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Adsorptive Recovery of Gold (III) from Acidic Chloride Media Using Carbonized Microalgal Residue

Khunathai K[1,2], Inoue K[1], Ohto K[1]*, Kawakita H[1], Kurata M[2], Atsumi K[2], Fukuda H[2] and Alam S[3]

[1]Department of Applied Chemistry, Faculty of Science and Engineering, Saga University, Honjo-machi 1, Saga 840-8502, Japan
[2]Research Laboratories, DENSO CORPORATION , Minamiyama 500-1, Komenoki, Nisshin, Aichi 470-0111, Japan
[3]Department of Chemical and Biological Engineering, College of Engineering, University of Saskatchewan, 57 Campus Drive, Saskatoon, SK, S7N 5A9, Canada

Abstract

Adsorption gel was prepared by carbonizing a residual waste of microalgae generated in the biofuel conversion process. The prepared gel exhibited high affinity and selectivity for Au (III) over other precious and base metal ions in hydrochloric acid. From the adsorption isotherms, the maximum loading capacity of Au (III) on the carbonized microalgae was evaluated as 1.20 mmol/g (236 mg/g), which is much higher than that on the original microalgal residue. Observations by microphotographs, scanning electron microscope, and X-ray diffraction confirmed the formation of metallic Au, suggesting that a redox reaction had taken place between the adsorbent and Au (III) ions during adsorption. Adsorption kinetics was interpreted in terms of pseudo-first order rate expression to evaluate the activation energy of this system as 33.7 kJ/mol.

Keywords: Adsorption; Gold (III); Microalgal residue; Carbonization; Acidic chloride media

Introduction

In recent years, recycling and recovery of valuable metals from spent electronic and electric appliances has become an emerging important research subject from both of environmental and economical viewpoints [1]. This is due to the facts that recycling of these metals reduces the cost of metallurgical treatments associated with their mining activity, and sharply decreases the destructive impacts on the environment. Adsorption techniques using activated carbons have played important roles in their recovery [2-6]. In recent years, biomass wastes have found their uses for the separation and recovery of toxic and precious metal ions from industrial solutions [7,8].

On the other hand, also in recent years, new energy or green energy has been strongly required as alternative to fossil fuels like petroleum and coal as well as atomic energy. One of such new energy is biodiesel oil produced from microalgae, on which much interests have been attracted all over the world [9]. However, the present production cost of such biodiesel oil is much higher than commercially available diesel oil produced from petroleum. One of the candidate methods to lower the total production cost of biodiesel oil is the effective use of the residue of microalgae after extracting biodiesel oil.

From such backgrounds, the authors attempted to develop a new technology for recovering gold from various industrial wastes using adsorption gel prepared from the above-mentioned microalgal residue only by treating in boiling sulfuric acid in the previous work [10]. Although the preparation of this gel is very simple, it suffers from a problem; that is, it leaves waste sulfuric acid containing polysaccharides and other organics after the preparation, which needs some costs for the post-treatment.

Activated carbons or the carbonized materials are carbonaceous material having well-developed porous structure consisting of hydrophobic graphene layer surface and hydrophilic surface functional groups. Main applications of activated carbon to the recovery of precious metals involve the adsorptive recovery of gold and silver from cyanide media in a so-called carbon-in-pulp (CIP) process in the gold industry [11]. Extensive research works have been conducted in this area [2-6]. Activated carbon is a good adsorbent as it has an extended surface area, porous structure, high adsorption capacity and a certain degree of surface reactivity. These properties are derived by both the nature of the raw materials and the processes employed for the production of the activated carbon.

In the present work, the authors attempted to prepare the new adsorption gel alternative to the above-mentioned gel at a low cost by means of the carbonization method. The microalgal residue mainly contains proteins and carbohydrates as its major components. The carbonization would convert such components to disorganized graphite-liked material. It was reported by Chand et al. [12] that carbonization of biomass waste at moderate temperature without prior chemical activation is attractive since the high specific surface area is retained and use of chemicals can be avoided. Also, due to its high surface area and porous structure, high adsorption capacity is also expected for the recovery of precious metals. The objective of the present work is to fundamentally investigate the adsorption behaviors of the prepared carbon material for the recovery of gold from acidic chloride media because, in hydrometallurgical processes for precious metals, feed materials are leached in acidic chloride media like aqua regia or hydrochloric acid containing chlorine gas.

Experimental

Materials and analytical methods

Analytical grade $HAuCl_4 \cdot 4H_2O$, $H_2PtCl_6 \cdot 6H_2O$, $PdCl_2$, $FeCl_3 \cdot 6H_2O$ (Wako Pure Chemical Industries, Ltd., Osaka, Japan), $ZnCl_2$ (Sigma Aldrich, St Louis, MO, USA), $CuCl_2$, and $NiCl_2 \cdot 6H_2O$ (Katayama

Chemicals, Osaka, Japan) were employed to prepare test solutions of Au (III), Pt (IV), Pd (II), Fe (III), Zn (II), Cu (II), and Ni (II), respectively. All other chemicals used for the preparation of the adsorbent and for the adsorption tests were of analytical grade and were used without further purification.

