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THE INFLUENCE OF THE UV IRRADIATION INTENSITY ON PHOTOCATALYTIC ACTIVITY OF ZnAl LAYERED DOUBLE HYDROXIDES AND DERIVED MIXED OXIDES

Layered double hydroxides (LDHs) have been studied to a great extent as environmental-friendly complex materials that can be used as photocatalysts or photocatalyst supports. ZnAl layered double hydroxides and their derived mixed oxides were chosen for the investigation of photocatalytic performances in correlation with the UV intensities measured in the South Pannonia region. The low supersaturation coprecipitation method was used for the ZnAl LDH synthesis. For the characterization of LDH and thermal treated samples powder X-ray diffraction (XRD), scanning electron microscopy (SEM), electron dispersive spectroscopy (EDS) and nitrogen adsorption-desorption were used. The decomposition of azodye, methylene blue was chosen as photocatalytic test reaction. The study showed that the ZnAl mixed oxide obtained by thermal decomposition of ZnAl LDH has stable activity in the broader UV light irradiation range characterizing the selected region. Photocatalytic activity could be mainly attributed to the ZnO phase, detected both in LDH and thermally treated samples. The study showed that the ZnAl mixed oxide obtained by the calcination of ZnAl LDH has a stable activity within the measured UV light irradiation range, whereas the parent ZnAl LDH catalyst did not perform satisfactory when low UV irradiation intensity is implied.

Keywords: ZnAl layered double hydroxides, ZnAl mixed oxides, UV irradiation, photocatalytic activity, methylene blue.

Recently there has been an increasing demand for catalysts that can eliminate environmental pollution because the environmental deterioration has become a major obstacle. The most extensive solution for these ongoing problems has been the introduction of the photocatalytic phenomena where a semiconductor is activated by light irradiation leading to the photocatalytic reaction that produces compounds harmless to the environment [1]. Many semiconductors have been utilized in these reactions but the most common is TiO₂ that has been very promising in the areas of environment and energy [2, 3]. However, this photocatalyst is activated by UV light (only up to 5%

of the solar light) that limits the application to this excitation source. This disadvantage could be overcome by surface modification of TiO₂ photocatalyst and catalyst support (texture, surface properties and composition) and therefore has been the topic of numerous studies [4,5]. For the past several years, layered double hydroxides (LDHs) have been studied to a great extent as environmental-friendly complex materials that can be used as photocatalysts or photocatalyst supports [6]. LDHs, also known as anionic clays and hydrotalcite-like materials, have a layered structure which enables specific properties of these materials and their thermally derived mixed oxides such as redox and acid-base properties, developed surface area, mesoporosity, ion exchange capability, etc. The LDHs layered structure is formed by isomorphic substitution of divalent cations in the brucite lattice with the trivalent cations, and the obtained positive charge is compensated by the intercalation of anions in the interlayer, the most common being CO₃²⁻.

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In this study, ZnAl layered double hydroxides and their derived mixed oxides were chosen based on the literature data stating that among the semiconductors, ZnO ($E_g \approx 3.2$ eV) has a photocatalytic potential assigned to its activation in the visible region of the solar spectra [1,7]. Layered double hydroxide composition and synthesis method were chosen based on our previous experience with LDH synthesis [8–10]. ZnAl layered double hydroxides were synthesized by low supersaturation coprecipitation method and thermally treated. Calcination of ZnAl LDH leads to the collapse of the layered structure and the formation of highly active mixed ZnAl oxides with high surface area and thermal stability. In selecting the test reaction and the measurement procedure the published literature data confirmed that there is good correlation in evaluating catalytic performance concerning the results obtained by spectroscopy and by TOC/COD [11,12]. This study is aimed to investigate photocatalytic activity of ZnAl layered double hydroxides and its derived mixed oxides at different UV irradiation intensities. The UV light irradiation intensities were chosen taking into consideration the intensities in the winter and summer periods measured in the South Pannonia region (Novi Sad, Serbia). These materials could further be applied as catalyst support with its involvement in catalytic act as the active phase, having also intrinsic activity shifted to the visible region of the solar light spectra. Additionally, this catalyst support provides a proper matrix for the optimal distribution of other possible active components (*e.g.*, TiO_2).

