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Influence of UV radiation on the catalytic activity of nanosized cobalt ferrite in the oxidative degradation reaction of dinitrophenol

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Abstract

Due to their multi-functionality, spinel ferrites, both doped and undoped, are promising materials for a wide range of practical applications, including catalysis, sustainable production of hydrogen and CO₂ deposition, electronic and magnet devices, as well as antibacterial agents. Recently, nanosized ferrites have been actively tested as catalysts in Fenton-like processes of deep oxidative degradation of organic substances in order to purify waste waters of different dyes, phenol and its derivatives, and antibiotics. The goal of this work was to establish the catalytic activity of CoFe₂O₄ nanopowder synthesised using citrate combustion in the reaction of oxidative degradation of 2,4-dinitrophenol upon the activation of the process with UV radiation.

Using citrate combustion, we synthesised the impurity-free nanopowder of CoFe₂O₄ cobalt ferrite with the average size of particles of about 70 nm and a pronounced agglomeration of particles. The cobalt spinel was tested as a catalyst of Fenton-like reaction of oxidative degradation of 2,4-dinitrophenol with UV radiation of $\lambda = 270$ nm. This process was differentiated with the sorption of dinitrophenol on a nanosized catalyst.

The degree of degradation of 2,4-dinitrophenol in a Fenton-like reaction without the CoFe₂O₄ catalyst was 14 %, while in the presence of a nanosized catalyst it increased up to 80 %. The effective oxidative degradation of the pollutant was performed in a less acidic environment as compared to a classic Fenton process with a rather large initial concentration of dinitrophenol. This allowed considering the nanosized CoFe₂O₄ as a promising catalyst of Fenton-like of waste waters purification through deep oxidative degradation of toxins.

Keywords: Catalysis, Nanosized ferrites, Wastewater treatment, Ultraviolet radiation

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1. Introduction

Today, the pollution of natural water with waste waters of industrial and agricultural productions has a significant antropogenic impact. Among the most widespread toxic agents entering the surface waters from oil refining, wood chemical, coke chemical, aniline dye, textile, and hydrolysis industrial enterprises are phenols and their derivatives. Nitrophenol compounds, such as nitrocece and dinitrophenol, are especially toxic. Nevertheless, dinitrophenols are rather widely used in agriculture due to their pronounced simultaneous herbicidal, fungicidal, and insecticidal effect, and they also act as precursors in the production of dyes and anticeptics for wood. It should be noted that pesticides of the dinitrophenol type have a good cumulation ability, which makes the task of their inactivation even more relevant [1].

It is considered that the most reliable and effective method is the complete oxidative degradation of phenol nitro-derivatives to non-toxic substances. In this regard, Fenton processes are of great interest, in the course of which hydrogen peroxide, by decomposing under the influence of a catalyst, acts as a source of stronger oxidants, primarily hydroxyl radicals with a redox potential from +2.0 to +2.8 V depending on pH [2, 3]. Ferriferrous materials based on mesoporous silicon dioxide, zeolites, and others are used as heterogeneous Fenton catalysts [4, 5].

Nanosized spinel ferrites MeFe_2O_4 [6–10] seem to be promising in this regard as their catalytic activity significantly increases due to a great specific surface area and ability of activation with ultraviolet radiation.

Spinel ferrites are already being used in a number of “green chemistry” technologies, such as wastewater treatment [11–13], carbon deposition [14], and antimicrobial treatment [15]. In case of photochemical processes due to a combination of radiation with the action of different oxidants, the most widespread of which is hydrogen peroxide, and catalysts, the redox potential of the system is considerably increased, and, as a result, the degree of oxidative degradation also rises. Thus,

the goal of this work was to establish the catalytic activity of CoFe_2O_4 nanopowder synthesised using citrate combustion in the reaction of oxidative degradation of 2,4-dinitrophenol upon the activation of the process with UV radiation.

2. Experimental

The synthesis of ferrite-spinel CoFe_2O_4 using the citrate combustion method was carried out according to [16]. Phase composition of the nanopowder was determined by X-ray diffractometry (Empyrean BV diffractometer with Cu anode ($\lambda = 1.54060$ nm)). The scanning was performed within a range of angles $2\theta = 10\text{--}80^\circ$ with a step of 0.0200. The JCPDC database [17] was used to identify the phases. The size and morphology of CoFe_2O_4 particles were determined by transmission electron microscopy (TEM, CarlZeiss Libra-120 transmission electron microscope). Quantitative elemental analysis was performed on a JSM-6380LV JEOL scanning electron microscope with an INCA 250 microanalysis system.

