

ADSORPTION ON ALUMINA OF DIFFERENT ORIGIN

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Abstract: The adsorbent used in the study was the alumina obtained from the bauxite found in the locality of Milici, Siroki Brijeg and alumina synthesized in the laboratory and obtained by sedimentation from the solution of aluminium nitrate and concentrated ammonia along with controlling pH solution in the course of sedimentation. The obtained sediment of alumina was annealed for 4 hours at 273 K. The adsorbates were triphenylmethane dyes, carboxylic acids – acetic and lauric one and aqueous solution of ammonia. Alumina was modified by the surface active agents (SAA), cation-active SAA, triethanolamine-di-estermethylsulfate called Propagen, and anion-active SAA, Na-salt alkyldiglycoethersulfate called Genapol. The textural characteristics of adsorbents were determined by the adsorption of nitrogen from the gas phase at the temperature of liquid nitrogen, and the results of the adsorption of acid and base adsorbates gave an insight in the changes, i.e. characteristics of surface active centers on which the adsorption takes place.

Keywords: alumina, adsorption, triphenylmethane dyes, carboxylic acids, modification with PAM.

1. INTRODUCTION

Aluminium oxide was one of the first materials industrially used as adsorbent, catalyst and catalyst carrier, which even nowadays remains significant and is used for the same purposes.

The reason for this significance of alumina as adsorbent and catalyst lies with its extraordinary mechanical, physical and chemical characteristics. Some characteristics of alumina, like porousness, specific surface, phase composition, are affected by the process of thermic treatment of minerals, conditions of aluminium hydroxide sedimentation, rinsing of pH environment, drying, and there could also be a strong effect of adding some additive into the solution from which sedimentation is performed.

The chemistry of γ -Al₂O₃ surface is very complex [1–5]. The surface of alumina is composed of a combination of Al³⁺ and O²⁻ ions, on which there is always a certain number of OH⁻ groups or water molecules with coordinate bond. The acidity of alumina is directly connected with the number

and manner of OH⁻ groups bonding. There are five types of OH⁻ groups with regard to their coordination number, i.e. depending on whether they are bonded with a tetrahedron or octahedron aluminium (Figure 1). Their acidity depends on the environment, and the most acid center originated from OH⁻ group bonded with three octahedron Al atoms (Figure 1d). The acidity of OH⁻ group on the surface of alumina essentially depends on the density of its electric charge, while it is related to the number of O²⁻ ions in the environment and distance of the observed OH⁻ group from them.

Figure 2 presents five types of OH⁻ groups in Al₂O₃·xH₂O, which are marked with A to E. Type A is directly surrounded by four O²⁻ ions, and because of that it is most negative. It is a potential base center that aspires to receive a proton – Brønsted base center. Type E represents OH⁻ ion, which in its immediate surroundings does not have any O²⁻ ions (it is surrounded by four distanced O²⁻ ions), it is most positive and represents the acid center aspiring to give an atom away – Brønsted acid center.

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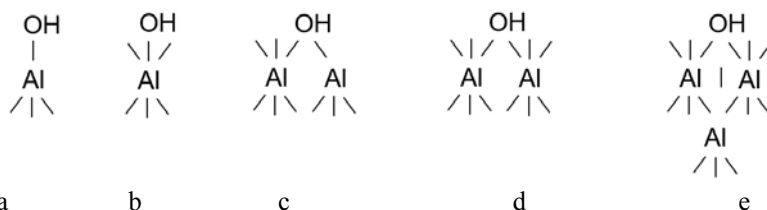


Figure 1. Different types of OH groups in alumina depending on the associated Al-ion (a-tetrahedron Al, b-octahedron Al, c-tetrahedron and octahedron Al, d-two octahedron Al, e-three octahedron Al)

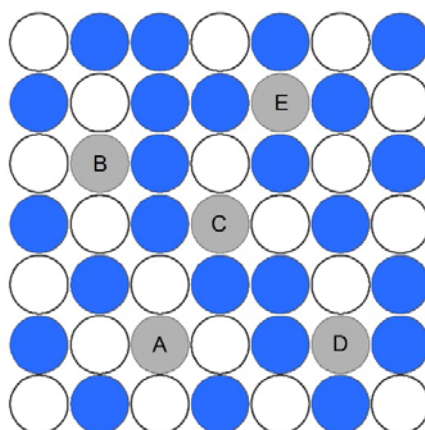


Figure 2. Different types of OH- ions (A-E) in $Al_2O_3 \cdot xH_2O$ depending on the number of O^{2-} ions from the immediate surroundings, \bullet O^{2-} ion, \circ Al^{3+} ion