EXPERIMENTAL

For the synthesis of ZnAl LDH, the low supersaturation (LS) coprecipitation method at constant pH (9–9.5) was used. The precursors, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (concentrations: $[\text{Zn}(\text{NO}_3)_2] = 0.7$ mol dm^{-3} ; $[\text{Al}(\text{NO}_3)_3] = 0.3$ mol dm^{-3}) were added at constant flow (4 $\text{cm}^3\text{min}^{-1}$), whereas carbonate and hydroxide ions (0.67 M Na_2CO_3 and 2.25 M NaOH solution) were added with the flow adjusted to maintain constant pH value. The coprecipitation took place at constant temperature (40 °C) and under vigorous stirring. After the coprecipitation, the precipitates were aged for 15 h, washed with water until pH 7, dried for 24 h at 100 °C and calcined for 5 h, at 500 °C in air. The dried samples were denoted as ZnAl_LDH and calcined as C_ZnAl (the lower dash indicating that the sample was synthesized within the study).

Scanning electron microscopy (SEM) was used to investigate the morphology of the samples and electron dispersive spectroscopy (EDS) for the surface

chemical analysis JEOL, JSM-6460LV instrument, at the working voltage of 20 kV and working distance of 10 mm.

The crystalline phases of the dried and calcined samples were investigated and identified by X-ray powder diffraction (XRD) using a Siemens D500 X-ray diffractometer with filtered $\text{CuK}\alpha$ radiation in 2θ range from 5 to 70° (angular steps of 0.02° with step time of 1 s).

For the investigation of the surface area and pore size distribution of dried and calcined samples low-temperature nitrogen sorption at -196 °C (Micromeritics ASAP 2010 instrument) was used. Prior to the isotherm measurements the dried and calcined samples were degassed at 110 °C. The surface area of the samples was determined using the Brunauer-Emmet-Teller (BET) method and pore size distribution was calculated using the Barret-Joyner-Halenda (BJH) equation.

The photocatalytic tests were performed in a Pyrex flask batch-type reactor under UV-A light (polychromatic black-light-blue in the range from 320 to 400 nm) using an Osram Eversun L40W/79K lamp at different intensities. The photocatalytic activity of both synthesized and calcined samples was estimated by methylene blue (MBlue) degradation monitoring the decrease of the MBlue concentration. A commercial UV radiometer (PMA 2100) was used for the measurement of UV irradiation intensity. The photocatalytic tests were performed under two UV light intensities: 0.71 (denoted as a low intensity) and 0.912 mW/cm^2 (denoted as a high intensity). In the 10 $\mu\text{mol}/\text{l}$ MBlue water solution (100 ml) 50 mg of the powdered sample (ZnAl_LDH, C_ZnAl) was added. In order to establish the adsorption/desorption equilibrium between the dye and the surface of the catalysts the reaction solution was vigorously stirred for 30 min in the dark. After establishing the equilibrium, the reaction solutions were UV irradiated and aliquots of the solution were taken after 30, 60, 90, 120 and 180 min of irradiation. Considering that the adsorption phenomena might occur in the reaction solution and influence the values of the photocatalytic activity, blank samples (reaction solution with the catalyst) were kept in the dark and MBlue concentration was measured at the same time intervals. UV-Vis spectrophotometry (Evolution 600 spectrophotometer) at $\lambda = 664$ nm was used for the measurement of MBlue concentration and photocatalytic activity was evaluated and calculated from the following formula: $A[\%] = 100[(c_0 - c)/c_0]$; c_0 being the concentration of MBlue in blank sample, c the concentration of MBlue in UV irradiated samples, both at the same defined time intervals. The UV intensities in the South Pannonia region were

measured in the period from January to August, 2011, by a commercial UV radiometer (PMA 2100) at noon (12:00 h) every day.

RESULTS AND DISCUSSION

The XRD diffraction patterns of the dried sample (ZnAl_LDH) and the mixed oxides obtained after the calcination (C_ZnAl) are presented in Figure 1. For the ZnAl_LDH sample, XRD peaks correspond to the characteristic diffractograms for the layered structure of the hydroxide-like materials and the symmetrical peaks indicate satisfactory level of order in the stacks [7,8,13,14]. High intensities of the peaks suggest well defined crystal structure of these layered double hydroxides. For this sample low intensity peaks were also observed that can be assigned to the presence of a crystalline ZnO phase dispersed over the brucite-like layers [7,15]. Calcination of the samples leads to the collapse of the layered structure and subsequently new diffraction peaks appear. The XRD peaks of the calcined sample (C_ZnAl) reveal the decomposition of the LDH phase leading to the zincite, ZnO phase

and the spinel phase (ZnAl_2O_4) [16]. The broadening of the peaks with lower intensities indicate lower crystallinity of these phases.

