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Application of cellulose acetate to the selective adsorption and recovery of Au(III)

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ABSTRACT

Cellulose acetyl derivatives were examined for the selective recovery of Au(III) from acidic chloride solutions as an adsorbent, and cellulose acetate fibers (CAF) were found to be effective for the separation of Au(III) from other metal ions, including the precious metal ions Pt(IV) and Pd(II). The amount of Au(III) adsorbed by the fibers increased with an increase in the hydrochloric acid concentration, but decreased with an increase in the ionic strength of the solution. The adsorption of Au(III) onto CAF took place quickly and an adsorption equilibrium was reached within 1 h. The maximum adsorption capacity of Au(III) was determined to be 110 mg/g at 2 M hydrochloric acid. The loaded Au(III) was readily recovered by incineration.

1. Introduction

Precious metals are of great importance currently because of their widespread applications in high-tech industries. Gold and palladium are especially indispensable in the manufacture of mobile phones and computers. The frequent replacement of these electronic devices causes the accumulation of large amounts of electronic and electrical waste, offering an important recycling opportunity for the secondary supply of precious metals. For example, the gold concentration in mobile phone handsets is 300–350 g/t and in computer circuit boards is 200–250 g/t, which is tens of times higher than that in gold ores (5–30 g/t) (Hageluken & Corti, 2010). Accordingly, the separation and recovery of gold from e-waste has attracted much interest (Mack, Wilhelmi, Duncan, & Burgess, 2007).

Conventional methods for metal recovery such as solvent extraction and ion exchange have been applied to the separation and recovery of gold. Several extractants have been designed for the recovery of gold (Baker, Baker, & Schulzke, 2011; Lu et al., 2011). In addition, various ion exchange resins have also been studied and evaluated for their adsorption performance toward Au(III) ions (Alguacil, Adeva, & Alonso, 2005; Nguyen, Jeong, Jha, Lee, & Osseo-Asare, 2010; Nguyen, Lee, et al., 2010). However, the capacity and selectivity of these conventional methods are still low. Moreover, secondary waste can be generated during these recovery processes, and post-treatment is always costly. From an environmental perspective, these petroleum-dependent reagents, solvents, and resins should be replaced by alternatives derived from renewable biomass resources.

Recently, natural polymers have attracted interest as alternatives to synthetic adsorbents. After treatment or modification, natural polymers such as cellulose, chitosan (Bratskaya, Azarova, & Matochkina, 2011; Chen, Lam, Mak, & Yeung, 2011; Ramesh, Hasegawa, Sugimoto, Maki, & Ueda, 2008), chitin (Schleuter et al., 2013), tannin (Huang, Wang, Liao, & Shi, 2010; Ogata & Nakano, 2005) and proteins (Kiyoyama, Maruyama, Kamiya, & Goto, 2008; Maruyama et al., 2007) have been studied, and are reported to be effective in the removal and recovery of metal ions. Despite its crucial importance, the adsorption selectivity of biosorbents toward metal ions has hardly been discussed, especially adsorption selectivity toward precious metal ions. Chitosan and its derivatives have been widely used for the adsorption of precious metal ions because of their nitrogen containing structure. Although amine groups and other nitrogen containing groups have been shown to have a large adsorption capacity for Au(III), no separation ability for Au(III) has been reported. On the other hand, oxygen containing functional groups shows better selectivity toward gold (Saitoh, Suzuki, & Hiraide, 2005).

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Among all the natural polymers, cellulose has great potential because of its abundance and low cost. Cellulose has overwhelmingly been developed as an adsorbent by blending with chitosan (Qu et al., 2009) or upon modification by functional groups (Tasdelen, Aktas, Acma, & Guvenilir, 2009). Despite several reports on cellulose-based adsorbents for the removal of heavy metal ions and the recovery of precious metal ions, few reports exist on the use of cellulose acetyl derivatives as an adsorbent.

Cellulose acetyl derivatives are produced by the esterification of acetic acid with naturally derived cellulose from wood pulp or cotton linter. The glucose unit that makes up the raw material polymer cellulose has three hydroxyl groups, and triacetate is the cellulose derivative in which most of the hydroxyl groups have been substituted with acetyl groups. Cellulose acetate is obtained by the partial hydrolysis of triacetate. These polymers, which are widely used in photographic film, cigarette filters, plastic materials and as a fiber in the textile industry, are thus industrially produced and a large amount of waste products are released. If cellulose acetyl derivatives can be used as an adsorbent without further modification, a cost-effective adsorption process is possible.