The acidity of alumina occurs by removing water from its surface, and the type of formed acid centers depends on the degree of water removal. By heating alumina and removing one molecule of water from two neighboring atoms of A, we get one Lewis acid and base center respectively. The first represents a place with a deficit of electrons, while the other one aspires to release electrons. The adsorption of water molecules on Lewis acid center results in the polarization of bonds in $H-O-H^+$, by which there a hydrogen atom gets created with a deficit of electrons – proton. This one is easily released and acts like Brønsted acid center. In the definition of acid/base character, Brønsted concept functions by the control of charge, while Lewis one functions by the control of orbitals, i.e. by the number of electrons in the shell.

2. RESULTS AND DISCUSSION

The samples of alumina used in the study were obtained from the bauxite of boehmite type from the locations in Milici and Siroki Brijeg,

Bosnia and Herzegovina. The presence of aluminum oxide hydrated with one molecule of water ($Al_2O_3 \cdot H_2O$) is a common characteristic of the bauxite from these locations, and that dominant mineral form is present in the quantity of 58.00% in the bauxite of Milici and 50.44% in the bauxite from the location of Siroki Brijeg [6]. The diffractograms of the sample of Milici bauxite and Siroki Brijeg bauxite are presented in Figures no. 3 and 4. The samples of alumina obtained in this manner were annealed at 600^0 C before adsorption.

The study comprises an overview of the research of adsorption from the gas and liquid phase on alumina. The adsorbate used in the gas phase was nitrogen at the temperature of liquid nitrogen, and the adsorbates from the liquid phase were the aqueous solutions of organic colors (methylene blue and methyl violet), ammonia and acetic acid, while the lauric acid was from the solution of ethanol.

The research was conducted on the effect of pH values in the course of sedimentation of aluminum hydroxide, conditions of rinsing, regime of drying and annealing of the obtained sediment (of alumina) on the specific surface of alumina.