The SEM images (Figure 2) show the morphology of the calcined sample (C_ZnAl). The SEM micrographs indicate the presence of agglomerates of plate-like particles in the sand-rose formation that is typical for the LDHs and their derived mixed oxides [15]. As compared to our previous study [8] the morphology of the sample additionally confirms the successful synthesis of ZnAl LDH, underlining that the morphology of layered double hydroxides is preserved after thermal treatment when non-stoichiometric mixed oxides are obtained [15,17].

Multipoint electron dispersive spectroscopy analysis was used for the generation of EDS spectra (results from the one of the measured locations are presented in Figure 3). The elemental chemical analysis of the sample surface confirmed successful incorporation of constituent elements Zn and Al into the LDH and mixed oxide matrix. The multipoint analysis did not detect any impurities.

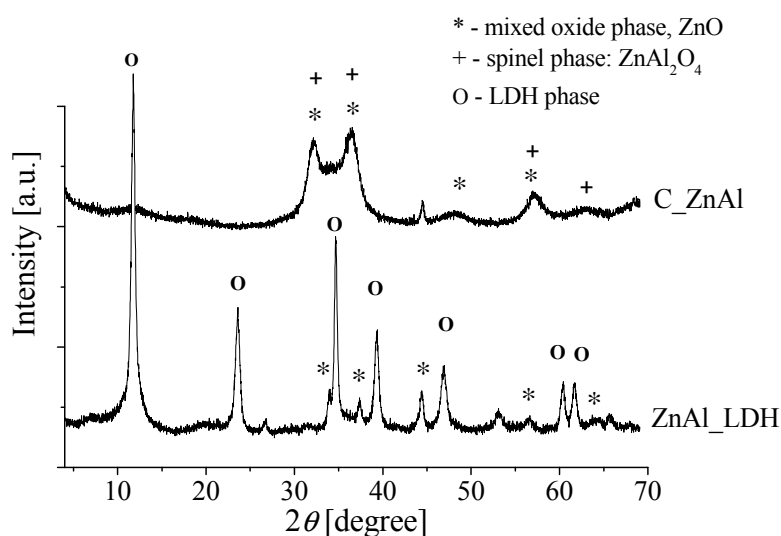


Figure 1. XRD Patterns of the dried sample (ZnAl_LDH) and the calcined sample (C_ZnAl).

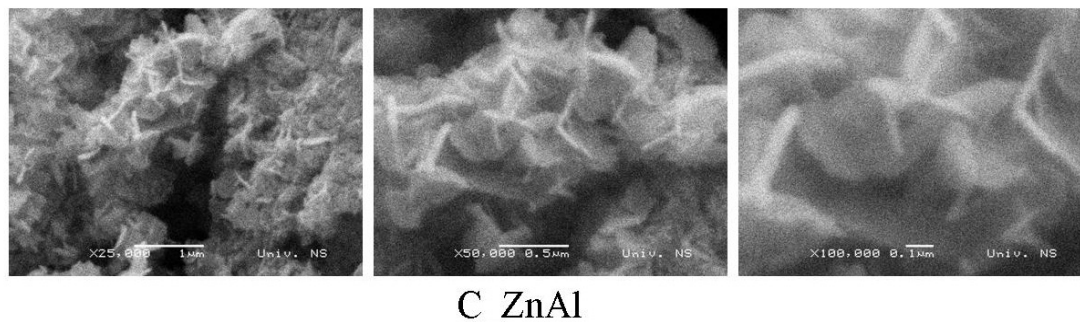


Figure 2. SEM Images of the calcined sample (C_ZnAl) at 25000, 50000 and 100000 magnification.