The catalytic activity of the cobalt ferrite nanopowder was studied on a reaction of 2,4-dinitrophenol (DNP) oxidation with hydrogen peroxide. To do this, we prepared a solution containing 0.084 g/l of 2,4-dinitrophenol and 10 wt. % hydrogen peroxide. pH of the solution, which was 4.4, was maintained by an acetate buffer. After that, 0.25 g of cobalt ferrite was added to a series of solution samples with a volume of 15 ml, and the concentration of DNP was measured at certain periods after the beginning of the reaction. Control measurements of the concentrations of DNP solutions without a catalyst were performed in the same manner. The experiment was conducted with daylight and ultraviolet radiation with $\lambda = 270$ nm (Nuobi UV lamp, 20 W). The DNP concentration was determined using photocolormetry (KFK-3-01 photocolormeter). The analytical wavelength for 2,4-dinitrophenol was 364 nm. The degree of DNP degradation was calculated by formula (1):

$$W = \frac{C_0 - C_t}{C_0} \cdot 100 \%, \quad (1)$$

where W is the degree of degradation %, C_0 is the concentration of the dye at the initial moment of time, and C_t is the concentration of the dye at the present moment of time.

To differentiate the catalytic oxidative degradation of DNP and its sorption on a nanosized catalyst, we performed an experiment using the above-described method without adding H_2O_2 oxidant to the solutions. In this case, oxidative degradation of DNP was not performed, while decolorisation of the solutions was only determined by the sorption of the pollutant on cobalt ferrite. The duration of static sorption was 2 hours. The sorption capacity of cobalt ferrite was determined using equation (2):

$$A = \frac{(C_0 - C) \cdot V}{m}, \quad (2)$$

where C_0 is the initial concentration of the DNP solution, mol/l; C is the concentration of DNP after a certain time after the beginning of the reaction, mol/l; V is the volume of the adsorbate solution, l; m is the weight of cobalt ferrite, g.

The validity of the results was established using methods of statistical analysis. The law of result distribution was determined using the Kolmogorov-Smirnov test. We determined the statistical significance by the Student's test.

3. Results and discussion

According to XRD results (Fig. 1), cobalt ferrite powder synthesised by citrate combustion did not contain impurities, and all reflections in the diffraction pattern belonged to $CoFe_2O_4$ spinel (JCPDC, card 22-1086).

The average size of coherent scattering regions (CSRs) of $CoFe_2O_4$ particles calculated using the Debye-Scherrer formula [18] was 61 ± 4 nm.

The average values of the weight and atomic percentages of the elements Co, Fe, and O according to the data of energy dispersion analysis are presented in Table 1, and the results correlate with the expected chemical composition.

According to TEM, cobalt ferrite powder (Fig. 2A) was highly agglomerated, which could be associated with specific features of the method of synthesis. Additional ultrasound dispersion of the nanopowder in an ultrasonic bath VU-09-“Ya-FP”-0 for 20 min allowed reducing