In this study, for the first time, we used cellulose acetyl derivatives as adsorbents for Au(III) and examined its practical use in the adsorptive separation of Au(III) from other metal ions.

2. Materials and methods

2.1. Materials

Cellulose acetate powders (CAP, degree of substitution DS: 2.4) and cellulose acetate fibers (CAF, DS: 2.4) derived from a cotton linter were provided by Daicel Chemical Industries, Ltd. (Tokyo, Japan), and were used as received.

Commercial reagent grade microcrystalline cellulose Avicel PH101 (Avicel) (Sigma–Aldrich, St. Louis, MO, USA), cellulose acetate (CA) and cellulose triacetate (CTA) (DS: 2.4 and 2.9, respectively, Wako Pure Chemical Industries, Ltd., Osaka, Japan) were used for comparison with the cellulose acetates CAP and CAF.

Standard aqueous solutions (1000 mg/L) of Au(III), Pd(II), Pt(II), In(III), Zn(II), Cu(II), and Ni(II) were purchased from Wako Pure Chemicals as metal ion sources. All other chemicals were of reagent grade.

2.2. Batch adsorption experiments

Batch adsorption experiments were carried out with aqueous solutions containing metal ions. An aqueous solution containing metal ions was prepared by diluting the standard solutions with deionized water. The pH of the solution was adjusted by an appropriate concentration of aqueous NaOH or HCl. The concentration of HCl in the metal solutions was adjusted using 5 M hydrochloric acid.

Five milliliters of an acid solution containing 10 mg/L metal ions was added to a known amount of a cellulose derivative as the adsorbent in a 15 mL centrifuge tube, followed by stirring in a thermostatic bath at 298 K to attain equilibrium.

After adsorption, the solution was filtered through a poly(tetrafluoroethylene) membrane filter. After appropriate dilution with deionized water, the metal ion concentrations in the aqueous phase were determined using an inductively coupled plasma atomic emission spectrometer (ICP-AES, Optima 5300 DV; Perkin-Elmer Co., MA, USA).

The adsorption percentage A (%) for each metal ion was calculated according to Eq. (1):

$$A(\%) = \frac{100(C_i - C_e)}{C_i} \quad (1)$$

The amount of metal adsorbed per unit mass of adsorbent q_e (mg/g) was calculated according to Eq. (2):

$$q_e(\mathrm{mg/g}) = \frac{V(C_i - C_e)}{m} \quad (2)$$

where C_i (mg/L) and C_e (mg/L) are the initial and the equilibrium metal ion concentrations, respectively. V (L) is the solution volume and m (g) is the mass of adsorbent.

2.3. X-ray diffraction (XRD) analysis

The X-ray diffraction patterns of the cellulose samples used in this study were obtained using an X-ray diffractometer (XRD) (Ultima IV, Rigaku, Akishima, Japan) at room temperature from 5 to 50° using Cu/Ka irradiation at 40 kV and 30 mA. The scanning speed was 2°/min and the data were collected in continuous mode.

2.4. Surface characteristics

The surface characteristics of Avicel, CA, CTA, CAP and CAF were analyzed by Brunauer–Emmett–Teller (BET) analysis of N_2 physisorption at 77 K using a sorption apparatus (BELSORP-miniII, BEL Japan Inc., Osaka). The BET surface area by physisorption of Kr (77 K) and the steam adsorption of H_2O (298 K) were also determined for CAP and CAF by BELSORP-max (BEL Japan Inc.).

2.5. Fourier transform infrared (FTIR) spectroscopy

To characterize the structure of the CAP and the Au(III)-loaded CAP, FTIR spectra of CAP and Au(III)-loaded CAP were analyzed with a Spectrum 100 FTIR spectrometer equipped with an attenuated total reflectance (ATR) unit (Spectrum 65, PerkinElmer Co.). The spectra were obtained over the range of 4000–700 cm^{-1} with eight scans recorded at a resolution of 4 cm^{-1}.

2.6. Thermogravimetric analysis (TGA)

TGA analysis for the Au(III)-loaded CAF and the original CAF was conducted using a TGA analyzer (Simultaneous DTA-TG Apparatus, TG7300, Brucker, Japan). Approximately 10 mg of the original CAF and the Au(III)-loaded CAF was placed in a platinum (Pt) crucible for the TGA analysis. A heating rate of 10 °C/min was used in a temperature range from 30 to 800 °C.

