

## Photocatalytic properties of ferrite/activated carbon composites for degradation of Malachite Green in aqueous medium

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Received: October 12, 2015; Revised: November 11, 2015

Ferrite/activated carbon (AC) composites of different formulation:  $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ,  $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ , and  $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  were prepared by coprecipitation and thermal treatment in nitrogen and studied as photocatalysts for oxidative degradation of Malachite Green dye in aqueous solution under UV-A light irradiation. The phase composition and magnetic behaviour of the prepared materials were established by powder X-ray diffraction analysis and Mössbauer spectroscopy. Synthesized ferrite/AC composites exhibit a superparamagnetic and collective magnetic excitation behaviour. A degradation degree of Malachite Green dye, used as model pollutant, reached 93% for  $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  and 99% for  $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  and  $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  composites, which shows that these materials are applicable as photocatalysts for purification of waters containing above-mentioned dye. Photocatalytic results indicate that  $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  manifested the highest photocatalytic activity. Comparison of the photocatalytic properties of the synthesized ferrite/activated carbon materials proved that the rate constants over the tested materials are increasing in the following order:  $7.1 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ) <  $24.1 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ) <  $28.4 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ).

**Keywords:** photocatalytic properties, Malachite Green dye, ferrite/activated carbon composites.

### INTRODUCTION

Nanodimensional spinel ferrites with general formula  $\text{MFe}_2\text{O}_4$  (M = Mn, Co, Ni, Cu, or Zn) are magnetic materials of certain chemical and thermal stability. The electromagnetic properties of ferrites are related to their microstructure and composition depending on the preparation conditions [1]. Activated carbons (ACs), also known as active charcoal, are very promising and versatile materials used for example as catalysts, catalyst supports, and effective adsorbents exhibiting highly developed porosity, large surface area, high degree of surface reactivity, and variable characteristics of surface chemistry. There are several possible approaches aimed at improving the application potential of activated carbons. One of them is formation of composite materials obtained by incorporation of appropriate inorganic (nano) particles in the structure of activated carbons [2]. Dye contaminants originating from the textile industries are among the major sources of environmental pollution. Semiconductor photocatalytic reactions have attracted considerable interest of the researchers in recent decades [3]. Activated carbon/cobalt ferrite/alginate composite beads, being a magnetically separable adsorbent prepared using a simple ionic polymerization route, have been used for effective removal of Methylene Blue dye from aqueous solution [4]. An  $\text{AC}/\text{CoFe}_2\text{O}_4$

composite has been prepared by a simple single-step refluxing route and used as adsorbent for removal of Malachite Green dye from water [5]. The catalytic activity of cobalt ferrite nanoparticles hosted in activated carbon [6] and nanostructured iron and mixed iron-cobalt oxides supported on activated carbon [7] has been studied for methanol decomposition [6,7].  $\text{CuFe}_2\text{O}_4/\text{AC}$  magnetic adsorbents prepared by a chemical coprecipitation method showed a high adsorption capacity for Acid Orange II (AO7) in water and later it could be separated from the medium by a magnetic technique [8].  $\text{MnFe}_2\text{O}_4/\text{AC}$  magnetic composites, prepared by a simple chemical coprecipitation procedure, have demonstrated high adsorption efficiency for tetracycline in water [9]. Wan *et al.* [10] have studied the removal efficiency of sulphamethoxazole in aqueous solution by using magnetic manganese ferrite nanoparticles on activated carbon synthesized by chemical coprecipitation at various temperatures ranging from 25 to 150°C [10]. Co, Cu, Li, Zn, Ni, and Mg ferrites have been synthesized by impregnation of iron and metal salt aqueous solutions onto activated carbon followed by calcination at low temperatures of 300–500°C [11]. They have been tested for hydrogen sulphide adsorption [11].  $\text{NiFe}_2\text{O}_4/\text{AC}$  composites were obtained by coprecipitation method [12] or facile hydrothermal synthesis [13] and used for removal of Alizarin Yellow R [12] and Methyl Orange [12,13] in water [12,13].  $\text{NiFe}_2\text{O}_4$  supported on activated carbon from petroleum coke (petcoke) was prepared

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and the magnetic properties of the nanoparticles indicated a strong dependence on particle size [14]. Zinc ferrites were obtained in the presence of activated carbon, activated carbon fibre, and Yallourn coal and their adsorption properties for H<sub>2</sub>S were examined [15]. ZnFe<sub>2</sub>O<sub>4</sub> supported on porous carbon was synthesized by pyrogenation of a mixture of ZnCl<sub>2</sub>, Fe(NO<sub>3</sub>)<sub>3</sub>, and novolac resin and it was used for catalytic degradation of mercaptan under alkaline-free conditions [16]. Manganese-zinc ferrites/AC nanocomposites (Mn<sub>0.5</sub>Zn<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub>) obtained by using the hydrothermal method demonstrated some fine adsorptive efficiency for Methylene Blue and a magnetic response [17].

