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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS AND STUDY OF FERRITE CATALYSTS FOR LOW-TEMPERATURE OXIDATION OF CARBON MONOXIDE USING SOL-GEL COMBUSTION AND MICROWAVE RADIATION

Specialty: 2316.01 – Chemical kinetics and catalysis

Field of science: Chemistry

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GENERAL CHARACTERISTICS OF WORK

Relevance of the topic and the degree of development. Carbon monoxide is one of the dangerous and toxic substances released into the atmosphere as a result of human activity (gas emissions from vehicles, thermal power plants, chemical and metallurgical industry enterprises) and natural disasters (fires, volcanic eruptions). Carbon monoxide can combine with hemoglobin in the blood stronger than oxygen and 200-300 times faster. It is typical for big cities that carbon monoxide concentration is 20-30 times higher than the maximum permissible limit (the maximum permissible concentration for CO is 0.05 mg/m^3). Several ways of neutralizing CO from atmospheric emissions are known: CO sorption by liquid and solid adsorbents, conversion of CO to CO_2 by high-temperature combustion, and catalytic oxidation of CO. Currently, numerous catalytic systems for the oxidation of CO to CO_2 are known, based on noble metals, oxides of transition and non-transition metals, perovskites and multicomponent systems with a spinel structure[1].

Catalysts made from platinum group noble metals, employed in transport and industry for neutralisation of carbon monoxide emissions, have high catalytic activity, but also have such shortcomings as being expensive and limited stock. As a result, this hinders their widespread implementation. Replacement of catalytic systems based on noble metals (Pd, Pt, Au, Ag) for low-temperature oxidation of carbon monoxide by oxide catalysts based on transition metals of variable valence is one of the most urgent issues in terms of reducing the cost of catalyst production and, at the same time, reducing energy consumption for the reaction.

It is known that the activity of catalysts depends on many factors, including its chemical composition and structure, the nature of active sites, the surface of the catalyst, dispersion and the presence

[1] Soliman, N.K. Factors affecting CO oxidation reaction over nanosized materials: A review. // Journal of Materials Research and Technology, - 2019, 8 (2), - p. 2395-2407.

of defects in the structure of the catalyst. These factors, in turn, are determined by the technology of obtaining catalysts. Therefore, the development of various technologies for obtaining nanoscale, nanostructured catalytic systems is always in the focus of researchers working in the field of catalysis.

One of the promising methods for the synthesis of catalysts is the sol-gel autocombustion method, which uses the energy of exothermic reactions[2]. The main advantages of this method are low energy consumption, short synthesis time, no need to use special equipment, and the possibility of one-step transformation of precursors into the final product using chemical reaction energy.

In recent years, the use of microwave radiation for the creation of environmentally safe technologies that save raw materials and energy is considered one of the promising directions. Currently, intensification with microwave radiation is used in many industrial processes. Heating with microwave radiation is distinguished by its high speed and efficiency.

Considering the low energy consumption and environmental safety, which are common advantages of sol-gel combustion and microwave technology, their combined use seems promising. In this regard, the development of efficient catalytic systems for low-temperature oxidation of carbon monoxide employing a combined sol-gel combustion method and microwave technology is an ongoing task and is of great relevance. This also leads to the solve of an important environmental problem.

The work objective and subject. The object of the study was ferrite-containing catalytic systems for low-temperature oxidation of carbon monoxide. The subject of the research was the joint synthesis of these systems with sol-gel combustion and microwave technology and the study of their catalytic properties in the reaction.

The work aims and tasks. The aim of this work is to prepare ferrite-containing catalysts by sol-gel combustion and microwave technology and to study their activity in the oxidation of carbon monoxide at low temperatures.

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[2] Bhagwata, V.R. Sol-gel auto combustion synthesis and characterizations of cobalt ferrite nanoparticles: Different fuels approach / V.R.Bhagwata, V.H.Ashok, S.D.Morec [et al.] // Materials Science & Engineering, – 2019, 248, – p. 11438.

For this purpose, it is planned to fulfill the following issues:

- Synthesis of copper, cobalt, nickel and manganese ferrites by sol-gel and sol-gel microwave combustion methods;
- Solid-phase microwave synthesis of ferrite catalysts from appropriate metal oxides;
- Studying the catalytic activity of ferrites synthesized by different methods in the conversion carbon monoxide into carbon dioxide at low temperatures;
- Synthesis of ternary component ferrite-containing catalytic systems by sol-gel combustion method and investigation on carbon monoxide conversion;
- Characterization of synthesized catalytic systems by modern Physico-chemical analysis methods.

The research methods. The catalytic activity of the synthesized catalysts was studied in continious flow reactor (CFR), and the products were analyzed by gas chromatography. The study of physical and chemical properties of catalysts is carried out using modern methods: X-ray phase analysis (XRD), infrared spectroscopy (IR), derivatographic analysis (TA/DTA), electron paramagnetic resonance (EPR), scanning electron microscope (SEM), dynamic light scattering (DLS), X-ray fluorescence analysis of catalyst composition, determination of specific surface area (BET).

The main provisions defended are as follows:

- Comparative study of the catalytic activity of Co, Ni, Cu, Mn ferrites synthesized by thermal sol-gel and microwave sol-gel combustion methods in carbon monoxide oxidation reaction;
- Effect of microwave thermal treatment on catalytic activity of ferrites obtained by sol-gel combustion method and solid-phase synthesis from oxides;
- Determination of synthesis conditions of active ferrite catalysts for carbon monoxide oxidation reaction by sol-gel microwave combustion method;
- Effect of microwave thermal treatment conditions on the specific surface of obtained ferrites.

Scientific novelty of the research.

- Ferrite-containing catalysts were synthesized by sol-gel combustion and microwave technology, and their activity was comparatively studied in the carbon monoxide oxidation reaction;
- The conditions of microwave application to the sol-gel combustion method in ferrite containing active catalysts synthesis for low temperature oxidation of carbon monoxide have been determined. The activity of ferrite-containing catalysts obtained using only microwave energy to 'ignite' the gel is not only comparable and even slightly superior to that of similar samples obtained by the traditional sol-gel combustion method;
- Long-term microwave radiation causes a low specific surface area of ferrites obtained by both sol-gel combustion and solid-phase synthesis from the corresponding oxides;
- Ternary component copper-manganese and cobalt-manganese ferrite-containing active catalysts have been investigated for low-temperature oxidation of carbon monoxide using sol-gel combustion and microwave thermal treatment.

Theoretical and practical significance of research. The practical significance of the work is that the production of ferrite-containing active catalysts was studied for the low-temperature oxidation of carbon monoxide by the sol-gel combustion method and using microwave radiation. The synthesis of catalysts by this method does not require long-term operations and additional thermal processing, which makes the process energy and economically effeciency.

Published works and approbation of the work.

