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Dissolution of periclase in excess of hydrochloric acid: Study of inhibiting effect of acid concentration on the dissolution rate

Pavel Raschman *, Alena Fedoročková

Faculty of Metallurgy, Technical University of Košice, Letná 9, Košice 042 00, Slovak Republic

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Abstract

Kinetics of the dissolution of particulate polycrystalline MgO in excess of hydrochloric acid were investigated, with special regard to the effect of activity of H^+ ions, temperature and particle size. The results are presented in terms of the shrinking particle model. The overall process is controlled by the chemical reaction of MgO with H^+ ions at the liquid–solid interface. The apparent activation energy was 58 kJ mol^{-1}. Acid concentration (from 0.5 M to 5.1 M) was surprisingly found to have a decelerating effect on the dissolution rate of MgO, resulting in negative values of reaction order for H^+ ions (from −0.16 to −0.18). We conclude that the regime studied here, characterised by decrease in the dissolution rate of MgO with an increase in H^+ activity, occurs under far from mass-transfer control conditions in the solutions with low Mg^{2+}:H^+ molar ratio.

Keywords: Magnesium oxide; Hydrochloric acid; Chemical dissolution; Rate; Mechanism

1. Introduction

Understanding the mechanisms of the chemical dissolution of oxides in acidic aqueous solutions is crucial for a wide variety of processes, including, for example, environmental and geochemical processes, hydrometallurgy, corrosion, catalysis and drug design. More reviews on this topic were published during last three decades [1–5], summarizing theoretical concepts and experimental evidence of how the physicochemical properties of both the oxide and the solution affect the rate and mechanism of the process.

Chemical dissolution of periclase (MgO), which, due to its simple structure, serves as a model (ionic) oxide, has been intensively studied. Generally, a very good correlation between experiment and theory was observed. The overall rate can be controlled by diffusion of liquid reactants/products or by the surface reaction, dependent on the reaction conditions [4–11]. However, in some cases, application of different theoretical approaches resulted in determining various potential rate-controlling steps for identical reaction conditions, with no clear basis for discrimination between these alternatives [4]. Furthermore, Jones et al. [8] found that there is an initial stage of the process of MgO dissolution in dilute strong acids, during which the rate increases rapidly despite a decrease in activity of H^+ ions. This observation was in conflict with the theories and, indeed, evidence in the literature [4]. A retardant effect of acid concentration on the dissolution rate of the magnesium was also observed during the leaching of burned magnesite using concentrated hydrochloric acid [12].

This study aims to investigate the rate and mechanism of MgO dissolution using concentrated hydrochloric acid, particularly to analyse the difference between the phenomena observed independently by Segall and co-workers [4,8], and Raschman and Fedoročková [12].

MgO is one of the few oxides that allow investigation of dissolution rate and mechanism at near-room temperature. This fact turned up to be crucial for application of various modern (and complementary) experimental techniques, from continuous kinetic measurements using a pH-meter [6] to atomic force microscopy [11,13,14]. On the other hand, this advantage (in addition to the difficulties resulting from non-ideality) has been probably the very reason why the kinetic experiments were carried out mostly at low temperatures using diluted acids (pH > 1). The aim of the present work therefore was also to extend the reaction conditions towards higher temperatures and acid concentrations—the situation is shown in Fig. 1. Single oxides are seldom extensively studied, which has been one of the major difficulties of reviewing past work and of attempt-

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* Corresponding author.
E-mail address: Pavel.Raschman@tuke.sk (P. Raschman).

