Morphological features of Co$_3$O$_4$ nanoparticles obtained by solution combustion method

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ABSTRACT

The global environmental crisis has made it imperative to enhance tools and techniques for monitoring and analyzing environmental parameters. Gas sensors, crucial for air quality assessment, continually undergo technological advancements to enhance accuracy and efficiency in detecting harmful substances. They play an essential role in ensuring safety in workplaces, urban areas, and industries, aiding pollution control efforts. Enhanced gas sensor performance hinges on careful selection and control of gas-sensitive materials and their structure. This involves optimizing gas-sensitive compounds, employing advanced materials, and developing technologies for sensitive and rapid substance detection. One promising compound for this purpose is Co$_3$O$_4$ oxide, synthesized efficiently using the solution combustion method. This method offers simplicity and allows for precise control over product structures and properties, enabling customization for specific requirements and ensuring high detection efficiency and accuracy. In this study, Co$_3$O$_4$ particles were synthesized from a mixture of cobalt nitrate and glycine with the addition of nitric acid using the solution combustion method. The influence of nitric acid addition and the fuel-to-oxidizer ratio on the morphological characteristics of the cobalt oxide was investigated. The results from SEM, TEM, XRD, and SAXS analyses confirmed that the addition of nitric acid and a fuel-rich mixture lead to nanoparticles with smaller diameter spread and more stable characteristics.

Keywords: metal oxide nanomaterials, Co$_3$O$_4$ nanoparticles, solution combustion method, exothermic redox reaction, gas sensors.

1. Introduction

Nowadays, gas sensors are attracting considerable attention from researchers due to the aggravated environmental problems. Gas sensors are of paramount significance in determining the type and concentration of pollutants in the air. There are different types of gas sensors such as semiconductor, photoionization, electrochemical, etc. [1].

Conductometric semiconducting metal oxide gas sensors, also, is one of the groups of gas sensors suitable for conducting gas measurements under atmospheric conditions. They have several advantages including flexibility in manufacturing, ease of use, low cost, and, most importantly, the ability to detect a wide range of gases [2].

An essential characteristic of conductometric semiconducting metal oxide gas sensors is the reversible interaction of gas with the material surface, which can be affected by the natural properties of the basic components, microstructure of the sensing layers, surface area, and external factors such as temperature and humidity [2-7]. The sensitivity of gas sensors plays an important role in their operation. However, there is currently no single and universal gas sensor for the detection of different gases. Therefore, various transition metal oxides like Fe$_2$O$_3$ [8], Cr$_2$O$_3$ [9], NiO [10], and non-transition metal oxides including pre-transition metals like ZnO [11], Al$_2$O$_3$ [12], SnO$_2$ [13], and so on are used in conductometric meters for the detection of combustible, oxidizing and reducing
gases. Inertness, structure stability, and the easiness of measuring the electrical conductivity, which depends on the electronic configuration, are the main parameters in the selection of oxides for gas sensors. Despite the wide range of oxides, transition metal oxides with $d^0$ and $d^{10}$ electron configurations have a practical application in gas sensors. Such oxides include TiO$_2$ [14], WO$_3$ [15], and Co$_3$O$_4$ [16].

Co$_3$O$_4$ stands out among the others because it is a p-type oxide with the structure of spinel AB$_2$O$_4$ and a forbidden band energy of 1.6–2.2 eV [17]. Due to its structure, Co$_3$O$_4$ has high stability, specific capacitance (500–700 F/g [18]), electrical conductivity (charging capacity 900–1000 mAh/g [19]), large surface area (more than 90 m$^2$/g [18]) and pore volume (135.72–292.66 cm$^3$/g [19]). Authors [17-20] investigated gas sensors based on cobalt oxide and found that it has a high potential in gas detection.

In addition, cobalt oxide nanoparticles can be used in magnetic materials [21], ceramic pigmentation [22], as a catalyst [23], electrochromic devices [24], and rechargeable batteries [19]. Therefore, the synthesis of cobalt oxide powder with improved characteristics has attracted huge interest recently.

