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Studies on Acidic Leaching of Waste Mobile Phones

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Abstract: The consumption of mobile phones is increasing day by day and the replacement speed is also accelerating. Thus, a lot of old mobile phones and their parts need to be processed. Mobile phones contain more than 40 elements and/or materials. This experimental study aims to establish the possibilities of recovering valuable metals from waste mobile phones by using acidic leaching. The process steps include dismantling of waste mobile phones, pre-processing to liberate the materials, and directing them to final treatment processes. Prior to leaching tests, the PCBs of waste mobile phone sample are chemically analyzed and its metal contents are found as 33.22% Cu, 9.42% Fe, 1.86% Zn, 2.77% Ni, 1.63% Al, 0.4% Pb, 0.02% Co, 0.14% Ag, and 251.2 ppm Au. In the leaching experiments, sulfuric, nitric, and hydrochloric acids are used. In the sulfuric acid leaching experiments, effects of acid concentration, temperature, hydrogen peroxide addition, and leaching periods on metal dissolution efficiencies are investigated. Results are compared and discussed in relation to the types of acids. XRD analyses of feed material and leach residues are also carried out. As a result of this experimental study, 97.9% Fe leaching in 8 M H_2SO_4 solutions, 89% Pb, 100% Ni, 90.4% Co, and 4.1% Cu leaching in 1M HNO_3 solutions, 100% Pb, and 100% Al leaching in 4 M HCl solutions in one hour are found. In conclusion, sequential leaching with different acids is recommended for the effective leaching of all metals.

Keywords: Recycling, Waste Mobile Phones, Acidic Leaching

1. Introduction

Electronic devices facilitate our lives in many ways such as health, education, work, transportation, and communication. Among them, communication is one of the most important concepts for people. Today, people use technological tools to communicate, such as laptops, desktop computers, cameras, televisions, and mobile phones. Nowadays mobile phones are one of the most used technological devices for communication because of their portable and convenient possibilities. With the development of technology, mobile phones are being developed and new features are added to them at the same time.

The consumption of electronic information products, mainly mobile phones, is increasing day by day, and the replacement speed is accelerating, leading to a surge in the number of used mobile phones. While enjoying social development and technological progress, people have also produced a lot of old mobile phones. As the world's largest consumer of mobile phones, 800 million mobile phones were used in China in 2017 [1]. The rapid development of the mobile phone's function and technology causes the mobile phones to be upgraded every 18 months or so, and as a result, a huge number of old mobile phones and their parts are processed in small underground businesses with the wrong methods. The improper disposal of the post-abandoned treatment can cause a great environmental burden and waste of resources. Therefore a reasonable scientific recycling system should be used to produce good environmental and economic benefits.

Mobile phone consumers have been motivated by the rapid growth of technology and encouraged to update their devices regularly to keep up with innovations, architectures, and capabilities [2]. Consequently, mobile/cell phone waste has risen significantly in the last decade. Due to their small size, it is convenient for users to keep outdated or unused mobile phones at home or the office, rather than recycling them appropriately. In today's modern world, most people tend to use a mobile phone in their daily lives. The technology of this device not only has enabled people to use it as a phone, but also offers them access to the Internet while providing a wide range of applications with which they can manage their work, access social platforms, play their favorite games, and even pay for products and services. Furthermore, with rapid developments in mobile phone technology, mobile manufacturers encourage users to change their devices frequently to take advantage of new features, designs, and capabilities. As a result, there has been a massive increase in mobile phone waste, and due to their small size, it is convenient to keep unused mobile phones at home or in the office [2].

Considering the usage rates of electronic devices in the world, mobile phones are in the first place since the devices are continuously improved with the renewed technology. In this case, the circuit boards of mobile phones take up a great deal of space when considering the amount of waste. As for the weight of mobile phones, plastic materials are the most prominent one. In addition to plastics, batteries, metals, and PCBs (printed circuit boards) have a large share in mobile phones [3]. Typically, a mobile phone can be split into four components: a PCB, a display unit, a battery, and a case, with the total mobile phone containing over 40 elements and/or materials [4]. The main elements are Cu, Ni, Fe, Co, In, Zn, Al, Pb, Sn, Cr, Hg, Cd, Ti, and some precious metals such as gold, silver, and palladium. The quantities of these metals vary according to the features of mobile phones. The metal contents of mobile phones have changed over the years. The reason for this is the development of technology and the effort to obtain a higher performance at a cheaper price. Another reason is that alternative resources are used because of the lesser amount of material used in nature.