Malachite Green (MG) is an N-methylated diaminotriphenylmethane basic dye [18]. Its chemical structure is shown in figure 1. The crystalline powder of this dye dissolved in water gives a bluish-green coloured solution [19]. MG is widely used as a colouring agent on wool, silk, jute, cotton, leather, paper, and acrylic. This dye is known to be carcinogenic, mutagenic, and teratogenic to mammalian cell. It has potentially harmful effects on the liver, kidneys, gills, gonads, and intestines in organisms [20]. The release of coloured wastewater containing dyes and their by-products has led to great damage to plants and aquatic animals [21]. Consequently, the elimination of Malachite Green from polluted water before its entrance into the food chain and environment is a very important and necessary action [22].

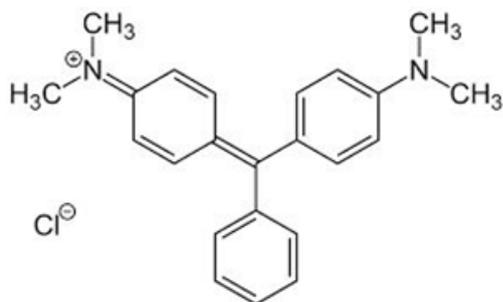


Fig. 1. Chemical structure of Malachite Green dye.

The present article deals with a study and comparison of photocatalytic activities of ferrite/activated carbon materials, such as Co<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>/AC, Cu<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>/AC, and Mn<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>/AC, prepared by coprecipitation technique and thermal treatment in nitrogen. Physicochemical methods including powder X-ray diffraction analysis and Mössbauer spectroscopy were applied to investigate the phase composition and magnetic behaviour of composite samples. In order to establish changes in photocatalytic efficiency of the ferrite/activated carbon composites, they were tested in the reaction of oxidative degradation of Malachite Green dye under UV irradiation.

## EXPERIMENTAL

Ferrite/activated carbon composites having different compositions: Co<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>/AC (sample 1), Cu<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>/AC (sample 2), and Mn<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>/AC (sample 3) were synthesized by the coprecipitation technique. Initial 0.03 M aqueous solutions of Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O (Alfa Aesar) and Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (GPR Rectapur/VWR Prolabo Chemicals), Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (Merck) or Mn(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O (Alfa Aesar) were mixed at a preset ratio. Activated carbon (2 g), obtained from peach stones (provided by Laboratory of Chemistry of Solid Fuels, Institute of Organic Chemistry with Centre of Phytochemistry, Sofia), was then added to the mixture. A precipitating agent, 0.3 M NaOH (Valerus Co.), was slowly added dropwise into the mixture under continuous stirring. Upon reaching pH = 12.5 for samples 1 and 3 and pH = 13 for sample 2, NaOH addition was discontinued and the mixture was aged under continuous stirring for one hour. Obtained samples were centrifuged and washed with distilled water until a neutral reaction (pH = 7) was attained. The so-prepared precipitates were dried at 110°C for 1 h in air. Afterwards all the samples were calcined at 400°C for 3 h in inert nitrogen.

The prepared ferrite/activated carbon materials were investigated by powder X-ray diffraction analysis (PXRD) and Mössbauer spectroscopy in order to determine their phase composition and magnetic behaviour.

The PXRD analysis was carried out using a TUR M62 apparatus with PC control and data acquisition applying an HZG-4 goniometer and CoK $\alpha$  radiation. Phases were identified by means of JCPDS database (Powder Diffraction Files, Joint Committee on Powder Diffraction Standards, Philadelphia PA, USA, 1997).

Mössbauer spectra were registered on a Wissenschaftliche Elektronik GmbH apparatus, working at a constant acceleration mode, and using a <sup>57</sup>Co/Rh source (activity  $\approx$ 50 mCi) and  $\alpha$ -Fe standard. The parameters of hyperfine interactions of Mössbauer spectral components: isomeric shift (IS), quadrupole splitting (QS), effective hyperfine magnetic field at iron nuclei (H<sub>eff</sub>), line widths (FWHM), and component relative weights (G) were estimated by computer fitting.

