to determine the structural properties using X-ray diffraction machine

3.0 Results and Discussion

3.1. Structural analysis

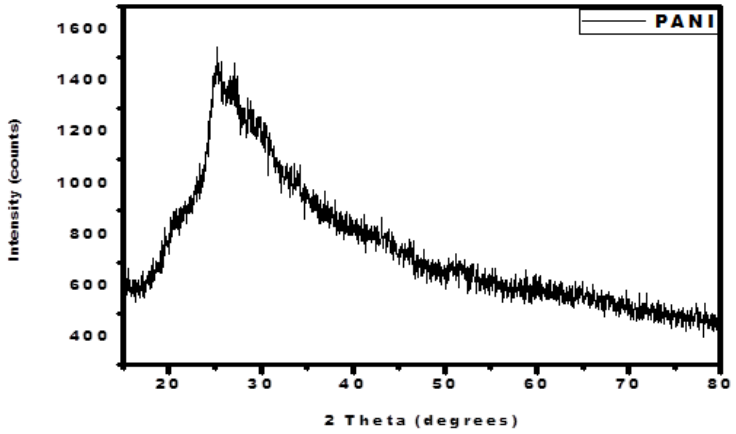

Figure 1: XRD pattern of Polyaniline (PANI) nanoparticles

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 (Roy et al., 2018). Also, the result corresponds to preferential orientation peak of 2θ angle of $25.19°$ for polyaniline obtained by (Vadiraj and Belagal, 2015).

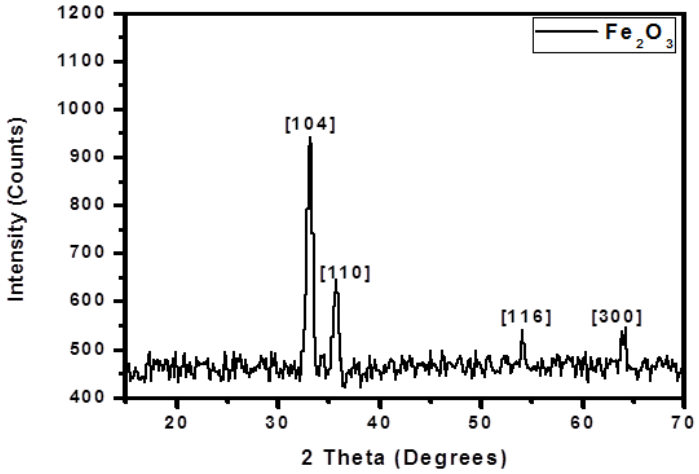

Figure 2: XRD pattern of Iron oxide (Fe_2O_3) nanoparticle

Figure 2 shows the x – ray diffraction pattern of iron oxide nanoparticles. The result shows that the fabricated iron oxide nanoparticles are of rhombohedral phase of iron oxide with mineral name Hematite and corresponding to JCPDS file number (00 – 033 – 0664) and space group R-3c. Two theta angles of $33.15°$, $35.70°$, $54.10°$ and $64.00°$ were obtained corresponding to miller indices of [104], [110], [116] and [300] respectively. Preferential orientation was observed at [104] plane. 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 are presented in Table 2. From Table 2, the average crystallite size of $20.68\ nm$ was obtained for Fe_2O_3. (Balaraju et al., 2017; Yufanyi et al., 2015; Farahmandjou and Soflaee 2015) obtained the same structural phase for their synthesized iron oxide nanoparticles with crystallite sizes of 39 nm, 45 nm and 30 nm respectively. Also, (Kostyukova, and Chung, 2016) obtained crystallite size between 21 nm and 30 nm for Fe_2O_3 phase which is close to the result of this work.

Table 2: Structural parameters of Fe_2O_3 nanoparticles

2θ (º)		D – spacing (Å)		[hkl]	FWHM (º)	Crystallite Size (nm)
Observed	**Standard**	**Observed**	**Standard**			
33.15	33.15	2.70	2.70	104	0.476	18.18
35.70	35.61	2.51	2.52	110	0.445	19.58
54.10	54.09	1.69	1.69	116	0.411	22.66
64.00	63.99	1.45	1.45	300	0.439	22.28

It could also be seen from Table 2 above that the results obtained for iron oxide nanoparticles compared favourably with the standard results. Figure 3 shows the x – ray diffraction pattern of PANI/Fe_2O_3 nanocomposites fabricated with different compositions of Fe_2O_3 nanoparticles ranging from 0.5 g to 2.0 g. It could be seen from the graph that intensity of PANI/Fe_2O_3 nanocomposites increases as the mass of Fe_2O_3 nanoparticles increases in the nanocomposite. The patterns show that the PANI/Fe_2O_3 nanocomposites have structural phase of iron oxide hydroxide with mineral name Goethite belonging to orthorhombic crystal structure with space group Pb nm and JCPDS file number (00 – 029 – 0713). 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/iron oxide nanocomposites are presented in Table 3. From the Table, the average crystallite sizes of $8.03\ nm$, $7.68\ nm$, $12.85\ nm$ and $13.91\ nm$ were obtained for different Fe_2O_3 compositions of polyaniline matrix. These results show that the polyaniline – iron oxide particles are in nanoscale. (De – Freitas et al., 2015) obtained similar Goethite structural phase for their synthesized iron nanoparticles.

