THE ACADEMY OF APPLIED
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BELGRADE

INTERNATIONAL SCIENTIFIC
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POLITEHNIKA 2023

CONFERENCE PROCEEDINGS

Belgrade, 15th December 2023

INTERNATIONAL SCIENTIFIC
AND PROFESSIONAL CONFERENCE

POLITEHNIKA 2023

CONFERENCE PROCEEDINGS

Belgrade, 15th December 2023

PUBLISHER
The Academy of Applied Technical Studies Belgrade
Katarine Ambrozić 3, Belgrade
www.atssb.edu.rs

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DESIGN OF THE CONFERENCE LOGO
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PRINT
The Academy of Applied Technical Studies Belgrade, Katarine Ambrozić 3, Belgrade

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FOREWORD

The International Scientific and Professional Conference POLITEHNIKA 2023 represents the seventh edition of the POLITEHNIKA scientific and professional events, occurring biannually since its inaugural event in 2011. POLITEHNIKA 2023 upholds a distinguished tradition and commitment to integrating higher education and practical application across a diverse spectrum of disciplines represented by defined thematic scopes.

Organized with the patronage of the Ministry of Education of the Republic of Serbia, the Ministry of Environmental Protection of the Republic of Serbia, the Ministry of European Integration of the Republic of Serbia, the Directorate for Occupational Safety and Health, the Office for Dual Education and National Qualifications Framework, the Conference of Academies of Applied Studies in Serbia, the Chamber of Commerce of Serbia, the Chamber of Commerce of Belgrade, the Institute for Standardization of Serbia, the Association of Belgrade Architects, the City of Požarevac and the Tourist Organization of the City of Požarevac, POLITEHNIKA 2023 stands as a collaborative platform at the intersection of academia, governmental institutions and industry.

This year heralds a notable progression with its international status and the incorporation of 10 conference scopes. Expanding beyond the thematic domains featured in previous events, the Conference now encompasses Environment and Sustainable Development, Occupational Safety and Health and Fire Safety, Smart Management Systems, Graphic Engineering, Design, Traffic Engineering, Biotechnology and Healthcare, Mechanical Engineering, Ecotourism and Rural development, and Mechatronics. By engaging experts, emerging professionals, and practitioners from these domains, the conference unifies fields of study programs of the Academy of Applied Technical Studies Belgrade. The thematic scopes, coupled with the structure of the compiled papers in this Proceedings, exhibit a rich diversity and multidisciplinary approach, fundamentally contributing to a holistic examination and resolution of societal and scientific challenges.

Comprising over 220 peer-reviewed contributions, the Proceedings represent a substantial intellectual asset, aligning with the conference's overarching objective of fostering the exchange of knowledge, research findings, and professional experiences among experts from industry, research institutions, and higher education establishments.

The Proceedings of the International Scientific and Professional Conference POLITEHNIKA 2023 serve as a comprehensive snapshot of the current landscape within the thematic realms of the conference, offering both insights and directives for ongoing scientific and professional development. Moreover, they proffer concrete solutions to practical challenges grounded in contemporary trends and pertinent insights.

The Academy of Applied Technical Studies Belgrade extends its sincere appreciation to all conference supporters whose financial contributions played a pivotal role in its successful realization. Special acknowledgment is reserved for the authors of the papers, whose diligence and eagerness to present their work to a wider audience, alongside the reviewers and members of the International Scientific Committee, Program Committee and Organizational Committee, have collectively contributed to the triumph of the International Scientific and Professional Conference POLITEHNIKA 2023.

Belgrade, December 2023
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ENVIRONMENT AND SUSTAINABLE DEVELOPMENT

