Al (III) removal from wastewater by natural clay and coconut shell

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Abstract

This paper describes the adsorption of Al³⁺ ions from aqueous solutions, by natural clay (from Sakarya’s Yenigün district) and coconut shell modified by means of acid treatment. Batch experiments were carried out to determine the effect of various factors such as initial pH (4-9), temperature (20, 40, 70 ⁰C), initial concentration (10 to 200 mg L⁻¹), and contact time (1-120 minute) on the adsorption process. The adsorption experiments were performed at a temperature of 20 ± 2 ⁰C, at 200 rpm agitation rate, with an adsorbent level of 1 g L⁻¹, and pH 6.8. Adsorption analyses of the clay variety used in adsorption analyses revealed it to be saponite clay, within the larger group of smectite clay minerals. The application of Langmuir revealed maximum adsorption capacity of 149.25 mg g⁻¹ for natural clay adsorbent (NCA), and 120.482 mg g⁻¹ for coconut shell adsorbent (CSA). Moreover, adsorption kinetics were found to be consistent with the second order kinetics (R² > 0.95). The result shows that, natural clay and coconut shell adsorbents are effective adsorbents to remove Al³⁺ from aqueous solutions with good adsorption rate (>92.8%).

Keywords: adsorption, Aluminium, clay, coconut shell, kinetic

1. Introduction

When released to the receiving environment, wastewater with a high level of metal content, produced in various anthropogenic activities, would cause significant toxicity in the water and biota, and may pose hazards for the environment and human health through gradual bioaccumulation. Advanced techniques such as chemical precipitation, adsorption, ion exchange, reverse osmosis, membrane separation are employed to remove various metal ions observable in high concentration levels in industrial wastewaters. These methods are, in turn, characterized by their relative advantages and disadvantages regarding supply, affordability, ease-of-implementation, waste production etc. Adsorption refers to the specific method which involves collecting and capturing the gas, liquid or dissolved substances on the surface of an adsorbent. It is a widely used, efficient, effective and popular method for removing metals from water. For years scientists have been using all kinds of clay minerals, zeolites, chitosan, wood, plant, fruit, peel, shells (Bhatnagar et al., 2010), seeds, resins, gel, silica, activated carbon etc. for metal adsorption, with significant levels of success (Internò et al., 2015).

Researchers used various clays in the context of adsorption of metal ions, such as: polygorskite clay from Dwaalboom area of S.Africa for Pb²⁺, Ni²⁺, Cr³⁺, Cu²⁺ metal ions at pH 7 (Potgieter et al., 2009); Ghassoul clay from Morocco for Cd²⁺ and Cu²⁺ at pH<6 (El Ass, 2018); montmorillonite from India’s Karnataka region for Cu²⁺ at pH 2.5 (Oubagaranadin and Murthy, 2010); montmorillonite and kaolinite for Fe³⁺ (pH 3), Co²⁺ (pH 5.8), Ni²⁺ (pH 5.7) (Bhattacharyya and Gupta 2008); Cæbentonite from Almeria (Spain) and a Na-exchanged bentonite from Milos (Greece) for Cr³⁺ (pH 4), Ni²⁺ (pH 6), Zn²⁺ (pH 6), Cu²⁺ (pH 5) and Cd²⁺ (pH 6) (Alvarez-Ayuso and Garcia-Sanchez, 2003); kaolinite from China’s Longyan region for Pb²⁺ (pH 6), Cu²⁺ (pH 6.5), Cd²⁺ (pH 7) and Ni²⁺ (pH 7) (Jiang et al., 2010); Amasya Celtek clay from Turkey for Pb²⁺ (pH 6), Cr³⁺ (pH 6) (Sari et al., 2007); Polygorskite clay for Pb²⁺ at pH 5 (Fan et al., 2009), illite from Tunisia for Pb²⁺ at pH 7 (Eloussaiief and Benzina, 2010); smectite from Tunisia for Pb²⁺ at pH 4 (Chaarfi et al., 2008); illite from Tunisia for Cd²⁺ and Cr³⁺ at pH 3.5 (Ghorbel-Abid et al., 2010). These studies led to significant successes in the removal of metal ions from aqueous solutions. Researchers have also been investigating various processes to make use of waste, by putting them to use, for instance, in water treatment
removal from water (Ihara et al., 2008; Pascu et al., 2016).