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Nomenclature

$a_{\mathrm{H^+b}}$	activity of H^+ ions in bulk liquid
$a^0_{\mathrm{H^+b}}$	initial activity of H^+ ions in bulk liquid
$a_{\mathrm{H^+w}}$	activity of H^+ ions on the solid particle surface
$c_{\mathrm{H^+b}}$	concentration of H^+ ions in bulk liquid ($\mathrm{mol\,dm^{-3}}$)
$c^0_{\mathrm{H^+b}}$	initial concentration of H^+ ions in bulk liquid ($\mathrm{mol\,dm^{-3}}$)
$c_{\mathrm{H^+w}}$	concentration of H^+ ions on the solid particle surface ($\mathrm{mol\,dm^{-3}}$)
D	diffusivity of HCl ($\mathrm{m^2\,s^{-1}}$)
E	apparent activation energy ($\mathrm{J\,mol^{-1}}$)
k	pseudo-first-order reaction-rate constant defined by Eq. (5) ($\mathrm{m\,s^{-1}}$)
k_{S}	rate parameter in Eq. (6) ($\mathrm{mol\,m^{-2}\,s^{-1}}$)
k_0	frequency factor in Eq. (7) ($\mathrm{mol\,m^{-2}\,s^{-1}}$)
k_{LS}	external mass transfer coefficient ($\mathrm{m\,s^{-1}}$)
K	parameter defined by Eq. (4) ($\mathrm{m\,s^{-1}}$)
L	radius of the solid particle (m)
M_{S}	mean mole mass of solid ($\mathrm{kg\,mol^{-1}}$)
n	reaction order for H^+ ions
N	number of leaching runs
r	rate of reaction (1) ($\mathrm{mol\,m^{-2}\,s^{-1}}$)
r_{c}	coefficient of correlation
R	original radius of the solid particle (m)
$\boldsymbol{R}$	gas constant = 8.314 $\mathrm{J.mol^{-1}\,K^{-1}}$
Re	Reynolds number
Sc	Schmidt number
Sh	Sherwood number
t	time of leaching (s)
t_{R}	time necessary to attain a complete dissolution of the solid (s)
T	temperature (K)
X	fraction of solid dissolved

Greek symbols

ϑ	temperature (°C)
ρ_{S}	bulk density of the solid ($\mathrm{kg\,m^{-3}}$)
τ	dimensionless time of leaching

ing to derive a general concept of the mechanism of metals oxide chemical dissolution [1], and we expect that these kinetic data will be of use for engineering applications, as well. The process parameters investigated include also particle size.

2. Theoretical

Chemical dissolution of MgO in hydrochloric acid is a liquid–solid reaction in which no solid product is formed. Due to sintering, porosity of polycrystalline periclase is less than 5%, which makes the model of non-porous shrinking particle applicable to describe the dissolution of the particulate solid. The overall process may be controlled by intrinsic chemical reaction or by external mass transfer [15,16]; it involves the following steps in series:

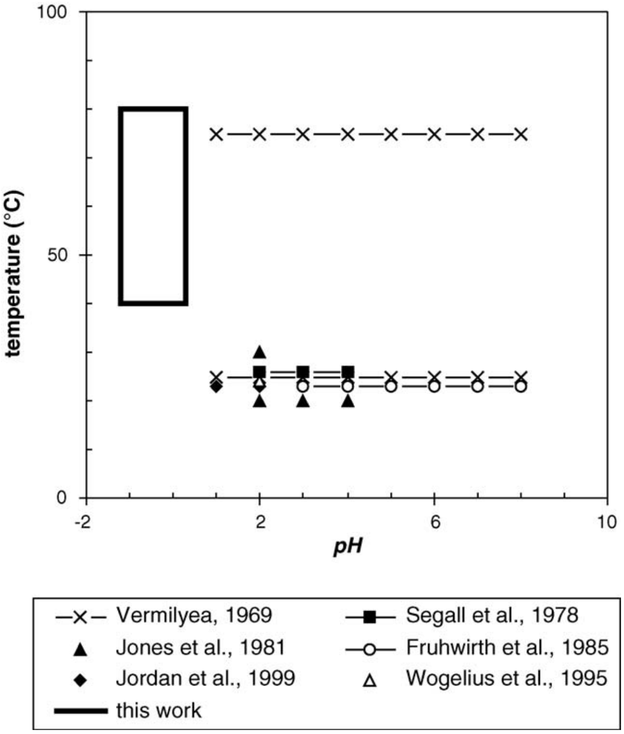

Fig. 1. Reaction conditions under which the chemical dissolution of MgO was studied in the present work, compared with those referred to in the literature.

1. diffusion of H^+ ions through the liquid film at the liquid–solid interface to the surface of the solid particles;
2. surface chemical reaction according to Eq. (1):

$$\mathrm{MgO_{(s)}} + 2\mathrm{H^+_{(aq)}} \rightarrow \mathrm{Mg^{2+}_{(aq)}} + \mathrm{H_2O_{(l)}}; \tag{1}$$

3. diffusion of liquid products of the reactions (1) from the interface through the film to the bulk liquid.