To measure the specific surface area and pore size distribution of the prepared carbonized gel, 0.2 g of sample was pretreated to remove water content using a model BELPREP-vacII powder/particle characterization analyzer (MicrotracBEL Inc., Osaka, Japan) at 150ºC for 42 h and N_2 adsorption-desorption isotherms at 77 K was measured using a model BELSORP-miniII-SP powder/particle characterization analyzer (MicrotracBEL Inc., Osaka, Japan). The data were analyzed using BET-Plot and t-Plot to evaluate the specific surface area. The classical pore size model developed by Barret-Joyner-Halande (BJH) was used for the pore size distribution calculation.

The infrared spectra of the microalgal residue were recorded using a FT-IR spectrophotometer, JASCO FT/IR-410 (JASCO Corp., Tokyo, Japan). The C, H, and N contents were determined by elemental analysis. The surface morphology of the adsorbent was recorded using a digital microscope KEYENCE model VHX-1000 (KEYENCE Corp., Osaka, Japan) and a scanning electron microscope (SEM), JEOL model JCM-5100 (JEOL Ltd., Tokyo, Japan) at an acceleration energy of 20 kV. The X-ray diffraction (XRD) analysis was carried out by using an X-ray diffractometer, Rigaku model L-094 (Rigaku Corp., Tokyo, Japan). The metal ion concentration in the solution was measured by using a Shimadzu model ICPS-8100 inductively coupled plasma atomic emission spectrometer (ICP-AES) (Shimadzu Corp., Kyoto, Japan).

The cyclic voltammogram was recorded to qualitatively investigate the electrochemical property of tested samples. The working electrode (WE) was prepared by grinding 2.0 mg of adsorbent with 1.0 mg of teflonized acetylene black (TAB-2) with works as a conductive binder. The equipment was set up by combination of WE together with a Ag/AgCl electrode as the reference electrode (RE) and a Pt plate as the counter electrode (CE) and it was carried out in 1.0 M (M = mol/L) $NaNO_3$ solution (electrolyte solution). The measurement was conducted with a two-electrode configuration using a model HSV-100 Automatic Polarization System (Hokuto Denko Corp., Tokyo, Japan) at a scan speed of 50 mV/s.

Preparation of the carbonized adsorption gel

Dried sample of the residue of *Pseudochoricystis ellipsoidea*, the microalga, generated in the biofuel extraction process was kindly provided by DENSO CORPORATION, Aichi, Japan, to be employed as the raw material of the adsorption gel. This sample was carbonized by the same method proposed by Chand et al. [12,13] as follows. Five g of dried sample was put in an alumina crucible, which was then put in a larger crucible and covered with graphite powder and capped with an alumina cover in order to avoid oxidation. This crucible was placed in a muffle furnace (Nitto Kagaku Co, Ltd., Aichi, Japan) for carbonization at heating rate of 5°C/min. Temperature was kept constant at 800°C for 1 h for carbonization. Then, the temperature was slowly lowered to the ambient temperature. The product was washed with distilled water and followed by hot water until the pH of filtrate reached neutral pH and was dried in a convection oven at 70ºC for 2 days. The obtained carbon material was ground and sieved to uniform particle size before adsorption test. Thus, prepared carbon material was abbreviated as CMR (carbonized microalgal residue), hereafter. It was compared with a couple of other carbon materials, a commercially available activated carbon purchased from Wako Pure Chemical Industries, Ltd., Japan,

which is termed as Wako activated carbon, hereafter, and a carbonized orange waste prepared by the same method from a dried sample of orange juice residue kindly donated from JA Beverage Saga Co. Ltd., Japan.

Batch-wise adsorption tests

All adsorption tests were carried out batch-wise to observe the adsorption behavior of various metal ions on the tested adsorbents from individual metal solutions at varying HCl concentrations. Mixtures of 20 mg of adsorbent and 15 mL of 0.2 mM metal solutions were prepared in sample vials, which were then shaken in a thermostated incubator (THOMAS AT24R) at 30°C for 24 h to attain equilibrium. Then, the samples were immediately filtered and the metal ion concentrations in the filtrate were measured. The percentage adsorption was calculated according to the following equation.

$$\text{Adsorption (\%)} = [(C_i - C_e)/C_i] \times 100 \qquad 1$$

where C_i and C_e are the initial and equilibrium concentrations of the tested metal ion (mM), respectively. The amount of metal adsorbed on the adsorbent (q) was calculated according to the following equation.

$$q = [(C_i - C_e)/M] \times V \qquad 2$$

where V is the volume of the test solution (L), and M is the dry weight of the adsorbent (g).

