SYNTHESIS OF BISMUTH-OXYHALIDE COMPOSITE PHOTOCATALYSTS, AND THEIR APPLICATION USING UV AND VISIBLE LED LIGHT SOURCES

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Abstract

BiOI, BiOCl, and their composites with different molar ratios have been synthesized and tested for photocatalytic application, using methyl orange as model pollutant, and three various LED light sources. Adsorption capacity of BiOI highly exceeds that of BiOCl. The composite having 80:20 molar ratio of BiOI:BiOCl showed similar adsorption capacity than BiOI, and the best photocatalytic activity: about three times higher than BiOI and nine times higher than BiOCl. Methyl orange effectively transformed under visible light radiation, but with lower rate than under UV radiation. HPLC-MS measurements proved that the transformation of methyl orange is initiated with a demethylation. Effect of methanol as HO• scavenger, and 1-4 benzoquinone proved that, the transformation of dye is initiated via direct charge transfer, and/or via photosensitization. Hydroxyl radical has no contribution to the transformation. The change of photocatalytic efficiency was followed during three cycles. After the first one, the transformation rate of methyl orange decreased, but there was no significant difference between the rates determined in the case of the second and third cycles. Ecotoxicity measurements confirmed that no toxic substances were dissolved from the catalyst under radiation, but the toxicity of methyl orange solution significantly increased during the treatment.

Keywords: Bismuth-oxyhalides, Photocatalysis, Visible light, LED

1 INTRODUCTION

One of the current challenges in water treatment is the removal of non-biodegradable and hazardous contaminants, having biological activity. These pollutants have a negative impact on the ecosystem and even endanger public health. Conventional biological-physical water treatment methods are sufficient and cost-effective processes to remove most of the biodegradable organic matter. Unfortunately, these conventional methods are often unsuitable for removing toxic, non-biodegradable pollutants, having low-concentration. Consequently, additional methods need to be developed that can be applied after biological treatment for this purpose. Advanced Oxidation Processes (AOPs), as additive water treatment methods, offer a solution to this problem. One of the widely investigated processes is heterogeneous photocatalysis, which is based on the application of semiconductors as photocatalysts, to initiate redox reactions. Due to the absorption of a photon having appropriate energy, an electron (e_{cb}) in the excited conduction band and a hole (h_{vb}^+) in the valence band forms in the semiconductor. These photogenerated charges can initiate the transformation of organic substances on the surface of photocatalyst. The transformation of organic pollutants can be initiated in different ways: direct charge transfer [1], photosensitization [2] or reaction with hydroxyl radicals (HO•) [1-3]. Their relative contribution depends on the properties of the photocatalyst, the properties of the substrate and the reaction parameters.

The most often used photocatalysts, TiO_2 and ZnO have wide band gaps (E > 3.0 eV), therefore they are only active in the UV region [3]. One of the main goals of material science is the synthesis of suitable catalysts, which efficiently works under visible light radiation, as sunlight is our cheapest and inexhaustible energy source. With the recent advances in materials sciences, several new, photoactive semiconductors are being synthesized and tested. Bismuth oxyhalides (BiOX: X=F, Cl, Br, I), as photoactive materials have received widespread attention. BiOF and BiOCl are active in the UV region (band gap: 3.6 eV for BiOF and 3.2 eV for BiOCl), while BiOBr, and especially BiOI are active in the visible region (band gap: 2.6 eV for BiOBr and 1.8 eV for BiOI) [4]. Their advantages include good adsorption capacity, they are easy to synthesize, but their chemical stability is relatively low, and the recombination of photoinduced charges is fast [5-7]. Several attempts have been made to improve the efficiency and stability of BiOX photocatalysts, one of which is the preparation of the composite catalysts. BiOCl/BiOBr, BiOCl/BiOI, SiO_2/BiOX and TiO_2/BiOX composites, with improved stability and enhanced visible light activity, were successfully synthesized [8-11]. Methods such as the size controlled synthesis of nanoparticles and the use of environmentally friendly, green synthesis methods are also investigated during the development of BiOX photocatalysts. [12-14].

Due to the intensive development of optoelectronics in recent years, the use of light emitting diodes (LEDs) radiating in the UV and visible light region has become increasingly popular. This makes it possible to improve the efficiency of additive water treatment methods based on photochemical processes. Compared to the conventional UV and visible light sources, LEDs have higher electric efficiency, lower price, better mechanical tolerance, and longer lifetimes, therefore they are a promising option to replace mercury-vapour and xenon lamps.

