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(Research Article)

Gold recovery from printed circuit boards in an acidic environment: A case study in Burkina Faso

Woulou SOMDA [1, *], Francis O. KONATE [1], Wendpagnagda COMBERE [2], Rafiatou LENGANE [1] and Arsène H. YONLI [1, 3]

[1] Laboratoire de Physique, de Chimie et de l'Environnement (LPCE), Département de Chimie, Université Joseph KI ZERBO, 03 BP. 7021 Ouagadougou 03, BURKINA FASO.
[2] Laboratoire Sciences et Technologie (LaST), Département de Chimie, Université Thomas SANKARA, 12 BP 417 Ouagadougou 12, BURKINA FASO.
[3] Centre de Recherche Yser Labs (CRYL), 11 BP 266 Ouaga CMS 11 Burkina Faso.

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Abstract

This study deal with a method for gold recovery from printed circuit boards (PCBs) obtained from WEEE collected in Burkina Faso. After grinding PCB samples and leaching with aqua regia, a solution rich in metal ions were obtained, containing notably 6.04 µg/g of gold, a concentration roughly 200 times higher than that of conventional gold ores. The study focused on the selective adsorption of Au^{3+} ions using a magnetic Fe-BTC/PpPDA MOF composite. This MOF exhibited a microporous microstructure (iodine index of about 2543.2 mg/g) and a point of zero charge (pH_{pzc}) of 5.6, promoting electrostatic interactions in an acidic environment. Batch tests demonstrated highly efficient and selective gold adsorption, with a recovery rate exceeding 98%. The Fe-BTC/PpPDA material also enables the reduction of Au^{3+} ions to metallic gold, catalyzed by the amine and hydroxyl groups in the structure. After desorption of gold-loaded composite a magnetic separation allowed a good regeneration of the adsorbent, with a recovery rate of 89% after a second cycle. Infrared spectroscopy analyses confirmed the involvement of N-H and O-H functions in the adsorption and reduction mechanism of gold ions.

Keywords: Gold; Adsorption; WEEE; Fe-BTC/PpPDA

1. Introduction

Waste from electrical and electronic equipment (WEEE) are both a major environmental challenge and an important secondary source of metals, particularly precious metals such as gold and silver [1-5]. Printed Circuit Boards (PCBs), as key components of WEEE, contain gold at levels much higher than those for natural gold ores, making them a promising alternative source for gold recovery [6-9]. However, the selective recovery of gold from WEEE remains a challenge due to the complexity of their matrix and the high content of base metals such as copper, iron, and nickel. Hydrometallurgical processes, particularly acid leaching followed by separation techniques, are among the most commonly used methods for solubilizing metals[10]. Aqua regia is recognized for its effectiveness in dissolving precious metals, but it generates multi-ionic solutions that require efficient selective separation steps[2; 4; 7].

In recent years, advanced porous materials, especially Metal Organic Frameworks (MOFs), have attracted an increasing interest for adsorption applications due to their high specific area, controlled porosity, and ability for chemical functionalization. Composite MOFs incorporating conductive polymers showed particular promising properties for both adsorption and reduction of metal ions[11-12].

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[*] Corresponding author: Woulou SOMDA

The present study aims to evaluate the effectiveness of a Fe-BTC/PpPDA composite MOF for the recovery of gold from solutions obtained from acid leaching of printed circuit boards, in the specific context of Burkina Faso. The influence of the material physicochemical properties, notably porosity, pH_{pzc}, and functional groups on selective adsorption and reduction of Au^{3+} ions is examined, as well as its regeneration and reuse.

2. Material and methods

2.1. Collection and treatment of WEEE

The treatment of printed circuit boards (PCBs) from WEEE aims to enrich the metallic particles to facilitate their recovery. In this study, printed circuit boards consisted in mobile phone boards, computer motherboards, RAM modules and processors. These boards were obtained from an association called “Association Burkinabè pour la Promotion des Emplois Verts” (ABPEV) following a previous study [1]. Collection, sorting and dismantling were carefully carried out until the printed circuit boards were obtained. The obtained printed circuit boards were crushed to reduce their particle size and facilitate dissolution. This particle size reduction involved shredding the PCBs to obtain particles smaller than 20 mm. Subsequently, we ground them further to reduce the particle size, and grinding produced particles equal to or smaller than 1.5 mm.