Adsorption isotherm tests were carried out by shaking 10 mg of adsorbent together with 10 mL of test solutions containing varying concentrations of Au (III) in 0.1 M HCl in a thermostated incubator at 30°C for 100 h to attain equilibrium.

Adsorption rate of Au (III) on the carbonized microalgae was measured at varying time intervals at 30, 40 and 50°C from 0.1 M HCl containing 0.6 mM Au (III).

Results and Discussion

Characterization of the carbonized microalgal residue

The infrared (IR) spectra of the original microalgal residue, the feed material, and CMR as well as WAKO activated carbon are shown in Figure 1 for comparison. In this figure, the spectrum of the original microalgal residue indicates the presence of O–H stretching at 3313 cm^{-1} and C–H stretching at 2910 and 2842 cm^{-1}. The sharp absorption at 1623 cm^{-1} indicates the presence of C=O groups. These spectra well described the presence of various functional groups in microalgal residue. On the other hand, the spectra of CMR indicate that most of OH groups and other functional groups existed in the microalgal residue were disappeared by heating during the carbonization process, whilst the presence of hydroxyl group was not observed in WAKO activated carbon.

From the elemental analysis, it was found that microalgal residue, the feed material, contains 6.33% H, 43.6% C, 4.04% N, and 46.1% O, while CMR contains 0.56% H, 49.8% C, 4.50% N, and 45.2% O as its major elemental compositions. The product yield of the carbonized microalgae was approximately 30% of the initial weight of the raw material.

From the measurement using scanning electron microscope (SEM) of CMR shown in Figure 2, it was observed that the morphological surface of CMR has a high degree of porosity which was considered to be well developed by the carbonization process.

From the measurement of the adsorption-desorption isotherms of nitrogen on CMR and WAKO activated carbon shown in Figure 3,
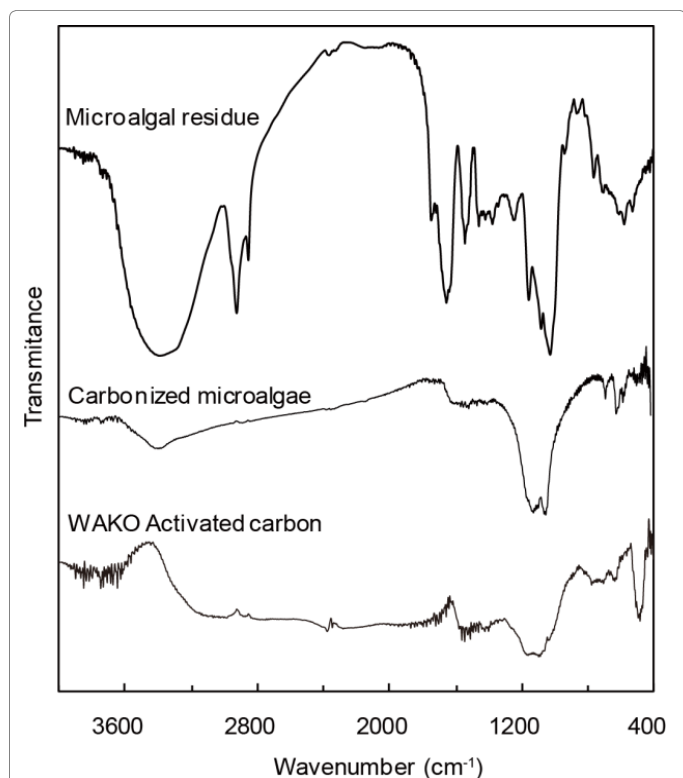
Figure 1: FT-IR spectra of the microalgal residue and CMR as well as WAKO activated carbon.
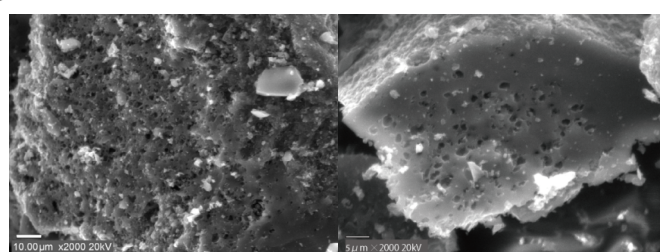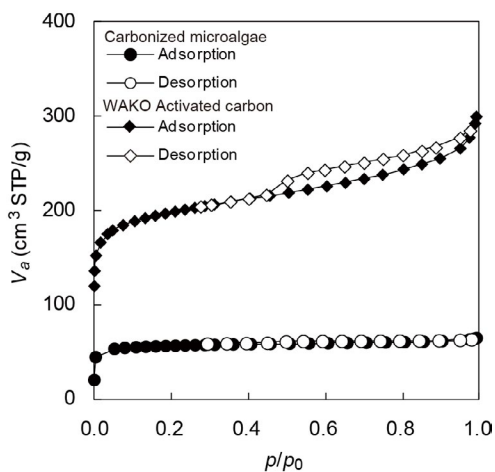

Figure 2: SEM images of CMR.

