Simple sol-gel auto-combustion synthesis and characterization of Lead Hexaferrite by utilizing cherry juice as a novel fuel and green capping agent

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Abstract

Lead Hexaferrite ($\text{PbFe}_{12}\text{O}_{19}$) nanoparticles have been developed by a new way preparing using the sol gel auto combustion route, without adding external surfactant. The present work focused on the synthesis and characterization of $\text{PbFe}_{12}\text{O}_{19}$ nanoparticles at low temperature using Cherry juice as a novel fuel and green capping agent for the first time. The effect of optimum amount of reductant, calcination temperature on the morphology and size of products were investigated. The size and agglomeration of the nanoparticles increase with decreasing Cherry juice content. When the amount of Cherry juice is 20 mL and calcination temperature is 800 °C, the product is pure and found to be $\text{PbFe}_{12}\text{O}_{19}$ nanoparticles. The products were characterized by X-ray diffraction pattern (XRD), scanning electron microscope (SEM), transmission electron microscopy (TEM), energy dispersive spectrometry (EDS). Furthermore, vibrating sample magnetometer (VSM) was used to study the magnetic properties of $\text{PbFe}_{12}\text{O}_{19}$ samples.

KEYWORDS: Nanostructures; Sol-gel; Auto combustion; Magnetic properties; Natural reductant.

1. Introduction

Hexagonal ferrites are group of ceramic magnets having large technological applications as permanent magnets, nanoelectromagnetic devices, microwave devices and magnetic recording media [1]. M-type hexagonal ferrites have appeared as novel materials with great technological and scientific interest considering their brilliant physical properties such as dependable magnetization, high coercive force, large magnetocrystalline anisotropy and relatively high Curie temperature, as well as notable chemical stability and low cost.

Among diverse inorganic materials, the hexagonal ferrites with a magnetoplumbite structure and a general formula $MFe_{12}O_{19}$ ($M=Ba, Sr, Pb$) are suitable candidate in chemistry and chemical engineering fields due to their great corrosion resistance and excellent chemical and thermal stability. In particular, the hexagonal ferrites are hard-magnetic magnets with a high saturation magnetization and a high theoretical maximum coercivity, which will profit the chemical process improvement [2-5].

There are numerous approaches to prepare hexagonal ferrites, such as phase inversion [6], electroless plating [7], electro spinning [8], template [9], high-energy ball milling [10], co-precipitation [11], hydrothermal [12], aqueous solution [13], micro-emulsion [14], glass-crystallization [15], salt melt methods [16] and sol-gel approaches [17]. As compared to other approaches, the sol-gel method has been confirmed to be an easy and adaptable method to construct hexagonal ferrites [18]. Different materials, such as metals, metal oxides and ceramics have been synthesized utilizing the sol-gel method [19, 20].

It is worthy to understand that properties of the powders depend on their particle size and morphology [21-28]. Consequently, exploring appropriate techniques to prepare ferrites and controlling the morphology and the size of the particles are noteworthy. Here, we extend the sol-gel auto combustion procedure to synthesize lead hexaferrite. This is a unique approach with an exclusive mixture of the chemical sol-gel procedure and the combustion procedure based on the gelling and subsequent combustion of an aqueous solution including salts of

the desired metals and the capping agent. Nowadays, for increasing biocompatibility and reducing pollution replacement of chemical compound by natural source is important in synthesis process because natural sources are green reagents and eco-friendly materials. The color of pomegranate, blueberry, black rice, cherry and etc., is due to anthocyanin pigments. These pigments which act as anti-oxidant reagent and in chemical reaction can be used as a reducing agent [29-30]. Furthermore anthocyanin pigment has a huge structure, so it can be used as a capping agent in nanomaterials synthesis procedure. In contrast to other works, our approach is clean, suitable, low-cost and eco-friendly practical process for green preparation of lead hexaferrite nanoparticles. This procedure is less complicated than the others. We have prepared lead hexaferrite from metal nitrates and cherry juice as capping agent. To the best of our knowledge, it is the first time that lead hexaferrite nanoparticles are formed at 800 °C by using cherry juice as novel fuel and capping agent in reactions.