The surface chemical reaction (1) involves the transfer of magnesium cations and the oxygen anions from the solid to the solution, in which the cations will be hydrated. The transfer of the anions will involve protonation or hydroxylation reactions at the surface of the solid, to form water. No oxidation or reduction (electron or hole transfer) is required [4].

In the first approximation, the periclase particles were considered to be spheres with original radius R; the particles shrink in time as the solid is consumed by reaction with H^+ ions according to Eq. (1). The shrinking-particle model in the form:

$$X = 1 - \left(1 - \frac{t}{t_{\mathrm{R}}}\right)^3 \tag{2}$$

was used to analyse the process. X is the fraction of solid dissolved and t_{R} the time necessary to attain a complete dissolution, defined as:

$$t_{\mathrm{R}} = \frac{2\rho_{\mathrm{S}} R}{M_{\mathrm{S}} K c^0_{\mathrm{H^+b}}} \tag{3}$$

where ρ_S is the bulk density and M_S the mole mass of the periclase; $c^0_{H^+b}$ the initial concentration of H^+ ions in solution; K the rate parameter defined by Eq. (4):

$$K = \frac{kk_{LS}}{k + k_{LS}}, \tag{4}$$

where k_{LS} is the mass transfer coefficient for the transfer of H^+ ions through the liquid film at the surface of solid particles; k the apparent pseudo-first-order reaction-rate constant:

$$k = \frac{r}{c_{H^+w}}. \tag{5}$$

The rate r of reaction (1) is expressed as a power-law function of the activity of H^+ ions on the particle surface, a_{H^+w}:

$$r = k_S a^n_{H^+w}. \tag{6}$$

The coefficient k_S is the apparent reaction-rate constant and n the apparent order of the overall surface chemical reaction (1). Temperature dependence of k_S can be expressed using the Arrhenius expression:

$$k_S = k_0 \exp\left(\frac{-E}{\boldsymbol{R}T}\right), \tag{7}$$

where k_0 the frequency factor; E the apparent energy of activation; $\boldsymbol{R}$ the gas constant.

3. Experimental

3.1. Materials

High-grade synthetic polycrystalline periclase HAMAG 99.5 produced from Slovak magnesites [17] in Hačava, Slovakia was used in the present study. Sized fractions were obtained by dry-screening. To eliminate the effect of very small particles adjacent to the surface of larger ones, the fractions were washed on the screens by acetone, dried and dry-screened again. The physico-chemical characteristics of the samples used for the leaching experiments (and named HAM/mean particle diameter) are given in Table 1. The samples were analysed by convenient analytical methods, the specific surface area was determined by the BET nitrogen adsorption technique. Presence of the periclase as the predominant mineral phase has been verified using X-ray diffraction method. Analytical reagent grade chemicals and distilled water were used in all experiments.

3.2. Experimental procedure and measuring kinetic data

Dissolution of the samples of periclase was tested in a 1.1 L isothermal well-mixed glass batch reactor with a six-blade turbine impeller, four baffles installed, and a thermometer (for more details see ref. [18]). The temperature was maintained to within 0.2 K by a heating glass coil connected to a thermostat.

When the HCl solution in the reactor had reached the required temperature, a small amount (0.5–1.0 g) of periclase was added. The 15–20 mL samples of the solution were withdrawn from the reaction mixture in the reactor through a sintered glass filter at appropriate time intervals. The solutions were analysed by EDTA method.

Table 1
Physico-chemical characteristics of samples of periclase

	Sample		
	HAM/67	HAM/113	HAM/335
(1) Particle diameter (μm)	−71 + 63	−125 + 100	−355 + 315
(2) Mean particle diameter (μm)	67	113	335
(3) Specific surface area ($m^2\ g^{-1}$)	0.11	0.09	0.02
(4) Chemical composition (wt.%)			
MgO		99.5	
CaO		0.15	
Fe_2O_3		0.12	
Al_2O_3		0.02	
SiO_2		0.04	
L.O.I.		<0.1	
(5) Rational analysis (wt.%)			
Periclase		99.6	
Magnesiumferrite		0.06	
Dicalciumsilicate		0.12	
Brownmillerite		0.10	
Dicalciumferrite		0.08	

