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Cobalt ferrite catalyst in the degradation of caffeine by hydrogen peroxide under induction heating

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Caffeine is an emerging contaminant that is difficult to remove with conventional remediation methods. Wet peroxide oxidation with heterogeneous cobalt ferrite catalyst was tested as an efficient and environmentally friendly technology for caffeine removal. Due to its ferromagnetic properties, cobalt ferrite is especially suitable for induction heating (IH) by alternating magnetic field. Cobalt ferrite was synthesized by “green” auto-combustion method using *Ginkgo Biloba* extract as a fuel. Spinel structure of the obtained material was confirmed by XRD. The degradation of caffeine in aqueous solutions was studied using cobalt ferrite suspension in a batch reactor under neutral conditions (pH = 7). The synthesized cobalt ferrite effectively accelerates the oxidative degradation of caffeine (Caf). Three independent process variables were tested: initial caffeine concentration (1–5 mg/L), hydrogen peroxide concentration (10–50 mM), and induction heating (IH). The reaction kinetics was monitored by measuring the concentrations of Caf and H₂O₂ by spectrophotometry. Kinetics of the Caf degradation was described by the first-order model and the corresponding reaction rate constants were measured. The efficiency of the Caf degradation was evaluated using percentage removal. The Caf degradation efficiency strongly depends on the initial concentration of H₂O₂ and catalyst dose. For the initial Caf concentration of 1.0 mg/L, the catalyst dose of 3.0 g/L and H₂O₂ concentration of 30 mM result in caffeine degradation efficiency of 92.45%. The results of the study show that CoFe₂O₄ synthesized using *Ginkgo Biloba* extract is a promising heterogeneous catalyst for wastewater remediation.

Keywords: cobalt ferrite; green synthesis; hydrogen peroxide; caffeine; catalyst.

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Introduction

Advanced oxidation is an efficient technique for waste water remediation [1,2]. The practical efficiency of this technology depends largely on the catalyst used [3]. Among heterogeneous Fenton-like catalysts, spinel ferrites stand out due to their high catalytic activity and excellent stability in aqueous solutions [4–6].

Cobalt ferrite is a particularly active catalyst because both the cobalt and iron ions are able to change their oxidation states, providing effective cyclic electron transfer [7–10]. In addition, cobalt ferrite has favorable

magnetic properties. A high saturation magnetization and remanence allows easy removal of the spent cobalt ferrite catalyst from the reaction medium [8]. Due to its high coercivity and magnetocrystalline anisotropy, cobalt ferrite heats up quickly in a high-frequency electromagnetic field. This effect makes cobalt ferrite suitable for hyperthermia treatment in oncological therapy [11,12]. The induction heating effect can be used to increase the catalytic activity of cobalt ferrite and nickel ferrites [9,13].

Various methods are suitable for the synthesis of cobalt ferrite [14,15]. A simple and economical method of

cobalt ferrite synthesis is sol-gel auto-combustion [8,12,16]. The “green” version of the auto-combustion method uses plant extracts as fuel and as a complexing agent [15]. In this work, cobalt ferrite was synthesized by the sol-gel autocombustion using aqueous extract of *Ginkgo Biloba* leaves.

Caffeine is an emerging contaminant with harmful effects on biological systems. Fortunately, wet catalytic oxidation is well suited to the degradation of caffeine [17]. This paper describes the catalytic degradation of Caf using CoFe_2O_4 nanoparticles under the influence of induction heating (IH). The influence of initial concentration of H_2O_2 and caffeine on the degradation efficiency was examined. The induction heating was found to accelerate Caf degradation. To our best knowledge, this is the first study on caffeine degradation catalyzed by CoFe_2O_4 nanoparticles synthesized using a *Ginkgo Biloba* leaf extract.

I. Experimental

1.1. The synthesis of cobalt ferrite catalyst

The synthesis procedure has been described in details elsewhere [18]. The starting materials were cobalt (II) nitrate hexahydrate $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and iron (III) nitrate nonahydrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$. The synthesis has been performed by sol-gel method using the extract of fresh *Ginkgo Biloba* leaves as a fuel [18]. The prepared extract (20 ml) was added to the nitrate salt solution (50 mL) and stirred for 30 min to homogenize. Then the reaction mixture was heated until gel was formed. Further heating caused spontaneous ignition. There were several centers of auto-combustion, brown gas was released. The resulted black powder was annealed at 500 °C for 2 h.