2.7. Laser scanning confocal microscopy

The residue of the Au(III)-loaded CAF after incineration by TGA was observed using 3D Laser Scanning Confocal Microscopy (VK-X100, KEYENCE Co., Japan).

3. Results and discussion

3.1. Characterization of adsorbents used

Fig. 1 shows the XRD patterns of Avicel, CA, CAP and CTA, from which morphological differences were determined. Fig. 1(a) refers to the diffraction pattern of Avicel. The peaks at 15° and 22.5° are characteristic of highly crystalline cellulose (Cheng et al., 2011). In Fig. 1(b) and (c), the main peak at approximately 8° in the XRD patterns of CA and CAP is the principal characteristic peak of a semicrystalline acetylated cellulose. The position of this peak indicates the generation of a disorder when cellulose is acetylated (Barud et al., 2008). For CTA, a more uniform structure causes better chain packing resulting in a high degree of crystallinity, as

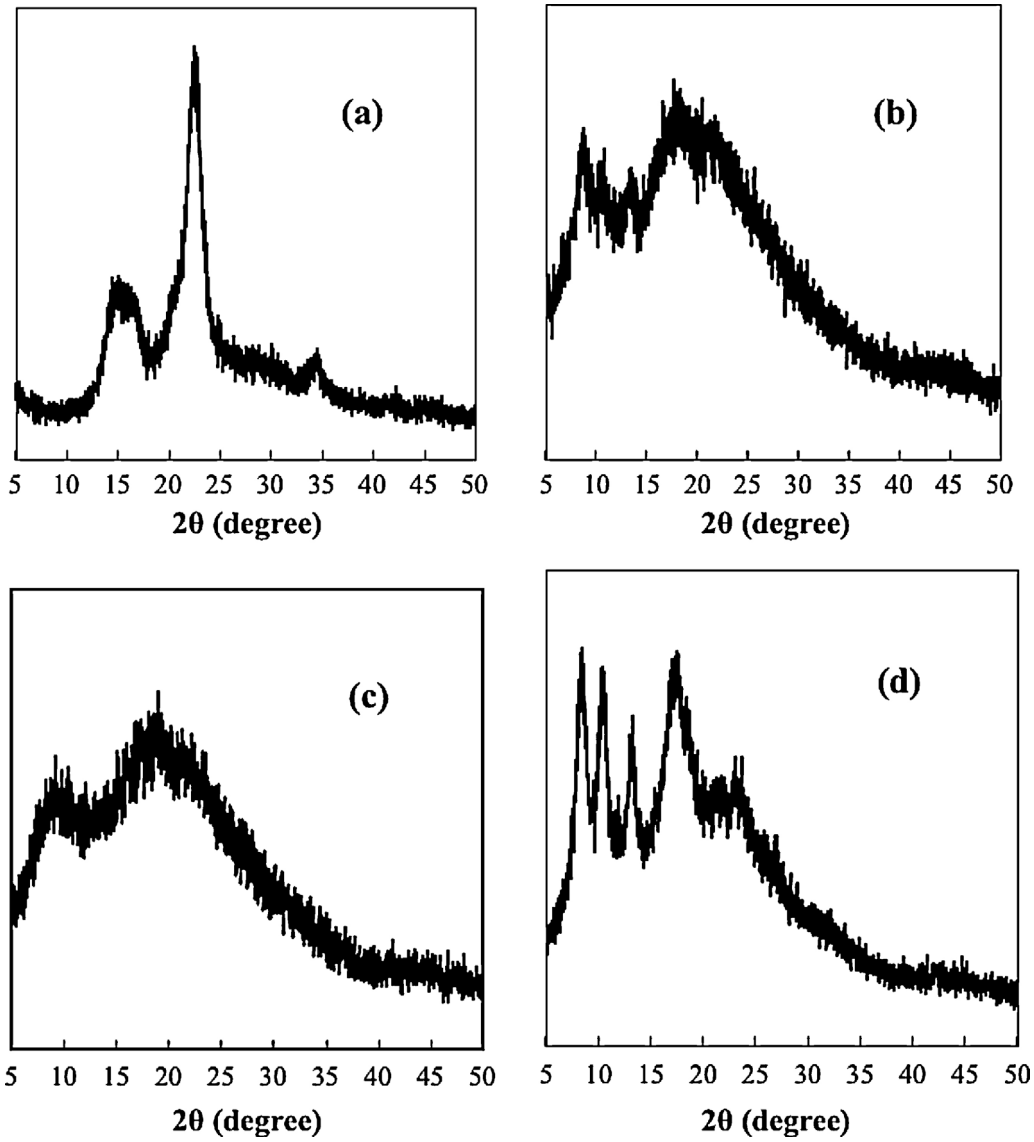


Fig. 1. XRD pattern for cellulose and cellulose acetyl derivatives. (a) Avicel; (b) CA; (c) CAP; and (d) CTA.

shown in the XRD pattern of CTA in Fig. 1(d) (Rodrigues et al., 2000).