ALKALI MODIFICATION OF FLY ASH FOR ADSORPTION OF SELECTED DYES

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Abstract: *Fly ash (FA) is an industrial waste material generated during coal combustion in thermal power plants. In order to reuse this kind of industrial waste, and at the same time to reduce its quantity, fly ash was used as a cheap material for the preparation of efficient adsorbents for the removal of selected dyes from water. For that purpose, different modification methods were used to obtain alkali-activated fly ash samples. The success of the modification process and characterization of the unmodified and modified materials was monitored by Fourier transform infrared spectroscopy, and scanning electron microscopy/energy dispersive spectroscopy. The adsorption properties of examined samples were studied through the adsorption of methylene blue, crystal violet, alizarin red, and methyl orange. All samples showed the highest adsorption efficiency for crystal violet removal, while the sample modified with a combination of NaOH, Na_2SiO_3, and diatomaceous earth (FA/geopolymer) was the most efficient adsorbent for all selected dyes. Therefore, the influence of initial pH value and contact time on adsorption was examined for the adsorption of crystal violet onto FA/geopolymer. It was found that alkaline modification improves the adsorption characteristics of fly ash making it a promising candidate to solve the problem of colored wastewater.*
Keywords: Fly Ash, Dyes, Alkali Modification, Adsorption.

1. INTRODUCTION

Discharge of water polluted with dyes from different industries leads to several problems in the environment. The presence of dyes in water affects its coloration, the permeability of sunlight, and thus the process of photosynthesis and the food chain, as well. Therefore, their removal from the environment is very important. Among the various methods (membrane filtration, photodegradation, ion exchange), adsorption stands out as a simple and energy-efficient technique [1,2]. The advantage of this method is reflected in the large number of available adsorbents. A significant amount of scientific research is focused on developing methods for repurposing agricultural, municipal, and industrial waste. In this way, waste materials gain a use value, their amount in landfills can be reduced, and thus, the harmful impact on the environment can be reduced as well. Fly ash represents a growing environmental problem, as it is an inevitable by-product of coal combustion. In Serbia, the share of coal in the production of electricity is 65 %. Large amounts of ash end up in landfills, and its

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disposal and transport represent a growing environmental problem. Therefore, numerous researches are focused on finding ways to reuse fly ash. One of the most important applications of fly ash is as a construction material, in the production of roads and various types of concrete. Today, fly ash is also used for the rehabilitation of surfaces degraded by mining activities, as a base and covering for sanitary landfills of municipal and other waste, for the production of activated carbon and artificial zeolite, etc. [3]. One idea is to use fly ash as an effective adsorbent for the removal of various pollutants, such as waste gases, heavy metals, dyes, and other organic compounds [4]. The adsorption efficiency of fly ash can be improved by its activation and modification [2,5].
In an attempt to reuse fly ash, in this work, an alkaline and thermal modification was applied to obtain cheap and efficient adsorbents for the removal of selected dyes from water. The influence of the applied modification method on the efficiency of obtained materials to adsorb methylene blue (MB), crystal violet (CV), alizarin red S (ARS) and methyl orange (MO) was examined. The adsorption process was studied through the influence of the initial pH value of the adsorbate solution, and contact time, and obtained experimental data were compared with the kinetic models.

2. EXPERIMENTAL

2.1 Material preparation

The starting fly ash, obtained from the power plant Nikola Tesla A (Obrenovac, Serbia), was sieved and washed with distilled water, and then modified, as it is shown in Table 1. After the modification, all samples were washed with distilled water to a neutral pH and dried overnight at 105 ^{o}C.

Table 1. Modified fly ash samples, labeling, and modification conditions
Source: original author's

Sample	Mass of fly ash	Modification agent	Modification condition
Ash	–	–	–
Ash_{300}	2.0 g	3.2 g NaOH pellets	300 °C, 3 h, electrical furnace
Ash_{ref}	2.0 g	20 ml 2M NaOH	5 h, reflux, boiling temperature
Ash_G	9.8 g	4.2 g diatomaceous earth 10 g of sodium silicate few drops 16 M NaOH	drying, 2 h, 105 °C aging, 7 days, room temperature
Ash_7	2.0 g	6.0 ml 2M NaOH	shaking (120 rpm), 7 days, room temperature

2.2 Material characterization

Scanning electron microscopy (SEM) with energy dispersive spectroscopy was used to examine the changes in surface and morphological characteristics induced by the modification of fly ash.
The presence of functional groups on the surface of unmodified and modified samples was determined by Fourier transform infrared spectroscopy (FTIR).

