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СИНТЕЗ НАНОМАТЕРИАЛОВ Ag/Fe_3O_4 ИЗ ЭКСТРАКТА ЛИСТЬЕВ ГУАВЫ И ИХ ПРИМЕНЕНИЕ В КАЧЕСТВЕ КАТАЛИЗАТОРА ВОССТАНОВЛЕНИЯ МЕТИЛЕНОВОГО СИНЕГО И РОДАМИНА Б

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В этой работе представлены результаты исследований по синтезу Ag/Fe_3O_4 методом зеленой химии с использованием экстракта листьев гуавы и их применения в качестве катализатора восстановления метиленового синего (MB) и родамина Б (RhB) борогидридом натрия ($NaBH_4$). Физико-химические характеристики материала определяли с помощью рентгеноструктурного анализа, EDX спектров и изображений SEM. Методами физико-химического анализа доказано, что полученный материал состоит из двух наноразмерных фаз: Ag (d = 20 нм) и Fe_3O_4 (d = 12 нм). Показано, что скорость восстановления метиленового синего и родамина Б борогидридом натрия увеличивается примерно в 26,3 раза и 57,8 раза в присутствии Ag/Fe_3O_4. Фаза Fe_3O_4 действует как носитель, определяя магнитные свойства, а фаза Ag выступает как каталитический центр, определяя каталитическую активность материала. Исследовано влияние условий экстракции листьев гуавы и условий синтеза наночастиц серебра на каталитическую активность полученного материала Ag/Fe_3O_4. По результатам исследований, материалы Ag/Fe_3O_4, синтезированные методом зеленой химии с использованием экстракта листьев гуавы при следующих условиях: 12,5 г листьев гуавы/200 мл дистиллированной воды, время экстракции: 20 мин, температура экстракции: 70 °С, 10 мл экстракта/50 мл 5 мМ раствора $AgNO_3$ при 40 °С в течение 30 мин, обладают лучшей каталитической активностью. Константы скорости восстановления метиленового синего и родамина Б борогидридом натрия в присутствии Ag/Fe_3O_4 составляют 0,3534 мин$^{-1}$ и 0,4886 мин$^{-1}$, что существенно выше, чем у аналогичных катализаторов. Катализатор был пригоден для вторичной переработки в течение 3 каталитических циклов, при этом эффективность удаления MB (RhB) снизилась с 92,8% (91,6%) до 75,2% (74,5%).

Ключевые слова: метод зеленой химии, экстракт листьев гуавы, наноматериал Ag/Fe_3O_4, катализатор, восстановления красителей

THE SYNTHESIS Ag/Fe_3O_4 NANOPARTICLES USING GUAVA LEAF EXTRACT (*PSIDIUM GUAJAVA L.*) AND ITS APPLICATION FOR CATALYTIC REDUCTION OF METHYLENE BLUE AND RHODAMINE B

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This article presents a study on green synthesis Ag/Fe_3O_4 using guava leaf extract and its application for catalytic reduction of methylene blue (MB) and rhodamine B (RhB) with $NaBH_4$.

The synthesized material was characterized using X-ray diffraction (XRD), EDX spectroscopy, and SEM images. The results of analyzing the physicochemical properties of materials showed that the obtained material consisted of two nano-sized phases: Ag (d = 20 nm) and Fe_3O_4 (d = 12 nm). The synthesized material was used as a catalyst for the reduction of methylene blue and rhodamine B with $NaBH_4$. It was shown that Ag/Fe_3O_4 material increases the reduction rate of methylene blue and rhodamine B by approximately 26.3 times and 57.8 times compared to without a catalyst. The results showed that the Fe_3O_4 phase acts as a carrier, determining the magnetism, while the Ag phase acts as the catalytic center, determining the catalytic activity of the material. The influence of synthesis conditions on the catalytic activity of Ag/Fe_3O_4 materials was studied. It was shown that Ag/Fe_3O_4 materials were synthesized under the following conditions: 12.5 g guava leaves/200 mL distilled water, extraction time: 20 min, extraction temperature: 70 °C, extract volume of 10 mL/50 mL of 5 mM $AgNO_3$ solution at 40 °C for 30 min have the best catalytic activity. The reduction rate constants of methylene blue and rhodamine B with $NaBH_4$ in the presence of Ag/Fe_3O_4 are 0.3534 min^{-1} and 0.4886 min^{-1}, significantly higher than similar catalysts. The catalyst was recyclable for 3 catalytic cycles with a drop from 92.8% (91.6%) to 75.2% (74.5%) in the removal efficiency for MB (RhB).

