PREPARATION AND APPLICATION OF ALUMINA ADSORBENT FROM WASTE

A. Miskufova[1)*]*, T. Havlik* [1)]*, M. Petranikova* [1)] *, A. Dorakova*[2)]*, K. Koralova*[1)]*, J. Briancin*[3)]
[1)]*Department of Non-Ferrous Metals and Waste Treatment, Technical University of Kosice, Faculty of Metallurgy, Letna 9, Kosice, Slovakia,*
[2)]*Department of Chemistry, Technical University of Kosice, Faculty of Metallurgy, Letna 9, Kosice, Slovakia*
[3)]*Institute of Geotechnics, Slovak Academy of Science, Watsonova 45, Kosice, Slovakia*

[*]*Corresponding author: andrea.miskufova@tuke.sk*

Abstract

The aim of the present work was to study the preparation of active alumina from industrial waste in laboratory scale and also verify the removal efficiency of hexavalent chromium from model water solutions by use of adsorbents prepared by different routes. Aluminum dross fines (-1+ 0 mm) were used for this purpose. In the first stage, fine dross was leached at room (or 40 and 60°C) temperature in NaOH solution. The next step was oriented to preparation of adsorbents by precipitation with three precipitating agent like H_2O_2, NH_4HCO_3 and HCl followed by calcination. The surface area of prepared adsorbent was measured. Further the adsorbents were used for study of the adsorption process and efficiency of Cr(VI) from model solution at different conditions in batch mode. The effects of genesis of adsorbents, surface area, pH, adsorbent dose, sorption time at initial concentration of Cr(VI) 100 mg/l were tested. The highest sorption efficiency was surprisingly achieved with adsorbent which exhibits the lowest surface area.

Keywords: Al dross, hydrometallurgy, alkaline leaching, precipitation, active alumina, Cr(VI) adsorption

1 Introduction

Waste utilization and recycling represents nowadays the general priority in the environmental strategies and ideas of sustainable development not only in EU, but also in other countries of the world. The prevention, waste minimization and application of production methods which produces reduced amount of waste, eco-design of product of consumption etc. are nowadays highlighted. The waste should be considered not as a waste but as a raw material. The recovery of useful materials from waste is a subject of many papers. Hydrometallurgy is a very good tool for recovery of pure substances from raw materials, especially when they are coming from secondary sources and they are of a low grade. The present study deals with utilisation of aluminium dross in preparation of various types of adsorbent based on alumina and their application in Cr(VI) removal from water.The objective of this study was to determine the influence of temperature, redox potential and chloride concentration on the dissolution rates of platinum, palladium and rhodium under atmospheric pressure in NaCl solutions. The dissolution behavior of PGMs has to be precisely known in order to determine suitable oxidizing agents and optimal conditions for laboratory scale leaching experiments using secondary material. The dissolution rates were measured using potentiostatic measurements.

2 Theoretical

Aluminium dross, especially fine dross fractions after mechanical pre-treatment are of low interest, due to low content of aluminium in elemental form (below 3-10 %) and they are mainly landfilled. Due to permanent legislative pressure (Directive 2008/98/ES) on more effective material valorization of wastes and energy saving it is demanding to find also solutions for utilization of problematic fine fraction of aluminium dross. The hydrometallurgical method is probably the most convenient way which enables a complex recovery of valuable substances from Al dross. However, dross is very heterogeneous material and applicability and efficiency of proposed method for individual dross samples should be experimentally investigated and set-up. One of the potential hydrometallurgical methods for aluminum dross treatment represents the alkaline leaching followed by precipitation of aluminium hydroxide. Moreover, the leaching residue free of hazardous matter can be then utilized for example in production of cement or refractory/ceramic materials. Obtained aluminium hydroxide can be used as a precursor for adsorbents production, preparation of abrasives, flocculants etc.). Active alumina can be recovered by calcinations of for example aluminium hydroxides. Active alumina is an excellent material for variety of industrial applications. It is broadly used in removal of dyes, fluoride, phosphates, heavy metals like Pb, Cr, Zn, Co and other substances from waste waters or gases.