The main results of the dissertation work were presented and discussed at the following international and national scientific conferences:

- XXIX Российская молодежная научная конференция с международным участием, посвященная 150-летию Периодической таблицы химических элементов (Екатеринбург-2019);
- XXI Mendeleev Congress on General and Applied Chemistry (Saint Petersburg-2019);

- III International School-Conference Applied Nanotechnology and Nanotoxicology (ANT-2019) (Sochi-2019);

- «Перспективные технологии и материалы» материалы всероссийской научно-практической конференции с международным участием (Севастополь-2020);

- IV Всероссийская молодежная научная конференция с международным участием - экологобезопасные и ресурсосберегающие технологии и материалы (Улан-Удэ-2020);

- Научный Вестник СамГУ, Scientific Reports of Samarkand State University, Ilmiy Axborotnoma, Електронный Выпуск материалов Международной конференции SOL-GEL 2020 (Samarkand-2021);

- Материалы XXIV Всероссийской конференции молодых ученых-химиков с международным участием (Нижний Новгород-2021);

- V Всероссийская научная конференция «Актуальные проблемы теории и практики гетерогенных катализаторов и адсорбентов» (Иваново-2021);

- IV Российский конгресс по катализу, Роскатализ (Казань, Россия-2021);

- Материалы Всероссийской научной конференции с международным участием, IV Байкальский материаловедческий форум (Улан-Уде-2022);

- Modern Problems of Theoretical & Experimental Chemistry devoted to the 90th Anniversary of Academician Rafiga Aliyeva (Baku-2022);

- Актуальные вопросы химической технологии и защиты окружающей среды. Сборник материалов IX Всероссийской конференции, посвященной 55-летию Чувашского государственного университета имени И.Н. Ульянова (Чебоксары-2022);

- XXIV Международная научно-практическая конференции студентов и молодых ученых. Химия и химическая технология в XXI веке (Томск-2023);

- Седьмая Международная конференция стран СНГ «Золь-гель синтез и исследование неорганических соединений,

гибридных функциональных материалов и дисперсных систем «золь-гель 2023 (Москва-2023);

- XXVII Всероссийская конференция молодых ученых-химиков (с международным участием) (Нижний Новгород-2024).

The name of the organization where the dissertation work was carried out. The dissertation work was performed in the "Preparation of catalysts" laboratory of the Institute of Catalysis and Inorganic Chemistry named after academician M. Nagiyev of the Ministry of Science and Education Republic of Azerbaijan.

The structure and scope of the dissertation work. Dissertation work including introduction (10988 symbols), 4 chapters (chapter I – 56180 symbols, chapter II – 26465 symbols, chapter III – 38735 symbols, chapter IV – 38553 symbols), main results (3008 symbols), and 190 citied references. The total volume of the work, including 73 pictures, 24 tables and 2 schemes, consists of 160 computer printed sheets and 173929 symbols.

Gratitude. The author is deeply grateful to the Department of Physico-Chemical Analysis (ICIC, Baku, Azerbaijan) for their support in conducting X-ray studies.

Research publication rate. 25 scientific works have been published on the subject of the dissertation work. 10 of them are articles (6 of them are included in the international summarizing and indexing systems), abstracts of 15 reports. All of thesis were published in international scientific conference materials.

The author's personal contribution. The author directly participated in the collection and analysis of literature materials for the dissertation work, the synthesis of catalytic systems, the experimental verification of their catalytic activity, the analysis of substances, and the conduct of physico-chemical analysis methods.

In the introduction, the relevance of the topic is justified, the purpose of the work, the issues to be solved, research methods, scientific innovations, the main provisions defended and the approval of the work are shown.

In the first chapter, reviewed the existing catalytic systems for the oxidation of carbon monoxide to carbon dioxide according to

the thesis topic, literature data on the synthesis of ferrite-containing catalysts by sol-gel combustion and microwave technology, catalytic oxidation of carbon monoxide and formulated the thesis topic.

The second chapter contains methods of synthesis of ferrite-containing catalysts using sol-gel combustion and microwave technology, methods of physicochemical analysis of catalyst samples and the equipment used, as well as the description and operation of the technological procedure of the flow-type laboratory experimental setup for the study of CO oxidation into CO_2 - is given.

In the third chapter, the physico-chemical properties of ferrites obtained by sol-gel combustion and microwave thermal processing methods and the catalytic activity were studied in carbon monoxide oxidation reaction.

In the fourth chapter, the results of the performed studies related to the study of the kinetic regularities of the catalytic oxidation reaction of carbon monoxide to carbon dioxide at low temperature and the formulation of the kinetic model are given of the process.

CONTENT OF THE WORK

Synthesis of ferrites using sol-gel combustion and microwave technology

According to the obtained previous results, $CuFe_2O_4$, $CoFe_2O_4$ ferrites showed high activity in the reaction of oxidation of carbon monoxide to carbon dioxide. Aqueous solutions of nitrate salts of metals (Co, Cu, Ni, Mn) and organic reagents (citric acid, glycine, urea) in calculated quantities (the molar ratio of metals and organic reagents was taken 1:2) were mixed with ammonia solution by heating in a magnetic stirrer for 1 hour, the pH of the solution was adjusted to 7, then it was evaporated until it solidified and formed a gel. Drying and burning of the gel was carried out in a drying cabinet and in a microwave oven. In the microwave synthesis of ferrites, an EM-G5593V (Panasonic) microwave oven with a resonator volume of 25 l, a magnetron power of 160-900 W, and a working frequency of 2450 MHz was used for the combustion of the gel. The sequence

of synthesis steps is given in figure 1 [17]. Figure 2 (a and b) shows photographs of some synthesized samples [8].

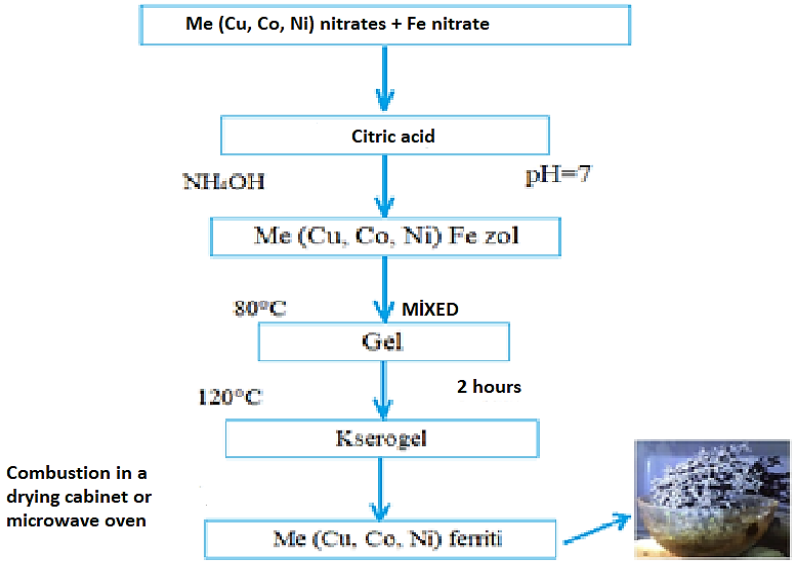


Figure 1. The scheme of obtaining ferrites by sol-gel combustion method [17]

$CoFe_2O_4$ $CuFe_2O_4$ $NiFe_2O_4$ $MnFe_2O_4$

a

$CuFe_2O_4$ $CoFe_2O_4$

b

Figure 2. Photographs of ferrites obtained by the sol-gel combustion method (a) and under microwave synthesis conditions (b) [8, 19]

In order to study the possibilities of using microwave technology in the production of ferrites, in addition to sol-gel combustion synthesis, ferrites of those metals were synthesized from the above-mentioned metal oxides and iron oxide by the solid phase synthesis method [9].

Nickel, cobalt, copper divalent oxides (MeO) and iron (III) oxide were used as starting materials for the solid-phase synthesis of cobalt, nickel and copper ferrites in the dissertation based on known methodology[3].

The ferrites obtained by both methods were mixed with aluminum oxide hydrogel as a binder, made into granules and used after thermal treatment at 500°C. The amount of active mass was taken as 1 gr.

Oxidation of CO was carried out in continuous flow mode in a quartz reactor with CO:air=1:(3-5) mole ratio and space velocity of 6000-12000 h^{-1}. The analysis of the products was carried out in a gas chromatograph.

Helium was used as a gas carrier. The gas flow was given at a rate of 15 ml per minute.