Figure 3: Nitrogen adsorption-desorption isotherms of CMR and WAKO activated carbon; V_a=Volume of N_2 adsorbed; p/p_0=Partial pressure of N_2.

it was found that the adsorption isotherms for both samples suggest the characteristic of microporous materials, while small hysteresis loop observed in the adsorption-desorption isotherms indicates the existence of mesopores [14,15]. The surface area of CMR was measured as approximately 204 to 272 m^2/g, whilst that of WAKO activated carbon is 704 to 900 m^2/g. Pore volumes of CMR and WAKO activated carbon were measured as 0.02 and 0.20 cm^3/g, respectively. Figure 4 shows the pore size distribution of CMR and WAKO activated carbon. It is obvious that the majority of pores of CMR are microporous having average pore size of 1.29 nm which is very close to that of WAKO activated carbon (1.21 nm).

Electrochemical property was investigated by recording cyclic voltammograms curves. Figure 5 shows the cyclic voltammograms curves of CMR and WAKO activated carbon. Both curves represent rectangular-like shapes without any redox current peaks on both positive and negative sweeps in the potential range, indicating good capacitive behavior similar to the supercapacitor [14,15]. In addition, the voltammetric charges on the positive and negative sweeps were approximately equal, which demonstrates that the charge-discharge behavior was highly reversible [16-18].
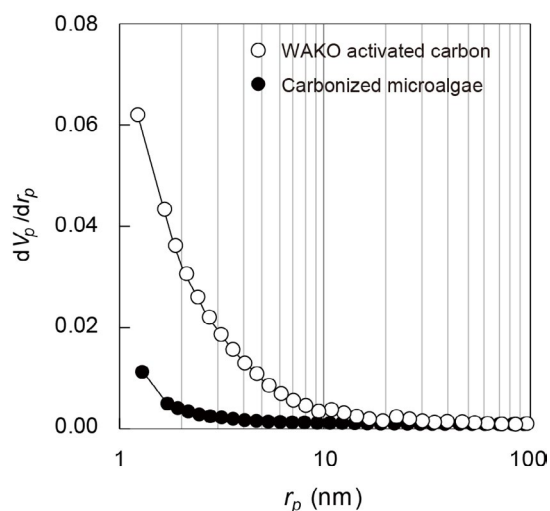

Figure 4: Pore size distribution diagrams of CMR and WAKO activated carbon. V_p: pore volume of adsorbent, r_p: pore radius of adsorbent.
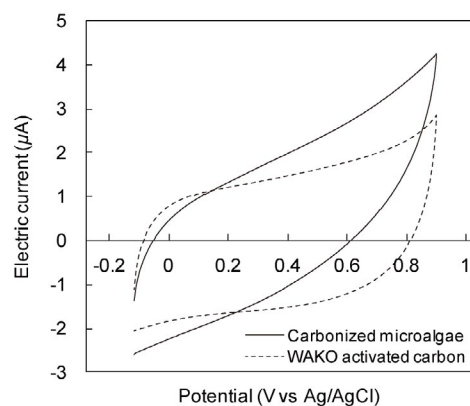

Figure 5: Cyclic voltammograms of CMR and WAKO activated carbon. Conditions: RE=Ag/AgCl, CE=Pt plate, electrolyte solution=1.0 M $NaNO_3$, scan speed=50 mV/s.

Effect of hydrochloric acid concentration on the adsorption of metal ions

In acidic chloride medium, Au (III), Pd (II), and Pt (IV) give rise to stable anionic chloride complexes such as $AuCl_4^-$, $PdCl_4^{2-}$, and $PtCl_6^{2-}$, respectively, while majority of base metal ions like Ni (II), Zn (II), and Cu (II) exist as cationic or neutral species and the concentration of anionic chloro-complexes is significant only at higher chloride concentration in the case of Fe (III). Figures 6a-6d show % adsorption of some precious and base metal ions on microalgal residue as it is, CMR, carbonized orange waste, and WAKO activated carbon, respectively, at varying HCl concentration.

From the comparison of Figures 6a and 6b, it is apparent that adsorption of Au (III) was greatly enhanced by the carbonization. Further, CMR exhibits high selectivity for Au (III) whereas adsorption of Pd (II) and Pt (IV) was much less than that of Au (III) over the whole HCl concentration range. Under the same experimental conditions, a group of base metal ions such as Ni (II), Fe (III), Zn (II), and Cu (II) were inert to be adsorbed on CMR, indicating high affinity and selectivity of this adsorbent towards precious metal ions.