Active alumina with high specific surface was prepared according to E.A. El-Katatny by leaching of aluminium dross in caustic soda. Aluminium hydroxide was obtained by use of different precipitating agents (H_2O_2, CO_2, NH_4HCO_3, $(NH_4)_2CO_3$, etc.). The highest surface area of 252 $m^2.g^{-1}$ exhibited γ-aluminium oxide after calcinations of obtained aluminium hydroxide by precipitating agent H_2O_2 at the temperature of 600°C for 3 hrs [1-2]. Other source [3] interprets methods how to recover hydroxides and oxides with high purity (Al_2O_3 and $MgO/MgAl_2O_4$) from aluminium dross (non-metallic residue). After digestion at temperature 98.8 °C in NaOH after 8 hrs the aluminium hydroxide was achieved by precipitation at pH=7 with H_2SO_4. Product on the base of aluminium oxide containing 99.75 % Al_2O_3 was recovered after drying. Experimental results in other work also showed that around 40% of aluminium from dross can by dissolved in NaOH after 2 hours and leaching residue consists mostly of spinel ($MgAl_2O_4$) and non-leached aluminium oxide [4-5]. In general, it is possible to state, that there is a lack of detailed information and experimental work dealing with recovery of active alumina from solution after alkaline leaching of wastes (aluminium dross) and even its application in Cr(VI) removal from waste waters. The active alumina is usually prepared by calcinations of hydroxide precursor most often in the temperature range of 200 – 600 °C in order to obtain high specific surface area. The alumina surface area of 300 $m^2.g^{-1}$ was obtained by calcinations of gibbsite at the temperature 400 °C [6]. The highest efficiency 99 % in removal of Cr(VI) by use of active alumina at pH 4 with dosage 5-10g of adsorbent per liter was achieved. For the comparison, active carbon showed acceptable adsorption effect of Cr(VI) (98 %) at much lower pH (2) and with higher dosage up to 50 g/l [7]. Generally, it is valid assumption that the higher surface area material has (usually more than 100 m^2/g) the more efficient adsorbent will be. But not in all papers dealing with sorption the relationship between the sorption efficiency and surface area or genesis of adsorbent is studied and correlated. The "secret" of adsorption activity of active alumina is meta-stable state (most often γ-alumina), defect and porous structure with fine granularity. The advantage of synthetically prepared adsorbents is that desired properties (for example grain size and porosity) can be more precisely adjusted.

The aim of this work was to investigate various methods for active alumina preparation from waste dross in laboratory scale and at the same time to verify the adsorption efficiency of hexavalent chromium from model water solutions by use of adsorbents with different surface area prepared by different routes.

3 Experimental

3.1 Input materials and methods

For experiments the aluminium dross from primary wrought alloys was used. The dross was crushed, sieved and only fraction *-1 +0 mm* was used for the experiments. The chemical analysis of fine dross fraction was realized by atomic absorption spectrometry AAS (Varian-AA20+) and composition of input dross sample is given in Tab.1. From the mineralogical point of view dross sample contains phases: Alelement, Al_2O_3, SiO_2, AlN, NaCl, $MgAl_2O_4$, KCl, CaF_2, Na_3AlF_6, $CaAl_2O_4$, $MgSiO_3$ and other probable minorities like MgF_2 and others. Mineralogical analysis was realized by use of X-PANalytical X'Pert PRO MRD.

Tab. 1 Chemical composition of Al dross (*-1 +0 mm*)

Element	**Fe**	**$Al_{element}$**	**Al_{total}**	**Si**	**Zn**	**Mg**	**Cu**	**Mn**	**Cl^-**
[w. %]	0.16	12.6	53.49	0.59	0.02	0.16	0.02	0.03	17.59

Dross fraction *-1+ 0 mm* was first leached without foregoing salt removal in NaOH (analytical grade) at the given conditions previously defined [4]. Leaching of dross charges of 20g or 50 g in 500 ml of alkaline solution (10 % NaOH) was realized at room temperature during 2 hrs at 400rpm. The extraction of Al was 35% or 23%, respectively and pH about 13.5. The obtained leaching liquors were used for recovery of active alumina.

