University of Belgrade, Technical Faculty in Bor

30th International Conference Ecological Truth & Environmental Research 2023

Proceedings

Editor
Prof. Dr Snežana Šerbula

University of Belgrade, Technical Faculty in Bor

30 EcoTER

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PROCEEDINGS

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PREFACE

The 30th international conference Ecological Truth & Environmental Research – EcoTER'23 kept three areas in focus: ecology, environmental protection and sustainable development. The conference will be held on Mt Stara Planina in hotel Stara Planina, Serbia, 20–23 June 2023. The monograph is published on the occasion of the 30th anniversary of the conference. On behalf of the scientific and organizing committee, it is a great honor and pleasure to wish all the participants a warm welcome to the conference.

The monograph is published on the occasion of the 30th anniversary of 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 30th international conference Ecological Truth & Environmental Research – EcoTER'23 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 103 papers from the authors coming from the universities, research institutes and industries in 11 countries: Australia, USA, Brazil, Spain, Portugal, Libya, Italy, Bulgaria, Bosnia and Herzegovina, North Macedonia, and Serbia.

As a part of this year's conference, the 5th Student Session – EcoTERS'23 is being held. We appreciate the contribution of the students and their mentors who have also participated in the conference.

The support of the Gold donor and their willingness and ability to cooperate has been of great importance for the success of the EcoTER'23. The organizing committee would like to extend their appreciation and gratitude to the Gold 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 the EcoTER'23. Sincere thanks go to all the people who have contributed to the successful organization of the EcoTER'23.

Prof. Snežana Šerbula,

President of the scientific and organizing committee

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MODIFIED MEMBRANES WITH GRAPHENE OXIDE – REMOVAL OF DYES FROM WASTEWATER

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Abstract
Improper disposal of wastewater containing synthetic dyes, due to carcinogenic, teratogenic and mutagenic effects, poses a danger to the environment and human health. Synthetic dyes form wastewater can be effectively removed by membrane separation processes. However, during wastewater treatment, contamination of the membranes can occur and increase the costs of wastewater treatment. Membrane contamination can be overcome by modifying the membranes with graphene oxide. Therefore, the modification of conventional membranes has attracted the attention of numerous researchers in the past few years. This paper provides an overview of scientific research on methods of membrane modification with graphene oxide and the effectiveness of removing synthetic dyes from wastewater.

Keywords: modified membranes, graphene oxide, synthetic dyes, wastewater treatment.

INTRODUCTION

Synthetic dyes are widely used in various industrial sectors, for dyeing paper, leather, textiles, plastics, etc. [1,2]. Contamination of aquatic ecosystems with synthetic dyes is a growing problem worldwide, because about 20% of the total amount of produced dyes is released into the environment without adequate treatment [3,4]. The presence of dyes in surface waters can reduce solar air infiltration and negatively affect photosynthetic activities [1,3]. In addition, consumption of water contaminated with synthetic dyes can lead to kidney disease, liver disease and damage to the central nervous system [1]. Therefore, the removal of synthetic dyes from industrial wastewater is important in protecting the environment and preserving human health.

Membrane separation processes represent an efficient and economical method for dyes removal, because they not require the use of flocculants, coagulants, etc. and do not lead to the formation of toxic by–products and secondary pollution of wastewater [1,5,6]. The main disadvantage of membrane separation processes is membrane contamination [7]. In order to overcome these shortcomings and avoid additional costs, numerous research teams propose the modification of conventional membranes with carbon–based nanomaterials.

MODIFICATION OF MEMBRANES WITH GRAPHENE OXIDE

Membrane modification with carbon–based nanomaterials (carbon nanotube, graphene, graphene oxide, etc.) can improve membrane performance and prevent contamination [8].

Graphene oxide (GO) is considered as the most suitable inorganic material for modification of membranes, because its structure contains specific functional groups (epoxy, hydroxyl, and carboxyl groups) that can increase hydrophilic, selectivity, and reduce pollutant permeability by reducing the membrane pore size and changing their surface charge [8–10]. In addition, the introduction of GO nanosheets can limit the migration of polyelectrolyte chains and improve membrane stability [9]. Modification membranes with graphene oxide can be carried out in two ways: by applying nanosheets of GO on the surface of the membrane (membranes surface modification with GO) or by incorporating nanosheets of graphene oxide into the polymer matrix of the membrane (GO–blended membranes).