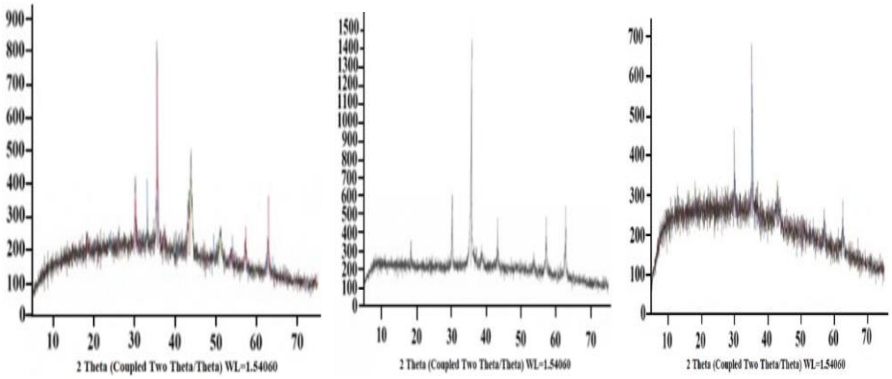
Physico-chemical study of cobalt, copper, nickel and manganese ferrites obtained by sol-gel combustion method

According to literature materials, citric acid was used as a complexing agent and "fuel" in most cases in the synthesis of ferrites by sol-gel combustion method. Therefore, these ferrites were mainly synthesized using citric acid. Figure 3 shows the diffractograms of ferrite samples synthesized in stoichiometric ratios of metals. According to X-ray phase analysis, in addition to ferrites, the samples contain small amounts of metal oxides and hematite.

In the diffractograms, the (111), (220), (311), (222), (400), (422), (511) and (440) maxima characteristic of the cubic spinel structure were determined in the obtained ferrite samples.

[3] Litvishkov, Yu.N. Microwave synthesis of ferrites of Co, Ni, Cu, Zn ferrites / Yu.N.Litvishkov, S.M.Zulfugarova, Z.F.Aleskerova [et al.] // The Russian Journal of Applied Chemistry, - 2018, 91 (5), - p. 679-687

Derivatographic studies are of particular importance in the synthesis of ferrites because the metal nitrates and citric acid-derived gel are subjected to thermal treatment.



1 **2** **3**

Figure 3. Diffractograms of ferrites synthesized by sol-gel combustion method: 1- $NiFe_2O_4$; 2-$CuFe_2O_4$; 3-$CoFe_2O_4$ [18]

Derivatographic studies can be considered in-situ studies, because the formation of ferrites actually occurs when the gel burns and the derivatogram is drawn. Figure 4 shows the derivatogram of the combustion of the gels prepared for the synthesis of cobalt and copper ferrite.

Exothermic peaks appearing in the derivatograms at temperatures of 180-200°C indicate the occurrence of the combustion reaction. During the burning of the gel up to 500°C, all transformations (melting, evaporation, formation of ferrite) already take place, above this temperature the weight loss is negligible [22].

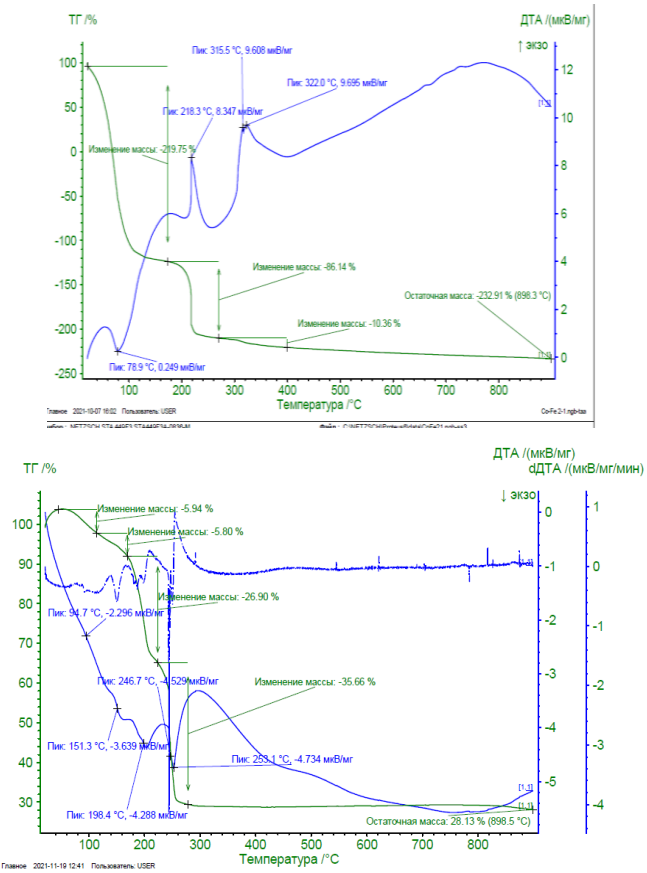
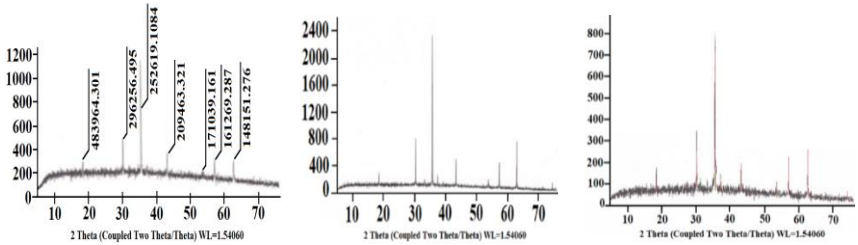


Figure 4. TGA-DTA analysis of [($Co(NO_3)_2$+$Fe(NO_3)_3$+$C_6H_8O_7$)](1) and [$Cu(NO_3)_2$ +$Fe(NO_3)_3$+ $CO(NH_2)_2$] (2) gels [22]

Microwave synthesis of ferrites

The duration of microwave processing is great importance in the synthesis of ferrites from oxides using the energy of microwave radiation, both by the "ceramic" method and by the sol-gel combustion method. Since controlling the temperature rise during microwave processing is not possible, the duration of the treatment will certainly affect the catalytic properties of the synthesised ferrites. During the synthesis of ferrites from metal oxides, a high

temperature is required to proceed for the solid-phase reaction. In the sol-gel combustion method, the microwave energy ignites the gel, as in the conventional thermal effect. Then, because the exothermic reaction proceeds in a manner of itself, in order to clarify questions about the continuance of radiation afterwards, the gel is ignited. In both methods, thermal treatment was carried out in a microwave oven. X-ray phase analysis of samples obtained from oxides by microwave thermal treatment showed the formation of ferrites (Figure 5).



1 **2** **3**

Figure 5. Diffractograms of cobalt (1), nickel (2) and copper (3) ferrites obtained by solid-phase synthesis from their oxides with microwave radiation [18]

Physico-chemical study of ferrites obtained by microwave sol-gel

The synthesis of ferrites by the microwave sol-gel method was carried out in different ways:

- The gel was thermally treated in a microwave oven for a few minutes until it completely burned to powder.
- Microwave thermal treatment is stopped at the moment of ignition of the gel. About 10-15 seconds are enough for the gel to ignite, even at the average power of the magnetron. In this case, microwave energy was used only to ignite the gel.
- The ferrite obtained by burning the gel in a drying cabinet was additionally thermally processed in a microwave oven [5].

