



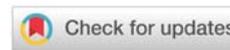
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Short Communication

Micro/nanosized ferrite and hexaferrite structures: The polyol processes for synthesis

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Abstract

In this research, controlled synthesis and properties of micro/nano-sized magnetic oxide powders via a polyol process are discussed. They are the original magnetic oxides that have been studied by many scholars because of their many applications. The most special forms are the diversity of FeO and Fe₂O₃, and the diversity of the Fe₃O₄ matrix which are the most important original structures with formulas to be FeO.Fe₂O₃ (Fe₃O₄) and Fe₂O₃ can be oxide systems of Fe_xO_y that are very common and different. On the basis of the original structures, we can synthesize the most special forms of ferrite and hexaferrite related to the four original structures, which are AB₂O₄-spinel-type SrFe₂O₄ and BaFe₂O₄ ferrites (M: metal elements, Sr, Ba; B: Fe), PbFe₁₁AlO₁₉-type MX₁₂O₁₉ (M: metal elements, Sr, Ba; X: Fe) SrFe₁₂O₁₉ and BaFe₁₂O₁₉ by polyol processes and technologies. We understand that elements that form original structures are incorporated into Fe₃O₄ to form ferrite and hexaferrite structures, typically such as SrFe₁₂O₁₉ by liquid-, solid- and gas-based phase chemical reactions via the polyol processes and heat treatment processes. In the future, we suggest that the original oxide structure and formula of natural minerals can be used as the new magnetic structures and compounds.

Introduction

Recently, micro/nanosized Co-, Fe-, and Ni-based magnetic metals, alloys, and oxides have been intensively studied for their practical applications in electronics and telecommunications, photonics, optoelectronics, information technology, and communication as well as catalysis, energy, and environment [1,2]. The polyol process was successfully used to synthesize magnetic Co, Fe, and Ni particles in various kinds of H₂O, ethanol, methanol, kinds of glycols, polypropylene glycol, ethylene glycol, or polyethylene glycol, especially ethylene glycol. The polymers, such as Polyvinylpyrrolidone (PVP), and Cetyltrimethylammonium Bromide (CTAB) are protective agents to the as-prepared particles [3,4]. It is known that the mineral is known as MgAl₂O₄ with the defined original spinel structure (S-type). Based on this spinel structure, the various kinds of spinel ferrites with the same formula of MgAl₂O₄-type MO.Fe₂O₃ (MFe₂O₄, M: Mn, Fe, Co, Zn, Mg, Ni, Cu, and others) (M: Ca, V, Cr, and others) have also been developed by sol-

gel technology and sintering, even combustion methods for magnetic recording, spintronics, hyperthermia, theranostics, and special areas [3-5]. In further experimental investigations, these led to the development of rare-earth garnets of Mg₂Al₂(SiO₄)₃-type R₃Fe₅O₁₂ or 3R₂O₃.5Fe₂O₃ (R: Y, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu) and was also focused on their applications [1,2]. In most special cases, the various kinds of hexaferrites based on magnetic coercivity were developed. They consisted of PbFe₁₁AlO₁₉-type BaFe₁₂O₁₉, SrFe₁₂O₁₉, MO.6Fe₂O₃ (M: Ba, Sr, Pb) (M-type, SrM; BaM; PbM) or BaO.2MO.8Fe₂O₃ (W-type) (M: Mn, Fe, Co, Ni, Zn, Mg), 2BaO.2MO.6Fe₂O₃ (Y-type) (M: Mn, Fe, Co, Ni, Zn, Mg), 3BaO.2MO.12Fe₂O₃ (Z-type, M: Mn, Fe, Co, Ni, Zn, Mg), and CaTiO₃-type magnetic oxide perovskite, typically such as BiFeO₃ (ABO₃, A: Bi, Ln, Ca, Sr, rare earth, and others, B: Fe) [2]. The use of Co in the defined original structures has led to the classification of the structures of ferrites and hexaferrites in the forms of S, M, W, X, Y, Z, and U (4BaO.2MO.18Fe₂O₃) by adjusting cations and anions at the sites of original crystal structures, respectively [1,2]. They

are the so-called mixed magnetic oxides and ceramics. The unlimited potential applications of the various kinds of ferrites and hexaferrites are clearly confirmed in electronics and telecommunications [1,2]. Additionally, magnetic alloys can be formed by the technologies of sintering or heat treatment with the change of magnetic oxides into magnetic alloys with reducing agents at high temperatures (CaH_2 , H_2 , solid and common gas-reducing agents by sintering processes) [4].

