

# Selective recovery of Au(III), Pd(II), and Ag(I) from printed circuit boards using cellulose filter paper grafted with polymer chains bearing thiocarbamate moieties

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**Abstract** This paper describes selective recovery of Au(III), Pd(II), and Ag(I) from waste printed circuit boards (WPCBs) by CFP-*g*-PHCTMA, an adsorbent based on cellulose filter paper (CFP) grafted with polymer chains bearing thiocarbamate moieties. As a preliminary study, the adsorption kinetics and the effect of acid concentration on the adsorption processes were studied in detail. The adsorption capacity for Au(III) and Pd(II) was not affected significantly by acid concentration, whereas that for Ag(I) was decreased under higher acid concentration. The adsorption proceeded smoothly, and reached equilibria for Pd(II) and Ag(I) within 90 min, and for Au(III) within 150 min. The selective adsorption toward these precious metal ions was maintained even in multi-element solutions. Only Ag(I), Pd(II), and Au(III) were adsorbed from a mixture containing Ag(I), Cu(II), Zn(II), Ni(II), Co(III), V(V), Cr(III), Fe(III), Mn(II), Cd(II), Au(III), Pd(II), Pt(IV), Ir(III), Os(III), Ru(III), and Rh(III). Encouraged by the fast and selective adsorption ability, the recovery of Au(III), Pd(II), and Ag(I) by CFP-*g*-PHCTMA was studied for a WPCB leachate in aqua regia. These precious metals were selectively adsorbed with negligible adsorption of coexisting ions contained in excess. This study demonstrates that this cellulose-based CFP-*g*-PHCTMA is an eco-friendly and economically viable adsorbent for the selective adsorption of precious metals.

## 1 Introduction

In recent decades, the technological innovations on electric and electronics equipment (EEE) and life style changes cause production of a large amount of waste electric and electronics equipment (WEEE) in a fast rate (Rahmani et al. 2014; Wang and Xu 2015; Yoshida et al. 2016). Precious metals like gold, silver, palladium are extensively used in manufacturing of EEE. Hence, WEEE are the vital sources for recovering precious metals (Akcil et al. 2015; Chancerel et al. 2009; Cui and Zhang 2008; Gurung et al. 2013). Printed circuit boards (PCBs) are the key components of EEE and are considered as the most valuable parts among them, since they contain precious metals in higher concentrations than natural high-grade ores (Chen et al. 2013; Hagelucken and Corti 2010; Park and Fray 2009; Van Eygen et al. 2016). In addition, natural occurrences of these precious metals are limited and, in some mines, already depleted. Hence, the effective recovery of these precious metals from secondary sources like WPCBs is quite important from economic and environmental points of view.

Hydrometallurgical and pyrometallurgical techniques have been widely used to recover precious metals from waste materials. Hydrometallurgical treatments are basically preferred over pyrometallurgical treatments for the recovery of precious metals from WPCBs due to the higher selectivity and the better environmental friendliness originating from the lower waste gas, no volatile metals, and the lower energy consumption (Akcil et al. 2015). In recent years, the impact of environmental friendliness is increasing in recovery of precious metals (Adhikari et al. 2008; Gurung et al. 2014; He and Xu 2015; Monier et al. 2014a, b; Navarro et al. 1999; Sharma and Rajesh 2014; Zhou et al. 2013). The leaching course is the first step in given

