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Sol–Gel Based Liquid-Mix Method for the Synthesis of Znfe₂O₄ Spinel

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ABSTRACT

he superparamagnetic $ZnFe_2O_4$ (ZFO) powder with spinel structure was synthesized by a sol-gel based liquid-mix method using deionized water, citric acid, iron nitrate, zinc nitrate, and propylene glycol. Based on the principles of the Pechini method, the obtained solutions were treated under reflux at 95 °C followed by drying to obtain the desired powder. Simultaneous thermal analysis (STA) showed that there are three main thermal events at 165, 300, and 800 °C, which are related to the evaporation of water and/or volatile chemicals, combustion of organic materials, and chemical reactions to form ZFO phase, respectively. Among different calcination temperatures, thermal treatment at 900 °C led to the formation of a powder which its XRD pattern is well-matched with the ZFO standard peaks. Scanning electron microscopy (SEM) showed that calcination at 400-600 °C led to some agglomerated sediment particles in the range of 50-100 nm, whereas the morphologies of the samples calcined at 700-900 °C consisted of some fused particles with larger size (~1 μ m). The vibrating sample magnetometer (VSM) results approved that although magnetization saturation (Ms) values were negligible in the samples calcined at lower temperatures (lowtemperature samples), Ms value of the sample calcined at 900 °C was about ± 7.5 emu/g. On the other hand, the S-shaped hysteresis curves of the high-temperature samples (calcined at 700-900 °C) and consequently zero/near-zero value for the coercivity (Hc) and remanence (Mr) parameters confirmed the superparamagnetic behavior of the as-synthesized ZFO compound. Prog. Color Colorants Coat. 14 (2021), 139-147[©] Institute for Color Science and Technology.

1. Introduction

Mixed-metal oxide compounds are extremely versatile engineering materials useful as catalysts [1, 2], sensors [3], solar cells [4], corrosion inhibitors and anticorrosive coatings [5-7], electronic and magnetic materials [8-10], medical applications [11], etc. Spinel are included in this category as they are composed of oxygen ions and bivalent and trivalent metal ions which are located in a cubic structure [12, 13]. The stoichiometric mixture of ZnO and α -Fe₂O₃ forms the iron zinc oxide or ZFO spinel structure. ZFO has attracted a great deal of attention due to its unique magnetic behavior, photochemical stability, and photovoltaic and photocatalytic performance [14-17]. Therefore, various methods and protocols have been developed to approach such a compound, including sol-gel [18], sol-gel template [17], sol-gel auto– combustion [16], solvothermal/hydrothermal-assisted sol-gel [19, 20], co-precipitation methods [21], etc. Although all the mentioned techniques are based on chemical methods, most of them have tried to improve the sol-gel technique and diminish its drawbacks. However, the liquid-mix technique, designed on the sol-gel method basis, is an improved sol-gel process which has been named after its inventor, Maggio Pechini [22].

Preparing a citrate-metal solution at the first step of this process provides conditions for the formation of a covalent polymer network which could embrace metal ions. At the next step, transesterification between citrate solution and alcohol should be occurred to form a metal–citrate chelate complex [23]. The advantage of this method is that it provides a homogeneous polymer network, making the metal ions disperse uniformly; therefore, it could be useful in the preparation of mixed-metal oxides such as spinel compounds.

In this work, a zinc-iron mixed oxide has been synthesized via the sol-gel based liquid-mix method for the first time. The main aim is to fabricate a homogenous mixed-oxide compound based on the Pechini sol-gel technique, which could be considered for synthesizing other mixed-oxide compounds. However, the effects of calcination temperature on the phase component, morphology, magnetic and colorimetric parameters have been also evaluated.

2. Experimental

2.1. Starting materials and chemical procedure

All chemicals were purchased from Merck and used without further purification.