Table 1

Phase composition of samples obtained by sol-gel combustion and microwave (m/w) treatment

Sample	Organic reagent	Preparation method	X-ray results
Cu-Fe=1:2	Citric acid	Sol-gel combustion	$CuFe_2O_4$
Cu-Fe=1:2	Glycine	Sol-gel combustion	$CuFe_2O_4$, Fe_2O_3, Cu
Cu-Fe=1:2	Glycine	Additional microwave thermal treatment after sol-gel combustion	$CuFe_2O_4$, $CuFeO_2$
Cu-Fe=1:2	Urea	Sol-gel combustion	$CuFe_2O_4$, $CuFeO_2$, Fe_2O_3
Ni-Fe=1:2	Citric acid	Sol-gel combustion	$NiFe_2O_4$, Fe_2O_3
Ni-Fe=1:2	Citric acid	Additional microwave thermal treatment after sol-gel combustion	$NiFe_2O_4$
Ni-Fe=1:2	Polyethylene glycol	Additional microwave thermal treatment after sol-gel combustion	$NiFe_2O_4$
Ni-Fe=1:2	Citric acid + Polyethylene glycol	Sol-gel combustion	$NiFe_2O_4$, Fe_2O_3, Ni
Ni-Fe=1:2	Citric acid + Polyethylene glycol	Additional microwave thermal treatment after sol-gel combustion	$NiFe_2O_4$
Mn-Fe=1:2	Citric acid	Sol-gel combustion	$MnFe_2O_4$
Co-Fe=1:2	Citric acid	Sol-gel combustion	$CoFe_2O_4$, Fe_2O_3
Co-Fe=1:2	Citric acid	Additional microwave thermal treatment after sol-gel combustion	$CoFe_2O_4$, Fe_3O_4

***m/w- microwave thermal treatment**

Table 1 presents information on the composition of ferrites obtained by sol-gel combustion and microwave thermal treatment methods using various organic reagents. As can be seen, in all cases, regardless of the use of various organic reagents such as complexing agent and "fuel", the corresponding ferrites were unambiguously formed in stoichiometric (1:2) ratios of primary metals.

Catalytic activity of synthesized ferrites in carbon monoxide oxidation reaction

The temperature dependence of the catalytic activity of ferrites synthesized was studied by the sol-gel combustion method in the oxidation of carbon monoxide. The activities of the catalysts were determined by the complete conversion of CO (figure 6).

As can be seen from Figure 6, among the synthesized ferrites, copper ferrite showed higher activity in the oxidation of carbon monoxide to dioxide. Complete oxidation of carbon monoxide in the presence of copper ferrite occurs at 270°C, cobalt ferrite - 380°C, and manganese ferrite - 450°C. Nickel ferrite showed less activity in this reaction. At 450°C, the conversion of CO in its presence is about 60%.

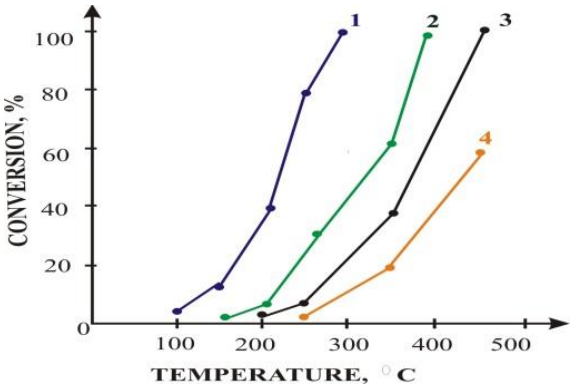


Figure 6. Temperature dependence of carbon monoxide conversion in the presence of copper, cobalt, manganese and nickel ferrites. CO:air=1:3 (mol). GHSV=12000 h^{-1}. 1. $CuFe_2O_4$; 2. $CoFe_2O_4$; 3. $MnFe_2O_4$; 4. $NiFe_2O_4$ [17]

Catalytic activity of Co-Fe system in carbon monoxide oxidation reaction

In order to study the influence of ferritic and oxide phases on their catalytic activity, binary component cobalt-iron oxide systems in the ratios Co:Fe= 1:1, 1:2 and 2:1 were synthesized in the ferrites [20]. Microwave technology was also used in the synthesis of these catalytic systems. Microwave sol-gel synthesis was carried out by two methods:

- The prepared gel was thermally treated in a microwave oven until combustion ceased and turned into powder.
- Microwave thermal treatment of the gel was stopped at the moment of its ignition.

The catalytic activities of Co-Fe systems prepared by different methods in different proportions of metals were studied in the oxidation reaction of carbon monoxide to dioxide and the results are shown in figure 7.

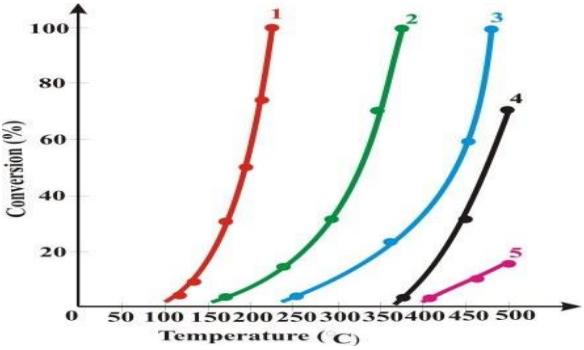


Figure 7. Temperature dependence of carbon monoxide conversion in the Co-Fe catalytic system synthesized by different methods: sol-gel combustion method (1 - Co-Fe=2:1; 2 - Co-Fe=1:1; 3 - Co-Fe=1:2), microwave combustion method (4-Co-Fe=1:2); microwave synthesis from direct oxides (5 - Co-Fe=1:2) [22]

As can be seen from Figure 7, in most samples, complete conversion of carbon monoxide is achieved at temperatures above 350°C. Only the Co:Fe =2:1 sample is an exception. In its presence, 100% conversion of CO to CO_2 occurs at 200°C. The conversion at 500°C is 16% and 64%, respectively, in samples prepared by microwave sol-gel combustion and microwave synthesis from oxides. Samples obtained by "calcining" the gel in a microwave oven show the same activity as samples obtained by the traditional sol-gel combustion method. The specific surface area of the samples is given in table 2. According to Table 2, exposure to microwave thermal treatment for a relatively long time leads to a decrease in specific surface area, which affects the catalytic activity.

Table 2

Specific surface area values of synthesized Co-Fe catalyst samples [19]

Metals ratio	Method of synthesis of catalysts	Specific surface area, m^2/g
Co-Fe=1:2	Solid-phase microwave synthesis	0.2
Co-Fe=1:2	microwave sol-gel	1.8
Co-Fe=1:2	Sol-gel combustion	12
Co-Fe=1:1	Sol-gel combustion	26
Co-Fe=2:1	Sol-gel combustion	28

Catalytic activity of the Cu-Fe system in the oxidation of carbon monoxide

The temperature dependence of the conversion of CO to CO_2 was studied in the presence of Cu-Fe catalysts obtained in the presence of various organic reagents (Figure 8). The nature of the temperature dependence of the catalytic activity of iron-copper-containing oxide systems obtained by sol-gel method using glycine, citric acid and urea as organic reagent is the same.

The specific surface area values of samples synthesized using different organic reagents are also different. While the specific surface area of samples prepared with urea ranges from 20.9-45 m^2/g, the specific surface area of samples prepared with citric acid and glycine is 9-23 m^2/g and 2.7-5.8 m^2/g, respectively.