Membranes surface modification with GO

Layer by layer (LBL) is an environmentally friendly, simple and most commonly applied method for membrane surface modification [9,11]. The principle of membrane modification by the LBL method is based on the adsorption of oppositely charged nanosheets on the membrane surface [9]. GO nanosheets are negatively charged due to the ionization of carboxyl groups and it is necessary to use positively charged polymers. Nanosheets of GO and other compounds can be applied by pressure–assisted filtration, vacuum filtration or immersion of the membrane into the modifying solution. Depending on the last applied layer, the modified membrane can be used to remove cationic or anionic dyes [1].

Numerous researchers [5,9,12–15] have investigated the possibility of modifying membrane surfaces using the LBL method. For example Ding *et al.* [12] modified a polyethersulfone membrane (PES) with GO and TiO_2. PES membrane was synthesized by the phase inversion method and immersed in an aqueous solution of GO@TiO_2 for 1h. After that, the membrane was washed with deionized water to wipe off the redundant solution and then dried. The results of the investigation conducted by Ding *et al.* [12] indicated that after the membrane modification, the contact angle decreased from 71.4° to 34.9°, which indicates that the hydrophilicity of the modified membranes is significantly improved. As already mentioned, GO contains hydroxyl and carboxyl groups that interact with water molecules, thus increasing the hydrophilicity of the membranes. Similar results and observations were obtained by Gu *et al.* [13] and Homem *et al.* [5].

In addition to hydrophilicity, the thermal and mechanical stability of membranes can be improved by modifying the membrane surface with graphene oxide [12]. The results of Fourier–transform infrared spectroscopy, given by Ding *et al.* [12], indicate that GO@TiO_2 nanosheets are bound to the surface of the PES membrane by interfacial interactions, improving its mechanical and thermal stability.

Homem *et al.* [5] modified polyethersulfone microfiltration membrane (mPES) with polyethyleneimine (PEI) and graphene oxide. A layer of polyethylenimine, then graphene oxide, and finally a layer of polyethylenimine were applied to the surface of the membrane using pressure filtration. The results of the study conducted by Homem *et al.* [5] indicate that the performance of the modified mPES membrane compared to the unmodified membrane is significantly improved. By modifying the mPES membrane with GO, the efficiency of removing sea blue colour from aqueous solutions increased from 11.8% to 92.4–97.8%, depending on the concentration of the GO solution used for membrane modification.

A summary of selected studies evaluating efficiency of removing synthetic dyes from aqueous solutions using GO modified membranes can be found in Table 1.

Table 1 *Efficiency of removal of synthetic dyes from wastewater using surface modified GO membranes*

Membrane	Dye	Efficiency (%)	References
GO/α–Ni(OH)$_2$ (6L)	Methylene blue	99.9	[14]
GO/α–Ni(OH)$_2$ (4L)	Victoria blue	99.7	[14]
(PEI/GO)$_3$	Rose Bengal	99.9	[9]
	Brilliant Blue B	98.1	
	Eosin Y	94.7	
	Methylene blue	85.1	
MLn (n– odd number)	Congo red	>99.0	[13]
	Methyl blue		
MLn (n– even number)	Methyl violet	>99.5	[13]
LBL$_3$(GO)	Methyl blue	79.0	[15]
mPES/PEI$_{3.0}$+GO$_{0.025}$+PEI$_{1.5}$	Dark blue	96.1	[5]

Based on the reviewed literature [5,9,13–15] and the data shown in Table 1, it can be concluded that with the use of modified membranes, a high removal efficiency of synthetic dyes from wastewater can be achieved. The high removal efficiency of synthetic dyes is achieved thanks to an efficient barrier that is formed on the surface of the membrane by applying GO nanofilms [5].

Wang *et al.* [9] investigated the effect of GO concentration (0.50–1.25 g/L) on the removal efficiency of Methylene blue (MB). As the concentration of GO increases from 0.5 to 1.0 g/L, defects on the membrane surface decrease, which leads to an increase in MB removal efficiency. However, with increasing GO concentration, the thickness of the films deposited on the membrane surface also increases, which leads to a decrease in membrane permeability. Li *et al.* [14] investigated the influence of the number of GO nanosheets on the flux of permeate. The test results indicate that with an increase in the number of applied layers of GO (1–6), permeate flux decreases from 92.6 L $m^{-2}h^{-1}MPa^{-1}$ to 6.4 L $m^{-2}h^{-1}MPa^{-1}$. Similar results were obtained by Gu *et al.* [13], Yan *et al.* [15] and Homem *et al.* [5]. Yan *et al.* [15] point out that the decrease in permeate flux occurs due to the reduction of membrane pores or due to their complete covering with a GO nanosheets. In order to increase the selectivity of the membrane, and maintain the permeate flux at an adequate level, it is necessary to optimize the parameters of the membrane modification process [1]. In research conducted by Ding *et al.* [12] the water flow through the optimized PES/GO@TiO$_2$ membranes was more than 4 times higher than through the unmodified PES membrane.

