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*Vasyl Stefanyk Carpathian
National University*

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Iryna Starko[1], Nazarii Danyliuk[1], Liubov Soltys[2]

Catalytic activity of Gd-doped Ni-Co ferrites in Fenton-like degradation of oxytetracycline

[1]*Educational and Scientific Center of Material Science and Nanotechnology, Vasyl Stefanyk Carpathian National University, Ivano-Frankivsk, Ukraine,* iryna.starko@cnu.edu.ua
[2]*Department of Chemistry, Vasyl Stefanyk Carpathian National University, Ivano-Frankivsk, Ukraine,* liubov.soltys@cnu.edu.ua

In this study, the efficiency of Gd-doped Ni-Co ferrites for the adsorption-catalytic removal of oxytetracycline (OTC) from aqueous solutions was evaluated. A series of ferrites with the composition $Ni_{0.5}Co_{0.5}Gd_xFe_{2-x}O_4$ (x = 0; 0.01; 0.25; 0.5) were synthesized by the sol-gel autocombustion method, and their catalytic activity was evaluated in the presence of 30 mM H_2O_2 and under the action of electromagnetic heating (EMH). The $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ sample demonstrated the highest OTC degradation efficiency, achieving 100% removal of 25 mg/L OTC in 30 min, with a reaction rate constant of 0.1416 min^{-1}. The $Ni_{0.5}Co_{0.5}Gd_{0.025}Fe_{1.975}O_4$ and $Ni_{0.5}Co_{0.5}Gd_{0.05}Fe_{1.95}O_4$ samples showed significantly lower activity with constants of 0.0446 and 0.0444 min^{-1}, respectively, and the degree of OTC removal decreased to 67-80% depending on the concentration. Electromagnetic field generates heat locally in the catalyst, which accelerates the formation of reactive oxygen species (•OH), increases the reaction rate constant and contributes to the effective destruction of the antibiotic. Excessive Gd doping leads to reduced catalyst efficiency. Increasing the OTC concentration reduces the degradation rate, probably by blocking the active centers of the catalyst. The obtained results demonstrate the promising potential of $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ in combination with H_2O_2 and EMH for the rapid and effective removal of antibiotics from water, which is important for environmental protection.

Keywords: Ni-Co ferrite; gadolinium; oxytetracycline; Fenton-like degradation; spinel; water treatment.

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Introduction

The intensive use of pharmaceuticals in medicine, veterinary medicine and agriculture in recent years has led to their accumulation in the environment. These recalcitrant compounds are not effectively removed by traditional treatment methods from wastewater and can accumulate in surface water, soils and bottom sediments, and then migrate to groundwater and drinking water. Long-term exposure to pharmaceutical residues in low concentrations negatively affects biota, in particular, causing endocrine disruption, chronic toxicity and the development of antibiotic resistance. Therefore, the tetracycline antibiotics pose a particular environmental threat. Oxytetracycline (OTC) is one of the most widely used antibiotics of the tetracycline series, which determines its systematic presence in wastewaters. Being widely used, OTC poses a huge threat to the environment [1]. It causes the adaptation and spread of antibiotic-resistant microbial populations [2], which poses a threat to human health. Due to its complex polyfunctional structure and high chemical stability, OTC is practically not removed by traditional biological treatment methods, which stimulates the search for effective approaches to its chemical destruction [3–5].

Among modern methods of water purification from persistent organic pollutants, advanced oxidation processes (AOPs) are of particular importance. AOPs are based on the formation of highly reactive oxygen radicals, primarily •OH. The classical Fenton reaction, based on the interaction of Fe^{2+} ions with hydrogen peroxide, demonstrates high oxidizing ability, but has significant limitations associated with a narrow operating pH range, formation of iron-containing precipitate and the difficulty of reusing the catalyst [6,7]. An alternative to homogeneous systems are heterogeneous Fenton-like catalysts, in particular transition metal oxide materials, capable of catalyzing the decomposition of H_2O_2 or peroxomonosulfate to form active radicals under a wider range of conditions [8].