In addition, the specific surface area of these adsorbents was measured by BET analysis of N_2 physisorption. As shown in Table 1, CAP showed the largest area and pore volume among the adsorbents measured. As for CAF, the surface area by adsorption of Kr, BET(Kr), was measured because the surface area could not be obtained via physisorption of N_2, and compared to that for CAP. The BET(H_2O) surface area of CAF and CAP was also measured via a steam adsorption of H_2O, and the results were summarized together in Table 2. It was found that the ratio of BET(H_2O) surface area toward BET(Kr) surface area of CAF was 32 times larger than that of CAP, that is, the surface of CAF is far more hydrophilic than that of CAP.

3.2. Adsorption ability of different cellulose acetyl derivatives toward Au(III)

The adsorption ability of different cellulose acetyl derivatives toward Au(III) was initially investigated using commercial reagents. Fig. 1 shows the Au(III) adsorption percentage on different cellulose acetyl derivatives from 0.1 M HCl solutions containing 10 mg/L Au(III).

As shown in Fig. 2, the adsorption of Au(III) was found to be affected by the acetylation degree of cellulose, and cellulose acetate CA showed the highest adsorption ability among Avicel, CA and triacetate CTA. The industrial product CAP is derived from cotton linter and it has a similar adsorption ability to CA. Acetylation was expected to contribute to the enhancement of the adsorption

Table 1
Surface characteristics of Avicel, CA, CAP and CTA.

Adsorbent	BET N_2 surface area (m^2/g)	Pore volume (cm^3/g)
Avicel	2.46	0.007
CA	4.6	0.048
CAP	12.7	0.127
CTA	1.86	0.0058

Table 2
Surface characteristics of CAP and CAF.

Adsorbent	BET surface area (m^2/g)		BET (H_2O)/BET (Kr)
	Kr	H_2O	
CAP	9.13	110.6	12
CAF	0.262	101.2	386

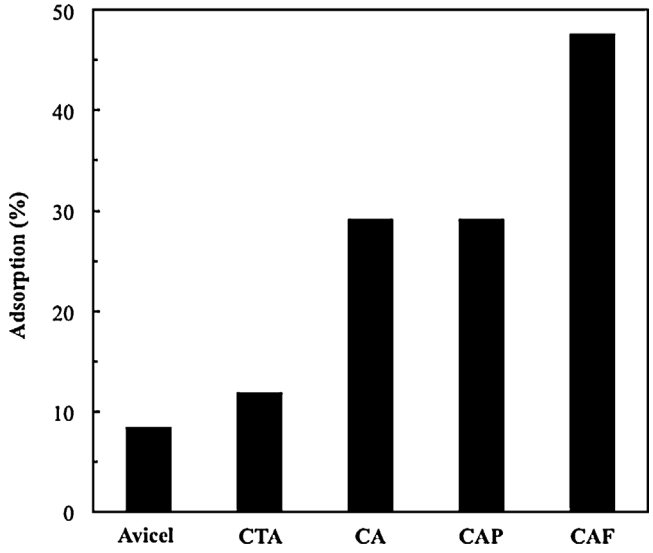


Fig. 2. Adsorption of Au(III) onto different adsorbents. Adsorbent dosage, 2 g/L; contact time, 3 h; agitation speed, 160 rpm; 298 K; 0.1 M HCl.

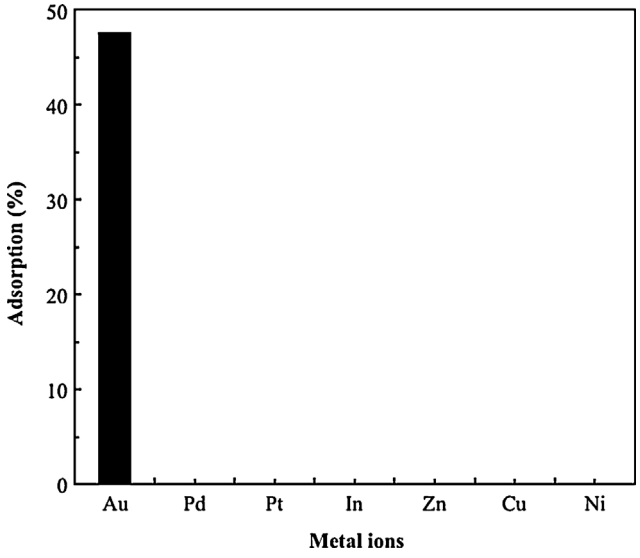


Fig. 3. Selective adsorption of Au(III) onto CAF. Adsorbent dose, 2 g/L; contact time, 3 h; agitation speed, 160 rpm; 298 K; 0.1 M HCl.