3.2 Precipitation and calcinations experiments

Alkaline leaching liquors (6-16 g Al/l) were subjected to precipitation at room temperature by use of agents like H_2O_2 (30 %), NH_4HCO_3 (analytical grade) and HCl (35 %). For the comparison, the pure precipitates were obtained also from synthetic pure Al solution. Synthetic solution was prepared by dissolving of pure Al (analytical grade powder) in NaOH (4 g of Al in 10 % NaOH). pH of prepared alkaline aluminate solution was around 13.5.

At the precipitation experiments the pH of chosen solution (first three cases) was first adapted to value in the range of 12 – 12.5 by use of 10 % HCl in order to avoid excess of used precipitation agents. Subsequently, the precipitation was realized by use of mentioned precipitating agents. The stoichiometric amount of agents were calculated according to proposed precipitation reactions (1-3). The dissolved agents in stoichiometric or multiple amounts (in distilled water) were added into the solutions at 24 °C under stirring (500 rpm) until solution reached pH around 10.

Precipitation by use of NH_4HCO_3

$$NaAlO_2 + 2NH_4HCO_3 = Al(OH)_3 + 2CO_2 + NaOH + 2NH_{3(g)} \quad \Delta G^\circ_{(25^\circ C)} = -90.914 \text{ kJ} \quad (1)$$

Precipitation by use of 30% H_2O_2

$$NaAlO_2 + 2H_2O_2 = Al(OH)_3 + NaOH + O_2(g) \quad \Delta G^\circ_{(25^\circ C)} = -157.492 \text{ kJ} \quad (2)$$

Precipitation by use of HCl

$$NaAlO_2 + 7NaOH + 2HCl = 4NaCl + 1.5Al_2O_3.3H_2O + 2NH_{3(g)} \quad \Delta G^\circ_{(25^\circ C)} = -717.470 \text{ kJ} \quad (3)$$

Obtained precipitates were filtered, dried at 105 °C and weighed. The precipitation efficiency was calculated from differences of aluminum content in solution before and after precipitation. The chosen samples were subjected to chemical analysis (AAS or XRF analysis), thermal gravimetry (TG) and differential thermal analysis (DTA) measurements (Derivatograph C, MOM Budapest, air, heating rate: 10 °C/min). Dried precipitates were further subjected to calcination in laboratory resistance oven at different temperatures 500, 600 °C for 1 or 3 hrs in order to obtain transition aluminas (meta-stable modification of Al_2O_3) with porous structure with higher surface area. Selected calcined samples were finally subjected to surface area measurement (Gemini 2360, Micrometrics, USA).

3.3 Adsorption experiments

The kinetic of sorption process and efficiency of toxic Cr(VI) from model solution by using of prepared adsorbents was investigated. Batch mode experiments of sorption were performed in order to study the effects of genesis of adsorbents, adsorbent dosage, sorption time and pH at the initial concentration of Cr(VI) around 100 mg/l. The temperature of sorption process was fixed in all experiments. In one experiment set, the best adsorbent was chosen and parameter like adsorbent dosage was fixed in order to investigate the influence of pH (4.5, 5.5, 6.5) on adsorption process.

Model Cr(VI) solution was prepared by dissolving of pure K_2CrO_4 (analytical grade) in distilled water. Initial concentration of Cr(VI) in solution was around 100 mg/l (90-130 mg/l). Experiments were realized at 24°C ± 1°C and the volume of model solution was in all experiments fixed to 200 ml. The amount of added adsorbents was 1 or 5 g/l. Desired pH of solution was adjusted by 0.5 M HNO_3 to 6 – 6.5 and the time of sorption was 60 minutes and in one case 24 hrs. Sorbent was added to the solution in glass baker under stirring (750 rpm). In chosen time intervals 2, 5, 10, 15, 30, 45 a 60 min. the liquid samples were taken off, filtered and analysed for Cr(VI) by AAS (Varian-20+).

