

Regular Article

Application of Activated Clay for Improvement of Water Quality in Wire Electric Discharge Machining

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In this study, the availability of activated clay treatment for decreasing the electrical conductivity in the real wire cut water was demonstrated. Activated clay (AC) was prepared, and its characteristics were investigated. The ability to adsorb Zn²⁺ and the removal ability through electrical conductivity were evaluated. It was found that the AC had a montmorillonite-like structure. Its specific surface area, micropore volume, mesopore volume, and macropore volume were 125.0 m²/g, 0.7 μL/g, 172.6 μL/g, and 13.7 μL/g, respectively. The cation exchange capacities at pH 5 and pH 10 were 56.2 and 67.2 cmol/kg, respectively. The effects of temperature, contact time, and pH of the solution on the adsorption of Zn²⁺ were measured. The amount of Zn²⁺ adsorbed by the AC increased with an increase in the adsorption temperature or in the pH. Adsorption isotherms data were fitted to the Freundlich equation compared to the Langmuir equation. Adsorption equilibrium was reached within 30 min, and kinetic data were fitted to the pseudo-second-order model compared to the pseudo-first-order model. Additionally, the AC was proven to effectively suppress the electrical conductivity. The suppression achieved by using washed AC was higher than that obtained by virgin AC. Washing AC with distilled water is useful for removing released ions (SO₄²⁻). Therefore, the column treatment packed with washed AC was evaluated in this paper. Finally, washed AC could remove Zn²⁺, resulting in the reduction of electrical conductivity (removal percentage is 47%). These findings provide significant information that can be useful for the removal of Zn²⁺ and reducing the electrical conductivity from wire cut water.

Key words activated clay, electrical conductivity, wire cut, zinc ion, adsorption

INTRODUCTION

Wire electric discharge machining is an innovative technology that can accurately and automatically cut even hard materials, such as carbides, into complicated shapes.¹⁾ Brass is mainly used as wire and is pulled through the workpiece from a supply spool onto the take-up equipment. During the application, an electric discharge occurs between the wire electrode and the work-piece in the presence of tap (or deionized) water with high resistivity.²⁾ This phenomenon induces its capability of machining any material with electrical conductivity more than 0.01 μS/cm (increasing the ion concentrations released from wire or workpiece).³⁾

In addition, it is important to achieve an optimal condition for the wire electric discharge machining. If the process is not performed in optimal conditions, serious problems such as short-circuiting of the wire, wire breakage, and work surface damage can easily occur.¹⁾ Therefore, many researchers suggested solutions that entail physicochemical techniques.^{1,3-7)} One of them ensures the water quality by decreasing the amount of anions or cations in the tap water. In general, this is achieved by controlling the electrical conductivity through an ion exchange treatment. However, this technology is relatively costly and thus not economically feasible. Therefore, an alternative technique must be envisioned.

In this study, we focused on using an activated clay (AC) to decrease the electrical conductivity (i.e. the amount of ions) in aqueous solutions. Our AC is a 2:1 layered clay mineral, with a tetrahedral layer centered on silicon (or aluminum), and an octahedral layer centered on aluminum (or magnesium).^{8,9)} Previous studies reported that AC is a much cheaper material; yet, it has a good adsorption capability which is beneficial for cation removal from aqueous solutions.^{10,11)} However, no study reported on AC being applied for maintaining the water quality in wire electric discharge machining. Therefore, we used AC for the improvement of water quality in wire electric discharge machining.

In this study, we prepared the AC and investigated its physicochemical characteristics. The effect of temperature, contact time, and pH of the solution on the adsorption of ions by the AC were also evaluated. Finally, the application of the AC using a column was demonstrated. The obtained results provide new insights for improving water quality and thus the performance of wire electric discharge machining.

MATERIALS AND METHODS

Materials A sample solution of zinc ions was prepared using zinc chloride (FUJIFILM Wako Pure Chemical Co., Japan). The AC was purchased from Kanto Chemical Co. Inc.,

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Japan, and the “Washed AC” was prepared by the following method. Three grams of AC were added to 500 mL of purified water. The suspension was shaken at 100 rpm for 24 h at room temperature. Subsequently, the suspension was filtered through a 0.45 μm membrane filter (Advantec MFS, Inc., Japan), and the obtained residue was the “Washed AC” used in this study. In addition, real circle water from an electrical discharge machining apparatus was obtained from Uehara sei shoji Co. Ltd., Japan.