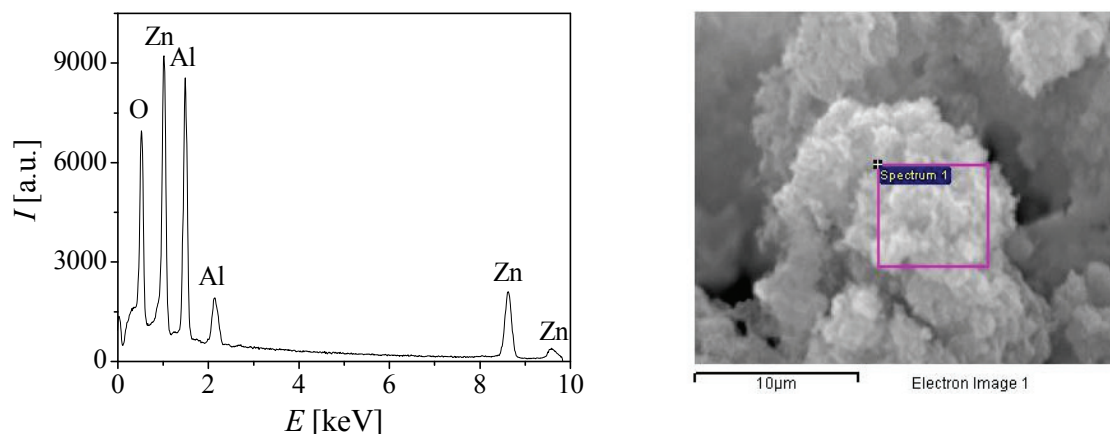


Figure 3. EDS Spectra of the calcined sample (C_ZnAl) and the location of analysis.

The textural properties (surface area and pore size distribution) of the dried (ZnAl_LDH) and calcined (C_ZnAl) samples were obtained by physico-chemical adsorption of nitrogen and the changes in the surface area values and pore size distribution before and after thermal treatment are presented in Table 1 and Figure 4. During the thermal treatment and decomposition of LDH drastic changes in sample structure occur, as confirmed by XRD analysis, leading to the collapse of the layered structure coupled with the evolution of gaseous products (water and CO₂). As a result of thermal decomposition, low crystalline, nonstoichiometric mixed oxides ZnO and ZnAl₂O₄ are formed having developed surface area due to the increase in smaller mesopore fraction (Figure 4) [15].

The adsorption-desorption isotherms of dried (ZnAl_LDH) and calcined (C_ZnAl) samples are presented in Figure 4a. A type IV isotherm, typical for mesoporous materials, with a broad type H3 hysteresis loop in the middle range of relative pressure was observed for the dried sample ZnAl_LDH. At low relative pressures a plateau was observed, which is characterized as the formation of a monolayer followed by multilayer adsorption and condensation in mesopores. The condensation process occurs at relative pressures higher than 0.8, giving rise to a sharp adsorption volume increase. The shape of the isotherm reveals that the sample ZnAl_LDH has a mesoporous character and the pore condensation shift to higher relative pressures indicates a presence of mesopores with larger diameters [18]. From the hyste-

Table 1. Surface area values of dried and calcined samples

Sample	Surface area, m ² g ⁻¹	BJH desorption cumulative pore volume, cm ³ g ⁻¹	Dominant mesopore diameter, nm bimodal pore size distribution		Detected shoulder, mesopore diameter range, nm
ZnAl_LDH	26.07	0.109	3.6	30	12-25
C_ZnAl	96.52	0.276	3.2	33	11.5-28

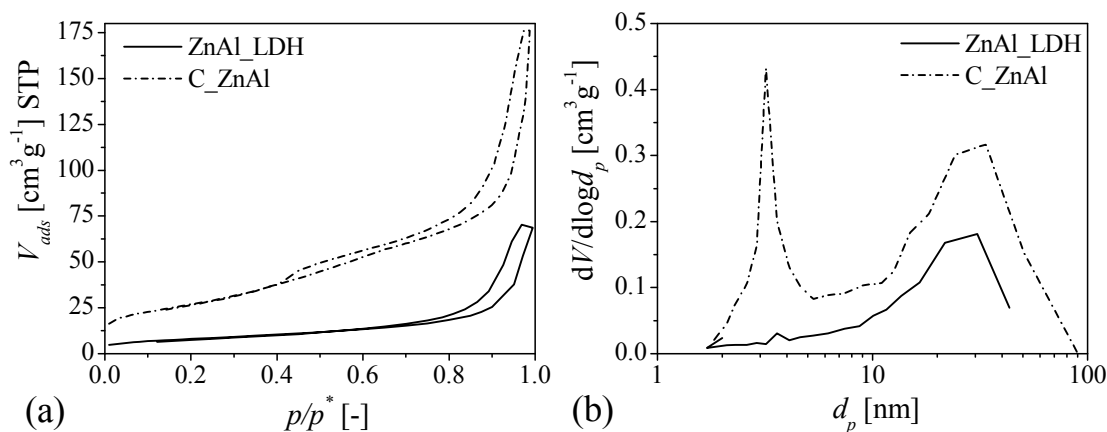


Figure 4. Textural analysis of the dried sample (ZnAl_LDH) and the calcined sample (C_ZnAl); a) adsorption-desorption isotherms and b) pore size distribution.