Keywords: green synthesis, guava leaf extract, Ag/Fe_3O_4 nanoparticles, catalyst, reduction of dyes

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INTRODUCTION

Textiles and clothing play a major role in the development and industrialization process of countries and their integration into the world economy. However, this industry is also considered highly polluting because of its intensive, hazardous discharge to the environment [1-3]. Textile wastewater has been found to contain a wide range of toxic dyes, heavy metals, as well as aromatic compounds. Textile dyes degrade the esthetic quality of water bodies by increasing biochemical and chemical oxygen demand, thereby impairing photosynthesis, inhibiting plant growth, entering the food chain, providing recalcitrance and bioaccumulation, and potentially promoting toxicity, mutagenicity, and carcinogenicity. Various technologies and methodologies were used for the treatment of dye-containing wastewater such as physically, chemically, biologically, or utilizing a combination of these methods [4].

Recently, the reduction of dyes into biodegradable pollutants in the presence of $NaBH_4$ was developed as a new and efficient technique for wastewater treatment. And silver nanoparticles (AgNPs) were found to be a good catalyst with excellent performance [5-7]. However, there are difficulties in the recovery of metal nanocatalysts and their reusability which causes considerable problems with traditional separation methods such as filtration, centrifugation, and precipitation considering environmental and economic points of view. Also, the direct use of silver nanoparticles often suffers from their susceptibility to coagulation and agglomeration, moreover, silver is a precious metal, and reducing the amount of silver used is one of the most effective ways to cut the cost. To overcome these difficulties, the composite of magnetic nanomaterials such as Fe_3O_4 with AgNPs is one of the promising materials for application in catalytic reactions. The composite of AgNPs and Fe_3O_4 nanoparticles can integrate individual excellence of each constituent which includes good electrical conductivity, high selectivity and reactivity, nanosize leading to large surface area to volume ratio, distinct catalytic activity, and easy magnetic separation [8-10]. So far there have been many reports on the composite of Ag and Fe_3O_4 based on various synthesis techniques [11-14]. Among them, the green synthesis method, which uses plant extracts instead of industrial chemical agents to reduce Ag^+ ions, is more beneficial than traditional chemical synthesis because it costs less, decreases pollution, and improves environmental and human health safety [15-17].

In the present paper, the results of Ag/Fe_3O_4 nanomaterials synthesis using guava leaf extract and the influence of synthesis conditions on its catalytic performance for the reduction of methylene blue (MB) and rhodamine B (RhB) with $NaBH_4$ are presented.

EXPERIMENTAL

Synthesis of the Fe_3O_4: Magnetite particles (Fe_3O_4) were synthesized by adding $FeCl_2$ (0.01 mol) and $FeCl_3$ (0.02 mol) to double-distilled water (100 mL) and then stirred at 70 °C for 5 min. After that 400 mL of solution 2 M NaOH was added into the reaction medium and the mixture was stirred at 70 °C for another 15 min. Afterward, Fe_3O_4 was separated with a strong magnet, washed with distilled water to neutrality, and dried at 60 °C for 4 h.

Preparation of guava leaf extract: guava leaves were collected in Da Nang City, Vietnam. The leaves were washed, shade-dried at room temperature, and cut into fine pieces. m g of fine pieces of leaves (m = 5-15 g) were taken in a beaker with 200 mL of double distilled water and placed in a water bath with a temperature of T °C (T = 50-100 °C) for t min (t = 5-50 min). The aqueous extract was cooled, filtered through Whatman No. 1 filter paper, and stored at room temperature for further nanoparticle synthesis process.