The aim of this study was to synthesize BiOCl/BiOI composite photocatalysts, and to test their effectiveness in the UV and visible region using different LED light sources. Methyl orange was used as a model compound. The adsorption capacity, stability and reusability of the photocatalysts, the transformation mechanism of methyl orange were investigated. Toxicity measurements were also performed to investigate the ecotoxicity of both the photocatalyst and the formed intermediates of methyl orange.

2. MATERIALS AND ANALYTICAL METHODS

2.1 Preparation of BiOI/BiOCl composite catalysts

The BiOI and BiOCl photocatalysts were prepared as described by Bárdos et al. [12]. $Bi(NO_3)_3 \times 5\ H_2O$ (Alfa Aesar, 98 %), KCl and KI (Molar Chemicals, 99.7 %) were used for preparation via solvothermal method. The $Bi(NO_3)_3 \times 5\ H_2O$ and KCl or KI were dissolved in 50 cm^3 ethylene glycol (Sigma-Aldrich, 99.95%) with continuous stirring and heating (up to 45°C). The crystallization was performed at 120 °C in a PTFE-coated steel autoclave for 3 hours. The solid material was washed with distilled water and ethanol (VWR, 96%), then vacuum-filtered with a 0.1 μm pore size filter (Durapore®, hydrophilic PVDF) and dried for 24 hours at 40 °C. The composite catalysts were prepared with the same method, with appropriate changing of the molar ratio of KCl and KI. The molar ratios have been calculated to result in composites with 5.0 to 95.0 n/n% BiOI content. The color of the prepared crystals changed from white to red with increasing BiOI content.

2.2 Photocatalytic experiments

During the photocatalytic experiments, 100 cm^3 suspension was irradiated in a cylindrical glass reactor (inner diameter: 45 mm). Due to the significant adsorption of methyl orange on the surface of catalysts, the suspensions were stirred in dark for 30 min before photocatalytic tests. The experiments were started with turning on the light source.

Three different LED light sources have been used, 'UV' (LEDmaster, $\lambda_{emission}$=397±10 nm, 288 lumen, 4.6 W), 'cool white' (LEDmaster, $\lambda_{emission}$=400-650 nm, 390 lumen, 4.6 W), and 'warm white' (LEDmaster, $\lambda_{emission}$=400-700 nm, 600 lumen, 4.6 W). Fig. 1. shows the emission spectra of the light sources. The LED tape (60 LED/m) was fixed on the inside of a 66 mm inner diameter, aluminum, double walled reactor. The reactor was equipped with water cooling system, to ensure the longer lifetime and constant light output of the LEDs.

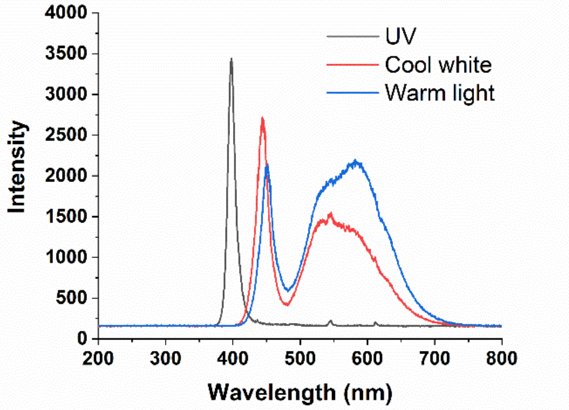

Fig. 1. Emission spectra of the LED light sources

2.3 Analytical methods

Methyl orange (and probably its main products) is adsorbed well on the photocatalysts surface. After sampling, 0.5 cm^3 NaF solution (0.5 M) was added to 1.0 cm^3 sample for desorption of methyl orange and its products from the surface. After keeping the samples for 10 minutes in the dark, they were centrifuged (Dragonlab, 15000 RPM) and filtered with syringe filters (0.22 μm, FilterBiO, PVDF-L).

