CHEMICAL SYNTHESIS AND CHARACTERIZATION OF LEAD DIOXIDE NANOPOWDER

Branislava Savić[1], Sanja Živković[1], Dalibor Stanković[1], Miloš Ognjanović[1], Tanja Brdarić[1], Gvozden Tasić[1], Ivana Mihajlović[2]

[1]*Vinča Institute of Nuclear Sciences, University of Belgrade, Mike Petrovića Alasa 12–14, 11000 Belgrade, Serbia*
[2]*University of Novi Sad, Faculty of Technical Sciences, Department of Environmental Engineering, Trg Dositeja Obradovića 6, 21000 Novi Sad, Serbia*
e-mail: savicbranislava87@gmail.com

Abstract
Lead dioxide (PbO_2) is one of the very interesting materials that have a lot of applications. In this work, nano-sized PbO_2 was synthesized by the chemical method. The composition, morphology and crystal structure of the nanopowder were characterized using Fourier Transform Infrared Spectrometry (FT-IR) and field emission scanning electron microscopy with energy dispersive X-ray spectrometer (FESEM-EDX). The obtained data indicate that a nanopowder PbO_2 with an average crystalline size of about 20 to 40 nm was formed. The synthesized nanomaterial PbO_2 could have a wide range of applications in wastewater treatment, e.g. to remove organic pollutants such as phenols.

Introduction
Nano-sized materials are of great scientific and technological importance in various fields of chemistry and environment protection [1]. A number of scientists investigations including physical, chemical and biological methods have been developed for nanoparticle preparation. It is well known that properties of nanoparticles depend on morphology, the size of the particles, the composition of the phases i.e. of the synthesis process.
The lead dioxide (PbO_2) is often cited as a promising material for chemistry and environmental protection applications, including its use for the oxidation of organic compounds in wastewater [2, 3], as a stationary phase for solid-phase microextraction [4]. Recently, it was reported that different morphologies of the PbO_2 nanoparticles (nano-rods, hollow spheres, trigonalnano-plates, nano-wires, macroporous, and hexagonal flower array) were synthesized by ultrasonic irradiation [5], hydrothermal method and electrochemical methods [6, 7].
The aim of the presented study is the synthesis and characterization of lead dioxide nano-powders. In the presented work, lead dioxide nano-powders were prepared using chemical method of preparation. Herein, the morphology and structure of PbO_2 were investigated using FT-IR and FESEM-EDX. The novel synthesized nanomaterial could be used in different applications for environmental protection.

Experimental
All chemical reagents were of analytical grade or better. Potassium persulfate ($K_2S_2O_8$), lead (II) acetate ($Pb(CH_3COO)_2$) and NaOH were all obtained from Sigma-Aldrich and used as received.
Functional groups were analyzed by Thermo scientific (Nicolet iS5, iD7) FT-IR spectrometer. The FT-IR spectra were recorded in the range from 400 cm^{-1} to 4000 cm^{-1}.
The morphology of the PbO_2 was determined by FE-SEM (FEI Scios 2). The micrographs were recorded with 25000 x magnification.

The synthesis of nanosized PbO_2 using PbO nanomaterial synthesis of nanosized lead oxides was conducted according to the procedure proposed by *M. Alagar et al.* [7], and *W. Li et al.* [9]. PbO nanomaterial, used as precursor for the synthesis of nanosized PbO_2, was prepared by mixing lead (II) acetate solution with NaOH solution and resulting mixture was heated up to 90 °C. The obtained precipitate was filtered and dried at 90 °C. Then, this material was mixed with deionized water and treated with potassium persulfate in hydrothermal reactor at 120 °C for 2 h. The obtained material was washed several times with deionized water and dried at 70 °C.
Obtained PbO_2 material was characterazed by FT-IR and FESEM-EDX methods.

Results and discussion

FT-IR studies:

The FT-IR spectra of PbO_2 nanoparticles (Figure 1.) showed a broad band at 445.58 cm^{-1} due to PbO_2 vibration mode. A peak at 511.34 cm^{-1} indicates the presence of oxides. This shows that the synthesised nanopowder consists of lead and oxide.

