

## Leaching kinetics of Chevreul's salt in ammonium chloride

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### Abstract

The study investigated the leaching kinetics of Chevreul's salt in ammonium chloride solutions by mechanical agitation experimental system. Reaction temperature, the concentration of ammonium chloride, stirring speed, and the solid-liquid ratio were selected as parameters. Experimental results were correlated using linear regression implemented in the statistical package program. The effect of parameters on Chevreul salt solubility was examined, and integrated rate expression of dissolution rate was found using kinetic models in solid-liquid heterogeneous reactions. The results revealed that the dissolution rate of Chevreul's salt decreased. At the same time, reaction temperature, concentration of ammonium chloride and stirring speed increased while dissolution rate decreased with the increase of the solid-liquid ratio. Based on the result of the applications of the obtained experimental results to the kinetic models, we can deduce that Chevreul's salt dissolution rate is controlled by diffusion through the ash (or product) layer. The activation energy of the dissolution reaction was 17.83 kJ/mol. The integrated rate expression, along with the effects of parameters on Chevreul's salt solubility, was found to be as follows:

$$1 - 3(1-X)^{2/3} + 2(1-X) = [2,96.10^{13} \cdot (CA)^{3,08} \cdot (S/L)^{-0,38} \cdot (W)^{1,23} e^{-9001,2 / T}] \cdot t$$

**Keywords:** Chevreul's salt, Ammonium Chloride, Leaching kinetics

### 1. INTRODUCTION

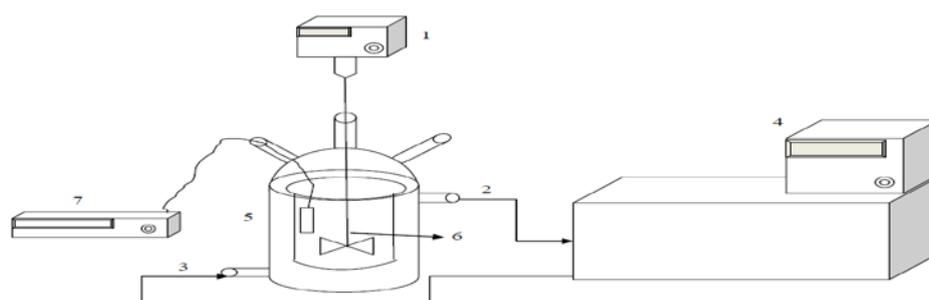
Copper is usually produced by cementation or electro-winning methods from solutions of soluble copper salts [1]. Precipitations of inorganic compounds from aqueous solutions generally are related to the physical or chemical processes. Due to hydrolytic action, ionic interaction or reduction, chemical processes may vary. Precipitation of copper sulphites from aqueous solutions, including copper, is significant in hydrometallurgical processes [2, 3]. Chevreul's salt is known as a stable mixed-valence sulphite, attracting many interests due to its intense brick red colour and highly stable mixed-valence state [4, 5]. The precipitation of Chevreul's salt is a critical stage in hydrometallurgical processes and is very important in aqueous systems [6-8]. In recent years, studies of compounds of this type have intensified. Chevreul's salt has been obtained using various methods and reagents [2, 3]. Conklin and Hoffmann have researched the metal ion-sulfur (IV) chemistry, thermodynamics, and kinetics of transient iron(III)-sulphur(IV) complexes. Their measurements indicated that sulphite binds the metal through oxygen [9]. Silva et al. [10] have examined the synthesis, identification, and thermal decomposition of double sulphites like  $Cu_2SO_3 \cdot MSO_3 \cdot 2H_2O$  (M=Cu, Fe, Mn or Cd). These salts have been obtained by saturation with sulphur dioxide gas from aqueous solutions of M(II) sulphates at room temperature. The thermogravimetry analysis and differential scanning calorimetric methods estimated the thermal behavior of double sulfites. They reported that these salts are thermally stable up to 200 °C and isostructural with Cu(II) replaced by Mn(II), Fe(II), and Cd(II) ions in  $Cu_2SO_3 \cdot MSO_3 \cdot 2H_2O$ . Çolak et al. [11] obtained 99.78% pure copper powder from Erzurum-Narman region oxidized copper containing 4.48% Cu. They precipitated Chevreul's salt ( $Cu_2SO_3 \cdot CuSO_3 \cdot 2H_2O$ ) using ammonia and sulphur dioxide. The best precipitation conditions of Chevreul's salt were found at pH: 4, the stirring speed: 600 rpm, the temperature: 60°C, passing time SO<sub>2</sub>: reaction time of 1 minute after passing SO<sub>2</sub>: 6 minutes. De Andrade et al. [12] have researched isomorphous series of double sulphites such as the  $Cu_2SO_3 \cdot MSO_3 \cdot 2H_2O$  (M=Cu, Fe, Mn or Cd) type. They found that the isomorphous Cu(II) in Chevreul's salt could be replaced by a divalent metal ion, forming an isomorphous series whose properties strongly depend on the nature of the M(II) cation. They determined that these mixed valence systems can be used as a model to identify intermediates under atmospheric conditions to evaluate the role of transition metals as catalysts of S(IV) autoxidation in the conversion of SO<sub>2</sub> in the atmosphere because of their attractive properties. Innoue et al. [13] Chevreul's salt synthesized by a reaction between  $CuSO_4$  and  $NaHSO_3$  and characterized by X-rays photoelectron spectroscopy, magnetic susceptibility, EPR, and electronic spectroscopy. Parker and Muir [14] determined some conditions for precipitation of Chevreul's salt from impure leach solutions. They obtained 75g of pure particulate copper per unit liter of solutions [14]. Çalban et al. [15] researched statistical modeling of Chevreul's salt recovery from copper leach solutions. They determined the optimum precipitation conditions of Chevreul's salt using leach solutions. They found at pH 3, temperature 62 °C, stirring speed 600 rpm, reaction time 12 minutes, SO<sub>2</sub> flow rate 358 L.h<sup>-1</sup> and concentration of  $CuSO_4$  solution 7.383 g Cu.L<sup>-1</sup>. Yeşilyurt and Çalban [16] precipitated Chevreul's salt from a mixture of  $CuSO_4$  and  $Na_2SO_3$  solutions. They determined the optimum precipitation conditions as temperature 60 °C,  $[SO_3^{2-}]/[Cu^{2+}]$  ratio 1.6, pH 3, stirring speed 500 rpm, and reaction time 20 minutes. Giovannelli et al. [17] investigated the surface layers formed on oxidized copper exposed to SO<sub>2</sub> in humid air under hypoxic conditions. Chevreul's salt exhibited orthorhombic symmetry at room temperature. A mechanical analogy with a bronze disease of archaeological artifacts has been indicated. Fischmann et al. [18] have