The recovery of methyl orange was checked with 2.0×10^{-4} M methyl orange in 0.50 g dm^{-3} BiOCl and BiOI containing suspensions, and found to be 98 %. An Agilent 8453 UV-Vis spectrophotometer was used for spectrophotometry measurements. The absorbance of the samples was measured at 464 nm (the maxima of the methyl orange spectrum) in a 2.0 mm quartz cuvette. The molar absorbance of methyl orange at this wavelength is $\varepsilon=25905$ mol^{-1} dm^3 cm^{-1}. For separation of the intermediates and determination of methyl orange concentration in treated suspension HPLC measurements were made. Agilent 1100 HPLC equipped with a diode array UV detector (DAD) was used. As a stationary phase, a Kinetex 2.6u XB-C18 100A (Phenomenex) reverse phase column was used, while the mobile phase consisted of 40 v/v% acetonitrile (VWR, UPLC-grade) and 60 v/v% formic acid solution (0.1%). The flow rate of eluent was 0.70 ml min^{-1} and the temperature was 25 °C. The product formed from methyl orange was determined via mass spectrometry (Agilent LC/MSD/VL). Measurement was performed in positive mode (4000 V capillary voltage, 65 V fragmentor voltage), the scan range was 100-1000 AMU.

Ecotoxicity tests (LCK480, Hach-Lange) based on the bioluminescence measurements of *Vibrio fischeri* bacteria were used to determine the acute toxicity of the samples. The inhibition of bioluminescence was measured using a Lumistox 300 (Hach Lange) luminometer after 30 min incubation time.

3. RESULTS AND DISCUSSION

Adsorption generally has an important role during heterogeneous photocatalysis, especially in the case of dyes. The adsorption capacity of photocatalysts was determined in suspensions containing 0.5 g dm^{-3} catalyst and 2.0×10^{-4} M methyl orange. The suspensions were stirred in dark for 2 hours. Adsorption equilibrium has been reached after 30 min.

Adsorption capacity of BiOI (29 % of the initial concentration of methyl orange; 1.16×10^{-3} mol/g) is highly exceed that of BiOCl (11 % of initial concentration methyl orange; 4.3×10^{-4} mol/g). The amount of adsorbed methyl orange increased with increasing the BiOI content, up to 80:20 molar ration of BiOI:BiOCl, when 37 % of methyl orang was adsorbed. After that, it slightly decreased with the further increase of the BiOI content (Fig. 2).

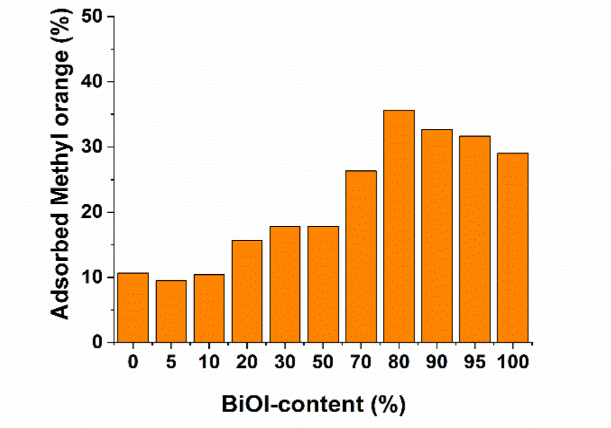

Fig. 2. The relative adsorbed amount of methyl orange ($c_0 = 2.0\times10^{-4}$ M; 0.5 g dm^{-3} photocatalyst)