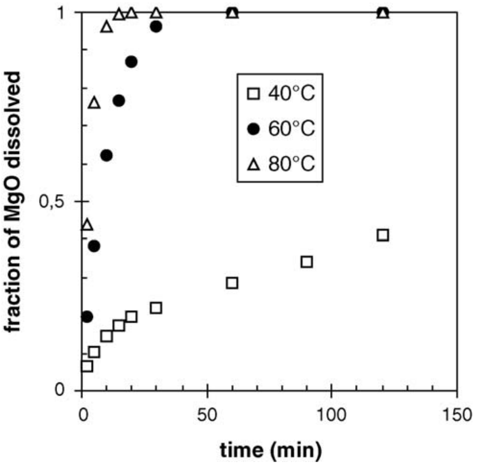

Fig. 2. Effect of temperature on fraction of MgO dissolved (reaction conditions: sample HAM/113; $c^0_{HCl} = 1.0\,M$).

The experiments were carried out under reaction conditions, which were: temperature from 40 °C to 80 °C and concentration of HCl from 0.5 M to 5.1 M.

4. Results

4.1. Effect of temperature

The rate of MgO dissolution is very sensitive to the temperature. A typical situation is shown in Fig. 2—the sample HAM/113 was dissolved in 1.0 M HCl over the temperature range of 40–80 °C.

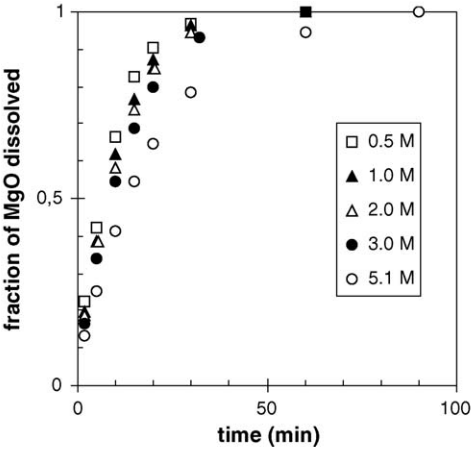

Fig. 3. Effect of hydrochloric acid concentration on fraction of MgO dissolved (reaction conditions: sample HAM/113; $\vartheta = 60\,^{\circ}\mathrm{C}$).

4.2. *Effect of acid concentration*

The experiments showed that the rate of transfer of magnesium to the solution decreased with increase in HCl concentration—an example is shown in Fig. 3.

4.3. *Effect of particle size*

From Fig. 4 it is seen that the rate of chemical dissolution of the magnesium increases with decrease in particle size of the periclase.

4.4. *Effect of lixiviant composition*

In selected experiments, two to three consequent runs were carried out under identical reaction conditions, using the product solution from each run as a lixiviant in the following one. It was observed that the rate of dissolution of the magnesium was not affected by the products of reaction (1) present in the solution under the conditions of the experiments in the present work.

5. Discussion

5.1. *Experimental method*

Dissolution of the periclase was tested under reaction conditions which were characterised by a relatively high excess of the acid, to eliminate possible effects of the changes in lixiviant composition during individual runs on the dissolution rate. In this study, HCl:MgO molar ratio ≥ 40 was used and the concentration of hydrochloric acid in the bulk aqueous phase was kept constant within 0.1–5% relatively during each run.

The value of heat-transfer coefficient for the transfer of heat through the liquid film at the surface of solid particles being $\approx 2 \times 10^4\ \mathrm{J\,m^{-2}\,s^{-1}\,K^{-1}}$ [19] indicates that the increase in surface temperature due to the heat generated by chemical reactions was ≈ 0.01 K. Hence, our conclusion is that the dissolution was practically isothermal, i.e. surface chemical reaction (1) took place at the temperature of bulk liquid.

5.2. *Mechanism of leaching*

Applicability of the non-porous shrinking particle model (NSPM) to the reaction system studied was checked using graphical analysis. The scatter diagram shown in Fig. 5 reveals that there is a very good correlation between model and experiment. We therefore conclude that the NSPM model can be used to analyse chemical dissolution of MgO under the conditions considered in the present work. The time necessary to attain a complete dissolution t_R was obtained for each run by quasi-linearised regression of X–t data using Eq. (2).

When the values of t_R were plotted against particle diameter $2R$, lines were obtained for various reaction conditions—the situation is illustrated by Figs. 6 and 7. These results are in a very good correlation with Eq. (3) and confirm applicability of NSPM model.