In this context, spinel-structured ferrites (MFe_2O_4, where M = Ni, Co, Cu, Mn, or a combination of these metals) have attracted considerable attention due to their high chemical stability, the presence of multiple redox pairs, and magnetic properties that facilitate catalyst separation after the reaction [9–11]. Special attention is paid to the modification of heterogeneous ferrites by doping with rare earth elements, such as Gd and La [12]. Doping spinel oxides with Gd^{3+} ions changes their electronic structure and energy gap, creates structural defects, increases number of active surface centers and improves the separation of charge carriers, which contributes to the generation of highly reactive •OH radicals in Fenton-like systems [13,14].

In particular, Gd^{3+}-doped spinel ferrites showed a significant increase in activity in the photocatalytic degradation of organic pollutants under visible light. The work [15] has shown that gadolinium-containing Mn-Co ferrites possess high photocatalytic activity in the degradation of the antibiotic tetracycline under the action of visible light. Under optimal conditions, the efficiency of tetracycline degradation reached 94.93% within 120 minutes of irradiation. The increase in photocatalytic activity is associated with the redistribution of metal ions in the octahedral cationic positions of the spinel structure, which contributes to the modification of the electronic structure and the improvement of the separation of photogenerated charge carriers.

Also, doping ferrites with Gd^{3+} ions significantly modifies their physicochemical characteristics. In particular, the introduction of Gd^{3+} contributes to an increase in the specific surface area and the formation of a mesoporous structure, which improves the adsorption interaction with pollutant molecules. In addition, the change in composition affects the pH_{PZC} value, which determines the nature of electrostatic interactions between the ferrite surface and various ionized forms of organic compounds in the aqueous environment [16,17]. The substitution of Fe ions by Gd ions reduces the crystallite size, increases the specific surface area and surface charge, which improves adsorption properties towards OTC [18].

Thus, the synthesis and testing of Gd-containing Ni-Co ferrites as Fenton-like catalysts in degradation of oxytetracycline is highly relevant for the purification of aqueous environments from pharmaceutical pollutants. This work describes the effect of Gd doping on the Fenton-like activity of Ni-Co ferrites synthesized by the sol-gel autocombustion method.

I. Experimental

1.1. Materials and methods

Gd-doped Ni–Co ferrites with the composition $Ni_{0.5}Co_{0.5}Gd_xFe_{2-x}O_4$ (x=0; 0.01; 0.25; 0.5) were synthesized by the sol–gel auto-combustion method using polyvinyl alcohol as the fuel for the combustion reaction. The synthesis procedure is described in detail in the paper [19]. In brief, stoichiometric quantities of metal salts (nitrates and acetates) were dissolved in a minimal volume of distilled water. A polyvinyl alcohol solution was prepared separately and slowly introduced into the metal salt solution at a 1:2 ratio. The resulting solution was heated until gelation, followed by autoignition and combustion. The formed magnetical powder was thoroughly washed and dried.

1.2 Testing of Fenton-like catalytic activity

Fenton-like catalytic properties of Gd-doped ferrites were estimated using antibiotic oxytetracycline (OTC) as a model pollutant. A typical procedure for the OTC degradation experiment was performed as follows: 120 mg of Gd-doped Ni-Co ferrites were added to 40 mL of OTC solution with a concentration of 25 mg/L. The degree of antibiotic adsorption on the surface of the synthesized catalysts was determined after 30 min of mixing. The experiments were performed with and without electromagnetic heating (EMH) [20]. Thermal energy generated by electromagnetic heating enhances the catalytic degradation of OTC. This localized heating at the catalyst surface increases the reaction rate by accelerating molecular collisions and facilitating the formation of reactive oxidative species (e.g., •OH radicals). As a result, the activation energy barrier is effectively reduced, leading to faster and more efficient degradation of OTC compared to conventional heating conditions [21,22]. Recording of OTC decomposition was started after the addition of 30 mM H_2O_2. During the degradation process, a 2 mL aliquot was taken over 60 min and filtered using a 0.35 μm syringe filter. The solution absorbance at the wavelength of 365 nm was measured using a ULAB 102-UV spectrophotometer. The calibration line has the correlation coefficient R^2 greater than 0.999. The residual OTC concentration was calculated using the equation:

$$\mathrm{C(OTC)} = \mathrm{A_x}/0.0141, \quad (1)$$

where C(OTC) is the OTC concentration (mg/L); A_x is the absorbance of the OTC solution at 356 nm. The initial concentration of OTC was varied in the range of 25-75 mg/L. The degradation efficiency was expressed by the following equation:

$$\text{OTC degradation (\%)} = \frac{C_0 - C_t}{C_0} \times 100, \quad (2)$$

where C_0 and C_t (mg/L) are the initial concentration of OTC and the concentration of OTC at a certain time t, respectively. The decrease in OTC concentration was fitted using the first order reaction kinetics:

$$\ln([\mathrm{OTC}]_t/[\mathrm{OTC}]_0) = -k[\mathrm{OTC}]\cdot t, \quad (3)$$

where k is the reaction rate constant, t is the reaction time, and $[OTC]_t$ and $[OTC]_0$ are the OTC concentrations at time t and 0 min, respectively.

II. Results and discussion

2.1. The effect of induction heating.

The catalytic activity of Gd-doped Ni-Co ferrites was determined by measuring the removal of OTC from aqueous solution. The adsorption-desorption equilibrium was reached after 30 min and remained unchanged (Fig. 1). The adsorption capacity values are quite high for all the synthesized samples. The highest OTC adsorption of 66.5 and 65.4% was recorded for the $Ni_{0.5}Co_{0.5}Fe_2O_4$ and $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ samples, respectively. The $Ni_{0.5}Co_{0.5}Gd_{0.025}Fe_{1.975}O_4$ and $Ni_{0.5}Co_{0.5}Gd_{0.05}Fe_{1.95}O_4$ samples showed lower efficiency of adsorptive OTC removal of 36.1 and 38.9%, respectively. Electromagnetic heating was used as an effective method of catalyst activation [20]. As shown in Fig.1-a, the OTC degradation rate using the $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ sample reaches 100% after 30 min of treatment with 30 mM H_2O_2 and the action of EMH. Without the influence of EMH, the OTC degradation rate is 93.5%, under the same conditions. The semi-logarithmic representation of the kinetic curves of OTC degradation is shown in Fig.1-b. The $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ catalyst showed the highest first-order reaction rate constant of 0.1416 min^{-1} (Fig.1-c). For the $Ni_{0.5}Co_{0.5}Gd_{0.025}Fe_{1.975}O_4$ and $Ni_{0.5}Co_{0.5}Gd_{0.05}Fe_{1.95}O_4$ samples, the reaction rate constants are only 0.0446 and 0.0444 min^{-1} under the action of EMH, respectively. It looks like as the structure of Gd-doped Ni-Co ferrites would allow for the effective destruction of OTC by accelerating the separation of generated charge carriers. However, overdoping Ni-Co ferrite with Gd ions leads to a decrease in the efficiency of OTC degradation. Compared with the optimal $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ ferrite, the degradation rate of $Ni_{0.5}Co_{0.5}Gd_{0.025}Fe_{1.975}O_4$ (after 60 min, without EMH) is reduced by 19.6% (Fig.1-d).