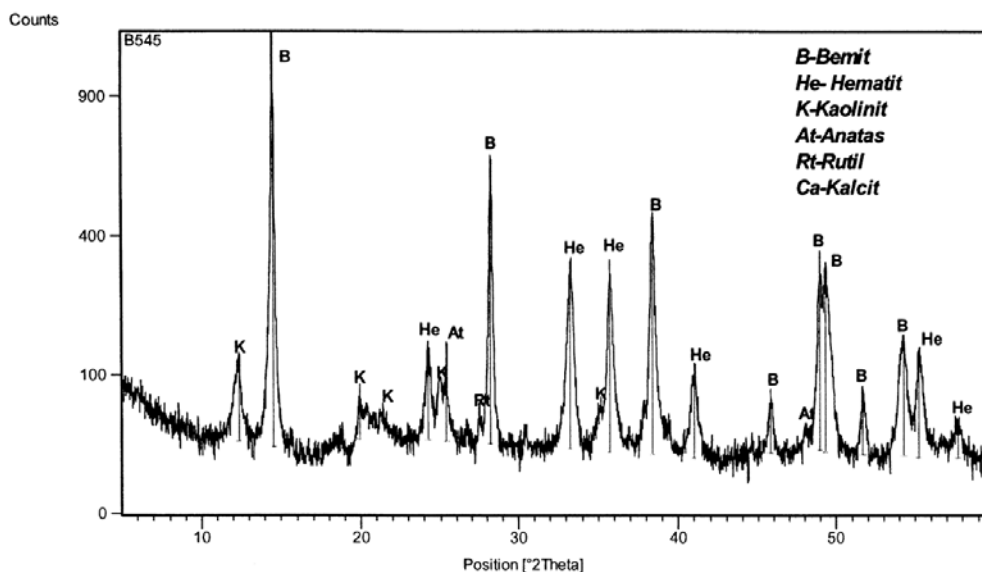


Figure 3. *Diffractogram of the Milici bauxite sample*

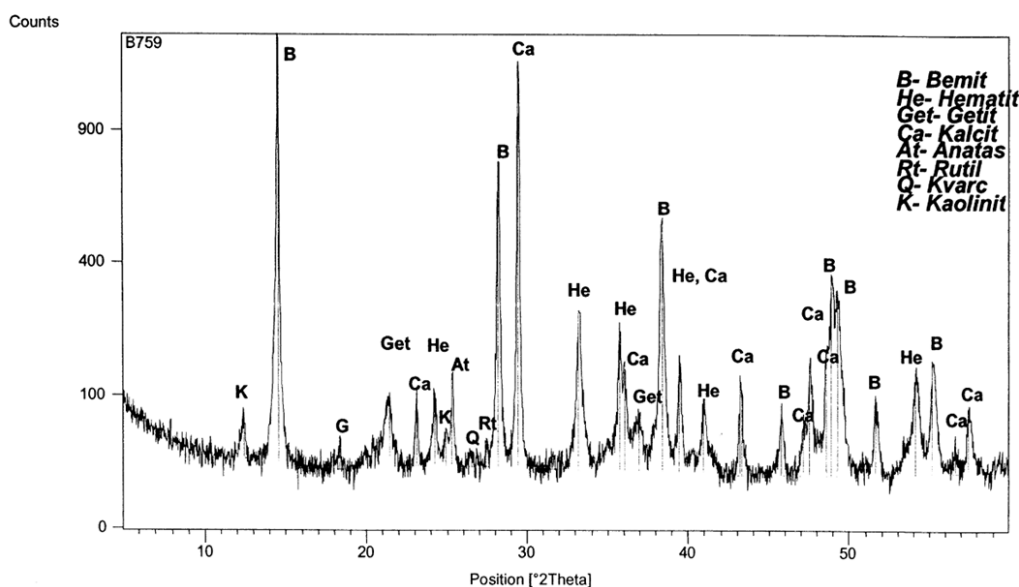


Figure 4. *Diffractogram of the Siroki Brijeg bauxite sample*

The experiments of adsorption of different adsorbates should provide an insight in the changes of the structure of surface active centers formed during the synthesis and alumina calcination.

The adsorption isotherms of the aqueous solution system of methylene blue and alumina Milici and alumina Siroki Brijeg were obtained in the way that 50 mL of the solution of color of different concentrations was placed in the contact with 2.0 g of adsorbent and thermostated at the experiment temperature.

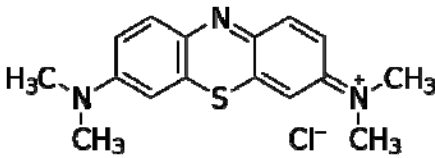
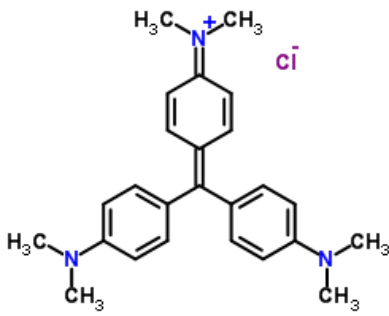
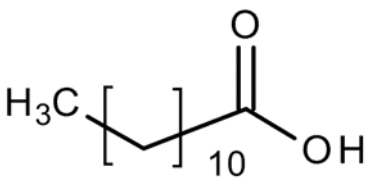
The standard solution of methylene blue color of the mass concentration of 100 mg/L was the one used. The color concentration was determined before

and after the adsorption by the spectrophotometric measurement at the wave length of 663 nm. Measuring was performed on the instrument Lambda 25 UV/VIS Spectrometer PERKIN ELMER. The volumetric method was used to determine the concentrations of the aqueous solution of ammonia with the standard solution of the chloride acid with the concentration of 0.1 mol/L, the concentration of the aqueous solutions of acetic acid with the standard solution of sodium hydroxide with the concentration of 0.1 mol/L, and alcohol solution of the lauric acid with 0.1 mol/L solution of the potassium hydroxide [7, 13].

Table 1. Basic data on adsorbents

	Adsorbent name	Chemical composition		Origin
		Compound	Content, %	
I.	Alumina from the Milici location	Al ₂ O ₃	98.89	„Birac“ Zvornik
		SiO ₂	0.008	
		Fe ₂ O ₃	0.012	
		Na ₂ O _{total}	0.34	
		ZnO	0.0092	
		CaO	0.02	
II.	Alumina from the Siroki Brijeg location	Al ₂ O ₃	97.50	„Birac“ Zvornik
		SiO ₂	0.010	
		Fe ₂ O ₃	0.010	
		ZnO	0.008	
		CaO	0.908	
		TiO ₂	0.557	
		P ₂ O ₅	0.507	
		Na ₂ O	0.500	