The decrease in the rate of transfer of the magnesium to the solution observed when increasing HCl concentration (see Fig. 3) indicates that the chemical reaction (1) might be the rate-

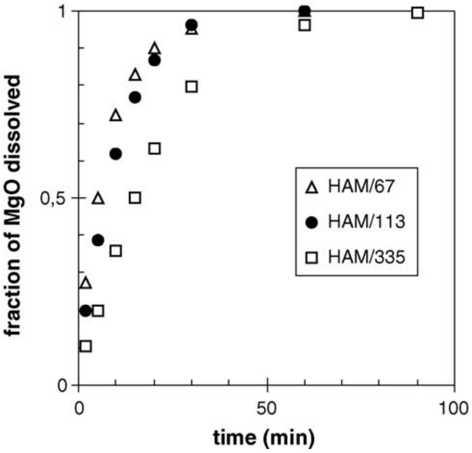

Fig. 4. Effect of particle size on fraction of MgO dissolved (reaction conditions: samples HAM/67, HAM/113 and HAM/335; $\vartheta = 60\,^{\circ}\mathrm{C}$; $c^{0}_{\mathrm{HCl}} = 1.0\,\mathrm{M}$).

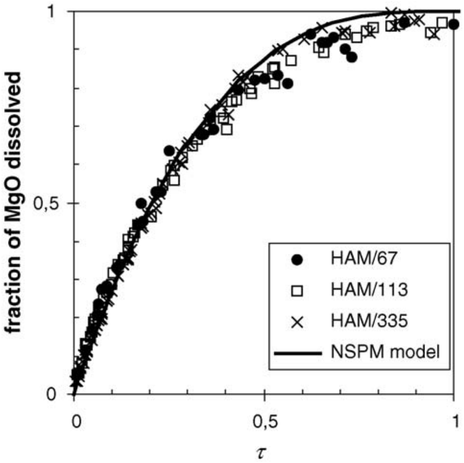

Fig. 5. Conversion vs. reduced time dependence for dissolution of the samples: points: measured data, full line: Eq. (2); $\tau = t/t_R$.

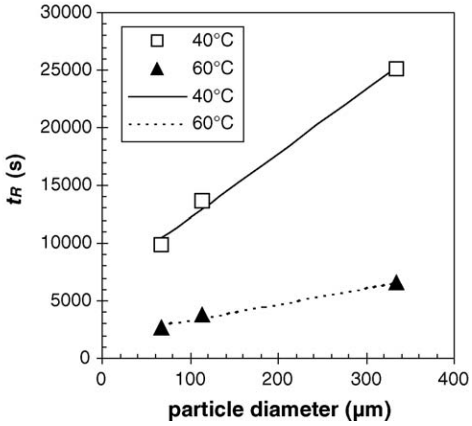

Fig. 6. Effect of particle diameter on t_R at constant concentration of H^+ ions (reaction conditions: $c^0_{HCl} = 5.1$ M; points: measured data, full line: regression line).

controlling step. To estimate the degree of interaction between chemical reactions and mass transfer, external mass transfer coefficient k_{LS} can be calculated using an appropriate expression available in the literature—for example [20]:

$$k_{LS} = \frac{Sh\,D}{L} \tag{8}$$

$$Sh = 1 + 0.38 Re^{1/2} Sc^{1/3} \tag{9}$$

where D is the diffusivity of HCl ($m^2\,s^{-1}$), L the radius of the solid particle (m), and Sh, Re and Sc are the Sherwood, Reynolds and Schmidt numbers, respectively. Conservative estimates of Sh calculated using Eq. (9) are from a range 3 to 4 which implies k_{LS} values $6 \times 10^{-4}\,m\,s^{-1}$ to $7 \times 10^{-4}\,m\,s^{-1}$. If the surface chemical reaction (1) is the rate-controlling step then the condition given by Eq. (10):

$$k \ll k_{LS} \tag{10}$$

holds, and one has:

$$k \approx \frac{2\rho_S R}{M^0_S t_R c^0_{H^+}}. \tag{11}$$

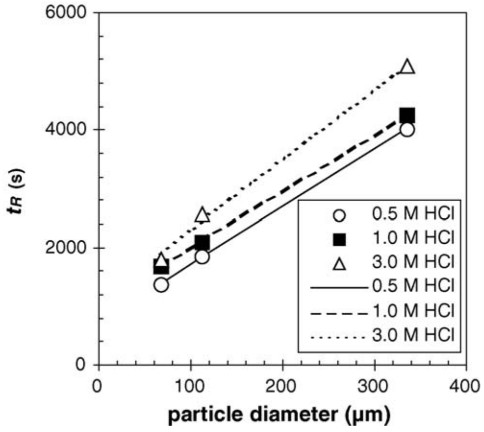

Fig. 7. Effect of particle diameter on t_R at constant temperature (reaction conditions: $\vartheta = 60\,°C$; points: measured data, full line: regression line).

