

International scientific conference
Environmental impact of illegal construction, poor
planning and design IMPEDE 2019

CONFERENCE PROCEEDINGS

10 – 11 October 2019, Belgrade, Serbia

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Photocatalytic Degradation of Methylene Blue by Catalysts Prepared from Serbian Clinoptilolite and SnO₂

Fotokatalitička degradacija metilensko plavog u prisustvu katalizatora na bazi klinoptilolita sa područja Srbije i SnO₂

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In this work, clinoptilolite-rich zeolitic tuff from the deposit Slanci (near Blegrade) was used for the preparation of a photocatalyst. Clinoptilolite surface was modified by Sn(IV) oxide in different amounts (3-15 wt.% Sn) using a simple procedure consisted of three steps: a) ion exchange, b) precipitation of Sn hydroxide and c) calcination under air at 400 °C. Photocatalytic activity was studied in water solution of methylene blue (MB) as a model pollutant with initial concentration of 10 ppm at room temperature, using different amounts of the prepared catalyst. The suspension was irradiated for 180 min in a solar simulator chamber with a visible-light lamp (8 mW cm⁻²). The prepared catalysts exhibited catalytic activity higher than pure SnO₂ indicating a significant role of the clinoptilolite lattice in the photodegradation of MB. It is showed that the initial concentration of MB decreased for 45-75 % depending of the catalyst amount. Reusability of the catalyst was tested in three catalytic cycles showing that photocatalytic activity partially decreases which is ascribed to partial blockage of catalytically active sites.

Keywords: photocatalysis, zeolite, organic dyes

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1. Introduction

Different organic dyes are discharged in water body mainly from textile and leather industries. These organics are generally toxic and carcinogenic and their presence in water even in low concentrations causes serious environmental problems. Although still important, traditional water purification methods such as mechanical separation, filtration, flocculation, coagulation or chemical treatments need to be invented as well as novel water treatment technologies more efficient than the traditional ones should be developed.

Organic dyes possess complex structures and their degradation is usually complicate. Advanced Oxidation Processes (AOPs) have been recognized as effective methods for a complete degradation of organic dyes. Effectiveness of

AOPs has mainly been ascribed to the formation of highly reactive hydroxyl radicals formed by an activated catalyst.

Photocatalysis is one of the AOPs considered as an environmental friendly and efficient method. Metal oxides such as TiO_2 , ZnO , SnO_2 , or Fe_2O_3 have been reported to exhibit significant photocatalytic performance [Kim et al., 2015; Dariani et al., 2016]. It has been reported that the catalytic activity and reusability of these oxides can be significantly improved by immobilization of the oxide particles onto suitable supports [Bahrami and Nezamzadeh-Ejhi, 2015; Maučec et al., 2018].

Due to their unique structural properties as well as due to low cost and environmental compatibility, in this work we used for the preparation of a photocatalyst zeolite – clinoptilolite which is the most abundant natural zeolite in Serbia. Clinoptilolite 3D structure is characterized with an open-framework lattice which enables an easy access to the channel system with nanometric apertures.

Catalytic activity was tested in the photodegradation of methylene blue (MB) as a model cationic dye under visible light.

2. Experimental

2.1 Synthesis

Zeolitic tuff (Z) obtained from Slanci deposit (near Belgrade) was used in this study. Semiquantitative X-ray diffraction analysis performed by Rietveld refinement method (Topas-Academic v.4) showed that zeolite – clinoptilolite is the major mineral phase (about 80 wt.%) whereas quartz (~ 4 wt.%) and feldspars (~ 16 wt.%) are accompanying mineral phases.

The grain size used in all experiments was in the range of 0.063-0.1 mm for which previous experiments showed to be optimal ones for the modification process.

The modification consisted of two phases. In the first one, the tuff sample was treated with HCl (1 mol dm^{-3}) and then with NH_4OH (0.2 mol dm^{-3}). The treatment resulted in the preparation of NH_4 -containing clinoptilolite ($\text{NH}_4\text{-Z}$). The calcination of the $\text{NH}_4\text{-Z}$ at $600 \text{ }^\circ\text{C}$ resulted in conversion of $\text{NH}_4\text{-Z}$ to HZ.

In the second phase the HZ was loaded by SnO_2 as follows. HZ was suspended in the ethanol solution of SnCl_2 containing different amounts of SnCl_2 and pH was adjusted to 10 using NH_4OH . The dried products were calcined at $400 \text{ }^\circ\text{C}$ under air yielding $\text{SnO}_2\text{-Z}$ (with 3-15 wt.% Sn).

2.2 Characterization

The crystallinity of the samples was tested by a powder X-ray diffraction method (PXRD) using an APD2000 Ital Structure diffractometer (CuK α radiation, $\lambda=0.15418$ nm).