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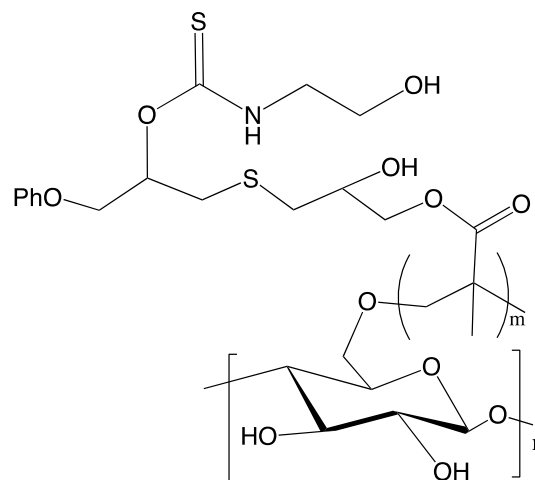
recycling chains through hydrometallurgical routes. The common leaching agents used for recovery of precious metals are aqua regia (Oh et al. 2003), alkaline cyanide (Parga et al. 2007; Warshawsky et al. 2001), chlorine (Kim et al. 2011), thiosulphate (Vinh et al. 2010), and thiourea (Orgul and Atalay 2002). Among them, aqua regia is a better leaching agent than others due to its ability for complete metal leaching from whole WPCBs along with the production of lesser toxic substances, while its strong acidity limits available adsorbents used in the next steps (Jadhav and Hocheng 2015). The leached solutions containing base and precious metals are subjected to the course of separation and purification, such as precipitation of impurities, solvent extraction, adsorption, and ion exchange to isolate and concentrate the metal of interest. The application of commercially available ion exchange resins and chelating resins is limited owing to their non-selective nature and low uptake capacity of precious metals (Chen et al. 2009; Lee et al. 2001; Ni et al. 2001). Therefore, it is important from technical, commercial, and environmental considerations that the precious metals are separated not only from the base metals with a high efficiency of recovery but also in a cost-effective method. From these points of view, it is desired to develop a new low cost metal adsorbent with high selectivity for these metals. Thus, the design of selective scavengers for precious metals would benefit from synthetic procedures involving accessible materials and facile protocols.

The selectivity to specific metal ions depends on the nature of chelating agents incorporated on the matrix substances. On account of high affinity of sulfur atoms to soft metal ions such as Au(III), Pd(II), and Ag(I), the selective recovery of these precious metals by chelating agents containing sulfur atoms was reported by the authors (Hyder and Ochiai 2017; Ochiai et al. 2009) and other (Nagai et al. 2010). Our adsorbent, CFP-*g*-PHCTMA (Fig. 1) based on accessible cellulose filter paper (CFP) and sulfur, is highly selective to precious metals (Hyder and Ochiai 2017). We subjected CFP-*g*-PHCTMA towards selective recovery of precious metals from a WPCB leachate. The adsorption behavior toward Au(III), Pd(II), and Ag(I) were studied in detail in order to understand the selective adsorption ability. Based on the results of these fundamental experiments, we demonstrate the practical recovery of Ag(I), Pd(II), and Au(III) from the WPCB leachate in aqua regia using CFP-*g*-PHCTMA.

## 2 Experimental

### 2.1 Materials

All the chemicals used were of analytical reagent grade. CFP (Adventec 5C) (Toyo Roshi, Tokyo, Japan) was



**Fig. 1** Structure of CFP-*g*-PHCTMA

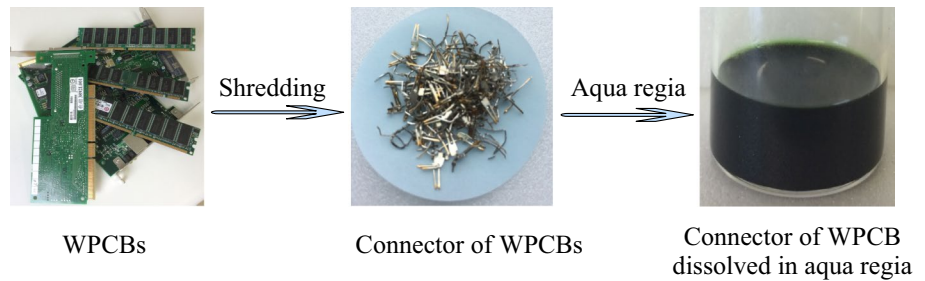
used for modification. Nitric acid, and Cu(II) and Ni(II) standard solution for ICP (1000 mg L<sup>-1</sup>) were purchased from Wako Chemicals (Tokyo, Japan). Pd(II), Au(III), and Ag(I) standard solution for ICP (1000 mg L<sup>-1</sup>), and multi-elemental solution (1) (Cu(II), V(V), Ni(II), Co(II), Fe(III), Mn(II), Cr(III), Ag(I), Zn(II) and Cd(II) and multi-elemental solution (2) (Pd(II), Au(III), Pt(IV), Ru(III), Rh(III), Os(III), Ir(III)) for ICP (100 mg/L) were purchased from Sigma-Aldrich (St. Louis, Missouri, USA). Water was purified with MINIPURE TW-300RU (Nomura Micro Science, Kanagawa, Japan). The scavenger, CFP-*g*-PHCTMA (Hyder and Ochiai 2017), and the precursors, *O*-1-mercapto-3-phenoxypropan-2-yl *N*-2-hydroxyethylcarbamothioate (HCT) (Hyder and Ochiai 2017) and 4-(phenoxyethyl)-1,3-oxathiolane-2-thione (DTC) (Kihara et al. 1995; Ochiai and Endo 2005) was prepared per the literatures.