2.3 Adsorption experiments

All adsorption experiments were performed in a batch system with constant shaking (170 rpm) at room temperature. To determine the adsorption efficiency of unmodified and modified fly ash samples (0.05 g), 20 ml of the selected dyes (MB, CV, MO, ARS) aqueous solution (20 mg/dm^3) was used, and adsorption experiments were performed for 2.5 h. Adsorption of CV onto Ash and Ash_G (0.05 g) was conducted from 20 ml aqueous solution (20 mg/dm^3) with initial pH adjusted at 3, 5, 7, and 10, to reveal the influence of the initial pH value on the adsorption. The influence of contact time on the adsorption capacities of Ash and Ash_G samples (0.25 g) was examined through the adsorption of CV (20 mg/dm^3) from 100 ml of aqueous solution. During this experiment, the concentration of

CV was measured in the specified periods of time (5, 10, 30, 60, 120, and 180 minutes). For measuring the dye concentration visible spectrophotometry was used. Obtained data were examined by pseudo-first [6] and pseudo-second [7] order kinetic models, given by equations 1 and 2, respectively:

$$q_t = q_e \cdot (1 - e^{-k_1 t}) \tag{1}$$

$$q_t = q_e - \left(\frac{1}{q_e} + k_2 t\right)^{-1} \tag{2}$$

3. RESULTS AND DISCUSSION

The surface morphology of fly ash samples was analyzed by scanning electron microscopy/energy dispersive spectroscopy and obtained results are shown in Figure 1a-1e and Table 1. All fly ash samples are characterized by a similar porous surface with visible cavities and cracks. Nevertheless, applied modifications in the presence of NaOH increase surface porosity, whereby the surface of sample Ash_G is the most porous, which can favor the adsorption of dyes. According to the results of EDS analysis, unmodified fly ash does not contain Na, while in modified samples atomic percentage of Na increases with the concentration of NaOH added and with a temperature of modification. A high content of Na in the modified samples can also favor the adsorption when it occurs through the mechanism of ion exchange.

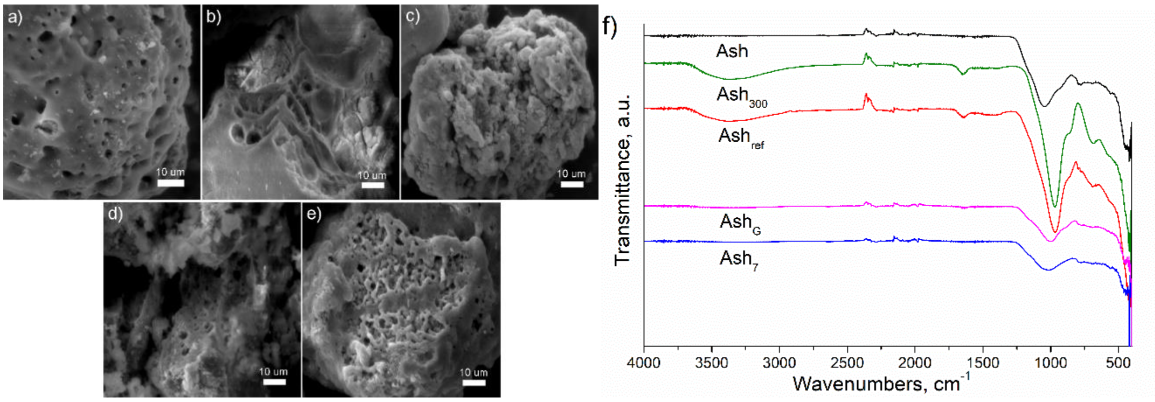

Figure 1. SEM photographs of fly ash samples: a) Ash, b) Ash_{300}, c) Ash_{ref}, d) Ash_G, e) Ash_7 and f) FTIR spectra of examined samples
Source: original author's

Table 2. The atomic percentage of elements obtained by EDS analysis
Source: original author's

Sample	O	Na	Mg	Al	Si	K	Ca	Ti	Fe
	atomic %								
Ash	69.87	-	0.81	9.47	16.43	0.55	1.06	0.19	1.62
Ash_{300}	62.02	5.20	0.87	10.43	18.08	0.28	0.80	-	2.31
Ash_{ref}	59.10	1.92	1.11	13.10	20.82	1.27	0.63	0.15	1.89
Ash_G	61.44	12.71	-	5.48	18.35	0.33	0.35	-	1.33
Ash_7	59.71	0.81	1.19	14.14	18.79	0.50	1.32	0.39	3.15

