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SCIENTIFIC PAPER

UDC 546.56:66:549.743.12

DOI 10.2298/CICEQ120113039K

INVESTIGATION OF THE USE OF AMMONIUM ACETATE AS AN ALTERNATIVE LIXIVIANT IN THE LEACHING OF MALACHITE ORE

Solutions containing ammonia allow for selective leaching of copper from a copper ore. In this study, the leaching and kinetics of malachite ore were examined using ammonium acetate solutions as an alternative lixiviant. The effects of some experimental parameters on the leaching of malachite ore were investigated. A kinetic model to represent the effects of these parameters on the leaching rate was developed. It was determined that the leaching rate increased with increasing solution concentration, temperature and stirring speed, and decreasing particle size and solid-to-liquid ratio. It was found that the leaching reaction followed the mixed kinetic control model. The activation energy of this leaching process was determined to be 59.6 kJ mol⁻¹. Consequently, it was determined that ammonium acetate solutions could be used as an effective leaching agent for copper extraction from malachite ore.

Keywords: copper, kinetic, leaching, malachite.

Metals are frequently produced after being extracted from an ore or its concentrates because most of them are found in nature as compounds, such as complex mixtures of sulfides, oxides, carbonates, silicates. The extraction of metals from metal sources like ores, concentrates, and secondary metal sources (various industrial wastes), is carried out by pyrometallurgical or hydrometallurgical methods [1,2].

Hydrometallurgy is essentially concerned with methods whereby metals, metal salts, or other metal compounds are produced by means of chemical reactions involving aqueous and organic solutions [2]. It covers a large variety of processes ranging from the leaching of metal values in an aqueous solvent through the purification of the solutions to the recovery of the metals or their compounds by chemical or electrochemical precipitation [2,3]. Leaching is the term applied to the process of recovering a metal from the metal source by a solvent or lixiviant, and it is the first step of any hydrometallurgical process. The metallic va-

lues in the metal source pass into the solution by dissolving in the leaching step [1,2].

Copper is among the most prevalent and valuable metals used in the industry. It is used mostly in the electrical and electronics industries because of its high conductivity [4-6]. In nature, copper is generally found in the form of sulfide and oxide minerals, such as azurite, malachite, tenorite, chrysocolla, bornite, brochantite, enargite, chalcopyrite, chalcocite, covellite [5,7,8]. At the present time, the production of copper is generally performed using the low grade oxidized copper ores and secondary sources containing copper due to depletion of high grade sulfide copper ores [9]. The hydrometallurgical methods are usually preferred for the recovery of copper from the low grade oxidized copper ores because of low cost, short construction time, operational simplicity, good performance, and environmental advantages [10]. For this aim, the strong acids, such as HCl, HNO₃, and H₂SO₄, are mostly used as the leaching reagents. Sulfuric acid is the preferred lixiviant for the leaching of the oxidized copper ores, like azurite, malachite, tenorite, and chrysocolla [5,8]. However, the acid leaching can be sometimes uneconomical because basic ores may cause excessive acid consumption during the leaching process due to the gangue minerals. Furthermore, some undesired impurities in ore matrix can be also dissolved along with the desired metal, and they pass

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Paper received: 13 January, 2012

Paper revised: 20 March, 2012

Paper accepted: 10 May, 2012

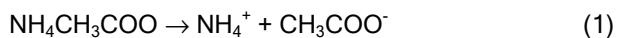
into the solution. Therefore, more basic lixiviants than inorganic acids (or weakly acidic reagents) may be more favorable in the leaching processes, allowing impurities in the solution to be separated more effectively [11]. So, the solutions containing ammonia have been used as lixiviant for the leaching of various ores, such as magnesite [12], colemanite [13,14], ulexite [15,16], smithsonite [17], and hemimorphite [18].

In acid leaching of oxidized copper ores, the impurities passing from ore to solution cause some problems in further processing of the leaching solution, such as electrowinning of copper [19]. For these reasons, basic or weakly acidic lixiviants containing ammonia have been also used for the leaching of the oxidized copper ores [5, 19-24].

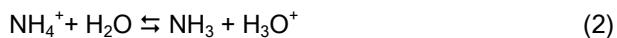
In ammoniacal leaching systems, the dissolution of ore is carried out at mildly acidic or basic conditions, and ammonia can be recovered by evaporation at the end of the process. Furthermore, some metals, which pass into the solution during dissolution process, precipitate because of the higher pH of solutions containing ammonia. Besides, ammonia forms stable complexes with copper ions, and this leads to an increase in the leaching rate of ore. Thus, ammonia allows for selective extraction of the copper from ore, leaving the undesired components in the residue [19,25,26]. As a result, a high purity solution for recovery of copper can be obtained at the end of such a leaching process.

The aims of this study were to investigate the usability of ammonium acetate solution as lixiviant in the leaching of malachite ore and to determine the effects of the experimental parameters, including ammonium acetate concentration, solid-to-liquid ratio, particle size, stirring speed, and reaction temperature.

