Processing Research

Adsorption of Cr(III) from aqueous media on zeolite A prepared from fused fly ash by hydrothermal synthesis

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In this work, the potential application of using zeolite A synthesized from fly ash to remove Cr (III) from aqueous solutions was studied. Zeolite A was synthesized from acid-washed fly ash by alkali fusion followed by hydrothermal treatment. The acid washing removed Ca and Fe from the fly ash, which allowed synthesis of the required zeolite. The acid-washed fly ash and NaOH were mixed at a mass ratio of 1:1.2 and heated at various temperatures. The hydrothermal zeolite synthesis was carried out at 600 °C temperature and the product characterized by X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). The zeolite achieved maximum adsorption capacity of Cr (III) attaining equilibrium within 30 min at pH 4.0. The adsorption isotherm for Cr (III) on this zeolite A was tested and showed that the adsorption data of Cr (III) were fitted best by a Langmuir isotherm.

Keywords: Fly ash, Acid leaching, Hydrothermal synthesis, Zeolite A, Cr (III) removal.

Introduction

Fly ash is a fine solid residue generated in coal-fired thermal and thermal-power stations (TPSs). A major problem for all coal power plants is the disposal of fly ash and bottom ash, due to environmental issues such as air pollution and groundwater contamination due to leaching of toxic metals from the ashes [1]. Since the fly ash from the TPSs is disposed of in large quantities, there is considerable interest in utilizing fly ash from the materials, environmental and ceramic scientists [2-4]. Therefore, finding an effective and economical way to use fly ash is an essential task for scientists.

One practical use of coal fly ash is its conversion into synthetic zeolites [5]. These are usually made by adjusting the fly ash's chemical composition to the desired zeolite composition and thermally processing the mixture. For this reason, synthetic zeolites are of relatively pure mineral composition [5]. Many review papers summarized the preparation of various types of zeolites from the fly ash and their application [5-10]. From the previous reports, synthesized zeolites from the fly ash depend on fly ash type, synthesizing method etc. [5, 8, 9].

Coal-fired thermal power and power stations produce over 90% of the electricity in Mongolia. The three main thermal power stations are placed in Ulaanbaatar, and the largest one mostly uses coal from the Baganuur deposit. More than 300,000 tons of fly ash is being generated annually in this power station, and only a small portion of this fly ash is used for the production of building materials such as concrete and cement. The chemical composition of fly ash indicates a high content of calcium oxide and a lower aluminum oxide content, making it a class C fly ash [11].

There have been previous attempts to prepare zeolitic compounds from this fly ash by adding aluminum oxide to adjust the chemical composition [12, 13]. It was suggested that whilst the calcium present in the fly ash does not directly interfere in zeolite formation, it does tend to produce a mix of various zeolitic compounds rather than a single phase. In our previous research, we reported a preliminary experiment using acid treatment to reduce the calcium and iron content [14] and showed that fly ash pretreated with acid preferentially formed zeolite A, which has a high CEC. However, there remain other crystalline phases such as mullite and quartz which are undesirable because of the slow dissolution of these crystalline compounds. One method to improve the homogenization of the reaction mixture is a preliminary fusion step with sodium hydroxide to facilitate the decomposition of the crystalline compounds present [15]. In that case, relatively pure zeolite can be obtained [15]. However, it is crucial to control the fly ash composition for fusion to facilitate the desired zeolite from the fused glass mixture.

Zeolites synthesized from fly ash can be used for

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removing heavy metals from wastewater due to a high cation exchange capacity (CEC) [15-23]. Heavy metal ions can contaminate water resources in many different ways, including industrial processes (tanneries, paint manufacture, fertilizer manufacture, and use), pesticides, animal manures, sewage sludge, wastewater irrigation, coal combustion residues, atmospheric deposition, etc. [24]. Of the various heavy metals, trivalent and hexavalent chromium is considered one of the significant sources of environmental pollution. Although Cr (VI) in its hexavalent form is toxic and carcinogenic, chromium in its trivalent state Cr (III) in trace amounts is a crucial nutrient for plant and animal metabolism [25]. However, Cr (III) can also be potentially hazardous, especially in the aquatic environment and is considered to be potentially toxic because it is a competitive inhibitor of many cellular processes [26]. It can also be oxidized to toxic hexavalent chromium during the processing of wastewater. Thus, the removal of Cr (III) from wastewater is an essential topic in environmental science.