investigated upgrading a chalcopyrite concentrate by copper (II) reaction and unexpected formation of Chevreul's salt. Aqueous copper (II) recovery onto a chalcopyrite concentrate was shown to be rapid at 60 °C in the presence of S(IV) in this instance sulphite. Amazingly, rather than partial conversion of chalcopyrite to a copper sulphite such as chalcocite or covellite, copper was precipitated in the form of Chevreul's salt. This process applies to increasing the copper grade of chalcopyrite concentrates using the leachate from a Galvanox™ leach of flotation tailings.

The study investigates the dissolution kinetics of Chevreul's salt in ammonium chloride solutions in a mechanical agitation system. There is no study reported in the literature about such a procedure. Investigation of the dissolution conditions and the dissolution kinetics of Chevreul's salt in ammonium chloride solutions are essential for industrial application. The dissolution kinetics of Chevreul's salt in disodium hydrogen phosphate solutions were examined according to the heterogeneous reaction models. In our study, reaction temperature, the concentration of ammonium chloride solutions, stirring speed, and solid/liquid ratio were chosen as process parameters.

## 2. MATERIAL AND METHOD

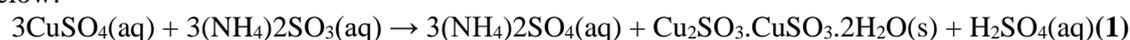
Leaching experiments were conducted under atmospheric pressure conditions. All reagents used in the experiments were prepared from analytical grade chemicals (Merck) and distilled water. A constant temperature water circulator was used in combination with the reactor to maintain the mixture in the reactor at a constant temperature. The experiments were carried out in a 500 mL spherical glass reactor. The reactor was equipped with a reflux condenser to prevent evaporation during heating and a mechanical stirrer to obtain a homogeneous suspension. The mechanical agitation experimental system is standard and can be seen in Figure 1.