2.2. Leaching of crushed printed circuit boards

The leaching solution was aqua regia. In a 200 mL beaker a mixture of 2 g of the various crushed cards were melt with 100 mL of an aqua regia solution (a mixture of HCl and HNO_3 in a volumetric molar ratio of 3:1). The mixture was heated to 70°C at atmospheric pressure under magnetic stirring. After 6 hours, the leachate was filtered and rinsed with acidified water (5% HCl). The resulting solution was rich in metal ions and used for gold recovery.

2.3. Iodine index

The pretreatment of Fe-BTC/PpPDA MOF and the standard iodine solution used for iodine adsorption was carried out in accordance with ASTM D4607-94 standard. 1 g sample of Fe-BTC/PpPDA was weighed and mixed with 100 mL of iodine solution (0.05 M) in an Erlenmeyer flask. The mixture was then stirred magnetically for 30 minutes; then centrifuged to separate the Fe-BTC/PpPDA from the iodine solution. 10 mL of the supernatant was withdrawn with a syringe and diluted with 100 mL of distilled water in a 250 mL beaker. This diluted solution was titrated with a sodium thiosulfate solution (0.1 M).

2.4. Methylene blue index

The pretreatment of the Fe-BTC/PpPDA MOF and the methylene blue solution was carried out in accordance with the NF P 94-068 standard. 1 g of Fe-BTC/PpPDA was weighed and placed in a 200 mL Erlenmeyer flask, then 50 mL of distilled water was poured on the MOF. Using a burette, 5 mL of the methylene blue solution was added and stirred for 5 minutes. A drop of the mixture was taken with a micropipette and placed on filter paper to observe the color of the spot. If the spot was colorless, 2 mL of the methylene blue solution was added each time, and a sample was taken to observe the color of the spot on filter paper, until the spot turns blue. The final volume of methylene blue added was noted.

2.5. Determination of the pH_{pzc} of Fe-BTC/PpPDA

The process consisted in introducing 20 mL of a $NaNO_3$ solution (0.1 M) into ten beakers, and the pH was adjusted with a NaOH solution (0.1 M) and a HNO_3 solution (0.1 M), varying between 3 and 12. Then, 20 mg of the Fe-BTC/PpPDA MOF was added to each beaker, and the mixture was homogenized and mechanically stirred. After 30 minutes, the mixtures were filtered, and the pH in each beaker measured using a pH-meter. The curve $\Delta pH = f(pH_{initial})$ was plotted and the intersection of the curve with the x-axis determined the pH_{pzc}.

2.6. Adsorption of Au^{3+} ions in batch

In a beaker containing 100 mL of a WEEE solution whose metal ion content had been previously determined, 1 g of Fe-BTC/PpPDA was added and mechanically stirred for 30 minutes and vacuum filtered through a Whatman paper. The filtrate was collected for analysis by a Perkin Elmer 5500 atomic absorption spectrometer. The adsorbent (Fe-BTC/PpPDA) loaded with gold was submitted to a magnetic separator to recover gold. Regenerated adsorbent was then reused another adsorption cycle.

The amount of gold loaded on MOF composite was determined according to Equation 1:

$$q_e = (c_0 - c_e)\frac{V}{m} \qquad \textbf{Equation 1}$$

Where:
q_e: Amount of sorbate loaded on the sorbent (mg/g);
C_0: Initial concentration of the solution in mg/L;
C_e: Equilibrium concentration of the solution in mg/L;
V: Volume of the solution in L;
m: Mass of the sorbent in g.

The percentage of adsorbed gold was determined by Equation 2.

$$\%\,Au = \frac{C_0 - C_e}{C_0} \times 100 \qquad \textbf{Equation 2}$$

Where:
% Au: Gold percentage on the composite
C_0: Initial gold concentration in mg/L
C_e: Gold concentration at equilibrium in mg/L.