In the study by Miah et al. [5] to identify and address various wastes in mobile phones, critical observations and research techniques were used. Major findings obtained from the study are as follows such as lead, beryllium, brominated flame-retardants, chromium, arsenic, cadmium, and antimony are considered toxic and harmful to the environment and human health. 90% of the materials within a mobile phone can be recycled. Mobile phone parts such as batteries, circuit boards, handset housings, and casings plastics, accessories including plastics can be recycled to make new products [5]. The paper addresses various wastes in mobile phones, which were hardly ever attempted before. Wastes activities are then individually attacked to reduce or eliminate them from the system. It is concluded that recycling mobile phone wastes make the environment safer and better and only 3% of mobile phones are currently being recycled in the world [5].

Waste management is carried out with different types of applications. The reason for the difference in their implementation is the structure of the material used. In general, these applications are classified as recycling, reuse, and recovery. Among these applications, recycling is the number one application for the recovery of valuable raw materials contained in electronic waste and mobile phones. Various methods are used in recycling processes [6] and typically, material recovering technologies for waste mobile phone (WMP) recycling consist of at least two of the following steps:

1) Pre-treatment, including disassembly and size reduction (crushing & grinding),
2) Electrostatic separation, gravity separation, density-based separation, jigging, Eddy Current separation, and magnetic separation [7].
3) Material extraction normally involves two processes: (i) leaching target metals using an acidic solution, followed by (ii) purification to concentrate the metals of interest. Other material extraction processes which have been investigated include solvolysis and reprocessing (for plastics).

Two main techniques are applied to recycle the PCBs of WMP; pyro-metallurgical and hydrometallurgical processes. During the pre-processing phase of the material, the battery and hardware parts are removed manually and only the PCBs are collected [8]. Then, it is shredded and ground to make it ready for chemical processing. Hydrometallurgical technologies are predictable and controllable methods for material extraction and have been applied to WEEE recycling for almost three decades [9, 10]. In hydrometallurgical processes, leaching of the target metals is usually followed by concentration and metal recovery using solvent extraction and electrowinning [9]. Pyrometallurgical processes such as smelting are also used to extract metals from WMP PCBs, but the technology is severely under-examined [11]. For WMP cases and whole units, other processes such as mechanical recycling and solvolysis are applied.

A series of factors have been identified to explain the low recycling rates of WMPs, including regional, educational, income personal or social norms, insufficient financial incentives, fear of privacy leakage, a lack of collection systems or knowledge of collection systems, and limited information availability, and technology availability [12, 6].

This study is important from the viewpoint of recycling and environmental protection by preventing these heavy metals from dissolving and contaminating soil & groundwater. When considering the decrease in primary resources and the cost of mining and processing, the recycling issue becomes more important, and thus this type of study.

2. Material and Method

This experimental study aims to investigate the possibilities of recovering the valuable metals in the PCBs of the waste (end-of-life/used) mobile phones. It includes preparation, characterization, chemical analyses, and hydrometallurgical studies. An experimental flow sheet showing all steps is presented in Figure 1. The process steps include dismantling of collected waste mobile phones, pre-processing to liberate the materials, and directing them to adequate subsequent final treatment processes. Hazardous substances such as batteries are removed and stored and/or treated safely while valuable components/materials are taken out for reuse or to be directed to efficient recovery processes. The batteries from the devices can be sent to dedicated facilities for the recovery of cobalt, nickel, and copper. After removal of the hazardous and other special components, the remainder is further separated by manual dismantling, mechanical shredding, and sorting techniques followed by acidic leaching. In leaching experiments, sulfuric acid, nitric acid, and hydrochloric acid are used. Effects of acid concentration, temperature, peroxide addition, and leaching times on metal dissolution efficiencies are investigated. Results are compared and discussed in relation to the types of acids. XRD analyses of feed material and leach residue are also carried out.