Therefore, original magnetic properties need to be enhanced, changed, and retained in the structural modifications of the above various structures. In this research, we understand that the new, modified, and easy-to-apply polyol processes, and heat treatment processes are effectively used for the controlled synthesis of the above original structures that are crucial to researchers. As a result, we present a typical experimental preparation of $\text{SrFe}_{12}\text{O}_{19}$ hexaferrite in our investigations and experiences.

Processes

In a typical process, the magnetic oxide powders based on the so-called original structures were carefully prepared with a reducing agent, and heat treatment after chemical synthesis under control [4,5]. The chemicals used included NaBH_4 , $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (or/and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$), $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ for the synthesis of the structure of $\text{SrFe}_{12}\text{O}_{19}$, ethylene glycol, polyvinylpyrrolidone (PVP), acetone, ethanol, NaOH , KOH , NH_4OH , and kinds of H_2O , respectively. Sr-Fe-O oxide powder products after cleaning polymer were obtained and dried into micro/nanosized magnetic powders at 200–240 °C. The powder samples of ferrite and hexaferrite were heated at 950 °C in a furnace to obtain the final powder with a metallic bright-black color. The as-prepared powders were measured by a vibrating sample magnetometer (VSM), Model10-VSM, MicroSense, and demagnetization field in the range of -1500 to +1500 Oe. The structures of Sr-Fe-O and $\text{SrFe}_{12}\text{O}_{19}$ samples were investigated by X-ray diffraction (XRD) (D2 Phaser/Bruker AXS diffractometer, Germany).

Results and discussions

In recent studies, we found out that Fe_3O_4 -type oxide exhibited original structures for the potential of producing other new structures due to their very porous structures of noncrystals or crystals that are very good for the integration of ion metal others or ion rare earth elements into the common porous matrix. It means that micro/nanosized ferrite and hexaferrite can be produced in a variety of porous Fe_3O_4 -type matrices. We also found that there was a coexistence of the two crystalline phases of $\alpha\text{-Fe}_2\text{O}_3$ (minor phase) and $\text{SrFe}_{12}\text{O}_{19}$ (major phase). The as-prepared structure of $\text{SrFe}_{12}\text{O}_{19}$ was assigned to the standard pattern to be PDF#33-1340 (Strontium iron oxide; $\text{CuK}\alpha$; Hexagonal; $\text{P}6_3/\text{mmc}(194)$; Eight strong lines; Strongest line: (114)). However, the XRD data and results were not presented here. It is evidenced that the most typical magnetic parameters of micro/nanosized $\text{SrFe}_{12}\text{O}_{19}$ oxides are calculated to be M_s about 33.35 (emu/g), M_r (remanent magnetization: M at $H = 0$) to be 18.64 (emu/g), and H_c (coercive field: Field at which M/H changes sign) to be 5072.22 (Oe) in

the average values, respectively (Figure 1). The high value of $\text{BH}_{\text{max}} = 2313$ MGOe (Maximum energy loss of the hysteresis loop) is measured. We show that superparamagnetism was transformed into ferrimagnetism when the Sr element was incorporated into the porous original structure of Fe_3O_4 to form the original crystal structure of $\text{SrFe}_{12}\text{O}_{19}$ at high temperatures.

In this study, we aim to focus on the future perspectives of mixed ferrites and hexaferrites from the defined original crystal structures, typically such as oxide systems of FeO , Fe_3O_4 , Fe_2O_3 , $\text{SrFe}_{12}\text{O}_{19}$, and $\text{BaFe}_{12}\text{O}_{19}$ by the polyol processes and technologies with heat treatment on industrial synthesis approach. At present, no evidence is available for the formation of hexaferrites through the chemical reaction in common solutions and solvents (i.e., H_2O , ethanol, methanol, and various industrial glycols) as that will be the new research trend for synthesis [6].

