

## A Comparison of the Leaching Kinetics and Mechanism of Calcined Magnesite with an Organic Leach Reagent

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**Summary:** The leaching kinetics of calcined magnesite in aqueous lactic acid solutions was investigated in a batch reactor by using parameters such as temperature, acid concentration, particle size, solid/liquid ratio, and stirring speed. A mathematical model was evaluated for the rate increased by decreasing particle size and by increasing temperature; and initially the leaching is increased in terms of acid concentration and then decreased by increasing concentration. It was shown that the new mathematical model for calcined magnesite particle was:

$$-1.9041 \cdot \ln((1 - X_B)^{1/3} + 1.157) + 0.952 \ln((1 - X_B)^{2/3} - 1.156(1 - X_B)^{1/3} + 1.338) - 3.298 \arctan(0.577(1.73(1 - X_B)^{1/3} - 1)) = \frac{3 \cdot b \cdot k_s \cdot C_{A0}}{\rho_B \cdot R} \cdot t$$

It was determined that experimental data complies with shrinking core model for fluid-solid systems. It was also found that the stirring speed did not change the dissolution and that the dissolution rate was controlled by surface chemical reaction. Additionally, the fact that leaching was controlled by the chemical reaction was also supported by the relationship between the rate constant and the particle radius. The activation energy of the process was determined to be 30.61 kJ mol<sup>-1</sup>.

**Keywords:** Calcined Magnesite; Lactic acid; Leaching; Kinetics; Organic reagent.

### Introduction

Leaching of the ores is a typical industrial application existing in fields such as ceramic, medicine, hydrometallurgy, and crystallography [1]. The increasing demand for metals and metallic compounds in the world has required intensive studies for the extraction of metals from the ores. Hydrometallurgical or pyrometallurgical methods can be used for the extraction of ores. Leaching processes constitute the first step of the hydrometallurgical methods and leaching kinetics an economically plays an important role in the extraction of metals and compounds [2].

For this purpose, many studies have been conducted with hydrometallurgical method. Inorganic acids have been commonly used as leach reagent in these studies [2, 3-7]. In addition to this, organic acids can also be used as leach reagent in recent years [8-14].

One of the ores is natural magnesite (Magnesium carbonate) which has played an important role in the production of magnesium and its compounds. Turkey posses an extensively natural magnesite ore. The magnesite ore can be used in three main forms: (1) crude magnesite, primarily for

use in chemicals, pharmaceutical industry as an anti-acid, agriculture, lime, in paper and sugar industries (2) dead- burned magnesia, a durable refractory for use in cement, pharmaceutical industry, glass, steel and which is used in metallurgical industries (3) caustic calcined magnesia, for use in making oxychloride and oxysulphate cements and various environmental and chemical applications [15].

Both natural magnesite and calcined magnesite (Magnesium Oxide) also contain some impurities such as silisium, iron and calcium which affect the quality of products. As inorganic acids H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub> or HCl are commonly used, the undesired impurities, particularly iron, can be appreciably dissolved in the leaching process. On the other hand, organic reagents have high selectivity since their dissolving capacity is low. This provides an advantage in leaching of metal oxide and carbonate compounds which include some impurities. Also, use of organic acids at high temperatures may be limited because of the low boiling temperatures and their decomposition [16, 17].

The leaching rate strongly depends on properties of the solid as well as the process

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conditions [7]. In general, the reactivity of calcined magnesite is much higher than the natural magnesite. Therefore, usage of the calcined magnesite has provided an important advantage [17, 18].

Many researchers have studied the leaching kinetics of calcined magnesite. They found that the layer of the product film formed on the mineral surface affects the reaction rate and the level of this effect differs for every acid [19]. Bayrak *et al.* [15] investigated the leaching kinetics of calcined magnesite in gluconic acid solutions and it was found that the dissolution rate was controlled by chemical reaction. Donmez *et al.* [18] investigated the leaching kinetics of calcined magnesite in acetic acid solutions and it was found that the dissolution rate was controlled by chemical reaction. Raschman and Fedorockova [7] researched the inhibiting effect of hydrochloric acid concentration on the dissolution rate of magnesium oxide during the leaching of dead-burned magnesite and they found that the dissolution of magnesium oxide is controlled by chemical reaction. Some of the examples of magnesite and

calcined magnesite studies are summarized in Table-1.