Table 2
Parameters of shrinking particle model (Eqs. (2)–(7)) fitted by experiments

	Sample		
	HAM/67	HAM/113	HAM/335
E ($kJ\,mol^{-1}$)	58	58	58
n (−)	−0.16	−0.18	−0.16
r^2_c (−)	0.95	1.00	1.00
N (−)	6	10	10

Since the reaction-rate constant k, calculated for individual runs using Eq. (11), is by up to three orders lower than the external mass transfer coefficient k_{LS}, we conclude that chemical dissolution of MgO was really controlled by the surface chemical reaction (1).

The values of model parameters were obtained by a multiple regression using Eq. (12):

$$\ln t_R = \text{const} - n \ln a^0_{H^+b} + \left(\frac{E}{\boldsymbol{R}}\right) T^{-1} \tag{12}$$

and are summarized in Table 2. Typical examples of Arrhenius and $\ln t_R$ versus $\ln a_{H^+}$ plots are shown for illustration in Figs. 8 and 9, respectively.

In general, fractional reaction order for H^+ ions in the empirical rate law in the form of Eq. (6) is compatible with, or even indicative of process controlled by surface reaction (e.g. [2–8,21]—mostly values in the range $0 < n < 0.6$ have been reported for the dissolution of oxides in acid solutions). The negative value of n obtained in the present study for all the samples is in conflict with the theories and, indeed, most evidence in the literature—we found just one reference in the literature (system Mn_2O_3–H_2SO_4, ref. [157] in [2]). However, in their work on MgO dissolution in dilute strong acids, Jones et al. [8] found that there are two distinct regimes in the dissolution process—an initial stage of dissolution of MgO, during which the dissolution rate increases rapidly despite a decrease in hydrogen ion concentration, and an advanced stage during which the rate decreases with increasing pH (this was particularly evident

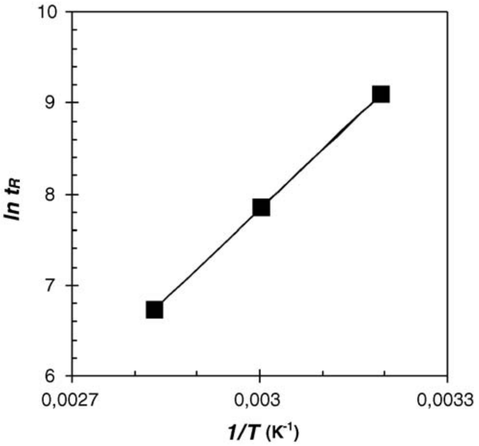

Fig. 8. An example of Arrhenius plot (reaction conditions: sample HAM/113; $c^0_{HCl} = 3.0$ M; points: measured data, full line: Eq. (12)).

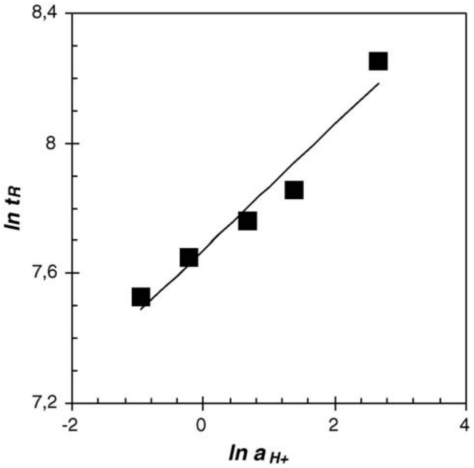

Fig. 9. An example of $\ln t_R$ vs. $\ln a_{H^+b}$ plot (reaction conditions: sample HAM/113; $\vartheta = 60\,°C$; points: measured data, full line: Eq. (12)).