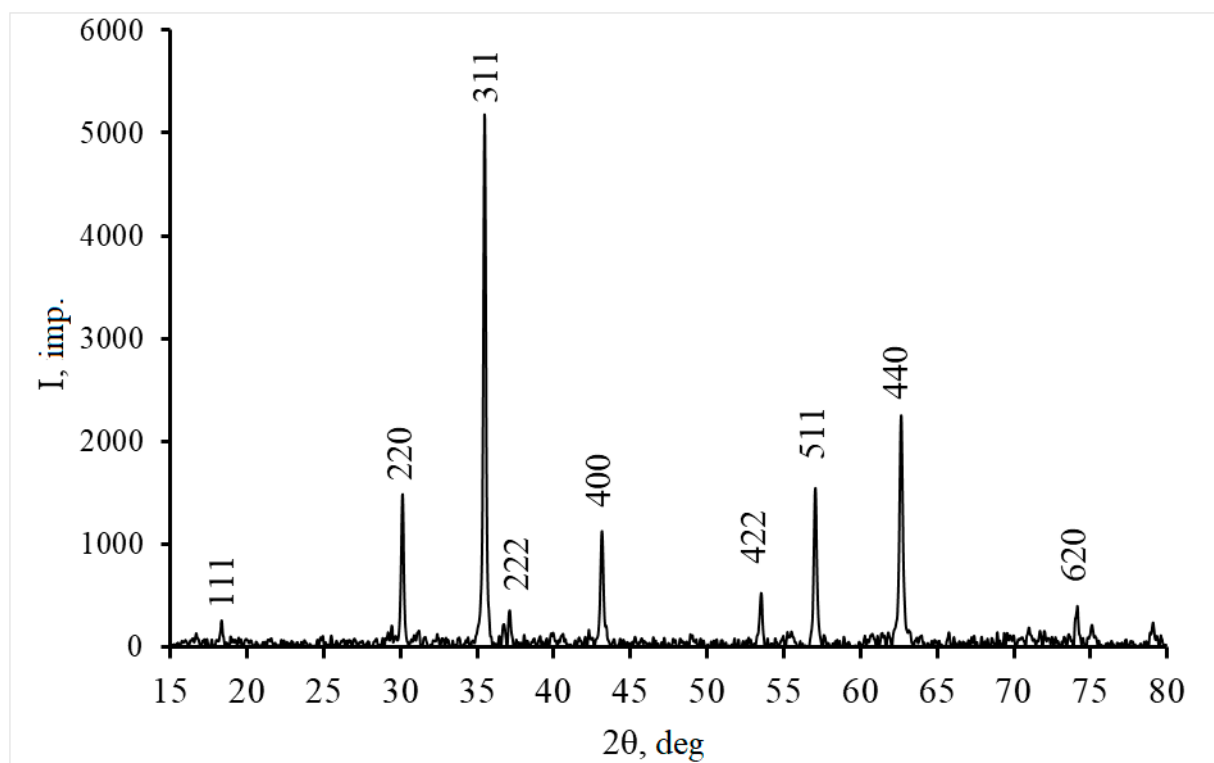


Fig. 1. Diffraction pattern of the $CoFe_2O_4$ powder synthesised by the citrate method