A few reports of Cr (III) adsorption on a synthetic zeolite have been published [27, 28]. Moreover, the types of zeolite synthesized from fly ash depend on the fly ash's chemical and mineralogical composition and the synthesizing method. Therefore, the zeolite synthesizing method designed for one type of fly ash cannot be used directly for other fly ash types. It requires modification depending on the fly ash's chemical and mineralogical composition. For the sustainable use of fly ash, it is vital to find the best way for zeolite synthesis and determine whether the synthesized zeolite acts as an adsorbent for hazardous metals, including Cr (III). This research aimed to extend knowledge on the preparation of zeolite from fly ash and elucidation of aqueous Cr (III) adsorption behavior on the prepared zeolite A. The influence of contact time, pH, chromium (III) concentration and adsorption kinetics of the chromium (III) by the synthesized zeolite were also studied.

Materials and Methods

Materials

A fly ash sample obtained from the 4th thermal power plant in Ulaanbaatar, Mongolia, was passed through a 100-mesh sieve before use. The chemical compositions of the raw and acid-washed fly ashes, determined by X-ray fluorescence (Rigaku, Primini), are listed in Table 1. The average particle size (Shimadzu, SALD-2100) of the fly ash was 27.91 μ m, and the BET specific surface area calculated from N₂ isotherms (Belsorp, mini) was 2.75 m²/g.

Acid washing of fly ash

The raw fly ash was pretreated by acid-washing to remove the calcium and iron using a 10% HCl solution in the mass ratio of 10:1 *liquid: solid*. The mixture was magnetically stirred at 80 °C for 1 h at 250 rpm. The fly ash was then filtered and washed with distilled water repeatedly and dried at 100 °C for 24 h.

It should be noted that acid leaching not only reduces the calcium and iron contents but also removes some of the aluminas from the glassy component of the fly ash. The loss on ignition also increases considerably, which indicates the appearance of strongly bonded water in the acid-washed fly ash microstructure.

The crystalline phases of the fly ash and newly synthesized zeolite were determined using X-ray diffraction (XRD) (Maxima X7000 Shimadzu, Japan).

Zeolite synthesis

The alkali fusion temperature of the acid-treated fly ash is an important factor in the formation of zeolite. The alkali-fusion process generates active Si and Al species in the synthesis mixture and removes the crystalline quartz and mullite phases [15, 29]. The alkali fusion was carried out by treating acid-treated fly ash with sodium hydroxide in a 1:1.2 weight ratio at 600, 700, and 800 °C for 2 h. The fused fly ash powder was added to 50 mL of water and agitated for 2 h at 60 °C, after which the solid was separated by filtration. The filtrate was then mixed with an aluminate solution (0.5 g Al metal with 20 mL 2M NaOH) in a Teflon vessel and agitated at 60 °C for 1h at 300 rpm. The zeolite syntheses were carried out on this mixture in a Teflon-lined 100 mL hydrothermal reaction vessel at 100 °C for 24 h. The reaction product was then filtered, washed and dried at 105 °C overnight. The experimental scheme for the zeolite synthesis is shown in Fig. 1. Initially, the cation exchange capacity (CEC) of the synthesized zeolite was determined by ammonium (NH_4^+) removal efficiency [30]. Then the synthesizing method which gave the highest CEC was used for further experiments.

Batch experiments for Cr(III) removal

All the adsorption experiments were conducted in 100 mL glass flasks containing 50 mL of solution at room temperature in a stirrer with continuous stirring at 150 rpm until equilibrium was attained. Stock solutions of 1,000 mg/L Cr (III) was prepared from analytical grade CrCl₃6H₂O and diluted to the desired concentrations by adding distilled water. After fixed time

 Table 1. Chemical composition of raw and HCl treated fly ashes, (weight %)