The desorption percentage was calculated from the ratio between the amount of Au^{3+} ions desorbed and the amount of Au^{3+} ions adsorbed within the composite for one cycle (Equation 3).

$$\%DES = \frac{Amount\ of\ Au^{3+}\ ion\ after\ desorption}{Amount\ of\ Au^{3+}\ ion\ adsorbed} \times 100 \qquad \textbf{Equation 3}$$

It was then possible to calculate the regeneration efficiency (%RE) per cycle (Equation 4):

$$\%RE = \frac{q_i}{q_1} \times 100 \qquad \textbf{Equation 4}$$

With q_1 and q_i, the adsorption capacities (mg/g) at cycle 1 and cycle i, respectively.

3. Results and discussion

The treatment and leaching of WEEE with aqua regia allowed the dissolution of several metal ions at different concentrations, including Au^{3+} ions. Table 1 gives the concentrations for the different metals.

Table 1 Concentrations of metals in WEEE

Metallic elements	Concentrations in mg/L
Ag	0.026
Au	1.51
Zn	4.62
Fe	26.70
Ni	26.12
Cu	927.27

Aqua regia allowed the leaching of both base metals and precious metals. Fe, Ni and Cu were the most abundant metals in the mixture [1, 3]. The concentrations of precious metals (Ag and Au) remain relatively low compared to those of other metal ions. However, when comparing the gold content in WEEE and in gold ores, there is about 200 times more gold in WEEE than in gold ores[1]. Copper has a very high content compared to the other metals, which could limit the recovery of precious metals like gold.

3.1. Iodine index and methylene blue index

The iodine and methylene blue indices provide an indication of the porosity and adsorption capacity of the adsorbent [13-17]. If the methylene blue index is low while the iodine index is high, that suggests that the adsorbent is microporous [18-20]. In this study, the iodine index of Fe-BTC/PpPDA was 2543.2 mg/g, and methylene blue index was 56 mg/g. Iodine has a diameter of approximately 0.6 nm, and its adsorption by Fe-BTC/PpPDA with such a high index indicates that the Fe-BTC/PpPDA material is microporous. Methylene blue, a bigger molecule [21], could not easily go inside the pores of Fe-BTC/PpPDA, explaining the low methylene blue index.

3.2. Determination of pH_{pzc} of Fe-BTC/PpPDA

The pH_{pzc} of an adsorbent provides crucial indications on its behavior in solution [22]. The type of interactions between the adsorbent and the adsorbate can be determined by knowing the charge of the adsorbate and the pH of the solution. Thus, if the pH_{pzc} is higher than the pH of the working solution, the surface of the adsorbent will be charged positively. Conversely, if it is lower, the adsorbent surface will be charged negatively [21, 23]. The pH_{pzc} of Fe-BTC/PpPDA was about 5.6 (Figure 1). The iodine solution was prepared at a pH = 5.3; under these conditions, the surface of Fe-BTC/PpPDA was positively charged, favoring the adsorption of I_3^- ions.

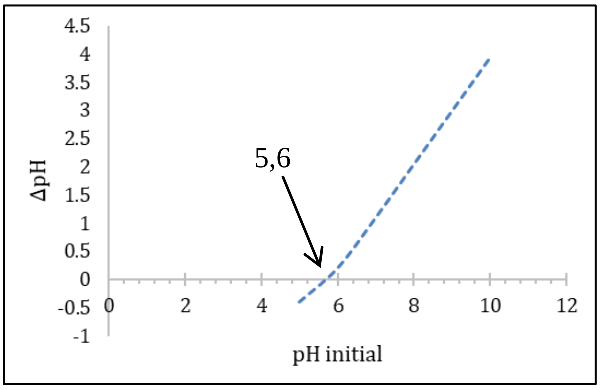


Figure 1 pH_{pzc} of Fe-BTC/PpPDA

3.3. Batch adsorption of metals on Fe-BTC/PpPDA

Batch adsorption of metal ions from dissolved WEEE showed a very high adsorption rate for Au^{3+} ions and Ag ions. A slighter adsorption for the other ions (copper, zinc, iron…) occurred[12; 24]. Figure 2 present the variation of metal concentrations in solution before and after adsorption on Fe-BTC/PpPDA MOF composite.