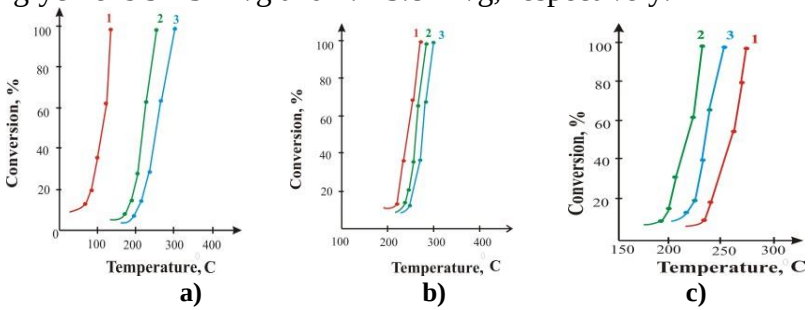


Figure 8. Temperature dependence of carbon monoxide conversion in iron-copper oxide samples: Cu:Fe=1:1 (a), 1:2 (b), 2:1 (c), synthesised by sol-gel combustion in the presence of urea (1), citric acid (2) and glycine (3). GHSV= 12000 h^{-1} [18]

Figure 9 shows the diffractograms of Cu:Fe=1:1 ratio samples obtained by the sol-gel combustion method in the presence of urea, citric acid and glycine. In the diffractograms, the amount of ferrite and copper oxide is about 90%, but in all samples the amount of ferrite is more than copper oxide. The amount of iron oxide in the sample obtained as a result of burning with urea is very low (3%), in the other two samples, the corresponding reflexes are quite clear, and in the sample obtained by burning glycine, it is more intense [18].

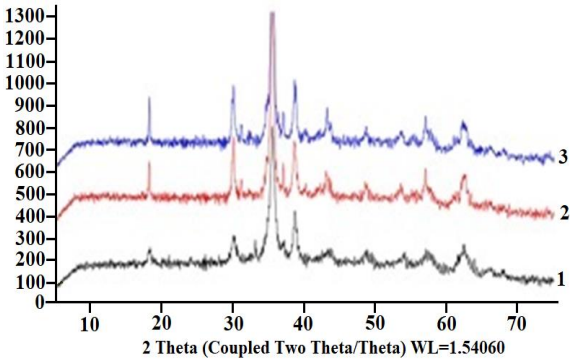


Figure 9. Diffractograms of Cu:Fe=1:1 ratio samples obtained by sol-gel combustion method in the presence of urea (1), citric acid (2) and glycine (3) [18]

Effect of microwave thermal treatment conditions on the catalytic activity of copper ferrite

It can be seen from Figure 10 that samples of copper ferrite obtained by sol-gel combustion method with both conventional and microwave ignition are more active in the oxidation of CO. While in these samples the full conversion of CO to CO_2 occurs at temperatures of 180-250°C, in the samples obtained by microwave afterburning and microwave solid-phase synthesis from oxides, the full conversion occurs at temperatures above 300°C.

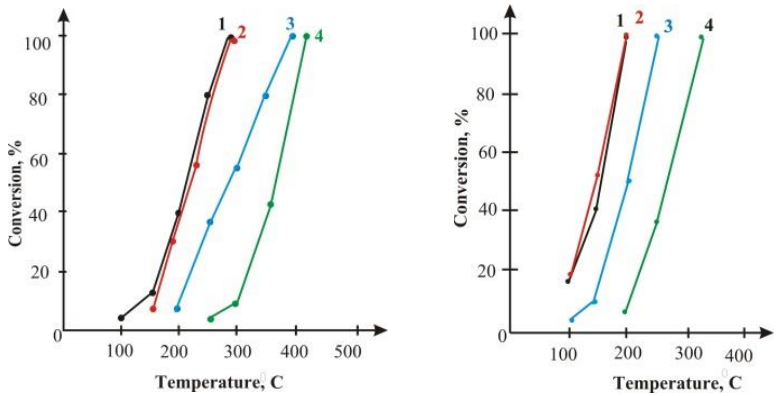


Figure 10. Sol-gel combustion by microwave ignition (1); Sol-gel combustion (2); Sol-gel combustion and additional microwave thermal treatment (3); Temperature dependence of CO conversion on the samples of copper ferrite (4) obtained by microwave solid-phase synthesis from copper oxide and magnetite: a) Cu:Fe=1:2, b) Cu:Fe=2:1. CO:air=1:3 (mol), GHSV= 12000 h^{-1} [5].

From the diffractograms of ferrites obtained by conventional sol-gel combustion and microwave ignition method, crystallite sizes found by Scherrer equation are quite close (30-35 nm). The same can be said about the values of their specific surfaces (Table 3).

Table 3

Specific surface areas of iron-copper-containing oxide samples synthesized by sol-gel combustion and microwave combustion [5]

Catalyst	Citric acid		Glycine		Urea	
	s.g*.	s.g.-m.w*.	s.g.	s.g.-m.w.	s.g.	s.g.-m.w.
Cu:Fe=1:2	14.8	18	4	5.4	22.6	25.5
Cu:Fe=1:1	6	9	5.1	5.8	23.8	25
Cu:Fe=2:1	10.5	15.4	2.8	3.6	18.7	20.9

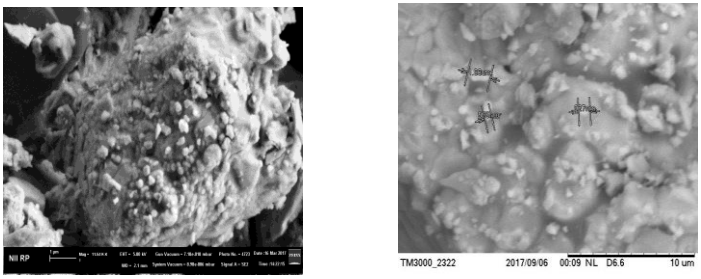
***s.g.-sol-gel combustion;**

***m.w.-microwave thermal treatment**

The value of the specific surface of ferrites obtained by the sol-gel combustion method decreases after additional microwave thermal treatment. This happens due to absorption of microwave radiation by ferrite particles and their aggregation [19].

Electron micrographs of copper ferrite samples obtained after oxide, sol-gel combustion and additional microwave thermal

treatment with microwave radiation demonstrate that both methods produce ferrite particles ranging in size from nanometre to micrometre. In addition, the sample treated with sol-gel combustion and additional microwaves shows a more uniform distribution of nanoparticles on the surface of slightly larger aggregates (figure 11).



a) **b)**

Figure 11. From copper ferritin oxides (a) sol-gel combustion and additional (b) electron micrographs obtained by microwave thermal treatment

Ternary component ferrite-containing systems

The investigated ferrites are spinel-type binary oxide systems. Unlike other binary oxide systems, structural changes in spinel can occur more easily, defects, mobile oxygen and oxygen vacancies are also exist in the structure.

Considering this factor and the fact that manganese is one of the main components of oxidation catalysts, ternary component systems using copper and cobalt ferrite as the third metal, using sol-gel combustion and microwave irradiation were synthesised and their catalytic properties were studied. It should be noted that, based on the results obtained with ferrites, in the synthesis of ternary component systems, microwave radiation was used only for igniting the gel [16].

Co-Mn-Fe oxide catalytic systems

The composition of the ternary component systems obtained by the sol-gel combustion method is more complex. The reflexes of $CoFe_2O_4$, $CoFe_{0,8}Mn_{1,2}O_4$, Mn_3O_4, and Fe_3O_4 phases were observed in the diffractogram of Co-Mn-Fe (1:1:1) oxide system. The diffractogram of the Co-Cu-Fe oxide system (1:1:1) shows reflexes

of the following phases: $CuFe_2O_4$, $CoFe_2O_4$, $CoCu_2O_3$, CuO. Thus, in the investigated three-component systems obtained by sol-gel combustion method, in parallel with copper, iron and manganese oxides, the formation of cobalt ferrite and copper ferrite was observed, as in the case of binary component oxide Co-Fe system. The results of the time dependence of the conversion of carbon monoxide in the presence of the obtained ternary component oxide systems are given in figure 12. It can be seen from the figure that these ternary systems are more active in the low-temperature oxidation of carbon monoxide. The temperature of complete conversion of carbon monoxide is lower and is 145-160°C.

