University of Belgrade, Technical Faculty in Bor

29th International Conference Ecological Truth & Environmental Research

EcoTER'22

Proceedings

Editor
Prof. Dr Snežana Šerbula

21-24 June 2022, Hotel Sunce, Sokobanja, Serbia

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PREFACE

In today's world, the environment has been endangered by the use of outdated technology, fossil fuels and environmental law violations. Therefore, environmental and many other scientists all over the world have been concerned about finding sustainable technology in resolving these issues. That is why environmental research and ecological truth are at the focus of the 29th International Conference Ecological Truth & Environmental Research 2022 (EcoTER'22), which will be held in Sokobanja, Serbia, 21–24 June 2022. On behalf of the Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the Conference.

We hope to convey the message of the conference, which is that a transformation of attitudes and behavior would bring the necessary changes. This is also an opportunity for the participants who are experts in this field to exchange their experiences, expertise and ideas, and also to consider the possibilities for their collaborative research.

The 29th International Conference Ecological Truth & Environmental Research 2022 is organized by the University of Belgrade, Technical Faculty in Bor, and co-organized by the University of Banja Luka, Faculty of Technology, the University of Montenegro, Faculty of Metallurgy and Technology – Podgorica, the University of Zagreb, Faculty of Metallurgy – Sisak, the University of Pristina, Faculty of Technical Sciences – Kosovska Mitrovica and the Association of Young Researchers, Bor.

These proceedings include 85 papers from the authors coming from the universities, research institutes and industries in 6 countries: Bulgaria, Italia, Albania, Bosnia and Herzegovina, Montenegro and Serbia.

As a part of this year's conference, the 4th Student section – EcoTERS'22 is being held. We appreciate the contribution of the students and their mentors who have also participated in the Conference.

Financial assistance provided by the Ministry of Education, Science and Technological Development of the Republic of Serbia is gratefully acknowledged by the Organizing Committee of the EcoTER'22 conference.

The support of the Platinum donor and their willingness and ability to cooperate have been of great importance for the success of EcoTER'22. The Organizing Committee would like to extend their appreciation and gratitude to the Platinum donor of the Conference for their donation and support.

We appreciate the effort of all the authors who have contributed to these Proceedings. We would also like to express our gratitude to the members of the scientific and organizing committees, reviewers, speakers, chairpersons and all the Conference participants for their support to EcoTER'22. Sincere thanks go to all the people who have contributed to the successful organization of EcoTER'22.

Prof. Snežana Šerbula,

President of the Organizing Committee

APPLICATION OF NANO- MnO_2 MODIFIED LIGNIN – BASED ADSORBENT FOR REMOVAL OF DICHROMATE IONS AND DICLOFENAC FROM WATER

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Abstract

The aim of this paper is to investigate the application of modified lignin-based adsorbent for successful removal of dichromate ions and sodium diclofenac (DCF) from aqueous solutions by adsorption. Modification of lignin-based adsorbent (LBA) was performed by inverse suspension copolymerization with branched poly (ethylene-imine) using epichlorohydrin as a crosslinker. After that, the functionalization of LBA was performed by chemical binding of amino modified nanoparticles of manganese (IV) oxide, in order to improve the adsorption properties. Characterization of LBA-MnO_2 microspheres was performed using FT-IR spectrometer, Scanning electron microscopy, BET/BJH analysis. The adsorption process was performed in a batch adsorption system. The results were obtained with the help of kinetic and corresponding equilibrium adsorption isotherms. The maximum adsorption capacity for the removal of $Cr_2O_7^{2-}$ ions and DCF was obtained using the Langmuir model and amounts to 88.4 mg g^{-1} and 52.8 mg g^{-1} at 45 °C, respectively. Adsorption kinetics was described using a second-order pseudo model. Based on thermodynamic parameters, it was concluded that the adsorption process is an endothermic and spontaneous. Based on the obtained results, LBA-MnO_2 material possess very good adsorption properties.