Although leaching kinetics of calcined magnesite was studied in a variety of acidic media, no study was found concerning the leaching kinetics of calcined magnesite in aqueous lactic acid solutions. Magnesium lactate, the magnesium salt form of lactic acid, is produced for food, pharmaceutical, refractory, textile, and cosmetic industries.

Therefore, the aim of this study is to investigate the leaching behavior of calcined magnesite by lactic acid solutions which have selective solubility. In this way, it is believed that the results obtained from experimental data may shade light upon magnesium lactate production. It is used as a drug against such a widespread illness known as common cold and can also be used as a dietary supplement, food additive and vitamin tablets.

Table-1: The Leaching Kinetics Of The Magnesite And Calcined Magnesite In Various Leach Reagents.

| Leach reagent (aq) | The leaching reactions of magnesite |  | E (kJ.mol <sup>-1</sup> ) | T (K) | R (µm) | t (dk) | Conc. (M) | Ref. |
|--------------------|-------------------------------------|--|---------------------------|-------|--------|--------|-----------|------|
| Gluconic acid      | $MgCO_3(s) + 2HC_6H_{11}O_7(aq)$    | $Mg(aq)^{2+} + 2C_6H_{11}O_7(aq)^{1-} + CO_2(g) + H_2O(l)$ | 61.7                      | 333   | 338    | 220    | 1         | [13] |
| Acetic acid        | $MgCO_3(s) + 2CH_3COOH(aq)$         | $Mg(aq)^{2+} + 2CH_3COO(aq)^{1-} + CO_2(g) + H_2O(l)$      | 78.4                      | 313   | 215    | 60     | 3         | [22] |
| Lactic acid        | $MgCO_3(s) + 2HC_3H_5O_3(aq)$       | $Mg(aq)^{2+} + 2C_3H_5O_3(aq)^{1-} + CO_2(g) + H_2O(l)$    | 50.3                      | 333   | 338    | 60     | 1.5       | [12] |
| Gluconic acid      | $MgO(s) + 2HC_6H_{11}O_7(aq)$       | $Mg(aq)^{2+} + 2C_6H_{11}O_7(aq)^{1-} + H_2O(l)$           | 32.9                      | 303   | 513    | 8      | 1         | [15] |
| Acetic acid        | $MgO(s) + 2CH_3COOH(aq)$            | $Mg(aq)^{2+} + 2CH_3COO(aq)^{1-} + H_2O(l)$                | 34.6                      | 303   | 1590   | 5      | 3         | [18] |
| Ammonium chlorur   | $2NH_4Cl(aq) + MgO(s)$              | $MgCl_2(aq) + 2NH_3(aq) + H_2O(l)$                         | 58                        | 323   | 90     | 16     | 1,3       | [7]  |

