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Review

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Synthesis of Transition Metal Ferrites (Co, Cu, Ni, Mn) by the Sol-Gel Method with Combustion and the Use of Microwave Processing

This review considers recent work on the synthesis of transition metal ferrites (Co, Cu, Ni, Mn) by sol-gel method with combustion, as well as the effect of microwave radiation. Ferrites are interesting not only for magnetic and optical properties, but also for their catalytic ones. In recent years, there has been an increasing amount of literature, which investigates the catalytic properties of transition metal ferrites in various reactions, including oxidative reactions. Given the fact that various organic components are used as complexing agents and as a fuel in the sol-gel method with combustion, the review considers the influence of the organic reagent nature, its ratio to precursors, the pH of the medium, the power and time of microwave exposure to the process of ferrite formation as factors influencing the size of formed particles and their textural characteristics, which are of great importance in the catalysis. Recently, the attention of chemists working in the field of catalysis has been attracted by studies of the effect of physical fields, to which the microwave field belongs, on various chemical processes, including the nanocatalysts synthesis. The use of microwave radiation in sol-gel synthesis of ferrite allows obtaining nanoferrites with high specific surface area. From this point of view, this paper considers the works of recent years devoted to the study of microwave sol-gel synthesis of ferrite.

Keywords: ferrites, sol-gel method with combustion, complexing agents, microwave technology, microwave power, dispersion, texture, specific surface area, particle size.

Content

List of abbreviations

XRD — X-ray diffraction method

FESEM — Field Emission Scanning Electron Microscopy
TEM — Transmission electron microscopy
SEM — Scanning electron microscope
EDS — Energy Dispersive X-Ray Spectroscopy
FTIR — Fourier transform infrared spectroscopy
TGA — Thermogravimetric analysis
DTA — Differential Thermal Analysis
BET — Brunauer-Emmett-Teller surface area analysis

Review Plan

Inclusion and exclusion criteria: The present review considers recent works on the synthesis of transition metal ferrites (Co, Cu, Ni, Mn), which are characterized by a wide range of applications, the sol-gel method with combustion, as well as the exposure to microwave radiation.

The review data are based on scientific publications of recent 7 years.

The review considers articles from the Scopus, Web of Science, Google Scholar databases.

The keywords used for the search are the "sol-gel method with combustion", "copper, cobalt, nickel, manganese ferrites", "microwave technology" and "texture". Statistical methods were not used in the review.

Introduction

It should be noted that ferrite materials, which have special magnetic properties and combine high magnetization with the characteristics of semiconductors and dielectrics, are widely used in magnetic, electronic, and microwave devices [1–7]. In addition to their promising magnetic properties, ferrites have catalytic properties [1, 8–12]. Oxides, salts and hydroxides of appropriate metals are usually starting materials for producing ferrites. There are various ways to obtain ferrites, namely co-precipitation, thermal decomposition of salts, sol-gel method, ceramic, burning solutions in a high-temperature stream, plasma-chemical. To date, the sol-gel method has become widespread among the methods for obtaining highly dispersed materials, including nanoferrites. The main advantage of this method is the high homogenization of precursors with the production of a sol and its transformation into a gel due to hydrolysis and condensation processes, followed by aging, drying and heat treatment.

The variation of the sol-gel method is the sol-gel with self-combustion. The process of drying and heat treatment in this method takes place in one stage. The method includes an exothermic and self-sustaining redox reaction of xerogel, which is obtained from an aqueous solution containing metal salts (oxidizing agent) and an organic component (reducing agent) also known as a "fuel". The organic component forms complexes with metal ions that prevents precipitation of metal salts and thereby improves gelation conditions. In addition to these advantages, owing to the results of the organic component combustion, a large amount of gaseous products is formed, which prevents the solid phase crystallites from sintering. The latter is obtained in the form of ash or a fine powder. The reaction proceeds quickly and at a sufficiently low temperature. The method is quite simple enough for practical implementation and economical in terms of time and energy consumption. Solutions can also be subjected to combustion, the so-called solution combustion.

In recent years, other than the thermal exposure, various types of radiation have also been used in the synthesis of compounds, in particular microwave [13–16]. Microwave technology has also found application in the synthesis of ferrites by the sol-gel method with combustion. Microwave technology is used in the synthesis of ferrites by the sol-gel method with combustion at the stage of drying and combustion. The literature data analysis shows that it is also possible to synthesize nanosized ferrites through the use of microwave radiation in the sol-gel method with combustion [17–22]. The brief review of recent works presents data on the synthesis of a number of ferrites (Co, Ni, Cu, Mn) by the sol-gel method with combustion, as well as using microwave treatment as an initiator of the combustion reaction, i.e. carrying out the combustion stage in the microwave field. The ferrites of these metals were chosen from the perspective of their use as catalysts. In recent years, more and more works have appeared in the literature in which the catalytic properties of transition metal ferrites in various reactions, including oxidative ones, are studied. For example, these are following reviews [8–10**]**. Previously, we carried out the microwave synthesis of these ferrites by the ceramic method from oxides [15]. The catalytic properties of copper ferrite synthesized by the ceramic and sol-gel method using microwave treatment were investigated by us in the oxidative conversion of carbon monoxide [23]. So this review focuses on the following issues:

Given the fact that various organic components are used as complexing agents and a fuel in the combustion sol-gel method, an emphasis is laid on the influence of the organic reagent nature on the formation of ferrites during combustion, as a factor affecting the size of the resulting ferrite particles and their textural indicators, which is of great importance in catalysis.

In microwave technology, the given parameters are power and time. It is important to find out how the time of microwave treatment affects the fineness of the formed ferrite particles when adjusted for the uncontrolled rise in temperature, using microwave energy in the sol-gel method with combustion.

1. Synthesis of ferrites by the sol-gel combustion method using citric acid as an organic reagent

An analysis of the literature data showed that the largest number of studies on the synthesis of both individual and mixed ferrites was carried out by the "citrate" method, i.e. using citric acid as a complexing agent and "fuel". That is why it became necessary to summarize them in a separate section.

The process essence is the reaction of hydrolysis and condensation of precursors, which are metal nitrates and citric acid, resulting in the formation of gel, upon further drying of which it passes into xerogel, followed by its spontaneous combustion and the formation of ferrite during combustion.

The synthesis procedure is as follows: a stoichiometric amount of metal nitrates is dissolved in a small amount of deionized water with stirring and a temperature of 80–90 °C. The pH of the medium is adjusted to 7 with an ammonia solution and the solution is slowly evaporated until a viscous gel is formed which self-ignites and burns to form a loose bulk powder.

$$3Me(NO_3)_2 + 6Fe(NO_3)_3 + 2C_6H_8O_7 = 3MeFe_2O_4 + 12N_2 + 12CO_2 + 8H_2O$$

There were also considered works in which the ferrites synthesis was carried out by burning the solution.