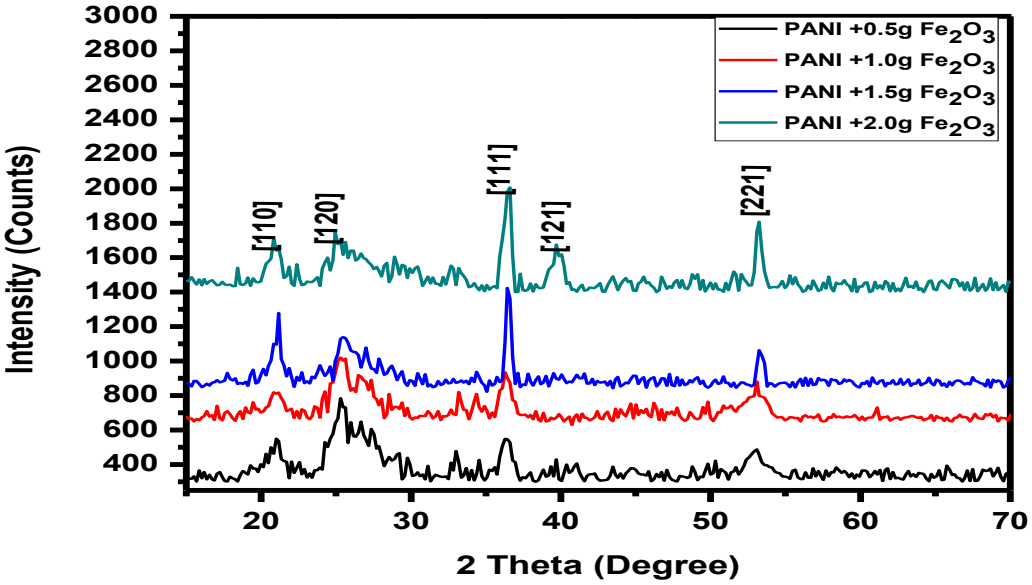

Figure 3: XRD pattern of polyaniline / iron oxide (PANI / Fe_2O_3) nanocomposite

Table 3: Structural Parameters of PANI / Fe_2O_3 nanocomposites

PANI/Fe_2O_3 (g)	2θ (°)		D – spacing (Å)		[hkl]	FWHM (°)	Crystallite Size (nm)
	Observed	Standard	Observed	Standard			
0.5	20.92	21.22	4.24	4.18	110	1.086	7.77
	26.00	26.32	3.42	3.39	120	2.846	2.99
	36.37	36.06	2.47	2.49	111	0.655	13.33
1.0	52.96	53.24	1.73	2.72	221	1.153	8.04
	20.99	21.22	4.23	4.18	110	0.987	8.55
	25.86	26.32	3.44	3.39	120	2.545	3.34
	36.35	36.06	2.47	2.49	111	0.709	12.31
1.5	52.95	53.24	1.73	2.72	221	1.421	6.52
	21.06	21.22	4.22	4.18	110	0.646	13.06
	26.03	26.32	3.42	3.39	120	2.547	3.34
	36.50	36.06	2.46	2.49	111	0.331	26.39
2.0	53.09	53.24	1.72	2.72	221	1.077	8.61
	20.89	21.22	4.25	4.18	110	0.866	9.74
	26.26	26.32	3.39	3.39	120	2.123	4.01
	36.44	36.06	2.46	2.49	111	0.490	17.82
	39.77	39.98	2.26	2.25	121	0.692	12.75
	53.21	53.24	1.72	2.72	221	0.368	25.21

It could also be seen from Table 3 that the results we obtained for PANI/Fe_2O_3 nanocomposites meet up with the standard results

4.0. Conclusion

Polyaniline nanoparticles, iron oxide nanoparticles, and polyaniline-iron 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 $25.36°$. X – ray diffraction pattern of iron oxide nanoparticles shows that the fabricated iron oxide nanoparticles are of rhombohedral phase of iron oxide with mineral name Hematite and corresponding to JCPDS file number (00 – 033 – 0664). Two theta angles of $33.15°$, $35.70°$, $54.10°$ and $64.00°$ were obtained corresponding to miller indices of [104], [110],

[116] and [300] respectively. The average crystallite size of $20.68\,nm$ was obtained for Fe_2O_3. PANI/Fe_2O_3 nanocomposites formed have structural phase of iron oxide hydroxide with mineral name Goethite belonging to Orthorhombic crystal structure with space group Pbnm and JCPDS file number (00 – 029 – 0713). Average crystallite sizes of $8.03\,nm$, $7.68\,nm$, $12.85\,nm$ and $13.91\,nm$ were obtained for different Fe_2O_3 compositions of polyaniline matrix. Since these crystallite sizes suggests: good potential for light interaction (absorption/emission), possible tunable band gap (important for devices) and balance between crystallinity and nanoscale effects, it is favorable for optoelectronics devices like LEDs, solar cells, transducers, and photodetectors.

5.0 Recommendation

I recommend more research for industrial application of polyaniline nanocomposites

Nomenclature

PANI = Polyaniline

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