There are numerous methods for the synthesis of cobalt oxide. However, each of them has its own advantages and disadvantages, and the choice of the method depends on the area and purpose of the material application.

As can be seen from Table 1, despite the various methods for the synthesis of cobalt oxide, obtaining homogeneous nanoparticles remains a costly method because controlling the size, shape, and morphology of the product, as well as the valence of cobalt ions like Co$^{3+}$ and Co$^{2+}$, is a labor-intensive process. Moreover, for extensive applications of Co$_3$O$_4$, including in gas sensors, the oxide must have high stability and dispersion. Also, it is necessary to properly approach the issue of synthesis waste disposal, for example, in the synthesis of nanoparticles by co-precipitation, in general, besides metal oxide, many compounds are formed, which are difficult to utilize. Therefore, a more ecological method of synthesis of Co$_3$O$_4$ should be preferred.

Among a variety of methods for the synthesis of cobalt oxide, the solution combustion method is highlighted. It is based on the self-propagating combustion of a mixture of fuel and oxidizer in the liquid phase. Gradual heating of reagents dissolved in water leads to an exothermic redox reaction resulting in the formation of metal oxide [17, 30]. Compared to other methods, this method is characterized by simplicity, practicality, and rapidity.

Furthermore, by changing the ratio of oxidizer and fuel, it is possible to obtain cobalt oxide nanoparticles with specified properties and structure, which expands the scope of the product application.

**Table 1. Methods of cobalt oxide synthesis**

<table>
<thead>
<tr>
<th>№</th>
<th>Method</th>
<th>Advantages</th>
<th>Disadvantages</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Thermal decomposition</td>
<td>1. Simple and inexpensive method</td>
<td>1. High temperatures required</td>
<td>[25]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Cobalt oxide with high purity</td>
<td>2. Complexity of particle size control</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Sol-gel</td>
<td>1. Cobalt oxide with high dispersibility</td>
<td>1. It is a time-consuming process, and specialized equipment is required</td>
<td>[26]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Suitable for making thin films and coatings</td>
<td>2. Does not have the ability to economically scale</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Spray pyrolysis</td>
<td>1. Can be used to create thin films and coatings</td>
<td>1. The specialized equipment is required</td>
<td>[27]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Process is highly controllable</td>
<td>2. The possibility of formation of irregular films</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Hydrothermal method</td>
<td>1. Applicable to produce nanostructured materials</td>
<td>1. High pressure and temperature are required</td>
<td>[28]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. The specialized equipment is required</td>
<td>2. The specialized equipment is required</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Co-precipitation</td>
<td>1. Control of composition and structure</td>
<td>1. Complex tuning of experimental conditions and/or expensive reagents are required</td>
<td>[29]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Formation of nanostructures</td>
<td>2. Tendency of synthesis products to aggregate</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3. Reduction of temperature conditions</td>
<td>3. Does not suitable for mass production</td>
<td></td>
</tr>
</tbody>
</table>
2. Experimental part

2.1. Materials

The following materials and equipment were used in this work: cobalt nitrate hexahydrate (Co(NO$_3$)$_2$·6H$_2$O), glycine (C$_2$H$_5$NO$_2$) and nitric acid (all reagents were chemically or analytically grade); magnetic stirrer (MS-H340-S4); and laboratory plate.