Green synthesis of the Ag/Fe_3O_4 nanocomposite using guava leaf extract: first a mg Fe_3O_4 magnetic nanoparticles (a = 5-25 mg) in 10 mL distilled water was sonicated for 15 min to complete dispersion after that 50 mL $AgNO_3$ C mM (C = 1-9 mM) was added to the solution. Added V mL of guava leaf extract (V = 4-14 mL) to the mixed solution and stirred for t min (t = 10-60 min) at T °C (T = 30-80 °C). Next, the desired Ag/Fe_3O_4 nanocomposite was separated using an external magnet, washed with distilled water to neutrality, and dried at 60 °C for 4 h.

Characterizations: The obtained sample was characterized by using various chemical and physical techniques. The XRD patterns were recorded on a Bruker D8-Advance X-ray powder diffractometer using Cu Kα radiation (λ = 1.5406 Å) with scattering angles (2θ) of 30-80°. Scanning electron microscopy (SEM) and mapping energy dispersive X-ray spectroscopy (EDX) were performed on an SEM-JEOL-JSM 5410 LV (JEOL JSM-IT200, Japan) with an acceleration voltage of 10 kV at a magnification level of 10 K.

The crystalline size is determined according to the Scherrer formula [18]:

$$D = \frac{K\lambda}{\beta \cos\theta}, \qquad (1)$$

where D is the average crystalline size (nm), K is Scherrer constant (K = 0.94); λ is a wavelength in Å (λ = 1.5418 Å), β is the FWHM in radian and θ is diffraction angle in degree. The major diffraction peaks appear at 2θ = 35.6° and 38.1°, which can be indexed as the (311) plane for Fe_3O_4 and the (111) plane for Ag.

Catalytic reduction of methylene blue and rhodamine B: The catalytic activity of green synthesized Ag/Fe_3O_4 was analyzed for the reduction of organic dyes (methylene blue and rhodamine B). To induce the reduction process, $NaBH_4$ was used as a reducing agent. The reaction was conducted in a cuvette containing 2 mg of samples dispersed in 4 mL of 20 ppm MB or 10 ppm RhB solution. Then 1 mL of 0.01 M $NaBH_4$ solution was injected into the mixture. The concentration of MB and RhB was measured with a UV–Vis spectrophotometer (Lambda 365, Perkin Elmer, USA). The characteristic spectra of the respective dye were monitored at different time intervals.

The recyclability of the catalyst was also investigated. After each catalytic cycle, the catalyst was separated using an external magnet, washed, and soaked in ethanol to remove any remaining dyes on the catalyst. It was then dried at 60 °C for 4 h.

RESULTS AND DISCUSSION

1. Characterization of materials

A comparative X-ray diffraction study of Fe_3O_4 and Ag/Fe_3O_4 to ascertain the crystalline phases of Ag has been shown in Fig. 1a.

The XRD patterns of both samples exhibit the characteristic peaks at 35.6, 43.3, 57.3, and 62.9° indexed (311), (400), (511), and (440) planes of Fe_3O_4 according to JCPDS 01-071-6336 [19]. Additionally, the diffraction peaks at 38.1, 44.3, 64.4, and 77.5 confirm the Ag crystalline phases. These correspond to the diffraction on *fcc* silver (111), (200), (220), and (311) planes (JCPDS 04-07-83) [17].

Analysis of the full-width at half-maximum (FWHM) of the diffraction peaks appear at 2θ = 35.6° and 38.1°, which can be indexed as (311) plane for Fe_3O_4 and (111) plane for Ag, reveals the crystalline size of the Ag/Fe_3O_4 particles. According to the calculation result, the average crystalline sizes of Fe_3O_4 and Ag are 12 nm and 20 nm, respectively.

The EDX spectrum shown in Fig. 1b confirms the existence of Fe (19.50%), Ag (10.44%), O (50.87%), and C (19.19%) in the obtained Ag/Fe_3O_4 material.