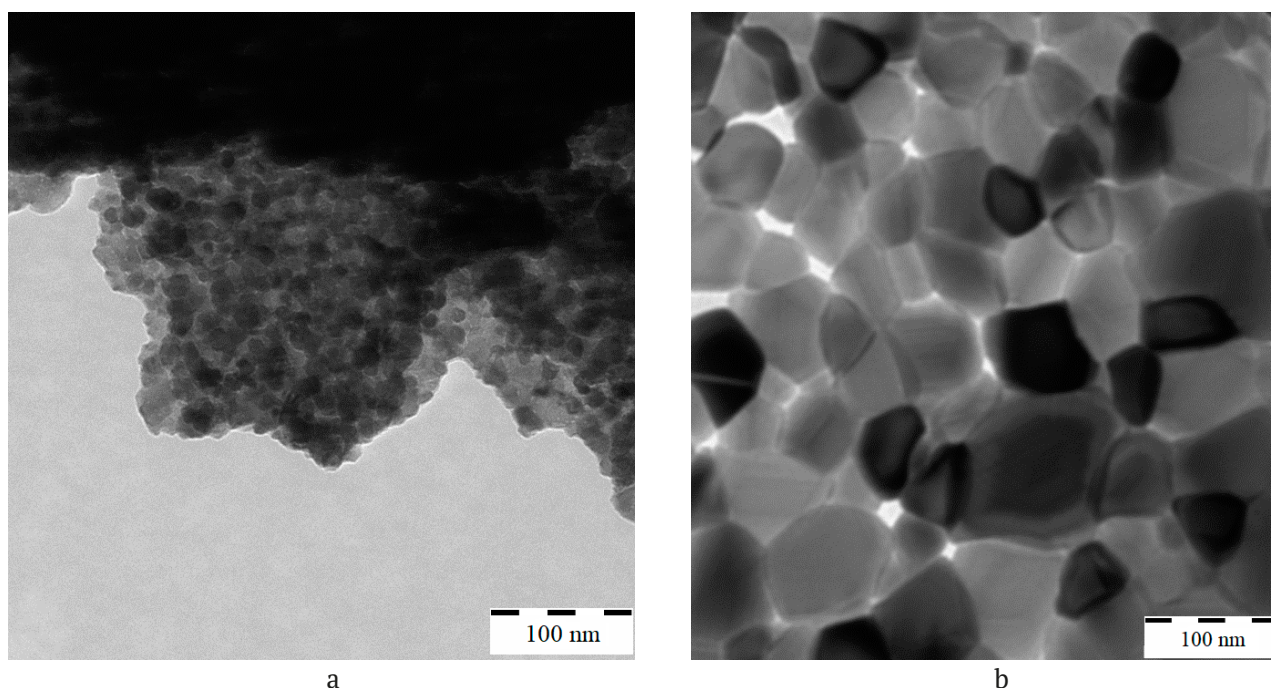


Fig. 2. TEM images of the CoFe_2O_4 sample before (a) and after the ultrasound dispersion (b)

Table 1. Results of energy dispersion analysis of spinel nanopowder

Element	Weight %	Atomic %
O	27.45	56.82
Fe	47.40	28.73
Co	25.15	14.44

the degree of agglomeration, thus making it possible to identify individual nanoparticles on the TEM image and assess their shape and size (Fig. 2b). The CoFe_2O_4 sample was represented by nanoparticles of irregular round shapes, and some particles were faceted. The majority of particles had a size in the range from 50 to 90 nm, while the average size of particles was about 70 nm. According to XRD, the calculated data of CSR values generally correlated with the TEM results.

It was experimentally established that cobalt spinel nanopowder synthesised using the citrate method is an effective heterogeneous catalyst for Fenton-like reactions. Oxidative degradation of DNP under the influence of UV radiation in the presence of the CoFe_2O_4 catalyst proceeded more intensely than in its absence (Fig. 3a). Therefore, the concentration of DNP during catalytic oxidation 2.5 hours after the beginning of the reaction decreased by 5 times. In the absence of a catalyst, after 2.5 hours of reaction, the

concentration of dinitrophenol decreased only by 1.4 times. As for the combination of the H_2O_2 oxidiser with UV radiation, the degradation degree of DNP was 29%, and the degree of degradation increased up to 80% with the introduction of a nanosized decomposition catalyst of hydrogen peroxide into the system.

The shape of kinetic curves of the oxidative degradation of DNP corresponded to the pseudo-first order of the reaction. The rate constant was evaluated by linearisation of the kinetic dependences in logarithmic coordinates (Fig. 3b). The rate constant of the oxidative degradation of DNP in the presence of CoFe_2O_4 with UV radiation was 0.102 min^{-1} , while in the absence of a catalyst, it was an order of magnitude smaller and amounted to 0.0019 min^{-1} . The value of the rate constant of dinitrophenol oxidation was comparable with the values in [19] obtained for a more acidic medium ($\text{pH} \leq 3$) and for the initial DNP concentration 8 times lower as compared to this work. In [10], the calculated constant of photodegradation rate of the methylene blue dye in the presence of nanosized MgFe_2O_4 was 0.0117 min^{-1} with UV radiation by two Xe arc lamps, 35 W each, for two hours. In [20], the rate constant of photodegradation of the same dye with the participation of CoFe_2O_4 as a catalyst and H_2O_2 as an oxidant with daylight but in a