Waste mobile phones subjected to this experimental study, are collected from friends & families and supplied by Exitcom Recycling Ltd. *Co*. (Turkey). They were dismantled with a screwdriver and their PCBs were removed one by one as shown in Figure 2a and plastic parts and batteries are separated. PCBs were then cut into pieces with a shredder machine below 4 mm in size (Figure 2b) and screened. A representative sample was taken from shredded PCBs and ground by a ring mill for chemical analysis. The remaining parts are dissolved in aqua regia and analyzed for different metals according to size ranges (Figure 2c). Finally, hydrometallurgical processes were conducted.

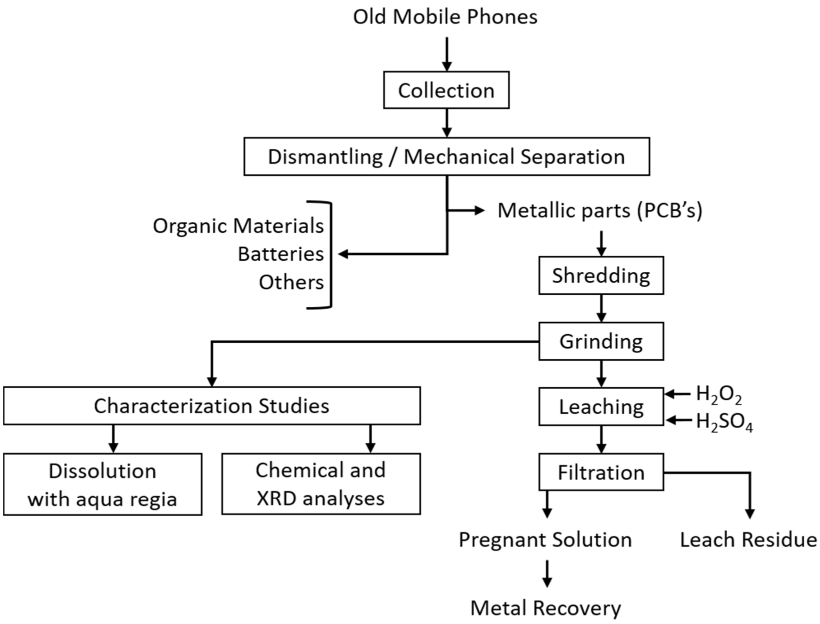


Figure 1. *Experimental flowsheet for the recovery of metals from old mobile phones.*

(a) (b) (c)

Figure 2. *(a) Dismantling batteries and capacitors of PCB-MP, (b) Shredding and classification, (c) Dissolving in aqua regia and filtering.*

3. Results and Discussions

Results are presented in two main parts namely size-reduction and characterization experiments, and leaching experiments as follows.

3.1. Size Reduction & Characterization Experiments

After dismantling of old mobile phones, PCBs are subjected to the size reduction process by the shredder, and cut samples are taken for sieve analysis. Chemical analysis of the sample related to the size ranges is given in Table 1. According to the table, all metal contents except Cu, Au, and Fe, do not change much with particle size range changes where Cu content decreases and Au content increases with decreasing size range and Fe content fluctuates without depending on size range.

Table 1. *Metal contents (%) related to particle size ranges.*

Particle Size Range, mm	Weight (%)	Metal Contents (%)								ppm
		Ag	Cu	Co	Ni	Zn	Fe	Al	Pb	Au
+8	17.72	0.05	43.27	0.01	2.56	3.00	3.73	1.84	0.31	166.2
-8+6	13.65	0.04	29.95	0.04	2.52	0.49	17.93	1.46	0.20	
-6+3.36	26.91	0.18	33.94	0.02	1.76	1.19	5.00	1.76	0.23	275.9
-3.36+2.36	19.81	0.14	25.20	0.06	3.44	1.67	21.91	1.50	0.38	
-2.36+1.19	11.86	0.31	35.76	0.03	4.37	3.68	5.90	1.31	0.38	
-1.19+0.5	3.83	0.28	27.48	0.03	4.95	1.69	6.29	1.27	1.43	
-0.5+0.3	3.38	0.17	22.91	0.02	2.62	0.60	2.95	1.51	2.17	440
-0.3	2.84	0.27	14.63	0.02	1.39	0.52	4.03	2.64	1.71	
Total	100.00	0.14	33.20	0.02	2.77	1.86	9.42	1.63	0.40	251.7