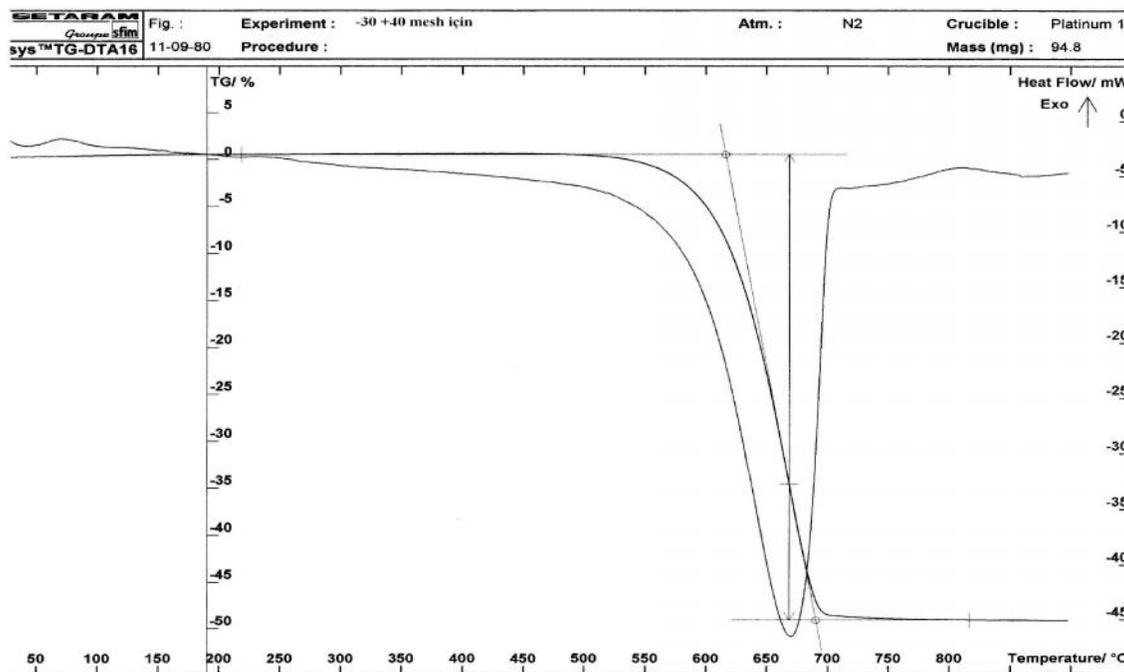


Fig. 1: TG/DTA curves for magnesite.

## Experimental

### Materials and Methods

The sample of magnesite ore that was used in experimental runs comes from the region of Refahiye, Erzincan, Turkey. After crushing and washing, the ore was gradually heated from 273 K to 973 K and maintained at 973 K for 2 hours using nitrogen atmosphere in furnace for calcination process as seen Fig. 1, and then sieved to different size fractions 1425 $\mu\text{m}$ , 725 $\mu\text{m}$ , 513  $\mu\text{m}$ , 338  $\mu\text{m}$ , 215 $\mu\text{m}$  using ASTM standard sieves. The chemical analysis was carried out by standard gravimetric and volumetric methods [20]. The analytical results are given in Table-2. X-ray diffraction patterns of the MgO were obtained by using a Philips PW) 3710 based diffractometer (XRD) with CuK $\alpha$  radiation (30 kV and 30 mA and automatic monochromator) at a scanning rate of 2 of 2 $^\circ$  min $^{-1}$  (Fig. 2).

Table-2: Chemical Analysis of the Calcined Magnesite.

| Component      | Wt %  |
|----------------|-------|
| MgO            | 97.80 |
| CaO            | 0.89  |
| Fe $_2$ O $_3$ | 0.51  |
| SiO $_2$       | 0.80  |

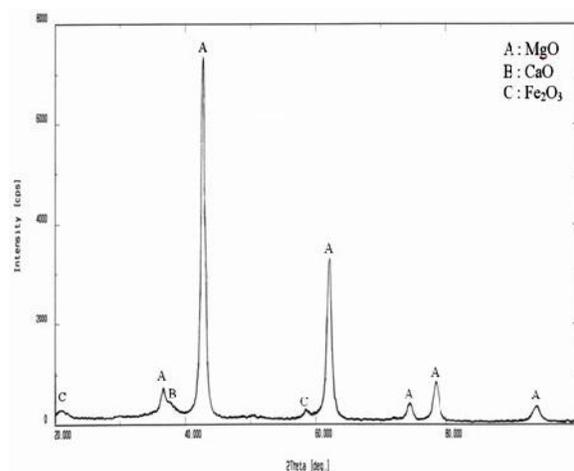


Fig. 2: X-Ray Diffractogram of the calcined magnesite.

Lactic acid, HC $_3$ H $_5$ O $_3$ - 2- hydroxy- d-propanoic acid, used for leaching experiments was reagent grade (90 wt % solution in water from Merck Chemicals).

The leaching process was carried out in a 500- mL cylinder glass batch reactor heated by a thermostat for keeping reactor contents at a constant temperature during the reaction, equipped with a

digital controlled mechanical stirrer for stirring, a thermometer for temperature control and a condenser to prevent losses by evaporation.

First, 250 mL volume of lactic acid solution at a definite concentration was poured into the reactor at the required temperature. When the required temperature of the reactor contents were reached, approximately 2.0 g of the calcined magnesite of required size was added into the reactor while the contents of the reactor were being stirred at a certain speed. From leaching solution, an amount of sample of the reaction mixture was taken out at pre-determined time intervals, filtered immediately and the amount of magnesium in the filtrate was analyzed complexometrically by EDTA at the medium of buffer solution (about pH 10) [21]. For each experimental condition, the experiment was repeated twice, and the arithmetic average of the result of the two experiments was used in the kinetic analysis. The leaching tests were performed as a function of several parameters, their range and values are given in Table-3.

