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# Magnetic behaviour of Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites synthesised by two different pathways

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 $ABSTRACT-Polyaniline/BaFe_{12}O_{19} \ \ composites \ \ synthesised$ bv two different polymerization pathways: chemical polymerization and solid-state polymerization, were studied. The influence was explored by the type of polymerization synthesis on the microstructure and on the magnetic properties of the composites. Structural, morphological and magnetic properties of the nanocomposites were performed by XRD, FTIR, SEM and VSM measurements, respectively.XRD analysis revealed the inorganic phase as bariumhexaferrite and FTIR analysis indicated an interaction betweenhexaferrite particles with Polyaniline regardless the synthesis method. The powder morphologies obtained from SEM revealed very similar particle for the while extent sizes tworoutes the ofagglomerationwashigherforthe chemical polymerization method.Polyaniline/BaFe<sub>12</sub>O<sub>19</sub>composites exhibited я ferromagnetic behaviour. The powders obtained by solid-state polymerization presnted better magnetic characteristics (for 70%Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composite, saturation magnetization 25.37 emu/g ) than those obtained by chemical polymerization.

KEY WORDS: Polyaniline, barium hexaferrites, chemical polymerization, solid-state polymerization, magnetic measurements.

#### 1. INTRODUCTION

Hybrid materials composed of both organic and inorganic units, offer wide opportunities for new materials with increased functionalities relative to that achievable with organic and inorganic materials alone [1-4].

Recently, a great attention has been focused on the synthesis of composites consisting of conducting polymer, specifically Polyaniline (Pani)with various inorganic nanomaterials such as oxide, sulphide, nitride, metal and clay [5-7].One of the most prevalent types of these composites is composed ofPani and ferromagnetic barium hexaferrite (BaFe<sub>12</sub>O<sub>19</sub>) which have potential application in antennas [8], radar absorbers [9], tunable filters [10], electromagnetic shielding materials for low and high frequency [11]....

Several studies have investigated the structural and magnetic properties of the obtained composite. However, few papers focused on the way of elaborating these composites and its influence on these properties. Barium hexaferritewith a general chemical formula BaO.6Fe<sub>2</sub>O<sub>3</sub>, have been coated with Pani by chemical oxidative polymerization [12-14].The magnetic properties of the obtained composite were influenced by the coating of hexaferrite by the polymer which is explained

as a result of electronic interactionbetween Paniat the interface offerromagnetic particles.

The present work was aimed at comparing the structural and magnetic properties of Polyaniline/BaFe<sub>12</sub>O<sub>19</sub>composites synthesized following chemical and solid state polymerization processes at room temperature.

### 2. EXPERIMENTAL

### 2.1. Materials

Aniline (analytical grade, Merck) was distilled twice under reduced pressure and stored at the refrigerator. Iron (III) nitrate nonahydrate (Fe ( $NO_3$ )<sub>3</sub>.9H<sub>2</sub>O), barium nitrate (Ba ( $NO_3$ )<sub>2</sub>, sodium hydroxide NaOH, aniline chloride (AnCl), ammonium persulfate (APS)and hydrochloric acid (HCl) were all of analytical purity and used without further purification.

### 2.2. Synthesis of BaFe<sub>12</sub>O<sub>19</sub> particules

The BaFe<sub>12</sub>O<sub>19</sub>powder was prepared by the hydrothermal process using an autoclave introducedinto a shaked furnace. A mixture of Fe(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>Oand Ba(NO<sub>3</sub>)<sub>2</sub> were added to 2.5 M NaOH solution according to desired stoichiometries. The mixture was thenpoured into the autoclave and heated at 220°C during 8 h. The resulting powderswere repeatedly washed with distilled water and dried at 100°C. Then, the obtained powder was calcined at 900°C for 2 h.

### 2.3. Synthesis of Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites by solid-state polymerization (SP)

Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites were obtained by solidstate polymerization (Fig.1a) as follow: the synthesis was carried out in an agate mortar by grinding an appropriate amount of An-Cl and BaFe<sub>12</sub>O<sub>19</sub> for 10 min. Then APS was added to the mixture and ground for 20 min until the colour turned dark green. Different mass ratios of BaFe<sub>12</sub>O<sub>19</sub> were used: 10, 30, 50, 70% with respect to aniline monomer and the samples were marked as 10SP, 30SP, 50SP and 70SP, respectively. The polymerization lasted for 24h at room temperature. The dark green product was filtered, washed by HCl until the filtrate became colourless then dried in vacuum at 60°C for 24h.Throughout the experiment the molar ration of aniline to APS was maintained at 1.25.