4 Results and discussion

4.1 Precipitation

The precipitation process was quantitative and efficiencies in all cases were achieved in the range 99-100 %. In the Table 2 are given precipitation route, conditions of individual adsorbent preparation (genesis) and surface area values.

Tab. 2 Precipitation route and surface area of prepared adsorbents

Sorbent	Precipitation route	Surface area [m^2/g]
MK 271	Precipitation from model solution with 30% H_2O_2 without previous pH modification; calcined at 600°C/3 hrs.	33.7657
MK 273	Precipitation from leaching liquor (10% NaOH, 20g dross, 60°C) with 30% H_2O_2 without previous pH modification; calcined at 600°C/3 hrs.	28.4785
MK 274	Precipitation from model solution with NH_4HCO_3 with pH modification to 12.5 by HCl; calcined at 600°C/3 hrs.	54.0220
MK 275	Precipitation from leaching liquor (10%NaOH, 20g dross, 60°C) with NH_4HCO_3 with pH modification to 12.5 by HCl, calcined at 600°C/3 hrs.	54.6912
MK(AD) 291.1	Precipitation from leaching liquor (10%NaOH, 50g dross, 20°C) with NH_4HCO_3 with pH modification to 12.5 by HCl, calcined at 500°C/1h.	22.214
MK 291.1R	Recalcined sample 291.1 after natural ageing on air. Prepared adsorbent MK 291.1 after long-therm natural ageing at ambient temperature and second calcination at 500 °C/1 h.	41.0300
AD 2	Precipitation from leaching liquor (10%NaOH,20g dross, 40°C) with HCl; calcined at 500°C/1 h.	133.6649
AD 3	Precipitation from leaching liquor (10%NaOH,20g dross,40°C) with NH_4HCO_3 without previous pH adjusting,; calcined at 500°C/1 hour	101.4916

It follows from Tab. 2 that surface area of individual adsorbents in some cases differs significantly. The surface area of adsorbents is influenced significantly by calcination temperature. Temperature of 600 °C it seems to be not suitable from surface area point of view. The positive fact is, that adsorbent prepared from model solution and leaching liquor and calcined at 600 °C for 3 hours does not differ so much (for H_2O_2 28.47 and 33.76 m^2/g, for NH_4HCO_3 54.02 and 54.69 m^2/g). The interesting is that adsorbent prepared by the same route from leaching liquor (MK 275 and MK(AD) 291.1) after calcination at lower temperature of 500 °C has lower surface area. At the same time, the adsorbent prepared with H_2O_2 showed much lower surface area in the contrary to published results [1-2]. This phenomenon could be caused by specific input materials and methods of adsorbent formation during precipitation. It seems that very small changes in route have significant influence on quality of precipitate. The highest surface area 133 m^2/g in these experiments was achieved by precipitation (neutralization) agent HCl. The precipitates were amorphous and by use of X-ray diffraction phase analysis it was not possible exactly identify the form of precipitated hydroxide. That is why the thermal analysis (DTA and TG) was used. Results of thermal analysis of „NH_4HCO_3“ precipitated sample from leaching liquor is shown in Fig.1a. TG analysis indicated that sample contains around 9 % of free water and decomposition of precipitate occurred in three steps (endo peaks at DTA curve). This suggested the presence of freshly precipitated aluminium hydroxide – gibbsite. In the first step (at 240 °C) the hydroxide loose 1.5 molecules of water and at 300 °C about 1.2 molecules of water and broad peak in the range 550 °C - 630 °C indicate the loose of the rest of 0.3 molecules of water. The position of third peak it is quite disputable due to fact that in synthetically prepared gibbsite it usually occur at around 500 °C. The decomposition of natural gibbsite shows maximum peak temperatures at around 275 °C, 311 °C and 505 °C, respectively

[8]. Together, the sample weight lost of bound water represents around 35 % (theoretically in pure $Al(OH)_3$ it is 36 %). It could point out the presence of some impurities based on sodium compounds. The sample precipitated with H_2O_2 showed the similar temperatures of decomposition 165 °C and 300 °C in DTA curves.