### 2.2 Adsorption experiment (typical procedure)

The adsorption experiment was carried out in batch mode. For adsorption experiments, CFP-*g*-PHCTMA (5 mg) was placed in a 10 mL plastic bottle containing 100 mg L<sup>-1</sup> of a metal ion solution (5 mL) at a determined acid concentration. The adsorption experiments for Au(III) were conducted using 200 mg L<sup>-1</sup> Au(III) solutions. The bottles were equilibrated at 25 °C on a thermostated shaker. Then the mixture was filtrated and the filtrate metal solution was analyzed for its residual concentration. The concentration of metal ions was determined by Perkin Elmer ELAN DRC II inductively coupled plasma (ICP) mass spectrophotometer.

The percentages of adsorption of metals were evaluated using the relations of initial and equilibrium concentrations of metal ions in solutions indicated in Eq. 1.

**Fig. 2** Leaching of metals in aqua regia from WPCBs



**Table 1** Concentrations of metals in leachate of WPCB connectors

Metals	Gold	Silver	Palladium	Copper	Iron	Nickel	Zinc	Vanadium
Concentration (ppm)	125	163	13	142,500	2650	13,000	375	200

$$\text{Percent of adsorption (\%)} = \frac{(C_i - C_e) \times 100}{C_i} \quad (1)$$

$C_i$  ( $\text{mg L}^{-1}$ ) is the initial metal ion concentration; and  $C_e$  ( $\text{mg L}^{-1}$ ) is the metal ion concentration in equilibrium.

The adsorption amounts were calculated from residual amounts of metal ions in solutions per Eq. 2.

$$Q_e = \frac{(C_i - C_e)V}{W} \quad (2)$$

$Q_e$  ( $\text{mg g}^{-1}$ ) is the adsorption capacity;  $V$  (L) is the volume of metal solution;  $W$  (g) is the mass of dry adsorbent, respectively.

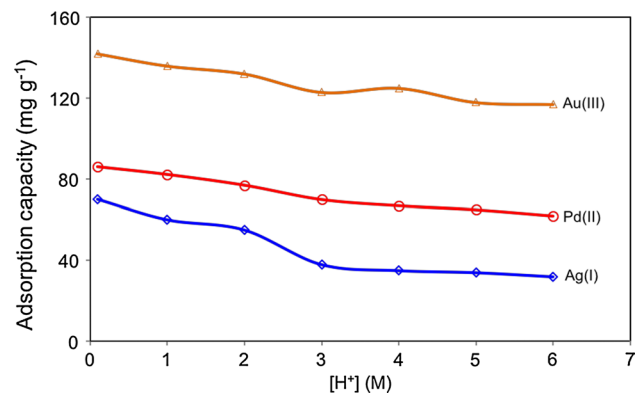
### 2.3 WPCB leaching in aqua regia

The sample of WPCB leachate was obtained as follows. Connectors were collected from WPCB, and treated with aqua regia (Fig. 2). The resulting mixture was filtrated and the filtrate was analyzed by ICP-MS after proper dilution. Detected metal ions are V(V), Fe(III), Ni(II), Cu(II), Mn(II), Zn(II), Pd(II), Au(III), and Ag(I), and the concentrations are indicated in Table 1.