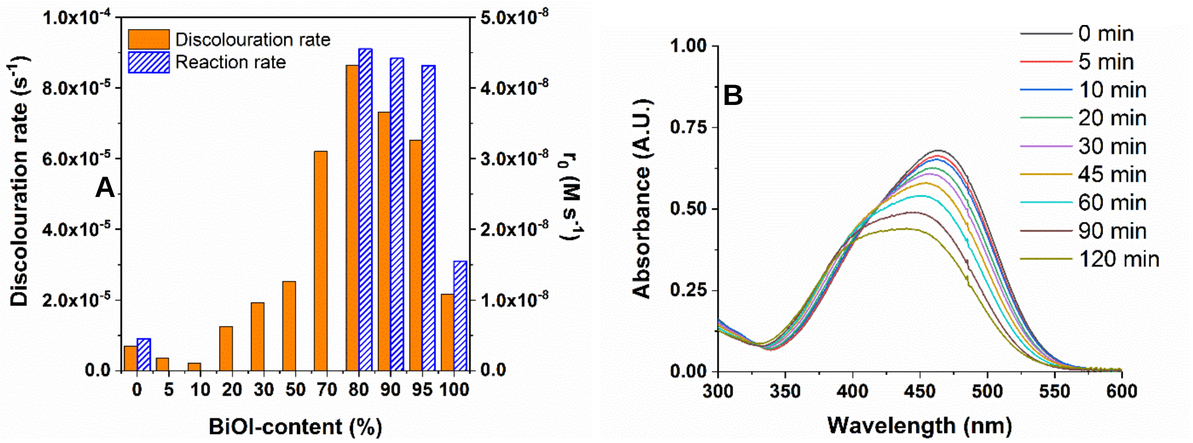

Fig. 3. The rate of discolouration and initial transformation rate of methyl orange (A), and UV-Vis spectra of the treated solutions in the case of the BiOI/BiOCl composit having 80% BiOI content (B)

The photocatalytic activity of pure BiOCl and BiOI, and of the composite catalysts were tested using the UV LED (397±10 nm) light source. Relative contribution of direct photolysis was negligible; there was no transformation without photocatalyst.

The discolouration of methyl orange solution was very slow in the case of BiOCl, while in the case of pure BiOI it was more effective. Significantly increased activity was determined in the case of the composite catalysts, which contain more than 50%

BiOCl. Activity reached the maximum value in the range of 80-95% BiOCl content (Fig. 3/A), having good correlation to the adsorption capacity (Fig. 2). During the spectrophotometric measurements, the characteristic change of the shape of methyl orange spectrum and the shift of the maximum to the lower wavelengths can be observed, which indicates the formation of products having significant absorption around 400-450 nm (Fig. 3/B).

In the case of the most promising composite catalysts (80, 90 and 95% BiOI content) and in the case of the pure BiOI and BiOCl, the initial transformation rate of methyl orange (r_0) and the formation rate of the main product were determined by using HPLC-DAD method. The identification of the main product (P1) having absorption maximum at 420(±5) nm was performed via mass spectrometry. This product forms due to the demethylation of methyl orange (Fig. 4.) and accumulates during the first period of the treatment. Its transformation became significant after the decomposition more than 50% of the methyl orange. The formation of this product was reported to form due to the transformation of methyl orange via direct charge transfer and/or sensitization [13].

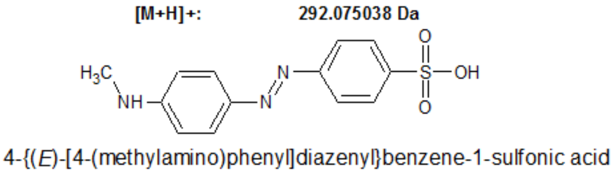

Fig. 4. The proposed chemical structure of P1 product

The activity of the BiOI/BiOCl composite catalyst, having 80% BiOI content, and of the pure BiOCl and BiOI were determined under visible light radiation, using cool white and warm white light instead of UV light. The direct photolysis of methyl orange was negligible in the case of each light source. The transformation of methyl orange and the formation of P1 product were followed during 120 min treatment time (Fig. 5.).

BiOCl has relative wide band gap (3.2 eV), and consequently its excitation requires radiation having shorter wavelength than 385 nm. The slow transformation of methyl orange ($r_0 = 4.53\times10^{-9}$ M s^{-1}), and the formation of the P1 product can be attributed to photosensitization (Fig. 5/B), as the cool white and warm white light do not have sufficient energy for the direct excitation of BiOCl. BiOI having 1.8 eV bandgap can theoretically be excited by radiation with light having shorter wavelength than 688 nm. In the case of BiOI, the initial transformation rates were significantly higher than in the case of BiOCl. An induction period can be observed at the beginning of the kinetic curves (up to 30 min.). During this period the transformation rate increased with the increased average energy of the photons (UV < Cool white < Warm white), but there was no significant difference between the overall efficiency after 120 min of treatment (Fig. 5/C).

In the case of the BiOI/BiOCl composite catalyst, induction period was not observed. There was no difference between the initial transformation rates determined in the case of cool white ($r_0 = 2.32\times10^{-8}$ M s^{-1}) and warm white light ($r_0 = 2.35\times10^{-8}$ M s^{-1}), while in the UV irradiated suspension that was found to be significantly higher ($r_0 = 4.55\times10^{-8}$ M s^{-1}). The formation of P1 product followed similar tendencies (Fig. 5/D); its accumulation and probably its transformation was faster in UV irradiated suspension.