FTIR spectra of fly ash samples are (Figure 1f) characterized by sharp, distinguish peaks at around 1000 cm^{-1}, and bands of low intensity around 780 cm^{-1}, which originate from the stretching of asymmetric Si - O -Si and symmetric Si - O -Si bond, respectively. The observed shift of the band around 1000 cm^{-1} to the lower wavenumber values is more pronounced for the samples modified at higher temperatures. This small shift, along with the disappearance, or decrease in intensity of peak around 780 cm^{-1} (observed for Ash_{300} and Ash_G, respectively) indicates the formation of alkali-activated materials. The bands around 3370 and 1630 cm^{-1} can be assigned to the stretching vibration of the O-H bond in hydroxyl groups from the material moisture.

The adsorption efficiency of unmodified and modified fly ash samples to remove cationic (CV and MB) and anionic (ARS and MO) dyes is given in Figure 2. Applied modifications increase adsorption efficiency for cationic dyes, up to 92 % for adsorption of CV onto Ash_G. On the other hand, these alkali treatments have no effect, or even decrease the adsorption efficiency for anionic dyes. Generally, all examined samples show the highest adsorption efficiency for CV, and Ash_G, as the sample with the highest efficiency in CV removal, was selected for further adsorption studies.

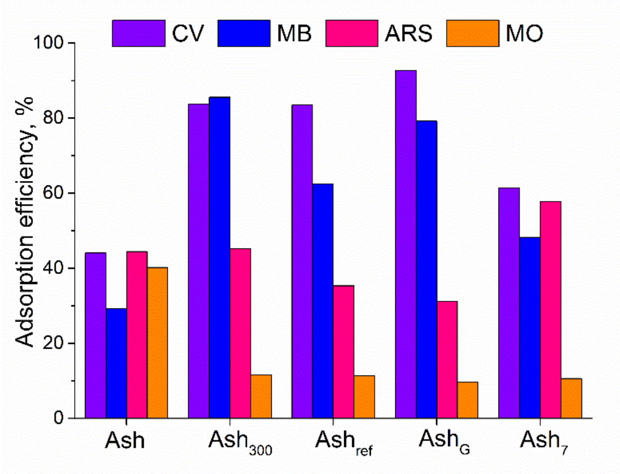

Figure 2. Adsorption efficiency of fly ash samples for removal of selected dyes
Source: original author's

The initial pH value of the dye solution can affect the adsorbent surface, as well as the distribution and the morphology of dye molecules, influencing the adsorption capacities of fly ash samples. The influence of the pH of CV solution on the adsorption capacities of Ash and Ash_G is shown in Figure 3a. Adsorption capacities increase with initial pH value, especially in the case of unmodified fly ash. The influence of contact time between adsorbent and adsorbate solution on adsorption capacities is shown in Figure 3b. The adsorption capacity for both samples increases with the contact time, reaching the equilibrium only for sample Ash.