Properties of the AC The characteristics of the AC were determined by the following method. Electron microscope images were obtained using a SU-1510 (SEM, JEOL, Japan), and the functional groups were measured by a FT-IR-460Plus (JASCO, Japan). X-ray diffraction (XRD) analysis was performed by MiniFlex II (Rigaku, Japan); specific surface area and pore volume were measured by a NOVA 4200e specific surface analyzer (Yuasa Ionics, Japan). The cation exchange capacity (CEC) was determined by the Japanese Industrial Standard (JIS 1478) method. The pH of the solution was measured with a digital pH meter (HORIBA, Ltd, Japan).

Analysis of Real Circle Water The electrical conductivity of a real circle water, obtained from an electrical discharge machining apparatus, was measured by a multi water quality checker conductivity meter, WA-2017SDJ (Sato Shoji, Japan). Concentrations of sodium, potassium, magnesium, calcium, zinc, copper, manganese, and silicon ions were measured by an inductively coupled plasma optical emission spectrometry, iCAP-7600 (ICP-OES, Thermo Fisher Scientific Inc., Japan). Concentrations of fluoride, chloride, nitrate, and sulfate ions were measured by ion chromatography (DIONEX ICS-900, Thermo Fisher Scientific, USA). The measurements were performed using the IonPac AS12A system (4 \times 200 mm, Thermo Fisher Scientific, USA). The mobile phase and regenerant comprised 2.7 mmol/L Na_2CO_3 + 0.3 mmol/L NaHCO_3 and 12.5 mmol/L H_2SO_4 , respectively. The flow rate was 1.5 mL/min at ambient temperature. The micro membrane suppressor was an AMMS 300 system (4 mm, Thermo Fisher Scientific, USA).

Adsorption Isotherms AC (0.05 g) was added to a zinc ion solution at 5–100 mg/L (50 mL). The suspension was shaken at 100 rpm for 2 h at 10, 25, and 50°C. The sample solution was filtered through a 0.45 μm membrane filter, and the filtrate concentration of zinc ion was measured by ICP-OES. The amount of zinc ion adsorbed was calculated using Eq. (1).

$$q = (C_0 - C_e) / VW \quad (1)$$

where q is the amount adsorbed (mg/g), C_0 is the initial concentration, C_e is the equilibrium concentration (mg/L), V is the solvent volume (L), and W is the weight of the adsorbent (g). The data are expressed as the mean \pm standard error (S.E.) of the mean ($n=3-6$).

Effect of Contact Time and pH on the Adsorption of Zinc Ion onto the AC To evaluate the contact time, AC (0.05 g) was added to a zinc ion solution at 50 mg/L (50 mL). The suspension was shaken at 100 rpm for different time intervals (0.17, 0.5, 2, 6, 22, and 24 h) at 25°C. To evaluate the pH, the adsorption experiments were performed with 0.05 g of AC and a zinc ion solution at 50 mg/L (50 mL) at different pH values (pH: 3, 4, 5, and 6). The pH level was adjusted by 0.1 mol/L of hydrochloric acid. The amount of zinc ion adsorbed in each case was calculated using Eq. (1). The data are expressed as the mean \pm standard error (S.E.) of the mean ($n=3-6$).

Applications of AC at Different Conditions AC (0.1 g or

0.2 g as condition I or condition II, respectively) and Washed AC (0.1 g as condition III) were added to real circle water (100 mL). The suspensions were shaken at 250 rpm for different time intervals (0.17, 0.5, 1, 2, 6, 22, and 24 h) at room temperature. In addition, an ion exchange resin (Uehara sei shoji Co., Ltd., Japan) was used as the control in this experiment. Changes in each concentration, pH, and electrical conductivity were evaluated by the aforementioned methods.

Finally, to evaluate the application of AC in the real field, the changes in each concentration, pH and electrical conductivity were measured by the aforementioned methods using a column with Washed AC (diameter \times height: 2.5 cm \times 10 cm). Washed AC (3.0 g) was added up to a level of 1 cm above the base of the column. The approximate condition for the column experiment was as follows: space velocity (S.V.), 61.1 1/h; linear velocity (L.V.), 0.61 m/h; flow rate, 5 mL/min (down flow). The column experiment was performed at ambient (room) temperature, and the volume of real circle water was 500 mL. The data are expressed as the one-time measurement.