1.2. The caffeine degradation without and in

the presence of induction heating

The caffeine degradation was performed in a 100 mL glass beaker with an overhead glass stirrer. The volume of Caf solution was 40 ml and the concentrations were 1 mg/L and 5 mg/L. The accuracy of the Caf solution preparation was checked by measuring the optical density at 274 nm on a ULAB 102 UV spectrophotometer, in 5 mm quartz cuvettes. The initial solution pH was 6.5. The dose of cobalt ferrite catalyst was 3 g/L. The suspension of catalyst in the Caf solution was stirred at 500 rpm for 30 min in order to attain the adsorption-desorption equilibrium. Then the specified amount of hydrogen peroxide was added to the reaction mixture to obtain the desired concentration of 10, 20, 30 or 50 mM. The reaction was carried out under continuous stirring and samples of 1.5 mL were taken at specified time intervals. The sample was filtered using a syringe filter to completely remove catalyst particles.

The experiments under the influence of induction heating were performed in the same way, but the reaction vessel was placed inside an induction coil with a diameter of 5 cm. The induction coil consists of six turns of water-cooled copper pipe. The alternating current generator operates at a frequency of 100 kHz and a power of 1 kW.

The residual caffeine concentration in the solution was determined spectrophotometrically by measuring the optical density at 274 nm using an ULAB 102 UV spectrophotometer and 5 mm quartz cuvettes. The residual caffeine concentration was calculated using the following equation:

$$C(\text{caffeine}) = A_{274}/0.0409, \quad (1)$$

where $C(\text{caffeine})$ is the caffeine concentration in mg/L and A_{274} is the optical density of the caffeine solution. The efficiency of caffeine degradation (%) was calculated using the initial and final caffeine concentrations:

$$\text{Caffeine degradation (\%)} = \frac{\text{Caf initial conc.} - \text{Caf final conc.}}{\text{Caf initial conc.}} \times 100 \quad (2)$$

The residual concentration of hydrogen peroxide in the solution was determined by the metavanadate method. 1 mL of the analyzed solution was mixed with 1.5 mL of 2M H_2SO_4 solution and 1.5 mL of 0.1 M NH_4VO_3 solution. The resulting solution was then filled with distilled water to 5 mL and left for 10 minutes to form colored solution. Optical density at 470 nm was measured in 10 mm glass cuvettes using an ULAB 102-UV spectrophotometer. The concentration of hydrogen peroxide was calculated by the equation:

$$C(\text{H}_2\text{O}_2) = \frac{A_{470}}{0.29917} * 5, \quad (3)$$

where $C(\text{H}_2\text{O}_2)$ is the concentration of hydrogen peroxide (mM); A_{470} is the optical density of the colored solution at 470 nm; 5 is the dilution factor.

The decomposition of caffeine in the presence of ferrite catalysts was described by the first-order law equation: $-d[\text{Caf}]/dt = k[\text{Caf}]$. The first-order law equation was re-written as follows:

$\ln([\text{Caf}]_t/[\text{Caf}]_0) = -k \cdot t$, where k is the experimental reaction rate constant, and $[\text{Caf}]_t$ and $[\text{Caf}]_0$ are the concentrations of caffeine in the solution at time t and 0 min, respectively. The rate constants for the decomposition of caffeine and hydrogen peroxide were determined as the slopes of the corresponding experimental plots $\ln([\text{Caf}]_t/[\text{Caf}]_0)$ vs. time.

II. Results and discussion

The degradation of caffeine in the presence of H_2O_2 alone was negligible. This fact indicates that the cobalt ferrite catalyst is definitely required for H_2O_2 activation and caffeine degradation. In the presence of cobalt ferrite, 30 mM H_2O_2 provides rather fast degradation of Caf. Caffeine solution of 1.0 mg/L was almost completely degraded within 60 min.