## 2.4. Synthesis of Polyaniline/BaFe<sub>12</sub>O<sub>19</sub>composites by chemical polymerization (CP)

Likewise, Polyaniline/BaFe<sub>12</sub>O<sub>19</sub>composites were prepared by chemical polymerization of aniline in the presence of BaFe<sub>12</sub>O<sub>19</sub>particles using APS as oxidant (Fig. 1b). In a typical procedure, a certain amount of BaFe<sub>12</sub>O<sub>19</sub>particles was added to 0.2M aqueous solution of hydrochloric acid containing aniline monomer sonicated in an ultrasonic bath for 30 min. Then, 5.71 g APS dissolved in 0.2M HCl solution was then added to the above mixture under agitation. The polymerization was carried out with the same barium hexaferrite and reagents ratio as abovefor 24h at room temperature. The products were isolated by filtration, thoroughly washed with HCl for removal of inorganic residues then dried in vacuum at 60°C for 24h.The composites were named as 10CP, 30CP, 50CP and 70CP, respectively



Fig.1Flow chart of (a) solid-state (b) chemical polymerization

### 2.5. Characterization

X-ray diffraction (XRD) patterns were recorded on aX'Pert PRO, PANalytical diffractometerusing Co  $K_{\alpha}$  radiation( $\lambda$ = 2.29Å)in the range of 2  $\theta$  = 5–80° with 0.002° increment. Scanningelectron microscopy (SEM) images were taken on a VP-SEM S-3400Nscanning electron microscope.Fourier transform infrared (FTIR) spectra were collected on a Perkin Elmer FTIR spectrometer using pressed KBr discs. The FTIR spectra were recordedwith a resolution of 1 cm<sup>-1</sup> over the range of 4000-400cm<sup>-1</sup>. Magneticproperties of the samples were measured at room temperature on vibrating sample magnetometer VSM Lake Shore 7400 model.

#### 3. RESULTS AND DISCUSSION

#### 3.1. Structure, composition and morphology

Fig.2 shows the XRD spectra of the Pani/BaFe<sub>12</sub>O<sub>19</sub> and BaFe<sub>12</sub>O<sub>19</sub> powders.All the peaks of the prepared BaFe<sub>12</sub>O<sub>19</sub>marked in Fig. 1 can be indexed to (101), (102), (110), (107), (114), (203), (205), (206), (217), (2011), and (220) planes of hexagonal BaFe<sub>12</sub>O<sub>19</sub> (space group P63/mmc, JCPDS cards 74-1121). The as-prepared  $BaFe_{12}O_{19}$  have the hexagonal crystal magnetoplumbite structure. As seen in Fig.1a and b, diffraction peaks positionand intensity of the Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites synthesised by chemical and solid-statepolymerization are similar to those of pure barium hexaferrite[15].Thecrystal phase of magnetic particles is well maintained after the synthesis process, based on the fact thatthere is no change in peak position between BaFe<sub>12</sub>O<sub>19</sub> and the composite. In addition, there is a wide peak in the range of  $2\theta = 20-30^{\circ}$  which is attributed to the characteristic diffraction peak of Pani and proves the existence of the polymer [16].However, the intensity of Pani peak decreased with increasing the amount of hexaferrite in the polymer.





Fig.2 X-Ray diffraction patterns of Polyaniline/BaFe $_{12}O_{19}$  composites obtained by: a) chemical polymerization and b) solid –state polymerization

Further investigations of the crystalline transitions and interactions between Polyaniline and hexaferrite in the composites were performed using FTIR spectroscopy. The aim was to obtain an improved understanding of the formation mechanisms and existing interactions of the composites. FTIRspectra were recorded from 4000 – 400 cm<sup>-1</sup> for the different samples, however figures show only expanded view in wavenumber region 400- 2000 cm<sup>-1</sup>, where major changes are observed as shown in Fig.3. The absorption at 578 cm<sup>-1</sup> and 430 cm<sup>-1</sup> for barium hexaferrite(Fig. 3a), correspond to the intrinsic vibrations of tetrahedral and octahedral sites for the prepared BaFe<sub>12</sub>O<sub>19</sub>, respectively [17].