1.1. Synthesis of individual ferrites

Although the ferrites formation at lower temperatures is one of the important advantages of the combustion sol-gel method, in many studies, the powders obtained after gel combustion are additionally annealed at higher temperatures. These are mainly works in which the magnetic properties of ferrites are investigated and where purity and high crystallinity are of great importance.

The synthesis of nanocrystalline cobalt ferrite was carried out in the works [24–26]. The powders obtained after burning the gel were additionally annealed at various temperatures from 600 °C to 900 °C for 2 h in air [24]. The particle size of the obtained ferrites strongly depends on the calcination temperature. At a temperature of 700 °C, the size of cobalt ferrite crystallites is 25–30 nm, they look like quasi-spheres. The crystallite sizes of the sample annealed at 900 °C are in the range of 40–50 nm, which is consistent with the results of X-ray diffraction (XRD) method. The authors suggest that the agglomeration increases as the annealing temperature increases, and some degree of agglomeration is unavoidable at higher annealing temperatures. The influence of the additional calcination temperature at 600, 700, and 800 °C on the structural and magnetic properties of $CoFe_2O_4$ was also considered in [25]. The authors found that as the calcination temperature increased, clearly defined sharp peaks were observed on the X-ray pattern in line with $CoFe_2O_4$ to indicate an increase in the ferrite crystallinity. Very pure and crystalline cobalt nanoferrite with a uniform size distribution was also obtained in [26].

Cobalt and copper ferrites obtained by the citrate method were characterized by X-ray diffraction method, Field Emission Scanning Electron Microscopy (FESEM), Transmission electron microscopy (TEM), Energy Dispersive X-Ray Spectroscopy) [6]. The average size of cobalt and copper nanoferrite crystallites was 24.7 and 37.7 nm, respectively.

The synthesis of nickel ferrite by the sol-gel method with combustion in the presence of citric acid was described in the works [27–31]. In the work [27], a mixed solution of nitrates (Ni and Fe) and citric acid was kept in an oven at 120 °C for 12 hours. The dried sample was ground and then annealed in a muffle furnace at 600 °C for 2 hours and slowly cooled to room temperature. X-ray diffraction analysis confirmed the formation of a cubic structure of nickel ferrite spinel. The average crystallite size was determined using the Debye-Scherrer formula and it was 21 and 60 nm, respectively, for the obtained samples before and after annealing. SEM analysis showed that the particles were spherical.

The authors [28] obtained nanoparticles of nickel ferrite with an average size of 23 nm calculated by the Scherrer formula.

In the work [30], the resulting powder after auto-combustion of the dried gel was also calcined at 550 °C for 4 hours for better crystallinity and purity. The average crystallite size of the prepared nickel ferrite sample was 22 nm.

In the work [29], the authors revealed that the resulting nickel ferrite particles had an approximately spherical shape and a crystal size in the range of 45–55 nm. The average grain size obtained from the TEM image of the sample was approximately 60 nm, which was in appropriate with the size determined from the X-ray diffraction patterns.

Nanosized spinel ferrites MFe_2O_4 (M = Ni, Co and Zn) were obtained in [31]. Gel ignition was observed at 623 K (350 °C). The resulting powder was sintered in two stages (at 500 °C/5 h and at 700 °C/5 h). The crystallite size (nm) for each sample was calculated from the broadening of the line of the most intense (311) diffraction peak using the Debye-Scherrer formula. It was found that sizes of crystallites depended on the nature of the M^{2+} cation. The lowest nanoscale crystallite value was found for nickel ferrite (30.6 nm), followed by zinc (34.4 nm) and cobalt (36.7 nm) ferrite.

Nickel ferrite was synthesized using various ratios of fuel (citric acid) to metal nitrate 1:1, 1:2, 1:3 and 1:4 [32]. All samples were characterized by Thermogravimetric (TG) and Differential Thermal (DT) analyzes. TG-DTA analysis confirmed the ferritization temperature, which varied slightly with the ratio of fuel to metal nitrate. All samples were sintered at an average temperature of 560 °C for 4 hours. X-ray diffraction analysis confirmed the cubic structure of the samples.

The authors [33–34] obtained copper and manganese ferrite particles with good composition uniformity and high phase purity.

1.2. Synthesis of substituted ferrites

When adjusted for the fact that mixed ferrites which are solid solutions, have the best magnetic characteristics, a promising method for their preparation is the sol-gel method with combustion. This is evidenced by quite a lot of works in the scientific literature dealing with the production of substituted ferrites by this method.

Consideration of these works is also important from the point of view of improving the catalytic properties of ferrites, in which atoms of other elements are introduced in a certain quantitative ratio.

A series of nanocrystalline copper-substituted cobalt-zinc ferrites $Co_{0.6}Zn_{0.4}Cu_xFe_{2x}O_4$ (x = 0.2, 0.4, 0.6, 0.8, and 1.0) with a cubic spinel crystal structure were synthesized by gel auto-combustion [35]. All samples after gel combustion were annealed at 400 °C, 600 °C, 800 °C and 1000 °C. X-ray diffraction analysis confirmed the formation of a cubic phase with space group *Fd*-3*m* for all nanoferrite obtained. The particles were 25 nm in size and spherical in shape. In this work, the same particle size of ferrite samples processed at different temperatures raises some doubts. High temperatures of heat treatment should undoubtedly lead to particle aggregation, which is noted in most works.

In the work [36], cobalt ferrite nanoparticles with substituted transition metals ($CoM_xFe_{2-x}O_4$, M = Cr^{3+}, Ni^{2+}, Cu^{2+} and Zn^{2+}, x = 0.2; 0.4; 0.6; 0.8 and 1.0) were synthesized. The resulting ferrite powders were annealed at 400 °C, 600 °C, 800 °C and 1000 °C for 2 hours. TEM images of $CoNi_{0.4}Fe_{1.6}O_4$ annealed at 400 °C and 1000 °C showed that ferrite particles had nano-sizes of 20 nm to 60 nm, respectively, and spherical morphology.

The authors [37] obtained nanoparticles of ferrites $Co_{1-x}Ni_xFe_2O_4$ (x = 0.02, 0.04, and 0.06 M) with a spherical shape and a particle size in the range of 26.31–31.13 nm.

Ruthenium-doped cobalt ferrite nanoparticles ($CoRu_xFe_{2-x}O_4$; x = 0.0, 0.02, 0.06) were obtained in [38]. The average size of ferrite crystallites was 32.12 nm for $CoFe_2O_4$, 17.77 nm for $CoRu_{0.02}Fe_{1.98}O_4$, and 18.45 nm for $CoRu_{0.06}Fe_{1.94}O_4$.