2.1. Effect of caffeine concentration on the degradation kinetics

Figs. 1a-1d and Figs. 2a-2d show UV spectra of selected solutions during caffeine degradation experiments. The spectral changes clearly indicate that caffeine degradation occurs rapidly in the presence of CoFe_2O_4 nanoparticles and under the influence of electromagnetic field.

The dose of CoFe_2O_4 catalyst is 3 g/L. The dashed line indicates the caffeine spectrum.

Figs. 3a, 3c show the dependence of the degree of caffeine decomposition on the reaction time in the presence of the synthesized cobalt ferrite catalyst. The effect of the initial Caf concentration on the Caf degradation kinetics is clearly seen. Increasing the Caf concentration from 1.0 mg/L to 5.0 mg/L reduces the final

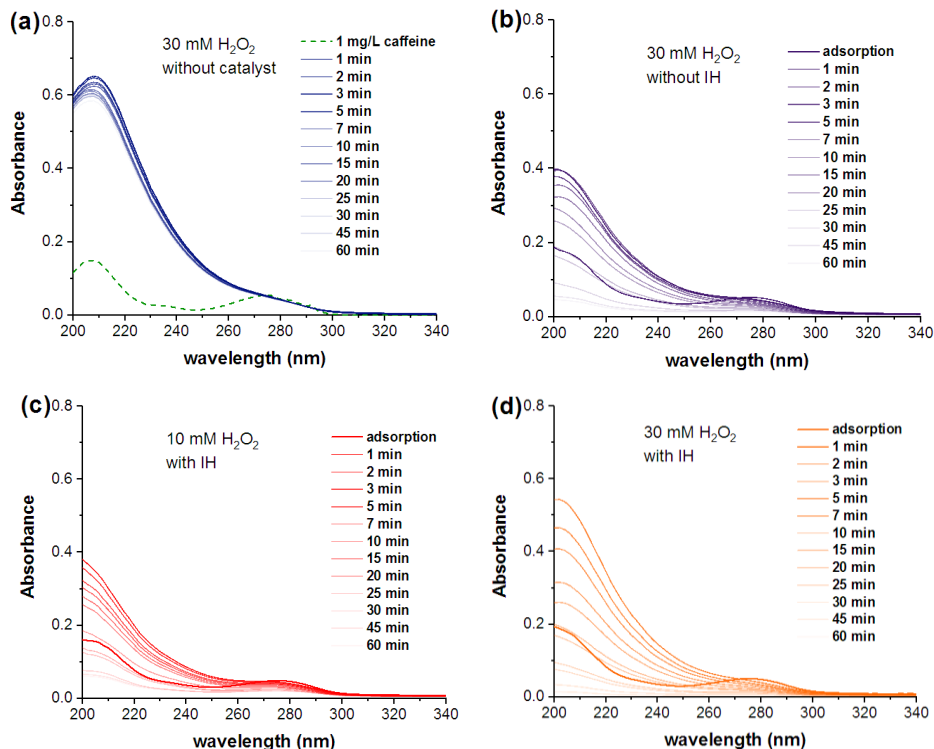


Fig. 1. Time-dependent changes of UV-vis spectra of reaction mixtures containing 1 mg/L caffeine.