To have the particle morphology of Ag/Fe_3O_4 nanocomposite, SEM analysis was provided. As shown in Fig. 1c, silver nanoparticles appeared after reduction by guava leaf extract and anchored on the surface Fe_3O_4.

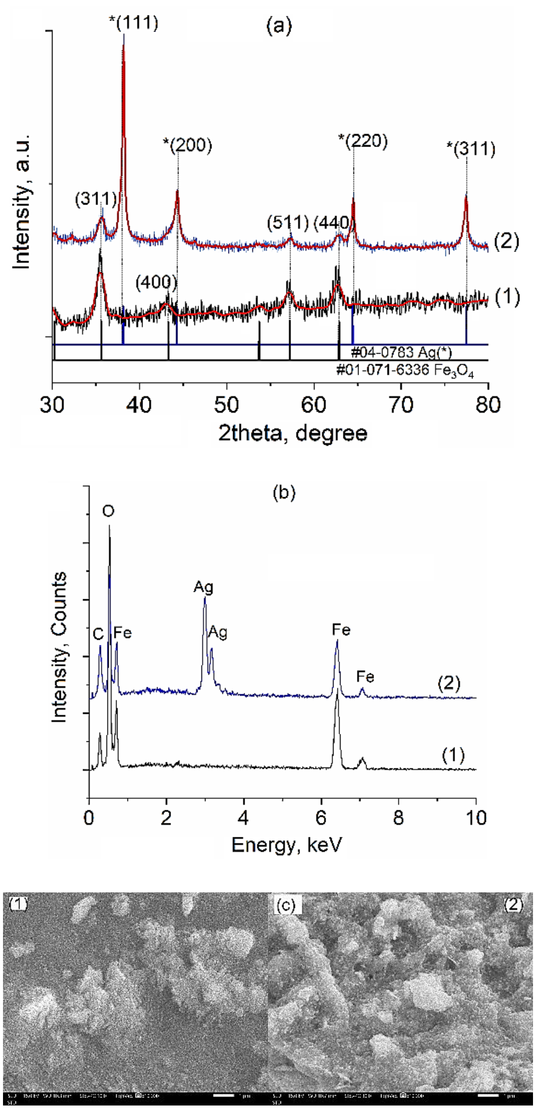

Fig. 1. (a) XRD patterns, (b) EDX, and (c) SEM images of the samples: (1) – Fe_3O_4; (2) – Ag/Fe_3O_4

Рис. 1. (a) Дифрактограмма, (b) EDX-спектры, и (c) SEM изображения образцов: (1) – Fe_3O_4; (2) – Ag/Fe_3O_4

Graphical representation of the green synthesis of Ag/Fe_3O_4 nanoparticles using guava leaf extract is presented in Fig. 2 [17]:

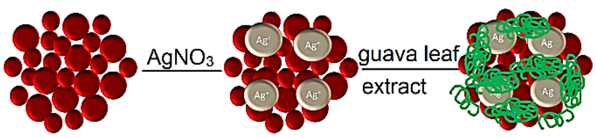

Fig. 2. Schematic synthesis of Ag/Fe_3O_4 nanocomposite meterials by guava leaf extract

Рис. 2. Схема синтеза наноматериалов Ag/Fe_3O_4 из экстракта листьев гуавы

The Fe_3O_4 nanoparticles function as magnetic carriers, guava leaf extract contains polyphenols, which act as a natural reducing agent for in situ chemical reduction of Ag^+ to Ag. The organic compounds of guava leaf extract have a significant tendency to accumulate over material and act as stabilizing agents [20].

2. Catalytic reduction of methylene blue and rhodamine B

The control experiments containing $NaBH_4$ and MB or RhB (without catalyst and presence of Fe_3O_4) did not show a significant change in the color of the solutions as well as a decrease in absorption spectra over time (Fig. 3a). The addition of Ag/Fe_3O_4 to the reaction mixture completely decolorized the dyes. The absorption spectra of dyes also disappeared within 8 min for MB (664 nm) and 5.5 min for RhB (554 nm) (Fig. 3b).