Ammonium acetate, chosen as the solvent in this study, is the salt of a weak acid (acetic acid) and a weak base (ammonia). According to the following reaction, it ionizes in an aqueous medium:

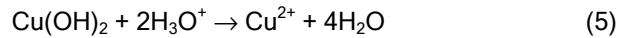
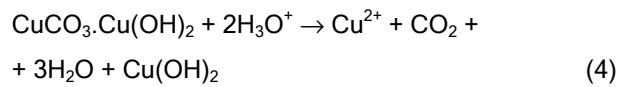


Ammonium and acetate both undergo hydrolysis, as shown in the following equations:

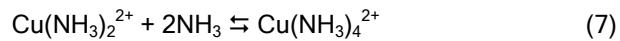
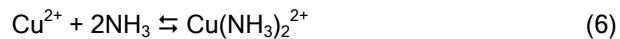


Ammonia/ammonium and acetic acid/acetate buffered systems form in the aqueous medium. Thus, pH of solution remains almost constant during leaching process. Ammonium acetate is a neutral salt, and pH of leaching solution is near neutral value at the end of dissolution process due to the formed buffer system [27]. This case can be an advantage in the leaching because some impurities present in ore mat-

rix, such as iron and aluminum, precipitate in the form of hydroxides. As a result of this, a high purity solution can be obtained at the end of leaching. In addition, ammonium acetate provides ammonium ions, which contributes the protons (hydronium ions) required for the dissolution of basic malachite ore. Ammonia and hydronium ions produced by reaction in Eq. (2) play an important role in the dissolution process. Basically, hydronium ions are responsible for the dissolution of basic ore. On the other hand, ammonia can form the stable complexes with copper (II) ions passing from ore to solution. The formation of copper amine complexes can ensure an increase in the dissolution of malachite ore. The copper in the malachite ore is in the form of $\text{CuCO}_3 \cdot \text{Cu(OH)}_2$. Thus, when malachite ore is added into ammonium acetate solution, the reactions occurring during the leaching process are probably as follows:

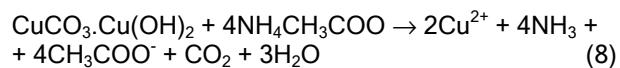


Copper ions pass into the solution in the form of the water-soluble copper amine complexes with ammonia, and thus, no precipitation of any copper compound occurs in the solution. The formation reactions of the copper-amine complexes are written as follows:



It is reported that the $\text{Cu}(\text{NH}_3)_2^{2+}$ formed during leaching reaction is an intermediate species, and it converts to the stable complex of $\text{Cu}(\text{NH}_3)_4^{2+}$ [5,18].

Consequently, the overall leaching reaction can be written as follows:



Because leaching is an essential unit operation in the hydrometallurgical treatment of ores, the kinetic analysis of leaching reactions is required for the efficient design of leaching reactor to be used in a hydrometallurgical plant. The reactions occurring in the leaching process are heterogeneous. Because of those reasons, leaching reactions do not often obey simple first- and second-order kinetics. The kinetic analysis of these kinds of reactions is generally performed using the heterogeneous reaction models [28,29]. Thus, the kinetic analysis of leaching process practiced in this study has been made using the shrinking core model.

MATERIAL AND METHODS

An oxidized ore sample (malachite) used in this leaching process was collected from Palu region Elazığ, Turkey. The ore sample was crushed, ground, and then sieved using ASTM standard test sieves to obtain desired particle size fractions. The mineralogical analysis of malachite ore sample was performed using a Rigaku RadB-DMAX II X-ray diffractometer. The result of the X-ray analysis (Figure 1) indicates that the ore sample consists of malachite, albite, chloride, laumontite, margarite, kaolinite, and quartz. The chemical composition of the sample is given in Table 1.

The leaching experiments were performed in a 1 L glass reactor equipped with a mechanical agitator, a reaction temperature control unit, and a cooler to avoid loss of solution from evaporation. The experimental procedure was initiated by adding 500 mL of an ammonium acetate solution into the glass reactor and bringing it to the desired reaction temperature. A given amount of solid sample was then added to the solution. The reactor content was agitated with a mechanical stirrer for required retention time. Aliquots of 5 mL were withdrawn at regular intervals during the reaction and were immediately filtered. The amount of dissolved copper in the solution was determined com-

plexometrically using EDTA solution as titrant and murexide as indicator. The fraction of dissolved copper (x) was calculated as follows:

$$x = (\text{amount of copper passing to the solution}) / (\text{amount of copper in the malachite ore})$$

RESULTS AND DISCUSSION

Effect of ammonium acetate concentration

The effect of ammonium acetate concentration on the leaching of malachite ore was investigated in concentrations of 0.5, 1.0, 2.0, 3.0, and 4.0 mol/L. In these experiments, the solid-to-liquid ratio, particle size, stirring speed, and reaction temperature were kept constant at 3/500 g/mL, 140 μm , 350 rpm and 45 °C, respectively. Figure 2 shows the experimental results concerning the effect of solution concentration. Under the experimental conditions mentioned above, it can be seen that the extraction rate of copper from malachite ore increases considerably with an increase in ammonium acetate concentration. It was determined that the extent of extraction of copper increased from 37.0 to 99.1% when ammonium acetate concentration increased from 0.5 to 4 mol/L.