2.2. Effect of initial OTC concentration.

Fig. 2a-c shows the efficiency of the adsorption and catalytic degradation of OTC in the concentration range from 25 to 75 mg/L. The high adsorption properties of Gd-doped Ni-Co ferrites played a key role in the removal of OTC. The catalyst dose was 3 g/L to achieve an effective effect of EMN on the OTC removal. (Fig. 2a-c). The $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ sample showed the highest efficiency for adsorptive removal of OTC. The adsorption degree was 66.5, 77.4 and 63.4% during the removal of 25, 50 and 75 mg/L of OTC, respectively. The addition of 30 mM H_2O_2 allowed to increase the removal efficiency of 25, 50 and 75 mg/L OTC to 93.8, 88.8 and 80.4%, respectively. This indicates that the removal of OTC by

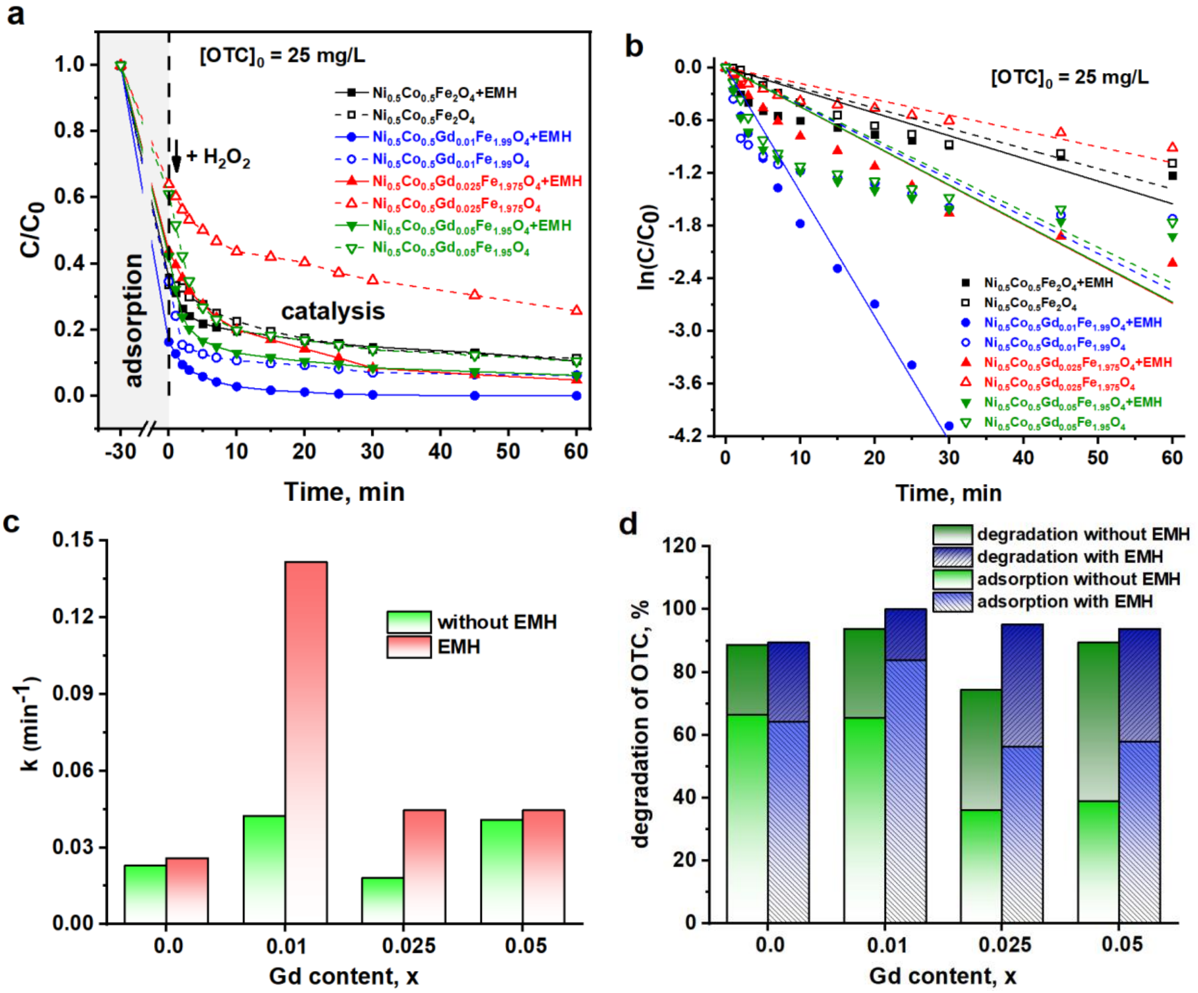

Fig. 1. The effect of EMH on the OTC removal. (a) Decrease in OTC concentration during the adsorption equilibrium stage and the following Fenton-like oxidation. (b) Pseudo-first order kinetic model. (c) The values of the kinetic rate constant *vs.* Gd content. (d) The OTC removal (in %) *vs.* Gd content. Conditions: [catalyst] = 3 g/L; [H_2O_2] = 30 mM; T = 20 °C.

Gd-doped Ni-Co ferrites is due to their ability to generate reactive oxygen species that destroy the adsorbed pollutant. The increase in the initial concentration of OTC led to a decrease in the efficiency of the catalytic removal. The adsorption–catalytic efficiency for OTC at concentrations ranging from 25 to 75 mg/L was analyzed using kinetic curves, as shown in Fig. 2d-f. For the $Ni_{0.5}Co_{0.5}Gd_{0.025}Fe_{1.975}O_4$ sample, the rate constant of the OTC decomposition reaction decreases by a factor of 2 from 0.0446 min^{-1} to 0.0229 min^{-1} with an increase in the OTC concentration from 25 to 75 mg/L (Fig. 2g). The highest reaction rate constant of 0.1416 min^{-1} was