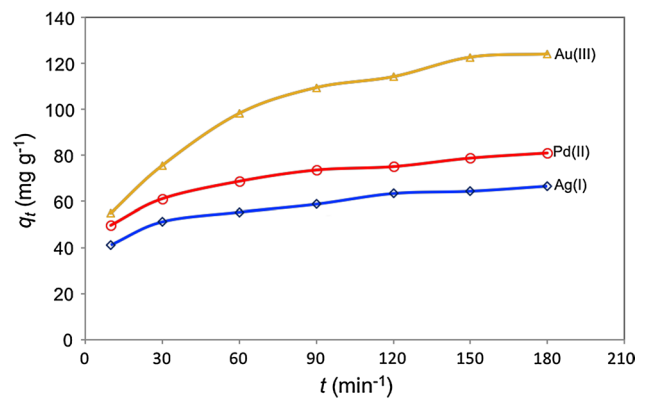
## 3 Result and discussion

### 3.1 Effect of $\text{H}^+$ ion concentration on adsorption processes

Selective adsorption of precious metals in strong acid is an important requirement for a metal scavenger applied in the practical hydrometallurgical collection of precious metals, as concentrated acids are used in metal leaching. Hence, adsorption isotherm studies under different acid



**Fig. 3** Effect of  $[\text{H}^+]$  concentration on the adsorption of Ag(I), Pd(II), and Au(III) onto CFP-g-PHCTMA. Initial concentration of Ag (I), and Pd (II) =  $100 \text{ mg L}^{-1}$ , and Au(III) =  $200 \text{ mg L}^{-1}$ , CFP-g-PHCTMA =  $1 \text{ g L}^{-1}$ , 24 h,  $25^\circ\text{C}$



**Fig. 4** Adsorption kinetics of Ag(I), Pd(II), and Au(III) on Cell-g-PHCTMA. Initial concentration =  $100 \text{ mg L}^{-1}$  (Ag(I), Pd(II)) and  $200 \text{ mg L}^{-1}$  (Au(III)), CFP-g-PHCTMA =  $1 \text{ g L}^{-1}$ ,  $[\text{H}^+] = 0.1 \text{ M}$ ,  $25^\circ\text{C}$

**Table 2** Kinetic parameters for Ag(I), Pd(II) and Au(III) adsorption by CFP-g-PHCTMA

Metals	Pseudo-first order model		
	$k_1 \times 10^{-3} \text{ (min}^{-1}\text{)}$	$Q_e \text{ (mg g}^{-1}\text{)}$	$R^2$
Ag(I)	16.27	27.33	0.9877
Au(III)	23.50	97.69	0.9810
Pd(II)	18.63	37.28	0.9848
Metals	Pseudo-second order model		
	$k_2 \times 10^{-3} \text{ (g mg}^{-1} \text{ min}^{-1}\text{)}$	$Q_e \text{ (mg g}^{-1}\text{)}$	$R^2$
Ag(I)	1.244	69.53	0.9984
Au(III)	0.340	137.6	0.9982
Pd(II)	1.049	84.47	0.9988

concentrations were carried out for Au(III) and Pd(II) in aqueous HCl, and Ag(I) in aqueous HNO<sub>3</sub> (Fig. 3). The high adsorption capacity of CFP-g-PHCTMA toward Au(III) and Pd(II) was maintained at a wide range of HCl concentration, though with slight decrements.

The trend of the decrement of the adsorption capacity for Ag(I) upon increasing the HNO<sub>3</sub> concentration was found to be significant. The decrease of the adsorption capacities can be explained by the slight protonation of sulfur atoms in the thione functional groups of CFP-g-PHCTMA as observed in adsorption of Au(III) and Pd(II) by phosphine sulphide-type chelating polymers (Sanchez et al. 2001) and the competition between the proton ion and Au(III), Pd(II), and Ag(I) for the exchange sites on the CFP-g-PHCTMA (Monier et al. 2014a).

### 3.2 Kinetics of Adsorption of Au(III), Pd (II), and Ag(I) by CFP-g-PHCTMA

Fast uptake ability is an important requirement on collection of metals from waste stream. In order to investigate the kinetics of the adsorption process, an adsorption experiment was performed using 1 g L<sup>-1</sup> CFP-g-PHCTMA for Ag(I) (100 mg L<sup>-1</sup>), Pd(II) (100 mg L<sup>-1</sup>), and Au(III) (200 mg L<sup>-1</sup>) solutions under 0.1 M acid concentrations at 25 °C (Fig. 4). The adsorption of these precious metals proceeded smoothly and the adsorbed amounts almost reached to plateaus after 50 min. The adsorption kinetics was studied using a pseudo-first order kinetic model (Eq. 3) along with its linear form (Eq. 4) and a pseudo-second model (Eq. 5) along with its linear form (Eq. 6).