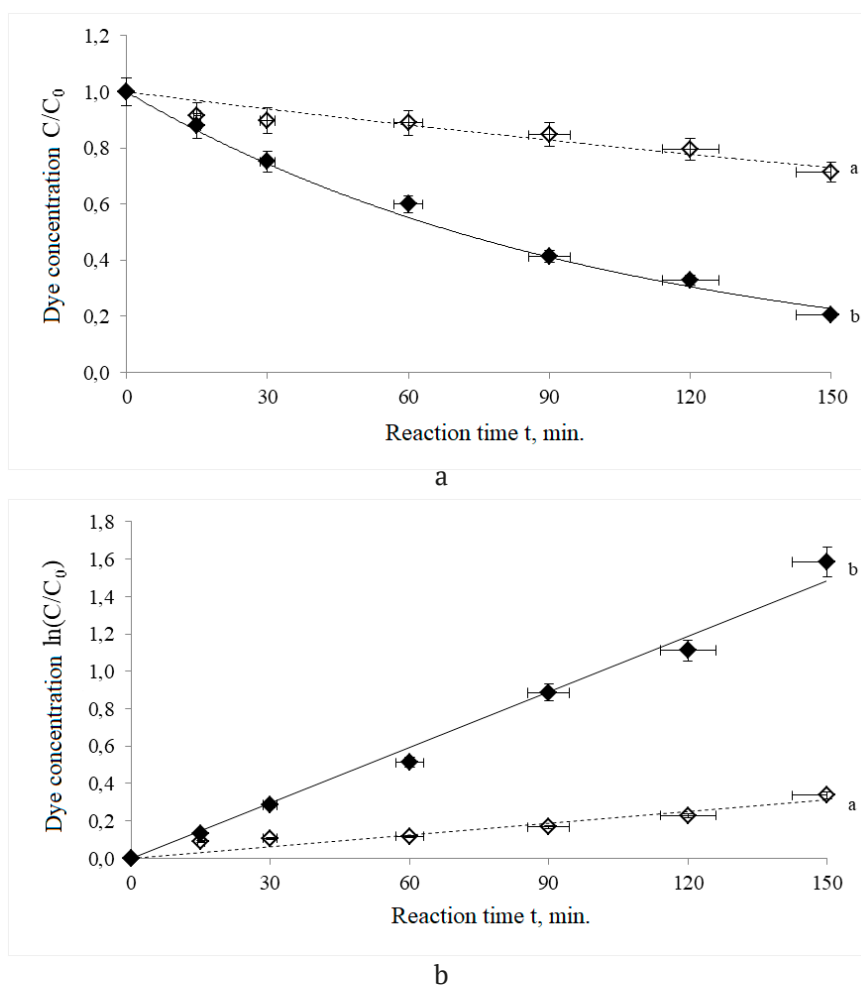


Fig. 3. Change in the concentration of DNP under the influence of UV radiation in the coordinates C/C_0 (a) and $\ln(C/C_0)$ (b) (a – without a catalyst, b – in the presence of the CoFe_2O_4 catalyst.)

considerably more acidic medium ($\text{pH} = 2.5$) was 0.078 min^{-1} . Decomposition of Rhodamine 6 dye in the presence of CoFe_2O_4 in the dark with $\text{pH} = 2$ [21] proceeded much slower (the rate constant was 0.0041 min^{-1}), which allowed concluding that pH of the reaction medium, as well as the type and lighting power were among the most important factors determining the rate of the reaction.

When the reaction proceeded with natural lighting, the catalytic effect of CoFe_2O_4 was feebly pronounced. The degree of DNP degradation was only 14%, although it should be noted that, in the absence of a catalyst, oxidative degradation of 2,4-dinitrophenol hardly occurred (the degradation degree was 0.13%).

The main parameters affecting the course of heterogeneous Fenton-type catalysis were concentration and availability of active catalyst centres, pH , H_2O_2 concentration, and the initial content of organic pollutants and accompanying

ions. As for the classical homogeneous Fenton process, the best pH value is from 2.8 to 3.5 [22]. With higher pH , Fe^{2+} ions become oxidised, thus forming complex hydroxo-compounds, and oxidative activity of H_2O_2 is also reduced.

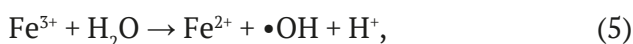
The mechanism of oxidative degradation in heterogeneous Fenton-like reactions consisted in the formation of strong oxidants ($\text{OH}\cdot$) in the course of the reversible transition from Fe^{2+} to Fe^{3+} under exposure to light. It was shown in [23] that tetrahedral centres of Fe^{3+} had electron-withdrawing properties, while the formed Fe^{2+} ions were active centres of Fenton-like processes:

$$\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \cdot\text{OH} + \text{OH}^- \quad (3)$$

In case of cobalt spinel and for cobalt ion (+2), the reaction proceeded with the formation of hydroxyl radicals, which was also confirmed by the data in [24]:



The Fe^{3+} ion is capable of regeneration into Fe^{2+} (especially upon UV radiation) with the formation of an extra amount of hydroxyl and perhydroxyl radicals participating in the oxidation of DNP, which increases the degree of degradation up to 80% as compared to daylight:



It seems that in our case, with daylight, the pH value = 4.4 and the high initial concentration of DNP determined a rather low degree of pollutant degradation.

