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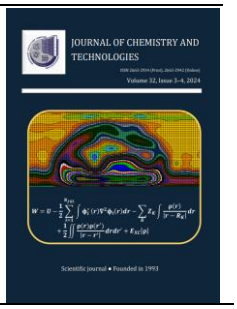

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NOVEL METHODS OF SPINEL FERRITES PRODUCTION: MINI-REVIEW

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Abstract

Spinel ferrites as a type of inorganic compounds have attracted the interest of scientists since their discovery in the last century until now. Spinel ferrites have become one of the best materials for various applications such as catalysts, adsorbents, gas sensors, lithium battery fillers, information storage systems, magnetic fluids, microwave absorbers, etc. The popularity of these ferrites is due to the ability to regulate their specific properties, such as coercive force, permeability, resistivity, and saturation magnetization, by changing their chemical composition and production technologies. In the review, a comparative analysis of various compositions of spinel ferrites, the structure of spinel ferrites, and possible applications was carried out. The classification of methods of obtaining was carried out. Liquid-phase methods can be considered more technologically feasible. When comparing hydrothermal, sonochemical, and sol-gel synthesis methods, it was found that the hydrothermal process makes it possible to adjust the size, shape, monodispersity, and crystallinity. The newest technologies for obtaining ferrites are also considered in detail; plasma, and ultrasonic. Their features are described.

Keywords: spinel; ferrite; self-deposition; ultrasound; plasma; saturation magnetization.

НОВІТНІ СПОСОБИ ОДЕРЖАННЯ ШПІНЕЛЬНИХ ФЕРИТІВ: МІНІ ОГЛЯД

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Анотація

Шпінельні ферити як різновид неорганічних сполук викликають інтерес вчених з моменту їх відкриття в минулому столітті і дотепер. Шпінельні ферити стали одними із найкращих матеріалів для різних областей використання, таких як виготовлення каталізаторів, адсорбентів, газових датчиків, наповнювачів літієвих батарей, систем зберігання інформації, магнітних рідин, мікрохвильових поглиначей тощо. Популярність цих феритів пояснюється можливістю регулювати їх специфічні властивості, наприклад, коерцитивну силу, проникність, питомий опір і намагніченість насичення, змінюючи їх хімічний склад і технології виробництва. В огляді проведено порівняльний аналіз різноманітних за складом шпінельних феритів, будову шпінельних феритів, можливості застосування. Проведено класифікацію методів отримання. Рідкофазні методи можна вважать більш технологічно доцільними. Під час порівняння гідротермального, сонохімічного та золь-гель методів синтезу з'ясовано, що гідротермальний процес дає змогу регулювати розмір, форму, монодисперсність, кристалічність. Детально розглянуто також новітні технології отримання феритів: плазмовий, ультразвуковий. Описані їх особливості.

Ключові слова: шпінель; ферит; співосадження; ультразвук; плазма; намагніченість насичення.

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Introduction

Spinel ferrites have attracted much attention of researchers from both fundamental and applied point of view, due to the unique properties of these compounds, which, accordingly, determines their use as catalysts, adsorbents, gas sensors, fillers for rechargeable lithium batteries, information storage systems, magnetic liquids, microwave absorbers, etc. [1–6].

Successful application of ferrites in these fields requires appropriate electrical and magnetic properties, which are determined primarily by the structure [7]. The general molecular formula of spinel ferrites is MFe_2O_4 (where M is Mn, Fe, Co, Ni, Cu, and Zn), and most ferrites exhibit superparamagnetic properties due to the nanoparticle size of about 20–50 nm [8]. The crystal chemistry of ferrites is a very important factor, and the correct choice of synthesis technology determines a certain structure of the crystal lattice and chemical composition and allows to regulate the physicochemical properties. Metal cations in octa- and tetra sites of the crystal lattice are distributed taking into account ionic radius, synthesis technologies, and synthesis parameters [9; 10].

Despite the expansion of the field of application of nanodispersed ferrites, the existing list of production technologies does not satisfy consumers in terms of purity, shape, size, monodispersity, stability and morphology of particles, cost, quantity and composition of the generated liquid waste [11].

Results and discussion

Recently, new methods for synthesizing nanodispersed ferrites have been developed, but all of them have certain disadvantages. The existing methods of synthesis are divided into two large groups, namely: solid-phase and liquid-phase [12; 13]. Solid-phase technologies require preliminary grinding and activation [12].