resis type it can be concluded that the aggregates of plate-like particles forming nonuniform slit-shape pores are present in this sample [18,19]. After thermal treatment adsorption-desorption isotherm changes. This could still be assigned as type IV isotherm, but the hysteresis loop is a combination of type H3 and type H4. The type H4 loops are associated with narrow slit pores [18] which evidently evolve after thermal decomposition, as confirmed by pore size distribution presented in Figure 4b. The corresponding pore size distribution of dried and calcined samples was obtained using BJH method from the desorption branch of the isotherms. A bimodal pore size distribution was identified in both samples. The dried sample exhibited a low intensity peak with maxima in the area of smaller mesopores (≈ 3.6 nm) and a broad peak in the area of larger mesopores (≈ 30 nm). Also, a broad shoulder shifting towards smaller mesopores (12-25 nm) was detected. This low cumulative pore volume is in correspondence with the value of the surface area for the dried sample, Table 1. After thermal treatment (C_ZnAl), due to the destruction of the lamellar structure, the surface area increases 3.8 times and consequently the cumulative pore volume value is doubled compared to the dried sample, Figure 4b. After thermal decomposition the low intensity peak detected for the ZnAl_LDH evolved into an intense sharp peak (≈ 3.2 nm) and the volume of the larger mesopores also is increased. For the larger mesopores, the broad shoulder shifting towards smaller mesopores was observed in a slightly different range of pore diameter (11.5-28 nm).

The UV intensities of solar light measured for the period from January until August in the South Pannonia region are presented in Figure 5. The winter months exhibited lower UV-A intensities with the average value of 0.362 mW/cm^2 (January to March) and a gradual increase in the intensity was detected after this period, Figure 5 and Table 2. The average value of the UV intensities of solar light measured in the period from January to August, 2011, is 1.882 mW/cm^2 . UV lamp intensities (0.71 and 0.912 mW/cm^2) lower than the average value measured from January to August were chosen for the experiment. The UV lamp intensity was selected to be lower than the average value measured taking into account the forthcoming autumn period (September to December), which would definitely reduce this average value. This selection was made in order to match as much as possible the expected year average value keeping in mind that the photocatalyst should perform efficiently in real conditions under solar light all year round.

The results for the photocatalytic activity of the samples induced by different intensities of UV irradiation are presented in Figures 6-9. In order to eliminate the influence of methylene blue concentration change due to other phenomena than the photocatalytic decomposition in the selected test reaction, methylene blue concentrations for both samples (in the dark and under UV irradiation) were monitored, Figure 6. The results show that the adsorption/desorption equilibrium of methylene blue was reached after 30 min in the dark for both ZnAl_LDH and

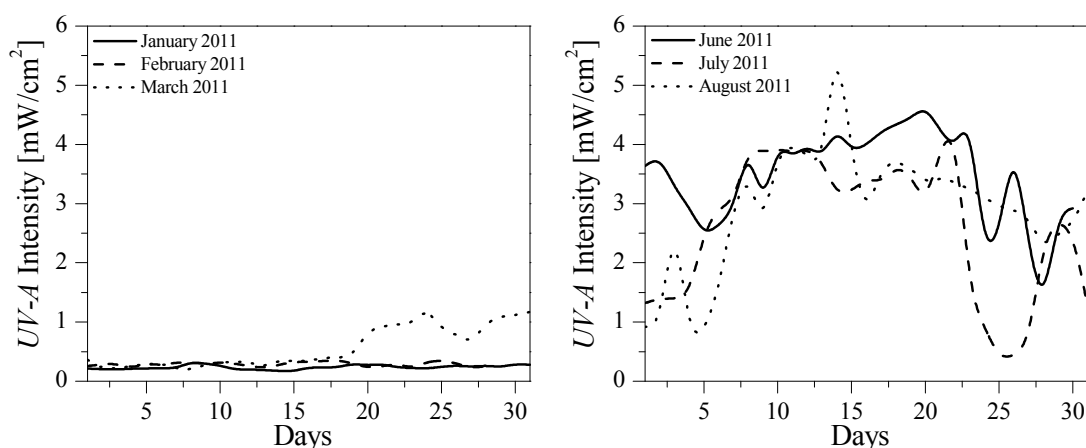


Figure 5. UV intensities for the winter period (January-March) and summer period (June-August) in the South Pannonian region (Novi Sad, Serbia).