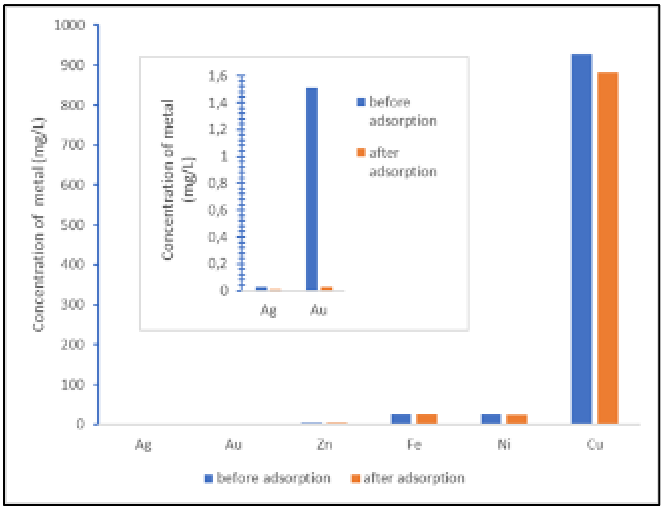


Figure 2 Adsorption of metal ions in a WEEE solution by Fe-BTC/PpPDA

The Fe-BTC/PpPDA MOF was able to predominantly adsorb Au^{3+} ions. The percentage of adsorbed gold was over 98%, indicating a good and preferential adsorption of Au^{3+} ions on Fe-BTC/PpPDA MOF composite. As the solution of ion metals had a pH lower than the pH_{pzc} of Fe-BTC/PpPDA, the surface of the MOF was positively charged, favoring the adsorption of metal ions by electrostatic interactions. MOF porosity and various chromophore groups such as amine functions allowed specifically the adsorption of gold more gold than other metal ions. Table 2 present the amounts and rates of metals adsorbed on MOF composite from WEEE solution.

Table 2 Amounts and rates of metals adsorbed on composite MOF Fe-BTC/PpPDA

	Ag	**Au**	**Zn**	**Fe**	**Ni**	**Cu**
Q_e (mg/g)	0.37	37,00	1.70	2.50	55.30	1150.53
% adsorbed	57.69	98.01	1.47	0.37	8.47	4.96

The Fe-BTC/PpPDA MOF not only allows the adsorption of metal ions but also their reduction into metallic elements. Optical microscopy of Fe-BTC/PpPDA before and after adsorption made it possible to visualize metallic particles adsorbed on the MOF (Figure 3).