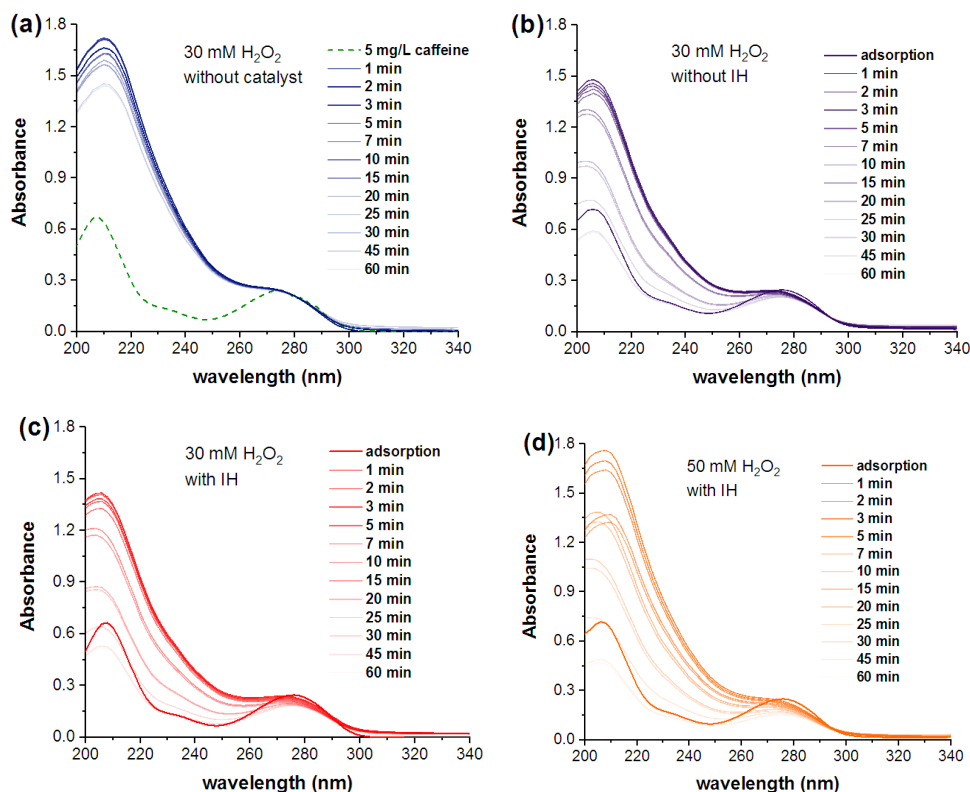


Fig. 2. Time-dependent changes of UV-vis spectra of reaction mixtures containing 5 mg/L caffeine.

degradation efficiency. It is obvious that when a higher initial caffeine concentration is used, a higher amount of residual Caf is present. Additionally, higher Caf amount reduces the formation of hydroxyl and superoxide radicals [19]. Figs. 3a, 3c show also the accelerating effect of induction heating. The highest decomposition efficiency of 92.45% was registered at 30 mM H₂O₂ in the presence of CoFe₂O₄ nanoparticles (NPs) under the influence of electromagnetic field. On the other hand, the degradation efficiency is only 17.07% without the action of IH (Fig. 3c).

Fig. 3b, 3d show the kinetic curves of caffeine decomposition transformed on the logarithmic scale. The straight lines of ln(C/C₀) versus time t (Figs. 3b, 3d) indicate that the catalytic decomposition of caffeine is a

first-order reaction. This conclusion is supported by high correlation coefficients ranging from 0.9482 to 0.9991. The corresponding values of first-order rate constant for caffeine degradation were calculated as the slopes of the plots in Figs. 4b, 4d and presented in Table 2. The rate constant of the caffeine degradation reaction with cobalt ferrite catalyst was found to be 0.0726 min⁻¹ (Table 1). This value is higher compared to the available literature data. The literature reports refer the catalytic degradation of caffeine using various catalysts such as cobalt-MCM41 [19] and AgFeO₂ nanoparticles [20].

2.2. Effect of H₂O₂ concentration

The estimation of the optimal concentration of H₂O₂ is one of the most important tasks for the catalytic wet