From the comparison with the carbonized orange waste, it is seen that the similar adsorption behavior was observed for both carbonized materials prepared by the same method. On the other hand, although WAKO activated carbon exhibits high affinity for Au (III) over a wide range of HCl concentration, it exhibited higher degree of adsorption also for Pd (II) and Pt (IV) than CMR and carbonized orange waste, suggesting that the selectivity for Au (III) over Pd (II) and Pt (IV) is higher for CMR and carbonized orange waste than WAKO activated carbon. On the other hand, adsorption of base metals such as Fe(III) is insignificant over the whole concentration range of HCl on all tested adsorbents except for WAKO activated carbon.

It is well known that the carbon surfaces of activated carbon are composed of graphene layer surfaces and functional groups on the edges. The functional groups on the activated carbon are mostly of oxygen functional groups, i.e., carboxylic, lactonic, phenolic, carbonyl, and ether groups. Although, the adsorption of organic compounds on activated carbon is mostly controlled by physical adsorption due to its porous characteristics, the majority of adsorption of ionic species like metal ions is considered to be governed by surface functional groups such as hydroxyl and carboxyl groups [19]. Chand et al. reported that the high selectivity of barley straw and rice husk carbons to precious metal ions is attributable to its positively charged surface, on which negatively charged species are adsorbed by electrostatic interaction [12]. In the cases of base metal ions, due to unfavorable cationic and neutral species, their adsorption on the carbonized materials is nearly negligible.

Adsorption isotherm of Au(III) on the carbonized microalgae

Adsorption isotherm of Au (III) on CMR at 30°C is depicted in Figure 7, which shows that the amount of adsorbed Au (III) increases with increasing Au (III) concentration and tends to approach the plateau region at high concentration of Au (III), suggesting that the Langmuir type adsorption takes place in this adsorption system. From this plateau region, the maximum loading capacity of Au (III) on CMR was evaluated as 1.20 mmol/g or 236 mg/g. Under the same conditions, the maximum loading capacities of WAKO activated carbon and carbonized orange waste for Au (III) was evaluated as 1.67 and 0.70 mmol/g, respectively. Table 1 lists the comparison of loading capacities of various carbonized materials for Au (III). Although the maximum loading capacity on CMR is lower than WAKO activated carbon, it is higher than other carbonized biomass wastes such as the carbonized orange waste and rice husk carbon.

Observation of the formation of metallic gold particles

In the previous work on the adsorptive recovery of Au (III) using waste microalgal gel prepared by the treatment in boiling sulfuric acid, we found the formation of metallic gold by means of the observation
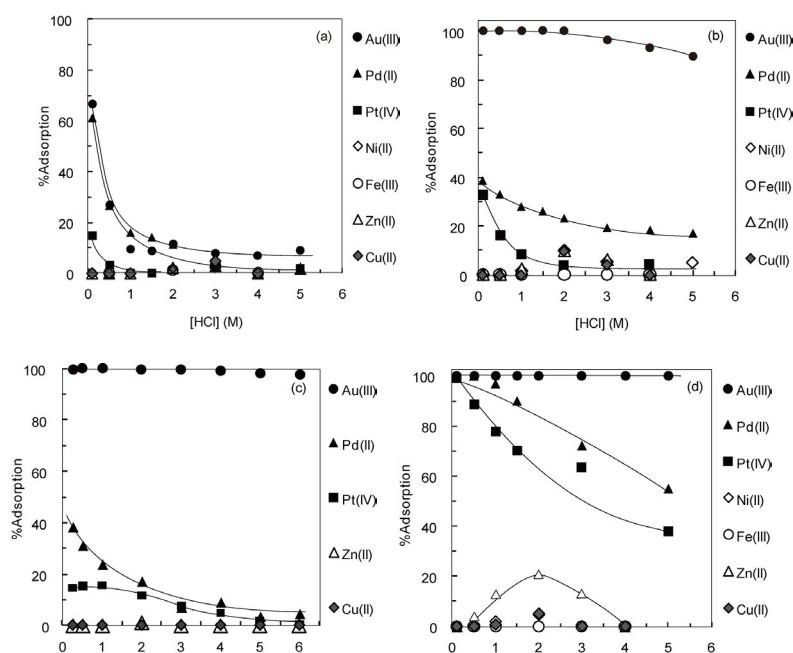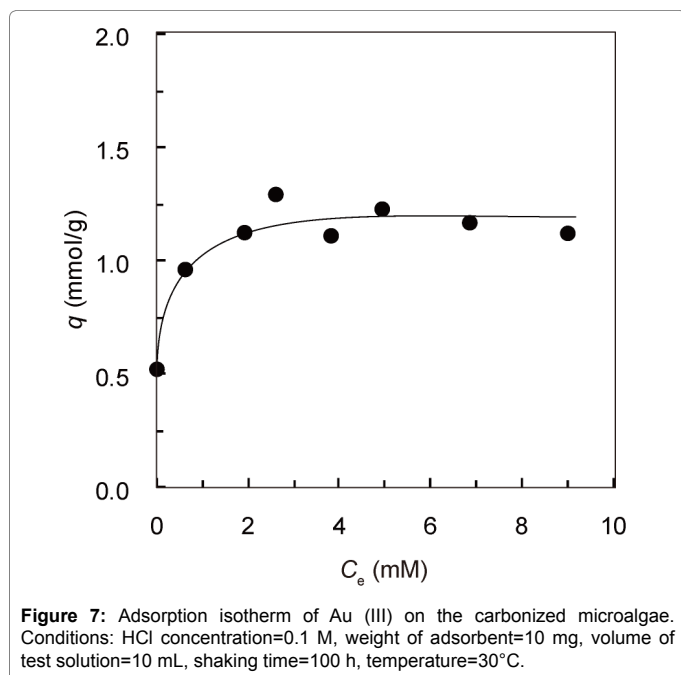