Table 2. Basic data on adsorbates

Adsorbate name	Chemical formula/Chemical composition	Origin
1. Nitrogen	N ₂	Sigma-Aldrich Chemie GmbH, Steinheim, Germany
2. Organic color – Methylene blue (MB)	C ₁₆ H ₁₈ ClN ₃ S 3,7-bis(Dimethylamino)-phenothiazine-5-ium chloride 	Merck KgaA, Darmstadt, Germany Used standard aqueous solution with the concentration of 0.1 g/dm ³ utilized to prepare solutions for adsorption by dilution.
3. Organic color – Methyl violet	C ₂₅ H ₃₀ ClN ₃ 4-{Bis[4-(dimethylamino)phenyl]methylene}-N,N-dimethyl-2,5-cyclohexadiene-1-iminiumchloride 	Merck KgaA, Darmstadt, Germany Used standard aqueous solution with the concentration of 0.1 g/dm ³ utilized to prepare solutions for adsorption by dilution.
4. Ammonia	NH ₄ OH	„Zorka“ Sabac Aqueous solution
5. Acetic acid	CH ₃ COOH	„Zorka“ Sabac Aqueous solution
6. Lauric acid	CH ₃ (CH ₂) ₁₀ COOH 	Merck KGaA, Darmstadt, Germany Standard solution in 96% ethanol (Kemika d.d., Zagreb, Croatia) with the concentration of 0.2 mol/L utilized to prepare solutions for adsorption by dilution.

Adsorbent	Specific surface, m ² /g
γ – Al ₂ O ₃ Siroki Brijeg pH 6,5, slow regime of drying	234.60
γ – Al ₂ O ₃ Siroki Brijeg, pH 8, fast regime of drying	112.90
γ – Al ₂ O ₃ Siroki Brijeg, pH 6,5, fast regime of drying	142.80
A Al ₂ O ₃ (0.5% NH ₄ NO ₃)	124.79
B Al ₂ O ₃ (1.0% NH ₄ NO ₃)	120.69
C Al ₂ O ₃ (1.5% NH ₄ NO ₃)	121.80

Freundlich adsorption isotherms of nitrogen are determined on the samples of synthesized alumina, results are presented in Figure 5.

The parameters of adsorption measurements, constants of Freundlich and Langmuir adsorption model for alumina adsorbents used in the study are presented in table 5.

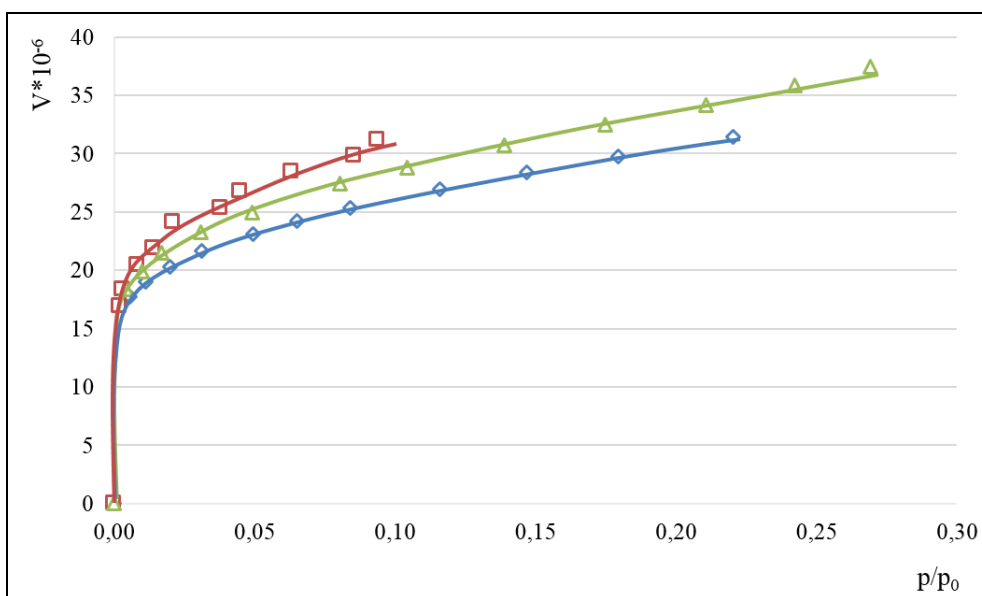


Figure 5. Freundlich adsorption isotherms of nitrogen (at 76K) on the synthesized alumina
 ▲ A (rinsed with 0.5% NH₄NO₃), weight form 5.2842 g, p₀=71.2 cmHg
 ■ B (rinsed with 1.0% NH₄NO₃), weight form 5.1747 g, p₀=71.4 cmHg and
 ◆ C (rinsed with 1.5% NH₄NO₃), weight note 5.5417 g, p₀=70.8 cmHg