$$Q_t = Q_e \left(1 - e^{-k_1 t}\right) \quad (3)$$

$$\ln(Q_e - Q_t) = \ln Q_e - k_1 t \quad (4)$$

$$Q_t = \frac{k_2 Q_e^2 t}{1 + k_2 Q_e t} \quad (5)$$

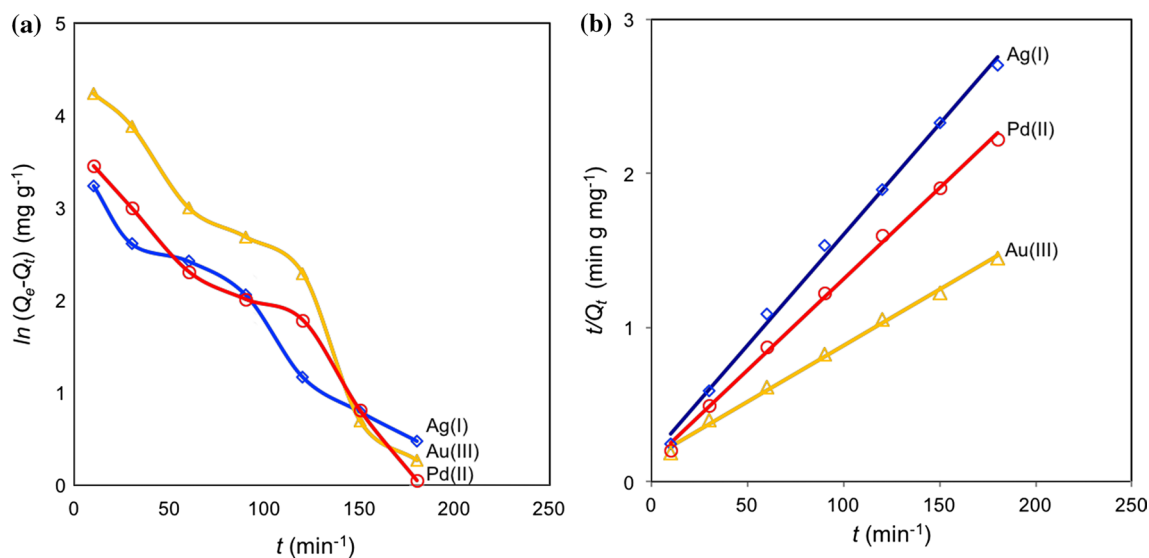
$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (6)$$

$Q_e$  (mg g<sup>-1</sup>) and  $Q_t$  (mg g<sup>-1</sup>) are the amounts of metal ions adsorbed per unit mass of the adsorbent at equilibrium and time ( $t$ ) respectively.  $k_1$  (g mg<sup>-1</sup> min<sup>-1</sup>) and  $k_2$  (dm<sup>-3</sup> mg<sup>-1</sup> min<sup>-1</sup>) are the pseudo-first order and pseudo-second order rate constants, respectively. The results of the kinetic were obtained analyzing Fig. 4. Table 2 lists the kinetic parameters of the pseudo-first order and pseudo-second order models.