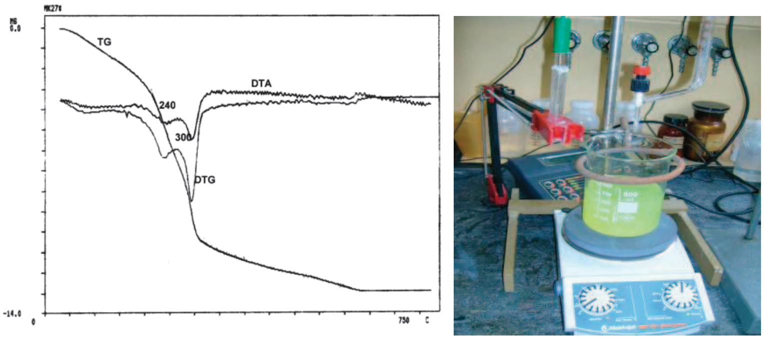

Fig. 1 a) Thermal analysis (TG, DTA and DTG curves) of precipitate by use of NH_4HCO_3 as precipitating agent from leaching liquor; heating rate: 10 °C/min in air, charge 28.9 mg; b) experimental setup at adsorption experiment

4.2 Adsorptions

Precipitates were calcined at 500 or 600 °C for 1 or 3 hours and subsequently used for adsorption of Cr(VI) from model solution. Experimental setup is shown in Fig. 1b. The impurities content of chosen calcined sample MK 291.1 prepared from leaching liquor with NH_4HCO_3 precipitation agent measured by use of XRF analyzator (Spectrometer Niton XL3 Goldd) is given in Tab. 3.

Tab. 3 The impurities content of calcined alumina adsorbent MK 291.1

Element	**Fe**	**Na***	**K**	**Si**	**Ca**	**Mg**	**Cl^-**
[w. %]	0	7.6	0.032	0.1375	0.158	0	0

* *measured by AAS*

It is shown from Table 3 that except of Al_2O_3 the adsorbent contains small amount of Si and Ca and for higher sodium could be responsible phases like sodium carbonate Na_2CO_3 (natrite or hydrated thermonatrite) and dawsonite - $NaAl(OH)_2CO_3$. These compounds could be created during the precipitation process from solution of $NaAlO_2$ with NH_4HCO_3 according to reaction 4 and 5 [9]. These compounds were indicated by XRD analysis of naturally long-period aged calcined adsorbent (Fig.4a).

$$NaAlO_2 + NH_4HCO_3 = NaAl(OH)_2CO_3 + NH_3(g) \qquad \Delta G^\circ_{298} = -63.203 \text{ kJ} \qquad (4)$$

$$2NaAlO_2 + CO_2(a) + 3H_2O = Na_2CO_3 + 2Al(OH)_3 \qquad \Delta G^\circ_{298} = -88.978 \text{ kJ} \qquad (5)$$

From XRD phase analysis also followed that calcined adsorbent MK(AD) 291.1 could contain at least two modifications of alumina, γ-Al_2O_3 and eta-Al_2O_3. Moreover, XRD analysis of re-calcined adsorbent AD 291.1R also showed possible presence of κ-alumina. Aluminium hydroxide undergoes decomposition and phase transformation in few steps when it is heated up to 1200 °C: $Al(OH)_3 \rightarrow AlOOH \rightarrow \gamma\text{-}Al_2O_3 \rightarrow \alpha\text{-}Al_2O_3$. However aluminium oxide is able to create also other allotropic modifications, for example delta, eta, theta, chi, kappa etc., but the most stable under normal condition are α and γ. The occurrence of individual phases in calcinations product depends on character of initial precursors, materials structure, impurities content, genesis of precipitate, the conditions and temperature of calcination process and other factors. By the variation of calcination temperatures (usually from 300 to 600 °C) it is possible to obtain active forms of alumina, which are suitable for adsorption process.