The effect of suspension concentration on the transformation rate was investigated using the most efficient UV-LED light source and BiOI/BiOCl composite photocatalyst. In the case of heterogeneous photocatalysis, the rate of transformation changes with increasing concentration of the photocatalyst according to a saturation curve. In this case, the transformation rate increased with the photocatalyst concentration linearly, within the range of 0 - 1.5 g dm^{-3} photocatalyst (Fig. 6/A), despite the fact that the emitted photons are completely absorbed at a suspension concentration of 1.0 g dm^{-3}.

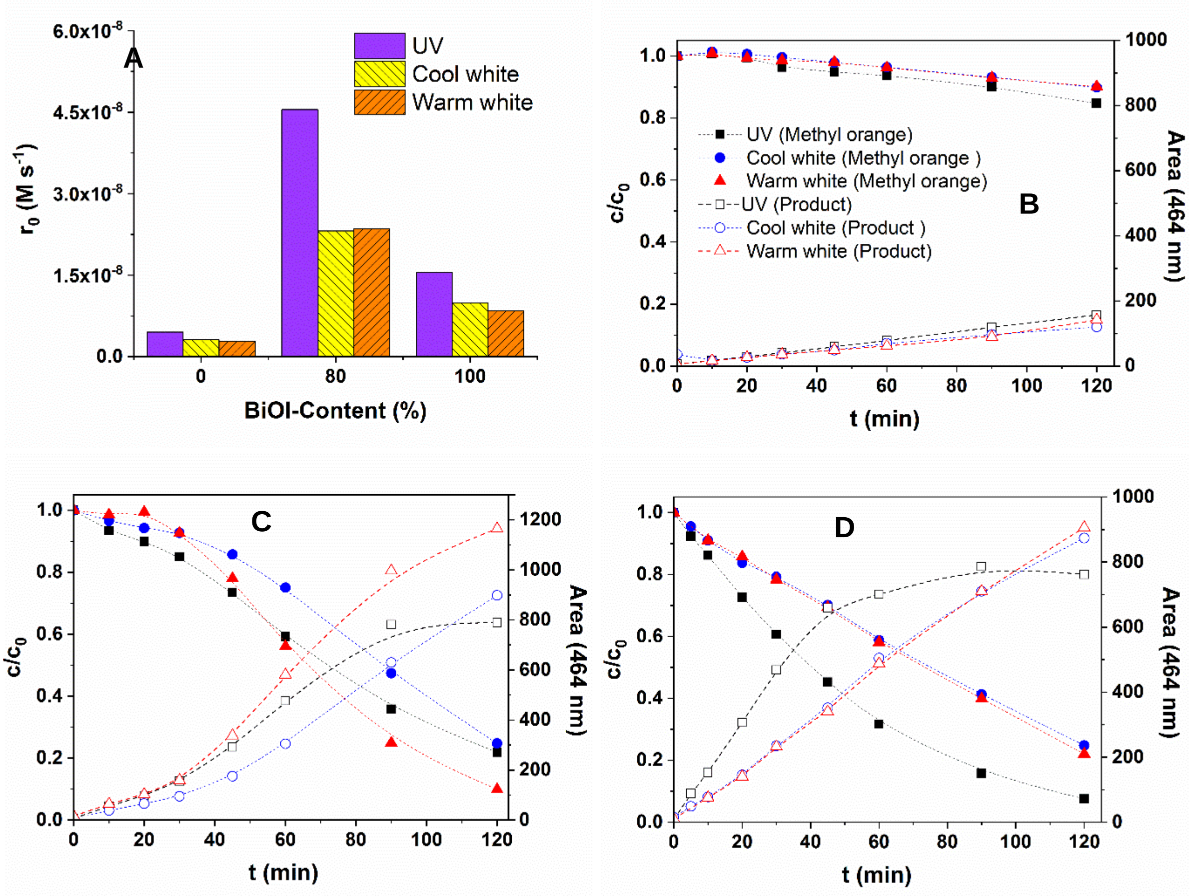

Fig. 5. The initial transformation rate (r_0) (A), the kinetic curves of methyl orange degradation, and the formation of P1 product in the presence of BiOCl (B), BiOI (C) and BiOI/BiOCl composite having 80% BiOI content (D) using various light sources