Table 2. The average values of UV-A intensities measured in Novi Sad, Serbia from January to August 2011

Month	January	February	March	April	May	June	July	August	Jan-Aug
Average UV-A intensity, mW/cm^2	0.237	0.287	0.562	2.444	2.468	3.433	2.628	2.943	1.882

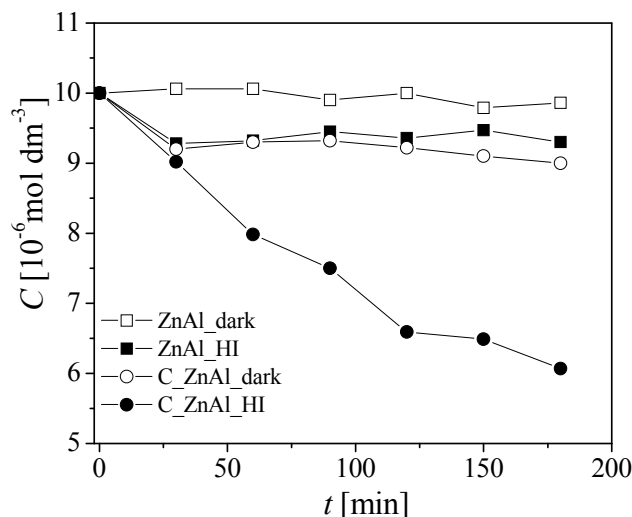


Figure 6. Methylene blue concentration curves for samples: ZnAl_dark, ZnAl_HI, C_ZnAl_dark and C_ZnAl_HI.

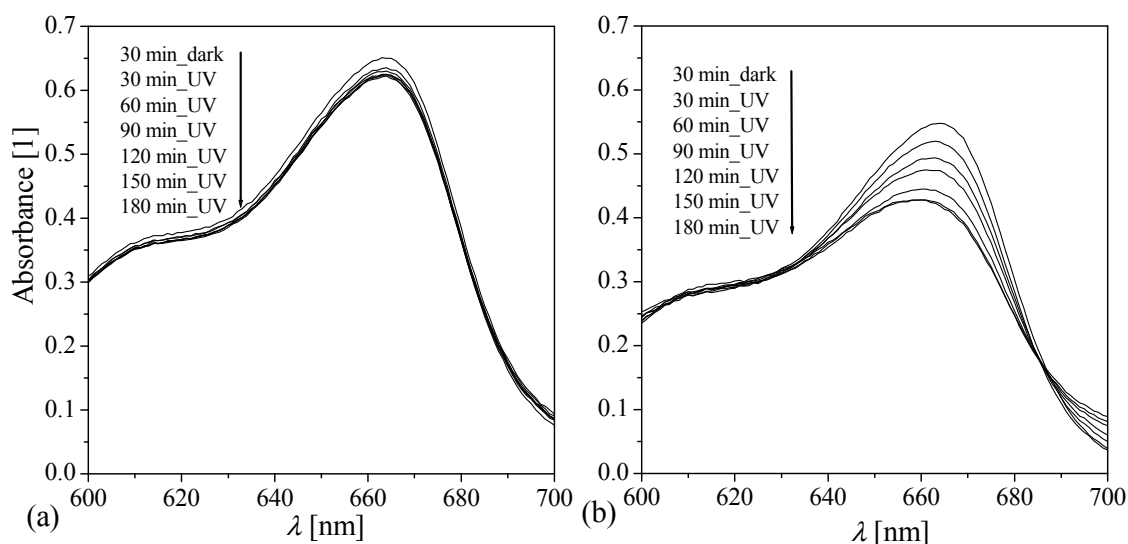


Figure 7. Absorption spectra of methylene blue photocatalytic degradation (absorption maximum at 664 nm) with a) ZnAl_LDH and b) C_ZnAl during HI UV irradiation.