**Figure 1.** Mechanical Agitation Experimental System (1.) Mechanical agitator, (2.) Water output, (3.) Water input, (4.) Water circulator heater, (5.) Glass reactor, (6.) stirrer, (7.) pH meter

### 2.1 Experimental Procedure to Form a Chevreul Salt

The current experiments were carried out in 2 stages. Primarily, using the optimum conditions, copper sulfate and ammonium sulphite were reacted to form a Chevreul salt. A typical experiment conducted was as follows: 250 mL of 0.25 M  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  solution was poured into a 500 mL water-quenched glass reactor. The reactor's temperature was set at 60 °C using a Memmert brand digital temperature controlled. The reactor contents were mixed with the Yellow Line brand mechanical mixer of stirring speed 600 rev/min. At the same time, a WTW brand pH-meter probe measuring the pH of 4 and temperature was immersed in the solution. When the reactor content reaches the desired temperature, the mixing operation is started by adjusting the mechanical mixer to the desired speed. The ammonium sulphite solution,  $(\text{NH}_4)_2\text{SO}_3$ , was carefully added dropwise over the desired period to allow the pH to remain at 4 during the reaction. The dissolution process continued during the reaction. At the end of the reaction, the reactor contents were drained, filtered using filter paper, and washed with distilled water. The resulting precipitate was placed in an oven set at 105 °C and allowed to stand for 2 hours to remove the moisture altogether. The precipitate removed from the oven was stored in a desiccator. The average grain diameter of the Chevreul salt obtained in our experiments was 0.05292 mm, and the density was found to be 3.57 g / cm<sup>3</sup>. The experimental setup in which the precipitation reaction takes place is given in Figure 1. The reaction of copper sulfate and ammonium sulphite into the reaction and forming the Chevreul salt is given below:



### 2.3 Experimental Procedure to Leach the Chevreul Salt

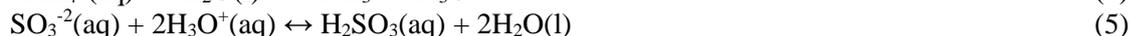
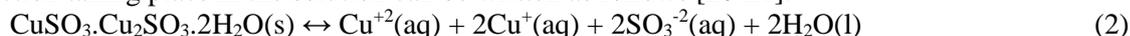
A typical experiment conducted was as follows: 300 mL of ammonium chloride solutions were poured into the reactor. The solution was heated to the desired temperature, which was kept constant. All experiments were carried out using 231 µm size fractions. After this, prominent quantities of solid Chevreul's salt were added to the solutions in the reactor. Stirring of the solution was started immediately there after. The duration of the treatment depended on the experimental conditions. At definite time intervals, 2-mL samples were taken for  $\text{Cu}^{2+}$  analysis by a complexometric method [19- 20] with EDTA using 5-sulfosalicylic acid as the indicator and for total Cu analysis by atomic absorption spectrophotometry (Shimadzu AAS-670). The degree of dissolution of Chevreul's salt was determined as a function of time. Each

experiment was repeated twice, and the arithmetic average of the results of the two experiments was used in the kinetic analysis. The suspension's homogeneity was obtained at a stirring speed of 630 rpm. Because of this, the stirring speed rate of 630 rpm was a constant value in all experiments to guarantee homogeneity in the batch reactor. The experimental setup in which the leaching reaction takes place is given in Figure 1.

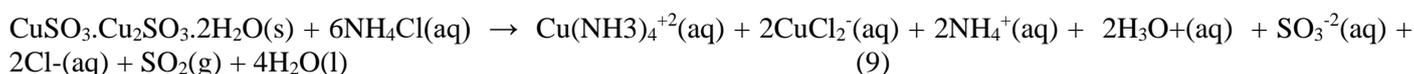
### 3. RESULT AND DISCUSSION

#### 3.1 Dissolution reactions

The reaction taking place in the solution can be written as follows [20-21]:



When Chevrel's salt is added to the ammonium chloride solutions, the total reaction taking place in the solution can be written as follows:



#### 3.2 Effect of parameters

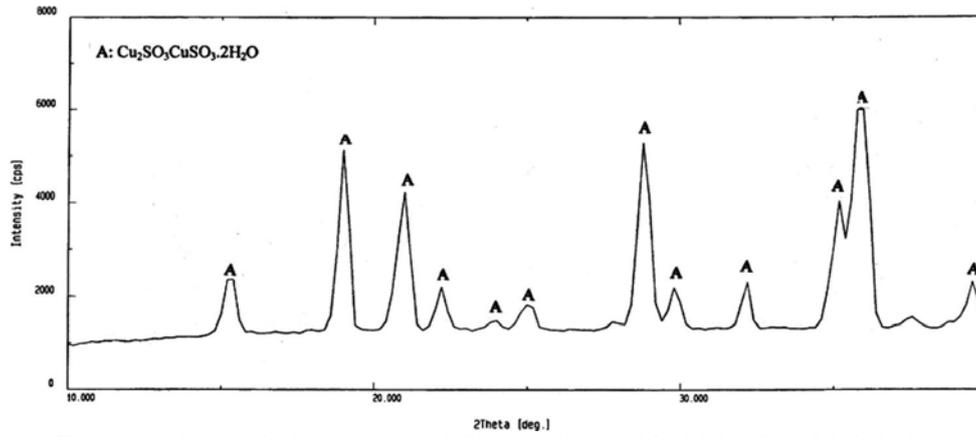
Reaction temperature, the concentration of ammonium chloride solutions, stirring speed, and solid/liquid ratio was selected as process variables to investigate their effects on the dissolution level of Chevrel's salt. The parameters are chosen, and their ranges can be seen in Table 1. In the experiments, while the effect of one parameter was studied, the values of other parameters shown with asterisks in Table 1 were kept constant. 300 mL of ammonium chloride solutions were used and kept constant in all experiments. Homogeneity of suspension in the reactor was obtained with a stirring speed of 700 rpm, kept constant in all experiments.