shown by the $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ sample during the removal of 25 mg/L of OTC. However, with an increase in the OTC concentration to 75 mg/L, a significant decrease in the reaction rate constant to 0.0128 min^{-1} is observed. This is primarily due to the blocking of the active centers of the catalyst by an excess number of OTC molecules that were adsorbed on its surface for 30 min. The unsubstituted $Ni_{0.5}Co_{0.5}Fe_2O_4$ sample exhibited almost the same reaction rate constants of 0.0258, 0.0286 and 0.02532 min^{-1} during the catalytic removal of 25, 50 and 75 mg/L of OTC. During the removal of 25 mg/L of OTC, the lowest efficiency (90.5%) was shown by the $Ni_{0.5}Co_{0.5}Fe_2O_4$ sample. However, when removing 75 mg/L of OTC, the lowest efficiency (67.1%) was shown by the $Ni_{0.5}Co_{0.5}Gd_{0.05}Fe_{1.95}O_4$ sample (Fig. 2h). According to the presented results, doping Ni-Co ferrites with Gd ions allows to significantly increase the efficiency of adsorption-catalytic removal of the OTC antibiotic from aqueous solution. In particular, the system $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4 + H_2O_2$ + EMH showed excellent activity in the decomposition of OTC with the removal efficiency of 100%.

The obtained results are in line with the literature reports. For example, the catalytic destruction of OTC in a fixed-bed reactor was investigated in the presence of 20 mM H_2O_2 solution using hematite (α-Fe_2O_3) as the catalyst [23]. The degradation percentage of 2 mg/L OTC was found to be 75%. Peroxymonosulfate (PMS) activated by $FeVO_4$ nanorods was successfully used for the degradation of 20 mg/L OTC [24]. OTC was almost completely destroyed in 40 minutes, and the reaction rate constant was 0.1073 min^{-1}. In particular, the $FeVO_4$ nanorods demonstrated good stability and reusability.