RESULTS AND DISCUSSION

Properties of Adsorbent The surface roughness of the AC was confirmed in this study (Fig. S1). The physicochemical properties of the AC were evaluated. The specific surface area, micropore volume, mesopore volume, and macropore volume were 125.0 m²/g, 0.7 μL /g, 172.6 μL /g, and 13.7 μL /g, respectively. The cation exchange capacity at pH 5 and pH 10 were 56.2 and 67.2 cmol/kg, respectively. The FT-IR of AC is shown in Fig. S2. The bands at 3699 cm^{-1} (Al-OH-Al stretching) and 915 cm^{-1} (Al-OH-Al binding) are indicative of dioctahedral smectite. The peaks at 3435 cm^{-1} (H-O-H stretching) and at 1637 cm^{-1} (H-O-H bending) indicate the presence of water. In addition, the broad at 1034 cm^{-1} represents the Si-O-Si stretching vibration.^{10,12} Finally, the chemical structure was confirmed (Fig. S3). The XRD pattern of the AC shows mainly the presence of montmorillonite. These results indicate that the surface properties of the AC have similar trends for raw and acid-treated bentonites.¹²

Concentration and Electrical Conductivity Decreasing the electrical conductivity in the circle water is one of the goals of this study. To this aim, we firstly measured the concentration and electrical conductivity of real circle water in wire cut condition (Table 1). Concentrations (and resulting contributions to the overall electrical conductivity) of Mg^{2+} , Zn^{2+} , and Cl^- were higher than those of other ions. The electrical conductivity of the wire cut water was 60.6 $\mu\text{S}/\text{cm}$. Mg^{2+} and Cl^- are usually present in tap water, yet it is Zn^{2+} that highly contributes to the electrical conductivity (12.5 $\mu\text{S}/\text{cm}$, 21%). Therefore, removal of Zn^{2+} from wire cut water is important for decreasing electrical conductivity, thus we focused in our experiments on removing Zn^{2+} from the aqueous solution system.

Adsorption Capacity of Zn^{2+} onto the AC The adsorption isotherms of Zn^{2+} onto the AC at different temperatures are shown in Fig. 1. The adsorbed amounts positively depended on temperature ($10 < 25 < 50^\circ\text{C}$). However, this trend was not confirmed at equilibrium concentration of over 40 mg/L in the case of adsorption isotherm at 25°C. Previous studies reported that Zn^{2+} adsorption in bentonite clay or in special modified raw bentonite was approximately 2–3 or 0.4–2.3 mg/g, respectively.^{13,14} In this study, the adsorbed amount was

Table 1. Concentration and Electrical Conductivity of Real Circle Water in Wire Cut Condition

Elements	Concentration (mg/L)	Electrical conductivity ($\mu\text{S}/\text{cm}$)
Na ⁺	1.38	3.01
K ⁺	0.23	0.44
Mg ²⁺	3.73	16.3
Ca ²⁺	2.13	6.34
Zn ²⁺	7.76	12.5
Cu ²⁺	2.24	3.89
Mn ²⁺	0.10	0.20
Si	0.27	0.00
F ⁻	1.61	4.69
Cl ⁻	4.62	9.96
NO ₃ ⁻	0.88	1.01
SO ₄ ²⁻	1.39	2.78

Table 2. Langmuir and Freundlich constants for the adsorption of zinc ions onto the AC

Temperatures (°C)	Langmuir constants			Freundlich constants		
	W_s (mg/g)	a (L/mg)	r	logK	1/n	r
10	10.8	0.01	0.689	-1.06	0.88	0.982
25	4.49	0.04	0.922	-0.31	0.45	0.961
50	4.90	0.13	0.996	0.03	0.35	0.966

approximately 4.0 mg/g. Therefore, the adsorption capability of the AC is sufficient for removing Zn²⁺ from an aqueous solution.