Keywords: sodium diclofenac, dichromate ions, adsorption, lignin-based adsorbent

INTRODUCTION

Rapid industrial development and increased number of inhabitants are continuously causing an increased amount of generated waste, which easily reaches water recipients. Heavy metals, widely spread in water, belong to the group of the most dominant pollutants [1]. Heavy metals accumulate in human body due to the consumption of contaminated water, which can lead to various diseases [2]. They are toxic, even at low concentrations, and not biodegradable [2]. Also, there is an increasing concentration of pharmaceuticals in drinking water and wastewater, which can pose a great danger to human life [3,4]. In recent years, sorbents based on natural materials, originating from renewable or waste sources, have been used for removing heavy metal ions and pharmaceuticals [5]. The adsorption process is mostly used because of its simplicity and efficiency. Economy, distribution and beneficial impact on the environment are the main reasons for using sorbents based on natural materials for removing heavy metals ions and pharmaceuticals from aqueous systems [6,7]. Lignin can be obtained by processing biomass, as a by-product in the pulp and paper industry [8–11]. In this study, the modified craft lignin has been used as a bio-sorbent for removing inorganic (dichromate ions) and organic pollutants (sodium diclofenac, DCF). In order to characterize bio-sorbent Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and BET/BJH were performed. Variation of adsorption efficiency and concentration of heavy metals ions were determined varying temperature, mass of the adsorbent, pH value of the solution and contact time duration in the scoring system. Langmuir, Freundlich and similar isotherm models were applied to determine the maximum adsorption capacities for the removal of heavy metal ions and DCF. Adsorption kinetics was calculated using first, second and pseudo second order.

The aim of this paper is to investigate the possibility of using MnO_2-modified lignin-based adsorbent to remove cations of dichromate and sodium diclofenac from aqueous solutions by adsorption process.

MATERIALS AND METHODS

A modified process based on the inverse suspension copolymerization [1] of kraft lignin with branched poly (ethylene-imine), PEI, to obtain an amino functional adsorbent and with the help of epichlorohydrin as a crosslinking agent, the synthesis of lignin bio-sorbent was performed. 10 mL of deionized water and 0.5 g of lignin were added to the three-necked balloon. Further, 2.0 g of PEI, 0.1 g of sodium dodecyl benzene sulfonate, as well as 10 mL of sodium alginate emulsifier solution, were added with stirring. Stirring was continued for 30 minutes at 60°C. In order to obtain a suspension, 80 mL of liquid paraffin was added to the flask. The copolymerization process was completed by adding dropwise 2.0 mL of crosslinker, epichlorohydrin, with the constant stirring for 120 minutes. The obtained copolymerized LBA, was centrifuged, washed with ethanol, ether and finally with water. The process of lyophilization was performed by drying LBA for 24 hours at 40°C. The synthesis of manganese (IV) oxide nanoparticles was performed by the reaction between potassium permanganate ($KMnO_4$) and ethyl alcohol (C_2H_5OH). 0.5 g of $KMnO_4$ was dissolved in 300 mL of distilled water. With constant stirring, 10 mL of C_2H_5OH was added dropwise to the mixture. A dark brown precipitate was formed, which was filtered and washed. Amino-functionalization resulted in the formation of nanoparticles, with the use of (3-aminopropyl) triethoxysilane (APTES) and the formation of amino-modified nanoparticles of manganese (IV) oxide (NP-OA-MnO_2). The LBA adsorbent was modified with the help of synthesized MnO_2 nanomaterial. Adsorbate solutions of sodium diclofenac and potassium dichromate (initial concentration 10 mg L^{-1}, both) were prepared by dilution of standard solutions (1000 mg L^{-1}).

RESULTS AND DISCUSSION

The textural properties of the LBA-MnO_2 adsorbent, determined by BET/BJH analysis, are presented in Table 1. Specific surface area (S_{BET}), pore volume (V_{tot}) and intermediate pore (V_{meso}), and mean pore diameter (D_m) were determined. The specific surface area of samples was calculated according to the Brunauer, Emmett, Teller (BET) method from the linear part of the nitrogen adsorption isotherms. The total pore volume (V_{tot}) was given at $p/p_0 = 0.98$. The volume of the mesopores was calculated according to the Barrett, Joyner and Halenda (BJH) method from the desorption branch of isotherm.