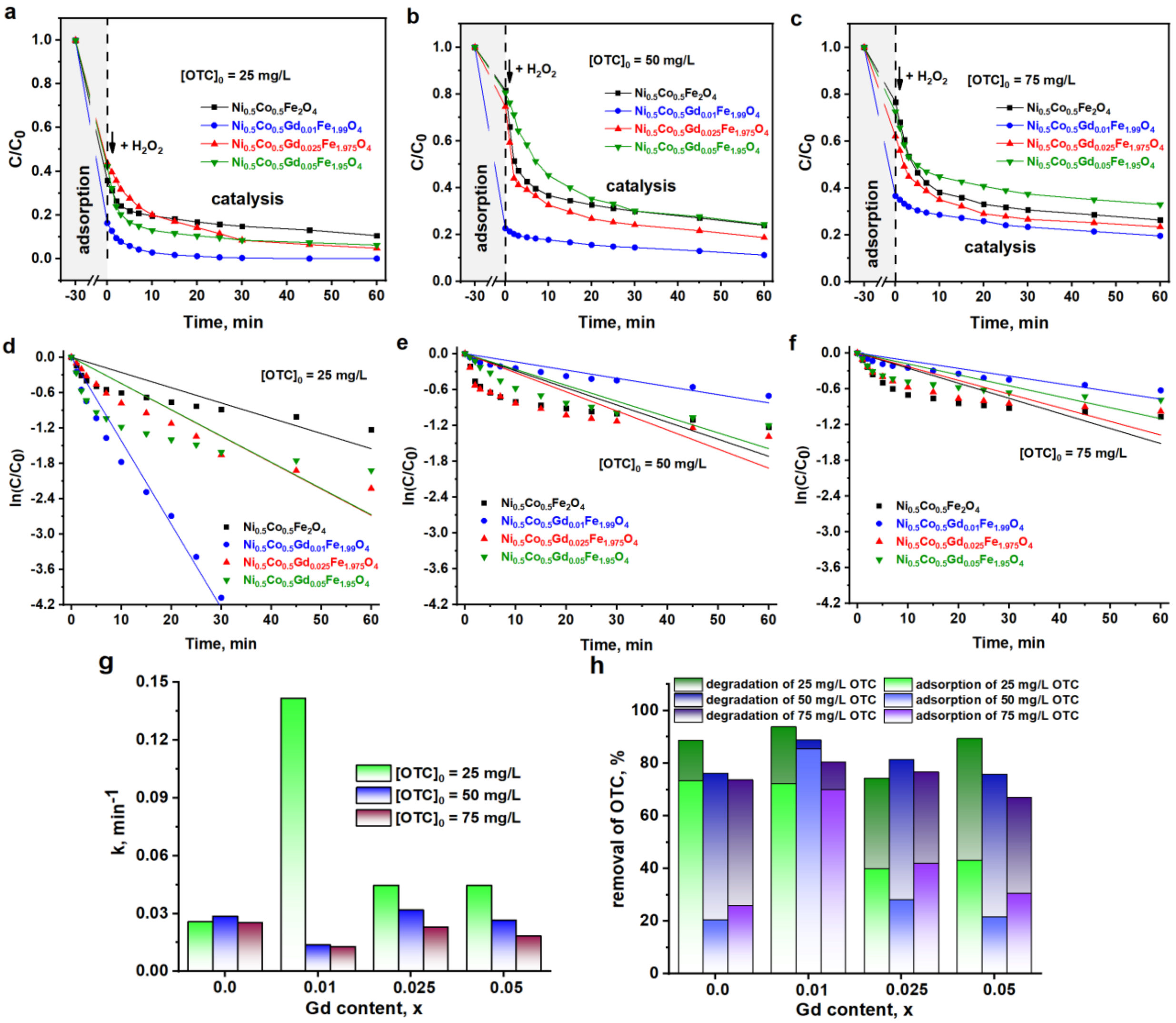

Fig. 2. The effect of Gd content in the Ni-Co ferrites on the oxytetracycline removal. (a–c) Decreasing in OTC concentration during the adsorption equilibrium stage and the following Fenton-like oxidation at different initial OTC concentrations. (d-f) Pseudo-first-order kinetic model. (g) The magnitudes of the kinetic rate constant *vs.* Gd content. (h) The OTC removal (in %) *vs.* Gd content. Conditions: [catalyst] = 3 g/L; [H_2O_2] = 30 mM; T = 20 °C.