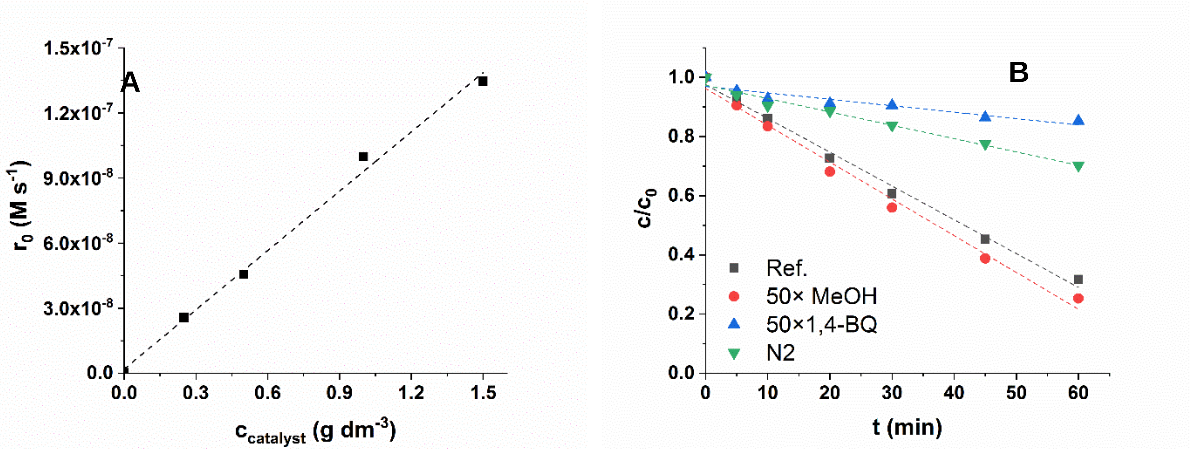

Fig. 6. The initial transformation rate (r_0) determined at various catalyst dosage (A), and the effect of methanol, 1,4-benzoquinone and dissolved O_2 on the transformation of methyl orange, using BiOI/BiOCl (molar ratio: 80:20) composite photocatalyst (B)

For BiOX photocatalysts, the mechanism of transformation is not yet clear; the HO•-based reaction [17], direct charge transfer [15,17] and/or reaction with O_2• [18], as well as photosensitization [4] are also supposed. Methanol (1.0×10^{-2} M), as HO• scavenger has no effect on the transformation rate, while addition of 1,4-Benzoquinone (1,4-BQ) (1.0×10^{-2} M) reduced that by 84 %. This suggest the dominant role of the reaction between methyl orange and $O_2^{\bullet -}$ and/or the reaction between methyl orange and photogenerated e_{cb}^- in the transformation. As dissolved O_2 also acts as a scavenger of e_{cb}^-, an experiment has been performed in deoxygenated suspensions (O_2 was eliminated by bubbling with N_2). The transformation rate was reduced by 70 %, which confirms the dominant role of the charge transfer reactions (Fig. 6/B).

The study of the stability and reusability of the catalyst has crucial role for practical application. Degradation of methyl orange was monitored for three consecutive cycles (Fig. 7/A). The initial transformation rate of methyl orange reduced by 77 % after the first cycle, but there was no difference between the initial transformation rate determined in the second and third cycle. The decreased activity can be caused by the accumulation of the products on the surface of the photocatalyst and the competition for active sites of the surface.

As the main goal of using BiOX catalysts for elimination of harmful organic substances, the change of toxicity during treatment was investigated. The toxicity of the composite photocatalyst, and the treated methyl orange solution was determined using *Vibrio Fisheri* test organism. The toxicity of methyl orange solution was negligible, but significantly increased during the treatment, most probably due to the formation of highly toxic intermediates. During radiation there the damage of photocatalysts can takes place, which can result in the formation of inorganic ions in the suspension. The toxicity of BiOI/BiOCl photocatalyst was also determined during 90 min radiation without methyl orange. After the removal of the catalyst particles, there was no significant effect of the filtered solution on the test organism. The inhibition effect was less than 10 %, and proved that no harmful substances are dissolved from the catalyst particles (Fig. 6/B).