Before leaching experiments, dissolution tests were carried out to find the metal content of the sample. For this purpose, samples were ground below 1.0 mm and then dissolved in aqua regia. An average weight loss of 53.11% was accepted as the total metal content. XRD analysis of the ground PCBs of the old mobile phone sample is shown in Figure 3. Metal contents are also given in the earlier part of Table 1. The highest peaks belong to Cu followed by Al, Pb, and Sn.

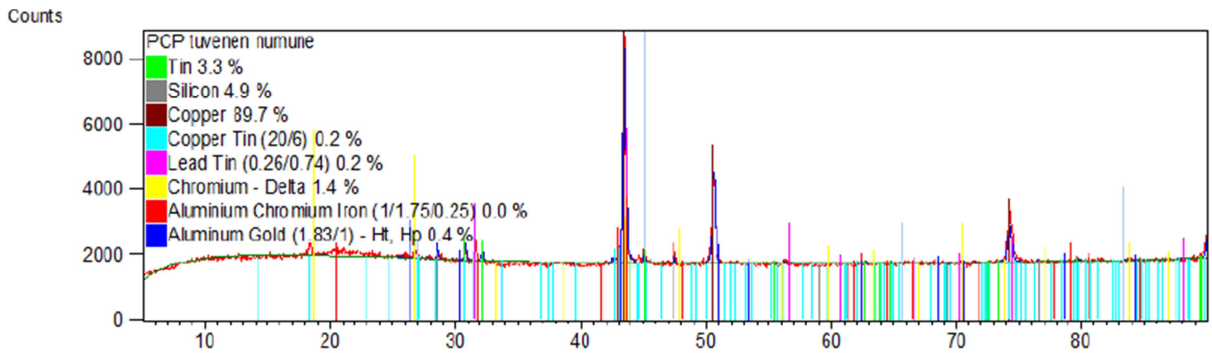


Figure 3. *XRD pattern of the PCB samples used in the tests.*

3.2. Leaching Experiments

Before starting the leaching experiments, the waste mobile phone sample is ground under 1 mm in size, and four groups of leaching tests were performed. In the first group experiments, the effect of sulfuric acid concentration on metal leaching efficiencies was studied. Experimental conditions were: solid/liquid ratio of 10%, the temperature of 60°C, stirring speed of 600 rpm, and duration of 1 hour at varying acid concentrations of 1-8 mole/L. According to the results shown in Table 2, Fe, Ni, and Co dissolutions increase with increasing acid concentrations while Pb, Zn, and Al are not affected by acid concentration changes.

Table 2. *Metal leaching efficiencies (%) at different acid concentrations.*

H_2SO_4, mole/L	Weight Loss, %	Metal Leaching Efficiencies, %							
		Cu	Pb	Fe	Ni	Zn	Co	Ag	Al
1	7.99	0.03	1.99	20.87	2.38	7.45	42.52	0.03	61.19
2	5.36	0.01	1.12	30.83	0.88	9.76	39.59	0.04	64.29
3	1.98	0.05	2.55	33.24	3.84	8.21	51.17	0.03	79.01
4	3.54	0.03	2.77	30.74	3.40	9.73	50.52	0.04	64.10
8	3.93	0.45	1.97	99.41	13.03	8.72	68.31	0.06	37.20

In the second group of experiments, the effect of hydrogen peroxide (H_2O_2) addition in sulfuric acid media was studied. Hydrogen peroxide is a strong oxidant, which is commonly used in combination with acids to enhance metal extraction.