Effect of particle size

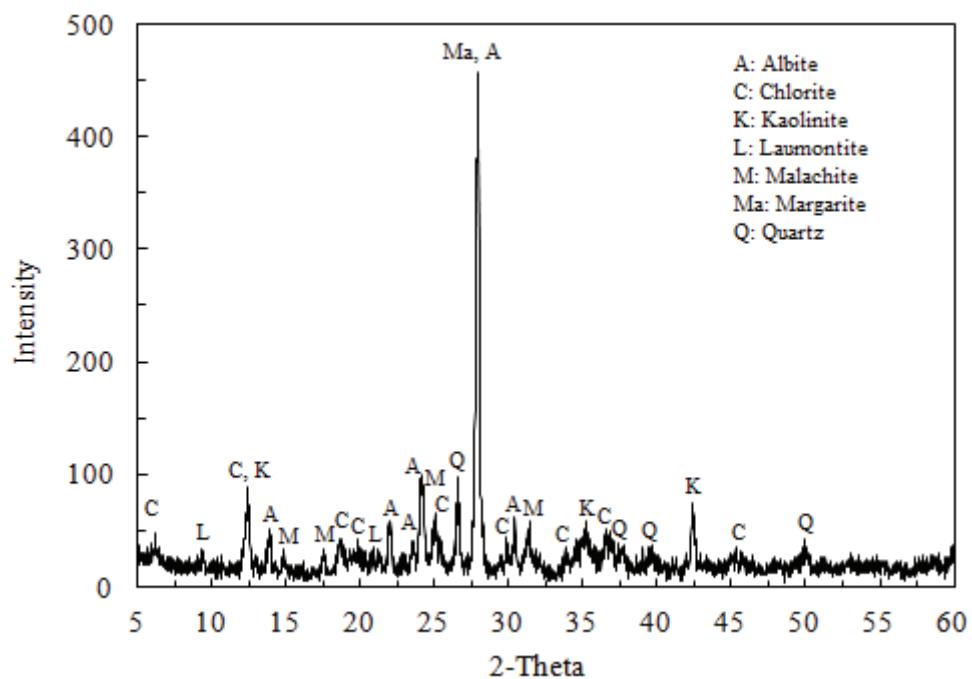


Figure 1. X-Ray diffractogram of malachite ore used in the study.

Table 1. Chemical analysis of malachite ore used in the study

Component	SiO_2	Al_2O_3	MgO	Fe_2O_3	CuO	CaO	Na_2O	Ignition loss	Other oxides
Value, %	46.40	17.00	7.30	6.87	5.20	3.30	2.30	8.00	3.63

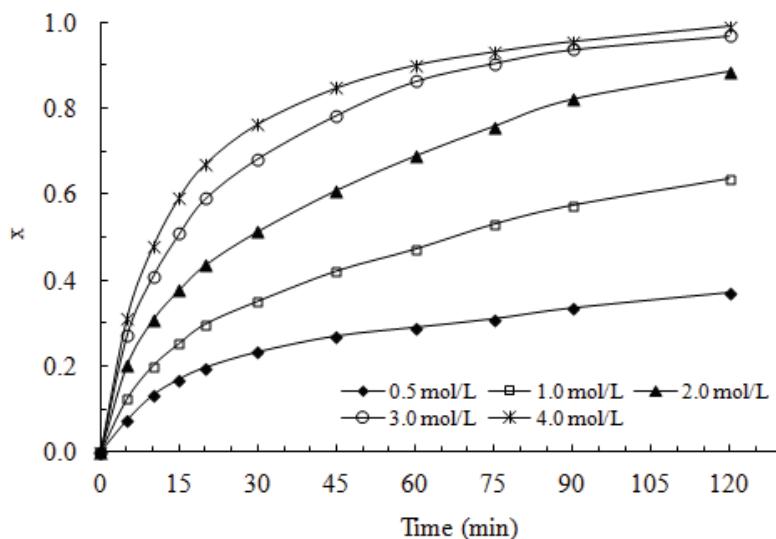


Figure 2. Effect of ammonium acetate concentration on leaching of malachite ore.

The effect of particle size on the leaching of copper from malachite ore was examined by using fractions of the sample with average particle size of 120, 140, 160, and 200 μm . In these experiments, the values of other parameters were kept constant at 3 mol/L, 45 °C, 3/500 g/mL, and 350 rpm. Figure 3 shows the effect of particle size on the leaching rate of copper from malachite ore. It is understood that the dissolution rate increases with decreasing particle size. After 120 min of leaching, it was found that the copper extraction from ore increased from 87.7 to 99.0% when average particle size of ore reduced from 200 to 120 μm . It is clear from these results that the amount of copper extracted from ore increases with decreasing particle size.

Effect of solid to liquid ratio

The effect of solid to liquid ratio on the leaching of the malachite ore was investigated by testing solid to liquid ratios of 2/500, 3/500, 5/500, 7/500, 10/500, and 15/500 g/mL while the values of ammonium acetate concentration, stirring speed, particle size, and reaction temperature were kept constant at 3 mol/L, 350 rpm, 140 μm and 45 °C, respectively. The variations of the leaching rate for various solid to liquid ratios are given in Figure 4. It was observed that the extent of the leaching of copper decreased slightly as solid to liquid ratio increased. This decrease may be due to the increase in solid amount in the reaction mixture. In fact, the leaching percentage of copper was 98.5% at solid to liquid ratio of 0.5/500 g/mL and