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	K ₂ O	TiO ₂	MgO	P_2O_5	SrO	BaO	LOI
Raw fly ash (%)	55.20	14.15	10.55	15.00	1.31	0.25	1.56	0.004	0.25	0.001	1.71
HCl treated fly ash (%)	67.53	7.75	7.85	6.96	1.39	0.48	0.97	0.03	-	0.14	6.23



Fig. 1. Experimental scheme for the zeolite synthesis.

intervals, the solutions containing 0.5 g of the zeolite, prepared as above, were filtered through a membrane filter (0.45-µm in size) and the residual Cr (III) in the filtrates were determined with a Unico spectrophotometer (UV-2102PCS). The Cr (III) was first converted into the hexavalent form by oxidation with ammonium persulfate at elevated temperature and acidic conditions (pH \approx 2) and the Cr (VI) concentration was determined at 543 nm by the 1,5-dephenyl-carbazide method [29]. The amounts of Cr (III) exchanged by the zeolite (mg/g) and the percentage Cr (III) removal were calculated by Eqs. (1) and (2).

Amount of exchanged Cr (III) =
$$\frac{C_o - C_e}{W} \times V$$
 (1)

Cr (III) removal (%) =
$$\frac{C_o - C_e}{C_o} \times 100$$
 (%) (2)

where C_o and C_e are the initial and equilibrium Cr (III) concentrations of the test solution (mg/L), (V) is the test solution at volume (L), and W is the amount of adsorbent (g).

Batch experiments were performed in duplicate and the average value used.

Results and Discussion

XRD characterization of fly ash

The XRD pattern of fly ash (Fig. 2) shows the major crystalline phase to be quartz (SiO₂), small amounts of microcline (KAlSi₃O₈), hematite (Fe₂O₃), magnetite (Fe₃O₄) and monocalcium aluminate (CaAl₂O₄).

Acid leaching could be caused the dissolution of crystalline microcline and monocalcium aluminate along with the amorphous part of the fly ash. However, the content of the potassium wasn't reduced by acid leaching. Therefore, it can be suggested that acid leaching dissolves the amorphous part of the fly ash but not the microcline. The fly ash obtained after acid leaching can be considered a medium level of calciumcontaining fly ash, though, the aluminum content is lower than that of the average fly ash.



Fig. 2. X-ray diffraction patterns of raw and acid pretreated fly ash.

Characterization of adsorbents

Fig. 3 shows the XRD pattern of synthesized zeolite. The zeolite A (Na-A) and small amounts of sodalite were assigned as the major and minor crystalline phases in the synthesized zeolite. In contrast to previous research on unwashed fly ash [11], relatively pure zeolite A with a small amount of sodalite was made from the high calcium and iron-containing fly ash. The presence of sodalite likely to be resulted by crystallization of it within zeolite A core due to increased pressure, as it described by [31]. Therefore, optimization of the zeolite synthesizing condition, possibly by reducing the crystallization time and temperature may decrease the content of the sodalite, though, a transformation of an initially formed sodalite into zeolite NaP has been observed [32].

Fig. 4 shows the SEM micrographs and corresponding EDS spectra of the acid-washed fly ash and synthesized zeolite. Fig. 4(a) shows the acid-washed fly ash is composed predominantly of polydisperse spheres with smooth surfaces, while the EDS data (Fig. 4(b)) of an ash sphere shows it to be an iron aluminosilicate glass.

Fig. 4(c) shows that the synthesized Na-A zeolite



Fig. 3. X-ray diffraction pattern of synthesized zeolite.

crystal is predominantly equiaxed cubic particles, which is similar to zeolite A crystals described in previous references. The EDS results indicate that the Na-A zeolite contains 8.0% Na, 13.55% Al and 13.77% Si (Fig. 4(d)). This is equivalent to a molar ratio of n(Na): n(Al): n(Si) = 0.34:0.50:0.49, close to the theoretical ratio of Si/Al = 1.

Although the adsorbent size is a potentially significant parameter in the kinetic test, this factor could not be examined due to the narrow size range of product zeolite particles.