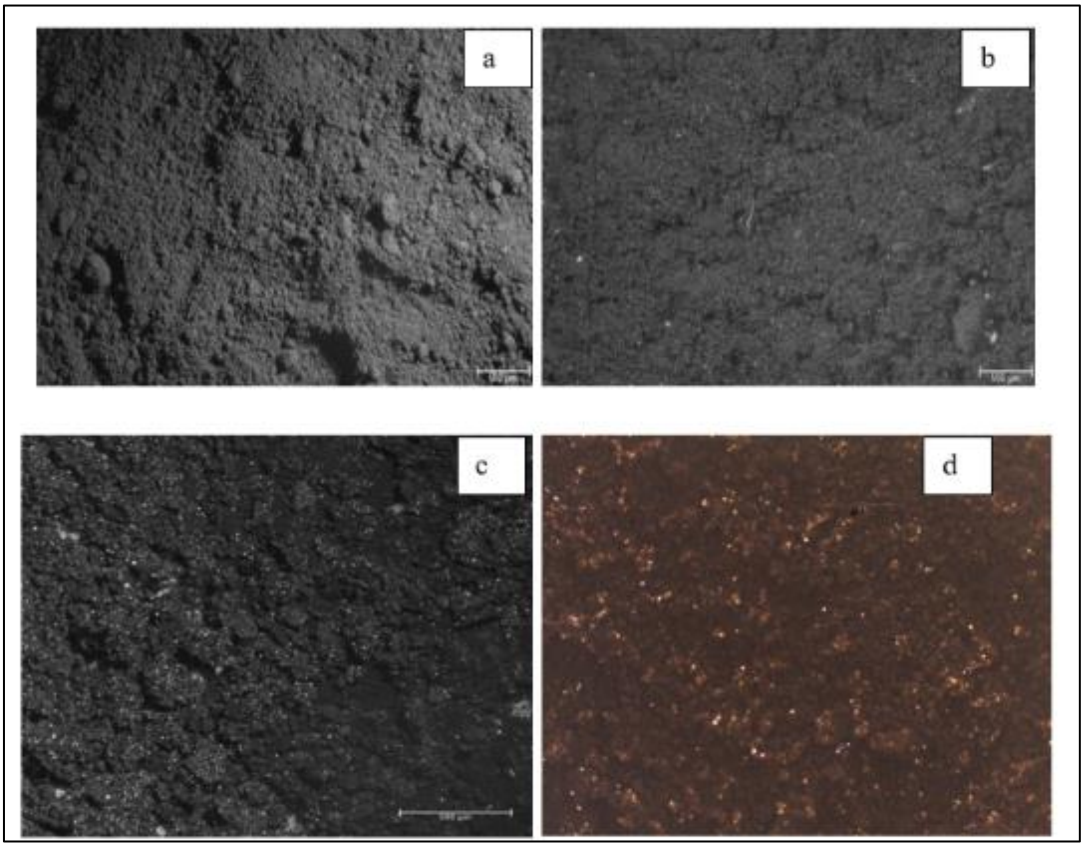
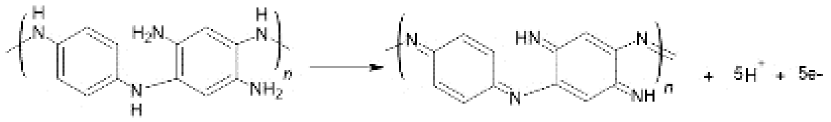


Figure 3 Optical microscopy images of Fe-BTC/PpPDA: (a) before adsorption; (b) after adsorption; (c) and (d): after desorption

The particles are much more visible after desorption (Figures 3d). The adsorbed metal ions are reduced to metallic elements and trapped in the pores; desorption has allowed these metals to be released, as shown of Figure 3d. Indeed, the amine groups of polyparaphenylenediamine are oxidized to imine, leading to the reduction of Au^{3+} ions and other ions (Equations 5 and 6).

$$AuCl_4^- + 3e^- \rightleftarrows Au + 4Cl^- \qquad \textbf{Equation 5}$$



Equation 6

The reuse of Fe-BTC/PpPDA for other adsorption cycles requires desorption and separation of the metals from the MOF. Since the metal element is gold and is mostly adsorbed, it is easy to separate it from Fe-BTC/PpPDA because it is non-magnetic while the MOF is magnetic. Desorption allowed the recovery of 98% of metals (mainly gold) for the first adsorption/desorption cycle and 89% for the second cycle (Figure 4).