performance for Au(III) because the introduction of acetyl groups, which has an affinity for Au(III), might provide more specific sites for Au(III) adsorption. Furthermore, crystalline structure of cellulose seemed to be partially destroyed by acetylation, as shown in Fig. 1. The adsorption ability of the cellulose without acetylation (Avicel) was significantly lower than that of CA.

The more acetylated CTA resulted in a decrease in the adsorption ability, because CTA was more crystalline than CA due to further acetylation, as explained in the last section. A crystalline structure is considered to inhibit the penetration of solutes. Therefore, cellulose acetate, with a more amorphous structure, was demonstrated to be suitable as an adsorbent for metal ions.

The fibrous cellulose acetate CAF had a far better adsorption ability compared with that of the cellulose acetate CAP in powder form, as shown in Fig. 2. As described above, the surface of CAF was observed to be more hydrophilic than that of CAP, thus resulting in a more easily accessible surface for hydrated Au(III) to be adsorbed. The fibrous cellulose acetate CAF was therefore studied as a model adsorbent instead of CAP.

3.3. Selective adsorption of Au(III)

Fig. 3 shows the results of the competitive adsorption of Au(III) onto CAF from an aqueous solution of 0.1 M HCl in the presence of several other precious and base metal ions. Among all the metal ions in solution, Au(III) was adsorbed selectively onto CAF with minimal absorption of the other metal ions. The adsorptive separation of Au(III) from other metal ions, especially other precious metal ions such as Pd(II) and Pt(IV) is difficult (Adhikari et al., 2007). The precious metal ions, Au(III), Pd(II) and Pt(IV) are negatively charged anions in the presence of hydrochloric acid, and they can be separated from positively charged base metal ions based on these differences in charge. The selective adsorption of Au(III) even in the presence of other precious metal ions such as Pd(II) and Pt(IV) has been realized as well with the use of CAF, whose mechanism would be discussed in the following study.

3.4. Adsorption mechanism of Au(III) onto cellulose acetate

The effect of HCl concentration on the adsorption performance of CAF toward Au(III) was examined, as it not only affects the surface charge condition of the adsorbent, but also affects the speciation of Au(III) in acidic chloride media. A previous study reveals that the predominant species of Au(III) are $AuCl_4^-$ at pH below 3.0. As increasing solution pH, species such as $AuCl_3(OH)^-$ appear in acidic chloride media (Ogata & Nakano, 2005).

As shown in Fig. 4(a), the adsorption percentage of Au(III) increased significantly as the HCl concentration increased, and reached a constant value at about 2 M HCl. Therefore, 2 M HCl was used as an experimental condition.

The adsorption of Au(III) onto CAF is unlikely to occur by ion exchange between a chloride anion Cl^- on the protonated cellulose acetate and the $AuCl_4^-$, as the hydroxyl and ester groups of the cellulose acetate cannot easily be protonated. Also, the fact that the Au(III) adsorption percentage increased as the HCl concentration increased cannot be explained by an ion exchange mechanism, as generally a decrease in Au(III) adsorption percentage will be observed because of increased competition from chloride anions.

It has been suggested that cellulose acetate has an affinity for Au(III) because of the interaction between the electron-donating oxygen atoms contained in the acetyl groups and $HAuCl_4$. In acidic chloride media the following equation is relevant (Van Nguyen et al., 2010):

$$AuCl_4^- + H^+ = HAuCl_4 \quad (3)$$

To confirm the electrostatic interaction between CAF and the Au(III) ions, the effect of the ionic strength of the aqueous solution on the adsorption efficiency was examined. Fig. 4(b) shows the relationship between the adsorption percentage and the concentration of sodium perchlorate in the feed solution from 0 to 1 M. As shown in Fig. 4(b), the adsorption efficiency of Au(III) onto CAF predictably decreased as the sodium perchlorate concentration increased because of the shielding effect of the electrostatic interaction between Au(III) and the surface of CAF by sodium ions.