Kinetic curves of adsorption process at pH = 6.5 and initial concentration of Cr(VI) 120 mg/l by use of various types of adsorbents in amount of 1g per liter at room temperature it is shown in Fig. 2a. Based on results shown in Fig. 2a in the next step the best adsorbent MK 291.1 was chosen in order to study the influence of pH on adsorption process. Results are given in Fig. 2b.

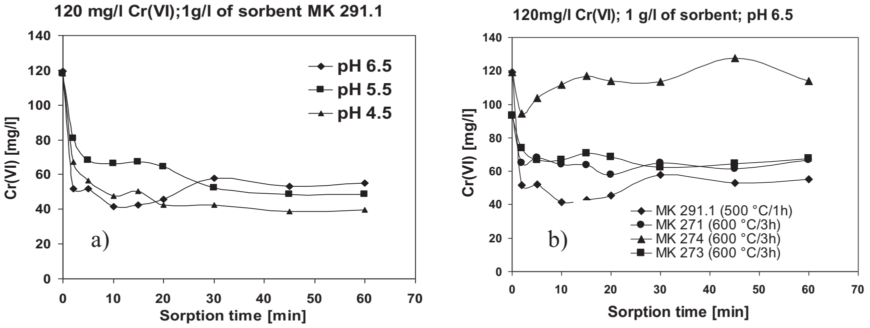

Fig. 2 a) Adsorption process of Cr(VI) from model solution for various adsorbents;
b) Influence of pH on adsorption process of Cr(VI) from model solution for adsorbent MK 291.1

In further experiments also other types of adsorbents were tested which were prepared by precipitation from leaching liquor by use of only HCl and by use of NH_4HCO_3 without pH modification before precipitation. Those adsorbents had the highest surface area and sorption process realized at optimized pH 4.5 with dosage of adsorbent 1g/l. Results are presented in Fig.3a. For the comparision in Fig. 3a is given also adsorbent MK(AD)291.1 which was suggested as the best adsorbent based on previous results. From the results it is obvious that sorbent MK(AD) 291.1 has a highest sorption capacity. In Fig.3b it is shown the kinetic of sorption process of MK(AD) 291.1 at the higher adsorbent dosage 5g/l. The adsorption process in this case was significantly improved and almost all Cr(VI) (more than 130 mg Cr(VI)/l) was removed within the 10 minutes). Adsorption process by use of the best adsorbent at pH 4.5 with the initial concentration 50 mg/l of Cr(VI) at higher dosage of adsorbent 5g/l was subjected to 24-hours sorption test in order to study the equilibrium of Cr(VI) adsorption. The efficiency of adsorption after 24 hours was 98.3 % with Cr(VI) concentration in final solution 1.53 mg/l. The increasing dosage has positive effect on adsorption process. Adsorption process was very rapid in general and equilibrium in this case was achieved approximately after 3 hours.

After 24 hours test adsorption and natural long-term ageing of used adsorbent MK(AD) 291.1 (sample AD 24) the XRD analysis (Fig. 4b) confirmed that adsorbent with adsorbed Cr(VI) contains the chromium oxide CrO_3 and at the same time this oxide preserved the structure of transition aluminas (γ-alumina, eta-alumina or probably also κ-alumina). That adsorbent was not active anymore and did not accept water into the structure even after long period. This is in the contrary to the adsorbent AD 291.1 without adsorbed Cr(VI) which was exposed to air in the same manner was hydrated and crystals of gibbsite, bayerite and norstrandite occurred, Fig. 4c.