The authors [39] synthesized polycrystalline nickel-substituted cobalt ferrites ($Co_{1-x}Ni_xFe_2O_4$, where x = 0, 0.25, 0.50, 0.75, 1). From TEM images of samples after heat treatment at 700 °C for 8 h, it was found that the nanoparticles were uniform in size, which corresponded to the average size obtained from peak broadening in X-ray diffraction analysis. The effective diameter of ferrite powder particles was 20–25 nm.

Polycrystalline Ni-Zn ferrites with the chemical formula $Ni_{0.5}Zn_{0.5}Zr_xCu_xFe_{2-2x}O_4$, with x values from 0.0 to 0.4 and 0.08 wt.%, were prepared by the sol-gel method with auto-combustion with the addition of ethylene glycol to citric acid as an additional combustible agent (Table 1) [40].

It was determined that the size of crystals of complex ferrites $Ni_{0.5}Co_{0.5}Nd_xFe_{2-x}O_4$, obtained in the work was 41, 37 and 35 nm for x = 0.025; 0.100 and 0.125, respectively [41].

Polycrystalline ferrites with chemical formulas $Ni_{0.5}Zn_{0.5}Fe_2O_4$, $Ni_{0.5}Zn_{0.5}Er_{0.025}Fe_{1.975}O_4$, $Ni_{0.5}Zn_{0.5}Er_{0.035}Fe_{1.965}O_4$, and $Ni_{0.5}Zn_{0.5}Er_{0.05}Fe_{1.95}O_4$ were obtained in [42]. The authors described in detail all ferrite synthesis process noting that it took less than 60 minutes from heating the sol to gelling it. And the time between the actual ignition and the end of the reaction was less than 5 seconds. An emphasis was laid

on the opportunity of using the combustion sol-gel method as a simple and affordable method for the synthesis of complex compounds.

Table 1

The sizes of the crystallite, particle and grain of Zr and Co substituted $Ni_{0.5}Zn_{0.5}Zr_xCo_xFe_{2-2x}O_4$

No.	Dopant concentration	Crystallite size, D (nm)	Particle size, (nm)	Grain size, (μm)
1	0.0	9.1121	15.5051	2.3137
2	0.08	10.9423	21.7809	3.043
3	0.16	10.2774	15.364	2.7436
4	0.24	10.1211	9.6893	2.6048
5	0.32	10.1474	4.1924	2.4618
6	0.4	10.3397	19.0597	2.2952

The average particle size of nickel–chromium ferrites with the general formula $NiCr_xFe_{2-x}O_4$ (x = 0.0; 0.1; 0.2; 0.3; 0.4 and 0.5) was calculated using the Scherrer formula from the data on the broadening of diffraction peaks and amounted to 23–43 nm [43].

The authors [44] developed a method for obtaining solid solutions based on nickel-zinc ferrite of the general composition $Ni_{0.75}Zn_{0.25}Fe_{2-x}La_xO_4$ (Ln = Nd, Gd, Lu, Yb). Citric acid was taken in a ratio of 1:1 for trivalent elements and 3:2 for divalent ones. A solution of nitrates and citric acid was evaporated to a jelly-like state at a temperature of 700 °C for 1 hour. Then the temperature was raised to the start of a self-developing process (T = 100 °C). The resulting powder was annealed at 700 °C until the remains of the organic phase were removed. The resulting reaction products were loose agglomerates consisting of nanoparticles. At an annealing temperature of samples up to 900 °C, coarsening of agglomerates and sintering of particles were observed. The average crystallite size for all synthesized compounds was 80–90 nm.

Samples of nickel-aluminum ferrites of the chemical composition $NiFe_{2-x}Al_xO_4$ were obtained in [45]. Drying of the xerogel was carried out in air at a temperature of 120–130 °C. Upon complete drying, spontaneous combustion of the porous xerogel occurred. The average particle size found from X-ray diffraction studies was 20–60 nm.

2. Synthesis of ferrites using various organic reagents

In addition to citric acid, the sol-gel combustion method uses such organic compounds that are easily oxidized and do not contaminate the resulting product, namely glycine, urea, ethylene glycol, polyvinyl alcohol.

In the work [46] nanoparticles of cobalt ferrite $CoFe_2O_4$ with a spinel structure were synthesized by the sol-gel method with auto-combustion using three different types of fuel (ethylene glycol, glycine and urea). The formation of a pure phase of cobalt ferrite with a cubic spinel structure was observed in X-ray diffraction patterns for all samples. The average crystallite size, lattice parameters and other structural parameters were calculated from XRD data. The experimental results showed that the average crystallite size of the prepared samples ranged from 15 to 22 nm. A lower average particle size of 15 nm was noted with urea as a fuel. The average grain size turned out to be in the range of 65–86 nm.

The influence of the ratio of fuel (glycine) to metal nitrates was considered in the work [47] when obtaining cobalt ferrite by the sol-gel method with combustion. The particle size of pure phase cobalt ferrite nanoparticles was found to be < 40 nm.

The authors of [48] synthesized cobalt ferrite ($CoFe_2O_4$) nanoparticles using several different methods: combustion, co-precipitation and precipitation. The average particle size obtained by combustion with glycine was 69.5 nm, by co-precipitation it was 49.5 nm and by precipitation it was 34.7 nm.

Ethylene glycol as a complexing agent and fuel was used to obtain copper ferrite in the work [49].

Magnesium-cobalt spinels ($Co_xMg_{1-x}Fe_2O_4$: x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) were synthesized by the combustion sol-gel method using urea [50] as a fuel. The results showed that the final products were a cubic spinel phase with spherical nanoparticle morphology.

In our work [51], the copper ferrite was obtained using various organic reagents. It revealed that the average size of ferrite crystallites obtained using urea was in the range of 15–19 nm, with citric acid — 20–23 nm and with glycine — 29–32 nm. Dynamic light scattering (DLS) measurements of isopropyl dispersions of ferrite samples synthesized using citric acid, glycine and urea were also carried out. Before DLS

measurements, the dispersions with the studied samples were subjected to ultrasonic (US) treatment. Ultrasound treatment was carried out for 10, 20 and 60 min. The results showed that the spectral pattern significantly depended on the processing time of the dispersions. The average particle size of the synthesized samples in isopropyl alcohol dispersions subjected to ultrasonic treatment for 20 minutes ranged from 381 to 447 nm. Large values of the distribution width indicated that the dispersion contained aggregates of particles of different sizes. These values were much larger than the average crystallite size determined by X-ray diffractometry. Most likely, the studied dispersions contained aggregates that were not destroyed by ultrasonic treatment. After 60 minutes of sonication, the particle size decreased and fluctuated in the range of 123–153 nm.

The racemic mixture of the right (*d*) and left (*l*) forms of alpha-alanine was used in the work [52] for the synthesis of highly crystalline cobalt nanoferrite. The synthesized samples were annealed at two different temperatures, namely 500 °C and 800 °C for 2 hours to study the effect of temperature on crystallite size, phase purity and thermal stability. The formation of single-phase spinel nanoparticles was observed both in the initial state and in the annealed state. The size of cobalt ferrite nanoparticles after gel combustion was 37.8 nm, after sample annealing at 500 and 800 °C, 38.8 and 43.7 nm, respectively.