Experimental conditions were: H_2O_2 addition of 10%, solid/liquid ratio of 10%, temperature of 60°C, stirring speed of 600 rpm, duration of 1 hour at varying acid concentrations of 1-8 mole/L. Results are given in Table 3. As it can be seen from the results, although weight losses are higher in terms of hydrogen peroxide addition, there are no significant changes in metal dissolution efficiencies. In both groups of leaching tests high Fe, Co, and Al leaching efficiencies are obtained. In an earlier study on the extraction of copper from scrap TV boards by Deveci et al [13], it was found that Cu dissolution was increased with H_2O_2 addition (0.3 M, 1-4 hours) in sulfide media.

Oh et al. [14] proposed sulfuric acid (H_2SO_4) leaching of PCBs in the presence of H_2O_2 as a first stage process where Cu, Fe, Zn, Ni, and Al were extracted at high recoveries of >95%. In the second stage, they targeted the recovery of precious metals using ammoniacal thiosulfate ($CuSO_4$-NH_4OH-$(NH_4)_2S_2O_3$) as lixiviant. Quinet et al. [15] also considered sulfuric acid leaching as the first stage process at which various oxidants including H_2O_2, O_2 and Fe^{3+} were tested for the extraction of copper by the chemical reaction given as;

$$Cu^o + H_2O_2 + H_2SO_4 \rightarrow Cu^{2+} + SO_4^{2-} + 2H_2O \quad (1)$$

Table 3. *Effect of H_2O_2 addition on metal leaching efficiencies (%) at different acid concentrations.*

H_2SO_4, mole/L	Weight Loss, %	Metal Leaching Efficiencies, %						
		Cu	Pb	Fe	Ni	Zn	Co	Al
1	6.23	0.09	0.54	31.45	3.30	7.69	36.09	61.09
2	6.99	0.04	0.87	26.46	2.85	7.32	36.61	78.38
3	5.90	0.07	1.23	28.26	3.41	8.72	40.62	74.31
4	5.05	0.09	1.12	40.26	4.24	18.99	35.25	67.63
8	4.95	0.16	3.32	97.90	7.75	9.59	61.78	24.02

In the third group of leaching experiments, the effect of leaching time on metal dissolution efficiencies with higher oxidant addition was investigated. In the leaching experiments, experimental conditions kept constant are 8 mole/L of H_2SO_4 concentration, the temperature of 60°C, 600 rpm of stirring speed, 10% of solid/liquid ratio, 20% of H_2O_2 addition. Leaching time changed from 1 to 5 hours. Experimental results are shown in Table 4. Increasing leaching time had an increasing effect on metal leaching efficiencies. At the end of 5-hour leaching, 98.44% Fe, 40.59% Ni, 92.09%Co, and 41.90% Al leaching efficiencies are reached. Cu, Pd, and Zn leaching efficiencies are found to be very low such as 3.87%, 4.20%, and 12.89%, respectively.

Table 4. *Effect of leaching time on metal leaching efficiencies.*

Leaching Time, h	Weight Loss, %	Metal Leaching Efficiencies, %						
		Cu	Pb	Fe	Ni	Zn	Co	Al
1	3.62	0.15	4.06	88.65	15.09	9.30	62.25	36.69
2	5.80	0.24	4.78	94.27	15.89	9.34	63.25	50.36
3	4.66	1.07	3.83	96.14	20.95	10.35	66.75	46.40
4	5.25	1.51	3.66	98.71	16.36	11.54	60.94	50.31
5	7.24	3.87	4.20	98.44	40.59	12.89	92.09	41.90

In the fourth group of experiments, the effect of acid type was studied. For this purpose, sulfuric, hydrochloric, and nitric acids are used. Acid leaching is often used as the first stage of leaching for the extraction of base metals, copper in particular. The acid leaching of metals from e-waste has been extensively investigated using various mineral acids and oxidants (HCl, H_2SO_4, HNO_3/H_2O_2, $HClO_4$, NaClO) [13]. Bacterial leaching is also studied [16] and ferric ion addition during bioleaching increased Cu extraction to > 99% according to Yang et al. [17].

Our experimental conditions were: solid/liquid ratio of 10%, a temperature of 60°C, stirring speed of 600 rpm, and duration of 1 hour at varying acid concentrations. Results are shown in Table 5. XRD analysis of leach residues of different types of acids is shown in Figure 4. In the experiments using HNO_3, higher Co, Ni, and Pb leaching efficiencies, and in the experiments using HCl, higher Pb, Co, Al, and Fe leaching efficiencies are observed. Cu leaching efficiency was still low in all cases with different types of acids.