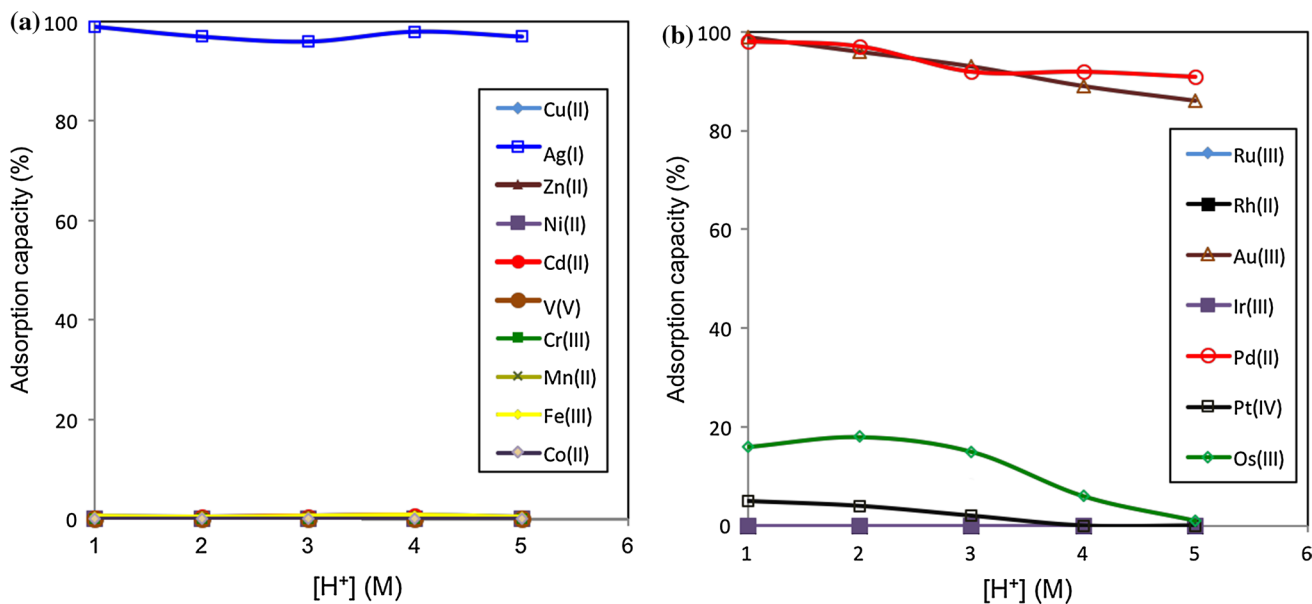
The linear plots of the pseudo-first order (Fig. 5a) and the pseudo-second order (Fig. 5b) kinetic models for the adsorption of Ag(I), Pd(II), and Au(III) on CFP-g-PHCTMA indicate that the second-order plots have better linearity, as can be confirmed by the  $R^2$  values (Table 2). As the adsorption of Ag(I), Pd(II), and Au(III) fitted the pseudo-second order model, the rate determining step of this adsorption is the chemical reaction between the ions and the adsorbent (Azizian 2004; Ho 2004, 2006; Ho and McKay 1999).

### 3.3 Selective adsorption of Ag(I), Pd(II), and Au(III) from multi-elemental solutions

Practical recovery processes of precious metals ions are carried out from multi-elemental solutions containing various base metals such as copper, zinc, and nickel in high concentration of acidic media. Accordingly, the adsorption experiments were carried out using two types of multi-elemental solutions with various acid concentrations to determine the selective adsorption behavior of CFP-g-PHCTMA even in highly acidic media (Fig. 6). Figure 6a shows the selective adsorption of Ag(I) from a multi-elemental solution (I) containing Ag(I), Cu(II), Zn(II), Ni(II), Co(III), V(V), Cr(III), Fe(III), Mn(II), and Cd(II) in HNO<sub>3</sub> aq. with the acid concentration varied from 1 to 6 M. This result indicates that CFP-g-PHCTMA adsorbs only Ag(I) from the mixture of various base metal ions without co-adsorption



**Fig. 5** Linear plots of **a** pseudo-first order kinetics and **b** pseudo-second order kinetics of adsorption of Ag(I), Pd(II), and Au(III) ions on CFP-g-PHCTMA. Initial concentration = 100 mg L<sup>-1</sup> (Ag(I), Pd(II)) and 200 mg L<sup>-1</sup> (Au(III)), Cell-g-PHCTMA = 1 g L<sup>-1</sup>, [H<sup>+</sup>] = 0.1 M, 25 °C



**Fig. 6** Effect of acid concentration on adsorption of **a** Ag(I) from multi-elemental solution (1) in HNO<sub>3</sub> aq. media, and **b** Pd(II) and Au(III) from multi-elemental solutions (2) onto CFP-g-PHCTMA