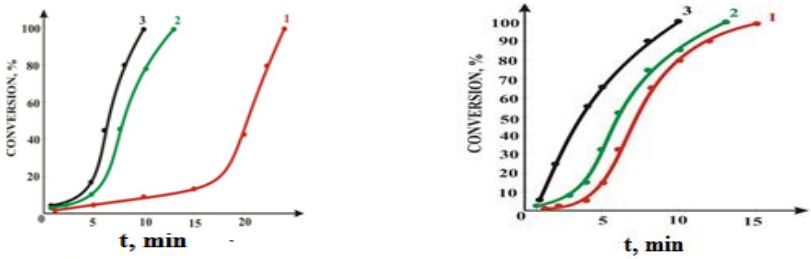


Figure 12. Time dependences of CO conversion rate in cobalt-containing ternary component oxide systems at different temperatures: a) Co-Mn-Fe (1-145°C, 2- 165°C, 3-180°C); b) Co-Cu-Fe (1-160°C, 2- 170°C, 3-215°C) [22]

Cu-Mn-Fe oxide catalytic systems

It is also interesting that Cu:Mn:Fe ternary systems exhibit activity in CO conversion. For this purpose, ternary component Cu:Mn:Fe oxide systems were synthesized by sol-gel combustion method in the ratio of components 1:1:1, 2:1:1, 1:2:1 and 1:1:2. In addition to double oxides of manganese and iron (Mn_3O_4 və Fe_3O_4) ferrites of manganese $Mn_{0.98}Fe_{2.02}O_4$ and copper $CuFe_2O_4$, copper manganite $CuMn_2O_4$, copper ferrites substituted with manganese manqanla ($Cu_{0.5}Mn_{0.5}Fe_2O_4$, $Cu_{1.2}Mn_{1.8}O_4$, $Mn_{0.43}Fe_{2.57}O_4$) is formed, these obtained systems can be considered as solid solutions.

The results of experiments on the oxidation of carbon monoxide in the presence of Cu-Mn-Fe catalysts synthesized by sol-

gel combustion showed that the complete conversion of CO occurs in the temperature range of 130-170°C (Table 4). During the first 3-5 minutes after starting the reaction, the conversion starts to increase dramatically and reaches 100% within the next 5 minutes. A sharp increase in conversion was observed starting from the first minutes and the maximum conversion was reached in a shorter time of 7-8 minutes at a temperatures which was slightly higher than the abovementioned. A similar situation is observed in the samples synthesized by sol-gel and microwave radiation (Table 4).

Table 4

Temperature results and rate of the oxidative conversion reaction of CO on Cu:Mn:Fe catalysts

Ratio of components in Cu:Mn:Fe catalyst	Specific surface area, m^2/g	100% conversion temperature, ^0C	100% conversion time, min
1:1:1	19.5	170	9
1:2:1	12.4	155	10
1:1:2	13.5	130	7-8
2:1:1	19	145	8

Co-Mn, Cu-Mn, Co-Cu binary oxide systems

In the studied iron-containing binary and ternary oxide systems, the main components of the composition are ferrospinels and oxides. Spinels of transition metals (cobaltites, manganites) show high activity in low-temperature oxidation of carbon monoxide. Among them, binary copper-manganese and cobalt-manganese systems can be mentioned. Therefore, the iron-free binary systems Co-Mn, Co-Cu and Co-Cr were synthesized and investigated by sol-gel combustion method [24].

In the Co-Mn system, according to the X-ray phase analysis, in addition to the spinel-type binary oxide-cobalt manganite $CoMn_2O_4$, cobalt and manganese oxides are also obtained, in the Co-Cu(1:1) system only Co_3O_4, CuO, Cu_2O oxides, and in the Co-Cr system Co_2CrO_4 is formed.

In Co-Mn and Cu-Mn systems, when the ratio of metals is 1:1, oxide of both metals is observed. In the diffractogram of the Cu:Mn=2:1 system, only can be seen reflections of copper oxide.

Table 5 shows the temperature and time dependences of the oxidation reaction of CO in the presence of these catalysts.

In copper-manganese 1:1 and 2:1 oxide systems, it is possible the oxidation reaction of carbon monoxide at a lower temperature.

Table 5

Temperature and time parameters ensuring complete conversion of CO in the presence of cobalt-containing binary oxide catalysts

Catalyst	100% conversion temperature, ^{0}C	100% CO conversion time, min
Co-Mn=1:1	200	9
Co-Mn=2:1	180	10
Co-Cu=1:1	150	8-9
Co-Cr=2:1	145	11
Cu-Mn=1:1	120	12-13
Cu-Mn=2:1	135	6-7

Feasibility study of solid-phase microwave synthesis of ferrites on a carrier

Co, Ni ferrites were obtained using metal oxides in order to clarify the possibility of microwave synthesis of ferrite in one step on the carrier. The possibility of forming ferrites by solid-phase synthesis directly on the carrier was studied by checking the example of synthesis of Co, Ni ferrites. For this purpose, precursors (metal oxides) and carrier (aluminum oxide) were first taken in the ratio of 20% ($MeO+Fe_3O_4$)+80%Al_2O_3 (where Me=Co, Ni). X-ray phase analysis of the samples was carried out to determine the acquisition of the corresponding ferrites after thermal treatment in a microwave oven. X-ray phase analysis showed the formation of only oxide phases and not ferrites in the diffractogram. Small reflections of cobalt ferrite and aluminum ferrite were determined only at 10-15% carrier concentration. Thus, the study of the microwave synthesis of ferrites from the appropriate oxides on the carrier (γ-Al_2O_3) with the

aim of using them as catalysts showed the impossibility of conducting such a synthesis.

In contrast to oxides, it was possible to obtain Cu:Mn=1:1 catalytic system by one-stage sol-gel combustion with a binder. The study of activity of the catalyst obtained by this method showed that complete CO conversion in the presence of this catalyst occurs at 155°C.

Study of thermodesorption of CO on synthesized catalysts

In the oxidation of CO, the adsorption stage of gas molecules on the surface of the catalyst is very important. The thermodesorption of carbon monoxide was studied from the surface of different samples of synthesized catalysts (figure 13). The interaction of CO with catalysts was studied at a linear heating rate of 20 °C /min in the temperature range of 20-300°C.