Elemental analyses were performed using a Carl Zeiss SupraTM 3VP field-emission gun scanning electron microscope (FEG-SEM) equipped with EDS detector (Oxford Analysis) with INCA Energy system for quantification of elements.

Porosity characteristics were determined by N $_2$ adsorption at -196 °C using a Micromeritics Instrument (ASAP 2020). The specific surface area (S_{BET}) was calculated according to the Brunauer, Emmett, Teller (BET) method up to relative pressures $p/p_0 = 0.15$.

The presence of SnO $_2$ onto samples was revealed from diffuse reflectance spectra (DRS) measured in the range 200-600 nm using V-650, JASCO UV-VIS spectrometer.

2.3 Photocatalytic tests

The photocatalytic experiments were performed in a batch 50 cm 3 reactor. The reaction mixture, contained a solution of MB (10 ppm) and catalyst (7.5-40 mg) was illuminated with a visible-light lamp (8 mW cm $^{-2}$) and mixed by bubbling O $_2$ at 25 cm 3 min $^{-1}$ from the bottom of the chamber during 180 min. The concentration of MB was measured colorimetrically at $\lambda = 664$ nm using Hach DR 2800 spectrophotometer.

The spent catalyst for which the photocatalytic experiments gave the best performance was chosen for recycling experiments. The catalyst was separated from suspension by filtration, left to dry at room temperature, washed 3 times with 0.01 M HNO $_3$ and dried at 90 °C for 1h prior to be reused.

3. Results and discussion

PXRD analysis showed that crystallinity of the zeolite - clinoptilolite remains intact after the conversion of Z to SnO $_2$ -Z (Fig. 1). The pattern of SnO $_2$ -Z does not exhibit any novel phase even at the highest amount of Sn which suggests that the formed SnO $_2$ (*vide infra*) is amorphous.

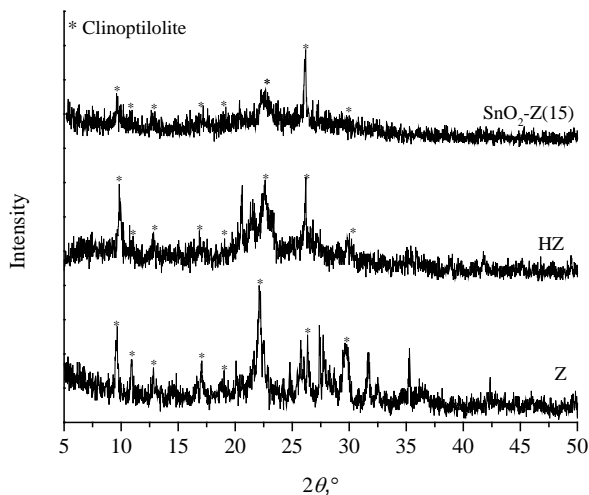


Figure 1. PXD patterns of Z, HZ and SnO₂-Z (with 15 wt.% Sn).

EDS analysis of the clinoptilolite phase of all the studied samples showed that conversion of the Z to HZ caused: a) a partial dealumination of the clinoptilolite lattice which results in increase of Si/Al molar ratio from 4.9 to 7.2, b) significant decrease of Na content (from 0.2 to 0.02 wt.%), c) removal of K, Ca and Mg present in the Z before its modification and d) the content of Sn varied from 3 to 15 wt.% in SnO₂-Z samples

The conversion of Z into HZ increased the specific surface area from 32 to 198 m² g⁻¹. The SnO₂ loading slightly influenced the specific surface area and decreased it to 170 m² g⁻¹. This could be ascribed to a partial pore blockage of the clinoptilolite lattice by formation of SnO₂ particles.

DRS revealed that the second phase of the modification process resulted in the formation of SnO₂. Fig. 2 clearly shows that the absorption maximum centered at 263 nm present in the spectrum of pure SnO₂ is also evident in the spectrum of SnO₂-Z. This maximum is not evident in the spectrum of HZ.

Photocatalytic tests showed that all SnO₂-Z samples are catalytically active. The activity increased with increasing of the Sn content (Fig. 3a) in SnO₂-Z as well as with increasing of applied catalyst amount. The highest degradation rate of MB (75 %) was achieved with 40 mg of the SnO₂-Z (Fig. 3b).

Fig. 3a shows an interesting phenomenon: all SnO₂-Z samples exhibited better catalytic performance than pure SnO₂. It indicates that the clinoptilolite lattice has a significant role in the photocatalytic degradation of MB.