Effect of alkali-fusion temperature

Fig. 5 shows the effect of the fusion temperature at 600, 700 and 800 °C after 2 h at a NaOH/fly ash ratio of 1.2:1 on the ammonium removal efficiency of the crystallized zeolite. The ammonium removal method was used as it is the most commonly used method for determining the CEC [33]. This parameter was chosen as the criterion for determining the optimum fusion temperature. In contrast to our previous study, the CEC of the newly synthesized zeolite (1.01 mg/g) was double that prepared without preliminary fusion by hydrothermal treatment [14]. It is clear that the predominantly formed zeolitic phases show the main influence on ammonium removal efficiency.

The results in Fig. 5 show that the most suitable fusion temperature is 600 °C. With the increase in temperature from 600 to 800 °C, the ammonium removal efficiency decreased from 48.5% to 15.9%. Our findings in good agreement with previous results reported that the increase of zeolite yield was observed from temperatures above the melting point of NaOH



Fig. 4. SEM images and EDS spectra of (a) and (b), acid washed fly ash, (c) and (d), synthesized zeolite.



Fig. 5. Effects of the alkali-fusion temperature on removal efficiency of ammonium at the condition of 1.2:1. NaOH/fly ash, fusion for 2 h, and hydrothermal reaction for 24 h at 100 °C ($C_0 = 10 \text{ mg NH}_4^+$ -N/L; pH = 8.0; T = 25 °C).

(318 °C) to a maximum crystallinity at 600 °C [34]. With the increased fusion temperature, the synthesized zeolite crystallinity decreases due to glass phase formation. Our preliminary study showed that an alkali fusion temperature of 500 °C was not sufficient for complete reaction. The CEC of the zeolite prepared at 500 °C was too low to consider further study.

Effect of contact time Cr (III) ion adsorption

The effect of time on Cr (III) sorption onto the synthetic zeolite is shown in Fig. 6. Fig. 6 shows the amount of Cr (III) adsorbed increased dramatically within the first 30 min, at which time 0.969 mg/g was adsorbed, representing almost complete removal of Cr (III). This figure indicates that adsorption was very rapid, with equilibrium achieved within 60 min.

The initial solution concentration and adsorbent dose show the main influence on the adsorbed amount of Cr (III) and removal efficiency.

Effect of pH

The effect of pH on the adsorption of Cr (III) ions by the zeolite at pH 2.0-8.69 is shown in Fig. 7.

Fig. 7 shows that as the initial pH of the solution is increased from 2.0 to 6.0, the chromium (III) uptake amount decreases from 0.985 to 0.884 mg/g, the maximum adsorption occurring at pH 2.0, then decreasing with increasing pH to a minimum value of 0.412 mg/g at pH 8.69. Thus, for chromium uptake by the synthesized zeolite, the optimal pH is in the range 2.0 to 6.0. Above this pH the adsorption decreased rapidly. Thus, for chromium removal, the optimal pH is from 2.0 to 6.0. When the pH is <4.0, Cr^{3+} is the dominant species, whereas at pH >4.0 (up to approximately 6.5), $Cr(OH)^{2+}$ predominates. Above this pH, Cr (III) removal will occur mainly by precipitation rather than absorption [35].



Fig. 6. Effect of contact time on the removal of Cr (III) by the synthesized zeolite ($C_0 = 10 \text{ mg/L } \text{Cr}^{3+}$ solution; adsorbent dose = 0.5 g; room temperature, pH 4.1).



Fig. 7. Effect of initial pH on the removal efficiency ($C_0=10 \text{ mg/}$ L Cr³⁺ solution; adsorbent dose = 0.5 g; room temperature).

Traditionally, Cr (III) was removed with precipitation of Cr (OH)₃ by adding lime water to increase the pH. The present research shows that Cr (III) removal can be achieved at lower pH than with the traditional precipitation method.

Effect of initial chromium (III) concentration

Cr (III) removal by the synthesized zeolite was studied for a range of initial Cr (III) concentrations from 11.3-510 mg/L. The results (Fig. 8) indicate that the initial Cr (III) concentration had a significant influence on both the amount of removal and the removal efficiency of chromium.

The US Environmental Protection Agency recommended that the Cr (III) concentration in the drinking water be 0.1 mg/L [33]. The allows discharge limit of Cr (III) from 0.05 mg/L (in surface waters) to 2.0 mg/L (in sewers).