***Table 1** Textural properties of LBA-MnO_2 adsorbent*

Samples	S_{BET}, m^2/g	V_{tot}, cm^3/g	V_{meso}, cm^3/g	D_m, nm
LBA-MnO_2	188.4	0.3256	0.3045	5.12

SEM micrographs of the LBA-MnO_2 sample are shown in Figure 1. As can be seen, small particles of irregular shape were observed, in which the agglomeration process can be noticed. The reason for this is the low concentration of emulsifiers (≤1.0 wt. %).

a) *b)*

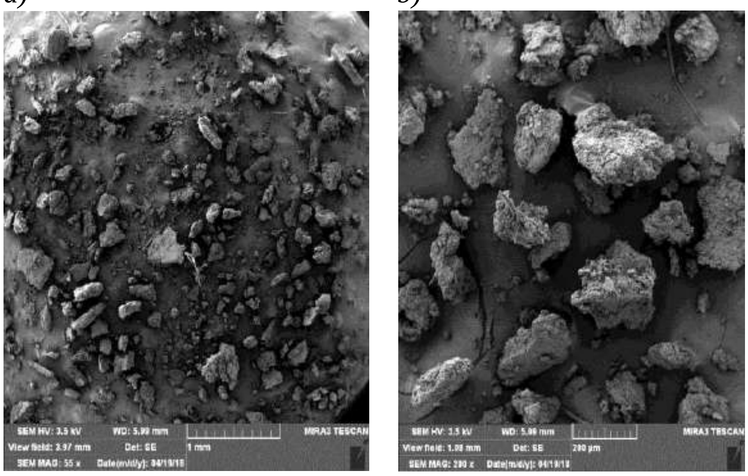

***Figure 1** SEM image of LBA-MnO_2 sample*

Figure 2 represents the FT-IR spectrum of the LBA-MnO_2 sample. The peak at 3329 cm^{-1} of the LBA-MnO_2 sample corresponds to the phenolic and hydroxyl vibrations of the O-H bond elongation [12,13]. Bands at 1593 cm^{-1}, as well as in the range of 1400–1470 cm^{-1}, indicate the presence of an aromatic structure from lignin [12,13]. The peaks at 2849 and 2917 cm^{-1} refer to the symmetric and asymmetric C-H tensile vibrations of the methylene group [14].

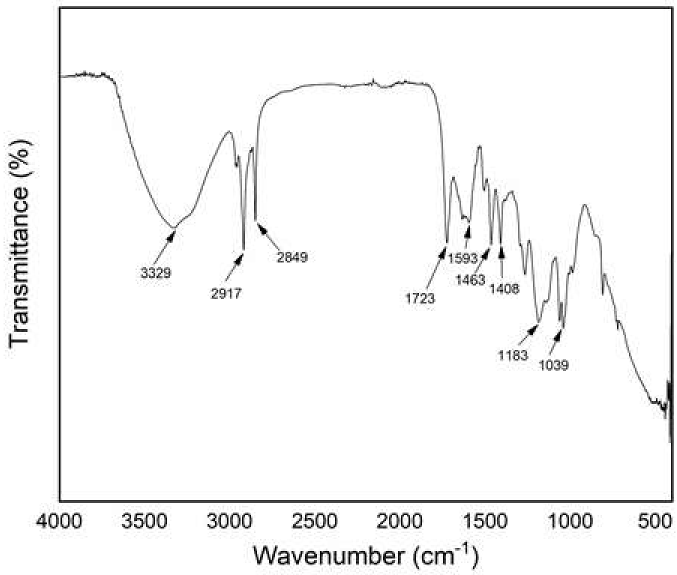

Figure 2 *FT-IR spectrum of the LBA-MnO_2 sample*

Nonlinear models of Langmuir, Freundlich, Temkin and Dubinin–Radushkevich adsorption isotherms are presented in Tables 2 and 3. Based on the results from Table 2, obtained with the help of Langmuir adsorption model, the adsorption capacity (q_e) increases with increasing temperature. Also, with increasing temperature there is an increase in Langmuir constant (K), which shows that at higher temperatures there is higher affinity of LBA-MnO_2 adsorbent. The maximum adsorption capacity (q_e) during the adsorption of $Cr_2O_7^{2-}$ ions and DCF is 88.4 and 52.8 at 45°C, respectively.

Table 2 *Results of Langmuir adsorption model when removing* $Cr_2O_7^{2-}$ *and DCF at 25, 35 and 45°C*

Lengmuir isotherm		q_m **(mg g^{-1})**	K_L**(L mg^{-1})**	**R^2**
LBA-MnO_2- $Cr_2O_7^{2-}$	25°C	78.653	0.14833	0.99091
	35°C	83.639	0.15172	0.99679
	45°C	88.434	0.15620	0.99748
LBA-MnO_2-DCF	25°C	43.498	0.54497	0.97590
	35°C	48.086	0.48902	0.97190
	45°C	52.831	0.44583	0.96781

Freundlich parameter (1/n) is less than 1 for both DCF and $Cr_2O_7^{2-}$ adsorption, indicating heterogeneity of the surface. A slight increase of 1/n value with increasing temperature due to subsequential heterogenization of the surface may be observed in Table 3. Also, from the same table, 1/n values for adsorption of sodium diclofenac and dichromate noticeably differ. It may be concluded that the surface is more homogenized after sodium diclofenac sorption, which may be explained as similiraty in structure of sodium diclofenac and lignine.