The removal efficiency of MB (RhB) was 9.2% (4.3%) in the presence of $NaBH_4$, while its removal efficiency was 7.0% (2.6%) and 92.8% (91.6%) within 8 min (5.5 min) when Fe_3O_4 and Ag/Fe_3O_4 were added to the reaction mixture. The results showed that Ag/Fe_3O_4 catalyzed the reduction of MB and RhB into leuco-methylene blue (leuco MB) and leuco-rhodamine B (leuco RhB) in the presence of $NaBH_4$ [12], [21]. The catalytic activity of Ag/Fe_3O_4 is determined by the Ag phase, the Fe_3O_4 phase only acts as a magnetic carrier to help recover materials. The mechanism of catalytic reduction of dye through silver nanoparticles involves electron transfer and the least activation energy [5], [22]:

$$BH_4^- + H_2O + Ag_n \rightarrow BO_2^- + Ag_n^- + H^+$$

$$Ag_n^- + MB(RhB) \rightarrow Ag_n + Leuco\,MB(RhB)$$

Since the amount of $NaBH_4$ used in this reaction is much higher than that of MB and RhB, its concentration remains practically constant during the reaction and hence the reaction may be considered to follow pseudo-first-order kinetics. So, the rate equation may be written as $\ln C_0/C = kt$, where ‘k’ is the pseudo-first-order rate constant, C_0 is the initial concentration of MB or RhB and C is the concentration at ‘t’ time. Fig. 3c shows the plots of $\ln(C_0/C)$ versus time. The good linear correlation between the variables reveals that the reaction strictly follows pseudo-first-order kinetics, and the rate constants of the reaction are 0.012 min^{-1} and 0.0076 min^{-1} for MB and RhB, respectively. The Fe_3O_4 reduces the reduction rate of MB and RhB by approximately 1.3 times (0.0091 min^{-1}) and 1.6 times (0.0048 min^{-1}). While the Ag/Fe_3O_4 composite increases the reduction rate of MB and RhB by approximately 26.3 times (0.3152 min^{-1}) and 57.8 times (0.4389 min^{-1}) compared to without a catalyst (Fig. 3d).

The synthesis conditions greatly affect the catalytic activity of Ag/Fe_3O_4 material (Fig. 4, Fig. 5).