X-ray diffractogram of Chevrel's salt [ $\text{Cu}_2\text{SO}_3 \cdot \text{CuSO}_3 \cdot 2\text{H}_2\text{O}$ ] can be seen in Fig. 2. The data obtained were plotted in the form of time versus fractional conversion, as appears in Fig. 3-6. In these figures, the fractional conversion  $X$  is defined:

$$X = \frac{\text{amount of dissolved Cu in the solution}}{\text{Amount of Cu in the original sample}} \quad (10)$$

**Table 1.** Parameters chosen and their ranges

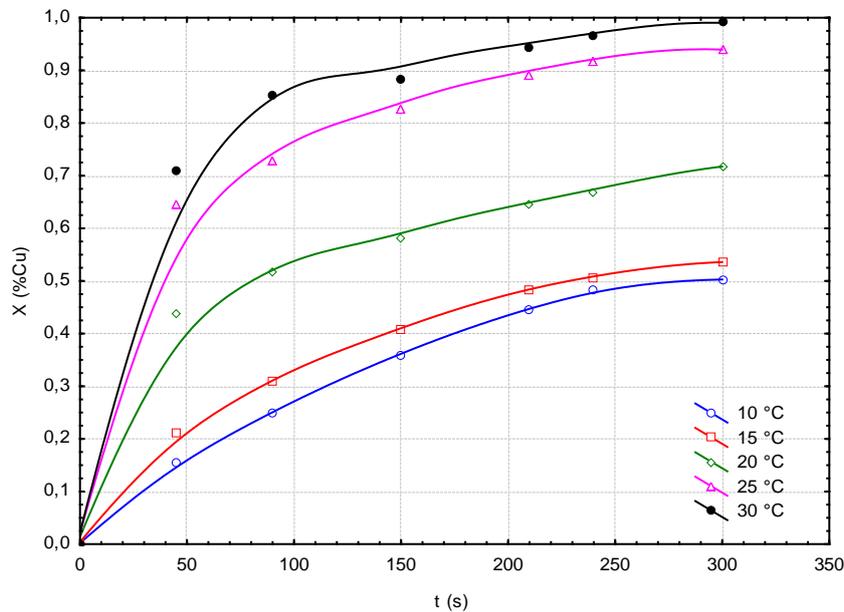
parameter	values
reaction temperature (°C)	10, 15, 20, 25, 30
concentration of ammonium chloride (mol L <sup>-1</sup> )	0,80 ; 1,00 ; 1,50 ; 1,75 ; 2,00 ; 2,25; 3,00
solid/liquid ratio (g/mL)	1/100, 1/75, 1/50, 1/25 and 1/12,5
stirring speed (rpm)	100, 200, 300, 400, 500, 600, 700 and 800
Reaction time (s)	45, 90, 150, 210, 240 and 300