C_ZnAl samples. For the ZnAl_LDH sample the decrease of methylene blue concentration due to the adsorption was insignificant ($\approx 0.5\%$) throughout the whole reaction test. The calcined sample C_ZnAl showed the decrease in of MBlue concentration in the initial period ($\approx 8.1\%$) and maintained a steady-state throughout the whole reaction test. Considering the well known memory effect of mixed oxides derived from LDHs [20-23], one can speculate that the methylene blue concentration decrease observed for the calcined sample in the dark could be attributed to the memory effect that did occur to some extent resulting in partial LDH structure reconstruction. Nevertheless, the difference in photocatalytic performance, originating mostly from the ZnO phase [7,15] implies that

larger amount of this active phase remains in the calcined sample.

Figure 7 shows the absorption spectra in the relevant wave-length region (600-700 nm) recorded at defined time intervals for the dried and calcined samples under HI UV intensity. The intensity decrease observed for characteristic methylene blue absorption curves is much more pronounced in time for the calcined sample. The same trend was observed for LI UV irradiation. In order to quantify the observed catalytic effect, the photocatalytic conversion was evaluated based on the intensity of the peak maxima at 664 nm and presented in Figure 8. For both UV intensities applied it was observed that with the increase of UV irradiation time, there is an increase in MBlue conversion in all investigated samples. Accord-

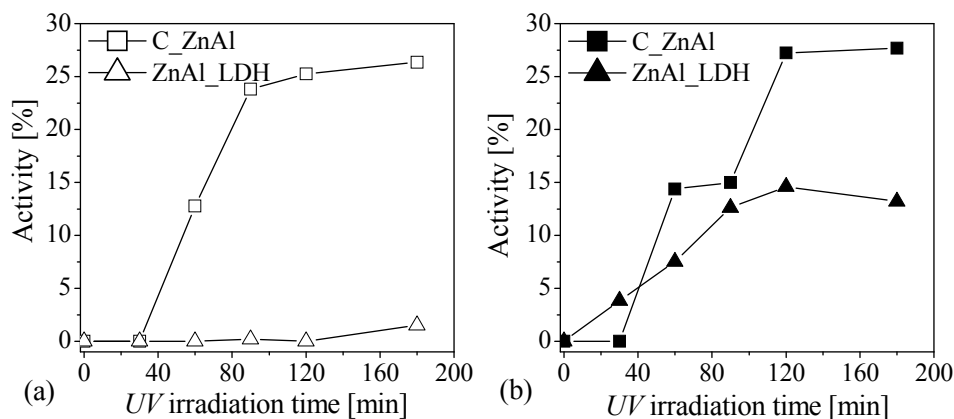


Figure 8. Photocatalytic activity of the dried (ZnAl_LDH) and the calcined (C_ZnAl) samples at a) low intensity UV irradiation - LI and b) high intensity UV irradiation - HI.

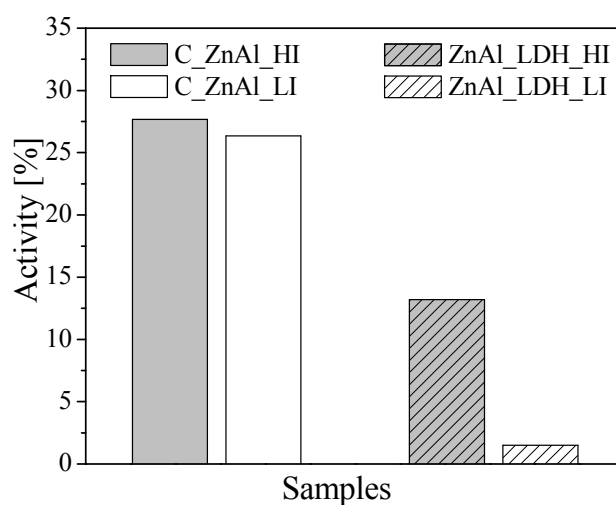


Figure 9. Histogram comparison of sample photocatalytic activities after 180 min at high (HI) and low (LI) intensities of UV irradiation.

ing to literature, this photodegradation process increase, evaluated by MBlue conversion, is probably due to the fact that the number of photons, essential for the photocatalytic reaction, grows with the increase of UV irradiation time [14]. The photocatalytic activity is highly improved for both, low and high UV intensity when the samples are calcined. These samples have much higher presence of the ZnO phase that is known as being responsible for the photocatalytic activity. Besides ZnO phase, ZnAl₂O₄ spinel phase, detected in calcined samples, also contributes to the photocatalytic activity due to its band gap of 3.8 eV suitable for photocatalytic processes, as suggested in recent publications [6,15]. The calcined samples also have much higher surface area which enables better utilization of the active sites present on the surface of the photocatalyst.