Furthermore, the FT-IR spectra of the original and the Au(III)-loaded CAP were recorded to investigate changes in CAP during the adsorption process. The results show only a slight change at wavenumber 1650 cm^{-1} in CAP before and after Au(III) adsorption, implying that Au(III) was adsorbed via a weak interaction related to ester groups.

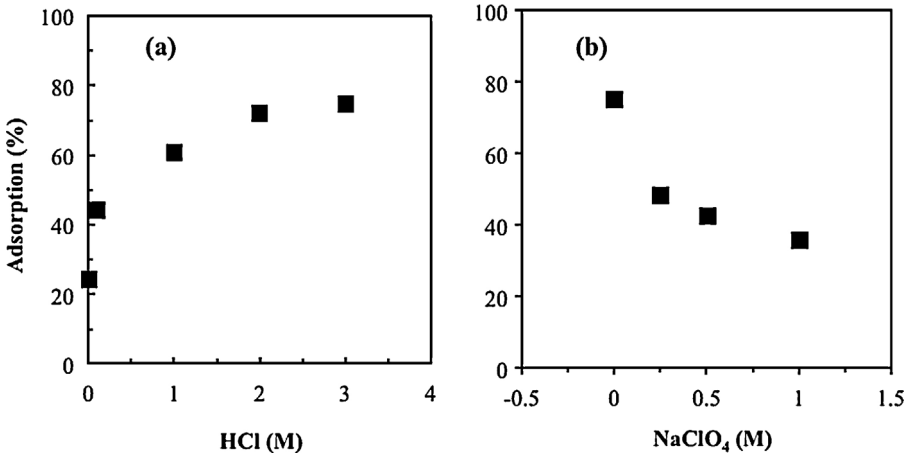


Fig. 4. (a) Effect of HCl concentration on the adsorption of Au(III) onto CAF. (b) Effect of ionic strength on the adsorption of Au(III) onto CAF. Adsorbent dosage, 2 g/L; contact time, 3 h; agitation speed, 160 rpm; 298 K.

3.5. Effect of adsorbent dose

The adsorbent dose plays a significant role in the degree of adsorption of metal ions from aqueous solutions. To estimate the effect of adsorbent dose, an adsorption experiment was conducted by varying the solid to liquid ratio from 2 to 10 g/L while the metal ion concentration was kept constant 10 mg/L at 2 M HCl.

A plot of the degree of Au(III) adsorption onto CAF against the adsorbent dose is shown in Fig. 5. As shown in Fig. 5, the degree of Au(III) adsorption increased as the adsorbent dose increased, as more adsorption sites are accessible for Au(III) when a larger adsorbent dose is used. The adsorption percentage reached about 95% when increasing the adsorption dose to 10 g/L, indicating that the Au(III) in the aqueous solutions almost completely adsorbed onto the CAF.

3.6. Effect of contact time

In the adsorption process, the adsorption rate, i.e. the time to achieve an adsorption equilibrium, is an important factor that affects the overall process efficiency. Fig. 6 shows the time course of the degree of Au(III) adsorption onto CAP and CAF, respectively, at 2 M HCl. As shown in Fig. 6, the degree of Au(III) adsorption increased sharply over the first 20 min, and reached equilibrium in about 60 min. A further increase in the contact time did not affect the adsorption efficiency; therefore, Au(III) was adsorbed as an ion and no other phenomenon such as reduction occurred under the present experimental conditions, as described later.

The rapid adsorption process is a significant advantage of this fibrous cellulose acetate as an adsorbent for use in a wastewater treatment process, and suggests the possible use of the fiber in continuous flow systems (Won, Park, Mao, & Yun, 2011).

3.7. Adsorption isotherms

An adsorption isotherm study for Au(III) adsorption onto CAF was conducted at 298 K by varying the initial Au(III) concentration in the aqueous solution at 2 M HCl. As shown in Fig. 7, the adsorption capacity of CAF increased with an increase in the equilibrium Au(III) concentration and it reached a constant value. The maximum adsorption capacity (q_{max}) was about 110 mg/g, and this was obtained at the plateau of the plots. This value is comparable to other adsorbents such as Amberlite XAD-7HP (58.82 mg/g) (Van Nguyen et al., 2010), lysine modified crosslinked chitosan resin