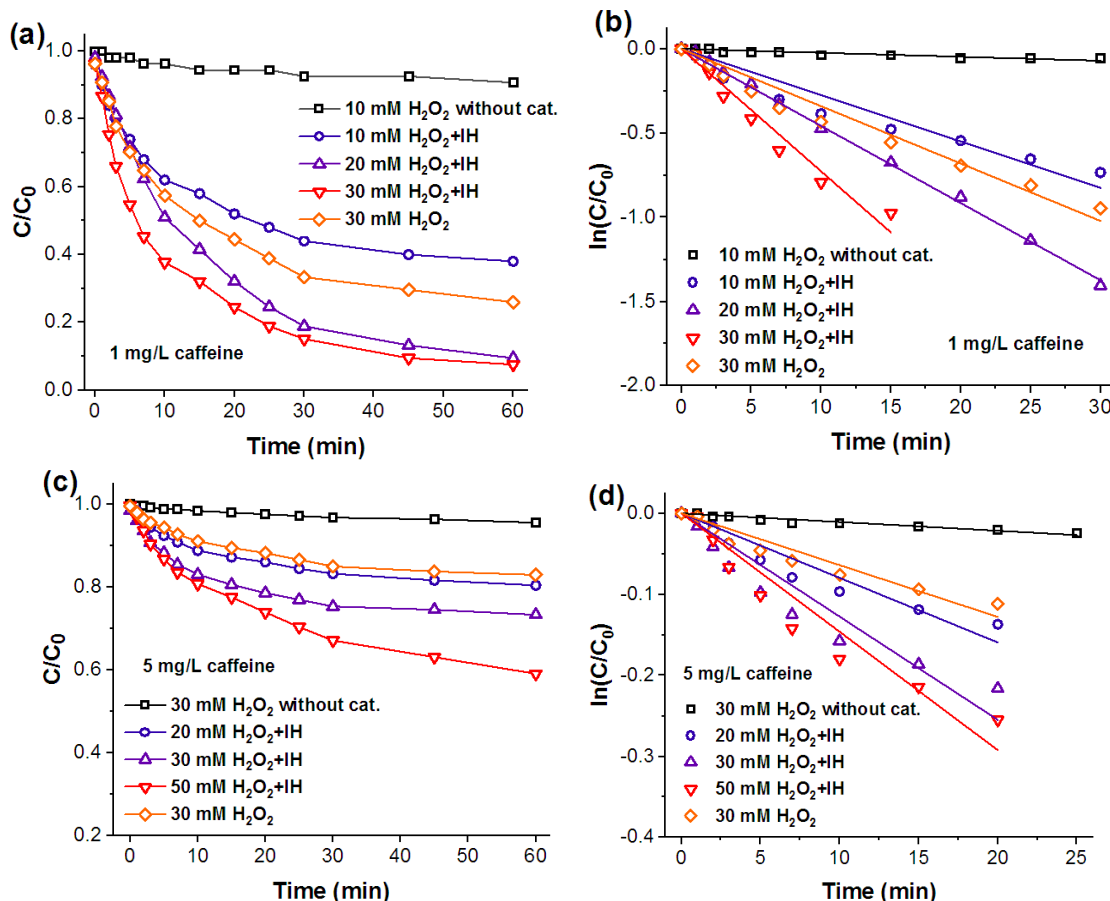


Fig. 3. (a,c) Changes of caffeine concentrations vs. reaction time in the presence of the CoFe₂O₄ catalyst. (b,d) The kinetic lines transformed according to the first-order kinetic model.

Table 1.

Rate constants of the first-order kinetic model for the degradation of caffeine with the CoFe₂O₄.

H ₂ O ₂ concentration (mM)	Caffeine concentration (mg/L)			
	1.0		5.0	
	k, min ⁻¹	R ²	k, min ⁻¹	R ²
30 *	0.0023	0.9482	0.0010	0.9741
30 **	0.0340	0.9847	0.0063	0.9655
10	0.0275	0.9691	-	-
20	0.0457	0.9991	0.0079	0.9658
30	0.0726	0.9844	0.0127	0.9568
50	-	-	0.0146	0.9687