The liquid phase involves the crystallization of a solid phase from a liquid under the influence of chemical or physical factors. There are several methods of liquid-phase synthesis: co-precipitation, thermal decomposition, hydrothermal, solvothermal, sol-gel, sonochemical synthesis, and microemulsion method. The solid-phase ones include ceramic, activated grinding methods, and processing in a microwave oven. Among these methods of synthesis, technologies using high-energy chemistry occupy a special place (modified method using diatomic alcohols, sonochemical and microwave irradiation, and plasma chemical). These are new methods, but they are used more and more often. These synthesis methods have many advantages, such as shorter reaction times, milder reaction conditions, and higher productivity [14].

Most methods for obtaining nanodispersed ferrites require the use of hazardous chemicals and significant energy consumption. In the last five years, the number of works devoted to the "green" synthesis of ferrites has increased.

It is promising to use natural reagents found in the environment, such as fruits, vegetables, microorganisms, plant waste, as reducing agents. Natural polymers are used as blocking substances that are easily decomposed, for example, marine and annual algae and sugars. Various parts of plants (roots, seeds, leaves, stems, shoots) are an effective fuel for the synthesis of nanoparticles. It is the "green synthesis" of nanoparticles that is gradually occupying a scientific niche alongside conventional methods, as it is simple, economically expedient, environmentally friendly and allows obtaining products with specified properties [15–18]. There are still few studies in this direction. There are several works on the use of plant extracts, fruit juices, bee honey, *Hibiscus esculentus, Limonia acidissima*, aloe vera leaves, and ginger roots [19; 20]. Plant extracts containing a significant amount of carbohydrates, protein, fat, and vitamins perform the function of a reducing agent and are necessary for the course of the main process of ferritization (microwave process, sonochemical, self-combustion). For example, the authors considered the possibility of using hibiscus extract as a blocking agent in the synthesis of cobalt ferrites. The chemical composition of this plant includes organic and phenolic acids, such as citric, malic, amber, lactic, and malic. Flavonoids such as quercetin, luteolin, gossypetin, and their glycosides are also involved in the process. The green chemical synthesis of cobalt ferrite was discussed in [21] Sesame seeds (*Sesamum indicum L*) were used as a gelling and chelating agent. It was shown that both self-combustion and ceramic technologies lead to the formation of a cubic spinel highly crystalline structure of $CoFe_2O_4$ [22]. *Nephelium lappaceum* was used for the synthesis of cobalt ferrite [23]. The authors [24] studied the physicochemical, photocatalytic, and antibacterial properties of ferrites obtained using cheese as fuel in the combustion method. Cheese contains lactic acid and carbohydrates,

which act as powerful reducing agents and are capable of forming active forms of oxygen, which are responsible for the burning process. Further, the structural, optical, morphological and luminescent properties of the obtained particles were studied.

The use of gum of plant origin for the synthesis of transition metal ferrites [25] in the Ni–Cu–Mg system [26] and Ni-Cu-Zn [27] is effective. The use of gel is a cost-effective natural, environmentally friendly way to synthesize nanoparticles that can be used as catalysts.

A method to produce ferrites from printed circuit board sludge by a combination of acid leaching, chemical precipitation and ferritization process has been proposed as green synthesis [28]. The obtained ferrite adsorbent showed high adsorption properties in relation to phosphate ions. The use of oxalic acid to obtain oxalates with subsequent decomposition at a temperature of 75 °C to obtain magnetic ferrites $CoFe_2O_4$, $NiFe_2O_4$, $ZnFe_2O_4$ is proposed by the authors [16].

Bacteria-assisted biosynthesis preserves chemically pure biocompatible ferrite $Zn_xFe_{1-x}O_2$ (x = 0.01, 0.1 and 0.15) [29] and $ZnFe_2O_4$ with high adsorption properties [30].

That is, the use of "green synthesis" of spinel ferrites does not involve the use of high-temperature stage, typical for auto-combustion technology, microwave processing. An alternative to the conventional method of providing chemical transformation of precursors into ferrites is the use of chemical high energy.