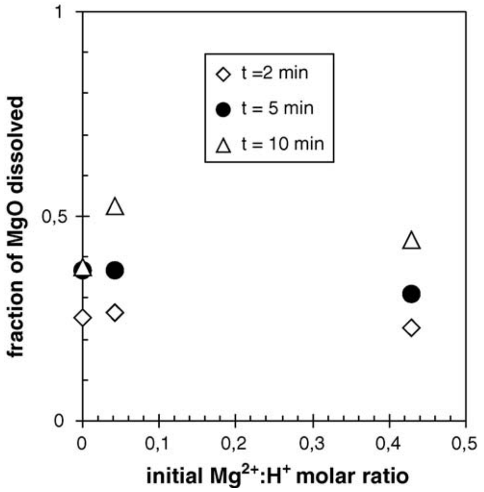

Fig. 10. Effect of Mg^{2+} to H^+ molar ratio on fraction of MgO dissolved (reaction conditions: sample HAM/113; $\vartheta = 25\,°C$; $c^0_{HCl} = 0.5$ M).

for lower pH values, i.e. pH between 2 and 3). Though the initial kinetic regime is still not understood [4], several features have been reported [8]:

(a) The dependence of initial dissolution rates on pH is either zero or very slight (kinetic order on H^+ ion concentration was found to be 0.05 ± 0.05, compared to values between 0.5 and 1.0 expected from the theory); proton supply to the surface is not limiting.
(b) Likewise, the anion concentration is not important.
(c) The dissolution rate is sensitive to the concentration of dissolved cation; values of Mg^{2+} ion concentration greater than a small percentage of the initial hydrogen ion concentration cause a considerable increase in rate.
(d) The early dissolution rates are not particularly sensitive to the nature of the anion or to the particular cation.

The values of the reaction order n summarised in Table 2 indicate that the dissolution rate of MgO was relatively insensitive to hydrogen ion concentration. Within an experimental error, these values are very close to the value reported by Jones et al. [8]. On the other hand, the stage of dissolution investigated under the conditions of the experiments in the present work was "initial" in respect to the composition of the liquid phase, while it was "advanced" in respect to the solid. Though the process was insensitive to Cl^- ion concentration, no accelerating effect of magnesium ion concentration on the rate of dissolution of MgO has been observed for Mg^{2+}:H^+ molar ratio up to 0.5—the situation is illustrated by an example shown in Fig. 10. The common feature of the "initial stage of dissolution" and the behaviour studied in the present work is that both occur under far from mass-transfer control conditions, in large excess of the acid. However, there are major differences in the nature of periclase and concentration of hydrochloric acid used by Jones et al. (nearly perfect MgO "smoke" crystals and $pH > 2$ [8]) and applied in the present study (sintered polycrystalline periclase and $pH < 0.5$). While the initial kinetic regime is probably controlled by solid-state factors [4], the advanced kinetic regime investigated in the present work was found to be insensitive to the presence of impurities [12] and is controlled by the composition of the liquid phase.

6. Conclusions

Kinetics of the dissolution of sintered polycrystalline periclase in excess of hydrochloric acid were investigated. The results are presented in terms of the shrinking particle model. Several facts indicate that the dissolution is controlled by the chemical reaction of MgO with H^+ ions at the liquid–solid interface:

1. The reaction-rate constant k, calculated for individual runs using Eq. (11), is by up to three orders lower than the estimates of external mass transfer coefficient k_{LS}.
2. The value of apparent activation energy is relatively high, $E = 58\,kJ\,mol^{-1}$.
3. Fractional reaction order for H^+ ions n in the empirical rate law (Eq. (6)) was obtained.

The negative value of n obtained in the present study is in conflict with the theories and most evidence in the literature. Though there are certain common features of the "initial stage of dissolution" referred to in the literature and the behaviour studied in the present work (both were observed under far from mass-transfer control conditions, in large excess of the acid, and are insensitive to the Cl^- ion concentration), major differences were observed. Not only periclase samples of different nature and different concentrations of hydrochloric acid were used, but also the stage of dissolution investigated in the present work was rather "advanced", in respect to the reacting solid. Furthermore, while the "initial stage of dissolution" is probably controlled by solid-state factors, the advanced kinetic regime investigated here is controlled by the composition of the liquid phase. We therefore conclude that a new kinetic regime of MgO dissolution has been presented in this paper. The rate-controlling mechanism could not be predicted using existing theories. In our opinion, the process is controlled by the removal Mg^{2+} ions hindered by the H^+ ions adsorbed at the surface of the solid.

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