In a reflux column, 0.45 mol citric acid monohydrate (C₆H₈O₇.H₂O) was added to 120 mL deionized water under moderate stirring. The solution was then heated to 70 °C for 10 min. In two different 0.1 mol iron nitrate nonahydrate beakers. (Fe(NO₃)₃·9H₂O) was dissolved in 120 mL deionized water and 0.05 mol zinc nitrate hexahydrate (Zn(NO₃)₂.6H₂O) was dissolved in 120 mL deionized water. The Fe-containing solution was added to the citric acid solution with a rate of 1 drop per second. Then, the Zn-containing solution was added at the same rate. Stirring at 70 °C was performed during these steps and continued 2 h afterward. At the next step, 0.8 mol propylene glycol was heated to 80 °C and added to the obtained solution under stirring. After the addition of the alcohol, 100 mL distilled water was added and

agitated for 3 h at 95 °C. Finally, the achieved solution was taken out from the reflux column, heated at 95 °C to evaporate the volatile constituents to obtain a gel. This gel was dried and fired in an electric furnace at 400-800 °C. These samples were named as S_T , where T shows the calcination temperature.

2.2. Characterization

Several analysis methods were utilized to evaluate and characterize the resultant powder samples. The phase component was determined by an X-ray diffractometer (PANalytical, Philips, Netherlands). The mass fractions of the crystallized phases in each sample were estimated on the matrix flushing model and its mathematical equation (Eq. 1) [24]. For this reason, the percentage of each phase (P_n) in a composite compound is related to the scale factor (*SF*) and reference intensity ratio (*RIR*) values of that phase. The RIR values which are achieved from the X'Pert software database are equal to 5.420, 3.010, and 3.800 for ZnO [JCPDS 76-704], Fe₂O₃ [JCPDS 73-2234], and ZnFe₂O₄ [JCPDS 22-1012], respectively [25]. SF values were achieved from the XRD results.

$$P_n = \frac{\frac{SF_n}{RIR_n}}{\sum_{i=1}^n \frac{SF_i}{RIR_i}} \times 100 \tag{1}$$

The thermal behavior of the samples was recorded through simultaneous thermal analysis (BAHR Thermoanalyse, Germany). Fourier-transform infrared (FTIR) spectroscopy was accomplished by а spectrophotometer (Spectrum One, PerkinElmer, USA). Scanning and transmission electron microscope micrographs were acquired by SEM (LEO 1455VP) and TEM (Zeiss 900) devices, respectively. The magnetic properties were evaluated by a vibrating sample magnetometer (VSM). The CIELAB color space was evaluated by a Gretag Macbeth spectrophotometer (Color Eye 7000a) to determine LAB parameters under D65, A, and TL84 standard illuminant and CIE1964 standard observers.

3. Results and Discussion

3.1. STA, XRD, and chemical mechanisms

The resulted gel should be calcined to provide conditions in which the crystallization process can occur. Thermal analysis of this system could determine the temperature range of physical and chemical events and reactions. Figure 1 shows an STA profile at the temperature range of 25-1000 °C. The DTG curve shows two main events at 165 and 300 °C that correspond to ~60% weight loss (TGA curve). On the other hand, the DTA curve shows an exothermic peak at ~ 300 °C. Based on these data, it is clear that the event at 165 °C is related to the evaporation of water and/or volatile chemical. Although the evaporation is an endothermic event, exothermic oxidation reactions caused to remove their trace on the DTA curve. The effects of these reactions on the thermal behavior of the system are obvious at 300 °C in DTA and DTG curves. However, it seems that the required temperature for this system should be more than 300 °C. Although there is no considerable weight change at the temperature range of 300-1000 °C, DTA shows an endothermic peak at 800 °C. This event starts from 400 °C and continues to 1000 °C, therefore 6 different calcination temperatures, i.e. 400, 500, 600, 700, 800, and 900 °C, were selected to evaluate the effects of firing temperature on the final products. XRD patterns are required to analyze this reaction.

The crystal structure and phase component of the samples were analyzed via XRD method and the obtained results are presented in Figure 2. Although trace of the goal phase, ZFO, appeared from S_{400} , its intensity increased by temperature. On the other hand, Fe₂O₃ and ZnO were crystallized, which was predictable. It seems that the crystallinity degree of these two phases is maximized at 700 °C. By increasing the calcination temperature to 800 °C, the intensity of their peaks decreased slightly, whereas ZFO peaks were intensified. Finally, calcination at 900 °C led to a sample which is matched well with ZnFe₂O₄ standard peaks [JCPDS 22-1012]. The crystal system of such a compound is a regular spinel cubic with a space group of Fd3m. Its main peaks are located at 35.265°, 29.920°, 62.213°, 56.631°, and 42.845° with miller indexes of (311), (220), (440), (511), and (400), respectively.