The effect of the ratio of glycine to oxidizing agent (metal nitrates) on the structural, morphological, and magnetic properties of $Co_{0.8}Mg_{0.2}Fe_2O_4$ was studied in [53]. Samples were prepared with stoichiometric fuel content to nitrates (G/N=1.48), lean fuel content (G/N=0.74) and high fuel content (G/N=2.22). After ignition at 180 °C and auto-combustion, the resulting powders were annealed at 600 °C for 2 hours. It was found that the crystallite size and the crystallinity of the spinel phase increased as G/N ratio increased (Table 2).

Table 2

Effect of heat treatment and fuel-to-oxidizer ratio (G/N) on the crystallite size nanoparticles (*a*) auto-combusted, (*b*) annealed at 600 °C

No.	Fuel	Crystallite size, D (nm)	
1	2.22 (fuel rich)	50 (a)	62 (b)
2	1.48 (stoichiometry)	46 (a)	54 (b)
3	0.74 (fuel lean)	43 (a)	49 (b)

In the work [54] for the synthesis of $Y_{0.2}CoFe_{1.8}O_4$, citric acid and polyvinyl alcohol were used as a fuel. The average crystal size of the synthesized ferrite was in the range of 20–70 nm.

Ferrospinels of $CuFe_2O_4$, $MgFe_2O_4$, $Ni_{0.5}Co_{0.5}Fe_2O_4$ composition were obtained using polyvinyl alcohol [55] as a fuel. Manganese ferrite was also obtained by the sol-gel method with the participation of polyvinyl alcohol [56].

As noted above, when synthesizing the compounds by the sol-gel combustion method, it is also possible to combust the solution. Therefore, works devoted to the production of ferrites by the solution combustion method are of interest [57–59].

Nanoparticles $CoFe_2O_4$, $CoFe_{1.95}Bi_{0.0\,5}O_4$, $CoFe_{1.9}Bi_{0.1}O_4$ and $Cu_{0.5}Co_{0.5}Fe_{1.9}Bi_{0.1}O_4$ were prepared by burning a solution using glycine as a fuel [57]. Aqueous solutions of the calculated amounts of precursors and fuel, after stirring on a magnetic stirrer, were placed in an oven at 500 °C for 5 minutes.

Nanoparticles $Co_{1-y}Cu_yFe_{2-x}Ce_xO_4$ ($x = 0$, $y = 0$), ($x = 0.05$, $y = 0$), ($x = 0$, $y = 0.5$) and ($x = 0.05$, $y = 0.5$) were also synthesized by burning a solution of metal nitrates and glycine in a preheated furnace at 350 °C for 5–7 min, after which they were sintered at 700 and 900 °C for 2 h [58].

Especially, there is a need to note the work [59], which studied the influence of the "fuel" concentration on the composition, structure and size of crystallites in the synthesis of cobalt ferrite by solution combustion. Glycine was used as a fuel, cobalt and iron nitrates were used as precursors. The solution of precursors and glycine was placed in an electric oven (400 °C) until complete combustion (approximately 15 minutes) was formed. Three syntheses were carried out with different fuel concentrations, namely low, stoichiometric and high ones. The authors concluded that higher concentrations of glycine led to the formation of secondary phases of cobalt oxide. The sample with low fuel concentration was the only one in which a pure nanosized $CoFe_2O_4$ phase was formed. Synthesis with an increased concentration of fuel led to rapid ignition and intense combustion during the reaction. With an excess of fuel, oxygen from the atmosphere was required to complete the reaction. This excess of oxygen could cause the formation of secondary oxide phases in addi-

tion to the primary cobalt ferrite phase. Sizes of crystallites were calculated using the Scherrer equation for various concentrations of glycine: 23.58 nm for low (smallest size among all samples), 31.14 nm for stoichiometric and 33.16 nm for high concentrations.

Thus, by varying the concentration of "fuel", it is possible to attain both a pure ferrite phase and complex systems consisting of ferrite and oxide phases. The second variant is most interesting for the heterogeneous catalysis. During the combustion of both the sol and the gel, as a result of an exothermic reaction, various physicochemical transformations occur, namely melting, chemical reaction, diffusion, which affect the formation of the composition and structure and result in various defects. At present, the phenomenon of growth in the catalytic and adsorption activity of solids with a defective surface in comparison with the structure of a perfect crystal is considered an established fact [60]. Therefore, using various organic reagents and their concentrations in the combustion sol-gel method, it is possible to obtain not only highly crystalline ferrites, but also solid solutions of complex composition and defect structure, which are promising materials for the heterogeneous catalysis.

3. Synthesis of ferrites by the sol-gel method with combustion in "green chemistry".

In recent years, works have appeared on the synthesis of ferrites by the sol-gel method with combustion, which belong to "green chemistry". According to these works, extracts of various plants are used as an organic reagent and "fuel". An example is the work [61], in which photocatalysts based on magnesium-doped nickel ferrite were synthesized by the sol-gel method using an extract from aloe *A.Vera* as a chelating agent, reducing agent and natural template. Stoichiometric amounts of nickel nitrate and iron nitrate were mixed with M^{3+}/M^{2+} in a molar ratio of 2:1, then a solution containing *A.Vera* extract and the calculated amount of magnesium nitrate was added dropwise. The mixture was continuously stirred for 1 hour at 80 °C until a gel formed. The resulting viscous gel was again heated to dry in an autoclave at 200 °C until auto-ignition started. Finally, the resulting $NiFe_2O_4$:Mg^{2+} (1 mol.%) nanoparticles were calcined at 350 °C for 1 h.

Cottage cheese was used as a fuel for the synthesis of cobalt ferrite with different contents of Zn^{2+} and the general formula [$Zn_xCo_{1-x}Fe_2O_4$ (x = 0.0, 0.2, 0.4 and 0.6) the ZCF] [62]. Solutions of metal nitrates and cottage cheese were continuously stirred for 30 min until a homogeneous mixture was obtained. The mixture was placed for 5 minutes in a muffle furnace heated to 500 °C. The resulting powder was calcined at 650 °C for about 5 hours.

A comparison of 2 methods for obtaining nickel ferrite nanoparticles, namely using citric acid and clove extract as a fuel was carried out in the work [63]. The structural properties of the resulting nickel ferrites were characterized by X-ray diffraction analysis, which revealed the cubic structure of the spinel. The average crystallite size was 24 nm and 26 nm for nanoparticles $NiFe_2O_4$ obtained respectively through the use of clove extract and citric acid.

The use of environmentally friendly, non-toxic natural materials in the sol-gel method with combustion expands the opportunity of using it for the synthesis of complex compounds, including ferrites.