The adsorption isotherms were evaluated by the Langmuir (2) or Freundlich (3) equations.

$$C_e/q_e = 1/aW_s + C_e/W_s \quad (2)$$

where q_e is the adsorption capacity at equilibrium (mg/g), C_e is the equilibrium concentration of Zn²⁺ (mg/L), and W_s and a are the maximum adsorption capacity (mg/g) and the equilibrium adsorption constant (L/mg), respectively.

$$\log q_e = \log K + 1/n \log C_e \quad (3)$$

where K is a constant indicative of the relative adsorption capacity of AC, and n is a constant indicative of the intensity of the adsorption and varies with surface heterogeneity.¹⁵⁾

Table 2 shows the Langmuir and Freundlich constants for the adsorption of Zn²⁺ onto the AC. The correlation coefficient of Freundlich (0.961–0.982) provides a better fit than that of Langmuir (0.689–0.996). The Freundlich-type isotherm hints toward surface heterogeneity. In addition, Zn²⁺ was easily adsorbed on the AC when $1/n$ was in the range of 0.1–0.5 but not when $1/n > 2$. These results suggest that Zn²⁺ adsorption readily occurred when $1/n < 2$ (0.35–0.88).¹⁶⁾

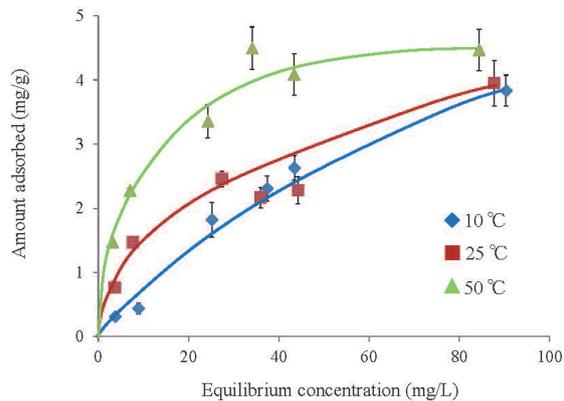
Effect of Contact Time and pH on the Adsorption of Zn²⁺ Figure 2 shows the effect of contact time on the adsorption of Zn²⁺ onto the AC. The adsorption equilibrium was reached within 30 min. Previous studies reported that the adsorption equilibrium of Zn²⁺ onto natural bentonite or kaolin was attained within 25 h or 80 min, respectively.^{17,18)}

The adsorption kinetics were used to evaluate the rate of adsorption and the adsorption mechanism.^{19,20)} Kinetic data were investigated by applying the pseudo-first-order (Eq. (4)) and pseudo-second-order (Eq. (5)) models, as follows:

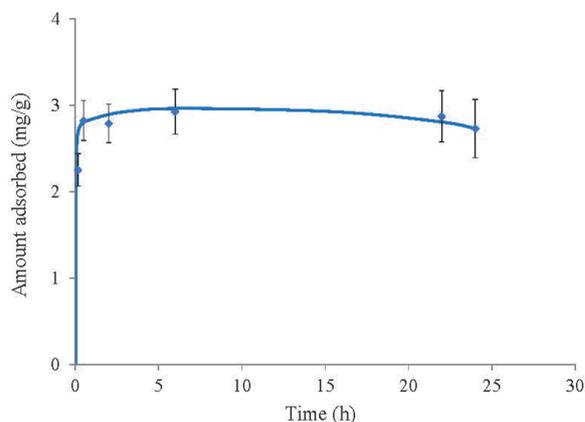
$$\ln(q_e - q_t)/q_e = -k_1 t \quad (4)$$

$$t/q_t = 1/(k_2 q_e^2) + t/q_e \quad (5)$$

where q_e and q_t correspond to the amount of Zn²⁺ adsorbed

**Fig. 1.** Adsorption Isotherms of Zinc Ion onto the AC at Different Temperatures

Initial concentration: 5–100 mg/L, solvent volume: 50 mL, adsorbent: 0.05 g, contact time: 2 h, temperature: 25°C, stirring: 100 rpm, pH in the solution: 5.7–6.5, the data are expressed as the mean±standard error (S.E.) of the mean (n=3–6).