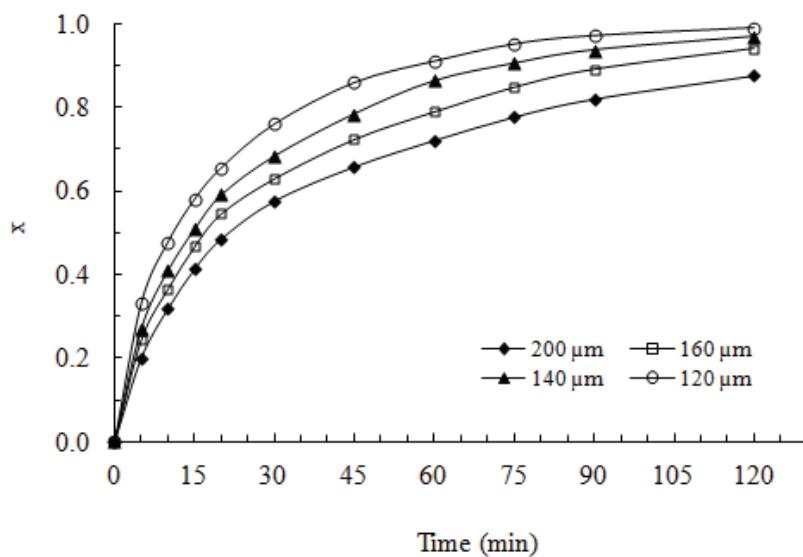


Figure 3. Effect of particle size on leaching of malachite ore.

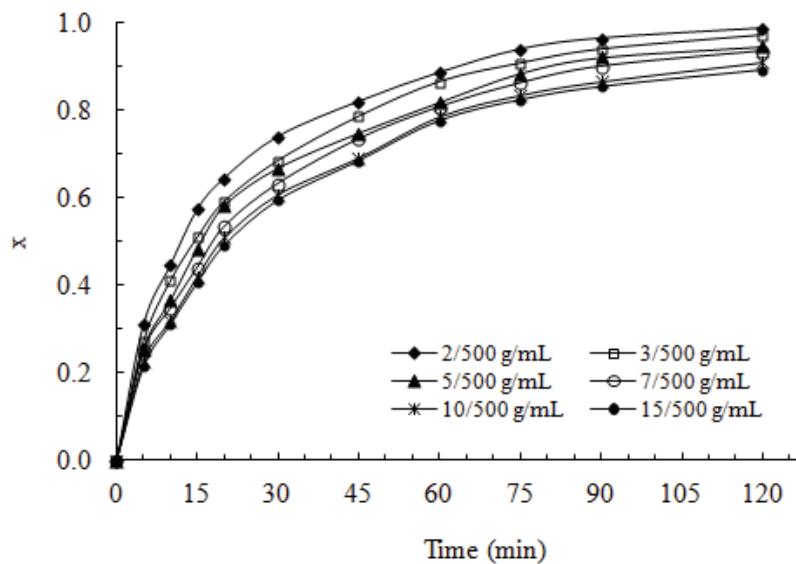


Figure 4. Effect of solid-to-liquid ratio on leaching of malachite ore.

decreased to 89% when solid to liquid ratio increased to 15/500 g/mL.

Effect of stirring speed

To examine the effect of stirring speed on the leaching of malachite ore, the experiments were performed at four different agitation rates in the range of 150–550 rpm. During experiments, the solution concentration, solid to liquid ratio, particle size, and reaction temperature were kept constant at 3 mol/L, 3/500 g/mL, 140 µm and 45 °C, respectively. The results obtained are graphically represented in Figure 5. When the agitation speed increased from 150 to 350 rpm, the leaching rate of copper increased. According to the experimental results, 92.7 and 96.9% of copper were extracted at stirring speeds of 150 and 350 rpm,

respectively. Further increase in agitation speed above 350 rpm provided a very small increase in the extent of extraction (98.0% at 450 rpm and 98.5% at 550 rpm).

Effect of reaction temperature

To determine the effect of reaction temperature on the leaching of malachite ore, the experiments were performed at temperatures of 25, 30, 35, 40, 45, 50 and 55 °C. In these tests, the solution concentration, liquid to solid ratio, particle size, and stirring speed were kept constant at 3 mol/L, 3/500 g/mL, 140 µm and 350 rpm, respectively. The results plotted in Figure 6 show that the leaching rate of malachite ore increased considerably with increasing reaction temperature. At a reaction temperature of 25 °C, 61.4% of copper was leached after 120 min of reaction, where-

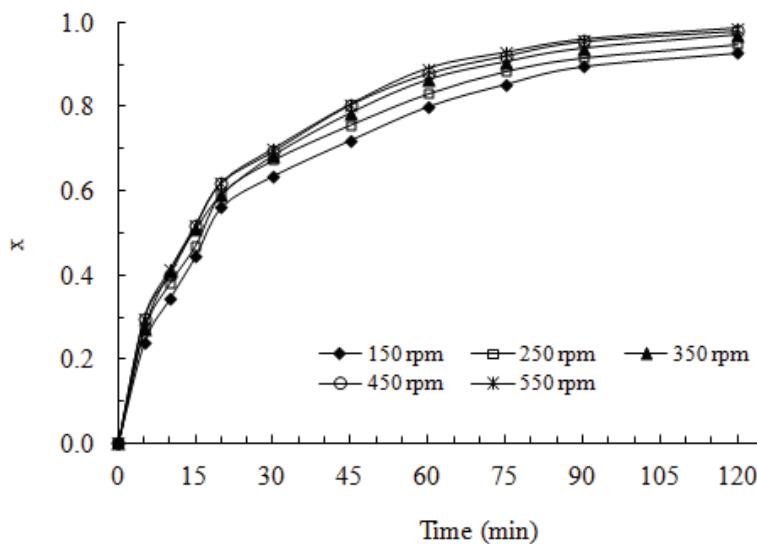


Figure 5. Effect of stirring speed on leaching of malachite ore.