Figure 1: STA profile in the temperature range of 400-1000 °C including TGA, DTA, and DTG curves.



Figure 2: XRD patterns of the samples calcined at different temperatures.

Based on the STA and XRD results, the observed endothermic peak at 800 °C maybe attributed to the formation of ZFO crystals. Since the crystallization and growth of ZFO crystals are controlled by diffusion, calcination at 900 °C led to the highest purity. According to the matrix flushing model evaluation, the mass fractions of the mentioned crystalline phases are presented in Figure 3, which shows the effects of the calcination temperature on the ZFO formation. It seems that Fe₂O₃ and ZnO are formed at a lower temperature. In the aqueous sol-gel method, oxygen is required to form a metal oxide. Usually, water provides this amount of oxygen, but not as efficient as it should be. Modified techniques, such as liquid-mix method, facilitate the oxygen supplying via using alcohol [26]. In such a system, increasing the temperature provides

the surfactant-controlled condition, in which metal ions transfer directly into metal oxide [27]. The suggested reaction sequences are presented below.

$$Fe(NO_3)_3 + 3H_2O => Fe(OH)_3 + 3HNO_3$$
(2)

$$Zn(NO_3)_2 + 2H_2O => Zn(OH)_2 + 2HNO_3$$
 (3)

Equations 2 and 3 show reactions, where the nitrate compounds were solved in water and took part in the hydrolysis reactions.

Metallic hydroxide compounds and citric acid form metal citrate complexes (Eq.4 and 5):

$$Fe(OH)_3 + C_6H_8O_7 = >FeC_6H_5O_7 + 3H_2O$$
 (4)

$$3Zn(OH)_2 + 2C_6H_8O_7 = Zn_3C_{12}H_{10}O_{14} + 6H_2O$$
 (5)



Figure 3: The mass fractions of the crystallized phases in each sample on the matrix flushing model.

The next series of reactions are transesterification which takes place between the citrate compounds and alcohol. Propylene glycol as a diol alcohol was used, so, the transesterification reaction led to the formation of metal/organic gel. This process is described in Figure 4.

$$FeC_6H_5O_7+C_3H_8O_2 =>$$
Iron-organic gel (6)

$$Zn_{3}C_{12}H_{10}O_{14} + C_{3}H_{8}O_{2} => Zinc-organic gel$$
(7)

Polyesterification and covalent network formation occurred by heating the obtained solution and gel. Heat treatment of the powder led to the combustion of organic matrix at about 300 °C (see DTA curves) and probably resulted in the formation of oxide phases, i.e. Fe_2O_3 and ZnO (XRD patterns). Further increase in calcination temperature caused the as-synthesized oxides to combine and form a spinel structure, ZFO (XRD patterns).

3.2. SEM observations

Figure 5 shows SEM images of the samples calcined at different temperatures. As can be seen, S_{400} , S_{500} , and S_{600} samples showed agglomerated sediment particles. This irregular structure is induced by the exhaust of gases during the combustion of organic compounds. The grains are in the range of 50-100 nm. By increasing the temperature to 700 °C, some bigger and different-shaped particles were observed in S_{700} . These particles became more obvious in S_{800} and S_{900} samples. These particles are most probably related to the desired phase of ZFO. Although the morphologies of these particles (S_{700} , S_{800} , and S_{900}) are not regular, the particle's shapes of the other samples (S_{400} , S_{400} , and S_{600}) are completely different.



Figure 4: Schematic illustration of transesterification reaction which leads to the formation of metal/organic gel.



Figure 5: SEM images of the samples.

3.3. FT-IR spectroscopy

Figure 6 shows the FTIR spectra of the samples calcined at different temperatures. The wide vibration at 3450 cm⁻¹ and a tiny vibration at 1640 cm⁻¹ are due to the hydroxyl stretching vibration and the hydroxyl bending vibration, respectively from the surface water [28]. These vibration peaks were intense in S_{400} - S_{600} samples, whereas the increases in the firing temperature led to a decrease in their intensities. This could be related to the water absorption in powder samples. As can be seen in SEM images, samples S_{800} and S_{900} contain some sintered particles with lower

porosity and holes which leads to lower water absorption. Another tiny peak at 2350 cm⁻¹ is attributed to the asymmetric stretching vibrations of CO₂. A little amount of residual organic compounds could cause this vibration. Increasing the firing temperature diminished it from S_{700} - S_{900} samples. The weak bands observed at 1440 and 1030 cm⁻¹ correspond to CH₃ bending and COH stretching vibrations, respectively. The last vibration in the range of 600-500 cm⁻¹ is attributed to M-O bonds (M = Zn and/or Fe) [29].