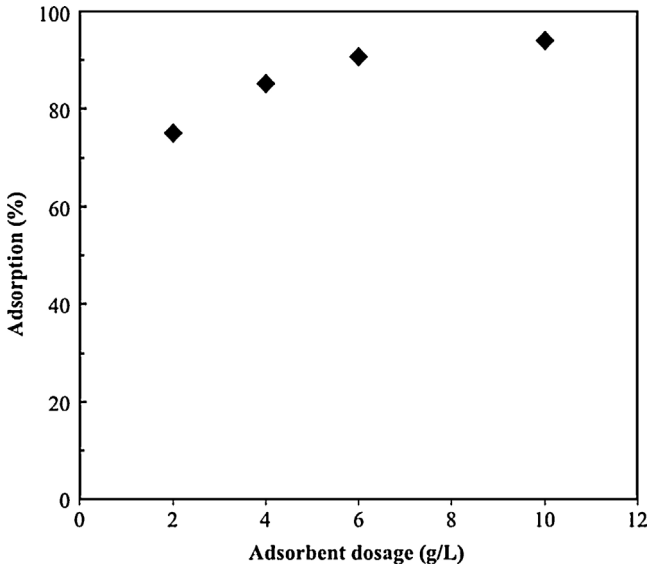


Fig. 5. Effect of adsorbent dose. Contact time, 3 h; agitation speed, 160 rpm; 298 K; 2 M HCl.

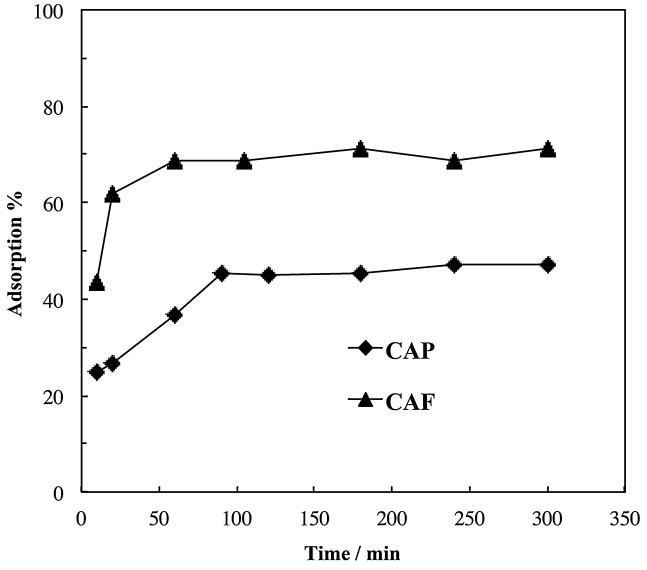


Fig. 6. Effect of contact time on Au(III) adsorption onto CAF. Adsorbent dose, 2 g/L; agitation speed, 160 rpm; 298 K; 2 M HCl.

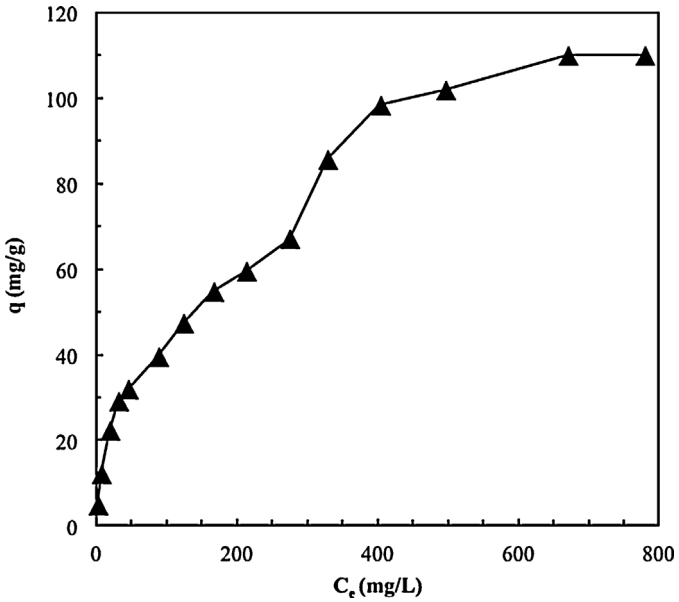


Fig. 7. Adsorption isotherm of Au(III) adsorption onto CAF. Adsorbent dose, 2 g/L; contact time, 3 h; agitation speed, 160 rpm; 298 K; 2 M HCl.

(70.34 mg/g) (Fujiwara, Ramesh, Maki, Hasegawa, & Ueda, 2007), and poly(ethylene glycol) gel (121 mg/g) (Kinoshita et al., 2006).