* without catalyst

** without induction heating

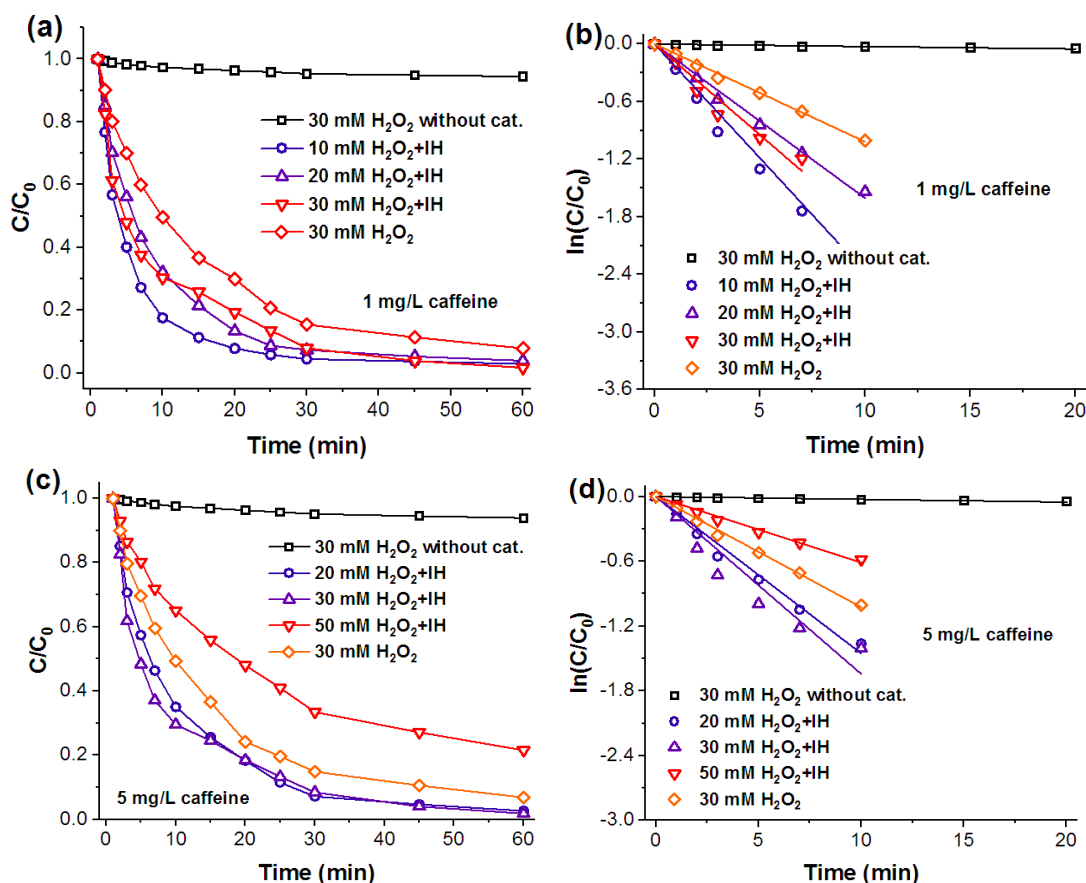


Fig. 4. (a,c) Changes of H_2O_2 concentration vs. reaction time in the presence of the CoFe_2O_4 catalyst. (b,d). The kinetic lines transformed according to the first-order kinetic model.

Table 2.

Rate constants of the first-order kinetic model for the degradation of H_2O_2 with the CoFe_2O_4 .

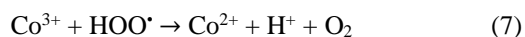
H_2O_2 concentration (mM)	Caffeine concentration (mg/L)			
	1.0		5.0	
	k, min^{-1}	R^2	k, min^{-1}	R^2
30 *	0.0025	0.9284	0.0025	0.9638
30 **	0.1017	0.9983	0.1023	0.9980
10	0.2372	0.9899	-	-
20	0.1604	0.9966	0.1452	0.9929
30	0.1889	0.9815	0.1642	0.9677
50	-	-	0.0612	0.9951

oxidation, since the amount of H_2O_2 used affects the cost of the overall water purification process. In addition, the residual H_2O_2 restricts release of the purified water into the environment [3]. The effect of H_2O_2 concentration on the caffeine degradation was studied by changing the amount of H_2O_2 from 10 to 50 mM under the action of IH. Fig. 4a, 4c show that no significant decomposition of hydrogen peroxide was observed without the catalyst even at a higher H_2O_2 concentration of 30 mM. After 90 minutes, the decomposition degree was approximately 6%. Instead, an the H_2O_2 decomposition was observed to be from 97.09% to 98.37% when the H_2O_2 concentration was from 10 to 30 mM during the destruction of 1.0 mg/L caffeine within 60 minutes. During the degradation of 5.0 mg/L caffeine for 60 minutes, a decrease in the H_2O_2 degradation degree from 97.36% to 78.45% was observed when the H_2O_2 concentration was increased from 20 to 50 mM.