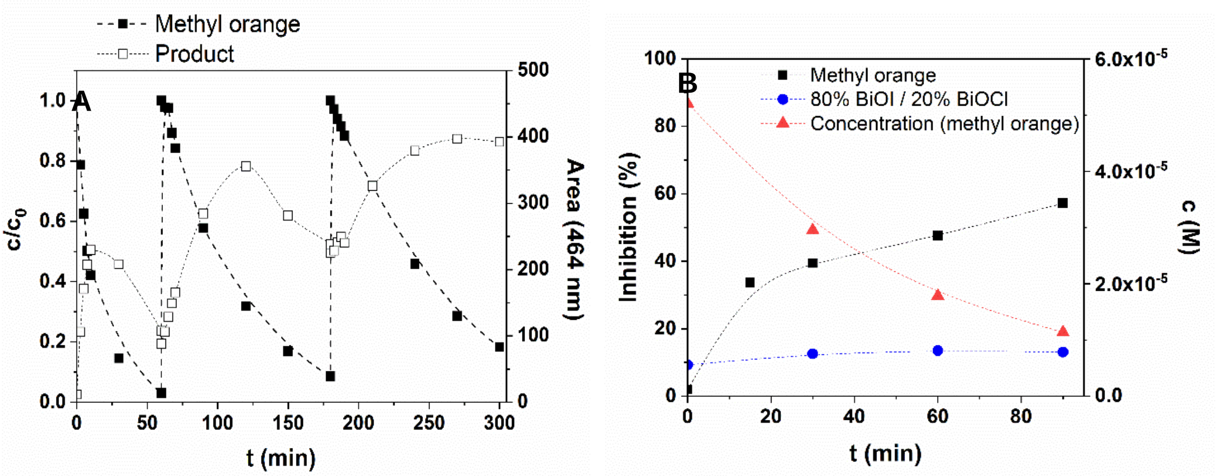

Fig. 7. Transformation of methyl orange and the formation of P1 product as a function of time during three cycles (A), and the ecotoxicity of methyl orange and the composite catalyst during UV-LED irradiation (B)

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Prof. Dr. Nesrin Kayataş Demir /Turkey
Dr. Muhammet Çelik / Turkey
Dr. Hacımurat Demir/Turkey
Dr. Gülşah Elden/Turkey
Dr. Sertaç Samet Seyitoğlu/Turkey
Dr. S. Buğra Selçuklu/Turkey
Dr. D. Andrew S. Rees/UK
Dr. Sibel Güneş/Turkey

PREFACE

This is the first year of International Conference on Energy, Environment and Storage of Energy, *ICEESEN 2020*. Symposium was held as virtually and brought together experts and scientists from many different fields.

103 presentations were presented in 18 virtual sessions at ICEESEN 2020 and four keynote speakers made presentation. The symposium was successfully held with the online participation of over 250 participants from thirty-two different countries.

I hope the studies and improvements that have been done and will be made in the future will contribute to leave a more livable world for future generations. As the scientific community, we have more jobs than expected and it is our duty to move forward with awareness of this.

I would like to thank the symposium honor, organization, management and scientific committee members, reviewers, invited speakers, session chairs, participants and the symposium secretariat who contributed to the successful implementation of the symposium.

Best Regards,

Prof. Dr. Selahaddin Orhan AKANSU

Conference Chair

INTERNATIONAL CONFERENCE ON ENERGY, ENVIRONMENT AND STORAGE OF ENERGY,

(*ICEESEN 2020)*

19-21 November 2020

Keynote Speaker: Prof. Dr. Marc A. Rosen

Speech Title: Nucleer Energy: Non-electric Application to Help Reduce Environmental Impacts

CV: Marc A. Rosen, Ph.D., is a Professor at University of Ontario Institute of Technology in Oshawa, Canada, where he served as founding Dean of the Faculty of Engineering and Applied Science. Dr. Rosen has served as President of the Engineering Institute of Canada and of the Canadian Society for Mechanical Engineering. He has acted in many professional capacities, such as Editor-in-Chief of various journals, and a Director of Oshawa Power and Utilities Corporation. With over 70 research grants and contracts and 900 technical publications, Dr. Rosen is an active teacher and researcher in sustainable energy, sustainability, and environmental impact. Much of his research has been carried out for industry. Dr. Rosen has worked for such organizations as Imatra Power Company in Finland, Argonne National Laboratory near Chicago, the Institute for Hydrogen Systems near Toronto, and Ryerson University in Toronto, where he served as Chair the Department of Mechanical, Aerospace and Industrial Engineering. Dr. Rosen has received numerous awards and honours, and is a Fellow of numerous societies.