Fig. 8 shows with an increase in the initial Cr (III) concentration from 11.3 to 510 mg/L, the chromium removal efficiency decreased from 91.1% to 71.4% for the synthesized zeolite. With increasing initial Cr (III) concentration, the chromium removal capacity of the synthesized zeolite increased, reaching a maximum value at 35.8 mg/g at an initial Cr (III) concentration of 510 mg/L. These results indicate that the adsorbents have limited exchange sites, and removal becomes restricted when these sites are saturated. Obviously, the increase in removal capacity by increasing the driving force generated by the concentration gradient. At the same time, the latter increases with increasing initial chromium concentration.

Table 2 summarizes the final chromium concentration and removal efficiency of the prepared zeolites after the equilibrium condition.

From Table 2, it can be seen that when the initial chromium concentration is 11 mg/L the residual chromium in the water fulfills the standard in sewages shown in the reference [38] i.e., Cr (III) < 10 mg/L. Higher levels require further treatment or using a larger mass of sorbent to reduce the final chromium concentration in the water medium. At an initial Cr (III) concentration of 370 mg/L, the optimum values for both



Fig. 8. Effect of initial Cr (III) concentration on the chromium removal efficiency and removal capacity by the synthesized zeolite (adsorbent dosage = 0.5 g; pH = 4.1; room temperature).

Table 2. The chromium final concentration and removal efficiency.

chromium removal efficiency and removal capacity are 86.5% and 26.25 mg/g. Although, there wasn't complete adsorption, this value could be the optimal condition of removal based on both chromium concentration and adsorbed amount.

Another important factor which wasn't studied in this research is the regeneration of the used zeolite. If occurs a full regeneration of the zeolite used at pH 4, it will overcome relatively poor adsorption of this zeolite. Such research is in the further study the goal of this zeolite.

Adsorption isotherm study

The adsorption data were analyzed in terms of the Langmuir isotherm, Freundlich and Dubinin-Raduskhevich (D-R) isothermal models as they are the most commonly used isotherms to describe solid-liquid adsorption systems [38].

Fig. 9 shows the equilibrium isotherm data for Cr (III) adsorption on the synthetic zeolite A, fitted to the Langmuir, Freundlich and Dubinin-Radushkevich, D-R adsorption models.

The Langmuir, Freundlich and Dubinin-Radushkevich (D-R) models sorption parameters calculated for Cr (III) are listed in Table 3. Fig. 9 indicates a better fit of the experimental data to the Langmuir isotherm than to Freundlich and D-R isotherms. From Table 3, the Langmuir model also well-fits to the experimentally



Fig. 9. Adsorption isotherms at pH 4.0 and T = 298 K.

C /conc/	Ci(initial) (mg/L)	Ce(equil) (mg/L)	W, Adsorbent mass (g)	Qe (Adsorbed amount) (mg/g)	R, Removal efficiency (%)
10	11	1	0.5102	1.006	91.127
50	60	5.6	0.5043	5.404	90.682
100	107	11.1	0.5047	9.476	89.602
200	228	25.5	0.506	19.990	88.806
300	370	55	0.5087	31.001	85.151
400	466	104.1	0.5047	35.853	77.661
500	510	145.9	0.5079	35.844	71.392

obtained data, in contrast to the Freundlich and D-R models in sorption isotherm and produced the highest R^2 value. Removal of Cr (III) occurs on a homogenous surface by monolayer adsorption as it fits to the Langmuir model.

A comparison of the present fly ash-derived zeolite results for the adsorption of Cr (III) from the water with literature results for other adsorbents is given in Table 4.

Table 4 shows the removal of Cr (III) by the present zeolite A is comparable with NaP1 zeolite synthesized from fly ash containing a medium amount of calcium. Although the CEC of the present zeolite A is higher than that of zeolite NaP1, removal of Cr (III) is greater in zeolite NaP1. Removal of Cr (III) is influenced by many parameters, including the chemical and mineralogical composition of the adsorbents, the chromium concentration, the pH, the contact time etc. One of the reasons for this behavior may be related with the presence of the calcium components such as CaCO₃, free CaO, along with the zeolite phase in the reaction mixture. As Sui et al. [28] suggested at the elevated pH the calcium components could increase ion exchange and adsorption; thus, chromium hydroxide precipitation will be improved.