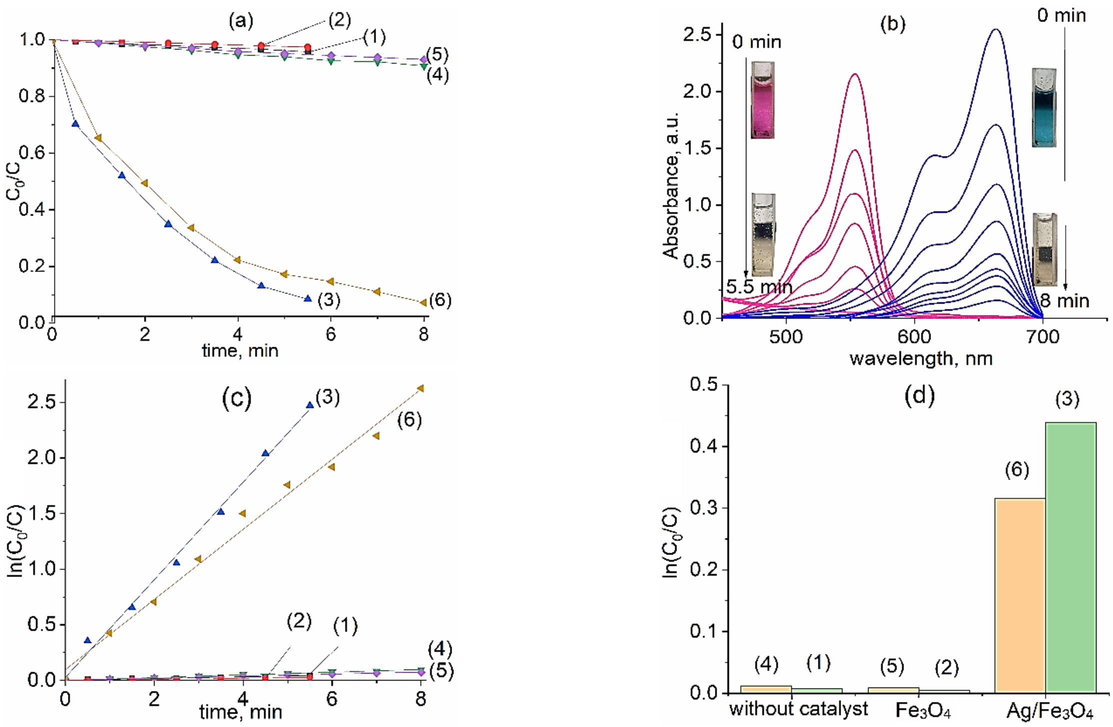

Fig. 3. (a) Plots of C/C_0 of MB and RhB versus time, (b) UV-Vis spectra of MB and RhB in the presence of $NaBH_4$ + Ag/Fe_3O_4; (c) Plots of lnC_0/C of MB and RhB versus time; (d) reduction rate constants of MB and RhB in reaction systems: (1) – RhB; (2) – RhB + Fe_3O_4; (3) – RhB + Ag/Fe_3O_4; 4 – MB; (5) – MB + Fe_3O_4; (6) – MB + Ag/Fe_3O_4

Рис. 3. (a) Зависимости C/C_0 от времени, (b) УФ-Вид спектры MB и RhB в присутствии $NaBH_4$ + Ag/Fe_3O_4; (c) Зависимости lnC_0/C от времени; (d) константы скорости восстановления MB и RhB в реакционных системах: (1) – RhB; (2) – RhB + Fe_3O_4; (3) – RhB + Ag/Fe_3O_4; 4 – MB; (5) – MB + Fe_3O_4; (6) – MB + Ag/Fe_3O_4

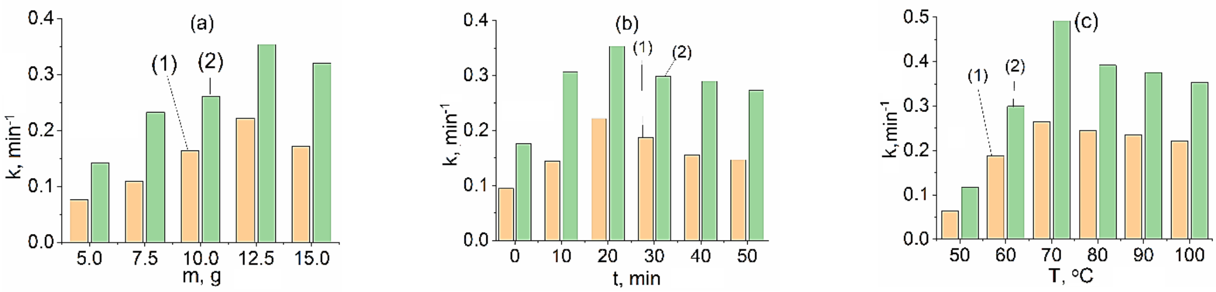

Fig. 4. Dependence of reduction rate constants of MB (1) and RhB (2) on (a) mass of guava leaves; (b) extraction time; (c) extraction temperature

Рис. 4. Зависимость константы скорости восстановления MB (1) и RhB (2) от: (a) массы листьев гуавы; (b) времени и (c) температуры экстракции

Fig. 4 shows that when increasing the mass of guava leaves from 5 g to 15 g, extraction time from 5 min to 50 min, and extraction temperature from 50 °C to 100 °C, the catalytic activity of the material increases and then gradually decreases.

This can be explained by the fact that when increasing the weight of guava leaves, temperature, and extraction time, the amount of reducing agents in the extract increases, thus the amount of Ag formed is greater. As a result, the catalytic activity of the material increases. However, when the amount of reducing agent in the extract is too much, the reduction of Ag^+ to Ag can occur right in the solution volume instead of taking place on the surface of the Fe_3O_4 support. The decrease in Ag content in the material is the cause of the decrease in catalytic activity of the material for the reduction of MB and RhB with $NaBH_4$.