**Figure 2.** X-ray diffractogram of Chevrel's salt [ $\text{Cu}_2\text{SO}_3.\text{CuSO}_3.2\text{H}_2\text{O}$ ].

### 3.3 Effect of reaction temperature

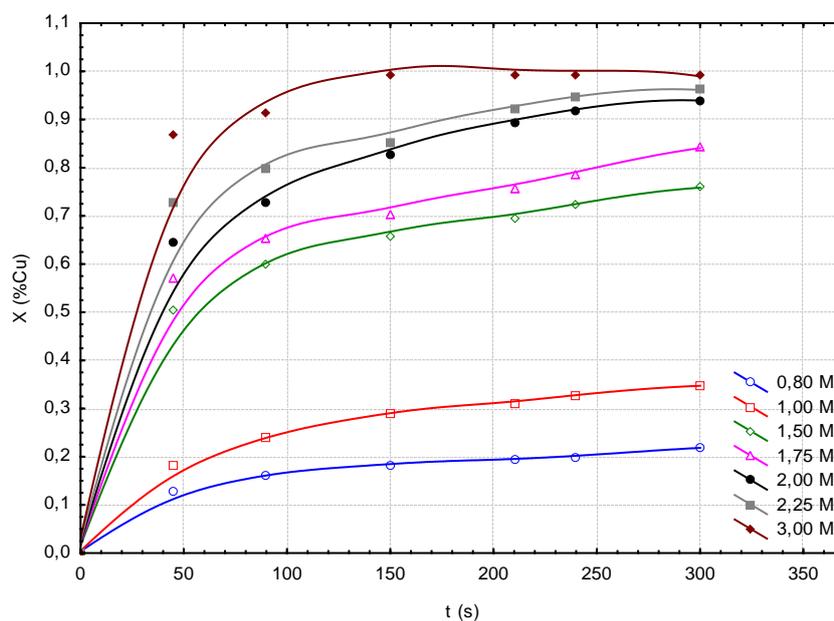
The reaction temperature is a factor of great importance for the leaching kinetics. The effect of reaction temperature was examined at 10, 15, 20, 25, and 30 °C. The difference in activation energies can probably be attributed to the differences in the chemical reaction mechanism. The dissolution curves obtained for 10-30 °C are shown in Fig.(3). It can be shown from Fig.(3) that the quantity of Chevrel's salt dissolved increases with increasing reaction temperature.



**Figure 3.** Effect of reaction temperature on dissolution rate of Chevrel's salt

### 3.4 Effect of concentration of $\text{NH}_4\text{Cl}$ solution

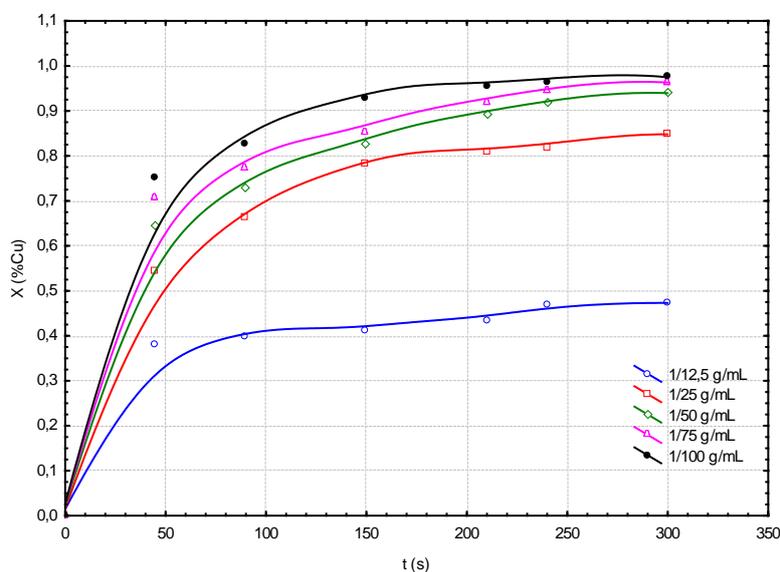
The leaching rate generally increases with increased reagent concentration, but only up to a certain maximum level. The effect of concentration of ammonium chloride solutions was studied by varying to 0,80 , 1,00 , 1,50 , 1,75 , 2,0 , 2,25 ve 3,0 M . The dissolution curves are given in Fig. (4). It can be seen from Fig. (4) that the dissolution level of the process increases with an increase in the concentration of ammonium chloride solutions



**Figure 4.** Effect of concentration of ammonium chloride on dissolution rate of Chevrel's salt

### 3.5 Effect of stirring speed

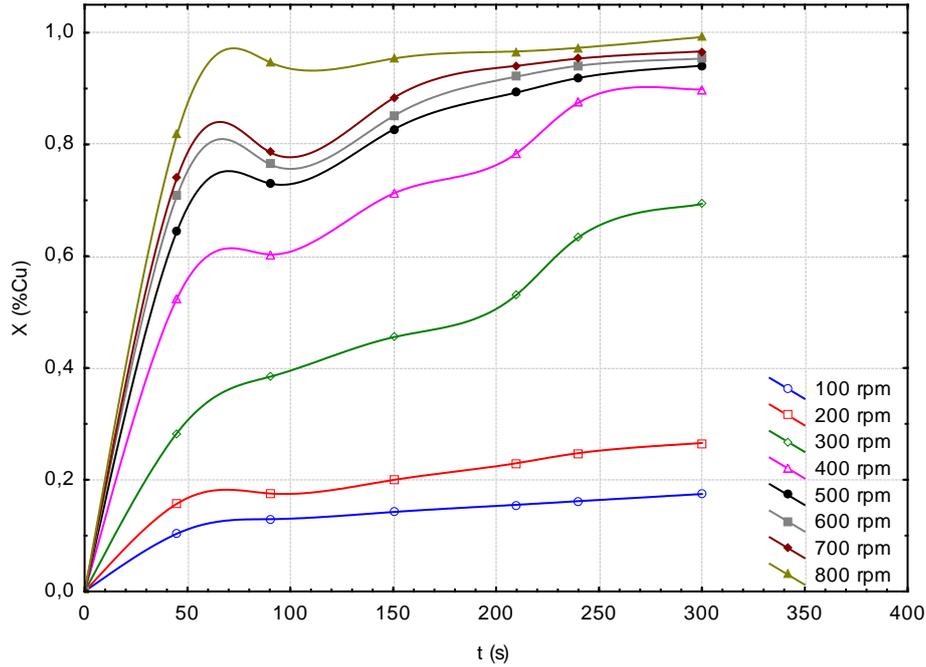
The effect of the stirring speed on the dissolution rate of Chevrel's salt was investigated at 100, 200, 300, 400, 500, 600, 700, and 800 rpm. The change between stirring speed and conversion can be seen in Fig. (5). It can be seen from Fig.(5) that the dissolution rate increases with an increase in the stirring speed rate.