From the analysis of the literature data presented above on the synthesis of transition metal ferrites by the sol-gel method with combustion, the following conclusions can be drawn:

– Regardless of the complexing agent nature, the formation of a cubic spinel structure is observed in the X-ray diffraction patterns for all samples;
– Method makes it possible to obtain ferrite nano-powders with a crystallite size in the range of 20–60 nm. Moreover, the average crystallite size of particles increases with an increase in the calcination temperature of the nanopowders obtained after combustion.
– Use of higher (superstoichiometric) concentrations of the complexing agent makes it possible to form the secondary phases.
– Ferritization temperature varies slightly with the ratio of fuel to metal nitrate.

In most of the considered works, the structure and magnetic properties of the synthesized nanoferrites are mainly studied, which are of interest to physicists, since ferrites are used mainly as magnetic materials in radio engineering, electronics, automation and computer technology. Substances in the nanocrystalline state exhibit special magnetic and optical properties, which are not characteristic of bulk materials.

The determination only of nanosizes of ferrites can be sufficient when studying their magnetic properties; however, ferrites are also used in catalysis, where textural indicators play an important role, namely, specific surface area, pore volume, and pore radius. Therefore, the study of these parameters is extremely important in the study of the catalysts activity. This caused the need to highlight the works in which the texture characteristics of ferrites synthesized by the sol-gel method with combustion were studied, as well as

their dependence on the type of organic reagent, on the ratio of fuel to metals, since during the combustion of various types of "fuel", different amounts of gases were released, which actually "loosen" the resulting mass of ferrite that also affected the texture of ferrites.

4. Textural parameters of ferrites synthesized by the sol-gel method with combustion

In the work [64], Ni, Co, Mn, Mg, and Zn ferrites were synthesized by the gel combustion method with the participation of glycine. The sizes of the synthesized ferrite nanoparticles were determined, as well as their specific surface area (Table 3).

Table 3

S_{BET} — specific surface area, average crystallite size, D

No.	Sample	S, m^2/g	D, nm
1	$NiFe_2O_4$	72.0	20.5
2	$CoFe_2O_4$	15.4	20.4
3	$MnFe_2O_4$	54.4	5.8

Nickel ferrite had the largest specific surface area (72 m^2/g) among Ni, Co, Mn ferrites obtained using glycine as an organic reagent, followed by manganese ferrite with a specific surface area of 54.4 m^2/g and cobalt 15.4 m^2/g.

Metal-substituted cobalt ferrite nanoparticles with the composition $M_xCo_{1-x}Fe_2O_4$ (M = Zn, Cu, Mn; x = 0.0; 0.25; 0.5 and 0.75)] were synthesized by the citrate sol-gel method in [65]. The specific surface of the samples was in the range from 37.99 to 107.05 m^2/g, $Zn_{0.5}Co_{0.5}Fe_2O_4$ nanoparticles had an average pore radius of 1.84 nm and a pore volume of 0.136 ml/g.

In the work [66], ferrites of the composition $Cu_{0.5}Ni_{0.5}Fe_2O_4$ were prepared using various ratios between citric acid and metal ions. A number of secondary phases (Cu, Cu-Ni alloy and hematite) were found in the obtained samples. An optimal ratio of citric acid to the sum of metal ions was experimentally established, at which the resulting ferrite samples were characterized by the largest surface area. The sample synthesized at the maximum ratio of citric acid to metal ions (3:1) was characterized by the smallest size of ferrite crystallites (9.65 nm) and a larger surface area (92 m^2/g).

The specific surface area of Ni-Zn-ferrite obtained by various "fuels" (citric acid, carbohydrazide and glycine) ranged from 12–41 m^2/g [67].

Nickel ferrite powders of the composition $NiFe_2O_4$ with a crystal size of 18.00 nm and a high specific surface area of 55.21 m^2/g were obtained by a combustion reaction using urea as a fuel [68].

The effect of pH and metal concentration on the textural characteristics of the formed cobalt ferrite nanoparticles was considered in [69]. Xerogels obtained from solutions with different pH values (≤1, 3, 7, and 10) of precursors showed different combustion behavior. Except for pH (≤1), all xerogels quickly ignited with the formation of a large amount of gases: combustion began in the hottest zones of the crucible and spread from bottom to top, as in a volcanic eruption.

The reaction was over in 10–30 seconds with the formation of a dark gray three-dimensional structure resembling a branched tree. Table 4 lists the synthesis conditions, nanoparticle sizes and specific surface area of the cobalt ferrite synthesized.

Table 4

Preparation parameters, BET surface area, XRD and BET average particle sizes of the sample

No.	Sample	Preparation parameters		BET surface area (m^2/g)	XRD average particle size (nm)	BET average particle size (nm)
		pH value	Metal concentration (mol L^{-1})			
1	A	7	0.1	38.6	24	29
2	B	7	0.2	37.9	26	30
3	C	7	0.3	31.2	28	36
4	D	<1	0.1	6.2	16	185
5	E	3	0.1	19.0	21	60
6	F	10	0.1	35.8	30	32

As can be seen from the Table 4, the specific surface area of nanoparticles formed from acidic solutions (samples D and E) was less than that of particles formed from neutral and alkaline solutions; they were also different in particle size.

Spinels $MeFe_2O_4$, in which the divalent cations Cu, Ni, Zn and Cd were synthesized by the sol-gel method with the participation of a 10 % solution of polyvinyl alcohol with a molecular weight of 145000 as a "fuel", were obtained [70]. The size of the obtained nanoparticles was 33–52 nm. The texture parameters are presented in Table 5 below.

Table 5

Powder characteristics of $MeFe_2O_4$ ferrites

Sample	Average agglomerat, size (nm)	Specific surface area, S_{BET} (m^2/g)	Pore volume, cc/g
$CuFe_2O_4$	550	1.48	0.002
$NiFe_2O_4$	500	3.86	0.0059
$CoFe_2O_4$	350	3.26	0.0048

In the work [71] there was established the specific surface area of ferrites synthesized by the sol-gel method with auto-combustion using various organic reagents (Table 6).

Table 6

Specific surface area of ferrites synthesized by the sol-gel method with various organic reagents

No.	Ferrite	Specific surface area, m^2/g		
		citric acid	glycine	urea
1	$CuFe_2O_4$	18	4	25.5
2	$CoFe_2O_4$	12	11.7	35.3
3	$NiFe_2O_4$	6.2	7.8	34.3
4	$MnFe_2O_4$	36.5	15	32.9

As can be seen from Table 6, the ferrites obtained by the sol-gel method during the combustion with urea have the largest specific surface and the smallest — with glycine. When burning with citric acid, the final product is more voluminous and branched; the process does not last long. With glycine, the combustion occurs in the form of a flash and rapid combustion. With urea, the process takes longer with the release of a large amount of gaseous substances.