3.4. VSM results

The magnetic properties of the synthesized samples were evaluated by the field dependence of magnetization as shown in Figure 7, providing remarkable insights into the effects of calcination temperature on characteristics of the powder samples such as magnetization saturation (Ms), coercivity (Hc), and remanence (Mr). Based on the magnetic hysteresis curves, Ms values for S_{400} - S_{600} samples were very low as compared to S_{700} - S_{900} samples, mainly due to the



Figure 6: FT-IR spectra of the samples.

lack of magnetic phase in these samples (XRD results). By increasing the calcination temperature, the Ms values were increased and reached to about ± 7.5 emu/g in S₉₀₀ samples. This is due to the higher amount of FeZn₂O₄ as well as its higher crystallinity and purity. In addition, the differences between Ms in S₈₀₀ and S₉₀₀ samples can also be related to the cation dispersion in the spinel structure and exchange interaction between the tetrahedral and octahedral sites [30]. The more similarity between a crystalline structure and perfect spinel structure, the higher the magnetization saturation level. On the other hand, VSM results showed no Mr and Hc, suggesting the superparamagnetic behavior of the synthesized compounds [31].

3.5. CIE LAB results

The colorimetric attributes of the samples are measured and presented in Table 1 and Figure 8. The parameters



Figure 7: VSM curves of the samples.

L*, a*, and b* correspond to lightness, rednessgreenness, and yellowness-blueness, respectively. It is clear that the lightness (L*) decreased slightly by increasing the calcination temperature. In other words, samples became darker by firing in higher temperatures. Unlike the first parameter, a* and b* values decreased sharply by increasing the calcination temperature. This means that the yellowness and redness indexes of the S_{700} - S_{900} samples decreased compared to the S_{400} - S_{600} samples. These results showed the differences between optical behaviors of the FeZn₂O₄ phase and Fe₂O₃/ZnO mixed oxide compound.





Illuminant ↓	Color Values↓	Samples					
		S ₉₀₀	S ₈₀₀	S ₇₀₀	S ₆₀₀	S ₅₀₀	S ₄₀₀
D65	L*	43.59	48.11	44.66	44.66	49.98	50.90
А		44.25	50.12	46.65	45.78	53.46	54.18
TL84		43.96	49.18	45.63	45.30	51.69	52.54
D65	a*	3.91	11.05	12.09	15.01	21.18	19.91
А		4.91	13.29	13.97	15.32	23.31	22.21
TL84		3.78	10.63	12.42	14.80	21.59	20.23
D65	b*	3.09	11.45	9.21	13.20	17.22	16.22
А		4.20	14.62	12.50	15.30	22.94	21.61
TL84		3.78	13.50	11.05	15.01	20.31	19.23

 Table 1: The colorimetric parameters for different samples under D65, A, and TL84 standard illuminant and CIE1964 standard observers.

4. Conclusions

In summary, $ZnFe_2O_4$ as a mixed-oxide compound with spinel structure has been synthesized via sol–gel modified Pechini method. Iron nitrate and zinc nitrate were used as metal-containing precursor and citric acid was used for the formation of metal citrate complexes. Propylene glycol as a diol alcohol was used to facilitate the transesterification reactions and form a metal/organic gel. This is a unique step, which makes the Pechini method prominent and leads to the formation of a covalent polymer network embracing metal ions uniformly. Such a uniform distribution

could provide suitable conditions for the synthesis of mixed-oxide compounds. Based on the STA results, a temperature range of 400-900 °C was selected for the calcination of the prepared powder samples. XRD patterns confirmed that 900 °C was the best temperature to achieve the highest amount of ZFO phase. SEM images showed some fused particles with sizes above 100 nm. The feature of the S-shaped hysteresis loop with Ms=7.5 emu/g, Mr=~0 emu/g, and Hc=0 Oe approved the superparamagnetic behavior of this sample.

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