3.8. Au(III) recovery via incineration

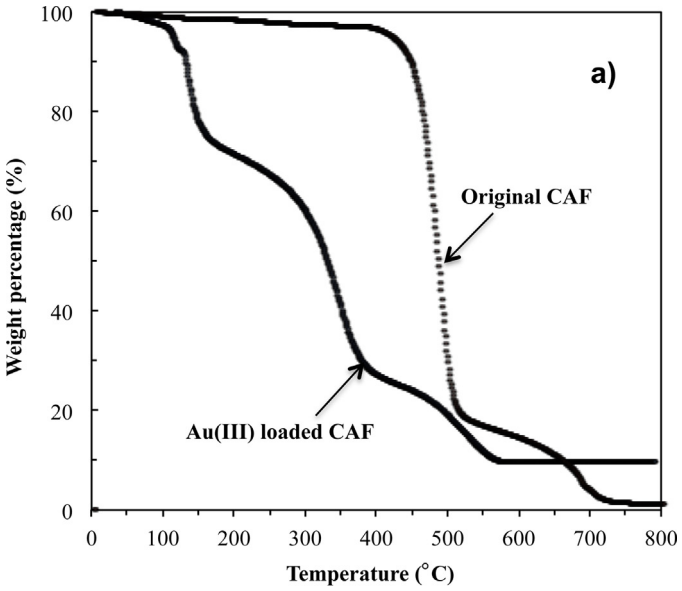
The desorption of precious metals from adsorbents is often difficult. In such cases, incineration is an effective method for the adsorptive recovery of metals. Plant derived hydrocarbon compounds such as cellulose derivatives can be removed by incineration, and Au(III) is thus reduced to metallic Au(0).

Fig. 8(a) shows thermograms for Au(III)-loaded CAF from the Au(III) adsorption experiments where the initial Au(III) concentration was 1000 mg/L, where the q_{max} was obtained, and for non-loaded CAF. Continuous weight loss was observed with an increase in the temperature until the weight percentage of the residue began to plateau. About 10% of the initial weight remained for the Au(III)-loaded CAF, while the CAF was almost completely removed. The difference in weight percentage between the Au(III)-loaded and non-loaded CAF is considered to approximately correspond to the amount of Au that was loaded. The residue of the Au(III)-loaded CAF after incineration was investigated by confocal microscopy and the image is shown in Fig. 8(b).

Because of the low cost of the cellulose acetate fiber used in this study, it is reasonable to employ incineration as a recovery method. In addition, different types of cellulose acetates are commercially produced, and are also generated as industrial waste, and these are considered to be available as a disposable adsorbent.

4. Conclusions

Cellulose acetate fibers that originate from cotton linters are shown to be effective in the selective adsorption of Au(III). Under the experimental conditions used in this study, the degree of Au(III) adsorption onto CAF increased with an increase in the hydrochloric acid concentration to around 2 M. The adsorption may occur by a proton-mediated electrostatic interaction between cellulose acetate and a Au(III) chloro-complex. The adsorption equilibrium was reached within 1 h. The relatively fast adsorption rate and the high adsorption capacity under acidic chloride conditions are



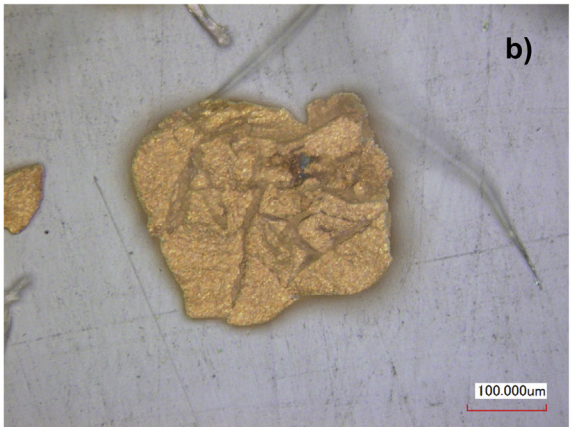


Fig. 8. (a) Thermogravimetric curves of Au(III)-loaded CAF, and the original CAF; (b) confocal microscope image of Au(III)-loaded CAF after incineration.

desirable attributes for the practical use of CAF in an adsorption process for the separation of Au(III).

Cellulose acetate fibers are commercially produced and are also generated as industrial waste. They can therefore potentially be used as cost-effective and environmentally friendly adsorbents for the selective recovery of Au(III).

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.carbpol.2014.05.003.

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