The photocatalytic properties of thermally treated ferrite/AC composites were tested for oxidative degradation of Malachite Green dye in aqueous solution under UV-A light illumination applying polychromatic (320–400 nm) radiation with maximal emission at 365 nm and a black light blue BLB lamp of 18 W. The reaction course was followed spectrophotometrically using a CamSpec M501 UV-vis

spectrophotometer based on the absorbance of dye solution in the UV-vis wavelength range from 200 to 800 nm. A semi-batch suspension photocatalytic reactor was used applying a fixed amount of photocatalyst and polluted water and continuous airflow through two frits to saturate the solution in oxygen. Thus, oxygen was in large stoichiometric excess, its concentration was practically constant, and it could be included in the value of the apparent rate constant. Therefore, the Langmuir-Hinshelwood mechanism of a bimolecular reaction is reduced to a pseudo-first order rate equation. The photocatalytic measurements were performed at a constant stirring rate (400 rpm) under ambient conditions. MG dye solutions had an initial concentration of 5 ppm. The investigated systems reached an equilibrium state after about 30 min in the dark before switching on the lamp. Sample aliquots of the suspension were taken out of the reaction vessel after regular time intervals in order to measure the photocatalytic activities of ferrite/activated carbon materials. The powder was then separated from the aliquot solution by centrifugation before UV-Vis spectrophotometric measurement of dye concentration. After that the aliquot solution together with the photocatalyst sample were placed back into the reaction vessel (constant volume operation). Apparent rate constants,  $k$ , were estimated by neglecting the dissolved oxygen con-

centration based on the straight-line slope of plotted  $-\ln(C/C_0) = k_{app} \cdot t$  logarithmic dependence.

## RESULTS AND DISCUSSION

Figure 2 illustrates powder X-ray diffraction patterns of the synthesized ferrite/activated carbon materials and activated carbon. As it can be seen, the presence of non-stoichiometric spinel cobalt ferrite  $\text{Co}_x\text{Fe}_{3-x}\text{O}_4$  (PDF-22-1086; PDF-75-0449), copper ferrite  $\text{Cu}_x\text{Fe}_{3-x}\text{O}_4$  (PDF-34-0425; PDF-75-0449), or manganese ferrite  $\text{Mn}_x\text{Fe}_{3-x}\text{O}_4$  (PDF-73-1964; PDF-75-0449), and also activated carbon phases were registered in presented PXRD patterns. A hematite phase (PDF-89-0599) was also observed in the case of thermally treated cobalt and copper ferrite-type/AC materials.

The registered Mössbauer spectra of the obtained samples at room temperature (RT) are displayed in figure 3. The spectra of  $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  (sample 1) and  $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  (sample 2) are a superposition of sextet and doublet components. Doublet lines were only recorded in the spectrum of  $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  (sample 3). A spinel phase, registered in the spectrum of sample 1, is presented by three sextet components: Sxt2 due to octahedrally coordinated  $\text{Fe}^{3+}$  ions, Sxt3 belonging to tetrahedrally coordinated  $\text{Fe}^{3+}$  ions, and Sxt4 – related to iron ions in a spinel phase with an average size below 20 nm. This phase

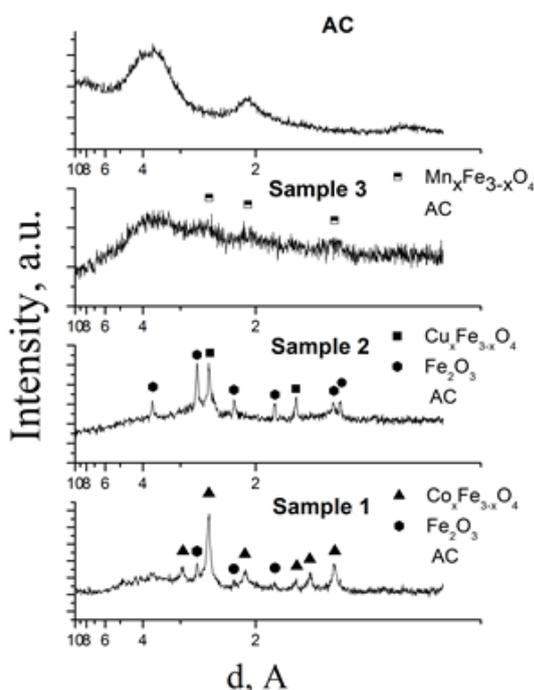


Fig. 2. PXRD patterns of thermally treated ferrite/AC composites and activated carbon.