2.2. Synthesis of Co$_3$O$_4$ by solution combustion method

The synthesis of ultra-disperse metal oxide particles is based on the exothermic process of interaction between the components of the solution system: fuel and oxidizer. Co$_3$O$_4$ nanoparticles were synthesized by the solution combustion method. Cobalt nitrate hexahydrate (Co(NO$_3$)$_2$·6H$_2$O) as oxidizer and glycine (C$_2$H$_5$NO$_2$) as fuel were used to produce cobalt oxide. The reaction equation of glycine-nitrate synthesis of cobalt oxide is as follows:

$$3\text{Co(NO}_3\text{)}_2 \cdot 6\text{H}_2\text{O} + 6\text{C}_2\text{H}_5\text{NO}_2 + 6.5\text{O}_2 \rightarrow \text{Co}_3\text{O}_4 + 12\text{CO}_2\uparrow + 6\text{N}_2\uparrow + 33\text{H}_2\text{O}\uparrow$$  (1)

The influence of the ratio of fuel and oxidizer on the morphology of the obtained nanoparticles was investigated. For this purpose, synthesis was carried out from a mixture of cobalt nitrate hexahydrate and glycine in the stoichiometric ratio $\phi=1$ (3 moles of cobalt nitrate to 6 moles of glycine) and under the condition of a fuel-rich mixture $\phi=1.5$ (for 3 moles of cobalt nitrate – 9 moles of glycine). The effect of nitric acid addition on the dispersion of cobalt oxide was investigated. Nitric acid was added in an amount of 10% by weight of cobalt nitrate.

The initial reagents were completely dissolved in 50 ml distilled water in a heat-resistant beaker and then evaporated to a volume of 5–7 ml. After evaporation, the reaction mixture was heated to 260 °C, whereupon spontaneous ignition of the solution was observed. The decomposition temperature of glycine was taken into account while selecting the self-ignition temperature. The ignition of the fuel mixture in solution leads to a temperature increase to 1200 °C and precipitation of the final product. The product of synthesis was washed with distilled water, and then dried at 80 °C for 24 h.

2.3. Methods of Co$_3$O$_4$ characterization

Currently, microscopic techniques are widely used to analyze the structure of various materials, including nanoparticles, and have an essential role for their characterization. These techniques include visible spectrum imaging, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). One of the significant advantages of visual methods is the ability to represent structure in different regions of the samples. Thus, the images obtained provide information useful for comparing localized structures throughout the sample. Despite these advantages, optical methods do not provide the quantitative data necessary for comparative analysis of different nanoparticles. Therefore, samples were examined by energy-dispersive X-ray spectroscopy (EDX) to determine the elemental composition.

2.3.1. Transmission electron microscopy

Transmission electron microscope (TEM) JEM-1011 in the Kazakhstan-Japan Innovation Center of the Kazakh National Agrarian University from JEOL company from Japan was used to study the structure of cobalt oxide. This microscope is equipped with a Morada digital camera (Olympus, Japan). Its main specifications include an accelerating voltage that can be adjusted from 40 to 100 kV, an accurate resolution of 0.3 nm, a linear resolution of 0.14 nm, a LaB6 electron gun, and a magnification range from 100 to 1,000,000. This technique plays an important role in the development and characterization of various nanomaterials, including nanoparticles, and is an integral part of modern nanotechnology and materials science.

2.3.2. Scanning electron microscopy

Quanta 200i 3D scanning electron microscope (FEI, USA) with an accelerating voltage of 30 kV was used to study the structure, size, and morphology of samples ("National Nanotechnology Laboratory of Open Type" Al-Farabi KazNU, Almaty, Kazakhstan). This method allows imaging of the surface, which makes it possible to determine both the structure and size of individual particles. This microscope also has an energy-dispersive X-ray analysis (EDX) system that can determine the chemical composition in the B to U range, with a resolution of 132 eV (Mn Kα). EDX analysis was used to determine the chemical
composition of cobalt oxide nanoparticles and quantitative data.