GO–blended membranes

Conventional membranes can be modified by incorporating graphene oxide into the polymer matrix of the membrane, which improves the hydrophilicity, resistance to contamination, mechanical and thermal stability of the membrane. The most commonly used method for membrane matrix modification is the phase inversion method. Within this method, there are two modification techniques: on–solvent induced phase separation (NIPS) and thermally induced phase separation (TIPS). The NIPS technique is suitable for the modification of membranes that will be used in wastewater treatment. By applying this technique, membranes with asymmetric morphology, a restricted pore size range and reduced mechanical strength are obtained. The TIPS technique produces membranes with a highly porous, symmetrical structure. By adjusting the process parameter, the morphology of the membrane can be changed depending on the desired mechanical properties, pore size and desired permeate flux [1].

GO–blended membranes are widely used in the treatment of wastewater containing synthetic dyes [1]. In summary, removal efficiency of dyes using GO-blended membranes is listed in in Table 2. Based on the data shown in Table 2, it can be concluded that a high degree of removal synthetic dyes can be achieved using GO–blended membranes.

***Table 2** Efficiency of removal synthetic dyes from wastewater using GO–blended membranes*

Membrane	Dye	Efficiency (%)	References
PES–0.5 wt% CeO2/GO	Reactive red 195	88.0	[16]
	Reactive red 43	93.0	
	Yellow 105	98.0	
CS/PAAm–DADMAc/GO	Methylene blue	99.0	[17]
	Congo red		
PVDF – 0.5 wt% GO NP	Rhodamine B	67.8	[18]
Fe/GO–TA20	Mixture of dyes	99.0	[19]
0.1 wt.% rGO/TiO_2/PES	Reactive Blue 21	81.4	[20]
0.1 wt.% GO/PES		69.7	
0.1 wt.% TiO_2/ PES		73.5	
GO/PAN/PEG	Acid Red 18	99.8	[21]
	Methyl Blue	100.0	
MMGO/PES/PVP	Direct Red 16	99.0	[22]

Modifying the membranes with graphene oxide, a higher efficiency of removing synthetic dyes from aqueous solutions can be achieved. Kadhim *et al.* [23] investigated the effect of modification PES membrane with GO nanoparticles on the efficiency of acid black removal and the performance of the newly synthesized membrane. The results of the investigation indicate that after the modification of the PES membrane with graphene oxide, the efficiency of acid black removal increased from 88% to 99%. By incorporating 0.5 wt% GO nanoparticles, the porosity of the membrane increases, while the contact angle is reduced from

60.82° to 39.21°, which indicates that the hydrophilicity of the membrane surface is improved. Zhu *et al.* [18] confirmed the positive impact of incorporating 0.5 wt% GO on membrane performance. Kadhim *et al.* [23] point out that with an increase in the concentration of GO from 0.5 to 1.5 and 2 wt%, the contact angle increases, without a significant decrease in the hydrophilicity of the membrane, which indicates that there has been an aggregation of GO nanoparticles, a decrease in the effective surface area of the particles, and thus a decrease in the number of GO functional groups on membrane surface.

Safarpour *et al.* [16] modified the PES membrane with a CeO_2/GO nanocomposite. The test results indicate that the modified membrane is characterized by greater hydrophilicity and porosity. In addition, with the introduction of CeO_2/GO nanocomposite, the resistance of the membrane to contamination increased from 39 to 69%.

Januario *et al.* [1] point out that the main disadvantage of membrane modification with GO is the reduction of permeate flux, due to the reduction of the pore size. However, the results of the study conducted by Safarpour *et al.* [16] indicate that the incorporation of CeO_2/GO nanocomposite into the polymer matrix of the PES membrane increases the permeate flux. With an increase in the mass fraction of nanocomposite from 0.01 to 0.2 wt%, the permeate flux increases. The permeate flux through the modified membrane containing 0.2 wt% CeO_2/GO nanocomposite was 64% higher than the pure water permeation through the unmodified PES membrane. The introduction of CeO_2/GO nanocomposite increases the number of hydrophilic groups on the surface of the membrane and the porosity, which resulted in an increase in permeate flow through the membrane. Similar results and observations were reached by Kadhim *et al.* [23] and Zhu *et al.* [18].