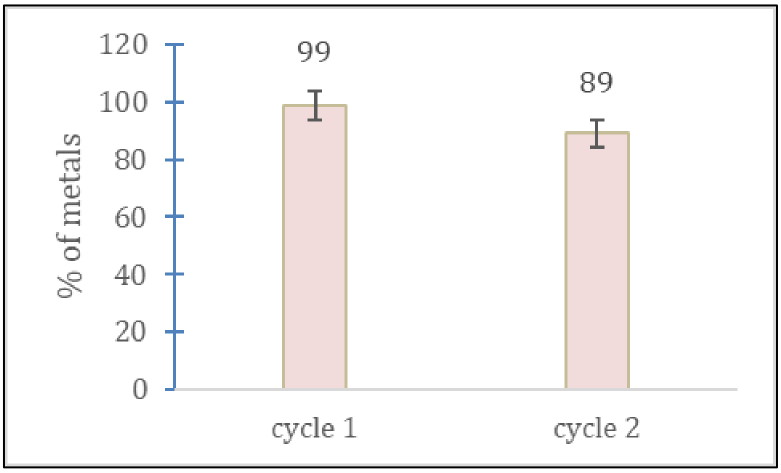


Figure 4 Adsorption/desorption cycles of Fe-BTC/PpPDA

The adsorption capacity of Fe-BTC/PpPDA decreases from one cycle to another, which is evident from the recovery percentages that dropped from 99% to 89%. This decrease is due to the incomplete release of all active sites as well as the blockage of the MOF pores. The IR spectrum of Fe-BTC/PpPDA before and after adsorption (Figure 5) showed that the amine and hydroxyl groups are responsible for the adsorption of metal ions and their reduction.

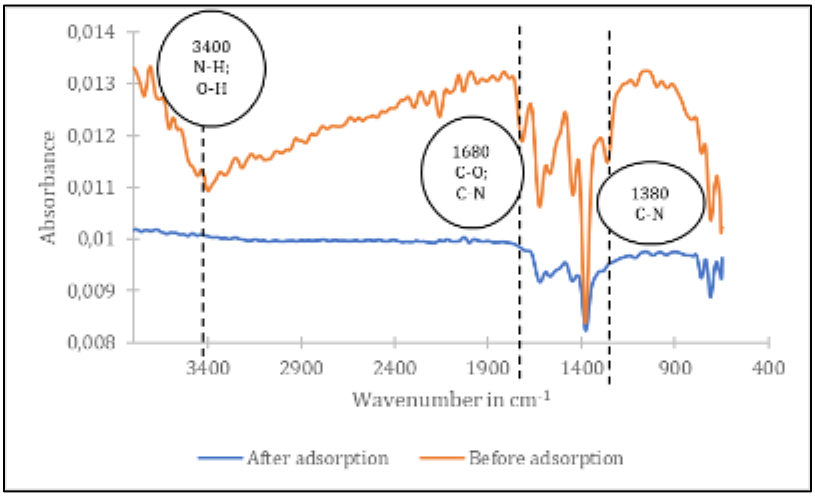


Figure 5 IR spectrum of Fe-BTC/PpPDA before and after adsorption

It can be observed that the broad band between 3000 and 3500 cm^{-1} has disappeared. This band was characteristic of the vibrations of N-H_2 and O-H bonds [25], which shows that these functional groups have reacted with the metal ions. The vibration bands at 1380 and 1680 cm^{-1}, characterizing the C-O/C-N bonds respectively [26-27], have also disappeared. The N-H_2 and O-H functional groups are responsible for the adsorption of gold ions but also for their reduction to the metallic element. Indeed, amines are oxidized to imines and alcohols to oximes, causing this reduction of bands intensity. Moreover, the absorption bands related to the Fe-O bond vibration have decreased considerably, highlighting electrostatic interactions between the iron metal centers and $AuCl_4^-$ ions.

4. Conclusion

This study highlighted the strong potential of Fe-BTC/PpPDA composite for the selective recovery of gold from complex solutions obtained from the acid leaching of printed circuit boards. Aqua regia leaching enabled the simultaneous solubilization of base and precious metals, confirming the richness of WEEE in gold compared to conventional gold ores. The physicochemical properties of Fe-BTC/PpPDA, particularly its predominantly microporous structure, its pH_{pzc} of 5.6, and the presence of reducing functional groups, facilitated the preferential adsorption of Au^{3+} ions, with an efficiency greater than 98%, despite the strong ionic competition, mainly from copper.

The adsorption of Au^{3+} ions in micropores followed by an in situ reduction of Au^{3+} ions to metallic gold, facilitated the recovery of gold using magnetic separation of gold and MOF adsorbent. Desorption and regeneration tests confirmed the reusability of the composite over multiple cycles, with a slight decrease in performance due to partial blockage of the active sites. These results demonstrate that Fe-BTC/PpPDA is a promising adsorbent for the valorization of WEEE and the sustainable recovery of gold, offering a relevant approach for developing countries such as Burkina Faso.

Compliance with ethical standards

Acknowledgments

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Disclosure of conflict of interest

The authors declare no competing financial interests.

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