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Research article

Optimization of HNO_3 leaching of copper from old AMD Athlon processors using response surface methodology

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ABSTRACT

The present study investigates the optimization of HNO_3 leaching of Cu from old AMD Athlon processors under the effect of nitric acid concentration (%), temperature (°C) and ultrasonic power (W). The optimization study is carried out using response surface methodology with central composite rotatable design (CCRD). The ANOVA study concludes that the second degree polynomial model is fitted well to the fifteen experimental runs based on p-value (0.003), R^2 (0.97) and Adj-R^2 (0.914). The study shows that the temperature is the most significant process variable to the leaching concentration of Cu followed by nitric acid concentration. However, ultrasound power shows no significant impact on the leaching concentration. The optimum conditions were found to be 20% nitric acid concentration, 48.89 °C temperature and 5.52 W ultrasound power for attaining maximum concentration of 97.916 mg/l for Cu leaching in solution.

1. Introduction

Discarded electronic equipments are rapidly growing worldwide. The technological advancement lead the world to a digital future. The wastes from electrical and electronic equipment have increased from 40 million tones/year to 70 million tones/year (Castro and Martins, 2009a; Bas et al., 2014). Electronic wastes contain precious metals as well as base metals. The recovery of precious metals from electronic wastes is gaining momentum (Birloaga et al., 2013; Kamberovic, 2011) due to the high demand for precious metals to be used in electrical and electronic instruments and limited availability of raw materials from ores. The computer processors, an advanced micro device (AMD) and RAM are typical examples of these type of wastes. The scrap composition varies from source to source. A PCB of computer can contain approximately 4–7% Fe, 13–20% Cu, 1–5% Al, 0.3–1% Pb, 0.1–1% Ni, 0.026–0.10% Ag and 0.05–0.250% Au along with some other metals (Cui and Zhang, 2008; Tuncuk et al., 2012).

Pyro metallurgical process and hydrometallurgical processes are being applied to recover the metals from ores and solid wastes (Seniunaite and Vasarevicius, 2017; Lu et al., 2016). Pyro metallurgical process consists of different steps like incineration, smelting, roasting, converting and refining to recover the metals. In pyro metallurgical process, metals are recovered without any chemical pre-treatment. The disadvantages associated are high energy requirement, NO_x and SO_x emissions and loss of metals during combustion (Jadhav and Hocheng, 2012). Hydrometallurgical process is gaining more attention due to less energy requirement are compared to Pyro-metallurgical process.

During hydrometallurgical process, leaching rates of metals are effected by the type of leaching agent, concentration of leaching agent and temperature (Chaurasia et al., 2013; Castro and Martins, 2009b). Different chemicals can be used as leaching agent like cyanides (Akcil et al., 2015; Dai and Breuer, 2013; Jeffrey and Breuer, 2000), sulfuric acid, aqua regia, thiourea (Jeffrey and Breuer, 2000; Örgül and Atalay, 2002; Jing-ying et al., 2012), thiosulfate and their mixtures with other chemicals (Breuer and Jeffrey, 2002; Ficeriová et al., 2005; Hilson and Monhemius, 2006; Feng and Van Deventer, 2007; Ha et al., 2010; Feng and Van Deventer, 2010). The leached solution can further be concentrated and purified by different separation processes including, precipitation, cementation, solvent

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extraction and ion exchange. Electrolysis or electrowinning process is the final step of hydrometallurgy for the recovery of metals (Jadhav and Hocheng, 2012).

Cyanide is very efficient for the leaching process but it is quite toxic as well (Jeffrey and Breuer, 2000). To overcome this problem, thiourea and thiosulfate are being studied. Thiosulfate has advantages over cyanide, as it is relatively economical and is less hazardous. However, its leaching mechanism is not well known. Leaching can be enhanced by the presence of some oxidant e.g., hydrogen peroxide along with acids (Ficeriová et al., 2005). Thiourea has been used as leaching agent to recover gold from PCBs (Birloaga et al., 2013; Kamberovic, 2011). Presence of copper imparts negative impact on the gold extraction. This problem can be eliminated by the pre-treatment. Aqua regia can also be used as leaching agent to leach the gold in the form of gold chloride, but it generates toxic waste that can affect the environment (Hilson and Monhemius, 2006; Kim et al., 2011). The leached solution can be concentrated and purified by the different separation processes including precipitation, cementation, solvent extraction and ion exchange (Cui and Zhang, 2008). However, these processes are very complex and gold is not recovered easily from the leached solution.

Nitric acid is a good lixiviant for the extraction of copper and other base metals from electronic waste because of its powerful oxidizing effect as compared to hydrochloric acid and sulfuric acid (Bas et al., 2014; Birloaga et al., 2013; Kim et al., 2011; Tuncuk et al., 2012). All base metals can be dissolved in nitric acid leaching except gold.