As discussed above at higher pH, the Cr (III) removal occurs by metal oxide precipitation. The present research suggests that with a lesser amount of calcium, at low pH, the Cr (III) is removed by adsorption mechanism.

These experimental parameters used are not the same in the other work which makes a direct comparison impossible. However, the relatively low removal efficiency of Cr (III) by the present zeolite A suggests further optimization of the synthesis parameters is required. The presence of the calcium and iron components in the raw fly ash may also have a beneficial effect on Cr (III) adsorption as it were shown by Wu et al. [27] and Sui et al. [28], though, it has lower CEC. The present research also suggests for the Cr (III) removal at high pH the purity of the synthesized zeolite is less important than the chemical and mineralogical composition of the used adsorbent. It is unnecessary to modify the high calcium fly ash's chemical and mineralogical compositions to remove Cr (III) from the waste water at high pH. However, at low pH, modification of the chemical and mineralogical composition of the raw fly ash is desirable for the preparation of zeolite A to exchange with the Cr (III).

Conclusions

Zeolite A was synthesized from fly ash by acid leaching followed by fusion with NaOH before hydrothermal reaction. Fly ash was subjected to fusion at 600 °C temperature to eliminate crystalline mullite and quartz and increase the starting mixture's homogeneity. The zeolite A was obtained by hydrothermal treatment at 100 °C for 24 h. The major crystallized zeolite was zeolite A, with minor amounts of sodalite. The dissolution of crystalline phases of the raw fly ash by fusion increases the CEC of the synthesized zeolite to 1.01 mg/g by 2-fold.

The optimal pH for Cr^{3+} removal by the synthesized zeolite was found to be in the range 2-6. The zeolite achieved equilibrium and became constant after 30 min, with the removal of 97% Cr (III) from the aqueous

Table 3. Langmuir, Freundlich and Dubinin-Radushkevich sorption isotherm parameters of Cr (III) by the zeolite synthesized from fly ash

T (K)	Langmuir model			Freundlich model			Dubinin-Radushkevich model		
298 -	Q _{max}	K (L/mg)	\mathbb{R}^2	K_{f} (mg/g)	n	\mathbb{R}^2	Q _{max}	K(mol/J)	\mathbb{R}^2
	46.78	0.0287	0.9897	3.8921	2.137	0.9413	34.61	235.2	0.951

Table 4. Adsorption of Cr (III) from solution by various adsorbents.

N	Raw materials and reaction product	Adsorption properties for Cr(III)	References
1.	Zeolite synthesized from various amounts of Ca containing fly ashes	1.NaP1 (low calcium fly ash) - 25.2mg/g 2.NaP1(medium calcium fly ash) - 39.4mg/g 3. Hydroxysodalite (high calcium fly ash)-75.5 mg/g	[27] [28]
2.	NaY zeolite	2.5 – 2.73 mequiv/g	[37]
3.	Faujasite (FAU)- type zeolite membranes have grown from ZSM-2 seeds coated on both as prepared and mesoporous silica-coated porous supports	removal from aqueous feeds (>95%).	[38]
4	Modified hydroxy-aluminum bentonites (OH-Al bentonites	s)Max. 24 mg/g	[39]
5.	Hydrous magnesium coated fly ash (MFA)	Max. adsorption 78.6 mg/g	[40]
6.	Sediment sample treated with fusion and boiling	38.9 to 75.8 mg/g	[41]
7.	Coal fly ash	0.44 mmol Cr/g pellet.	[42]
8	Zeolite A synthesized from fly ash	35.8 mg/g	Present research

solution at concentration of 10 mg/L. The batch study indicated that at an initial Cr (III) concentration of 317 mg/L, the optimum values for both chromium removal efficiency and removal capacity were 86.5% and 26.25 mg/g.

Equilibrium data tested against the Langmuir, Freundlich and Dubinin-Radushkevich isotherm model equations. The Langmuir model yield better fit ($R^2 =$ 0.9987) than the Dubinin-Radushkevich ($R^2 =$ 0.951) and Freundlich model ($R^2 =$ 0.9413).

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