The objective of this study, in turn, is to use adsorbents prepared by treating natural materials such as natural clay and coconut shells with acids, for the removal of aluminum ions, which are found in excessive volumes in water, and which pose significant health hazards.

2. Material and methods

2.1. Preparation of clay and coconut shell adsorbents

The natural clay mineral collected from Sakarya’s Yenigün district and the shells of coconuts purchased at supermarkets were crushed and washed multiple times using pure water, and then dried in an oven at a temperature of 105 °C overnight. Then, 10-gm samples were taken from the clay and coconut shells, and treated separately in 10% H₂SO₄, 37% HCl, and 65% HNO₃. Thereafter, the samples were dried once again in the oven at a temperature of 105 °C, for two hours. 6 individual adsorbent samples prepared through these activation procedures were then used in adsorption experiments.

2.2. Adsorption studies

The batch experiments for Al³⁺ adsorption were performed on an orbital shaker at 200 rpm using glass erlenmeyer flasks containing 500 mL of solution and 0.5 g of the adsorbent. Experiments were performed at room temperature (20 ± 2 °C) and at optimal pH values, except those in which the effects of temperature and pH were investigated. Adsorption studies were performed by using 1 g adsorbent (clay and coconut shell) with various initial Al (III) concentrations ranging from 10 to 200 mg L⁻¹. The preliminary experiments revealed that about 30 min was required for the adsorption process to reach equilibrium. Therefore, a contact period of 120 min was finally selected for all equilibrium tests. The working solutions of 10-200 mg L⁻¹ Al(III) were prepared by appropriate dilution of the Al stock solution (Merck nr. 119770) right before use. The concentration of the Aluminium ions was determined by a Spectro Arcos ICP (Inductively Coupled Plasma) spectrometer. The pH of the solution was adjusted to desired values with 0.1 N HNO₃ and 0.1 N NaOH.

The adsorbed Al (III) concentration levels were assessed based on the difference between the total initial Al (III) and final Al (III) concentrations. The adsorption capacities of clay and coconut shell adsorbents as milligram per gram of adsorbents (mg g⁻¹ sorbent) were calculated by the following equation:

$$ q_t = (C_0 - C_t)V/W $$

where:

- $q_t$ is the initial concentration of Al³⁺ ion (mg L⁻¹),
- $C_t$ is the Al³⁺ ion concentration after adsorption time t (mg L⁻¹),
- $V$ is the volume of metal ion solution (mL) and
- $W$ is the weight of adsorbent (g).

The data gathered through...
the initial concentration experiments were used in calculations in accordance with Langmuir Freundlich, Temkin and Dubinin-Radushkevich isotherms.

3. Results and discussion

3.1. Characterization of clay and coconut shell sorbents

The structure and chemical composition of the clay collected from Sakarya's Yenigün district were analyzed through X-ray diffractometer (XRD) and X-ray fluorescence spectrometer (XRF) instruments. The results of the analyses are provided in Table 1 and Fig.

1. XRF chemical analysis of clay revealed it to be unrefined saponite.

Saponite is a member of Smectites clay group, and contains magnesium and/or iron as well as alkalis other than potassium, and certain alkaline earth elements. Saponite is a naturally occurring 2:1 trioctahedral layered silicate wherein the anionic layer charge originates from the isomorphous substitution of Al(III) for Si(IV) in the tetrahedral sheet. The gallery cations can be readily replaced by a variety of functional cations for potential applications in catalysis and adsorption (Shao and Pinnavaia, 2010).