The microwave radiation method is a new way to synthesize ferrites. The advantages of this method are speed, simplicity, short reaction time, high product yield, cost-effectiveness, selectivity, and yield compared to conventional technologies. It is hypothesized that microwave storage greatly increases the rate of chemical reactions due to "specific microwave effects" and "non-thermal microwave effects" [31]. However, the use of this ferrite synthesis technology on an industrial scale is impossible due to low product yield and cumbersome apparatus design compared to other methods, such as co-precipitation, hydrothermal, or ceramic methods. In this method, the thermal energy is internal rather than external as in the case of conventional combustion reactions, and microwave energy is used to burn the raw material to form ferrites. The production of heat is due to the transformation of microwave energy into thermal energy by absorption of electromagnetic radiation and leads to an increase in the temperature of the substance. The temperature is usually 100 to 200 °C. Some nanodisperse ferrites were synthesized by this method: Fe_3O_4, $CoFe_2O_4$, $Zn_{1-x}Mn_xFe_2O_4$, $NiFe_2O_4$ [32–35], and $ZnFe_2O_4$ [36; 37].

Under the influence of microwave radiation, solid-phase synthesis of cobalt, nickel, copper, and zinc ferrites with structural spinel based on the oxides of these metals and natural magnetite was carried out [38]. The power of the magnetron and the reaction time were varied. The optimal radiation frequency was 2.45 GHz.

Ultrasound treatment is widely used for the synthesis of oxide compounds both in liquid phase and in heterogeneous systems [39; 40.]. This method is particularly aimed at achieving uniform mixing and controlling the particle size distribution. In the process of synthesis, as a result of irradiation with ultrasonic waves, bubbles are formed in the reaction medium, and hydroxides are subjected to thermal effects due to the introduction of high energies into the reaction volume. With the help of ultrasonic exposure, it is possible to achieve such a degree of homogeneity in the system that the ferritization process occurs in the liquid phase or at low temperatures. Ferrites synthesized by this method include $MnFe_2O_4$, $NiFe_2O_4$, $Ni_xCu_yZn_zFe_2O_4$, and Fe_3O_4 [41–44].

In [45], data on the comparison of the effects of microwave exposure and ultrasound in hydrophase synthesis are presented. The process of ferritization significantly increased with the previous use of microwave irradiation, ultrasound treatment did not affect the formation of $CoFe_2O_4$. On the contrary, the authors [46] claim a significant acceleration of the formation of Mn-ferrite from the corresponding acetates under the action of US radiation at high temperatures (650, 750, and 850 °C).

There are works related to the formation of Co-Ni ferrites under the action of ultrasound at a temperature of 80 °C, doped with rare earth elements [47] Crystalline nanoparticles of spinel ferrites of the composition $Co_{0.3}Ni_{0.5}Mn_{0.2}Eu_xFe_{2-x}O_4$, where 0.00 ≤ x ≤ 0, 10 were synthesized using the sonochemical method in the pH static mode at pH = 14, (frequency 20 kHz and power 70 W), and as the Eu^{3+} content increases, the magnetic properties decrease. A series of $CoTm_xFe_{2-x}O_4$ nanoparticles (0.0 ≤ x ≤ 0.08) were synthesized by the sonochemical method at pH = 11. As the content of thulium cations increases, the saturation magnetization and final magnetization increase [48]. Cobalt,

nickel ferrites were synthesized using ultrasound treatment. Coercive force of cobalt ferrite was found to be 1200 Oersted.

The synthesis of cobalt ferrites and the influence of the conditions of treatment with ultrasound radiation on the coercive force and saturation magnetization are considered. X-ray analysis showed that the particles have different crystal lattice structure of cubic spinel structure depending on the pH of synthesis. The sequence of formation phases can be represented as follows: $(CoFe)_2O_3$>$(CoFe)_3O_4$>$CoFe_2O_4$. Measurements of the magnetic properties of the powders confirmed the stated assumption. The saturation magnetization (Ms) of the samples ranged from 1.5 to 58 Emu/g; the coercive force (Hc) was 45–450 E depending on the synthesis conditions[49].

A decreasing trend of Hc, Ms, nB, and Mr values was detected with Nd^{3+} substitution in ferrites obtained by the sonochemical method at a constant pH = 7 [50].

Spinel ferrites $Ni_{0.4}Cu_{0.2}Zn_{0.4}La_xY_xFe_{2-x}O_4$ (x = 0.00–0.10) were produced using ultrasonic irradiation of the suspension at pH=11. The saturation magnetization and residual magnetization decrease, and the coercivity slightly increases with the substitution of La^{3+} and Y^{3+} [51]

Similarly, nanoparticles of $Mn_{0.5}Zn_{0.5}Dy_xFe_{2-x}O_4$ ($x \leq 0.03$) were obtained [52]. The value of the band gap energy was in the range from 1.61 to 1.67 eV. The magnetization parameters are significantly increased due to the increase in the concentration of rare earth ions Dy^{3+}.