Table-3: Parameters and their range used on the experiments.

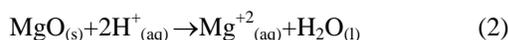
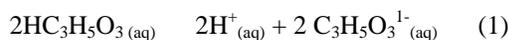
| Parameter                           | Value  |
|-------------------------------------|--|
| temperature ( $^\circ$ C)           | 13, 20, 25*, 30, 35                          |
| acid concentration (mol.L $^{-1}$ ) | 0.5, 1.0*, 2, 3.0, 4.0                       |
| particle size ( $\mu\text{m}$ )     | 1425, 725, 513*, 338, 215                    |
| stirring speed (rpm)                | 500, 600*, 700, 800                          |
| solid/liquid ratio (g.mL $^{-1}$ )  | 0.5/250, 1.0/250, 2.0/250*, 4.0/250, 6.0/250 |

\* The constant values used when the effect of other parameters was investigated.

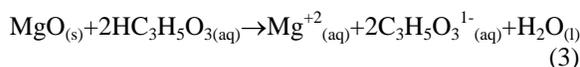
## Results and Discussion

### Leaching Reactions

Reactions occurring between calcined magnesite and lactic acid can be written as follows:



The overall reaction can be written as follows:



### The Effects of Parameters

#### Effect of Particle Size

In order to examine the effect of the particle size on the leaching rate, experiments were carried out using five different particle sizes of the sample ranging from 1425  $\mu\text{m}$ -215  $\mu\text{m}$ . The other parameters namely the temperature, stirring speed, solid/liquid

ratio and concentration of lactic acid were kept constant at 25°C, 600 rpm, 2/250 g mL<sup>-1</sup> and 1.0 M, respectively. The results are given in Fig. 3. As seen in Fig. 3, the leaching rate greatly increases with decrease in particle size of calcined magnesite used, which can be attributed to the increase of the contact surface, in which the main dissolution reaction occurs, with the decrease of the particle size per amount of the solid. In addition, values of the rate constants were plotted versus the reciprocal of the particle radii, yielding a linear relationship with a correlation coefficient of 0.998 (Fig. 4). The results showed that the particle size had an important effect on the leaching of calcined magnesite.

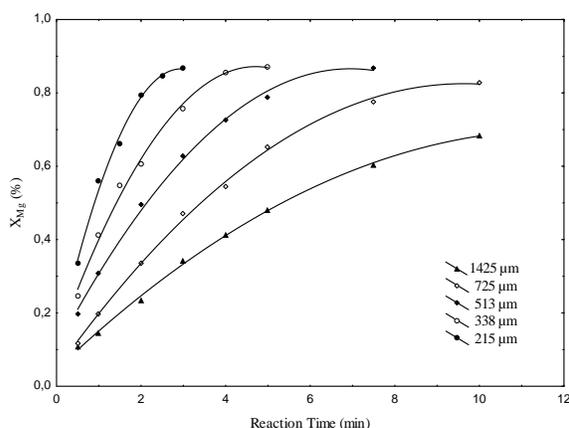


Fig. 3: Effect of particle size on the dissolution of calcined magnesite.

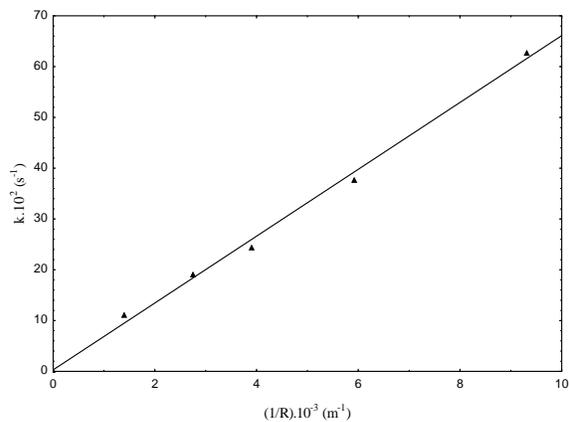


Fig. 4: Plot of  $k$  versus  $1/R$ .

#### Effect of Reaction Temperature

The leaching experiments of calcined magnesite were carried out in a temperature range of 13–35°C, in 1.0 M lactic acid concentration, at 600 rpm stirring speed at a constant particle size of 513

μm. Experimental results are shown in Fig. 5. From this Fig, it can be seen that increasing the reaction temperature strongly increases the leaching rate, as expected from the exponential dependence of the rate constant in the Arrhenius equation.