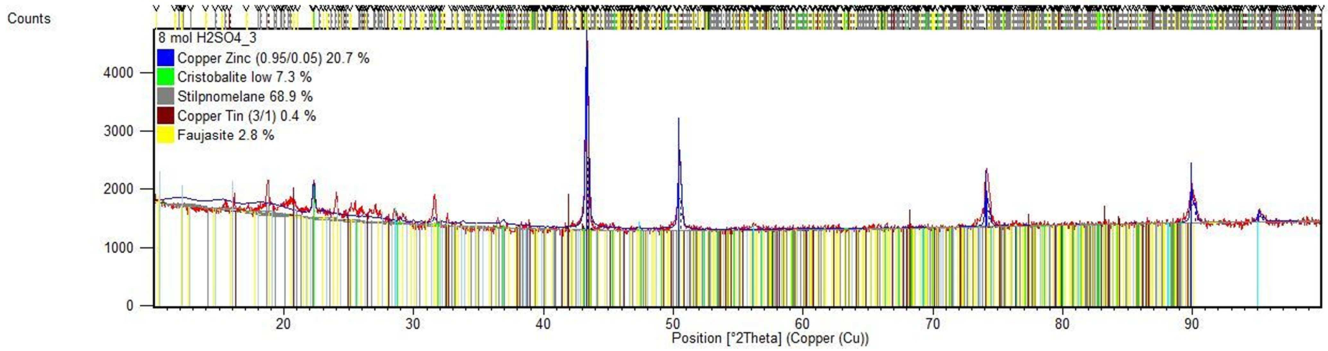
In the studies done by Mecucci & Scott [18] high Cu and Pb extractions (>85%) were observed by nitric acid (HNO_3) leaching according to the following reaction;

$$3Cu^o/Pb^o + 8HNO_3 \rightarrow 3Cu/Pb(NO_3)_2 + 4H_2O + 2NO \quad (2)$$

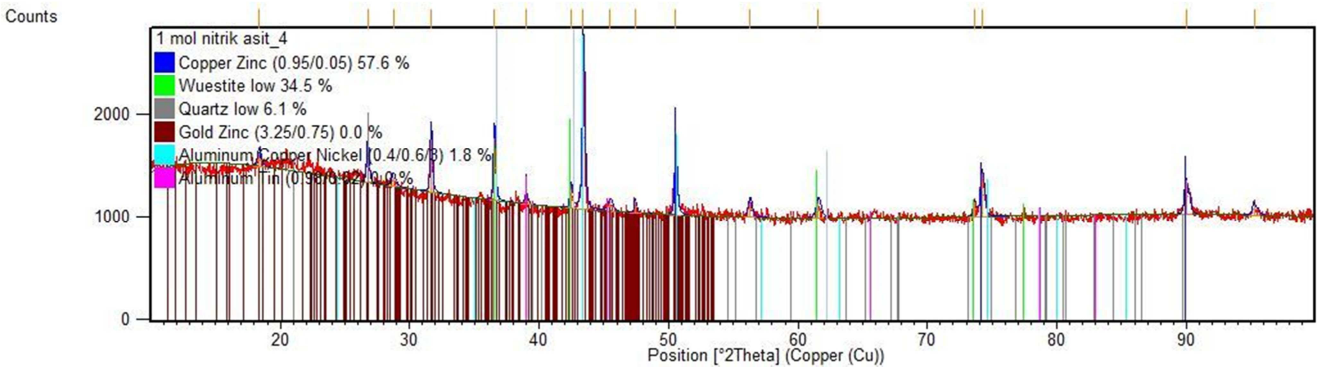
In our study, 100% Pb extraction was also observed while Cu leaching efficiency reached only >4%. Castro and Martins [19] similarly reported that Cu extraction from PCBs found <0.01% in sulfuric acid leaching supporting our results and about 30% in cases of H_2SO_4+HCl, HCl, and HCl+HNO_3 solutions. According to Madenoğlu's report [20], the highest extraction of Cu and Au from PCBs was obtained by using HNO_3/HCl media (e.g. Aqua regia) as expected. XRD analysis of leach residues (Figure 4) in all cases also showed the existence of Cu and Zn as their low dissolutions.