A series of experiments involving the oxidation of DNP in the combination of H_2O_2 + CoFe_2O_4 + UV and in the presence of CoFe_2O_4 with UV radiation without hydrogen peroxide as an oxidising agent certainly showed that in the first case, the degradation of DNP occurred primarily due to the formation of strong oxidants in the course of the catalytic decomposition of H_2O_2 . The absence of an oxidant revealed a weak sorption capacity of cobalt spinel regarding DNP, and the concentration of the toxic agent decreased only by 5% (Fig. 4). Although under these conditions, Fe^{3+} can be considered as a possible oxidant of dinitrophenol, the data we obtained allowed concluding that this contribution to the overall process was insignificant.

The decrease in the DNP concentration due to sorption was approximately 5% (Fig. 4b), which correlated with the calculated sorption capacity of cobalt ferrite, which was very low, and it may

be associated with the strong agglomeration of the CoFe_2O_4 nanopowder. It should be noted that a small specific surface area was also typical for cobalt spinel synthesised by co-deposition [25]. The sorption capacity of CoFe_2O_4 reached its maximum value (only $0.2 \mu\text{mol/g}$) in 15 min (Fig. 4, insert), and then remained almost unchanged. As a result, it can be concluded that the contribution of sorption to the overall process of DNP degradation in the presence of the CoFe_2O_4 nanosized catalyst in a Fenton-like process was insignificant.

Therefore, the performed assessment of the catalytic activity of nanosized cobalt spinel in the reactions of oxidative degradation of 2,4-dinitrophenol allowed considering CoFe_2O_4 as the basis for creating heterogeneous Fenton catalysts, including composite ones.

4. Conclusions

We synthesised CoFe_2O_4 spinel with an average particle size of about 70 nm using citrate combustion. The strong agglomeration of particles as a result of the selected synthesis method was a specific feature of the nanopowder. We established high catalytic activity of nanodispersed CoFe_2O_4 in Fenton-like reactions of oxidative degradation of 2,4-dinitrophenol upon radiation by ultraviolet $\lambda = 270 \text{ nm}$ in a less acidic medium as compared to a classical Fenton process and with a significantly higher initial pollutant concentration. The degradation degree of DNP without a catalyst was 14%, while in the presence of a nanosized catalyst CoFe_2O_4 it was 80%.

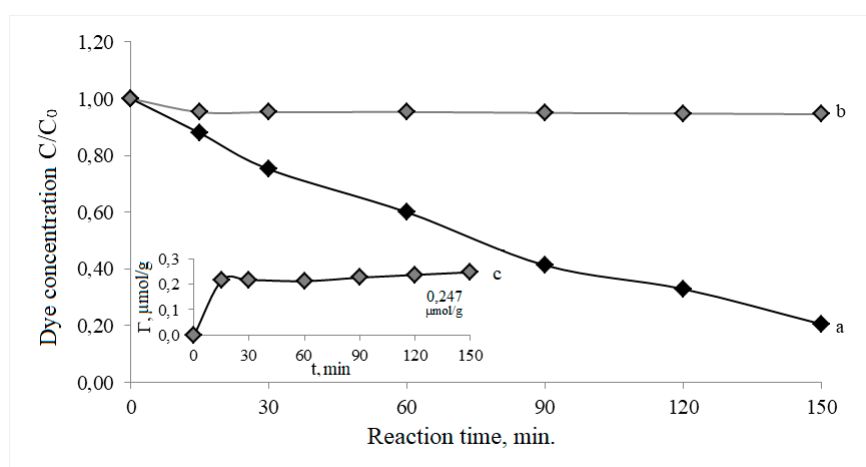


Fig. 4. Curves of catalytic oxidation (a) and sorption (b) of DNP in the presence of CoFe_2O_4 , (c) – sorption capacity of the sample

Contribution of the authors

The authors contributed equally to this article.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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