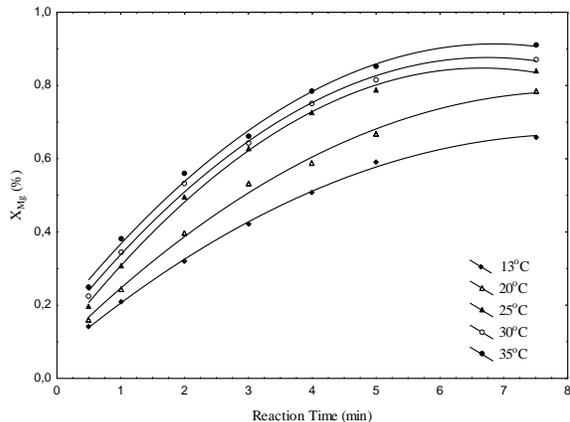


Fig. 5: Effect of temperature on the dissolution of calcined magnesite.

#### Effect of Stirring Speed

To observe the effect of stirring speed on the leaching rate, experiments were carried out by using four different stirring speeds in a range of 500–800 rpm. As seen from the experimental results given in Fig. 6, the leaching rate is nearly independent of the stirring speed. Also, in order to see this result better, the conversion data at various times versus stirring speed are shown as a graph in Fig. 6. The fact that the leaching rate was independent of the stirring speed indicates that the diffusion through the fluid film does not act as a rate-controlling step.

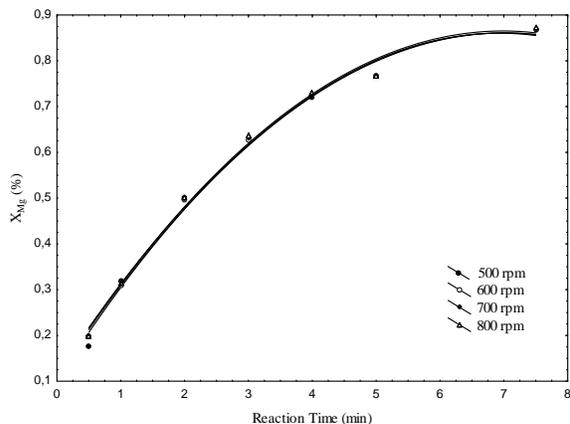


Fig. 6: Effect of stirring speed on the dissolution of calcined magnesite (500-800 rpm).

### Effects of Acid Concentration

In order to examine the effect of the lactic acid concentration on the leaching rate, some experiments were performed with six different lactic acid concentrations in the range of 0.5–4.0 M. The experiments showed that the leaching rate increases by increasing the acid concentration until 2.0 M of the lactic acid concentration and more increase in acid concentration caused a decreasing rate as can be seen in Fig. 7 (a) and 7 (b). It could be attributed to the fact that the intensity of negative effect of the water-the solvent- decreases after a certain value of acid concentration, and which was more dominant than that of positive effect of increase of acid concentration. Again, when the acid concentration exceeded a definite value, the number of hydrogen ions in the medium might decrease due to decrease of water amount more and more [22]. In addition, this behavior could be explained by the fact that as the acid concentration in the medium is increased, the appearance rate of the product increases and as the product reacted to the saturation value near the solid particle, it forms a sparingly soluble product film layer around the particle. Consequently, the leaching process slowed down after acid concentration of 2.0 M [23]. However, the leaching kinetics was basically investigated for the concentration range of 0.5–2.0 M. Additionally, the constant concentration value was selected as 1.0 M in the experiments.

### Effect of Solid–Liquid Ratio

The effect of the solid/liquid ratio on the leaching rate was studied in the range of 0.5/250–6/250 g mL<sup>-1</sup> as shown in Fig. 8. According to Fig. 8, the leaching rate decreased as the solid/liquid ratio increased. When solid/liquid ratio was increased, the amount of dissolved solid per unit liquid increase and hence the rate of leaching decrease.