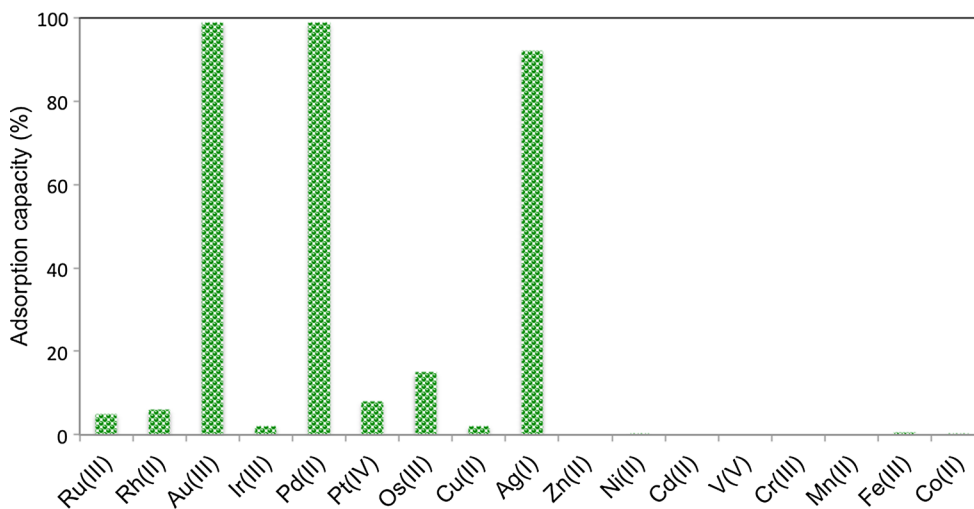
in HCl aq. Initial concentration of M<sup>n+</sup> = 100 mg L<sup>-1</sup>; CFP-g-PHCTMA = 6 g L<sup>-1</sup>; 25 °C

of other metals. Figure 6b presents the metal adsorption behavior of CFP-g-PHCTMA from a multi-elemental solution (2) containing Au(III), Pd(II), Pt(IV), Ir(III), Os(III), Ru(III) and Rh(III) in HCl aq. with the acid concentration varied from 1 to 6 M. It was observed that only Au(III) and Pd(II) was bound to the adsorbent, whereas the trace amounts of Os(III) and Pt(IV) was adsorbed and no other precious metals were captured at all by CFP-g-PHCTMA.

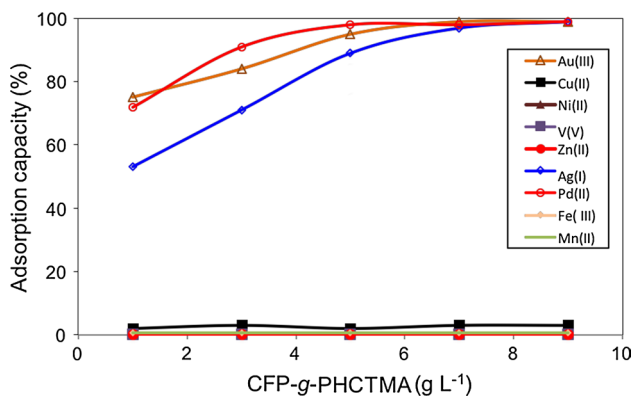
The adsorption of Os(III) and Pt(IV) was suppressed under high acid concentrations.

As PCB metals are often leached by aqua regia, 1 in HNO<sub>3</sub> media and 2 in HCl media were mixed together and the mixture was employed for the adsorption experiment with CFP-g-PHCTMA (total acid concentration 1.53 M) (Fig. 7). CFP-g-PHCTMA selectively and efficiently adsorbed Au(III), Pd(II), and Ag(I)