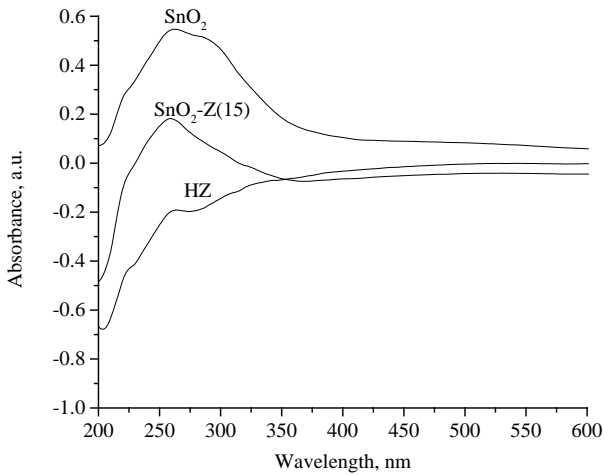
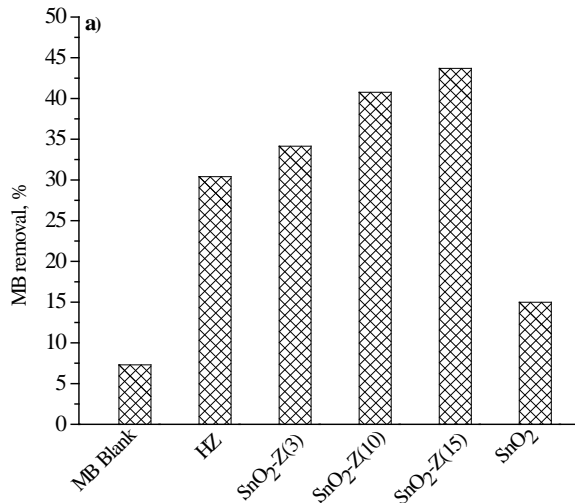


Figure 2. DR spectra of SnO₂, HZ and SnO₂-Z with 15 wt.% Sn.

The recycling experiment was performed using the highest amount of the SnO₂-Z (Fig. 4). During three cycles the photocatalytic activity of SnO₂-Z decreased to about 30 %. The effect could be ascribed to a partial blockage of the active sites on the catalyst surface by the degradation products of MB. This shows that future work should be directed towards preservation on active sites on the catalyst as well as to optimization of recovering process.



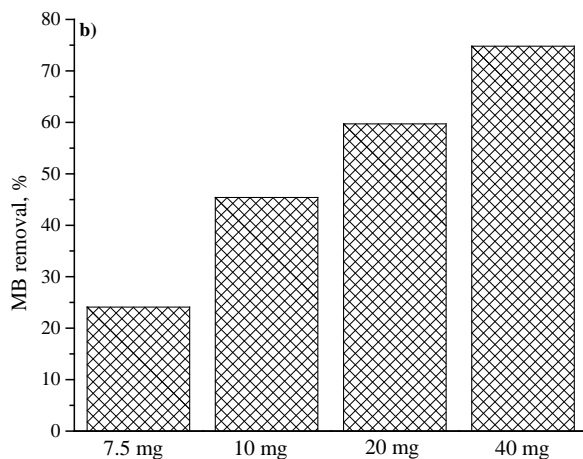


Figure 3. Results of photocatalitical degradation of MB: a) in the presence of 10 mg catalyst. Different amounts of Sn (in wt.%) are in parentheses; b) by using a different amount of SnO₂-Z(15).

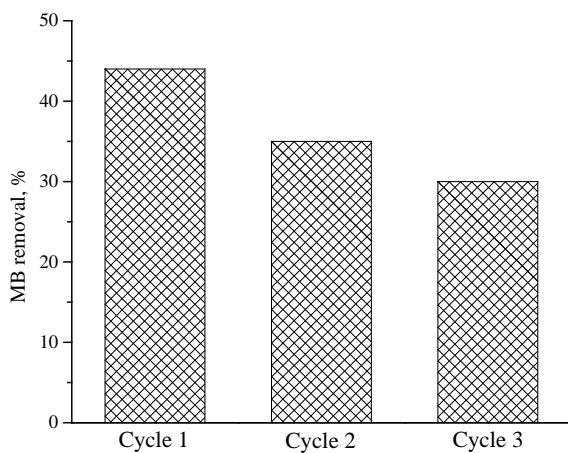


Figure 4. Reusability of SnO₂-Z(15) in photocatalitical degradation of MB.

4. Conclusion

Present results show that the clinoptilolite-rich zeolitic tuff from a Serbian deposit can be used in the preparation of catalysts active in the photodegradation of organic dyes under visible light. By a simple procedure sample of the tuff was converted in the catalyst with a high catalytic activity in the photodegradation of methylene blue. Since the photocatalytic activity partially decreased during reuse experiments future investigation will be directed towards the preservation of its activity.

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