Processes based on technologies using various types of plasma discharges are considered promising and competitive. Among the plasma chemical discharges, contact non-equilibrium low-temperature plasma (CNP) is promising from the point of view of practical application. A plasma discharge is generated between an electrode located in the gas phase and the surface of a liquid in the volume of which the second electrode is located. Thus, chemical transformations at the boundary of the phase distribution are caused by the complex influence of electrochemical oxidation-reduction; photolysis reactions initiated by UV-irradiation; and by the flow of charged particles from the gas phase to the surface of the liquid medium. So far, the authors have established [53; 54] that by varying the composition of liquid phases it is possible to a certain extent to control the routes of chemical transformations and the composition of the obtained products.

For example, several ferrites of the MFe_2O_4 system (M = Ni, Mn, Zn, Co) were synthesized using contact low-temperature non-equilibrium plasma. The simplex method determined the effect of the mutual influence of the content of various cations on the saturation magnetization and coercive force. It was established that low values of magnetization are observed for Ni-Zn ferrites and high for the entire range of Co-Zn, Co-Ni ferrites. An increase in the content of cobalt cations leads to an increase in the coercive force in all compositions. [2; 55–58]. A comparative study of $Co_xFe_{3-x}O_4$, $Ni_xFe_{3-x}O_4$, and $Mn_xFe_{3-x}O_4$ cobalt ferrites with different compositions (x = 0.25, 0.5, 0.75, 1.0) synthesized using CNP was conducted. Compositions other than stoichiometric have a defective structure, which is confirmed by crystal-chemical calculations. As the cobalt content increases, the orientation of the spinel increases, and the number of vacancies decreases. [59; 60].

Conclusion

That is, the quality of synthesized ferrites mainly remains factors such as composition, synthesis methods, type of raw material, nature of precipitant, surfactant, nature of solvent. There is currently no unequivocal conclusion regarding the universal method of obtaining ferrites. Everything depends on the characteristic by which the technology is evaluated. Liquid-phase methods are traditionally considered more technologically feasible. For example, when comparing hydrothermal, sonochemical, and sol-gel synthesis methods, the hydrothermal process is proposed as one of the most convenient for the synthesis of ferrites due to its advantages, such as achieving the desired size, shape, monodispersity, and high degree of crystallinity. In addition, the hydrothermal method helps to reduce agglomeration, thus it is possible to synthesize parts with a narrow size distribution, phase homogeneity and controlled morphology. A variation of the hydrothermal-solvothermal method has a number of advantages and the absence of a large amount of toxic liquid waste, which ensures its widespread use. According to our findings, the matching method makes it possible to regulate the morphology and properties of magnetic nanoparticles. Particle size and magnetic properties can vary with pH, salt concentration, temperature and ionic silicone solution. The ceramic method is proposed as the most convenient in terms of possible significant

production capacity. The sol-gel method makes it possible to obtain single-phase monodisperse nanosized ferrite. On the other hand, the synthesis of monodisperse nanodisperse particles MFe_2O_4 (M = Ni, Co, Mn, Zn $Co_xFe_{3-x}O_4$) and γ-Fe_2O_3 both at the water-toluene interface and in microwave hydrothermal conditions using readily available salts (nitrates, chloride) and oleic acid as a dispersant is productive and effective.

Both methods are important for the synthesis of monodisperse nanodisperse MFe_2O_4 ferrite particles with a size distribution of 5 to 10 nm. Regarding the comparison of electrochemical, hydrothermal, co-plantation and sol-gel methods, from the point of view of convenient control of particle size, regulation of electrooxidation current density or potential in the system, preference is given to electrochemical methods. With this analysis, we can conclude that there is a consensus on a single, universal approach to synthesizing ferrite nanoparticles.

In termsof simplicity of synthesis, coprecipitation is preferred, and solvothermal synthesis is preferred to control the size and morphology of ferrite particles. On the other hand, the method of forward and reverse emulsions is better for the synthesis of monodisperse particles of different sizes.

The sonochemical method and the microwave treatment method are new methods and are finding more and more applications nowadays, due to: shorter reaction time, homogeneous reaction conditions, higher yield of the final product, high selectivity compared to other traditional methods. In general, all the synthesis methods listed above are characterized by the presence of both disadvantages and advantages for the synthesis of dispersed ferrites. Thus, the choice of synthesis method dependson the required production capacity and application. That is, it is very important not only to choose the factors affecting the synthesis (type of solvent, surface-active substance, concentration, pH, temperature, stirring speed), but also to use the optimal technology in terms of required quality and productivity.

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