The authors, N.V.Long and N.T.N. Hang have used the original equation of controlled synthesis of porous Fe_3O_4 crystal oxides (the Elmore reaction [7], an important and meaningful reaction, i.e. $\text{FeCl}_2 + 2\text{FeCl}_3 + 8\text{NaOH} \rightarrow \text{Fe}_3\text{O}_4 + 8\text{NaCl} + 4\text{H}_2\text{O}$ for small scale synthesis in the laboratory, and the ideas of industrial synthesis approach at large scale; NaOH , KOH , and NH_4OH are also important in experimental; NaBH_4 , KBH_4 , and other agents are suitable catalysts for the formation according to time and reaction temperature) as a discovery of black box equation for the synthesis of nano/microsized ferrites and hexaferrites that are formed in heat treatment. The original systems of nano/microsized ferrites and hexaferrites are formed by heat treatment. It means that the mixed oxides such as Mn-Fe-O, Co-Fe-O, Ni-Fe-O, Mg-Fe-O, Zn-Fe-O, and others based on the original Fe_3O_4 -type forms are effectively formed in the highest crystallization of MFe_2O_4 (M: Mn^{2+} , Co^{2+} , Ni^{2+} , Mg^{2+} , Zn^{2+} etc) by sintering or heat processes [3-8]. To improve the ferrimagnetism of MFe_2O_4 , the Co element is added in the experiments to form the modified and mixed oxide structures with the formula of $\text{M}_x\text{X}_y\text{Fe}_2\text{O}_4$ (M: Mn^{2+} , Ni^{2+} , Mg^{2+} , Zn^{2+} and others; X: Co^{2+} ; $x + y = 1$; $x = y = 0.5$). Rare earth can be incorporated into Fe_3O_4 -type matrix for the formation of RFeO_3 and ABO_3 (original structure of perovskite; R: Y, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu; typically as HoFeO_3 ; A: Bi; B: Fe; typically as CaTiO_3 -type BiFeO_3). Thus, the same polyol process for the synthesis of hexaferrite with the original formula of $\text{MFe}_{12}\text{O}_{19}$ (M: Ba, Sr; $\text{BaFe}_{12}\text{O}_{19}$; $\text{SrFe}_{12}\text{O}_{19}$; $\text{Sr}_x\text{Ba}_y\text{Fe}_{12}\text{O}_{19}$; $x + y = 1$; $x = y = 0.5$) in our research open a new way of controlled synthesis for the new kinds of mixed ferrites and hexaferrites, by industrial

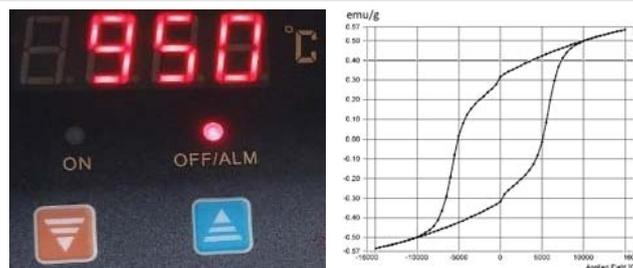


Figure 1: Hysteresis of $\text{SrFe}_{12}\text{O}_{19}$ heated at 950 °C (0.017 g powder for VSM measurement).

synthesis approach according to their potential applications for electronics and telecommunications, especially such as the various kinds of new ferrite and hexaferrite magnets.

Conclusion

Based on their crystal structures, original ferrite and hexaferrite structures are meaningful for the further investigation of new, modified, and mixed ferrites and hexaferrites. The polyol process can lead to polyol technology (N.V. Long and N.T.N. Hang's new ideas of "original equation for synthesis", and "original structures and formulas of materials for research") for the controlled synthesis of new magnetic mixed nanomaterials in the future. In this study, the Fe_3O_4 matrix (a special form of ferrite) is very important in order to synthesize the structure of MFe_2O_4 (M: metal; micro/nanosized ferrites; Fe: major element), and $\text{MFe}_{12}\text{O}_{19}$ (M: Sr, Ba; micro/nanosized hexaferrites; Fe: major element).

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