CONCLUSION

Based on previous researches, it can be concluded that the hydrophilicity, selectivity and stability of the membrane can be improved by modifying the membranes with graphene oxide. The modification of membranes with GO can be carried out in two ways: applying GO nanosheets on the surface of the membrane or incorporating GO nanosheets into the polymer matrix of the membrane. The modification of the membrane surface is based on the adsorption of nanosheets of GO and other compounds on the membrane surface, whereby there is a reduction or complete covering of the membrane pores, a change in the charge of the membrane, an improvement in the hydrophilicity, selectivity and stability of the membranes. Using surface–modified membranes can achieve high efficiency in removing synthetic dyes from aqueous solutions. However, due to covering the pores of GO membranes, the flow of pure water through the membrane is reduced, which is the main drawback of this method of membrane modification. Conventional membranes can be modified by incorporating GO nanosheets into the polymer matrix of the membrane. Applying this method of modification increases the porosity, hydrophilicity and selectivity of the membrane, as well as the permeate flow.

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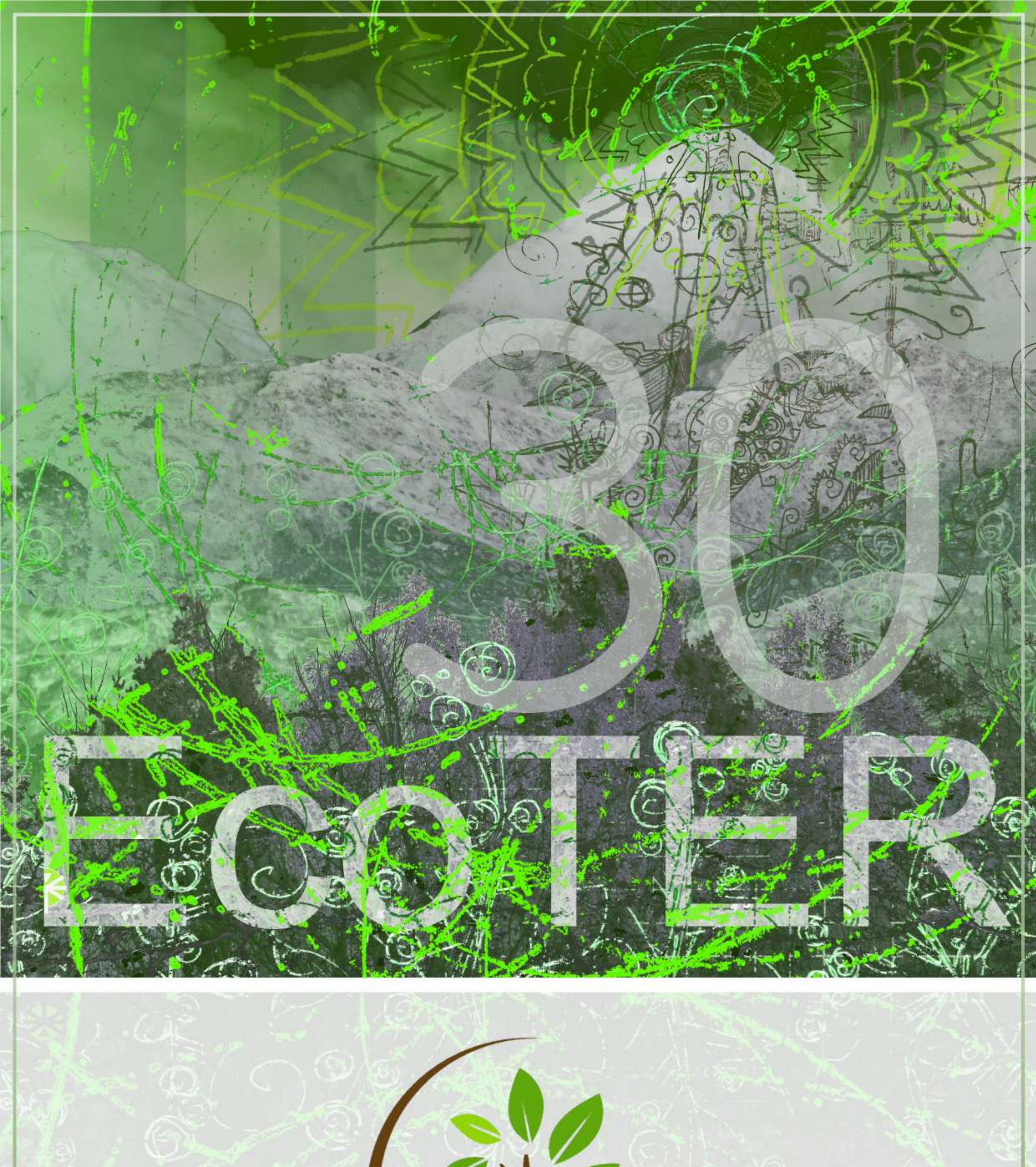

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