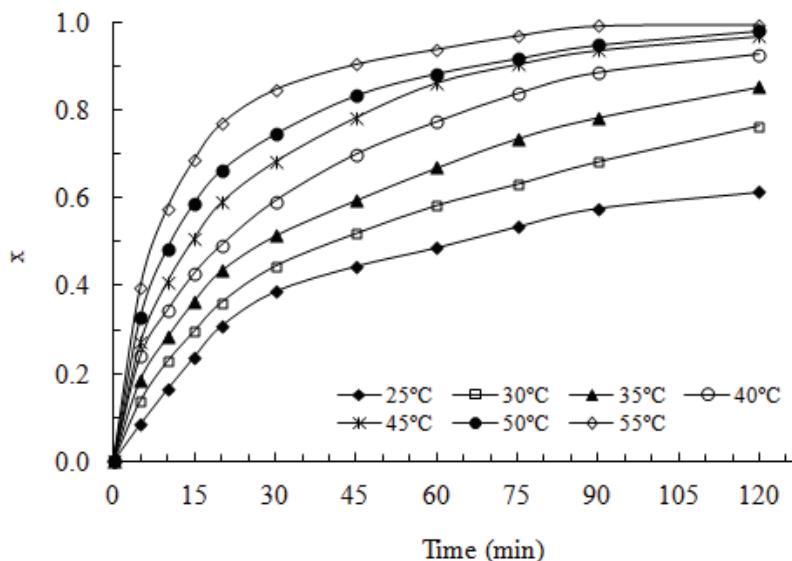


Figure 6. Effect of reaction temperature on leaching of malachite ore.

as at a temperature of 55 °C, 99.5% of copper was extracted after the same period of time. The results obtained from the temperature tests indicate that the reaction temperature has a significant effect on the extraction of copper from malachite ore.

Kinetic study

The leaching reaction of mineral particles with a reagent can be represented by the following reaction:



where A, B, and b represent the fluid reactant, the solid undergoing leaching, and stoichiometric coefficient, respectively. Because the leaching reactions are heterogeneous, the kinetic analysis of these kinds of reactions is generally performed by non-catalytic heterogeneous reaction models. The kinetics of leaching reactions is often described by the shrinking core model. According to the shrinking core model, it is thought that the reaction between solid and fluid reactants takes place on the outer surface of solid. The solid reactant is initially surrounded by a fluid film through which mass transfer occurs between the solid and the bulk fluid. As the reaction proceeds, the unreacted core of the solid shrinks towards the center of the solid, and a porous product layer forms around the unreacted core. However, it is considered that the initial outside radius of the solid does not change while the leaching reaction continues [6,30,31].

In this model, the rate of reaction between the solid particle and leaching reagent may be controlled by one of the following steps: diffusion through the fluid film, diffusion through the product layer, or the chemical reaction at the surface of unreacted [31].

The integrated rate equations of these steps can be written as follows:

$$x = k_f t \text{ (the film diffusion control)} \quad (10)$$

$$1 - 3(1-x)^{2/3} + 2(1-x) = k_d t \text{ (the product layer diffusion control)} \quad (11)$$

$$1 - (1-x)^{1/3} = k_r t \text{ (the chemical reaction control)} \quad (12)$$

where x is the conversion fraction of solid particle, k_f is the apparent rate constant for diffusion through the fluid film, k_d is the apparent rate constant for diffusion through the product layer, k_r is the apparent rate constant for the surface chemical reaction, and t is the reaction time.

Generally, the kinetics of any leaching reaction fit to one of the above models. In addition to these models, the mixed kinetic models can be used to determine the rate expressions of leaching reactions. The rate equations for the mixed kinetic models are given in the literature [30,32].

To determine the kinetic parameters and the rate-controlling step of the dissolution of malachite in ammonium acetate solutions, the experimental data obtained in the leaching step were analyzed based on the shrinking core model using the rate expression given in Eqs. (10-12) and the mixed kinetic models introduced in the literature. Among the mixed kinetic models applied to the leaching data, it was observed that the following model could be more suitable to demonstrate the kinetics of this leaching system:

$$(1 - (1-x)^{1/3})^2 = k_m t \quad (13)$$

Table 2. The apparent rate constants, k_f , k_r , k_d , k_m for kinetic models and correlation coefficient values