Figs. 4b, 4d show the kinetic curves of hydrogen peroxide decomposition. Figs. 4b, 4d show the transformations on the logarithmic scale. The obtained plots are described by high coefficients of determination R^2 ranging from 0.9284 to 0.9983 (Table 2). The values of the first-order rate constants of the H_2O_2 decomposition reaction were calculated from the slopes of the lines in Fig. 4b, 4d. Table 2 lists the obtained numerical values. The highest rate constant of 0.237 min^{-1} was registered for 10 mM H_2O_2 , 1.0 mg/L caffeine and a catalyst dose of 3 g/L under the influence of IH.

Literature reports indicate that Co^{2+} and Fe^{2+} ions catalyze the H_2O_2 decomposition through a cyclic electron transfer reactions, which lead to the formation of hydroxyl and hydroperoxyl radicals [21]. The occurring reactions are as follows:





The especially high catalytic activity of cobalt ferrite can be attributed to the presence of ions Co^{2+} in octahedral positions of the ferrite sublattice. In addition, electron transfer between ion pairs $\text{Co}^{2+}\text{-Co}^{3+}$ i $\text{Fe}^{2+}\text{-Fe}^{3+}$ also occurs.

Conclusions

Cobalt ferrite nanoparticles were synthesized by auto-combustion method using *Ginkgo Biloba* leaf extract as a reducing agent. The obtained material was used as a catalyst in the oxidative degradation of caffeine. The highest degradation efficiency of 92.45% within 90 minutes was achieved using 3.0 g/L of catalyst, 30 mM H_2O_2 and 1.0 mg/L caffeine under the influence of

induction heating. Thus, the simple green synthesis of CoFe_2O_4 nanoparticles can be used to obtain catalysts for wastewater remediation. This work is the first study on the caffeine degradation by hydrogen peroxide activated by CoFe_2O_4 synthesized using *Ginkgo Biloba* leaf extract. The used CoFe_2O_4 nanoparticles can be easily recovered by magnetic field and reused.

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Ферит кобальту як каталізатор розкладу кофеїну в присутності пероксиду водню при індукційному нагріві

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Кофеїн є поширеним забруднювачем, який важко видалити стандартними методами очищення води. Пероксидне окиснення з гетерогенним каталізатором фериту кобальту було перевірено як ефективну та екологічно чисту технологію видалення кофеїну. Завдяки своїм феромагнітним властивостям ферит кобальту особливо підходить для індукційного нагріву змінним магнітним полем. Ферит кобальту було отримано методом «зеленого» синтезу з використанням екстракту *Гінкго білоба*, як палива. Структуру шпінелі отриманого матеріалу було підтверджено методом X-променевої дифрактометрії. Деградацію кофеїну у водних розчинах вивчали з використанням суспензії фериту кобальту в реакторі періодичної дії в нейтральних умовах (рН = 7). Синтезований ферит кобальту ефективно прискорював деградацію молекул кофеїну. Було перевірено три незалежні змінні процесу: початкова концентрація кофеїну (1–5 мг/л), концентрація пероксиду водню (10–50 мМ) та вплив індукційного нагріву. Кінетику деградації кофеїну описано моделлю першого порядку та визначено відповідні константи швидкості реакції. Виявлено, що ефективність деградації кофеїну залежить від початкової концентрації H₂O₂ і дози каталізатора. Для початкової концентрації кофеїну 1,0 мг/л, дози каталізатора 3,0 г/л і концентрації H₂O₂ 30 мМ ефективність розкладу кофеїну становить 92,45%. Результати досліджень показують, що CoFe₂O₄, синтезований з використанням екстракту *Гінкго білоба*, є перспективним гетерогенним каталізатором для очищення стічних вод.

Ключові слова: ферит кобальту; зелений синтез; пероксид водню; кофеїн; каталізатор.