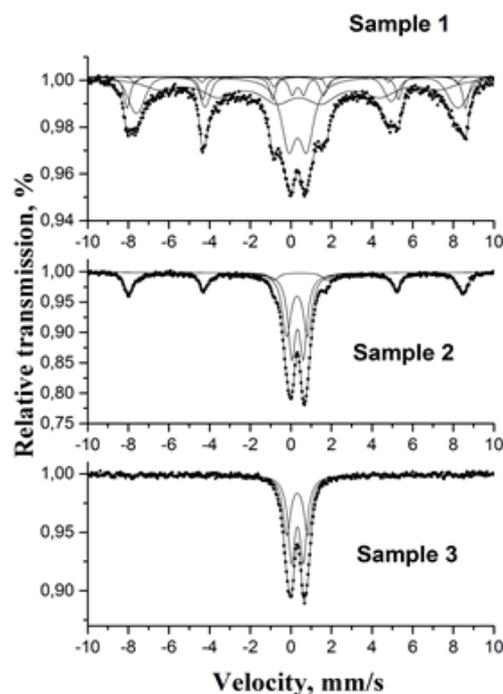


Fig. 3. Mössbauer spectra of ferrite/AC composites at room temperature.

exhibits a high dispersion degree and collective magnetic excitation (CME) behaviour. In the spectra of sample 1 and sample 2, the sextet component, Sxt1, is attributed to ferric ions ( $\text{Fe}^{3+}$ ) in a hematite phase. Calculated hyperfine parameters of doublet components could be assigned to the presence of ferrite particles of superparamagnetic (SPM) behaviour and an average size below 10 nm [23] (Table 1). Mössbauer measurements confirmed the powder X-ray diffraction studies.

The degree of degradation of the Malachite Green dye as a function of time interval under UV-A light irradiation during photocatalytic tests over prepared ferrite/AC composites is given in figure 4. The results indicate that samples 1 and 2 manifested the highest photocatalytic activity (99%) in the oxidative degradation of Malachite Green dye after 120 min.

The apparent rate constants of ferrite/AC photocatalysts determined by the slopes of the curves

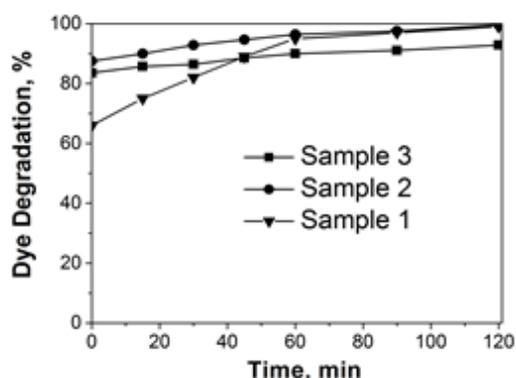
using pseudo-first order kinetics [3],  $-\ln(C/C_0) = k_{\text{app}} \cdot t$ , are shown in figure 5. Differences in photocatalytic activity of tested materials proved that a maximum in the rate constant was observed with sample 1 ( $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ). The photocatalytic activity of the ferrite/AC samples is decreasing in the order:  $28.4 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ) >  $24.1 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ) >  $7.1 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ).

## CONCLUSIONS

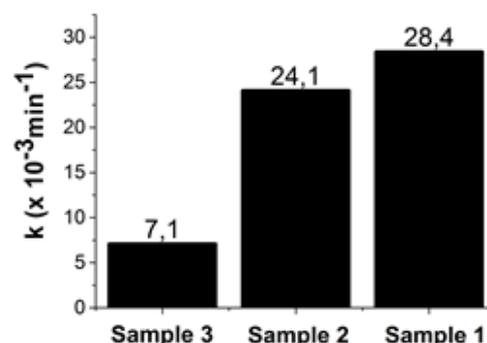
A coprecipitation procedure followed by thermal treatment in inert nitrogen at  $400^\circ\text{C}$  for 3 h was successfully used to prepare ferrite/activated carbon photocatalysts of different composition:  $\text{M}_x\text{Fe}_{3-x}\text{O}_4/\text{AC}$  ( $\text{M} = \text{Co}^{2+}, \text{Cu}^{2+}, \text{Mn}^{2+}; x = 0.5$ ). A powder X-ray diffraction study showed that spinel ferrite and activated carbon phases were registered