Parameter	Diffusion through the liquid film, x		Surface chemical reaction, $1-(1-x)^{1/3}$		Diffusion through the product layer, $1-3(1-x)^{2/3} + 2(1-x)$		Mixed kinetic model $(1-(1-x)^{1/3})^2$	
	k_f / min ⁻¹	R^2	k_r / min ⁻¹	R^2	k_d / min ⁻¹	R^2	k_m / min ⁻¹	R^2
Concentration (mol/L)								
0.5	0.0040	0.467	0.0015	0.581	0.0005	0.978	0.0002	0.994
1.0	0.0067	0.660	0.0028	0.826	0.0017	0.988	0.0007	0.995
2.0	0.0096	0.541	0.0049	0.881	0.0043	0.994	0.0020	0.996
3.0	0.0113	0.200	0.0069	0.824	0.0071	0.980	0.0039	0.995
4.0	0.0118	0.067	0.0077	0.787	0.0082	0.962	0.0050	0.997
Temperature (°C)								
25	0.0067	0.605	0.0029	0.673	0.0017	0.979	0.0007	0.995
30	0.0080	0.660	0.0037	0.828	0.0026	0.995	0.0011	0.994
35	0.0093	0.541	0.0046	0.804	0.0038	0.997	0.0017	0.995
40	0.0104	0.413	0.0058	0.810	0.0055	0.992	0.0027	0.995
45	0.0113	0.208	0.0069	0.766	0.0071	0.977	0.0039	0.995
50	0.0117	0.113	0.0074	0.623	0.0077	0.948	0.0044	0.999
55	0.0123	0.055	0.0089	0.510	0.0096	0.842	0.0064	0.999
Stirring speed (rpm)								
150	0.0107	0.320	0.0060	0.805	0.0057	0.981	0.0029	0.997
250	0.0110	0.220	0.0064	0.793	0.0064	0.976	0.0033	0.997
350	0.0113	0.200	0.0069	0.824	0.0071	0.980	0.0039	0.995
450	0.0115	0.161	0.0073	0.840	0.0076	0.980	0.0043	0.996
550	0.0116	0.158	0.0075	0.858	0.0078	0.982	0.0046	0.996
Particle size (μm)								
120	0.0119	0.132	0.0080	0.704	0.0085	0.944	0.0052	0.998
140	0.0113	0.463	0.0069	0.761	0.0071	0.977	0.0039	0.995
160	0.0107	0.217	0.0060	0.764	0.0058	0.993	0.0039	0.996
200	0.0098	0.012	0.0050	0.722	0.0044	0.994	0.0020	0.998
Solid/liquid ratio (g/mL)								
2/500	0.0117	0.097	0.0076	0.736	0.0080	0.966	0.0047	0.997
3/500	0.0113	0.463	0.0069	0.761	0.0071	0.977	0.0039	0.995
5/500	0.0110	0.325	0.0064	0.728	0.0063	0.972	0.0034	0.996
7/500	0.0107	0.127	0.0060	0.761	0.0059	0.982	0.0030	0.995
10/500	0.0104	0.010	0.0056	0.753	0.0052	0.982	0.0026	0.995
15/500	0.0102	0.048	0.0054	0.736	0.0049	0.974	0.0023	0.997

where k_m is the apparent rate constant for the mixed kinetic model.

For each experimental parameter, the apparent rate constant values and their correlation coefficients are given in Table 2. According to data in Table 2, the biggest regression coefficients were obtained for the mixed kinetic model. To test the validity of the model in Eq. (13), the left side of Eq. (13) *versus* time were plotted for the solution concentration, stirring speed, solid-to-liquid ratio, particle size, and temperature. The graphs obtained are given in Figures 7-11. The apparent rate constants determined from the slopes of the straight lines in Figures 7-11 and their correlation coefficients are given in Table 2. According to the results in Figures 7-11 and in Table 2, it can be

said that the kinetic model in Eq. (13) is appropriate to represent this leaching process.

To determine the effects of the reaction parameters on the reaction rate constant, the following semi-empirical model can be suggested:

$$k_m = k_o (C)^\alpha (PS)^\beta (SL)^\gamma (SS)^\nu \exp(-E_a/RT) \quad (14)$$

where C , PS , SL , SS , E_a , R , T represent the concentration, particle size, solid-to-liquid ratio, stirring speed, activation energy, universal gas constant, and temperature, respectively. The constants α , β , γ and ν are the reaction order for relating parameters, and k_o is the frequency or pre-exponential factor. Combining Eqs. (13) and (14), the following equation is obtained:

$$(1-(1-x)^{1/3})^2 = k_o (C)^\alpha (PS)^\beta (SL)^\gamma (SS)^\nu \exp(-E_a/RT) t \quad (15)$$

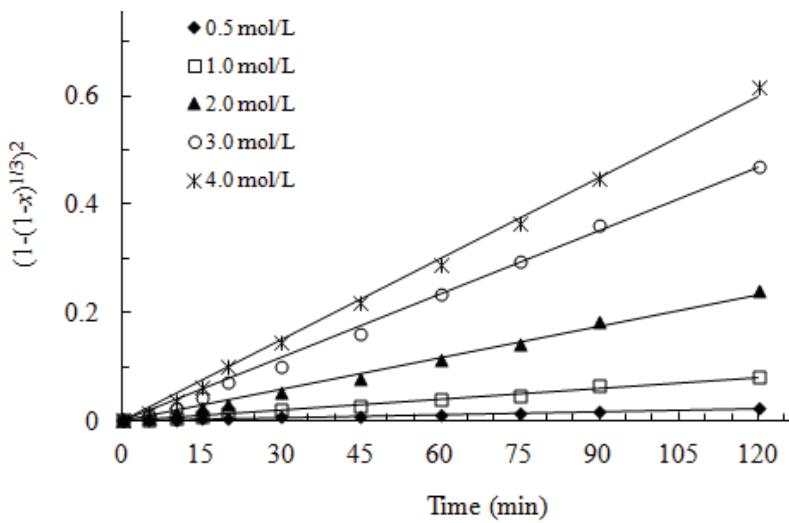


Figure 7. Plot of $(1-(1-x)^{1/3})^2$ versus t for various solution concentration ore.