Table 5. Parameters of adsorption measurements, constants of Freundlich and Langmuir adsorption model for the synthesized alumina rinsed with NH₄NO₃

Synthesized alumina	Freundlich constant		Plateau height V _A	Number of adsorbed molecules at the plateau	Langmuir constant	Maximum adsorption capacity
	n	K _F			K _L	q _m
	-	-	m ³ /g	molecule	cm ³ /g STP	cm ³ /g STP
A (rinsed with 0.5% NH ₄ NO ₃)	5.84	43.88	2.50 · 10 ⁻⁵	6.72 · 10 ²⁰	101.00	33.03
B (rinsed with 1.0% NH ₄ NO ₃)	6.61	43.03	2.50 · 10 ⁻⁵	6.72 · 10 ²⁰	159.00	31.44
C (rinsed with 1.5% NH ₄ NO ₃)	6.39	38.01	2.50 · 10 ⁻⁵	6.72 · 10 ²⁰	78.25	31.95

The conditions of the synthesis of γ - Al_2O_3 marked with A, B and C alumina (controlled pH and different concentrations of NH_4NO_3) proved not to significantly affect the size of the specific surface. The differences of the values of the specific surface for these three samples are within 3.5% of the experimental error and all three samples adsorb an equal number of nitrogen molecules at the adsorption isotherm plateau (table 5.).

The obtained sample of Siroki Brijeg alumina had the specific surface of $1.3 \text{ m}^2/\text{g}$. By processing of alumina with a highly concentrated solution of NaOH, by heating to the boiling point, we get the

alumina sediment that was along with the controlled pH (6.5-8.0) dried with different regimes [4]. With the slow regime of drying, the sediment is kept for 48 hours at the temperature of 50°C , 48 hours at 100°C , 8 hours each time at the temperatures of 200 and 300°C . Then the sediment is annealed for 12 hours at the temperature of 400°C . This procedure of drying significantly affected the specific surface that increased to $142.8 \text{ m}^2/\text{g}$. Figure 6 presents the adsorption isotherm of ammonium hydroxide on Siroki Brijeg alumina and of aluminium hydroxide from the same location at 293K.

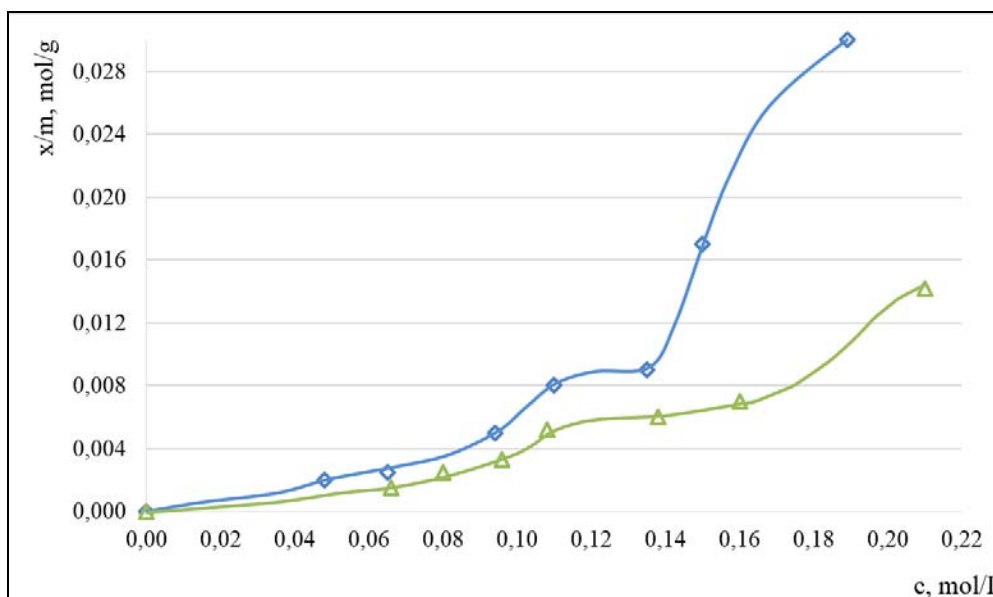


Figure 6. Adsorption isotherms of ammonium hydroxide (temperature of 293 K) on: Aluminium hydroxide (hydrate) Al_2O_3 hydrated alumina