Table 5. *Effect of acid type on metal leaching efficiencies.*

Acid Type	Metal Leaching Efficiencies, %						
	Cu	Pb	Fe	Ni	Zn	Co	Al
1 mole/L H_2SO_4	0.09	0.54	31.45	3.30	7.69	36.09	61.09
8 mole/L H_2SO_4	0.16	3.32	97.90	7.75	9.59	61.78	24.02
8 mole/L H_2SO_4 + 20% H_2O_2	0.15	4.06	88.65	15.09	9.30	62.25	36.69
1 mole/L HNO_3	4.13	87.98	21.84	100.00	25.66	90.40	37.20
4 mole/L HCl	0.74	100.00	48.84	17.69	18.80	77.57	100.00
8 mole/L HCl	0.85	100.00	49.25	17.61	19.97	71.36	100.00



(a)



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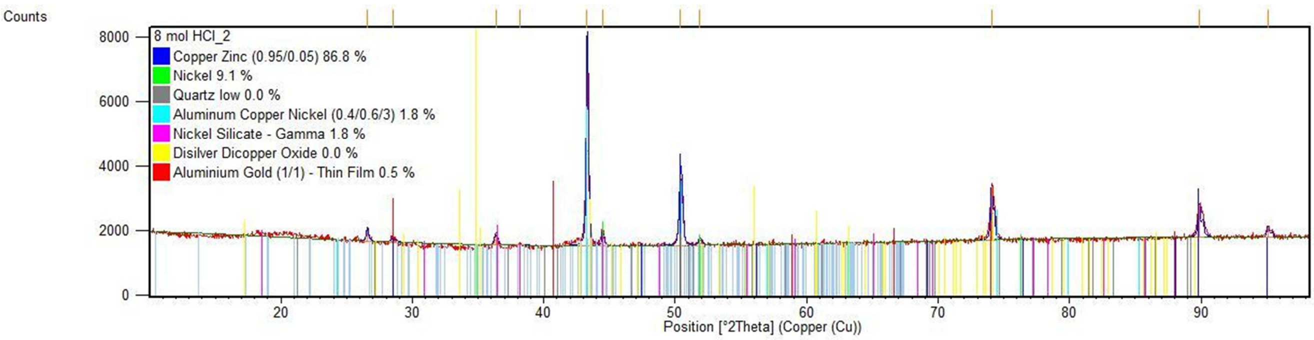

(c)

Figure 4. *XRD analyses of leach residues carried with different types of acids (a) 8 mole/L H_2SO_4, (b) 1 mole/L HNO_3, (c) 8 mole/L HCl).*

4. Conclusions

In this experimental study, metal recoveries from waste mobile phones containing 33.22% Cu, 9.42% Fe, 1.86% Zn, 2.77% Ni, 1.63% Al, 0.4% Pb, 0.02% Co, 0.14% Ag, and 251.2 ppm Au, by acidic leaching were studied. The experimental study includes dismantling, size reduction (shredding & grinding), screening, dissolution, and leaching tests. After shredding and grinding of the PCBs of waste mobile phones, four groups of leaching tests were performed. Effects of acid concentration, H_2O_2 addition, leaching time, and acid type on metal leaching efficiencies were studied. As a result of this experimental study, 97.9% Fe leaching in 8 M

H_2SO_4 solutions, 89% Pb, 100% Ni, 90.4% Co, and 4.1% Cu leaching in 1M HNO_3 solutions, 100% Pb, and 100% Al leaching in 4 M HCl solutions in one hour were found. In conclusion, sequential leaching with different acids is recommended for the effective leaching of all metals. Precious metals can be dissolved by using cyanide, thiourea, and aqua regia. For metal recoveries from leach solutions solvent extraction/ion exchange, precipitation, cementation, and electrowinning depending on metal types and compounds can be used.

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