Figure 6: Effect of HCl concentration on the adsorption of various metal ions on (a) microalgal residue as it is, (b) CMR, (c) carbonized orange waste, and (d) WAKO activated carbon. Conditions: metal concentration=0.2 mM, dry weight of adsorbent=20 mg, volume of test solution=15 mL, shaking time=24 h, temperature=30°C.

Figure 7: Adsorption isotherm of Au (III) on the carbonized microalgae. Conditions: HCl concentration=0.1 M, weight of adsorbent=10 mg, volume of test solution=10 mL, shaking time=100 h, temperature=30°C.

Figure 8: (a) Image of the optical microphotograph of CMR with deposited metallic Au on its surface taken at 2000X magnification. (b) SEM image of CMR with deposited metallic Au.

| Adsorbent	Loading capacity (mmol/g)	Condition	References
Microalgal residue as it is	0.34	0.1 M HCl	This work
CMR	1.2	0.1 M HCl	This work
Orange waste carbon	0.7	0.1 M HCl	This work
WAKO activated carbon	1.67	0.1 M HCl	This work
Rice husk carbon	0.76	0.1 M HCl	[12]
Barley straw carbon	1.47	0.1 M HCl	[12]
Heated immobilized *C. vulgaris*	0.14	pH 3.0	[20]
Heated rice husk	0.26	pH 3.0	[20]
Activated carbon (Sigma C-5261)	0.37	pH 3.0	[20]

Table 1: Comparison of maximum loading capacities of various carbonized materials for Au (III).

by optical microscope and SEM as well as XRD [10]. Consequently, the formation of metallic gold was confirmed also in this system by means of SEM and XRD. The optical microphotograph shown in Figure 8a and a SEM image shown in Figure 8b clearly reveal the formation of metallic gold deposited on the surface of CMR. It is apparent from these figures that Au(III) was reduced to metallic Au (0) during the adsorption also under the present experimental conditions according to the following equation.

$$AuCl_4^- + 3e^- = Au (0) + 4Cl^- \quad E^0 = 1.00 \text{ V vs SHE} \qquad 3$$
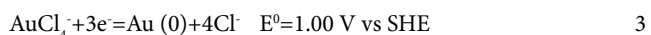
Based on these results, it can be concluded that CMR is an excellent electrical conductor, it functions as the electrode of redox reaction in the adsorption of Au (III) from solution. In the same way as electrochemical cementation reduction, there are two respective half-cell areas on the carbon surface. In the cathodic region, Au (III) and Au (I) consume electrons and are reduced to its metallic state (Au (0)).

Figure 9 shows the XRD pattern of CMR after the adsorption of Au (III), which indicates the presence of four identical peaks at 38, 44, 64, and 77° 2θ, which are assigned to Au (111), Au (200), Au (220), and Au (311) of fcc (face-centered cubic) gold crystal phase, respectively.