The leaching rates of copper can be increased using ultrasound waves. Zhang et al. (2008) studied the ammonia leaching on copper bearing tailings and reported 13.5% increase in leaching of copper using ultrasound waves (Zhang et al., 2008). Ultrasonic cavitation is affected by temperature, power or intensity, the solvent vapor pressure and the nature of the solvent used (Ficeriová et al., 2005). It is previously reported that cavitation is better attained at a lower temperature. Solvent vapor fills the cavitation bubbles, which then tends to collapse less violently, that is, the sonication effects are less intense than expected. Hence a compromise between temperature and cavitation must be achieved (Camelino et al., 2015).

Various studies reported on leaching of copper using ultrasonic irradiation and process optimization have used response surface methodology (RSM). However, selective nitric acid leaching of copper from old AMD Athlon processors has never been reported in the literature. Although RSM has been commonly used for obtaining optimal conditions, there were no reports describing the use of experimental design (Expert-Design Pro) software to optimize such processes. Current study intends to estimate the effect of variables for identifying the optimum conditions using small composite design. To the content of the present study, it is also needed to investigate the effect of ultrasonic radiation at high temperatures on the leaching of copper.

RSM is rapidly replacing one variable—one time approach to the collective effect of all factors on a particular process. It is less laborious and depicts complete effect of various parameters on the process. Hence, present work is designed to assess the effect of variables such as nitric acid concentration, temperature and ultrasonic power to identify the optimum conditions using small composite design. The interaction among various factors especially ultrasonic power and temperature may not be ignored to get true optimization of the process which ultimately would lead to finding significant and non-significant factors. This will help to ignore the involvement of unnecessary parameters during leaching of copper. The characterization of solid particles is carried out by XRD and concentration of copper in the leached solution is determined by Atomic Absorption Spectroscopy.

2. Materials and methods

2.1. Leaching of copper in nitric acid

Nitric acid was used to leach the metals from AMD Athlon processors. The concentration of metals present in the digested solution was determined using atomic absorption spectrophotometer (G8442AA, Agilent Technologies, USA). X-ray diffraction pattern was investigated on grounded waste PCBs powder by X-Ray diffractometer (X'Pert Powder PANalytical, DY-3805), and the result indicates that the copper in the PCBs exists as metal copper.

Leaching procedure of waste AMD Athlon processors begin by taking the specimen without gold coated pins and these were shredded to 1 mm final fineness. Analytical grade commercial concentrated nitric acid (50%) was used as received in experiments and was diluted as per requirement. Leaching experiments were carried out with 1.5 g crushed AMD Athlon processors in a 300 mL conical flask and it was stirred in a magnetic stirrer at temperatures ranging from 25° C to 50° C for one hour. The effect of ultrasonic irradiation on the leaching of copper at this temperature range was studied by irradiating the reaction solution at different power ranges from 0 to 300 W. During the process, NO_x emissions occur that could be identified by the brown colored gas emitted from the solution (Equation (1)). Therefore, reflux condenser was used for re-dissolving these gases in solution.

$$Cu + 4HNO_3 \rightarrow Cu(NO_3)_2 + 2NO_2 + 2H_2O \quad (1)$$

The leached solution was further analyzed for copper metal concentration by atomic absorption spectroscopy.

AMD Athlon processors contain gold coating pins on small printed circuited chip which were detached and were separately leached in the nitric acid solution. The collected solid particles were washed few times with the distilled water and were placed in a heating oven at 100 °C for 1 h, leaving behind the dry gold particles. The purity of gold was confirmed by X-Ray diffractometer (X'Pert Powder PANalytical, DY-3805). The XRD pattern of the sample suggested the crystalline behavior of gold. The spectrum showed that there was no other metal present except the recovered gold (Fig. 1). The five dominant peaks of the plane (111), (200), (220), (311) and (222) were dictating the crystalline structure of gold. This pattern exactly matched the standard XRD pattern of gold.

As soon as the coated layer of gold is removed, the metal present inside is now exposed. These pins were separated and then sent for X-Ray diffraction. The major peaks of XRD pattern confirmed the presence of copper like Cu (111), Cu (200) and Cu (220) (Fig. 2).

2.2. Experimental design and optimization

In this study, the main objective is to apply RSM to build a mathematical model and to maximize leaching concentration of copper (Y, mg/l) from AMD Athlon processors. Response surface methodology (RSM) is a most commonly used procedure to explore functional relation between a set of input variables with the responses and to optimize these responses. RSM is used to study the effect of three input variables (factors):nitric acid concentration (X_1, %), temperature (X_2, °C) and absolute ultrasound power (X_3,W). Two level factorial with small CCRD was used. The small design generated 15 experiments (supplementary Table 1) which are comprised of six axial points, four factorial points and five central points. The minimum and maximum levels of each input variable were selected in accordance with previous studies (Bas et al., 2014; Kamberovic, 2011; Tuncuk et al., 2012; Seniunaite and Vasarevicius, 2017; Lu et al., 2016; Ghosh et al., 2015). The coded levels and actual values of input factors are listed in Table 1. The Design-Expert9.4