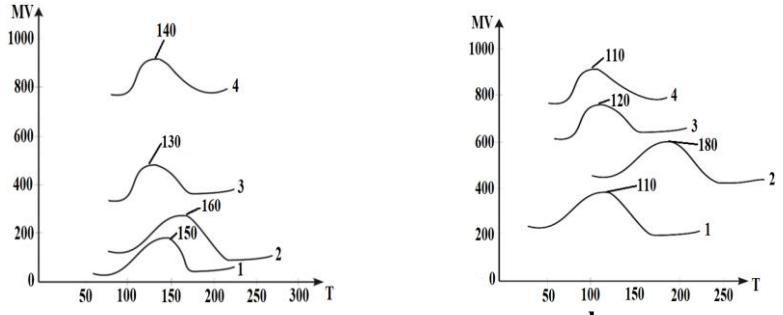


Figure 13. Thermodesorption of carbon monoxide from the surface of binary and ternary component catalytic systems: a) 1 – Cu:Mn:Fe=1:2:1; 2 - 1:1:1; 3 - 1:1:2; 4 - 2:1:1; b) Co:Cu=1:1(1), Co:Mn=2:1(2), Cu:Mn=2:1(3) and Cu:Mn=1:1(4)

From the obtained results (figure 13), it is clear that there is only one form of adsorbed carbon monoxide on the surface of the catalysts in the temperature range of 20-200°C. Given that the weights of the catalyst samples are the same (about 1.0 gr) and their specific surfaces are quite close, the thermodesorption curves can be compared. According to catalytic experiments, these samples completely convert carbon monoxide to carbon dioxide in this temperature range. At relatively high temperatures, the thermodesorption curves do not have clearly noticeable peaks. This

applies to catalysts active at higher temperatures, such as cobalt and copper ferrites. Thus, according to the thermodesorption data, carbon monoxide adsorption is also observed in the same interval for all studied catalysts that carry out the oxidation reaction of carbon monoxide at low temperature, in the temperature range of 110-180°C.

Study of kinetic regularities of carbon monoxide oxidation in the presence of synthesized ferrite catalysts

In order to study the kinetic regularities of chemical reactions, it is first necessary to determine whether the reaction takes place in the kinetic region. In the presence of CuMnFe, the most active catalyst synthesised using a known technique, it was observed that the linear rate remained constant with no change in CO conversion when the linear rate was varied by 0.6-2.5 cm^3/sec and the catalyst size was varied between 0.16-0.5 mm. In other word, it was determined that the oxidation reaction of CO does not have an internal or external diffusion effect, that is, the reaction takes place in the kinetic region.

Table 6

Experimental results of kinetic studies of CO oxidation reaction in the presence of Cu-Mn-Fe catalyst

T, °C	GHSV, h^{-1}	$P_{CO,\ atm}$	$P_{O_2,\ atm}$	X_{CO}, %	Yield CO_2, %
140	3000	0.015	0.06	97.3	97.2
140	3000	0.015	0.09	97.6	97.5
140	3000	0.015	0.12	98.1	98.0
140	3000	0.010	0.08	98.0	98.0
140	3000	0.020	0.08	97.5	97.4
140	3000	0.040	0.08	96.6	96.5
160	3000	0.015	0.06	97.7	97.6
160	3000	0.015	0.09	98.2	98.0
160	3000	0.015	0.12	98.4	98.2
180	3000	0.015	0.06	98.0	98.5
180	3000	0.015	0.09	99.2	99.0
180	3000	0.015	0.12	99.5	99.3
140	6000	0.015	0.06	96.3	96.2
160	6000	0.015	0.09	99.9	99.9
180	6000	0.015	0.12	99.6	99.4
160	9000	0.015	0.06	99.9	99.9
180	9000	0.015	0.09	99.8	99.8

At the same time, other technological parameters in a wide variation range are temperature 140-180°C, contact time 0.1-0.3 sec, space velocity 3000-9000 h^{-1}, $CO:O_2$=1:1-1:8 ratio, the effect of the oxidation reaction rate of carbon monoxide has been studied. It was determined that 100% conversion of carbon monoxide in the presence of CuMnFe catalyst is ensured under optimal conditions (T=160°C, t=0.3 sec, GHSV=3000 h^{-1}, $CO:O_2$=1:6) (Table 6).

Also, the carbon monoxide oxidation reaction proceeds in a redox scheme, with the oxidation rate of the catalyst surface exceeding the reduction rate. It was determined that during the reduction of the catalyst surface CO is supplied to the surface, CO absorption stops after 4-6 minutes and oxidation of CO occurs rapidly only after oxygen is supplied to the surface (Figure 14).

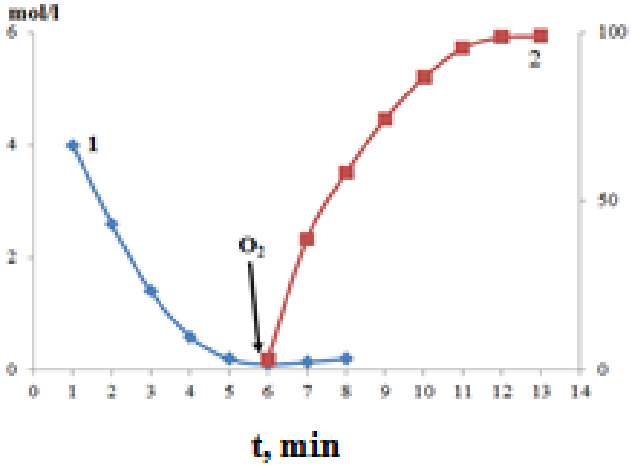


Figure 14. The interaction of the catalyst surface with CO and the time dependence of air supply to the system.
1. CO is given 2. O_2 is given.

In the literature, the mechanism of carbon monoxide oxidation was shown to occur according to associative and dissociative simple schemes by Langmuir-Hinshelwood, Mars-Van Krevelen, and later by Kazansky, Rozovsky, Alkhasov and others. In order to determine the share of this or that mechanism, by analyzing the kinetic curves

obtained by the kinetic method, in other words, by giving the catalyst surface separately (CO, CO, CO) O_2 and mixed (CO, CO, CO)($CO+O_2$) gas mixture the rate of the $CO+O_2$ catalytic oxidation reaction was found to be high in all cases. In other words, although both mechanisms work, but prevails the Langmuir-Hinshelwood mechanism. This can be mainly explained by the strong bonding and structural position of oxygen in the volume of the catalyst, as well as the bonding type of the metal cation. Based on the analysis of literature materials and the experimental results we obtained, we can describe the oxidation mechanism of carbon monoxide with the presence of CuMnFe catalyst as follows:

$$O_{2(gas)} + 2Z \underset{k_{-1}}{\overset{k_1}{\rightleftarrows}} 2ZO_{(ads)}$$

$$CO_{(gas)} + Z \underset{k_{-2}}{\overset{k_2}{\rightleftarrows}} ZCO_{(ads)}$$

$$ZO_{(ads)} + ZCO_{(ads)} \xrightarrow{k_3} ZCO_{2(ads)}$$

$$ZCO_{2(ads)} \underset{k_{-4}}{\overset{k_4}{\rightleftarrows}} Z + CO_2$$

Z-is the active site of the catalyst.

Here, for CO oxidation, it is assumed that the catalyst surface is completely covered by oxygen due to the increase in oxygen concentration, the rate of the oxidation reaction exceeds the reduction rate, and both O_2 and CO are adsorbed at the same active sites. Also considering the complete coverage of the catalyst surface

$$C_Z+C_{ZO}+C_{ZCO}+C_{ZCO_2}=1 \qquad \textbf{(1)}$$

Solving the rates of each stage and the concentration of substances as a system of differential equations, we obtain

$$W_{CO_2} = \frac{k_1 \cdot k_2 \cdot k_4 P_{O_2} P_{CO}}{(1+k_1 P_{O_2}+k_2 P_{CO})^2} \qquad \textbf{(2)}$$

In order to obtain the equilibrium constant in the oxidation reaction, we use the Kutta-Meyerlon method and integrate it within the "MATLAB" computer program to obtain the calculated values of the concentrations for the kinetic model. For this, from the method of minimizing the function

$$F = (k_i - k_4) = \sum_{i=1}^{4}\{C_{i_n}\left(k_{1_n} - k_5\right) - C_1\}^2 \quad \textbf{(3)}$$

can be used to calculate the value of the parameters. Here, $C_{1n}(k_1...k_4)$ i=1-4 is the solution of the system of differential equations at the indicated value of each parameter. C_{it}=1:4 are experimental values of concentrations. K is taken as the equilibrium constant. Taking K_i=1-4 as a starting point, the quadratic function *f(x)* is calculated and minimised based on Powell's method. Then, the values of the activation energy are calculated by plotting the lnk= f $(\frac{1}{T})$ graphical dependence using the well-known "Poisk" program to calculate the parameters k_0 and the values of the activation energy (E) of the selected model. Calculations were performed for three minimum temperatures of 140°C, 160°C and 180°C and the obtained values are given in table 6. The relative error of the values of the parameters calculated on the basis of the kinetic model with the values obtained from the experimental results was 8-10% (Table 7).