2. Experimental

2.1. Materials and experiments

All the chemicals used in our experiments, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and $\text{Pb}(\text{NO}_3)_2$ were of analytical grade, were purchased from Merck and were used as received without further purification. XRD patterns were collected with a diffractometer of Philips Company with X'Pert Promonochromatized Cu K_α radiation ($\lambda=1.54 \text{ \AA}$). FE-SEM images were obtained on MIRA3 FEG-SEM. TEM images were obtained on a Philips EM208S transmission electron microscope with an accelerating voltage of 100 kV. EDS analysis was obtained on Philips EM208. The magnetic properties of the samples were detected at room temperature using a vibrating sample magnetometer (VSM, Meghnatis Kavir Kashan Co., Kashan, Iran).

2.2. Synthesis of $\text{PbFe}_{12}\text{O}_{19}$ nanoparticles

The $\text{PbFe}_{12}\text{O}_{19}$ nanoparticles were synthesized by the sol-gel auto combustion route. First, $\text{Pb}(\text{NO}_3)_2$ was dissolved in distilled water to form a clear solution. Then, an aqueous solution from different amount of cherry

juice including: in absence of cherry juice, 10 mL, 15 mL, 20 mL, 25 mL and 30 mL was added into the lead nitrate solution drop-wise under strong magnetic stirring at room temperature. The solution was heated by stirring at 60 °C. After stirring the solution for 30 min, an aqueous solution including $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was added to the above solution and was heated at 120 °C by stirring for 1h. The experiments were carried out with molar ratio between Pb and Fe, 1:6. Evaporation of the mixed solution affected the formation of a very viscous gel. Then the gel was dried in an oven at 120 °C. The final remain was calcined at diverse temperatures 800 °C for 2h. Table 1 lists the reaction conditions for the synthesis of $\text{PbFe}_{12}\text{O}_{19}$. This work is compared with the other work in Table 3.

3. Results and discussion

Fig. 1a-c shows the XRD patterns of samples were prapered in the presence of 5 mL, 10 mL and 15 mL cherry juice. When the amount of cherry juice was 5 mL a mixture of PbFe_4O_7 (JCPDS No.09-0048), PbFe_2O_4 (JCPDS No. 04-0705) and PbO (JCPDS No. 85-1414) and (JCPDS No. 01-0824) were obtained. In the presence of 10 mL cherry juice PbFe_4O_7 (JCPDS No.09-0048) and PbO (JCPDS No. 01-0824) were synthesized. With increasing the amount of cherry juice to 15 mL $\text{PbFe}_{12}\text{O}_{19}$ (JCPDS No.17-0660) was formed with impurity of PbO (JCPDS No.85-1287). Fig. 2a-e illustrate the XRD patterns of as-synthesized hexaferrites. When the amount of cherry juice was 20 mL and was calcined at 800 °C (sample no 5), the products had the peaks corresponding to pure hexagonal $\text{PbFe}_{12}\text{O}_{19}$ (JCPDS No. 31-0686) (Fig. 2a). When the cherry juice content increased to 25 mL (sample no 7), and 30 mL(sample no 8), a mixture of $\text{PbFe}_{12}\text{O}_{19}$, Fe_2O_3 (Fig. 2b) and only Fe_3O_4 (Fig. 2c) were obtained, respectively. Changing of the calcined temperature to 700 °C and the amount of 20 mL juice (sample no 6), the products had the peaks corresponding to hexagonal $\text{PbFe}_{12}\text{O}_{19}$ (JCPDS No. 31-0686) and Fe_2O_3 (JCPDS No. 04-0755). (Fig. 2d).Fig. 2e, exhibits XRD pattern of as-synthesized hexaferrites without cherry juice (sample no 1). in absence of cherry juice, only Fe_2O_3 was obtained. Therefore, These

patterns confirm that the minimum of cherry juice content for preparation of $\text{PbFe}_{12}\text{O}_{19}$ is 20 mL and the calcination temperature of 800°C is ideal temperature.