**Figure 5.** Effect of solid/liquid ratio on dissolution rate of Chevrel's salt

### 3.6 Effect of solid/liquid ratio

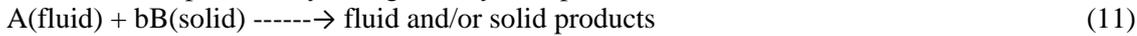
The effect of the solid/liquid ratio on the dissolution rate of Chevrel's salt was investigated by varying ratios to 1/100, 1/75, 1/50, 1/25, and 1/12.5 g.mL<sup>-1</sup>. The dissolution curves are given in Fig. 6. It can be seen from Fig. (6) that the dissolution rate decreases with an increasing solid/liquid ratio. This situation can be explained by the decrease in the number of Chevrel's salt particles per solution amount.



**Figure 6.** Effect of stirring speed on dissolution rate of Chevrel's salt

### 3.7 Analysis of Kinetic

The solid-fluid heterogeneous reaction rate can be obtained from the heterogeneous reaction model. The leaching reaction of ore particles by a reagent may be represented as follows:



The shrinking core model often describes the leaching kinetic reactions. The reaction between solid and fluid reactants takes place on the surface of the solid. A fluid film surrounds the solid reactant. Mass transfer occurs between the solid and the bulk fluid. The experimental data were analyzed using the unreacted shrinking core model to evaluate the rate-controlling step [21]. The heterogeneous reaction model gives rate equations for each control mechanism. The step with the highest resistance is the rate-controlling step. The model has been used in analytical and numerical methods for solid-liquid heterogeneous systems. Integrated rate equations for the unreacted shrinking-core model and the other models are shown in Table 2. According to the model, the kinetic data were treated by equations in Table 2.

**Table 2.** Integrated rate equations for the un-reacted shrinking core model and the other models

rate-controlling step	rate equation		
surface chemical reaction	$t/t^* = [1 - (1 - X_B)^{1/3}]$	$t^* = \rho_B R / bksC_{Ag}$	(12)
the film diffusion control	$t/t^* = X_B$	$t^* = \rho_B R / 3bkgC_{Ag}$	(13)
diffusion control through the ash or product layer	$t/t^* = [1 - 3(1 - X_B)^{2/3} + 2(1 - X_B)]$	$t^* = \rho_B R^2 / 6bDeC_{Ag}$	(14)
For the first-order pseudo-homogeneous model		$-\ln(1-X) = kt$	(15)
For the second-order pseudo-homogeneous model		$(1-X)^{-1} = kt$	(16)
Avrami model		$-\ln(1-X) = kt^m$	(17)

The data were analyzed based on the shrinking core model using the rate expression given in Eqs. [12-17] into Table 2 to determine the rate-controlling step of the leaching of Chevrel's salt in ammonium chloride solutions. . To