The above few works on the determination of the specific surface area of transition metal ferrites obtained by the sol-gel method with combustion involving various organic components make it possible to conclude that the nature of the organic reagent affects the specific surface area of the synthesized ferrites. It varies from a few square meters per gram to tens of square meters per gram. The different nature of combustion leads to the formation of different surface morphology and texture, which ultimately can affect the catalytic activity.

5. Synthesis of ferrites by the sol-gel method with microwave combustion

During the synthesis of ferrites by the sol-gel method with combustion, microwave technology, as noted above, is used at the stage of drying and combustion. In a microwave oven, the parameters to be set are power and time. These parameters, along with the nature of the organic reagent, determine the surface morphology, texture, and fineness of the resulting ferrites.

In [72], the synthesis of cobalt ferrite doped with nickel was carried out using the method of microwave combustion of a solution using metal nitrates and urea as a reducing agent. Metal salts were dissolved in water; urea was added and homogenized on a magnetic stirrer. The combustion reaction was carried out in a domestic microwave oven at its maximum value for 5 minutes. The solution first boiled, then it dehydrated, then decomposed with the release of a large amount of gas and then spontaneous combustion occurred with the release of a large amount of heat and the formation of a free-flowing powder.

Cobalt ferrite was obtained by burning solutions of metal nitrates (precursors) and glycine and ammonium nitrate as organic promoters in a household microwave oven at a power of 900 W for 30 min [73].

Nickel ferrite nanoparticles were obtained by microwave combustion of a solution containing nickel, iron nitrates and trisodium citrate ($Na_3C_6H_5O_7$) and finally, the sodium salt of citric acid [74]. The mixture

was placed in a microwave oven and irradiated for 30 minutes. The resulting mass was then ground into powder.

Nickel-magnesium ferrites were obtained with the participation of urea as a "fuel" and microwave treatment for 10 minutes at a magnetron power of 850 W [75].

Nickel ferrite was prepared by the sol-gel method from solutions of nickel and iron nitrate with the addition of citric acid and ammonia, evaporation at 100^0C, followed by ignition and the formation of a free-flowing powder, which was further subjected to microwave treatment [76].

In [77], the effect of an organic reagent on the synthesis of $NiFe_2O_4$ nanoparticles by combustion using microwave radiation was studied; urea, glycine, and citric acid were used as fuel reagents. The synthesized nanoparticles were characterized by X-ray diffraction, scanning electron microscopy, Brunauer-Emmett-Teller (BET) surface area. The authors found that the type of fuel affects the surface properties of nanoparticles. According to the results of X-ray diffraction analysis, the highest crystallinity was observed in nanoparticles synthesized with glycine, while nanoparticles prepared with urea had the highest surface area. The SEM micrographs showed that all nanoparticles had nanocrystalline behavior and the particles were cubic in shape. In the same work, nickel ferrite was also obtained by the "dry" method as a result of an exothermic reaction of a mixture of metal nitrates and urea. This method did not use water or any other solvent. Reagents, namely nitrates of nickel and iron, as well as urea were mixed in certain stoichiometric ratios. Owing to the crystallization water of metal nitrates, a thick mixture was formed. This mixture was placed in a laboratory type microwave oven at a maximum power of 800 W for 10 minutes. When the mixture reached the point of spontaneous combustion, it began to burn, releasing gas and heat, and the sample instantly became solid.

In [78], nickel-cobalt ferrite $Ni_xCo_xFe_2O_4$ was obtained by mixing solutions of nitrates and urea, heating to 100 °C, and microwave treatment for 10 minutes until combustion.

Flash synthesis of $NiFe_2O_4$ nanoparticles with a particle size close to 4–5 nm and a high specific surface area (about 240 m^2/g) was carried out using a RAMO autoclave microwave heater developed by the authors [79].

The microwave combustion method was used to synthesize nanocrystalline $Zn_xNi_{1-x}Fe_2O_4$ from a stoichiometric mixture of the appropriate metal nitrates and urea powders [80]. The resulting ferrite had a high-purity spinel structure with a calculated crystallite size of ~20 nm.

The paper [81] reported on the synthesis of $Zn_{0.7}Ni_{0.3}Fe_2O_4$ nanoparticles using a microwave combustion method using urea as a fuel. X-ray diffraction and FT-IR analyzes confirmed the composition and structure of the spinel ferrite. The crystallite size was estimated using X-ray diffraction (16.4 nm). The morphological study of the products was carried out using TEM, which revealed the presence of spherical, spheroidal and polygonal crystallite shapes.

In [82], the authors synthesized a rhombohedral nanostructure of nickel ferrite by the method of rapid combustion with the help of microwave radiation using ethylenediaminetetraacetic acid as a chelating agent.

Spinel nanoparticles $Zn_{1-x}Co_xFe_2O_4$ with different particle sizes were prepared by microwave combustion with urea as a fuel [83]. Composites were prepared with the addition of cobalt in various molar ratios (x = 0.0–0.5) to $ZnFe_2O_4$. The obtained spinel ferrites were characterized by powder X-ray diffraction (XRD) and the average grain size and morphology were determined by high resolution scanning electron microscopy (HR-SEM). The formation of a single cubic phase of spinel was confirmed by X-ray diffraction and Rietveld analysis with an average crystallite size in the range of 43–49 nm.

Nanoparticles of copper ferrite $CuFe_2O_4$ were obtained by microwave combustion using an extract of the plant Hibiscus rosa sinensis (Chinese rose) as a fuel. X-ray diffraction and analysis by the Rietveld method confirmed the formation of a single cubic phase with a crystallite size of 25 to 62 nm due to grain growth after calcination [84].

Copper ferrite nanoparticles were prepared using starch as a fuel. The starch solution was poured into solutions of copper and iron nitrates with continuous stirring. The clear solution was placed in a household microwave oven (2.45 GHz, 950 W) for 20 minutes. After the solution reached the point of spontaneous combustion, it instantly evaporated and became solid. The obtained solids were well washed with ethanol, dried and were designated as nanostructures of $CuFe_2O_4$ obtained by microwave technology [85].