2.3.3. X-ray diffraction analysis

The crystal structure of the nanomaterial samples was investigated through X-ray diffraction analysis (XRD) using a DRON-3M multipurpose X-ray diffractometer with copper radiation, which has an IBMPC-based control and recording system in digital form. XRD was used to obtain data on the lattice parameters of the substances, to determine their phase composition and the degree of amorphousness of the sample. The samples were examined under the following imaging settings: the X-ray tube voltage reached 30 kV, the tube current was 30 mA, and the goniometer was moved with an angular step of 0.05° 2θ while measuring the intensity up to 1.0. The sample was rotated in its plane at a speed of 60 rpm. Analysis of the X-ray data to determine the angle and intensity of reflection was performed using the program "Fpeak". Phase analysis was performed using the program "PCPDFWIN" and the diffraction data database. The obtained spectra were identified using the JCPDS X-ray database. The apparatus error of X-ray pulse measurement was less than 0.4%.

2.3.4 Small-angle X-ray scattering method

To investigate the nanoscale catalyst particles, small-angle X-ray scattering was applied. SAXS curves were analyzed using a Hecus S3-MICRO diffractometer with a Cu-C radiation filter. The value of the scattering vector modulus $q=\frac{4\pi \sin\Theta}{\lambda}$, where $2\Theta$ is the scattering angle, $\lambda$ is the wavelength of the radiation used, thus $\lambda$ was equal to 1.54 Å and $2\Theta$ was equal to 0.008÷8) was used as the scattering coordinate. The scattering intensity was recorded in the range $q$ from 0.006 to 0.6 Å⁻¹; $q$ is linearly related to the correlation $L\sim 2\pi/q$. The small-angle curves for glycerol and several samples of nanoparticles dissolved in glycerol and obtained by the SAXS method were used to determine the size distribution (radius histogram) in the spherical approximation for nanoparticles.

3. Results and discussion

To establish the chemical and phase composition of the synthesized cobalt oxide nanoparticles, X-ray diffraction analysis was carried out, which showed that in all cases monophasic cobalt oxide with the formula Co₃O₄ was obtained (Fig. 1).

![Fig. 1. Diffractogram of cobalt oxide obtained by solution combustion method of cobalt glycine-nitrate mixture at $\varphi=1.5$.](image)

![Fig. 2. Small-angle curves of glycerol and Co₃O₄ nanoparticles ($\varphi=1$): (a) Co₃O₄ nanoparticles obtained without addition of nitric acid ($\varphi=1$); (b) Co₃O₄ nanoparticles obtained with addition of nitric acid ($\varphi=1$).](image)
The diffractogram shows a characteristic diffraction pattern with a series of distinct peaks corresponding to planes (111), (220), (311), (222), (400), (511) and (440). The diffraction peaks and reflections are consistent with JCPDS card: 00-042-1467 [31]. All reflections are attributed to the typical Co$_3$O$_4$ phase. The monophasic cobalt oxide Co$_3$O$_4$ has a crystal symmetry corresponding to the crystal structure of spinel. The spinel structure is a type of cubic crystal structure with a space group known as Fd3m (face-centered cubic structure), which is also referred to as a "cubic densely packed" structure. In the spinel structure, Co$_3$O$_4$ consists of two different types of cations (Co$^{2+}$ and Co$^{3+}$ cobalt ions) distributed in a specific order within the crystal lattice. This arrangement results in the characteristic symmetry of the Fd3m spinel structure. In the Co$_3$O$_4$ spinel structure, oxygen ions (O$^2-$) form a tightly packed face-centered cubic (FCC) lattice, while cobalt ions occupy both octahedral and tetrahedral positions within this oxygen lattice. The arrangement of cobalt ions within these positions contributes to the unique symmetry of Co$_3$O$_4$ as a spinel. To establish the chemical and phase composition of the synthesized cobalt oxide nanoparticles, X-ray diffraction analysis was carried out, which showed that in all cases monophasic cobalt oxide with the formula Co$_3$O$_4$ was obtained (Fig. 1).