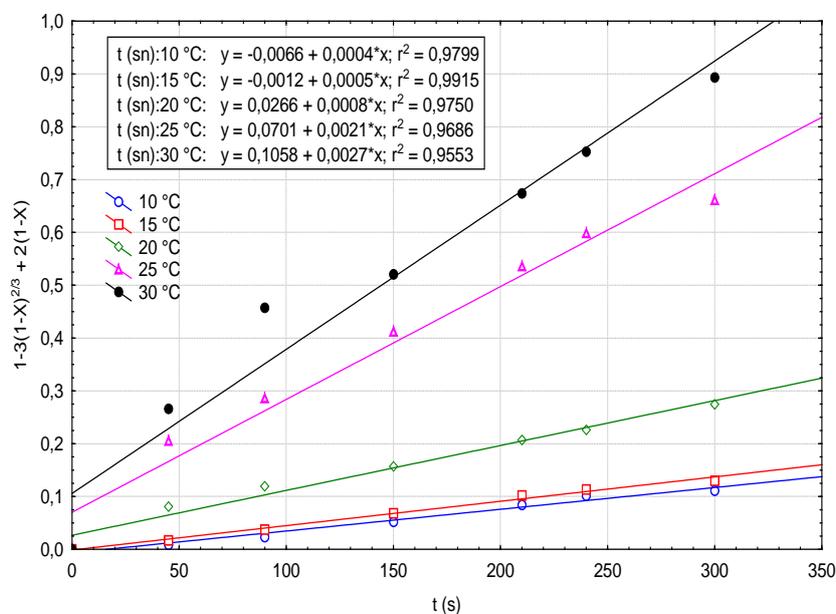
obtain the apparent rate constants of each step, the left side of Eqs. [12-17] was plotted vs. the reaction time, and the slopes of the straight lines were equal to the rate constants. The apparent rate constants,  $k_1$ ,  $k_r$ , and  $k_d$ , for kinetic models and correlations coefficients values for reaction temperatures are shown in Table 3. The high regression coefficients were obtained for the chemical reaction models. Applying the above models to the experimental data will help to determine the dissolution kinetics of the process. In cases where the chemical reaction is much faster than the diffusion, the leaching is said to be diffusion-controlled. The leaching mechanism often becomes diffusion controlled when, during the leaching, a porous product layer forms on the surface of the particle to be leached. The diffusion-controlled leaching mechanism of the spherical particle is often called the shrinking core model [23-26]. Experimental data fits the heterogeneous chemical reaction controlled in the form of  $t/t^* = 1 - 3(1-X)^{2/3} + 2(1-X)$ .

**Table 3.** The Correlations Coefficients Values based on the models.

	Diffusion Through the Liquid Film	Surface Chemical Reaction	Diffusion Through the Product Layer
<b>T (°C)</b>	<b>X</b>	<b><math>1-(1-X)^{1/3}</math></b>	<b><math>1-3(1-X)^{2/3}+2(1-X)</math></b>
		<b><math>r^2</math></b>	
<b>10</b>	0,9398	0,9630	<b>0,9799</b>
<b>15</b>	0,8953	0,9345	<b>0,9915</b>
<b>20</b>	0,7403	0,8403	<b>0,9750</b>
<b>25</b>	0,6693	0,8748	<b>0,9686</b>
<b>30</b>	0,6059	0,8884	<b>0,9553</b>

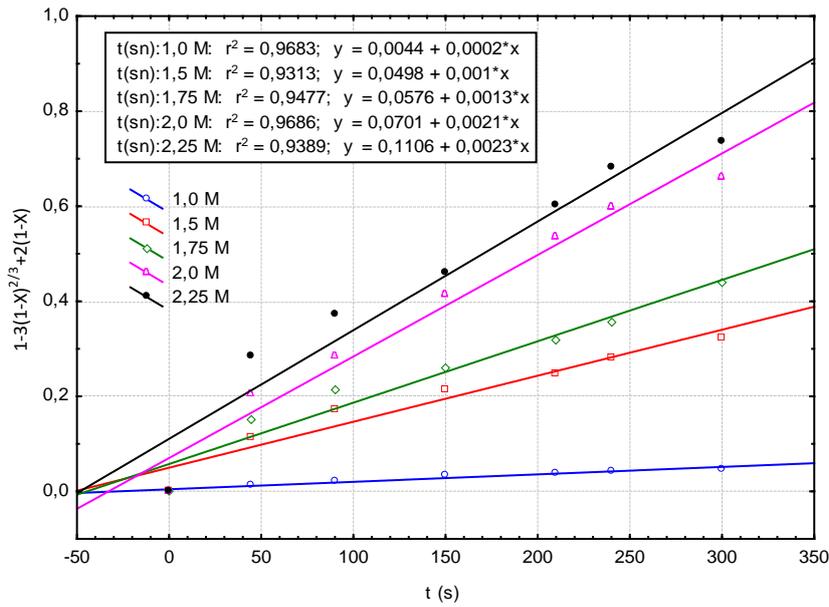
The evidence for this proposal is as follows:

Variations of  $1 - 3(1-X)^{2/3} + 2(1-X)$  with time for reaction temperatures and concentration of  $\text{NH}_4\text{Cl}$  can be seen in Fig. (7 & 8), respectively.