Fig.3FTIR Spectra of a)  $BaFe_{12}O_{19}$ ; b) Polyaniline/BaFe<sub>12</sub> $O_{19}$  composites obtained by chemical polymerization and c) Polyaniline/BaFe<sub>12</sub> $O_{19}$  composites obtained by solid –state polymerization

In Fig.3 b and c, Polyaniline/BaFe<sub>12</sub>O<sub>19</sub>composites spectra obtained by the two synthesesexhibitthe characteristic peaks of Paniemeraldine salt [18, 19]. The band 1564-1573 cm<sup>-1</sup> can be associated mainly to C=N and C=C stretching of the quinoiddiimineunit, while the band near~ 1484-1489 cm<sup>-1</sup> is assigned to the C-C aromatic ring stretching of the benzenoiddiamine unit. TheC-N stretching of the secondary aromatic amine and aromatic C-H out of plane banding vibration, respectively, are attributed to the bands at ~ 1297 -1303 cm<sup>-1</sup>and~806-817cm<sup>-1</sup>. However, it can be observed that the two bands at 572 and 429 cm<sup>-1</sup>corresponding to the Fe-O band of BaFe12O19red shift and decrease clearly withthe decrease in weight percentage of hexaferrite in the composite. This shift reveals a strong interaction between Polyaniline on the surface of barium hexaferrite[20,21].

Scanning electron microscopy was used to examine the morphology of the prepared barium hexaferrite and the different Polyaniline/BaFe12O19 composites obtained by chemical and solid-state polymerization (Fig.4, 5 and 6).As seen in Fig.4, BaFe12O19 particles exhibit a platelet like shapeand that the average size is estimated at 300 nm. Agglomerationappears due to interactions between magnetic particles. SEM micrographs of the composites consist of irregularly shaped and aggregated particles due to the coating of BaFe<sub>12</sub>O<sub>19</sub> by Polyaniline in the two syntheses. Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites obtained by chemical synthesis present much more morphological heterogeneity in terms of particle size than those obtained by solid-state polymerization (Fig.5and 6). In fact, Wang et al.[22] have shown that in acidic solution, there is an electrostatic force between hydrogen of amine groups of Polyaniline backbone and oxygen atom of the metal oxide in ferrite, and supported by the chloride ions which are attracted by the positive charges of ferrite and could be adsorbed by Polyaniline cations. Whereas, the solid-state polymerization present a smaller particle size composites compared to chemical polymerization due to the less contact between the monomer and oxidant according to Bahadra et al. [23]. In addition for the both syntheses, with the increase cotenant of Polyaniline in the composites, a bigger segregation of the magnetic grain is produced which can be explained by a secondary nucleation of Pani growing on the existing Pani [24].



Fig.4SEM microphotographs of prepared BaFe12O19



Fig.5SEM microphotographs of Polyaniline/BaFe $_{12}O_{19}$  composites synthesised by chemical polymerization: a) 10 CP; b) 30 CP, c) 50 CP and d) 70 CP.



Fig.6SEM microphotographs ofPolyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites synthesised by solid-state polymerization:a) 10SP; b) 30SP, c) 50SP and d)70SP.

### 3.2. Magnetic properties

The hysteresis loops of barium hexaferrite and the composites at room temperature at a maximum applied field at 2T are shown in Fig.7and 8.The magnetization under applied magnetic field for the as-prepared Polyaniline/barium hexaferrite composites exhibits clear hysteresisbehaviour.



Fig.7Hysteresis loop BaFe12O19.



Fig.8Hysteresis loops of Polyaniline/BaFe $_{12}O_{19}$  composites obtained by: a) chemical polymerization and b) solid –state polymerization.

The magnetic parameters, namely, the saturation magnetization (Ms), the remanent magnetization (Mr) and the

coercivity field (Hc)as obtained from the curves at Fig.7 and8are listed in Table 1.

 $Table1\ Magnetic\ properties\ of\ BaFe_{12}O_{19} and\ Polyaniline/BaFe_{12}O_{19} \\ composites\ prepared\ using\ chemical\ and\ solid-state\ polymerization$ 

Sample	Synthesisp athway	Ms (Am²/Kg)	Mr (Am²/Kg)	Hc (T)
BaFe2 <sub>12</sub> O <sub>19</sub>	Hydro- thermal	66.65	29.91	0.1118
10	СР	0.55	0.35	0.1212
	SP	5.77	2.19	0.1083
30	СР	8.25	3.5	0.1117
	SP	13.18	5.57	0.1094
50	СР	13.36	5.76	0.1026
	SP	18.52	7.64	0.1030
70	СР	20.06	8.72	0.1070
	SP	25.7	11.12	0.1026

The real content of barium hexaferrite in the composites after each synthesis was determined using the magnetic parameters. The weight fraction of  $BaFe_{12}O_{19}$ in the composites were convertedinto volume fractions by using densities of barium hexaferrite $p_{f}= 5.32 \text{ gcm}^{-3}$ , and Polyaniline,  $\rho_P = 1.45 \text{ gcm}^{-3}$ . The density of barium hexaferrite and Pani are written as follow:

$$\rho_f = \frac{m_f}{v_f} \tag{1}$$

$$\rho_P = \frac{m_P}{v_P} \tag{2}$$

where  $m_{f}$  and  $v_{f}$  are the weight and the volume of barium hexaferrite, respectively.  $m_{P}$  and  $v_{P}$  are the weight and volume of Polyaniline, respectively.