The adsorption of ammonium hydroxide on the γ - $\text{Al}(\text{OH})_3$ hydrate is according to the form of isotherm equal to the isotherm on γ - Al_2O_3 obtained by annealing at 600°C . It is a multilayer physical adsorption. By the Perry model, only cca 0.7 nm^2 of the surface goes to one molecule of ammonium hydroxide, so we can conclude that one molecule of ammonium hydroxide gets bonded to one hydroxyl group with the vertical orientation. Now the postulate of why adsorption occurs in many layers is logi-

cal, because on the polar molecule of ammonia hydroxide building the first adsorption layer there will be built the next layer of molecules. It is assumed that the unit being adsorbed is not the molecule of ammonia, but NH_4^+ ion.

The same form of isotherm both on γ - Al_2O_3 and γ - $\text{Al}(\text{OH})_3$ may possibly confirm the assumption that in both cases it is the surface of adsorbent covered by hydroxyl groups.

Table 6. Parameters of adsorption measurements; constants of Freundlich and Langmuir adsorption model for adsorption of nitrogen on Milici alumina at the temperature of 75 K

Alumina Milici	Freundlich constant		Plateau height VA	Number of adsorbed molecules on the plateau	Langmuir constant	Maximum adsorption capacity
	n	K_F			K_L	q_m
	-		m^3/g	molecule	$\text{cm}^3/\text{g STP}$	$\text{cm}^3/\text{g STP}$
	4.09	31.66	$1.80 \cdot 10^{-5}$	$2.29 \cdot 10^{19}$	29.07	24.57

The behavior of aluminosilicate minerals, as well as of alumina, depends on the characteristics of their surface. The surface electric charge and capacity of ion exchange are directly connected to the active centers that are placed on the sides of the crystal structure, on the basal plains or within the pores. The process of modification with organic surface active matters (SAA) results in a partial neutralization of negative electric charge and it is expected for the obtained organic material to have a bigger efficacy of adsorption of the components from water and air [11,12]. Alumina (Milici and Siroki Brijeg)

is modified by the anion and cation SAA. The concentration of SAA was 4.0 g/L, the procedure is to mix it up with 150 mL SAA and 30 g of alumina. The matter used was cation SAA (pröpagen) and anion SAA (genapol).

Table 7. presents the data on the quantity of the adsorbed ammonia on the alumina of different origin and characteristics determined at the temperature of 293 K.

Table 8. presents adsorption parameters for acetic and lauric acids adsorbed from the solution on alumina at the temperature of 293 K.

Table 7. Height of plateau on Freundlich adsorption isotherm, number of adsorbed molecules of ammonia from the solution on alumina at 293 K

Adsorbent	Temperature of alumina processing, °C	Height of plateau on isotherm, x/m, mol/g	Number of adsorbed molecules of adsorbates by 1g of adsorbent
Alumina Milici	600	I=7.0·10 ⁻⁵ II=19.0·10 ⁻⁵	0.42·10 ²⁰ 1.14·10 ²⁰
Alumina Siroki Brijeg	600	I=0.50·10 ⁻³ II=0.90·10 ⁻³	3.01·10 ²⁰ 5.42·10 ²⁰
Aluminium hydroxide γ-Al(OH) ₃ Siroki Brijeg	105	I=0.002 II=0.006	12.04·10 ²⁰ 36.12·10 ²⁰
SAA Alumina Milici GENAPOL	105	I=0.700·10 ⁻³ II=0.865·10 ⁻³	4.21·10 ²⁰ 5.20·10 ²⁰
SAA Alumina Milici PRÖPAGEN	105	I=0.073·10 ⁻³	0.44·10 ²⁰
A Al ₂ O ₃ synthesized (0.5% NH ₄ NO ₃)	600	I=2.4·10 ⁻⁴	1.45·10 ²⁰

Table 8. Height of plateau on Freundlich adsorption isotherm, number of molecules of acetic and lauric acid adsorbed on 1g of alumina Siroki Brijeg at 293 K