Ag/Fe_3O_4 materials were synthesized using guava leaf extract, which was obtained under the following conditions: 12.5 g guava leaves/200 mL distilled water; extraction time: 20 min; extraction temperature: 70 ℃, showed the best catalytic performance.

Experimental results showed that, when increasing the synthesis time and temperature, volume of extract, and $AgNO_3$ concentration, the catalytic activity of the synthesized materials increases, then gradually decreases (Fig. 5). The influence of nanosynthesis conditions on the catalytic activity of the material can be related to the amount of Ag formed and the dispersibility of Fe_3O_4 suspension. Accordingly, when the amount of Ag formed and the dispersibility of Fe_3O_4 is high, the Ag content in the Ag/Fe_3O_4 product is high, so the catalytic activity of the material is good.

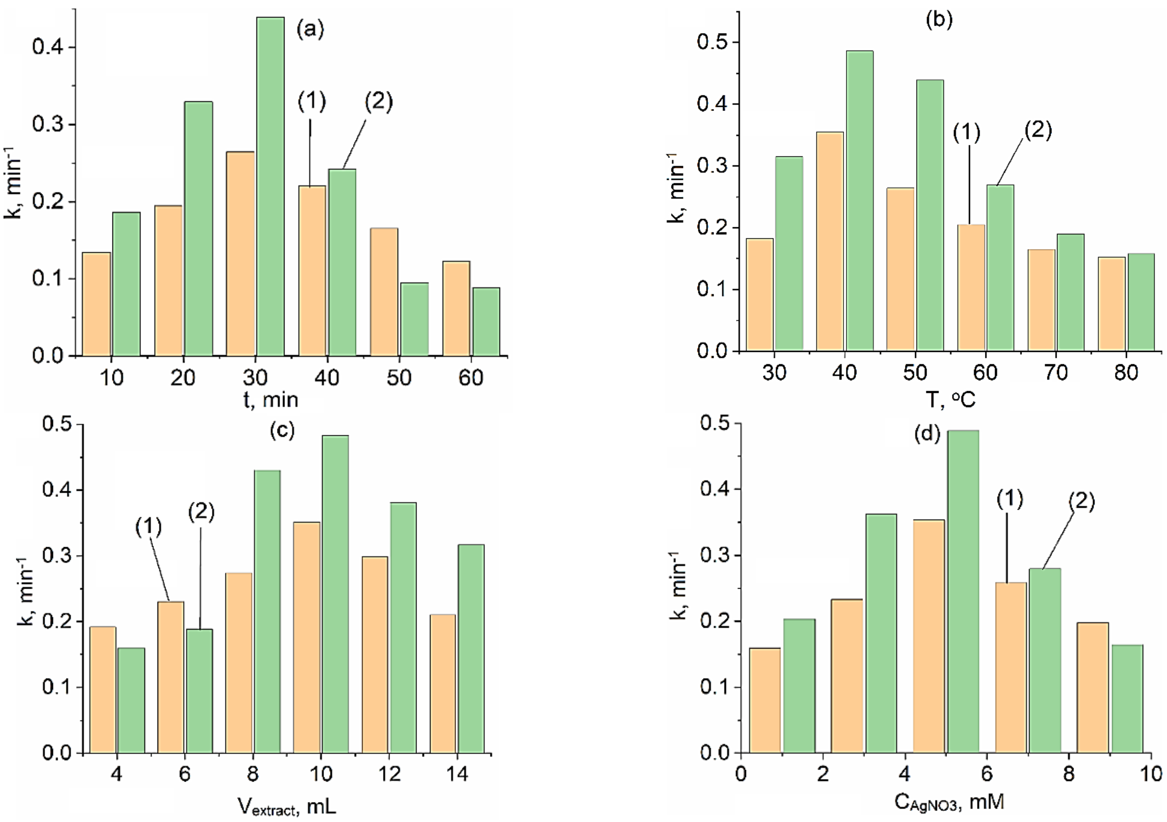

Fig. 5. Dependence of reduction rate constants of MB (1) and RhB (2) on (a) synthesis time; (b) synthesis temperature; (c) volume of extract; (d) concentration of $AgNO_3$