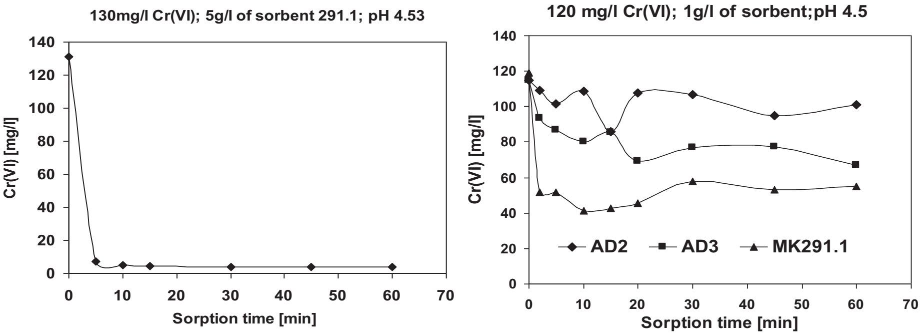

Fig. 3 a) adsorption of Cr(VI) from model solution for various adsorbents; b) adsorption kinetic for adsorbent MK 291.1 at pH 4.5 at sorbent dose 5g/l

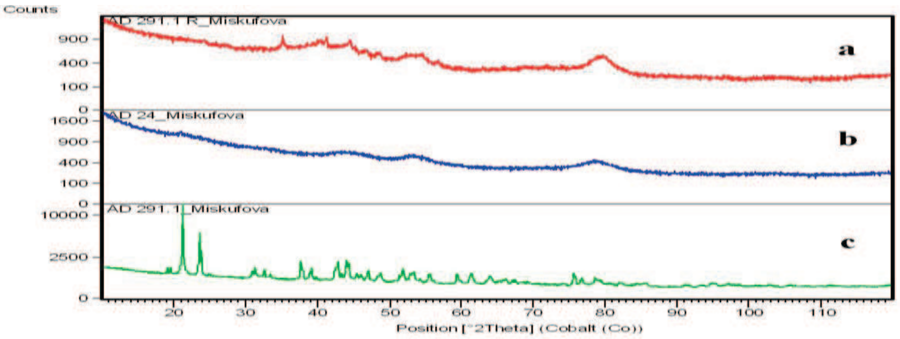

Fig. 4 XRD patterns of :a) calcined adsorbent AD 291.1 (R) at 500 °C not used for adsorbed; b) after adsorption during 24 hours by use of best adsorbent AD 291.1 at pH 4.5 at dosage of adsorbent 5g/l and after long-term ageing, c) long-term aged adsorbent AD 291.1

5 Conclusion

By alkaline leaching of aluminum dross and precipitation with various agents (NH_4HCO_3, H_2O_2, HCl) the amorphous gibbsite precipitate were obtained. After calcination of precipitates at 500-600 °C the active aluminas were achieved and used for hexavalent chromium removal from model solution. Adsorption process was very rapid and equilibrium was achieved approximately after 3-6 hours. Adsorption process was significantly influenced by pH, sorbent dosage and genesis (precipitation route). The highest efficiency of Cr(VI) removal in the range of pH 4.5-5.5 was achieved. The maximum removal of Cr(VI) 98.3 % after 24 hours was reached by use of adsorbent (in amount of 5g/l) recovered from waste aluminate leaching liquor by precipitating agent NH_4HCO_3 and subsequent calcining at 500°C for one hour. The interesting phenomenon in adsorption process was found out the influence of surface area of adsorbents. By use of adsorbent with the lowest surface area 22.214 m^2/g surprisingly the best removal efficiency was achieved and adsorbent exhibits the highest sorption capacity of 19.74 mg/g. Experiments showed that an effective adsorbent can be prepared from industrial waste. But on the other hand, the influence of surface area and other aspects on sorption process as well as mechanism should be studied more precisely. From results also followed that sorption activity of adsorbent is influenced probably by other factors like preparation genesis, impurities content and other parameters, not only by surface area. Presence of sodium (sodium carbonate) or chloride compounds probably positively influence the hydrophilic character of alumina surface and finally the sorption activity and binding capability through complexation via -OH groups [10].

Acknowledgements
This work was supported by Ministry of Education of the Slovak republic under Grant MS SR 1/0123/11, then with financial support of project No. APVV-20-013405. This contribution is also the result of the project implementation Research excellence centre on earth sources, extraction and treatment supported by the Research & Development Operational Program funded by the ERDF, ITMS number: 26220120017.

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