Conclusions

The study demonstrated that the catalytic activity of Gd-doped Ni-Co ferrites is largely dependent on the Gd content and the concentration of oxytetracycline (OTC). The optimal $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ sample achieved 100% OTC removal at a concentration of 25 mg/L in 30 min in the presence of 30 mM H_2O_2 and under electromagnetic heating, with a rate constant of 0.1416 min^{-1}. Samples $Ni_{0.5}Co_{0.5}Gd_{0.025}Fe_{1.975}O_4$ and $Ni_{0.5}Co_{0.5}Gd_{0.05}Fe_{1.95}O_4$ exhibited significantly lower efficiencies of 80.4% and 67.1% OTC removal, respectively, with rate constants of 0.0446 and 0.0444 min^{-1} under the action of EMH. The unsubstituted $Ni_{0.5}Co_{0.5}Fe_2O_4$ provided 90.5% removal at 25 mg/L OTC with a rate constant of 0.0258 min^{-1}. With increasing OTC concentration to 75 mg/L, the removal efficiency for all samples decreased, and the rate constants dropped to 0.0128-0.0229 min^{-1}, which is explained by the blocking of active sites by OTC molecules. The results confirm that the control of Gd content and experimental conditions is critical to ensure high catalytic activity. The $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ + H_2O_2 + EMH system demonstrates significant potential for practical water purification.

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Ірина Старко[1], Назарій Данилюк[1], Любов Солтис[2]

Каталітична активність Gd-вмісних Ni-Co феритів у фентонподібній деградації окситетрацикліну

[1]Навчально-науковий центр хімічного матеріалознавства та нанотехнологій, Карпатський національний університет імені Василя Стефаника, Івано-Франківськ, Україна, iryna.starko@cnu.edu.ua
[2]Кафедра хімії, Карпатський національний університет імені Василя Стефаника, Івано-Франківськ, Україна, liubov.soltys@cnu.edu.ua

У цьому дослідженні було оцінено ефективність Gd-вмісних Ni-Co феритів для адсорбційно-каталітичного видалення окситетрацикліну (ОТЦ) з водних розчинів. Серію феритів складу $Ni_{0.5}Co_{0.5}Gd_xFe_{2-x}O_4$ (x=0; 0.01; 0.25; 0.5) було синтезовано методом золь-гель самозаймання, а їхню каталітичну активність оцінювали у присутності 30 мМ H_2O_2 та під дією електромагнітного нагрівання (ЕМН). Зразок $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ продемонстрував найвищу ефективність деградації ОТЦ, досягнувши 100% видалення 25 мг/л ОТЦ за 30 хв, з константою швидкості реакції 0.1416 хв$^{-1}$. Зразки $Ni_{0.5}Co_{0.5}Gd_{0.025}Fe_{1.975}O_4$ та $Ni_{0.5}Co_{0.5}Gd_{0.05}Fe_{1.95}O_4$ мали значно нижчу активність з константами 0.0446 та 0.0444 хв$^{-1}$ відповідно, а ступінь деградації ОТЦ знизився до 67-80% залежно від концентрації. Електромагнітне поле генерує тепло локально в каталізаторі, що прискорює утворення активних форм кисню (•OH), збільшує константу швидкості реакції та сприяє ефективному руйнуванню антибіотика. Надмірне легування Gd призводить до зниження ефективності каталізатора. Збільшення концентрації ОТС знижує швидкість деградації, ймовірно, шляхом блокування активних центрів каталізатора. Отримані результати демонструють перспективний потенціал $Ni_{0.5}Co_{0.5}Gd_{0.01}Fe_{1.99}O_4$ у поєднанні з H_2O_2 та ЕМН для швидкого та ефективного видалення антибіотиків з води, що важливо для захисту навколишнього середовища.

Ключові слова: Ni-Co ферит; Гадоліній; окситетрациклін; фентонподібна деградація; шпінель; очищення води.