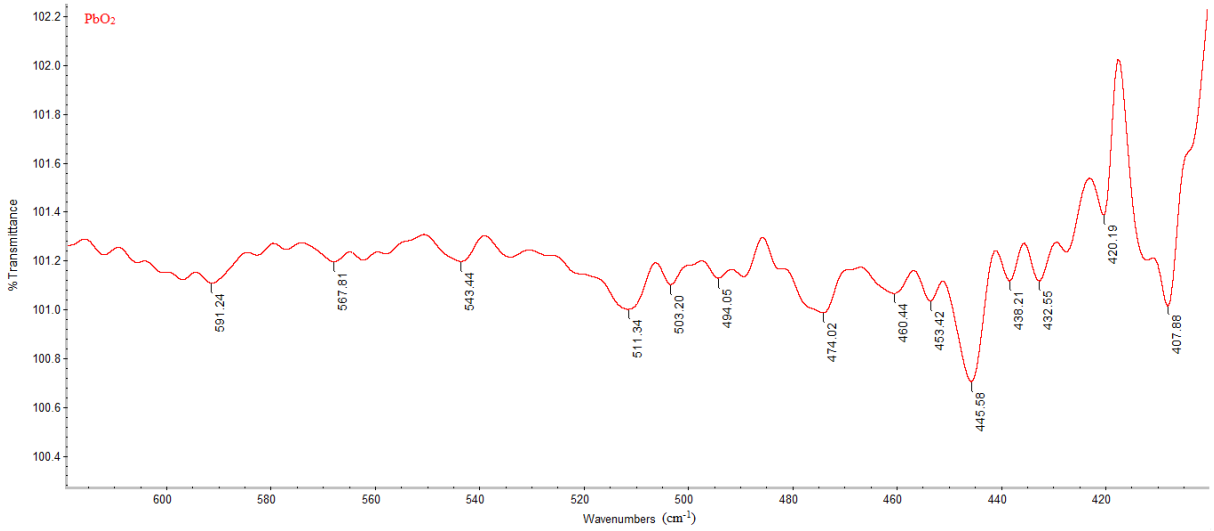

Figure 1. FT-IR spectrum of PbO_2 nanoparticles

SEM studies:

SEM morphological and nanostructural studies of the PbO_2 nanoparticles are shown in Figure 2. The size of nanoparticles is from 20 nm to 40 nm.

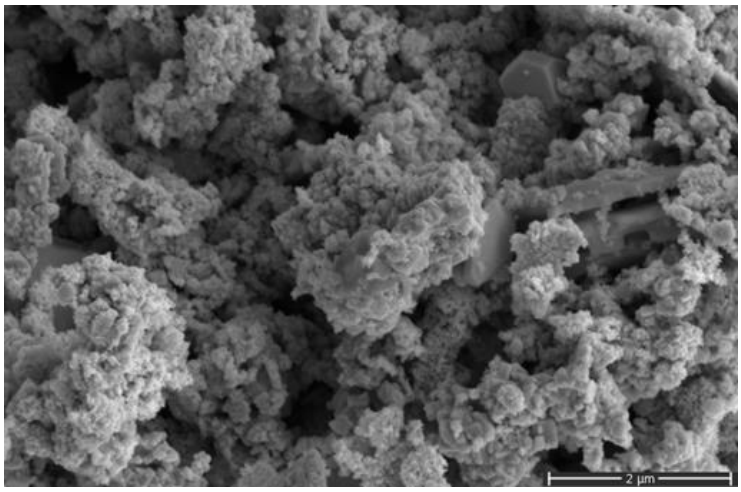

Figure 2. SEM picture showing PbO_2 nanoparticles

Conclusion

In summary, PbO_2 nanoparticles were synthesized by chemical method. The nanopowder contains a crystallites from 20 to 40 nm. The synthesized nanopowder could be appropriate for electrochemical degradation of organic pollutants and can be used as electrode material. The future investigation should be focused on the detailed physical characterization of synthesized material, as well as the application of nano-sized PbO_2 in wastewater treatment of organic pollutants.

Acknowledgements

This study was financially supported by the "Serbian Ministry of Education, Science and Technological Development" (project no. TR 37021).

References

[1] R.F. do Nascimento, O.P. Ferreira, A. J. De Paula, V. de Oliveira Sousa Neto (Eds.), Nanomaterials Applications for Environmental Matrices, Water, Soil and Air, Elsevier, Netherlands, 2019, pp. 528.
[2] S.P. Tong, C.A. Ma, H. Feng, A novel PbO2 electrode preparation and its application in organic degradation, Electrochem. Acta 53 (2008) 3002–3006.
[3] X. Duan, F. Ma, Z. Yuan, L. Chang, X. Jin, Electrochemical degradation of phenol in aqueous solution using PbO2 anode, J. Taiwan Inst. Chem. Eng. 44 (2013) 95–102
[4] A. Mehdinia, M.F. Mousavi, M.J. Shamsipur, Chromatogr A. 2006 Nov 17;1134(1-2):24-31. Epub 2006 Sep 25.
[5] S. Ghasemi, M.F. Mousavi, M. Shamsipur, H. Karami, Sonochemical-assisted synthesis of nano-structured lead dioxide. Ultrason. Sonochem. 15 (2008) 448-455.
[6] Fu, Z. A. Hu, L. J. Xie, X. Q. Jin, Y. L. Xie, Y. X. Wang, Z. Y. Zhang, Y. Y. Yang, H. Y. Wu, Int. J. Electrochem. Sci., 4 (2009) 1052.
[7] H. Xu, D. Shao, Q. Zhang, H. Yang, Y. Wei, Preparation and characterization of PbO_2 electrodes from electro-deposition solutions with different copper concentration, RSC Adv. 4 (2014) 25011–25017.
[8] M. Alagar, T. Theivasanthi, A, Kubera Raja, Chemical Synthesis of nano-sized particles of lead oxide and their characterization studies, J. of Applied Sci., 12 (4) (2012) 398-401.
[9] W. Li, Huayun Y., Q. Liu, Hydrothermal synthesis of PbO_2/RGO nanocomposite for electrocatalytic degradation of cationic red X-GRL, J. of Nanomaterials, (2017), Article ID 1798706.