It was found that the existence of impurity peaks is due to the lack or excess of enough cherry juice as fuel and reductant. The ceramic samples were first prepared via the four basic steps: powder preparation, green body forming, sintering and finishing. Powders of $\alpha\text{-Fe}_2\text{O}_3$ and PbO in a 6:1 molar ratio were mixed in solvent on the stirrer [32], which at the beginning of the reaction Pb was reacted because of the potential reduction of less than Fe and PbO was formed, when the amount of reductant was low the powders of PbO and $\alpha\text{-Fe}_2\text{O}_3$ were not mixed completely and lead hexaferrites formation reaction was not done, with increasing the amount of cherry juice to 20 mL pure lead hexaferrites was obtained. Following excess of enough reductant, intermediate Fe_2O_3 was formed and in the presence of 30 mL cherry juice, Fe^{+3} was reduced to Fe^{+2} and Fe_3O_4 was synthesized. Therefore for the formation of pure $\text{PbFe}_{12}\text{O}_{19}$ is needed 20 mL cherry juice.

In continuance, the effect of cherry juice on the morphology of the hexaferrite nanoparticles was considered, while the molar ratio between Pb/Fe was chosen to be 1:6. Fig. 3a-d exhibit the SEM images of the hexaferrite nanoparticles achieved with different amount of cherry juice. It can be seen that the size and agglomeration of the nanoparticles decrease through the increasing of the cherry juice content. Fig. 3d with the amount of 20 mL cherry juice has even more distribution than other samples. Fig. 3f exhibits nanoparticles achieved without the presence of cherry juice. In fact, in absence of cherry juice as green capping agent, serious agglomeration of nanoparticles happened and only Fe_2O_3 was obtained (Fig. 3e). Therefore, cherry juice has critical effect on preparation of hexaferrite nanoparticles. The effect of the calcination temperature on the morphology of hexaferrite nanoparticles was considered. By decreasing the temperature from 800°C (Fig. 3d) to 700°C (Fig. 3e) and the amount 20 mL cherry juice, the agglomeration of nanoparticles increased and the homogeneity decreased. Therefore, the calcination temperature of 800°C is ideal temperature.

Typical histograms of the particle diameters for the samples Nos. 1, 2, 3, 4, 5 and 6 are seen in Fig. 4a–f, respectively. By comparing the particle size distribution of the products, it is found that $\text{PbFe}_{12}\text{O}_{19}$ prepared in the presence of 20 mL cherry juice and calcined at 800°C has smaller particle size distribution (15–45 nm). Also, $\text{PbFe}_{12}\text{O}_{19}$ prepared without cherry juice has the largest particle size distribution.

Fig. 4 reveals the TEM images of the hexaferrite achieved at calcination temperature 800°C for 2h (sample no 5) in various scales. These images exhibit formation of agglomeration of nanoparticles with width ranging nearly 10–15 nm.

Chemical purity and stoichiometry of the $\text{PbFe}_{12}\text{O}_{19}$ were tested by EDS. The strong peaks related to Fe and Pb are found in Fig. 5. Moreover, the EDS spectrum of sample no. 5 shows the oxygen that further confirms the formation of $\text{PbFe}_{12}\text{O}_{19}$.

Fig. 6 exhibits FT-IR spectrum of as-prepared hexaferrite nanoparticles and reveals two absorption peaks nearly 442 cm^{-1} and 587 cm^{-1} , corresponding to the Fe–O band in hexaferrite. FT-IR spectrum exhibits that the product does not show any strong IR-active peak consistent to impurities.

The hexaferrites are all ferrimagnetic materials, and their magnetic properties are intrinsically linked to their crystalline structures [31]. They all have a magneto crystalline anisotropy, that is the induced magnetization has a preferred orientation within the crystal structure. These materials are divided into two main groups: uniaxial hexaferrites (with an easy axis of magnetization) and ferroplana or hexaplana ferrites (with an easy plane, or cone, of magnetization) [31]. Fig. 7 shows the magnetization versus applied magnetic field ($M-H$) curve at 300 K for the as-synthesized lead hexaferrites (samples no. 5, 6). The hysteresis loops in this figure exhibit a ferromagnetic behavior for the $\text{PbFe}_{12}\text{O}_{19}$ nanostructures. The specific saturation (M_s) and coercivity (H_c) of sample no. 5 and 6 are also obtained from VSM measurement. The specific saturation magnetization and coercivity of sample no 5 (Fig. 6 a) are about 7.6 emu g^{-1} and 300 Oe respectively. M_s and H_c for sample no 4 (Fig. 6 b) is about 15.2 emu/g and the coercivity (H_c) of the sample is about 1100 Oe, This sample with a high

Hc is characterized as a hard ferrite and is appropriate for using permanent magnetic materials. Table 2 compares magnetic hysteresis parameters of samples.