**Fig. 2.** Effect of Contact Time on the Adsorption of Zinc Ion onto the AC

Initial concentration: 50 mg/L, solvent volume: 50 mL, adsorbent: 0.05 g, contact time: 0–24 h, temperature: 25°C, stirring: 100 rpm, pH in the solution: 5.8, the data are expressed as the mean±standard error (S.E.) of the mean (n=3–6).

at equilibrium and at time t (mg/g), respectively; k_1 is the pseudo-first-order rate constant (1/h); and k_2 is the pseudo-second-order rate constant (g/mg/h).

The correlation coefficient of the pseudo-second-order model (0.999) was higher than that of the pseudo-first-order model (0.417). In addition, the value of $q_{e,\text{exp}}$ (2.93 mg/g) was closer to that of $q_{e,\text{cal}}$ in the pseudo-second-order kinetic model (2.79 mg/g) than in the first-order model (0.23 mg/g). These results indicate that the adsorption of Zn²⁺ is controlled by chemical sorption (chemisorption), involving valence forces.²¹⁾

Subsequently, the effect of pH on the adsorption of Zn²⁺ onto the AC was evaluated (Fig. 3). It was observed that the adsorption capacity of Zn²⁺ onto the AC increased with the increasing pH of the solution. At lower pH values, the decreasing of adsorption capacity might be attributed to the electrostatic repulsion between Zn²⁺ and the protonated surface functional group. However, at higher pH values, the adsorption capacity was enhanced due to an increase of the deprotonated surface functional group (Scheme is below).¹¹⁾

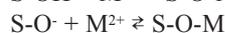
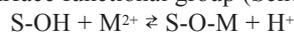
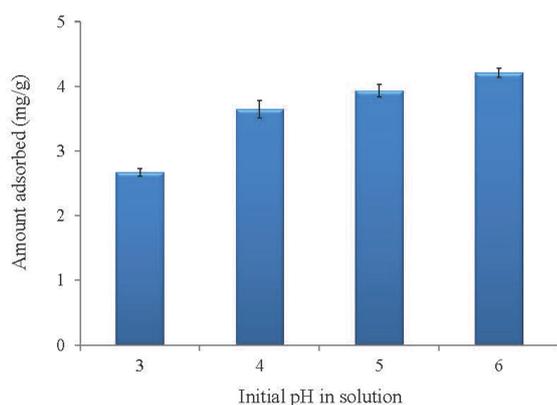


Table 3. Changes in Element Concentrations, pH, and Electrical Conductivity with the AC

Time (h)	0	0.17	0.5	1	2	6	22	24
Elements (mg/L)								
Zn ²⁺	8.11	5.11	4.57	4.28	4.02	3.84	3.72	3.62
Cu ²⁺	2.59	0.53	0.41	0.32	0.29	0.29	0.35	0.32
Mn ²⁺	0.10	0.08	0.07	0.07	0.07	0.07	0.06	0.07
Al ³⁺	0.02	0.00	0.01	0.01	0.03	0.05	0.09	0.08
Si	0.23	0.26	0.33	0.44	0.75	1.32	3.23	3.40
Na ⁺	0.86	0.89	0.88	0.81	0.90	0.84	0.95	0.91
K ⁺	0.25	0.52	0.47	0.42	0.45	0.35	0.50	0.41
Mg ²⁺	4.71	3.51	3.32	3.20	3.12	3.01	2.96	2.91
Ca ²⁺	1.95	1.72	1.67	1.49	1.54	1.38	1.35	1.28
F ⁻	1.52	1.50	1.56	1.55	1.58	1.40	1.54	1.53
Cl ⁻	1.91	1.90	1.96	1.86	2.06	2.10	2.14	2.84
NO ₃ ⁻	0.18	0.16	0.18	0.17	0.44	0.25	0.23	0.25
SO ₄ ²⁻	0.79	6.55	6.80	6.79	6.88	6.38	6.44	6.90
pH	7.1	6.8	6.8	6.7	6.7	6.6	6.6	6.5
Electrical conductivity (μS/cm)	72.7	62.2	59.7	57.2	55.1	52.8	52.9	53.2

Solvent volume: 100 mL, adsorbent: 0.1 g, contact time: 0–24 h, temperature: room temperature, stirring: 250 rpm, pH in the solution: 7.1

**Fig. 3.** Amount of Zinc Ion Adsorbed onto the AC at Different Values of pH

Initial concentration: 50 mg/L, solvent volume: 50 mL, adsorbent: 0.05 g, contact time: 2 h, temperature: 25°C, stirring: 100 rpm, pH in the solution: 3.0–6.0, the data are expressed as the mean±standard error (S.E.) of the mean (n=3–6).

where S represents the active surface functional groups (silanol or aluminol site) onto AC, and M represents the metal ions. In this study, similar trends were observed compared to previous studies.