With the help of Dubinin – Radushkevich and Temkin's model, significant data on the adsorption process were obtained. Based on the values of energy adsorption (E) for the adsorption of $Cr_2O_7^{2-}$ and DCF obtained on the basis of the Dubinin – Radushkevich model, it can be concluded that the ion exchange mechanism is dominant and its value ranges around 8 kJ mol^{-1}. Also, based on the value of E, it is concluded that adsorption bonding is physisorption (E<8 kJ mol^{-1}) [15]. The bond constant, A, based on the Temkin model, represents the maximum binding energy (dm^3 g^{-1}). The maximum binding energy, for $Cr_2O_7^{2-}$ adsorption is reached at a temperature of 45°C, while for sodium diclofenac adsorption is reached at 25°C. The value of the mean index, B, for both types of pollutants also confirm conclusion made by comparison of energy activation that adsorbate bonding occurs *via* physical adsorption [16].

Table 3 *Results of Freundlich, Dubinin Radushkevich and Tempkin adsorption model when removing* $Cr_2O_7^{2-}$ *and DCF at 25, 35 and 45°C*

Isotherm models and parameters			**Temperature**		
			25°C	**35°C**	**45°C**
Freundlich isotherm	K_F (mg g^{-1})(dm^3 mg^{-1})$^{1/n}$	LBA-MnO$_2$-$Cr_2O_7^{2-}$	9.915232	10.70843	11.56561
	1/n		0.77341	0.77612	0.77780
	R^2		0.99286	0.99407	0.99457
	K_F (mg g^{-1})(dm^3 mg^{-1})$^{1/n}$	LBA-MnO$_2$-DCF	17.79673	18.23528	18.08722
	1/n		0.29308	0.31726	0.34001
	R^2		0.99421	0.99491	0.99422
Dubinin Radushkevich	q_m (mg g^{-1})	LBA-MnO$_2$-$Cr_2O_7^{2-}$	25.16	2.638	27.59
	E (KJ mol^{-1})		8.09316	8.11841	8.14253
	B		7.63	7.59	7.54
	R^2		0.87000	0.86472	0.85912
	q_m (mg g^{-1})	LBA-MnO$_2$-DCF	28.20	719.34	29.71
	E (KJ mol^{-1})		7.34852	8.60338	7.36927
	B		9.26	6.76	9.21
	R^2		0.69894	0.99711	0.68413
Tempkin isotherm	A (dm^3 g^{-1})	LBA-MnO$_2$-$Cr_2O_7^{2-}$	2.492848	2.586535	2.693991
	b		197.03	193.19	189.92
	B (mg g^{-1})		12.58	13.26	13.93
	R^2		0.95123	0.95045	0.94929
	A (dm^3 g^{-1})	LBA-MnO$_2$-DCF	12.359	9.667758	7.98153
	b		346.34	314.28	288.71
	B (mg g^{-1})		7.16	8.15	9.16
	R^2		0.95185	0.94362	0.93564

CONCLUSION

The subject of this paper refers to the application of sorbents based on natural polymer, kraft lignin, which is further modified by manganese dioxide nanoparticles, to remove heavy metal ions, such as $Cr_2O_7^{2-}$ and sodium diclofenac from aqueous solutions by adsorption. According to Langmuir's adsorption model, the highest adsorption capacity of 88.3 mg g^{-1} for removal of $Cr_2O_7^{2-}$ ions was reached at 45°C. When removing sodium diclofenac from water, the maximum adsorption capacity was 52.8 mg g^{-1} at 45°C. The mechanism of adsorption was subjected to Freundlich's model (chemisorption process), with a great contribution of Temkin's model. Based on the value of the mean free adsorption energy, it was determined that an overall removal process is a physisorption. Based on the presented results, it can be concluded that nano-modified, porous lignin-based adsorbent, LBA-MnO_2, can be used to remove heavy metal ions and sodium diclofenac from the aqueous solutions.

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