![Fig. 1. XRD patterns of Saponite clay mineral](image)

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>K.K.</th>
<th>Humidity</th>
<th>SiO₂</th>
<th>CaO</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>MgO</th>
<th>K₂O</th>
<th>Na₂O</th>
<th>P₂O₅</th>
<th>TiO₂</th>
<th>MnO</th>
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<th>SrO</th>
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<tr>
<td>%Amount</td>
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<td>9.1</td>
<td>43.54</td>
<td>17.22</td>
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<td>7.104</td>
<td>2.01</td>
<td>2.63</td>
<td>0.963</td>
<td>0.196</td>
<td>0.851</td>
<td>0.16</td>
<td>0.085</td>
<td>0.057</td>
</tr>
</tbody>
</table>

Humidity : 105 °C  
K.K.: 1000 °C

There are various forms of clay in the nature, such as kaolinite, smectite, illite, chlorite, and talc. Given clay’s abundance in nature, as well as its affordability and ease of supply, coupled with its high level of specific surface area and replacement capabilities, it is only natural to see many studies in the literature, discussing the use of clay minerals as adsorbents (Silva et al., 2016; Araujo et al., 2013). In order to ascertain the surface characteristics of clay and coconut shell adsorbents, SEM images were taken following activation using H₂SO₄ and adsorption process (See Fig. 2 a, b, c, d).
3.2. The effect of initial pH, temperature and initial Al$^{3+}$ concentration

In order to determine the most optimal pH value where the best adsorption results were achieved, the experiments were carried out at a temperature of 20°C, at 200 rpm agitation rate for 2 hours, at 4, 5, 6, 7 pH values. Samples taken at the 1$^{st}$, 5$^{th}$, 10$^{th}$, 30$^{th}$, 60$^{th}$, 90$^{th}$ and 120$^{th}$ minute of the process were analyzed (See Fig. 3 for results).

Fig. 3 shows that the pH levels, which ensure the highest level of adsorption are pH 7 for CSA, and pH 6 for NCA. As Aluminum is inclined to transition to a colloidal structure in the pH range 5.5 - 7, its perfect precipitation can be achieved at around pH 8. The pH values used in the experiments, below the range of transition to a complete colloidal structure and sedimentation of Aluminium, are hypothesized to accelerate and increase the efficiency of adsorption.

The temperature analyses were carried out at 20°C, 40°C and 70°C at optimal pH values at 200 rpm agitation rate for 2 hours. The results are provided in Fig. 4.

The initial concentration analyses were carried out at 20°C, at 200 rpm agitation rate for 2 hours, at pH6 for clay and pH 7 for coconut shell. The samples taken at 1$^{st}$, 5$^{th}$, 10$^{th}$, 30$^{th}$, 60$^{th}$, 90$^{th}$ and 120$^{th}$ minutes were analyzed in ICP (See Fig. 5)
3.3. Adsorption equilibrium isotherms and kinetics

Four isotherm equations were applied for this study: The Langmuir (Equation (1)) (Langmuir, 1918), Freundlich (Equation (2)) (Freundlich, 1906), Temkin (Equation (3)) (Temkin and Pyzhiev, 1940) and Dubinin-Radushkevich (Equation (4)) (O’Connor et al., 2001) isotherms.

\[ q_e = \frac{K_e C_e}{1 + a_1 C_e} \]  \hspace{1cm} \text{linearized form as} \hspace{1cm} \frac{C_e}{q_e} = \frac{1}{K_e} + \frac{a_1}{K_e} \log C_e \hspace{1cm} (2)

\[ q_e = K_F C_e^{1/n} \] \hspace{1cm} \log q_e = \log K_F + \frac{1}{n} \log C_e \hspace{1cm} (3)

\[ q_e = B \ln A + B \ln C_e \] \hspace{1cm} \text{and} \hspace{1cm} B = R.T/b \hspace{1cm} (4)

\[ q_e = q_m e^{-\beta_1 \varepsilon} \] \hspace{1cm} \text{where} \hspace{1cm} \varepsilon = RT \ln[1+(1/C_e)] \hspace{1cm} (5)

where: \( q_e \) is equilibrium capacity in mg g\(^{-1}\); \( C_e \) is the equilibrium concentration in mg L\(^{-1}\); \( K_e \) and \( a_1 \) are Langmuir constants related to the maximum capacity (L g\(^{-1}\)); \( K_F \) (mg g\(^{-1}\)) and \( n \) (g L\(^{-1}\)) are Freundlich constants; \( A \) and \( B \) are Temkin isotherm constants; \( q_m \) is the maximum capacity (mg g\(^{-1}\)); \( \beta \) is a D-R constant (m g\(^2\) J\(^{-1}\)), \( \varepsilon \) Polanyi potential and \( \beta_1 = \frac{1}{\sqrt{2}} \).