### Kinetic Analysis

The fluid-solid reaction rate can be obtained using the heterogeneous and homogeneous reaction models. In the homogeneous reaction model, it is visualized that a reactant liquid enters the particle and reacts throughout the particle at all times. Thus the solid reactive behave as if it were dissolved. As a result the rate of the reaction can be given in the same form as for a homogeneous reaction. In the heterogeneous model, the reaction is considered to take place at the outer surface of the unreacted particle. With increase in conversion the unreacted core of the particle shrinks and the layer of the solid product thickens. According to this model, the

following three steps are considered to occur in succession during reaction [24].

1. Diffusion of the fluid reactant through the film surrounding the particle to the surface of the solid.
2. Diffusion of the fluid reactant through the ash to the surface of unreacted core.
3. Fluid-solid chemical reaction at the reaction surface.

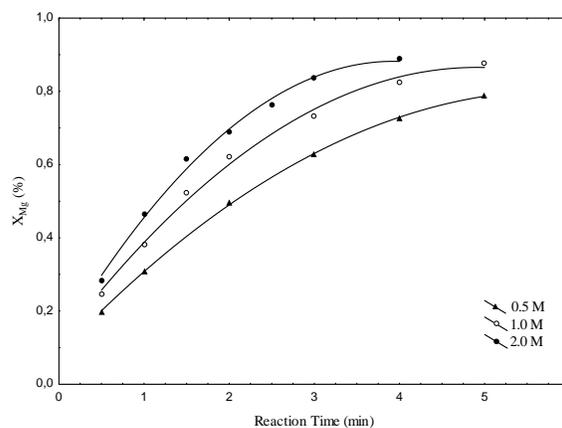


Fig. 7: (a). Effect of acid concentration on the dissolution of calcined magnesite (0.5–2.0 M).

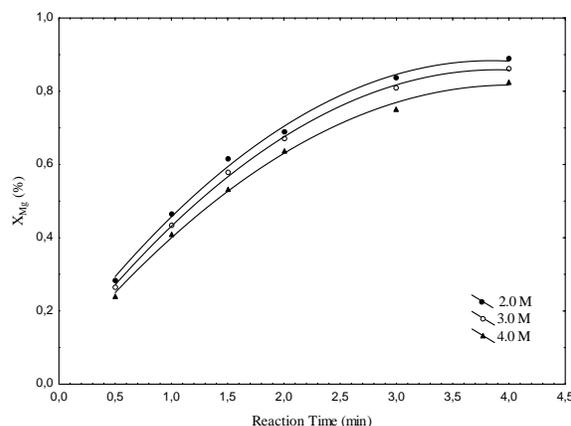


Fig. 7: (b). Effect of acid concentration on the dissolution of calcined magnesite (2.0–4.0 M).

According to the steps given above a heterogeneous reaction may be controlled by the fluid film diffusion (Step 1), the product layer diffusion (Step 2), or chemical reaction (Step 3). The fact that the leaching rate was independent of the stirring speed indicates that the diffusion through the fluid film does not act as a rate-controlling step. So, the experimental data have been applied to the step 2 of shrinking core models.

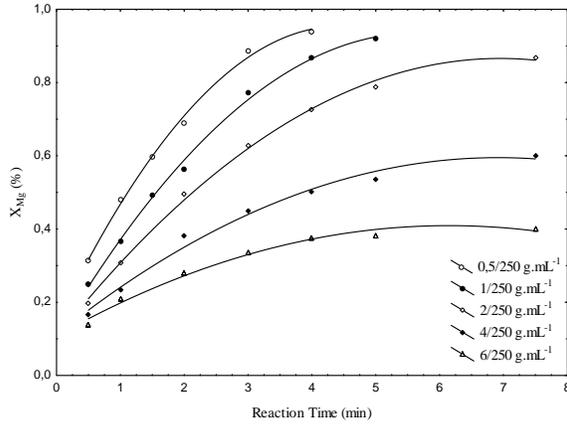


Fig. 8: Effect of solid/liquid ratio on the dissolution of calcined magnesite (0.5/250–6/250 g.mL<sup>-1</sup>).