Figure 9: XRD pattern of CMR after Au (III) adsorption.
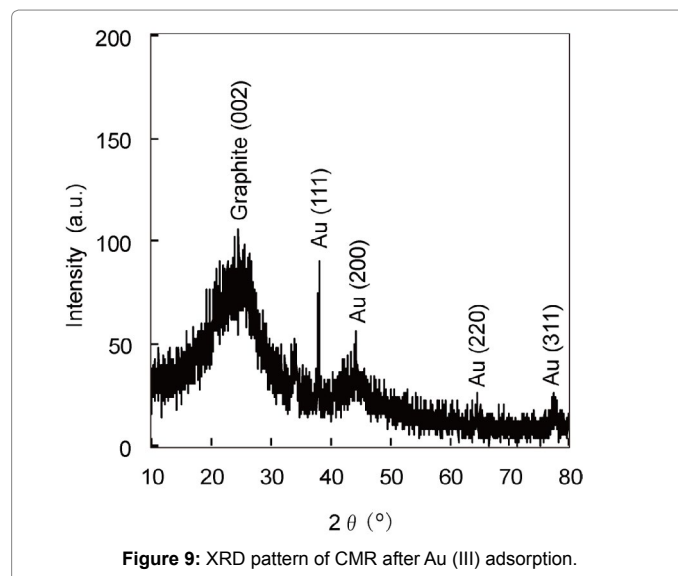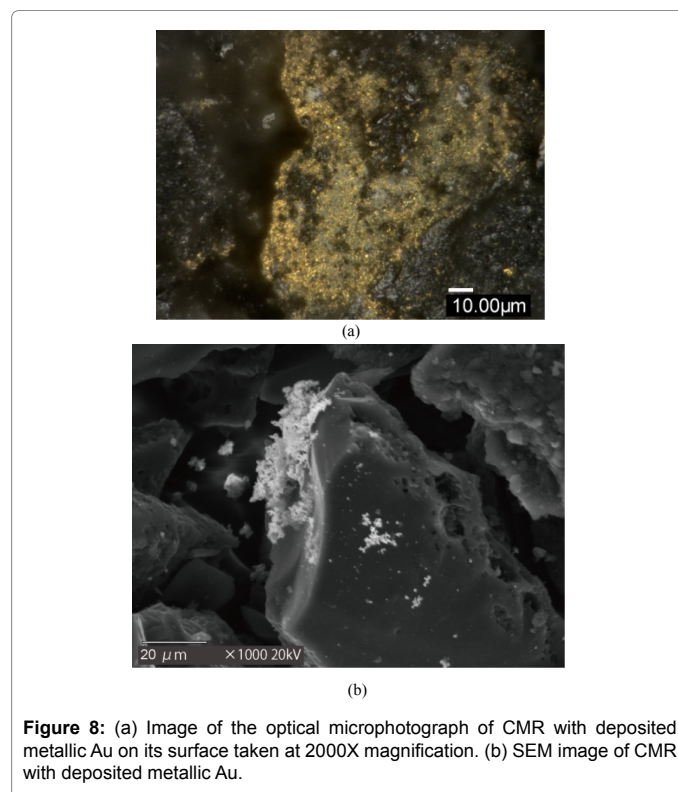
The standard reduction potentials for $AuCl_4^-$ species, the predominant species of Au (III) in this study, is 1.00 V which is relatively higher than other precious metal complexes like $PdCl_4^{2-}$ (0.62 V) and $PtCl_6^{2-}$ (0.68 V). Unlike the case of Au adsorption from alkaline cyanide solution by the activated carbon [20,21], Au adsorption from acidic halide solution involves the electrochemical reduction. This is attributable to the relative instability of $AuCl_4^-$ complexes and the ability of the activated carbon to function as a reducing agent. Figure 10 shows a comparative cyclic voltammograms of CMR before and after saturated adsorption of Au (III). It is obvious that the positive and negative sweep of CMR becomes notably small after Au (III) adsorption, which
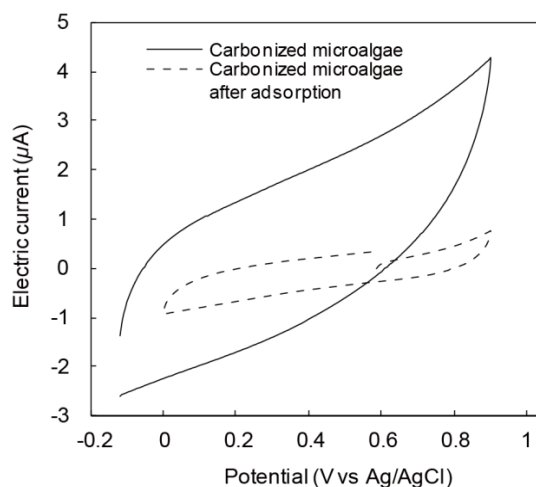
Figure 10: Cyclic voltammograms of CMR before and after Au(III) adsorption. Conditions: RE=Ag/AgCl, CE=Pt plate, electrolyte solution=1.0 M NaNO$_3$, Scan speed=50 mV/s.

indicates that CMR have lost their electron donation ability due to the slow redox reactivity.