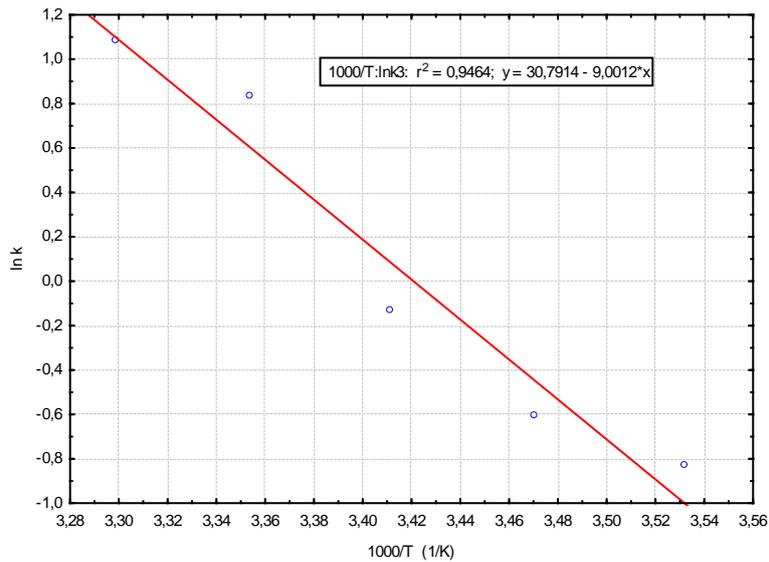


**Figure 7.** Variation of  $1 - 3(1-X)^{2/3} + 2(1-X)$  with time for reaction temperatures

Variation of apparent reaction rate constant with all reaction temperatures is shown in Table 3. The regression analysis has shown that experimental data correlate well with Eq. 14 in Table (2), which means that the dissolution is controlled by diffusion through the ash (or product) layer. The regression coefficient was found to be 0.9874 as higher linearity. The Arrhenius plots of  $\ln k_s$  versus  $1/T$  were drawn to find the activation energy of the reaction [23-26] and presented in Fig. (9). From the slopes of the straight lines of Fig.(9), the activation energy of the reaction is found to be 17.83 kJ/mol.



**Figure 8.** Variation of  $1 - 3(1-X)^{2/3} + 2(1-X)$  with time for concentration of ammonium chloride



**Figure 9.** Arrhenius plot of the dissolution process for reactions temperatures

#### 4. CONCLUSIONS

It was concluded that the dissolution extent increases with an increased stirring speed rate between 100-700 rpm. The dissolution process follows a shrinking core model with the heterogeneous reactions controlled by diffusion through the ash (or product) layer as the rate-controlling step. The activation energy of the reaction is found to be 17.83 kJ/mol. The integrated rate expression, along with the effects of parameters on Chevreul's salt solubility, was found to be as follows:  $1-3(1-X)^{2/3}+2(1-X) = [2,96.10^{13} \cdot (CA)^{3,08} \cdot (S/L)^{-0,38} \cdot (W)^{1,23} e^{-9001,2 / T}] \cdot t$

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## NOMENCLATURE

A	=	fluid reactant,
B	=	solid mineral or ore
b	=	stoichiometric coefficient
C	=	concentration of ammonium chloride solutions (mol m <sup>-3</sup> )
C <sub>A</sub>	=	concentration of A in the bulk solution (mol m <sup>-3</sup> )
D	=	mean particle size (m)
D <sub>e</sub>	=	diffusion coefficient (m <sup>2</sup> min <sup>-1</sup> )
E <sub>A</sub>	=	activation energy (kJ kmol <sup>-1</sup> )
k <sub>dm</sub>	=	mass transfer coefficient (m min <sup>-1</sup> )
k <sub>s</sub>	=	reaction rate constant for surface reaction (mol min <sup>-1</sup> )
k <sub>o</sub>	=	frequency or pre-exponential factor (min. <sup>-1</sup> )
L	=	amount of liquid (mL)
n	=	mol number (mol)
r	=	correlation coefficient
R	=	universal gas constant (kJ kmol <sup>-1</sup> )
R	=	initial radius of a solid particle (m)
S	=	amount of solid (g)
T	=	reaction temperature (°C)
t	=	reaction time (h)
t*	=	reaction time for complete conversion (min.)
X	=	fractional conversion of B <sub>2</sub> O <sub>3</sub>
W	=	stirring speed (rpm)
B	=	molar density of solid reactant (kmol m <sup>-3</sup> )
k <sub>1</sub>	=	apparent rate constants for diffusion through the liquid film
k <sub>r</sub>	=	apparent rate constants for surface chemical reaction
k <sub>d</sub>	=	apparent rate constants for diffusion through the product layer