4. Conclusion

Lead hexaferrites were synthesized via a simple sol-gel auto combustion route. This approach confirmed a new, simple, green, cost-effective and rapid method. The effect of the various amount of cherry juice and calcination temperature on purity and crystallinity of product was investigated, and it was found that lead hexaferrite nanoparticles are formed at 800 °C by using cherry juice as novel fuel and green capping agent for the first time. Moreover, concerning the purity of the product, crystallinity, and magnetic properties, this sample is the best one. The obtained product is a hard magnetic material. The specific saturation magnetization and coercivity are 15.2 emu/g and 1100 Oe, respectively.

Acknowledgment

Authors are grateful to the council of Iran National Science Foundation (91053846) and University of Kashan for supporting this work by Grant No (159271/1021).

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Figure captions

Fig 1. XRD patterns of the products synthesized in the presence of different amounts of cherry juice: (a) 5 mL (sample no. 2), (b) 10 mL (sample no. 3) and (c) 15 mL (sample no. 4).

Fig. 2. XRD patterns of lead hexaferrites prepared from $\text{Pb}(\text{NO}_3)_2$ and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ at calcinations temperature 800°C for 2h, with different amount of cherry juice: (a) 20 mL (sample no. 5) , (b) 25 mL (sample no. 7), (c) 30 mL (sample no. 8), (d) 700°C for 2h and 20 ml cherry juice (sample no. 6) and (e) without cherry juice (sample no. 1).

Fig. 3. SEM images of $\text{PbFe}_{12}\text{O}_{19}$ of (a) sample no. 2, (b) sample no. 3, (c) sample no. 4, (d) sample no. 5, (e) sample no. 6 and (f) SEM images of sample obtained in the absence of cherry jouce (sample no. 1).

Fig 4. Particle size distribution of samples Nos. (a) 1, (b) 2, (c) 3, (d) 4, (e) 5 and (f) 6.

Fig. 5. TEM images of lead hexaferrite (sample no. 5).

Fig. 6. EDS spectrum of sample no. 5.

Fig. 7. FT-IR spectrum of sample no. 5.

Fig. 8. Magnetization versus applied magnetic field at room temperature for the pure $\text{PbFe}_{12}\text{O}_{19}$ (sample no. 5, 6).

Table 1. The reaction conditions of lead hexaferrites synthesized in this work.

Fig 1.

Fig 2.

Fig. 3.

Fig 4.

Fig. 5.

Fig. 6.

Fig. 7

Fig. 8

Sample no	Amount of Reductant	Calcination Temperature (°C)	Calcination Time (min)	Particles size (SEM) (nm)	Product	Crystal phase
1	-	800	120	320	Fe ₂ O ₃	Hexagonal
2	5 mL	800	120	240	PbO and PbFe ₄ O ₇	
3	10 mL	800	120	170	PbO, PbFe ₂ O ₄ , PbFe ₄ O ₇	
4	15 ml	800	120	110	PbFe ₁₂ O ₁₉ , PbO	
5	20 ml	800	120	15	PbFe ₁₂ O ₁₉	Hexagonal
6	20 ml	700	120	22	PbFe ₁₂ O ₁₉	Hexagonal
7	25 ml	800	120	-	PbFe ₁₂ O ₁₉ , Fe ₂ O ₃	
8	30 ml	800	120	-	Fe ₃ O ₄	

Table 2.

Sample no.	M_r (emu/g)	M_s (emu/g)	H_c (Oe)
5	6.69	15.20	1100.90
6	1.90		300

Table 3.

Calcined temprature	Reductant	Particle size (nm)	Refrence No
900 °C.	Citric acid	150-400	33
920 °C	NaOH/Na ₂ CO ₃	500	34
900 °C	Maltose	200-400	20
900 °C	Maleic acid	200-500	35

Lead Hexaferrite Nanoparticles

Pb/Cherry Juice

1:6.5

Pb/Cherry Juice

1:13

Pb/Cherry Juice

1:26

Without Cherry Juice