The weight fraction of hexaferrite *f*<sub>w/</sub>is given by

$$f_{wf} = \frac{m_f}{m_f + m_P} \tag{3}$$

Introducing Eq. (1) and (2) into Eq. (3) leads to

$$\frac{v_P}{v_P} = \frac{1 - f_{wf}}{f_{wf}} \frac{\rho_f}{\rho_P} \tag{4}$$

In The same way, volume fraction of hexaferrite  $f_{vf}$  is written:

$$f_{vf} = \frac{vf}{vf + vP} \tag{5}$$

To convert weight fraction to volume fraction, Eq. (4) is introduced in Eq. (5)

$$f_{vf} = \frac{\rho_P f_{wf}}{\rho_P f_{wf} + \rho_f (1 - f_{wf})}$$
(6)

The weight and volume fractions of Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites were calculated using the magnetic parameters obtained from VSM measurements then listed in Table 2.

Table2weight fraction  $f_w$  and volum fraction  $f_v$  of Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites prepared using chemical and solid-state polymerization

Sample	Synthesis pathway	$f_w$ before synthesis	$f_w$ after synthesis	$f_{v}$	Yield%
10	СР	0.10	0.008	0.002	8
	SP	0.10	0.086	0.025	87
30	СР	0.30	0.12	0.036	41
	SP	0.30	0.19	0.062	66
50	СР	0.50	0.20	0.063	40
	SP	0.50	0.23	0.075	46
70	СР	0.70	0.30	0.105	43
	SP	0.70	0.38	0.143	54

From the Table 1,it is evident that the Ms and Mrfor all samples increase with increasing  $BaFe_{12}O_{19}content$ , while Hc changes a little. The saturation magnetization of pure  $BaFe_{12}O_{19}$  was 66.65 Am<sup>2</sup>/Kg at room temperature which is close to the theoretical onecalculated for single-crystal barium hexaferrite, i.e. 72 Am<sup>2</sup>/Kg, asreported by Shirk and Buessem [25]. The Ms values for the composites (Fig.9) are lower than that of pure barium hexaferrite which depends of  $BaFe_{12}O_{19}$  content and uneven distribution in the composite[26]. The calculated weight and volume fraction in Table 2 confirms more these values.





Fig.9Variation of saturation magnetisation and coercivity of Polyaniline/BaFe $_{12}O_{19}$  composites obtained by: a) chemical polymerization and b) solid –state polymerization.

In fact, magnetisation saturation Ms is related to the volume fraction  $\phi$  of the particle and saturation moment of a single particle according to the following expression [27]

$$Ms = \varphi m_s \tag{7}$$

It is considered that the magnetization saturation of Polyaniline/BaFe<sub>12</sub>O<sub>19</sub> composites depends mainly on the volume fraction of the magnetic ferrite. In Table 2, the different values of weight and volume fraction of particles are depicted together with the yield. The yield is globally between 40 and 50% independently of the chemical route and quantity of ferrite, except for the lower amount of barium hexaferrite, 10wt%, witch correspond to a weight fraction of 0.83% and 8.6% while the volume fraction of 0.02% and 0.25% for chemical polymerisation and for solid-state polymerization, respectively. This is presumably due to a relative heterogeneity of the mix, so that the sample may not be representative of the mix.

The coercive force of the composites doesn't show significant change. However, a slight decrease with increasing fraction of ferrite is observed. This may be due to increasing magnetostatic interactions between particles as the distance decrease as the morphology and size of particles doesn't depend on the mixing method.

#### 4. CONCLUSION

Two types of polymerization methods were studied, namely, chemical and solid-state polymerization, in view of synthesizing Polyaniline/Barium ferrite composites. The particles have irregular shapes and are agglomerated. In fact, the powders produced by the solid-state polymerization exhibited a much higher degree of homogeneity than those obtained by chemical polymerization. The magnetic properties of the samples are mainly related to the weight and volume fraction of ferrite in the composites not the synthesis route. It is thus demonstrated that the solid-state polymerization, which is less complicated and less expensive than the classical chemical polymerization, can be used to prepare powders of Polyaniline/barium hexaferrite nanocomposites with good magnetic properties in view of applications.

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