Application of AC in Real Circle Water Table 3 shows the changes in element concentrations, pH, and electrical conductivity with the AC (Condition I). The AC treatment was performed to decrease the concentration of Zn²⁺ (8.11→3.62 mg/L) and Cu²⁺ (2.59→0.32 mg/L). However, we also observed increases in the concentration of Si (0.23→3.40 mg/L) and SO₄²⁻ (0.79→6.90 mg/L). These results suggest that Zn²⁺ and Cu²⁺ were adsorbed onto the AC, whereas Si and SO₄²⁻ were desorbed from it during the treatment. Finally, it can be concluded that the electrical conductivity of real circle water can be reduced approximately 27% by the AC treatment. In the following experiments, we focused on the concentrations of Zn²⁺ and SO₄²⁻, and on the electrical conductivity (Fig. 4 and Table 4).

We confirmed the increase of SO₄²⁻ after the AC treatment, and therefore washed the AC with distilled water to prevent the release of SO₄²⁻ from the AC. The removal rate of Zn²⁺ from real circle water was similar for Conditions I–III. However, the released concentration of SO₄²⁻ from the AC was in

Table 4. Changes in the Electrical Conductivity in Different Conditions

Time (h)	Electrical conductivity (μS/cm)			
	Condition I	Condition II	Condition III	Ion exchange resin
0	72.7	77.0	72.3	71.9
0.17	62.2	66.4	48.6	66.5
0.5	59.7	65.5	47.0	54.1
1	57.2	63.6	43.7	48.2
2	55.1	65.6	39.7	23.9
6	52.8	66.3	35.8	3.4
22	52.9	66.6	36.2	2.8
24	53.2	66.0	36.0	3.4

Amount adsorbent of virgin AC is 0.1 g or 0.2 g in condition I or condition II, and amount adsorbent of washed AC is 0.1 g in condition III.

Solvent volume: 100 mL, contact time: 0–24 h, temperature: room temperature, stirring: 250 rpm

the following order: Condition III < Condition I < Condition II. This phenomenon indicates that the washed treatment with distilled water is useful for the removal of SO₄²⁻ from the AC. In addition, the reduction rate of the electrical conductivity after 24 h was in the order: Condition II (14%) < Condition I (27%) < Condition III (50%) < Ion exchange resin (95%). However, Condition III shows the ability of reducing the electrical conductivity in a short time, i.e. within 10 min. Therefore, we could establish a very cheap and rapid treatment for the removal of electrical conductivity.

Finally, the column packed with washed AC was used to exemplify a real application of the AC in field conditions (Table 5). The experimental results confirmed that the adsorption capability of Zn²⁺ was higher (7.93→1.51 mg/L), and the released SO₄²⁻ from the AC was lower (1.22→3.66 mg/L) compared to untreated AC. Moreover, the reduction rate of the electrical conductivity was approximately 47%. This result suggests that the column treatment packed with washed AC is useful for reducing the electrical conductivity by ion removal.

In summary, an AC was prepared for removing Zn²⁺ from wire cut water. The physicochemical properties of the AC were investigated, and the adsorption capability was evaluated in this paper. The AC has a montmorillonite-like structure, and a specific surface area of 125.0 m²/g. The amount of Zn²⁺ adsorbed onto the AC increased with the temperature increasing (10–50°C). The experimental data were fitted to the Freun-