Shrinking core model is given as in [24]. For the first order chemical reaction rate model,

$$-\frac{1}{S} \cdot \frac{dN_B}{dt} = -\frac{b}{4f \cdot r_c^2} \cdot \frac{dN_A}{dt} = b \cdot k_s \cdot C_{A_s} \quad (4)$$

$$dN_B = b \cdot dN_A = \dots_B \cdot dV = -4f \cdot \dots_B \cdot r_c^2 \cdot dr_c \quad (5)$$

$$\frac{-1}{4} \frac{1}{r_c^2} \frac{dr_c}{dt} = -\frac{1}{B} \frac{dr_c}{dt} = b k_s C_A \quad (6)$$

The variation of  $C_A$  is too big to neglect [25]. Hence,

$$-\int_R^{r_c} dr_c = b k_s \int_0^t C_A dt \quad (7)$$

$$C_A = C_{A0} \left( 1 - \frac{N_{B0}}{VbC_{A0}} X_B \right)$$

$$\text{and } \frac{r_c}{R} = (1 - X_B)^{1/3} \quad (8)$$

consequently,

$$dr_c = -\frac{R}{3} \cdot (1 - X_B)^{-2/3} \cdot dX_B \quad (9)$$

By combining equations (7)-(9);

$$\frac{(1 - X_B)^{-2/3} dX_B}{(1 - MX_B)} = \frac{3 \cdot b \cdot k_s \cdot C_{A0}}{B \cdot R} \cdot dt \quad (10)$$

where

$$M = \frac{N_{B0}}{VbC_{A0}} \quad (11)$$

$$\int_0^{X_B} \frac{dX_B}{(1 - X_B)^{2/3} \cdot (1 - MX_B)} = \frac{3 \cdot b \cdot k_s \cdot C_{A0}}{B \cdot R} \cdot \int_0^t dt \quad (12)$$

The integration of the left-hand side of Eq. (12) is analytically tested by using Matlab 2007B Package Program. The integrated form is Thus, Eq. (13) could be given as

$$\begin{aligned} & -1.9041 \cdot \ln((1 - X_B)^{1/3} + 1.157) + 0.952 \ln((1 - X_B)^{2/3} - 1.156(1 - X_B)^{1/3} + 1.338) \\ & - 3.298 \arctan(0.577(1.73(1 - X_B)^{1/3} - 1)) = \underbrace{\frac{3 \cdot b \cdot k_s \cdot C_{A0}}{\rho_B \cdot R}}_K \cdot t \end{aligned} \quad (13)$$

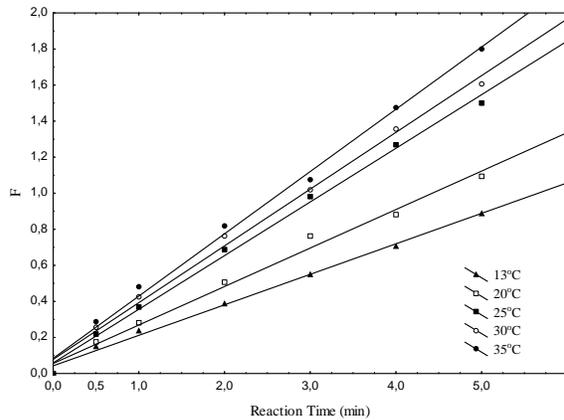


Fig. 9: F vs. reaction time for various reaction temperatures

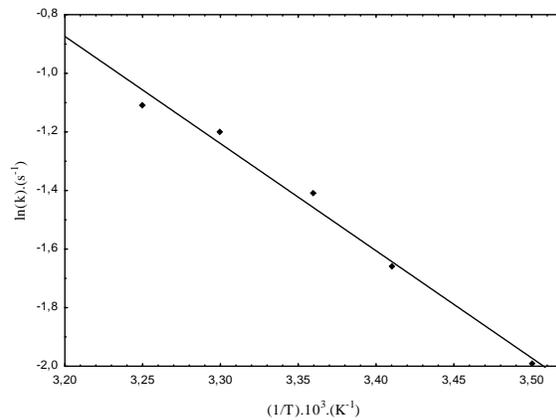


Fig. 10: Arrhenius plot

The fit of all the experimental data into the integral rate was tested by multiple regressions using Statistical Package Program, and the multiple regression coefficients obtained for integral rate expression were calculated. In the calculations, it was seen that the very high value of regression coefficient correcting the rate expression was for surface reaction control (step2). The coefficient value was calculated as 0.987. Additionally, the fact that the dissolution rate was independent of the stirring speed indicates that the diffusion through the fluid film does not act as a rate-controlling step. To confirm the results of these statistical analyses, the experimental data for each parameter were analyzed by graphical methods.