All the parameters calculated from the studied isotherm models along with the correlation coefficients (\( R^2 \)) are given in Table 2. Al adsorption effected through CSA and NCA adsorbents is found to be best compatible with the Freundlich (See. Fig. 6) equation. It is also found to be compatible with Langmuir equation as well. As the surfaces of both adsorbents exhibit significant imperfections, and as adsorption with both adsorbents initially develop very quickly, only to slow down gradually, Freundlich isotherm is considered the best representation of adsorption with both. Temkin isotherm, on the other hand, is a match for adsorption with CSA, while adsorption with NCA does not offer a match. Dubinin-Radushkevich equation is unable to identify any isotherm equilibrium.

### Table 2. Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isotherm parameters for Al\(^{3+} \) adsorption by NCA and CSA

<table>
<thead>
<tr>
<th></th>
<th>Langmuir isotherm</th>
<th>Freundlich isotherm</th>
<th>Temkin isotherm</th>
<th>Dubinin-Radush. iso.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( a_e ) (L/mg)</td>
<td>( K_e ) (L/g)</td>
<td>( q_m ) (mg/g)</td>
<td>( R^2 )</td>
</tr>
<tr>
<td>NCA</td>
<td>0.09293</td>
<td>13.8669</td>
<td>149.254</td>
<td>0.9389</td>
</tr>
<tr>
<td>CSA</td>
<td>0.05552</td>
<td>6.689</td>
<td>120.482</td>
<td>0.9692</td>
</tr>
</tbody>
</table>

![Graph showing adsorption isotherms](image)
In kinetic studies; Intraparticle diffusion model (Weber and Morris, 1962), Lagergren pseudo-first-order (Lagergren, 1898), Pseudo-second-order (Ho and McKay, 1999) and Elovich (Elovich and Larinov, 1962) models were used to test the experimental data and the associated formulas.

In order to analyze the adsorption kinetics of Al adsorption, Intraparticle diffusion model (Weber and Morris, 1962), Lagergren pseudo-first-order (Lagergren, 1898), Pseudo-second-order (Ho and McKay, 1999) and Elovich (Elovich and Larinov, 1962) kinetic models were applied with the experimental data. The intraparticle diffusion model is expressed as follows:

\[ q_t = k_{int} t^{1/2} \quad (6) \]

The pseudo-first order model is represented by the equation:

\[ \ln(q_e-q_t) = \ln q_e - k_1 t \quad (7) \]

The pseudo first-order model describes adsorption in solid–liquid systems based on the adsorption capacity of solids.

The pseudo-second-order equation based on equilibrium adsorption is described as:

\[ \frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \quad (8) \]

Finally, the Elovich’s model is expressed as,

\[ q_t = \frac{1}{\beta} \ln(\alpha \beta) + \frac{1}{\beta} \ln t \quad (9) \]

Where \( q_e \) (mg g\(^{-1}\)) and \( q_t \) (mg g\(^{-1}\)) are the values of amount adsorbed per unit mass at equilibrium and at any time \( t \), respectively. Where \( k_{int} \) (mg g\(^{-1}\) min\(^{-1/2}\)) is the intraparticle diffusion rate constant. Values of \( k_{int} \) were calculated from the slope of the linear plots of \( q_t \) versus \( t^{1/2} \).

\( k_1 \) (min\(^{-1}\)) is the rate constant of the pseudo-first order equation, \( k_2 \) (g mg\(^{-1}\) min\(^{-1}\)) is the rate constant of the second-order equation. The parameter \( \alpha \) is the initial sorption rate constant (mg/g min) and \( \beta \) is related to the extent of surface coverage and activation energy for chemisorptions (g mg\(^{-1}\)).

Table 3 and 4 summarizing the results obtained from the graphs reveal that adsorption of Al\(^{3+}\) metal ions with both adsorbents is a very good match for second order kinetics (See Fig. 7). Their correlation factors calculated on the basis of the second order kinetics are 0.99.