To determine the effect of nitric acid, experiments were carried out without and with the addition of nitric acid to the initial mixture. The obtained samples were investigated by small-angle X-ray scattering. Glycerol was used as a matrix. Small-angle curves for glycerol and Co$_3$O$_4$ nanoparticles (φ=1, without the addition of nitric acid) and Co$_3$O$_4$ (φ=1, with the addition of nitric acid) (Fig. 2a, 2b). The contribution of small-angle scattering of glycerol was subtracted from the curve to determine the size distribution of
nanoparticles (the sphere radius is indicated in the histogram) in the spherical approximation.

As can be seen from the graph of the distribution of cobalt oxide nanoparticles by diameter, 8% of particles have a diameter close to 5 nm, about 9–10% of nanoparticles have diameters up to 4 nm, a diameter of 5–6 nm corresponds to 1.5% of the studied nanoparticles, the contribution of the remaining nanoparticles is less 1%. The remaining fraction of nanoparticles has dimensions much larger than the maximum permissible diameter, so their contribution is not taken into account and is not displayed on the graph.

As can be seen from the distribution plot of \( \text{Co}_3\text{O}_4 \) nanoparticles obtained without the addition of nitric acid (Fig. 3), the main fraction of particles has a diameter up to 6 nm, but particles with diameters up to 25 nm are also present.

As can be seen from the distribution graph of \( \text{Co}_3\text{O}_4 \) nanoparticles obtained with the addition of nitric acid (Fig. 4), the bulk of the particles have diameters up to 8 nm. In this case, the nanoparticles have diameters up to 10 nm and there are no particles with larger diameters. The results obtained showed that the addition of nitric acid allows obtaining more monodisperse particles with a small spread.

As can be seen from the obtained scanning and transmission electron microscope images (Fig. 5), for cobalt oxide particles at stoichiometric fuel-to-oxidizer ratio \( \varphi=1 \), the particle size range is from 23 to 60 nm, and agglomerates larger than 500 nm are also present. The formation of agglomerates can be attributed to high-temperature fluctuations during the self-ignition of the mixture. For the cobalt oxide nanoparticles at a ratio of \( \varphi=1.5 \), the particle size ranges from 20 to 65 nm, without large agglomerates.

As can be seen from the obtained scanning and transmission electron microscope images (Fig. 5), for cobalt oxide particles at stoichiometric fuel-to-oxidizer ratio \( \varphi=1 \), the particle size range is from 23 to 60 nm, and agglomerates larger than 500 nm are also present. The formation of agglomerates can be attributed to high-temperature fluctuations during the self-ignition of the mixture. For the cobalt oxide nanoparticles at a ratio of \( \varphi=1.5 \), the particle size ranges from 20 to 65 nm, without large agglomerates.
A comparison of the two samples based on SEM and TEM images illustrates the positive effect of fuel addition above the stoichiometric ratio. The reaction of the fuel with the oxidizer leads to the decomposition of the initial components with the formation of gaseous products that lead to further dispersion of the final product. Thus, the obtained results confirm the efficiency of the synthesis of Co$_3$O$_4$ nanoparticles by the solution combustion method. Changing the composition of the initial mixture can significantly change the morphology of the obtained product and clearly illustrates the possibility of controlled synthesis. The synthesized Co$_3$O$_4$ nanoparticles are perspective materials for application in gas sensors. Co$_3$O$_4$ as a transition metal oxide has chemical, phase, and structural stability, which allows increasing the temperature if necessary, and high electrical conductivity, which allows recording the chemoresistive response that occurs during the redox reactions of metal oxide with the detected gas [2, 32]. For this purpose, in metal-oxide gas sensors, the sensitive material Co$_3$O$_4$ is heated at a certain temperature. The flow of electricity within this material depends on the number of free electrons. When the sensing material is in clean air, the oxygen (O$_2$) in the atmosphere adsorbs on the surface of the sensing material, attracts free electrons, and keeps the electrons on the surface as ions. This leads to an increase in the resistance of the sensor, resulting in a decrease in the flow of electrons within the sensing material. In the presence of reducing gases such as methane or propane, these gases interact with the adsorbed oxygen, releasing bound electrons within the sensing material. This results in a decrease in the resistance of the sensor, allowing more electrical current to flow. As the concentration of reducing gases increases, the resistance of the sensor decreases further, allowing even more electrical current to flow. According to the sensitivity characteristics of Co$_3$O$_4$, there is a certain relationship between the sensor resistance and the gas concentration in the atmosphere, which provides information about the concentration of pollutants in the air [33, 34].