Table 7

Values of the parameters of the kinetic model of the carbon monoxide oxidation reaction

Rate constant	gmol/g cat.sec			lnk_0	E (Q), kJ/mol
	140°C	160°C	180°C		
k_1	0.3453	0.3112	0.2874	-5.842	19.43
k_{-1}	$0.6421•10^{-5}$	$0.6073•10^{-4}$	$0.5321•10^{-3}$	-4.056	16.27
k_2	2.3760	$2.8431•10^{-1}$	$4.2433•10^{-2}$	8.3710	17.62
k_{-2}	$0.7214•10^{-4}$	$0.7642•10^{-3}$	$0.3561•10^{-2}$	1.6362	15.58
k_3	1.4361	2.3780	3.1532	3.7864	53.54
k_4	$0.7682•10^{-7}$	$0.8412•10^{-8}$	$0.9347•10^{-9}$	-2.5763	12.64
k_{-4}	$0.4336•10^{-3}$	$0.6872•10^{-2}$	$1.4652•10^{-2}$	0.7854	10.31

CONCLUSIONS

1. Binary and ternary catalytic systems containing Cu, Co, Ni, Mn ferrites were obtained by sol-gel combustion method and microwave technology, and their high activity was investigated in the reaction of low-temperature oxidation of carbon monoxide [8, 20, 22, 24].

2. Using various organic reagents by sol-gel method and in stoichiometric amounts of precursors according to $MeFe_2O_4$ formula, ferrites (Me=Cu, Co, Ni, Mn) have been shown to obtain a small amount of Fe_2O_3. In these ferrites, the conversion of carbon monoxide into carbon dioxide was determined at temperatures higher than 270°C. In non-stoichiometric amounts of precursors, it was determined that metal oxides, along with ferrites, were formed in the catalyst, and that the samples showed high activity at lower temperature [5, 18, 22].

3. During the use of microwave technology in the synthesis of ferrite catalysts, the influence of the microwave treatment time of the obtained gel on the catalytic activity was determined and it was shown that it is enough to use the energy of microwave radiation to "ignite" the gel to obtain an active catalyst. At this time, the formation of crystallites was determined with a smaller size and a larger specific surface area (19-28 m^2/g) [17].

4. It was determined that ferrites obtained by microwave synthesis from oxides in the solid phase are not active in the conversion of carbon monoxide at low temperature. A similar situation is observed after additional microwave thermal treatment of ferrite powder obtained by sol-gel combustion method. The specific surface area of ferrites obtained by both methods is small (0.4-2.1 m^2/g), which is attributed to the aggregation of ferrite particles obtained due to the microwave treatment time and rapid increase in temperature [19, 22].

5. According to the results of the X-ray phase analysis of the ternary component Cu-Mn-Fe oxide system synthesized by the sol-gel combustion method, it was shown that they have a complex phase composition. In addition to the double oxides of manganese and iron, the formation of manganese ($Mn_{0.98}Fe_{2.02}O_4$) and copper

ferrite ($CuFe_2O_4$), copper manganite $CuMn_2O_4$, copper ferrites substituted with manganese ($Cu_{0.5}Mn_{0.5}Fe_2O_4$, $Cu_{1.2}Mn_{1.8}O_4$, $Mn_{0.43}Fe_{2.57}O_4$) has been determined. In the Co-Mn-Fe oxide system, $CoFe_2O_4$, $CoFe_{0,8}Mn_{1,2}O_4$, Mn_3O_4, Fe_3O_4 phases, and in the Co-Cu-Fe oxide system are determined $CuFe_2O_4$, $CoFe_2O_4$, $CoCu_2O_3$, CuO. These catalysts ensure complete conversion of carbon monoxide to carbon dioxide at a temperature of 130-170°C [16].

6. The kinetic regularities of the oxidation reaction of CO in the presence of the CuMnFe catalyst synthesized by sol-gel and microwave combustion methods were studied and it was determined that the reaction takes place according to the redox scheme. The rate of oxidation of the catalyst surface is several times higher than the rate of reduction. The oxidation of CO occurs according to the Langmuir-Hinshelwood mechanism. The mechanism, kinetic model of the process was proposed and were calculated its parameters [16].

7. The study of solid-phase microwave synthesis of ferrites from appropriate oxides on a carrier (γ-Al_2O_3) showed the impossibility of such synthesis. Simultaneously, the possibility of a one-step synthesis of the binary Cu:Mn=1:1 catalytic system on the carrier by the sol-gel combustion method was determined, and the complete conversion of carbon monoxide in this system was studied to occur at a temperature of 155°C [9, 24].

The main results of the dissertation work were published in the following articles and abstracts:

1. Азимова, Г.Р., Алескерова, З.Ф., Зульфугарова, С.М. Синтез феррита никеля микроволновой обработкой смеси оксида никеля и магнетита // XXIX Российская молодёжная научная конференция с международным участием «Проблемы теоретической и экспериментальной химии», Екатеринбург, – 23 – 26 апреля, – 2019, – с. 246.

2. Azimova, G.R. Sol-gel synthesis of ferrite copper with exposure microwave radiation / G.R. Azimova, S.M. Zulfugarova, Z.F. Aleskerova, N.V. Shakunova // XXI Mendeleev Congress on

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3. Azimova, G.R. Synthesis and study of cobalt ferrite in the reaction of carbon monoxide oxidation / G.R. Azimova, Z.F. Aleskerova, S.M. Zulfugarova, N.M. Gasanguliyeva // II International School-Conference «Applied Nanotechnology &Nanotoxicology» («ANT-2019»), Sochi, – 10 – 13 October, – 2019, – p. 125-126.

4. Азимова, Г.Р. Влияние способа приготовления на каталитические свойства феррита меди в реакции окисления монооксида углерода / Г.Р. Азимова, С.М. Зульфугарова, З.Ф. Алескерова, Э.Г. Исмаилов // «Перспективные технологии и материалы» материалы всероссийской научно – практической конференции с международным участием, Севастополь, – 14 – 16 октября, – 2020, – с. 199-203.

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6. Азимова, Г.Р. Исследование ферритов Co, Cu, Ni и Mn в реакции окисления моноксида углерода / Г.Р. Азимова, С.М. Зульфугарова, З.Ф. Алескерова, Ю.Н. Литвишков // IV Всероссийская молодежная научная конференция с международным участием–экологобезопасные и ресурсосберегающие технологии и материалы, часть 2, Улан-Удэ, – 2020, – с. 9-11.

7. Азимова, Г.Р. Применение микроволновой активации при получении медь-марганец-железных катализаторов окисления монооксида углерода в диоксид золь-гель методом с горением / Г.Р. Азимова, С.М. Зульфугарова, З.Ф. Алескерова, А.Х. Гулиева // Научный Вестник СамГУ, Scientific Reports of Samarkand State University, Ilmıy Axborotnoma, Електронный Выпуск материалов Международной конференции SOL-GEL 2020, – Samarkand, – 2021, – с. 14-15.

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