**Table 3.** Kinetic parameters and correlation coefficient for the Al\(^{3+}\) adsorption on NCA

<table>
<thead>
<tr>
<th>( C_0 ) (mg/L)</th>
<th>( q_e ) (mg/g)</th>
<th>Intraparticle diffusion model</th>
<th>Pseudo first order kinetic</th>
<th>Pseudo second order kinetic</th>
<th>Elovich model</th>
</tr>
</thead>
<tbody>
<tr>
<td>( k_{int} ) (mg/g dak ( ^{1/2}))</td>
<td>( R^2 )</td>
<td>( k_1 ) (1/dak)</td>
<td>( R^2 )</td>
<td>( k_2 ) (g/mg dak)</td>
<td>( R^2 )</td>
</tr>
<tr>
<td>10</td>
<td>9,9</td>
<td>0,696</td>
<td>0,916</td>
<td>0,0322</td>
<td>0,914</td>
</tr>
<tr>
<td>50</td>
<td>43,59</td>
<td>2,1447</td>
<td>0,512</td>
<td>0,0281</td>
<td>0,769</td>
</tr>
<tr>
<td>100</td>
<td>78,91</td>
<td>4,7774</td>
<td>0,925</td>
<td>0,0273</td>
<td>0,884</td>
</tr>
<tr>
<td>150</td>
<td>116,49</td>
<td>11,1030</td>
<td>0,968</td>
<td>0,0257</td>
<td>0,892</td>
</tr>
<tr>
<td>200</td>
<td>133,03</td>
<td>11,2990</td>
<td>0,952</td>
<td>0,0146</td>
<td>0,897</td>
</tr>
</tbody>
</table>

Among other kinetics models, intraparticle diffusion model defines adsorption achieved with the adsorbent obtained from clay. As water which, through capillary effect diffuses into the weak Van der Walls bonds of clay, is able to expand the gaps between clay molecules by up to 7 times, the clays are compatible with the intraparticle diffusion model. Elovich equation, on the other hand, offers a partial definition of both adsorption cases.
3.4. Thermodynamic properties

Thermodynamic parameters can be determined using the equilibrium constant $K_e$, which depends on temperature. The Gibb’s free energy ($\Delta G^o$), enthalpy ($\Delta H^o$) and entropy ($\Delta S^o$) changes for $\text{Al}^{3+}$ ions adsorption have been determined using Eq. (10).

$$\Delta G^o = - R \cdot T \cdot \ln K_e$$

where, $K_e$ is the equilibrium constant, $C_s$ (mg g$^{-1}$) and $C_a$ (mg L$^{-1}$) are the equilibrium of adsorbate and residual concentration in the solution, respectively. $R$: the universal gas constant (8.314 J mol$^{-1}$ K$^{-1}$) and $T$: temperature in Kelvin ($K$), $\Delta H^o$ and $\Delta S^o$ parameters can be calculated from the slope and intercept of the plot of $\ln K_e$ versus $1/T$ yields (Van’t Hoff), respectively (Yurtsever and Şengil, 2009).

Thermodynamic analysis was run as well, on the basis of the results of the adsorption experiment carried out at 293, 313, 343 K using clay and coconut shell as adsorbent. Parameters such as the thermodynamic equilibrium constant, Gibb’s free energy ($\Delta G^o$), enthalpy change ($\Delta H^o$) and entropy change ($\Delta S^o$) for the adsorption of $\text{Al}^{3+}$ ions were calculated, and results are presented in Tables 5 and 6.

Table 4. Kinetic parameters and correlation coefficient for the $\text{Al}^{3+}$ ion adsorption on CSA