**Table 1.** Mössbauer parameters of prepared ferrite/AC materials.

Sample	Component	IS $\text{mm.s}^{-1}$	QS $\text{mm.s}^{-1}$	$H_{\text{eff}}$ T	FWHM $\text{mm.s}^{-1}$	G %
sample 1	Sxt 1 – $\text{Fe}^{3+}$ hematite	0.37	-0.21	51.8	0.35	10
	Sxt 2 – $\text{Fe}^{3+}$ octa	0.24	0.05	49.3	0.31	2
	Sxt 3 – $\text{Fe}^{3+}$ tetra	0.34	-0.05	49.2	0.33	22
	Sxt 4 – Fe - spinel, CME	0.41	0.02	42.8	1.94	40
	Dbl 1 – SPM	0.35	0.57	–	0.39	3
sample 2	Dbl 2 – SPM	0.34	0.90	–	0.85	23
	Sxt 1 – $\text{Fe}^{3+}$ hematite	0.36	-0.21	51	0.54	28
	Dbl 1 – SPM	0.33	0.58	–	0.43	37
sample 3	Dbl 2 – SPM	0.31	1.04	–	0.51	35
	Dbl 1 – SPM	0.33	0.59	–	0.41	55
	Dbl 2 – SPM	0.32	1.04	–	0.45	45



**Fig. 4.** Degree of degradation of MG dye calculated as  $[(C_0 - C)/C_0] \times 100\%$ , where  $C_0$  is the initial concentration before switching on illumination and  $C$  is current concentration of the solution based on changes in peak intensity having maximal absorbance at 615 nm with time under UV-A irradiation using synthesized ferrite/AC samples in a semi-batch reactor.



**Fig. 5.** Comparison data of calculated apparent rate constants,  $k$  (disregarding dissolved oxygen concentration) under UV-A illumination using ferrite/AC photocatalysts in photodegradation of Malachite Green dye.

in all the samples. A hematite phase was also observed in the case of calcined cobalt and copper ferrite-type/AC materials. Mössbauer measurements at room temperature verified that the synthesized ferrite/AC composites exhibited a superparamagnetic and collective magnetic excitation behaviour and indicated a highly dispersed ferrite phase of an average particle size below 20 nm. Photocatalytic activity tests demonstrated a high degree of degradation of the Malachite Green dye, used as model pollutant, being within 93–99% for obtained ferrite/AC composites. The present results give evidence that the prepared ferrite/activated composites could be successfully applied to purify polluted waters containing MG dye as contaminant. A certain effect of the different metal ions on the photocatalytic activity was established. The photocatalytic apparent rate constants (a pseudo-first order rate equation) of the investigated samples are increasing as follows:  $7.1 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ) <  $24.1 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ) <  $28.4 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ). Among the three materials, the  $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  composite manifested the highest photocatalytic activity.

**Acknowledgements:** The authors thank the Bulgarian Science Fund for financial support by Contract DFNI E02/2/2014.

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## ФОТОКАТАЛИТИЧНИ СВОЙСТВА НА КОМПОЗИТИ ФЕРИТ/АКТИВЕН ВЪГЛЕН ЗА РАЗГРАЖДАНЕ НА МАЛАХИТОВО ЗЕЛЕНО ВЪВ ВОДНА СРЕДА

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Постъпила на 12 октомври 2015 г.; Преработена на 11 ноември 2015 г.

(Резюме)

Композити ферит/активен въглен с различен състав,  $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ,  $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  и  $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ , бяха получени чрез съутаяване и термична обработка в азотна среда и изследвани като фотокатализатори в окислителното разграждане на Малахитово Зелено (МЗ) багрило във воден разтвор под УВ-А облъчване. Фазовият състав и магнитното поведение на получените материали бяха установени чрез рентгенофазов анализ и Мьосбауерова спектроскопия. Синтезираните композити ферит/активен въглен притежават суперпарамагнитно поведение и колективно магнитно възбуждане. Високите степени на разграждане на багрилото, използвано като моделен замърсител, възлизат на 93% за образеца  $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  и 99% за  $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  и  $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  композити, което показва, че тези материали са приложими като фотокатализатори за пречистване на води съдържащи Малахитово Зелено. Получените фотокаталитични резултати показаха, че образецът  $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$  има най-висока фотокаталитична активност. Сравнение на фотокаталитичните свойства на синтезираните материали ферит/активен въглен установи, че скоростните константи на изследваните образци нарастват в реда:  $7.1 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Mn}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ) <  $24.1 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Cu}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ) <  $28.4 \times 10^{-3} \text{ min}^{-1}$  ( $\text{Co}_{0.5}\text{Fe}_{2.5}\text{O}_4/\text{AC}$ ).