Jia and Demopoulos suggested that hydroquinone and phenolic groups is mainly responsible for the redox characteristic of carbon [19]. Due to the reduction of Au (III) to Au (0), hydroquinone simultaneously undergoes the oxidation and form the quinones. In addition, graphene layers exhibit the characteristic of Lewis bases and behave as electron donors due to the enrichment of π-electrons on the basal plane surfaces. Therefore, it is also considered that the reduction of Au(III) to Au (0) on CMR might partly occur on the graphene layer surfaces.

Adsorption kinetics of Au(iii) on the carbonized microalgae

Since it was observed to take long time to reach equilibrium in the adsorption of Au (III) on the adsorption gels prepared from various biomass wastes including the microalgal residue by treating in boiling sulfuric acid, the adsorption kinetics of Au (III) on the carbonized microalgae was investigated in detail.

Figure 11a shows the time variation of the amount of Au (III) adsorbed on CMR at varying temperatures. This observed kinetic data were interpreted according to the pseudo-first order rate equation described by Equation 4 as depicted in Figure 11b.

$$\ln (C_t/C_i) = -k_1 t \qquad 4$$

where C_t and C_i are the concentration of Au (III) in the aqueous phase at time (t) and its initial concentration, respectively, while k_1 is the pseudo-first order rate constant of the adsorption reaction.

As expected from Equation 4, the plots lie on straight lines corresponding each temperature in this figure. From the slopes of each straight lines, the pseudo-first order reaction rate constants were evaluated at each temperature and plotted according to the Arrhenius equation described by Equation 5 as shown in Figure 11c to evaluate the activation energy (E_a).

$$\ln k = \ln A - (E_a/RT) \qquad 5$$

where A is the frequency factor and R is the ideal gas constant (8.314 J/(mol·K)).

As expected from Equation 5, the plots in Figure 11c lie on a straight line, from the slope of which the activation energy in this adsorption reaction was evaluated as 33.7 kJ/mol. This value is in the range of interaction controlled for the chemical adsorption (30-120 kJ/mol) according to Saeed et al. [22]. Similar to other heterogeneous reactions between solids and fluids, it is inferred that the process of gold chloride adsorption on activated carbon includes the diffusion of AuCl$_4^-$ through the liquid film surrounding the carbon particles followed by the reduction of Au (III) to Au (I) and finally to metallic gold on the carbon surface [19].

Conclusion

It can be concluded that CMR prepared in the present work was effective for the selective recovery of Au(III) from acidic chloride media. The adsorption of Au (III) is nearly independent of HCl concentration, which is an advantage for the practical applications. The reduction of Au (III) to metallic Au (0) was found to be involved in the adsorption of Au (III) on the surface of CMR. The results of the present work suggest a promising application of CMR prepared by a simple treatment for the selective and effective recovery of Au (III).
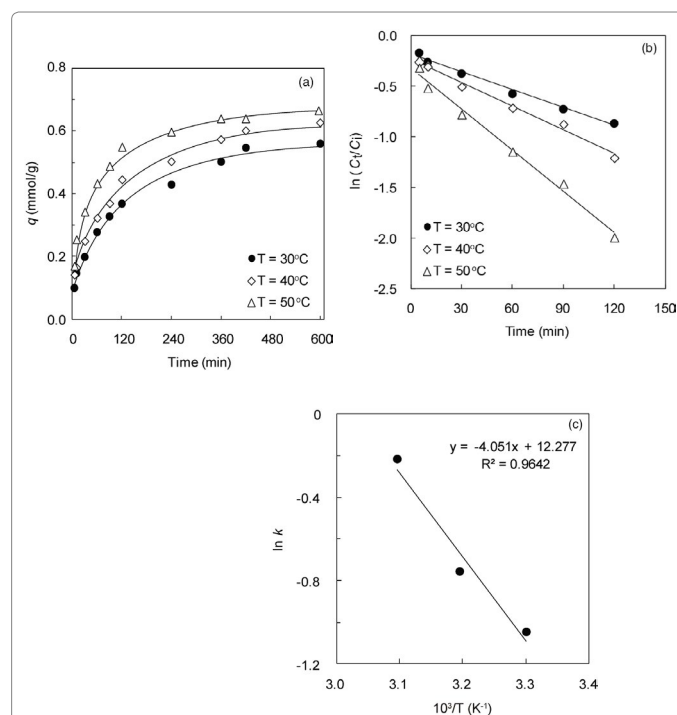
Figure 11: Kinetic aspect of the adsorption of Au (III) on CMR. Conditions: weight of adsorbent=10 mg, volume of test solution=10 mL, initial Au (III) concentration=0.6 mM, HCl concentration=0.1 M. (a) Time variation of amount of Au (III) adsorbed on CMR. (b) Plots of the kinetic data according pseudo-first order rate expression expressed by Equation 4. (c) Arrhenius plot of Au (III) adsorption on CMR.

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