In Table 2, examples of Co$_3$O$_4$ applications in gas sensors for the determination of a wide range of gases are shown. The main parameters of Co$_3$O$_4$ - based gas sensors are given. Successful results of Co$_3$O$_4$ application in gas sensors show the prospect and relevance of the development and optimization of methods for obtaining cobalt oxide nanoparticles with controlled parameters of morphology and structure to obtain stable and repeatable results.

### 4. Conclusion

Co$_3$O$_4$ nanoparticles were obtained by solution combustion method as a result of the exothermic redox reaction of cobalt nitrate hexahydrate.
(Co(NO$_3$)$_2$·6H$_2$O) and glycine (C$_2$H$_5$NO$_2$). The effect of the addition of nitric acid and the fuel: oxidizer ratio on the structure and dispersibility of cobalt oxide nanoparticles was investigated. The positive effect of the addition of nitric acid was established. The addition of nitric acid allows to obtain of cobalt oxide nanoparticles with a more uniform distribution of particles, which is proved by SAXS investigations. Moreover, it was found that the use of a fuel-rich mixture (φ=1.5) leads to the formation of more homogeneous Co$_3$O$_4$ crystals with a size of 23–60 nm. Thus, by changing the composition and ratio of components of the initial mixture it is possible to qualitatively change the structure and morphology of the final product. Co$_3$O$_4$ nanoparticles obtained by solution combustion method is a potential and perspective sensitive material for gas sensors, while manipulation of the synthesis process allows to obtain nanoparticles with a given structure and properties. Thus, it allows Co$_3$O$_4$ nanoparticles-based gas sensors to be used further in the military, metallurgy, and oil-producing industries, which expands the scope of potential application of cobalt oxide nanoparticles obtained by solution combustion method.

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Сұйық фазалы жану эдісімен алынған Co₃O₄ нанобөлшектерінің морфологиялық ерекшеліктері

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АННОТАЦИЯ

Жаһандық экологиялық дағдарыс қоршаған орта параметрлерін бақылау жəне талдау құрал-діріліп отырады. Газ датчиктері, азық материалдары жəне олардың құрылы-мын мұқият таңдау мен бақылауға байланысты. Бұған газға сезімтал қосылыстарды оңтайландыру, азық материалдарды пайдалану жəне заттарды сезімтал жəне жылдам анықтау технологияла-рын əзірлеу кіреді. Осы мақсат үшін перспективалы қосылыстардың бірі – сұйық-фазалық жану əдісімен алынған Co₃O₄ бөлшектері. Бұл əдіс қа-рапайымдылығымен ерекшеленеді жəне оның куралымы мен құрылысын өзін-өзі таңдауға мүмкіндік береді. Бұл қосылыстардың морфологиялық ерекшеліктерін сізге белгілі."
ной работе частицы Co₃O₄ были синтезированы из смеси нитрата кобальта и глицина с добавлением азотной кислоты методом жидкофазного горения. Было исследовано влияние добавления азотной кислоты и соотношение горючего к окислителю на морфологические характеристики получаемого оксида кобальта. Результаты СЭМ, ПЭМ, РФА и МУРР анализов подтверждают, что добавление азотной кислоты и использование топливо-богатой смеси приводят к образованию наночастиц с меньшим разбросом по диаметру и более стабильными показателями.

Ключевые слова: металл-оксидные наноматериалы, наночастицы Co₃O₄, метод жидкофазного горения, экзотермическая окислительно-восстановительная реакция, газовые сенсоры.