<table>
<thead>
<tr>
<th>$\text{Al}^{3+}$ Model</th>
<th>Intraparticle diffusion model</th>
<th>Pseudo first order kinetic parameters</th>
<th>Pseudo second order kinetic parameters</th>
<th>Elovich model</th>
</tr>
</thead>
<tbody>
<tr>
<td>$C_s$ (mg/L)</td>
<td>$q_e$ (mg/g)</td>
<td>$k_{int}$ (mg/g.dak$^{-1}$)</td>
<td>$R^2$</td>
<td>$k_1$ (1/dak)</td>
</tr>
<tr>
<td>------------------</td>
<td>----------------</td>
<td>----------------</td>
<td>-------</td>
<td>-------------</td>
</tr>
<tr>
<td>10</td>
<td>9.28</td>
<td>0.3676</td>
<td>0.745</td>
<td>0.0251</td>
</tr>
<tr>
<td>50</td>
<td>38.15</td>
<td>0.1528</td>
<td>0.833</td>
<td>0.009</td>
</tr>
<tr>
<td>100</td>
<td>71.41</td>
<td>0.64425</td>
<td>0.964</td>
<td>0.0246</td>
</tr>
<tr>
<td>150</td>
<td>88.35</td>
<td>0.34483</td>
<td>0.819</td>
<td>0.0267</td>
</tr>
<tr>
<td>200</td>
<td>105.36</td>
<td>0.69002</td>
<td>0.973</td>
<td>0.0171</td>
</tr>
</tbody>
</table>

Many studies showed that agricultural wastes are highly efficient for the removal of metals. Abdel-Ghania et al. (2015) studied the removal of $\text{Al}^{3+}$ ions from aqueous solution onto rice hull in order to explore their potential use as low-cost activated carbon for effective metal removal from wastewater. Adsorption isotherms were determined at ~20 °C and the experimental data obtained were modelled with the Langmuir, Freundlich, Temkin, Dubinin-Radushkevich isotherms, the pseudo-first order, pseudo-second order and Elovich kinetic equations. On the basis of the experimental results and adsorption models applied in this study, one can reach to the conclusion that equilibrium data were represented well by a Langmuir isotherm equation with maximum $\text{Al}$ adsorption capacity of 34.48 mg g$^{-1}$ (pH 5) for Rice Hull Activated Carbon (RHAC). Singh et al. (2007) using rice husk carbon (RHC) and commercial granular activated carbon (GAC) for $\text{Al}^{3+}$ removal from drinking water, observed 1.6 mg g$^{-1}$ maximum adsorption capacity with rice husk char at an optimal pH of 4.2. Lodeiro et al. (2010) studied Aluminium removal from wastewater by refused beach cast seaweed and found maximum adsorption capacity of 22.5 mg g$^{-1}$ at pH 4. Denizli and Say (2001) investigated the adsorption behavior of magnetic poly(2-hydroxy ethyl methacrylate) (mPHEMA) adsorbent in the removal of $\text{Al}^{3+}$ ions from drinking and dialysis water. They found maximum $\text{Al}^{3+}$ adsorption was 722 μmol g$^{-1}$ polymer at pH 5.0. In particular, it is evident that the use of a waste material in removing toxic pollutants from wastewater would bring multiple environmental benefits.

In the present study, natural clay and waste coconut shells were successfully used as a raw material for the preparation of a cost effective adsorbent, which was previously untested in $\text{Al}^{3+}$ removal.

4. Conclusions

This study is based on the adsorption studies of $\text{Al}^{3+}$ ions to remove it from aqueous solutions, using coconut shell and clay-based adsorbents, which are low cost, natural, and easy to procure materials. The results revealed that the optimal pH value for maximum adsorption was pH ~7 for CSA, and pH 6 for NCA. These pH ranges are actually the pH range in which $\text{Al}$ assumes a perfect colloidal form. The adsorption equilibriums achieved with both adsorbents match the Freundlich
and Langmuir isotherm. Maximum adsorption calculated for Al$^{3+}$ ions on the basis of Langmuir isotherm is found to be 120.482 mg g$^{-1}$ for CSA, and 149.25 mg g$^{-1}$ for NCA. The maximum Al$^{3+}$ removal efficiencies were 98.95% for NCA at pH 6 and 92.83% for CSA at pH 7. Thermodynamic analyses revealed that adsorption based on CSA was endothermic ($\Delta S$: 78.32 J/mol.K), whereas adsorption based on NCA was exothermic ($\Delta S$: -46.35 J/mol.K). Adsorption kinetics were, on the other hand, found to match the second order kinetic model. In this study, the potential use of natural materials such as coconut shells (an agricultural waste) and clay (from Turkey) in the adsorption of Al$^{3+}$ from aqueous solutions were investigated. These materials can be highlighted as promising adsorbents with a noticeable adsorption capacity for the removal of Aluminium ions from aqueous solution even around neutral pH values.

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