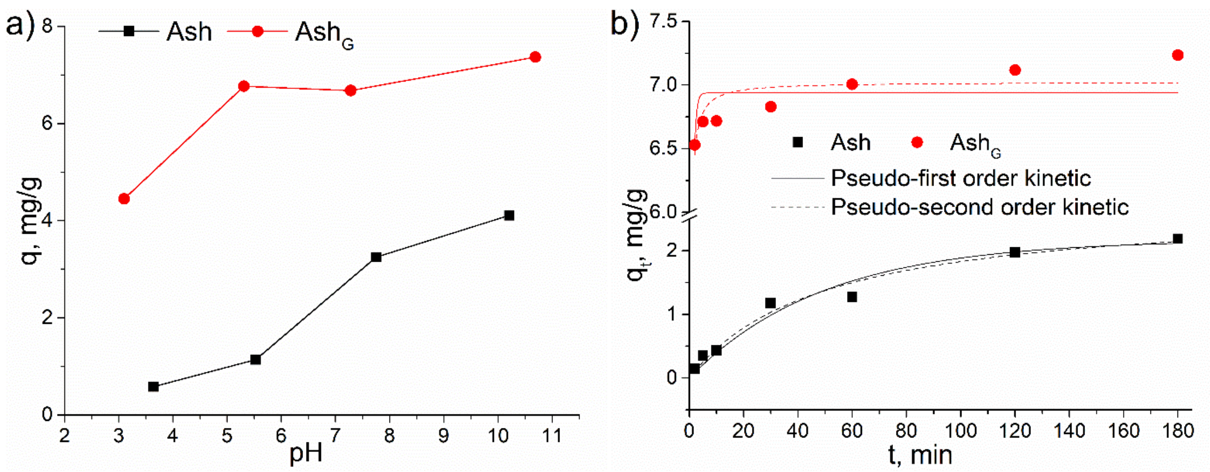

Figure 3. The influence of initial pH value (a) and contact time (b) on adsorption capacities of Ash and Ash_G
Source: original author's

Experimental data are fitted with pseudo-first and pseudo-second kinetic models and also shown in Figure 3b, while obtained kinetic parameters are summarized in Table 3. According to the correlation coefficients (R^2) adsorption of CV onto both samples fits better with pseudo-second order kinetic. However, a better agreement between the experimental adsorption capacity and the one calculated by the pseudo-first order model, indicates that CV adsorption onto Ash can be better described by this model.

Table 3. Kinetic parameters of CV adsorption onto samples Ash and Ash_G
Source: original author's

Sample	Pseudo-first order			Pseudo-second order			$q_{e,exp}$, mg/g
	$q_{e,cal}$, mg/g	k_1	R^2	$q_{e,cal}$, mg/g	k_2	R^2	
Ash	2.175	0.02014	0.95994	2.735	0.0074	0.97308	2.190
Ash_G	6.938	1.40664	0.25695	7.023	0.8015	0.59680	7.235

4. CONCLUSION

Fly ash, obtained from the power plant Nikola Tesla A, was modified using different alkali treatments and used as an adsorbent for the removal of selected dyes from water. Material characterization showed that applied modifications led to the formation of alkali-activated materials, the increase of the surface porosity, and the atomic percentage of Na. Changes in material structure induced by applied treatments led to an increase in material efficiency in adsorb cationic dyes. All samples showed the highest adsorption efficiency for crystal violet removal, while the sample modified with the combination of NaOH, Na_2SiO_3, and diatomaceous earth (Ash_G) was the most efficient adsorbent for all selected dyes. Adsorption of crystal violet onto unmodified fly ash and Ash_G increases with initial pH value and contact time, following the pseudo-first, and pseudo-second order rate, respectively. It was found that alkaline modification improves the adsorption characteristics of fly ash making it a promising candidate to solve the problem of colored wastewater.

This research was supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia (Contract No. 451-03-47/2023-01/200135 and 451-03-47/2023-01/200287), and the Science Fund of the Republic of Serbia, GRANT No 7743343, Serbian Industrial Waste towards Sustainable Environment: Resource of Strategic Elements and Removal Agent for Pollutants - SIW4SE, Program IDEAS.

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6(082)(0.034.2)
5(082)(0.034.2)
331.45/.46(082)(0.034.2)
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INTERNATIONAL Scientific and Professional Conference Politehnika (2023 ; Beograd)

Conference Proceedings [Електронски извор] / International Scientific and Professional Conference Politehnika 2023, Belgrade, 15th December 2023 ; [organizer] The Academy of Applied Technical Studies "Belgrade", Belgrade. - Belgrade : The Academy of Applied Technical Studies "Belgrade", 2023. - 1 USB fleš memorija ; 1 x 1 x 5 cm

Sistemski zahtevi: Nisu navedeni. - Nasl. sa naslovne strane dokumenta. - Tiraž 400. - Bibliografija uz svaki rad.

ISBN 978-86-7498-110-8

а) Техника -- Зборници б) Примењене науке -- Зборници в) Заштита на раду -- Зборници г) Менаџмент -- Зборници

COBISS.SR-ID 132801289

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