From the photocatalytic results it could be concluded that the calcined samples have stable activity within the measured UV light irradiation range, since the change in the UV intensity did not significantly in-

fluence the photocatalytic activity of these samples (Figures 8 and 9). The difference in the activity between dried and calcined samples at lower UV intensity is more evident. The interesting point of this part of the investigation is the photocatalytic activity of the dried samples at different UV intensities. Low UV intensity did not provoke the photocatalytic reaction of the dried sample, whereas the 30 % increase in the UV intensity (HI) induces photocatalytic activity of this sample (Figures 8 and 9). The obtained value of 12% for ZnAl_LDH_HI sample can be still considered as quite low, especially when roughly compared to the other published data using different test procedures and test compounds [7,23].

These very different activity values of the dried sample could be explained by the presence of photons and ZnO phase. The number of photons emitted by low UV intensities was probably not enough to trigger the photoactivation of the small amount of the ZnO phase in dried sample, revealed by XRD analysis. On the contrary, the calcined samples have large

amounts of the ZnO phase and photocatalytic active spinel phase and even a small number of photons emitted by low UV intensity is sufficient for good photocatalytic activity of these samples.

CONCLUSION

The study showed that the ZnAl mixed oxide obtained by the calcination of ZnAl LDH has a stable activity within the measured UV light irradiation range usual for the average value of the UV intensities of solar light in the South Pannonia region (Novi Sad, Serbia). On the contrary, the parent ZnAl LDH catalyst did not perform satisfactory, since the low UV intensity did not provoke the photocatalytic activity in this sample. The phase responsible for the photocatalytic activity is ZnO phase together with the ZnAl₂O₄ spinel phase, detected in the calcined samples. The results showed that the efficiency of the photocatalyst depends also on the location of application and climate of the selected regions. The stable photocatalyst can be obtained by ZnAl LDH thermal treatment showing satisfactory photocatalytic performance even at lower UV intensities common for the Northern European region. In order to enhance the photocatalytic activity of ZnAl LDH derived photocatalysts, it would be interesting for future investigation to combine TiO₂ and ZnAl LDHs with the intention to enhance the overall photocatalytic activity in the solar light.

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NAUČNI RAD

UTICAJ INTENZITETA UV ZRAČENJA NA FOTOKATALITIČKU AKTIVNOST ZnAl DVOSTRUKIH SLOJEVITIH HIDROKSIDA I NJIHOVIH MEŠOVITIH OKSIDA

Istraživanje dvostrukih slojevitih hidroksida (LDH) se u poslednje vreme usmerava ka ispitivanju ovih kompleksnih materijala kao fotokatalizatora ili nosača fotokatalizatora u oblasti zaštite okoline. Zlispitivane su fotokatalitičke performanse u korelaciji sa intenzitetima UV zračenja izmerenim u oblasti Južne Panonije, korišćeni su ZnAl dvostrukih slojevitih hidroksida i njihovih mešovutih oksidobijenih termičkom aktivacijom. Sinteza ZnAl LDH je izvršena metodom koprecipitacije niske prezasićenosti. Defrakcija X-zraka (XRD), skenirana elektronska mikroskopija (SEM), elektron dispreziona mikroskopija (EDS), kao i nisko-temperaturna adsorpcija azotom su primenjene za karakterizaciju sintetisanih LDH i termički tretiranih mešovutih oksida. Kao fotokatalitička test reakcija odabrana je reakcija razgradnje azo boje (metilensko plavo). Istraživanja su pokazala da ZnAl mešoviti oksidi, dobijeni termičkom razgradnjom ZnAl LDH, imaju stabilnu aktivnost u širem opsegu intenziteta UV zračenja karakterističnog za oblast Južne Panonije. Fotokatalitička aktivnost ovih materijala se može pripisati ZnO fazi detektovanoj kod ZnAl LDH i njihovih mešovutih oksida. Ova studija je pokazala da ZnAl mešoviti oksidi dobijeni kalcinacijom ZnAl LDH imaju stabilnu aktivnost u opsegu merenog UV zračenja, a ZnAl LDH nisu pokazali zadovoljavajuću fotokatalitičku aktivnost na niskim intenzitetima UV zračenja.

Ključne reči: ZnAl dvostruki slojeviti hidroksidi, ZnAl mešoviti oksidi, UV zračenje, fotokatalitička aktivnost, metilensko plavo.