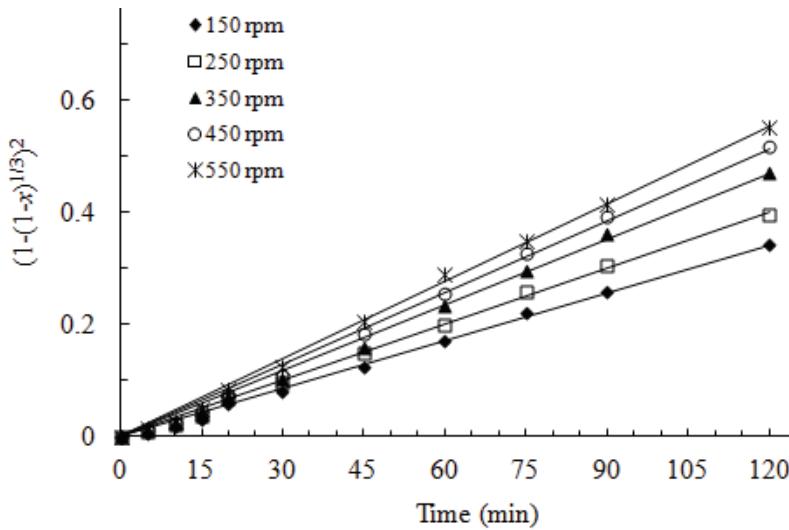


Figure 8. Plot of $(1-(1-x)^{1/3})^2$ versus t for various stirring speeds.

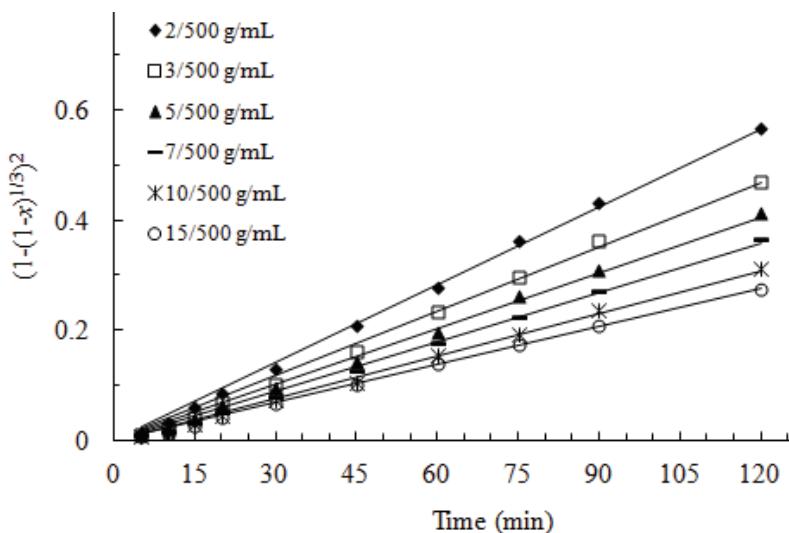
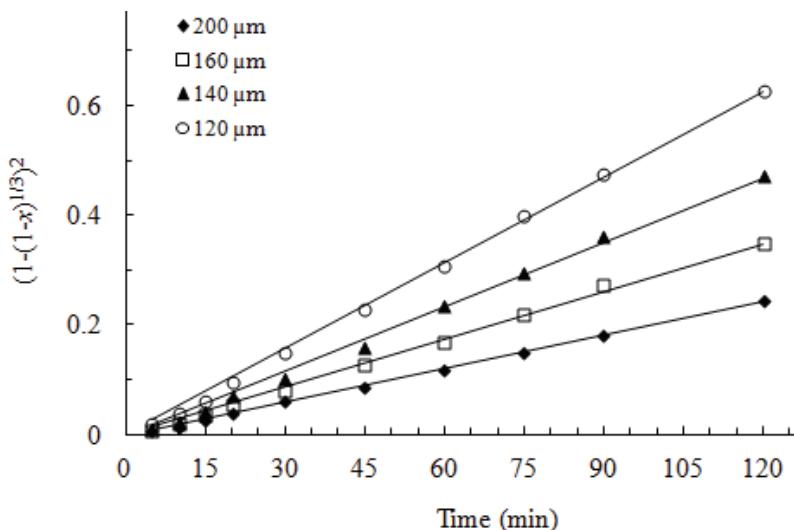
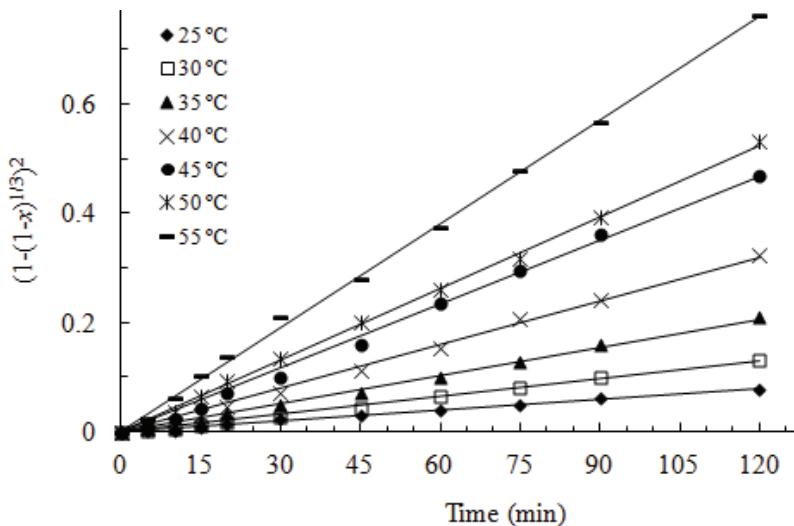


Figure 9. Plot of $(1-(1-x)^{1/3})^2$ versus t for various solid-to-liquid ratios.