Рис. 5. Зависимость константы скорости восстановления MB (1) и RhB (2) от: (a) времени наноформирования; (b) температуры наноформирования; (c) объема экстракта; (d) концентрации $AgNO_3$

Ag/Fe_3O_4 materials were synthesized under the following conditions: extract volume of 10 mL/50 mL of 5 mM $AgNO_3$ solution at 40 °C for 30 minutes have the best catalytic activity. The reduction rate constants of MB and RhB with $NaBH_4$ in the presence of Ag/Fe_3O_4 are 0.3534 min^{-1} and 0.4886 min^{-1}, respectively. Compared with similar catalysts, the Ag/Fe_3O_4 material synthesized by the green synthesis method using guava leaf extract in this work has significantly higher catalytic activity for the reduction of MB and RhB by $NaBH_4$ (Table 1).

The results of the investigation into the reusability of the catalyst show that, after 3 cycles, there is a decrease in the MB and RhB elimination rate with the removal efficiency dropping from 92.8% (for MB) and 91.6% (for RhB) in the first catalytic cycle to 75.2% (for MB) and 74.5% (for RhB) in the third catalytic cycle (Table 2). After consecutive catalytic cycles, the Ag/Fe_3O_4 material might slightly lose silver, which can, in turn, affect its catalytic performance.

Table 1

Comparison of results for the reduction rates of MB and RhB by $NaBH_4$ in the presence of different catalysts

Таблица 1. **Сравнение скоростей восстановления MB и RhB борогидридом натрия в присутствии разных катализаторов**

Catalysts	k (min^{-1})	Reference
MB		
AgNPs	0.101	[23]
Fe_3O_4@Nico-Ag	0.40	[22]
Fe_3O_4-Ag	0.26	[24]
Ag/Fe_3O_4	0.3534	This work
RhB		
AgNPs	0.1542	[25]
Fe_3O_4@Nico-Ag	0.23	[22]
Ag-Fe_3O_4	0.42	[26]
Ag/Fe_3O_4	0.4886	This work

Table 2

The removal efficiency of MB and RhB in three consecutive catalytic cycles

***Таблица 2.* Эффективность удаления MB и RhB в трех последовательных каталитических циклах**

	Cycle 1	Cycle 2	Cycle 3
MB	92.8%	85.4%	75.2%
RhB	91.6%	82.3%	74.5%

CONCLUSIONS

Ag-Fe_3O_4 materials were successfully synthesized by the green synthesis method using guava leaf extract and characterized by various techniques.

The synthesized Ag/Fe_3O_4 material has good catalytic activity for the reduction of MB and RhB with $NaBH_4$. The Ag/Fe_3O_4 composite increases the reduction rate of MB and RhB by approximately 26.3 times and 57.8 times compared to without a catalyst. The results showed that the Fe_3O_4 phase acts as a carrier, determining the magnetism, while the Ag phase acts as the catalytic center, determining the catalytic activity of the material.

Ag/Fe_3O_4 materials were synthesized under the following conditions: 12.5 g guava leaves/200 mL distilled water, extraction time: 20 min, extraction temperature: 70 °C, extract volume of 10 mL/50 mL of 5 mM $AgNO_3$ solution at 40 °C for 30 min have the best catalytic activity. The reduction rate constants of MB and RhB with $NaBH_4$ in the presence of Ag/Fe_3O_4 are 0.3534 min^{-1} and 0.4886 min^{-1}, significantly higher than similar catalysts.

The catalyst was recyclable for 3 catalytic cycles with a drop from 92.8% (91.6%) to 75.2% (74.5%) in the removal efficiency for MB (RhB).

The authors declare the absence of a conflict of interest warranting disclosure in this article.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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