Adsorbate / Adsorbent	Temperature of alumina treatment, °C	Height of plateau on isotherm, x/m, mol/g	Number of adsorbed molecules of adsorbates by 1g of adsorbent
CH ₃ COOH / Al ₂ O ₃	600	I=0.335·10 ⁻³ II=0.440·10 ⁻³	2.02·10 ²⁰ 2.65·10 ²⁰
CH ₃ COOH / PAM Al ₂ O ₃ [genapol]	105	I=0.390·10 ⁻³	2.35·10 ²⁰
C ₁₂ H ₂₄ O ₂ / Al ₂ O ₃	600	I=0.205·10 ⁻³	1.23·10 ²⁰
C ₁₂ H ₂₄ O ₂ / PAM Al ₂ O ₃ [genapol]	105	I=0.300·10 ⁻³	1.80·10 ²⁰

The adsorption of methylene blue color was observed on the Milici alumina and on the modified alumina with the surface active matter pröpagen. Methylene blue color did not get adsorbed on the SAA-alumina. The results of adsorption of methylene blue color on γ-alumina Milici are presented in table 9.

Figures 7 and 8 present adsorption isotherms of organic colors from the aqueous solution on γ-alumina Siroki Brijeg at 293 K annealed at 600°C and methylene blue color on γ-alumina Milici at 293 K.

Table 9. Height of the plateau on Freundlich adsorption isotherm, number of adsorbed molecules of methylene blue color from the solution on the Milici alumina at different temperatures

Adsorbent	Temperature of the alumina treatment, K	Temperature of the adsorption experiment, K	Height of the plateau on the isotherm, x/m , mol/g	Number of adsorbed molecules of adsorbates by 1g of γ -alumina
γ -alumina Milici	873	278	$I=0.230 \cdot 10^{-3}$	$1.38 \cdot 10^{20}$
		293	$I=0.250 \cdot 10^{-3}$	$1.50 \cdot 10^{20}$
		303	$I=0.034 \cdot 10^{-3}$	$0.205 \cdot 10^{20}$

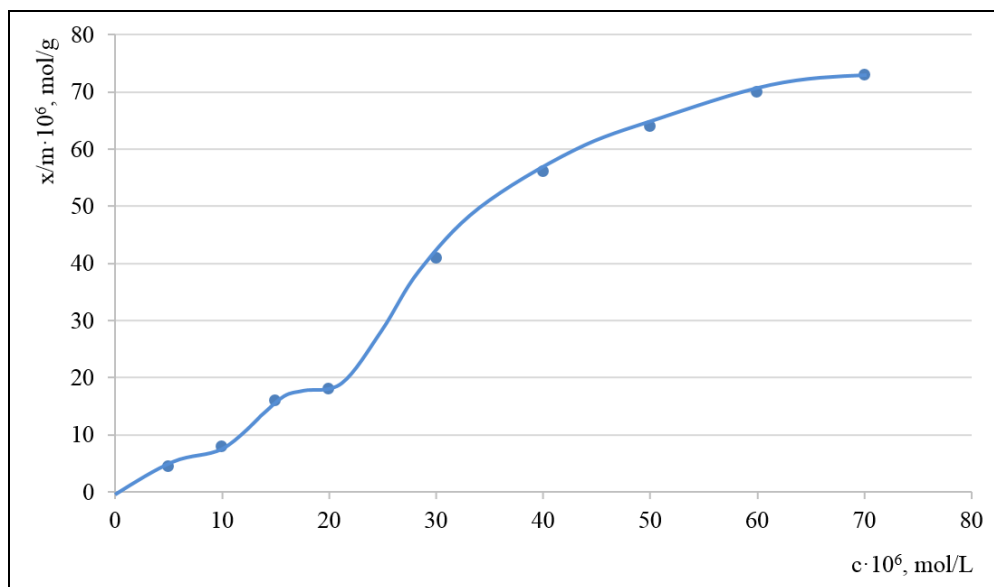


Figure 7. Adsorption isotherm of the organic color of methyl violet on the Siroki Brijeg alumina