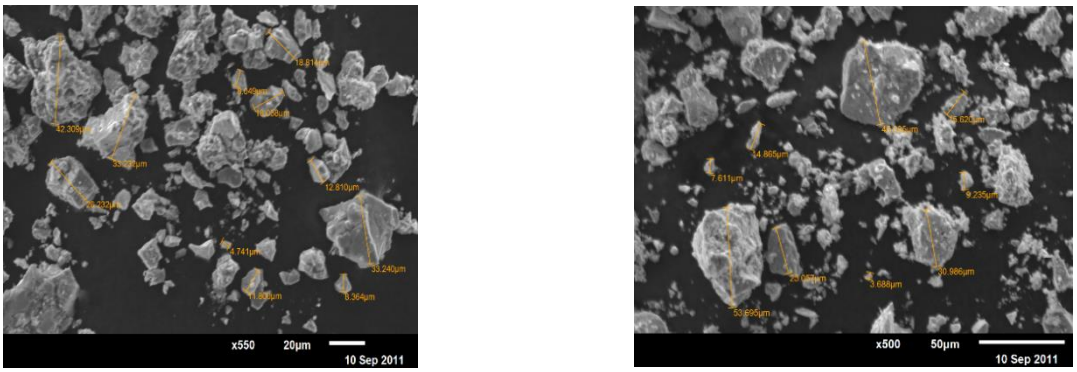
Copper ferrites were synthesized by us on the basis of the ceramic method from oxides and by the sol-gel method using microwave treatment, following which the values of the specific surface area of the samples were determined (Table 7) [15, 23]. The specific surface area was also determined after additional microwave treatment of the ferrite powder obtained after burning the gel.

Table 7

Values of the specific surface area of copper ferrite obtained by various methods

No.	Method for synthesis of copper ferrite $CuFe_2O_4$	Specific surface area, m^2/g
1	"Ceramic method" from oxides of copper and iron in microwave field	0.4
2	Sol-gel combustion method with citric acid	18
3	Sol-gel combustion method with citric acid and additional microwave treatment	1.0
4	Sol-gel combustion method with urea	25.4
5	Sol-gel combustion method with urea and additional microwave treatment	2.1

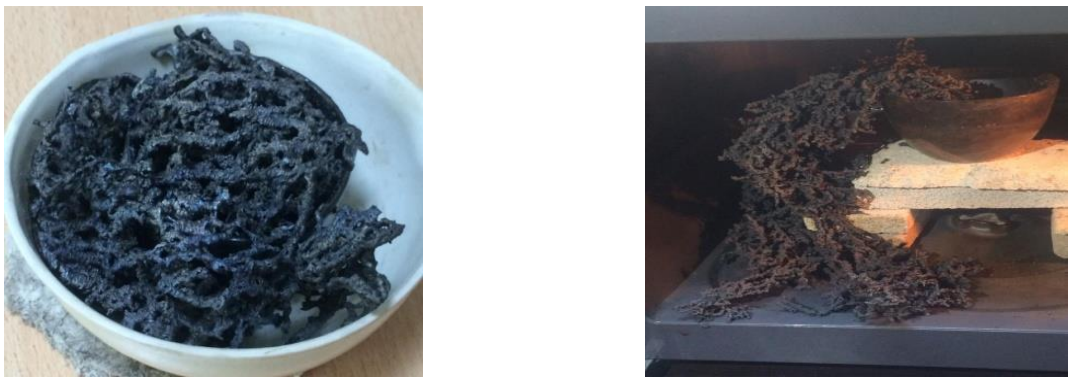
It can be seen from the Table 7, additional microwave treatment of copper ferrite powder leads to a decrease in its specific surface area. The same is true of a sample obtained by a ceramic method in a microwave field. When ferrites are obtained by solid-phase microwave synthesis from oxides, as a result of a very rapid rise in temperature and a long processing time, aggregation of the resulting ferrite particles is observed (Fig. 1)



a *b*

Figure 1. Micrographs of (*a*) nickel and (*b*) cobalt ferrite samples obtained by the solid-phase microwave synthesis from oxides. Reprinted from [15]

A similar picture is also observed during additional microwave heat treatment of the obtained sol-gel method with the combustion of ferrite powder. The ferrites obtained by these two microwave processing methods are characterized by low specific surface area. Based on these experimental data, we decided to use microwave energy to "ignite" the gel without further prolonged irradiation. This process took place even at low magnetron values within a few seconds. Figure 2 shows photographs of the resulting copper ferrite during the combustion of the gel in air (*a*) and "ignition" in a microwave oven (*b*). In both cases, branched, voluminous structures are observed.



a *b*

Figure 2. Samples of copper ferrite obtained by the sol-gel method with combustion (*a*) and gel treatment in a microwave field. The organic reagent is citric acid. Reprinted from [87]

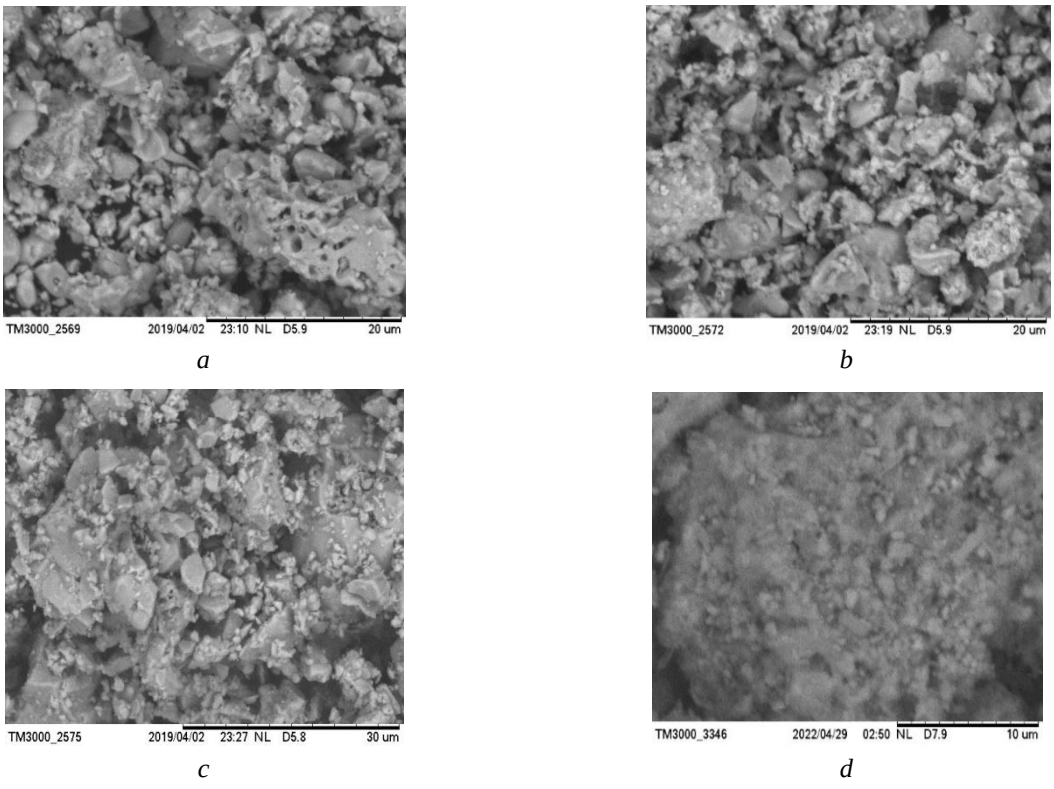
From the diffraction patterns of ferrites obtained by the sol-gel method with conventional combustion and microwave ignition, the crystallite sizes found using the Scherrer equation are quite close (30–35 nm). The same is true of the values of their specific surface area (Table 8) [86].