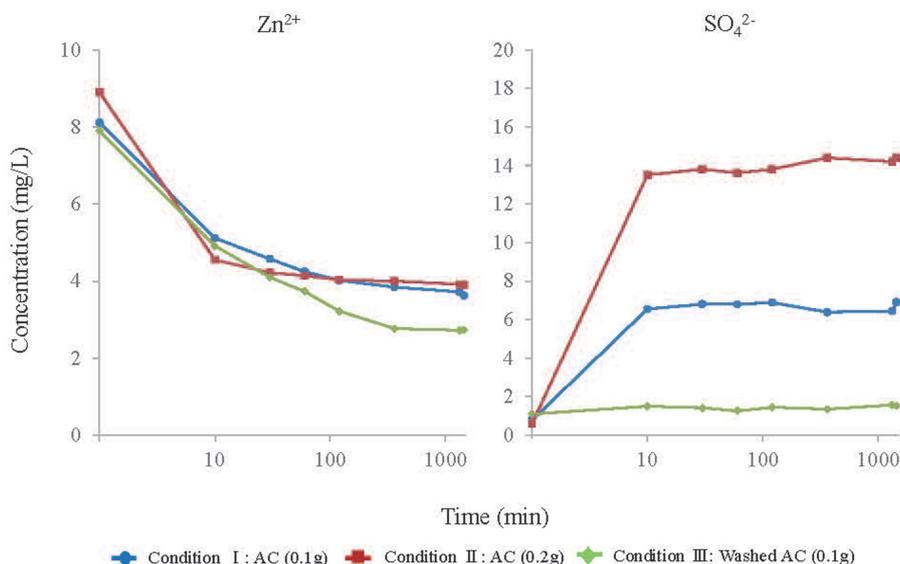


Fig. 4. Changes in the Concentration of Zinc Ion and Sulfate Ions Using the AC in Real Circle Water

Solvent volume: 100 mL, adsorbent: 0.1–0.2 g, contact time: 0–24 h, temperature: room temperature, stirring: 250 rpm, pH in the solution: 6.8–7.5, the data are expressed as the one-time measurement.

Table 5. Changes in Element Concentrations, pH and Electrical Conductivity Using a Column Containing Washed AC

Time (h)	0	0.17	0.5	1	2	6	22	24
Elements (mg/L)								
Zn ²⁺	7.93	7.20	4.71	1.73	1.30	1.12	1.56	1.51
Cu ²⁺	2.65	3.02	1.31	0.13	0.58	0.14	0.61	0.60
Mn ²⁺	0.10	0.10	0.06	0.03	0.03	0.04	0.03	0.03
Al ³⁺	0.00	0.00	0.00	0.02	0.04	0.06	0.04	0.03
Si	0.23	0.36	0.46	0.64	0.75	1.39	3.41	3.56
Na ⁺	0.70	0.68	0.63	0.60	0.64	0.57	0.54	0.56
K ⁺	0.78	0.71	0.84	1.50	0.91	0.68	0.65	0.52
Mg ²⁺	4.83	4.37	2.97	1.37	1.24	1.22	1.26	1.23
Ca ²⁺	1.91	1.93	1.41	0.85	0.89	0.84	0.68	0.72
F ⁻	0.07	0.07	0.06	0.06	0.07	0.06	0.07	0.07
Cl ⁻	2.70	2.53	2.46	3.39	2.96	2.74	2.37	2.63
NO ₃ ⁻	0.28	0.33	0.21	0.21	0.20	0.18	0.19	0.22
SO ₄ ²⁻	1.22	3.61	2.84	3.16	3.45	3.58	3.69	3.66
pH	6.8	6.6	6.5	5.8	5.2	4.8	4.9	4.8
Electrical conductivity (μS/cm)	69.9	66.9	51.9	36.9	39.5	41.7	37.1	36.9

Solvent volume: 500 mL, flow rate: 5mL/min, S.V.: 61.1 l/h, L.V.: 0.61 m/h

dlich equation (correlation coefficient: 0.961–0.982). Moreover, the adsorption capability was found to be affected by the contact time and pH of the solution. Adsorption equilibrium of Zn²⁺ onto the AC was reached within 30 min. Adsorption rate data were shown to fit better to the pseudo-second-order model (correlation coefficient: 0.999) than to the pseudo-first-order model (correlation coefficient: 0.417).

Additionally, the AC was proven to be able to reduce the electrical conductivity of wire cut water. The reduction of electrical conductivity using washed AC was higher (50%) than that achieved using untreated AC (27%). A column treatment packed with washed AC was useful for reducing the electrical conductivity (47%). Taken together, these results indicate that washed AC could have a high potential for reducing the electrical conductivity (through Zn²⁺ removal) from wire cut water.

Conflict of interest The authors declare no conflict of interest.

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