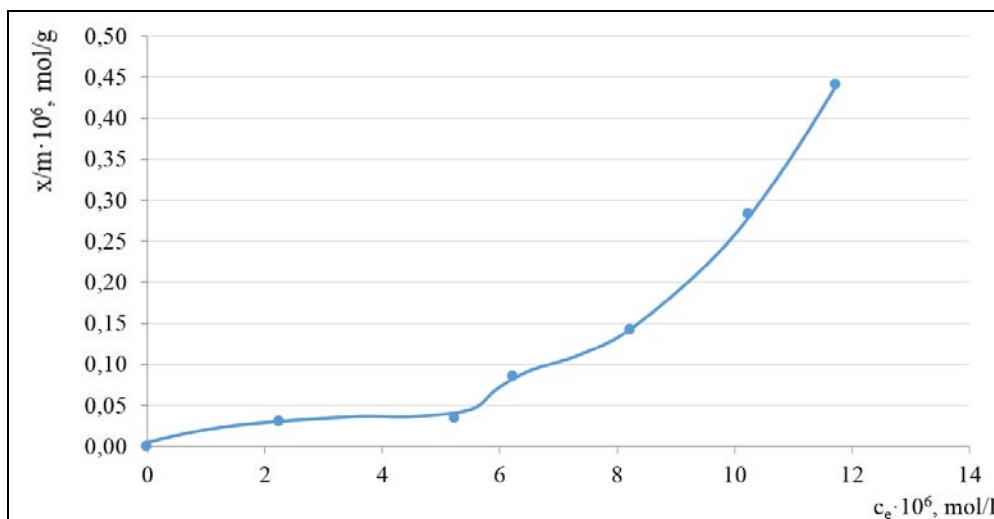


Figure 8. Adsorption isotherm of the organic color of methylene blue color on the Milici alumina

3. CONCLUSIONS

➤ The change of concentration of ammonium nitrate during the alumina synthesis did not significantly affect either the specific surface of alumina (differences within 3.5%) or the form of isotherm. All three samples give the isotherm of

Langmuir type. The number of adsorbed molecules of nitrogen at the temperature of liquid nitrogen is the same and amounts to $6.72 \cdot 10^{20}$ molecules, and the adsorption capacity goes from 33.03 to 31.44 cm^3/g on STP.

➤ In the course of processing $\text{Al}(\text{OH})_3$ from the location of Siroki Brijeg, the change of pH envi-

ronment value proved to affect significantly less (below 1%) the size of specific surface of the obtained alumina, which differs from the regime of fast and slow drying of alumina during which the value of specific surface increased from 1.3 m²/g to even 234 m²/g (cca 200%).

➤ The thermic treatment of alumina provides the samples of big specific surface. For Al(OH)₃ processed by annealing in the temperature interval from 300°C to 600°C, the desorption in the vacuum with the pressure of cca 10⁻³ Pa results in the fall of the value of specific surface from 234 to 131.2 m²/g.

➤ The adsorption of ammonia from the aqueous solution on the alumina of different origin and modified by SAA (table 7) almost does not show any bigger difference. The reason most probably lies in the water, solvent that also gets adsorbed on the alumina surface and can be found in a greater quantity than the adsorbate NH₃ and most probably transfers the surface active centers to hydroxyl groups of equal acidity, so that the ammonia in the form of NH₄⁺ ions gets bonded with the hydroxyl aluminate surfaces.

➤ The adsorption of ammonia is defined as a multilayer physical adsorption.

➤ The adsorption of organic acids was registered on the calcinated and SAA-modified alumina. The results speak in favor of weaker acid and discovered base places on the alumina surface.

➤ The organic colors of methylene blue and methyl violet provide the isotherm of S4 type according to Giles. The adsorption is performed from very much diluted solutions, water is certainly a competitor for the molecules of organic color, for which a higher base level of active centers is more suitable. The adsorption of color did not take place on the SAA modified alumina.

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АДСОРПЦИЈА НА АЛУМИНИ РАЗЛИЧИТОГ ПОРИЈЕКЛА

Сажетак: Као адсорбенси у раду коришћена је алумина добијена из боксита са локалитета Милићи, Широки Бријег и лабораторијски синтетизована алумина добијена таложењем из раствора алуминијум-нитрата и концентрованог амонијака уз контролисан рН раствора током таложења. Добијени талог алумине је жарен четири сата на 873 К. Адсорбати су били трифенилметанске боје, карбоксилне киселине, сирћетна и лауринска, и водени раствор амонијака. Алумина је модификована површински активним материјама (ПАМ), катионактивном ПАМ, триетаноламин-диестерметилсулфат под називом Пропаген и анионактивном ПАМ, Na-со алкилдигликолетерсулфат под називом Генатол. Адсорпцијом азота из гасне фазе на температури течног азота одређена су текстуална својства адсорбенса, а резултати адсорпције киселих и базних адсорбата дали су увид у промјене, односно карактеристике површинских активних центара на којима се адсорпција одвија.

Кључне ријечи: алумина, адсорпција, трифенилметанске боје, карбоксилне киселине, модификација са ПАМ.