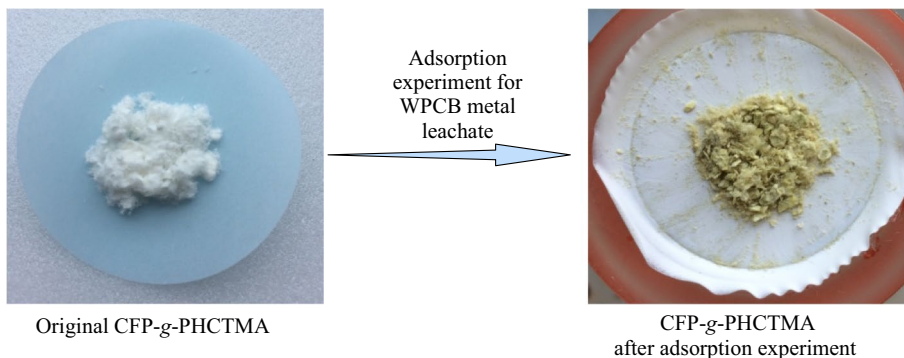
**Fig. 7** Selective adsorption of Ag(I), Pd(II), and Au(III) from a mixture of multi-elemental solution (1) and multi-elemental solution (2) onto CFP-g-PHCTMA at (HCl + HNO<sub>3</sub>) acid media. Initial concentration  $M^{n+} = 50 \text{ mg L}^{-1}$ ; CFP-g-PHCTMA =  $6 \text{ g L}^{-1}$ ,  $[\text{H}^+] = 1.53 \text{ M}$ ,  $25 \text{ }^\circ\text{C}$



**Fig. 8** Adsorption of metal ions by CFP-g-PHCTMA from WPCB connector leachate in aqua regia for 3 h at  $25 \text{ }^\circ\text{C}$

even from the mixture of the two multi-elemental solutions as well as the individual and the original multi-metal solutions.

**Fig. 9** States of CFP-g-PHCTMA before and after adsorption from WPCB metal leachate in aqua regia



### 3.4 Selective recovery of Au(III), Pd (II), and Ag(I) from WPCB leachate

On the basis of the excellent adsorptive recovery of Au(III), Pd(II), and Ag(I), we carried out collection of the precious metals from WPCB leachate with aqua regia by CFP-g-PHCTMA, which involves difficulty in selective adsorption due to the presence of various organic components and base metals such as Cu(II), Ni(II), V(V), Fe(II), and Zn(II). The leachate consists of Pd(II), Au(III), and Ag(I), and significantly higher amounts of base metals as indicated in Table 1. Figure 8 represents the adsorption behavior of metals in the WPCB metal leachate with CFP-g-PHCTMA. It was observed that Cu(II), Ni(II), Zn(II), V(V), and Fe(II) was negligibly adsorbed onto the adsorbent CFP-g-PHCTMA. By contrast, Au(III), Pd(II), and Ag(I) were completely adsorbed. The concentrations of CFP-g-PHCTMA in the metal leachate in aqua regia for the complete recovery of Au(III) and Pd(II) are  $3 \text{ g L}^{-1}$ , while that for Ag(I) is  $7 \text{ g L}^{-1}$ . This differences in the efficiencies agrees with the ability of adsorption of Cell-g-PHCTMA

towards Au(III), Pd(II) and Ag(I) observed in the single ion experiments at higher acid concentration. These amounts of CFP-*g*-PHCTMA for entire recovery of Au(III), Pd(II), and Ag(I) from the WPCB leachate is not so high considering the expensiveness of these precious metals than higher than organic materials accessible from inexpensive resources. Figure 9 shows that the color of CFP-*g*-PHCTMA was changed from white to yellow in a similar manner with the adsorption of individual metal ions (Hyder and Ochiai 2017). These results clearly demonstrated that the precious metals Au(III), Pd(II), and Ag(I) were successfully recovered by CFP-*g*-PHCTMA from the metal leachate from WPCBs. In other words, CFP-*g*-PHCTMA is a promising candidate for practical recovering of precious metals.

#### 4 Conclusion

The adsorbent CFP-*g*-PHCTMA was found to be effective for the selective recovery of Au(III), Pd(II), and Ag(I) from WPCB. The successful result originates from the specific adsorption ability of the thiocarbamate ligand and the stability under very high concentration of acid media. The high selectivity was manifested by the negligible adsorption of base metals, namely Cu(II), Zn(II), Ni(II), Co(III), V(V), Cr(III), Fe(III), Mn(II), and Cd(II), and other precious metals, namely Pt(IV), Ir(III), Os(III), Ru(III), and Rh(III). The kinetic study revealed that the adsorption rates are fast and the experimental data well fit the pseudo-second order model. This features are vital for practical recovery of precious metals, and CFP-*g*-PHCTMA is a promising adsorbent for a wide range of hydrometallurgical recovery of precious metals from various wastes.

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