Table 8

Specific surface of ferrites synthesized by the sol-gel (s-g) method with combustion and the use of various organic reagents with conventional heating and microwave exposure (m.e)

Sample	Citric acid		Glycine		Urea	
	s-g	s-g + m.e	s-g	s-g +m.e	s-g	z-g + m.e
Cu:Fe = 1:2	18	14.8	4	5.4	25.5	22.6
Co:Fe = 1:2	12	13.3	11.7	12	35.3	30.6
Mn:Fe = 1:2	36.5	27.2	15	13.2	32.9	27.6
Ni:Fe = 1:2	6.2	14	7.8	5.8	34.3	28

Microphotographs of Ni, Co, and Cu ferrite samples obtained by the sol-gel method and additional microwave treatment are shown in Figure 3. For comparison, a micrograph of a copper ferrite sample obtained by the conventional sol-gel method is also presented. As can be seen from the photographs, additional microwave treatment of the samples intensifies the crystallization; the photographs show an increase in the amount of ferrite nanoparticles.



a *b* *c* *d*

Figure 3. Micrographs of Ni, Co, Cu ferrite samples obtained by the sol-gel method with combustion and additional microwave treatment (*a*, *b*, *c*) and a copper ferrite sample without microwave treatment (*d*), an organic reagent is citric acid

The above-mentioned works on the sol-gel synthesis of ferrites using microwave radiation were indicative that in most works the synthesis of ferrites by the sol-gel method with combustion was carried out in ordinary household microwave ovens, in which only the magnetron power and exposure time could be controlled, but not the temperature. Hence, the matter was about discrepancies in both the time of microwave treatment from several minutes to half an hour and power from 120 to 700 watts.

A feature of the synthesis by the sol-gel method with combustion is that the beginning of the combustion process is initiated by an external thermal effect, in some local volume of it an exothermic reaction be-

gins. The heat released in this case is transferred to the neighboring layers, in which the reaction is occurred and so the combustion is spread throughout the system. The difference between microwave exposure and conventional thermal exposure lies in the fact that the initiation of combustion occurs simultaneously throughout the entire volume of the system, thereby reducing the duration of the combustion process. But a longer microwave exposure can lead to particle aggregation due to the high rate of temperature rise, which is especially clearly observed in the ceramic synthesis of ferrites [87]. Therefore, when using microwave radiation in the sol-gel method with combustion, along with the nature of the organic reagent, the power and time of exposure to the radiation play an important role in the formation of the surface morphology, texture of ferrites.

Conclusions

The latest studies on the production of cobalt, nickel, copper and manganese ferrites by the sol-gel method with self-ignition and combustion in a microwave field were considered briefly. Also, the influence of various reaction parameters, namely the organic reagent nature, its ratio to precursors, pH of the medium, power and time of microwave exposure on the process of ferrite formation as factors affecting the size of the formed particles and their textural characteristics were analyzed.

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Өтпелі металл ферриттерінің (Co, Cu, Ni, Mn) золь-гель әдісімен жануы және микротолқынды өңдеуді қолдану арқылы синтездеу

Мақалада өтпелі металл ферриттерінің (Co, Cu, Ni, Mn) золь-гель әдісі арқылы жануы, сондай-ақ микротолқынды сәулеленудің әсерінен синтездеу бойынша соңғы жылдардағы жұмыстар қарастырылған. Ферриттер магниттік және оптикалық қасиеттерімен ғана емес, сонымен қатар каталитикалық қасиеттерімен де қызықты. Әдебиеттерде өтпелі металл ферриттерінің әртүрлі реакциялардағы, соның ішінде тотығу реакцияларындағы каталитикалық қасиеттері зерттелген еңбектер қатары көбейіп келеді. Золь-гель әдісінде жану кезінде комплекстүзуші және отын ретінде әр түрлі органикалық компоненттер қолданылатынын ескере отырып, мақалада органикалық реагенттің табиғаты, оның прекурсорларға қатынасы, ортаның pH-ға әсері, феррит түзілу процесі түзілген бөлшектердің мөлшеріне әсер ететін факторлар ретінде және олардың катализде маңызы зор текстуралық көрсеткіштері зерттелген. Соңғы жылдары катализ саласында жұмыс істейтін химиктердің назарын микротолқынды өріске қатысты физикалық өрістердің әртүрлі химиялық процестері, соның ішінде нанокатализаторлар синтезіне әсері туралы зерттеулер аударуда. Әдеби деректерге сәйкес, ферриттердің золь-гель синтезінде микротолқынды сәулелендуді қолдану гетерогенді катализаторлар үшін өте маңызды, яғни меншікті бетінің ауданы жоғары наноферриттерді алуға мүмкіндік береді. Осы тұрғыдан алғанда, мақалада микротолқынды золь-гель синтезін зерттеуге арналған соңғы жылдардағы жұмыстар зерттелген.

Кілт сөздер: ферриттер, золь-гель әдісімен жану, комплекстүзуші заттар, микротолқынды технология, ұсақтық, текстура, дисперстілік, меншікті беті, бөлшектердің мөлшері.

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Синтез ферритов переходных металлов (Co, Cu, Ni, Mn) золь-гель методом с горением и использованием микроволновой обработки

В обзоре рассмотрены работы последних лет по синтезу ферритов переходных металлов (Co, Cu, Ni, Mn) золь-гель методом с горением, а также воздействием микроволнового излучения. Ферриты интересны не только своими магнитными и оптическими свойствами, но и каталитическими. В литературе появляется все больше работ, в которых исследуются каталитические свойства ферритов переходных металлов в различных реакциях, в том числе и окислительных. Ввиду того, что в золь-гель методе с горением в качестве комплексообразователя и топлива используют различные органические компоненты, в настоящей работе рассмотрено влияние природы органического реагента, его соотношения к прекурсорам, pH среды на процесс образования ферритов как факторов, влияющих на размер образующихся частиц и их текстурные показатели, имеющих большое значение в катализе. В последние годы внимание химиков, работающих в области катализа, привлекают исследования по изучению влияния физических полей, к которым относится микроволновое поле, на различные химические процессы, в том числе на синтез нанокатализаторов. Согласно литературным данным, применение микроволнового излучения в золь-гель синтезе ферритов позволяет получать наноферриты с высокой

удельной поверхностью, что очень важно для гетерогенных катализаторов. С этой точки зрения авторами рассмотрены работы последних лет, посвященные изучению микроволнового золь-гель синтеза.

Ключевые слова: ферриты, золь-гель метод с горением, комплексообразователи, микроволновая технология, дисперсность, текстура, удельная поверхность, размер частиц.

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