Figure 10. Plot of $(1-(1-x)^{1/3})^2$ versus t for various particle sizes.Figure 11. Plot of $(1-(1-x)^{1/3})^2$ versus t for various reaction temperatures.

The constants α , β , γ and ν were estimated from the apparent rate constant values given in Table 2. The plots of $\ln k_m$ vs. $\ln C$, $\ln k_m$ vs. $\ln (PS)$, $\ln k_m$ vs. $\ln (SL)$, and $\ln k_m$ vs. $\ln (SS)$ were constructed using the values given in Table 2 for each parameter. The slopes of the straight lines in the graphs obtained assign the reaction order for each parameter. The values of the constants α , β , γ and ν were determined to be 1.56, -1.86, -0.34 and 0.36, respectively. The activation energy of this leaching process was found from the Arrhenius equation. The Arrhenius plot of the reaction is shown in Figure 12. The value of the activation energy of this leaching reaction was calculated to be 59.6 kJmol^{-1} .

As a result, the kinetic expression including the reaction parameters used in this leaching process can be written as follows:

$$(1-(1-x)^{1/3})^2 = 3.6 \times 10^5 (C)^{1.56} (PS)^{-1.86} \times \\ \times (SL)^{-0.34} (SS)^{0.36} \exp(7172/T) t \quad (16)$$

CONCLUSIONS

In the present paper, the leaching and kinetics of copper from malachite ore in ammonium acetate solutions was studied. It was determined that the leaching rate of copper increased with increasing solution concentration, reaction temperature and stirring speed, and decreasing particle size and solid-to-liquid ratio. As a result of kinetic analysis, it was found that the leaching reaction followed the mixed kinetic control model. A mathematical model containing experimental parameters was developed to represent the leaching kinetics of malachite ore in ammonium acetate solutions. The activation energy for this leaching

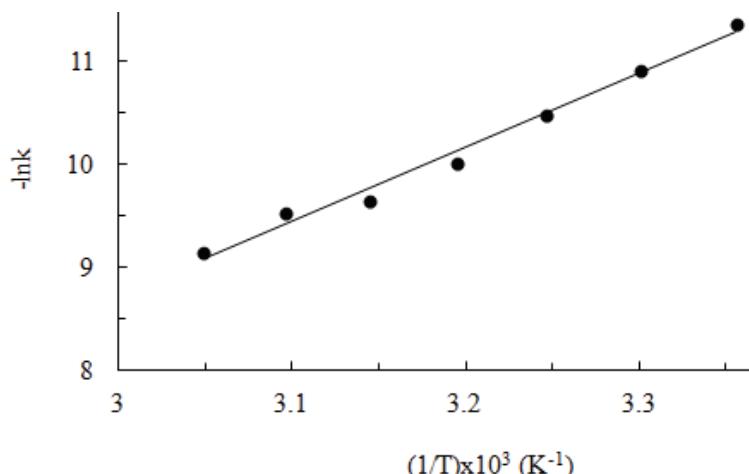


Figure 12. Arrhenius plot for leaching process.

process was to be 59.6 kJ/mol. It was observed that ammonium acetate solutions could be used as an effective leaching agent for leaching of copper from the malachite ore. The leaching process in this study can produce a pure solution for further processing of leach liquor due to the neutral pH operating conditions.

Acknowledgments

This work was supported by Inonu University Research Fund (Project No: 2010/18).

Nomenclature

b	stoichiometric coefficient, in Eq. (9)
C	concentration of ammonium acetate (mol/cm ³)
PS	particle size (cm)
SL	solid to liquid ratio (g/cm ³)
SS	stirring speed (rpm)
T	temperature (K)
t	reaction time (s)
x	converted fraction
k_d	apparent rate constant for diffusion through the product layer (1/s)
k_f	mass-transfer coefficient for fluid film (cm/s)
k_m	apparent rate constant for mixed kinetic model (1/s)
k_0	frequency or pre-exponential factor (1/s)
k_r	apparent rate constant for surface chemical reaction (1/s)
$\alpha, \beta, \gamma, \nu$	reaction orders, in Eq. (14)
R	universal gas constant (J/molK)
E_a	activation energy (J/mol).

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NAUČNI RAD

UPOTREBA AMONIJUM-ACETATA KAO ALTERNATIVNOG MEDIJUMA ZA IZLUŽIVANJE BAKRA IZ RUDE MALAHITA

Rastvori koji sadrže aminijak mogućavaju selektivnu luženje bakra iz njegovih ruda. U ovom radu proučavana je kinetika luženja malahitne rude pomoću amonijum acetata kao alternativnog lužnog rastvora, kao i efekti nekih eksperimentalnih parametara ovo izluživanje. Razvijen je kinetički model koji reprezentuje uticaj ovih parametara na brzinu izluživanja. Nađeno je da se brzina izluživanja povećava sa povećanjem koncentracije rastvora, temperature i brzine mešanja, kao i sa smanjenjem veličine čestica i odnosa čvrsto-tečno. Nađeno je da reakcija luženja sledi mešani kinetički kontrolni model. Određeno je da aktivaciona energija za ovaj proces luženja iznosi 59.6 kJ/mol. Prema tome, određeno je da rastvor amonijum acetata može biti efikasni lužni rastvor za ekstrakciju bakra iz rude malahita.

Ključne reči: bakar, kinetika, luženje, malahit.