If the left hand of equation (13) was called as F, the plot of F vs. t was shown in Fig. 9 for the reaction temperatures (13-35°C). From the slopes of the straight lines the apparent rate constants were evaluated. From Arrhenius equation k term was known as:

$$k = k_0 e^{-E_A/R_g T} \quad (14)$$

As shown Fig. 10, the plot of  $\ln k$  vs.  $\ln 1/T$  was obtained for the each value of the temperature and the following values were calculated:

$$E_A = 30.61 \text{ kJ mol}^{-1} \quad k_0 = 5.47 \times 10^4 \text{ s}^{-1}$$

Such a value of activation energy indicates

$$\begin{aligned} & -1.9041 \cdot \ln((1 - X_B)^{1/3} + 1.157) + 0.952 \ln((1 - X_B)^{2/3} - 1.156(1 - X_B)^{1/3} + 1.338) \\ & - 3.298 \arctan(0.577(1.73(1 - X_B)^{1/3} - 1)) = 5.47 \cdot 10^4 \cdot e^{\frac{-30.61}{R_g T}} \cdot t \end{aligned} \quad (15)$$

## Conclusions

The dissolution kinetics of calcined magnesite in lactic acid solutions was studied at the various parameter levels. On the basis of the results of this study, the following conclusions can be expected:

- The rate of dissolution of magnesite in lactic acid was independent of stirring speed, which indicates that the reaction is not controlled by the diffusion in the liquid phase.
- The dissolution kinetics follows a shrinking core model with the surface chemical reaction as the rate-controlling step. The activation energy of the reaction was 30.62 kJ.mol<sup>-1</sup> and the reaction rate was very sensitive to the temperature in the range of 13-35 °C.

that the process is a chemically controlled reaction and agrees with the values obtained in the similar research of fluid– solid reaction systems [11-15,17-18].

To assess the agreement between the experimental conversion values and the model conversion values, the graph of  $X_{exp.}$  vs.  $X_{cal.}$  was plotted as shown in Fig. 11. It is observed that the agreement between the experimental and calculated values is very good.

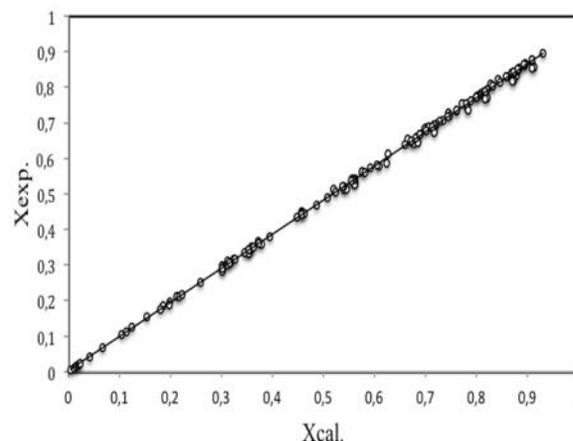


Fig. 11: Agreement between experimental and calculated conversion values.

- Increasing the lactic acid concentration increases magnesite dissolution in the range of 0.5-2.0 M and decreases it in the range of 2.0-4.0 M.
- With decrease in particle size of magnesite, the dissolution rate greatly increases.

## NOMENCLATURE

|          |   |
|----------|---|
| $C_{AS}$ | Molar concentration, mol.L <sup>-1</sup>                      |
| $C_{A0}$ | Initial molar concentration, mol.L <sup>-1</sup>              |
| $E_A$    | Activation energy, kJ.mol <sup>-1</sup>                       |
| $K$      | Reaction rate constant, s <sup>-1</sup>                       |
| $b$      | Stoichiometric coefficient, -                                 |
| $k_S$    | Rate constant for surface reaction, m.s <sup>-1</sup>         |
| $R$      | Average radius of solid particles, m                          |
| $r_C$    | Radius of unreacted core, m                                   |
| $t$      | Time, min   |
| $T$      | Temperature, K  |
| $V$      | Reaction volume, L <sup>-1</sup>                              |
| $P_B$    | Molar density of solid reactant, mol.m <sup>-3</sup>          |
| $X_B$    | Conversion of solid reactant                                  |
| $R_g$    | Universal gas constant, kJ.mol <sup>-1</sup> .K <sup>-1</sup> |
| $k_0$    | Frequency or pre-exponential factor, s <sup>-1</sup>          |

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