

# The Conversion Kinetics of Tincal to Boric Acid in Nitric Acid Solutions

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**ABSTRACT:** Pure borax and various boron compounds are produced from the solutions in which tincal is dissolved with various reagents. Economically important boron compounds such as boric acid, borax, boric oxide, and refined hydrated sodium borates and perborates are produced from boron-containing ores. The production of boric acid by using nitric acid from tincal is more advantageous because it evaluates by-product  $\text{NaNO}_3$  as fertilizer production. In this study, the conversion kinetics of tincal to boric acid in nitric acid solutions were investigated by such parameters as particle size, 300-1500  $\mu\text{m}$ ; acid concentration, 0.5-4 mol/L; solid-to-liquid ratio 0.04-0.10 g/mL; stirring speed 20.93-62.8  $\text{s}^{-1}$ , and reaction temperature, 30-60 °C. The conversion was found to increase with decreasing particle size and solid-to-liquid ratio and while it increased with increasing reaction temperature, acid concentration, and stirring speed. The conversion kinetics was examined using the heterogeneous and pseudo-homogeneous reaction models. The experimental results were found to be in better agreement with the correlation given in the following equation and the activation energy being 37.5 kJ/mol.

$$\ln\left(\frac{1-X_{\text{HNO}_3}}{1-X_{\text{tincal}}}\right) = (C_{0,\text{HNO}_3}, 2C_{0,\text{tincal}})kt$$

**KEYWORDS:** Borax; Conversion kinetics; Nitric acid; boric acid.

## INTRODUCTION

Turkey holds approximately 70% of the world's boron reserves. Boron is mostly found in nature as borates

of calcium and/or sodium. Tincal is the natural mineral of borax decahydrate with the chemical formula  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$

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and prismatic crystals structure. Tincal, one of the most widespread boron minerals, is used in the production of borax hydrates and boric acid ( $\text{H}_3\text{BO}_3$ ) [1].

Economically important boron compounds such as boric acid, borax, boric oxide, and refined hydrated sodium borates and perborates are produced from boron-containing ores [1-3].

Boron compounds find many applications in various fields: i.e., The preparation of disinfectants and drugs in medicine, production of optic and chemically stable glasses, hardness modulator in enamel, metal oxidation inhibitor in soldering, electrolytes additives in nickel plating. In addition, they can be used in nuclear engineering, as fuel for rocket motors, in the preparation of hard and refractory alloys, high quality steels, boron-containing gasolines, and heat resistant polymers, and also as catalysts [4].

Various studies have been conducted on the reaction of boron ores with different chemicals to obtain boron compounds. The dissolution of boron minerals has been carried out using nitric acid [5], hydrochloric acid [6], and sulfuric acid [7]. These earlier investigations show that product film layer formed on the mineral surface affects the reaction rate, and the level of this effect being different for each acid. Karazhanov [8] studied the kinetics of the dissolution of borates and other natural salts, and it was found that the dissolution mechanism of salts was highly depended on the experimental conditions in general, and temperature and the type of solvent used in particular. Perchloric acid [9], phosphoric acid [10], and acetic acid [11] were also use in the dissolution of borates and the rate of dissolution was comparable to that obtained by hydrochloric acid, nitric acid, and sulfuric acid solutions. Yeşilyurt studied the optimum conditions for boric acid extraction from colemanite in nitric acid media [12], and obtained an extraction yield of 99.7 %. The dissolution processes of ulexite and colemanite minerals in  $\text{CO}_2$ -saturated water have been investigated [13, 14], and the optimum dissolution conditions for ulexite in  $\text{CO}_2$ -saturated water have been identified [15]. Künkül et al. [16] investigated the dissolution kinetics of ulexite in  $\text{CO}_2$ -saturated ammonia solution. They observed that the dissolution rate increased with increasing ammonia concentration, reaction temperature, and calcination temperature, and they also found that the process could be described by a first-order pseudo-

homogeneous reaction model. Kocakerim and Alkan [17] studied the kinetics of dissolution colemanite in water saturated with  $\text{SO}_2$  and found that the dissolution rate was chemically controlled. Özmetin et al. [18] investigated the dissolution kinetics of colemanite in aqueous acetic acid solution and found an increase in the dissolution rate with increasing temperature and decreasing particle size, with no significant effects of stirring speed. They found that the conversion rate formed a maximum at the acid concentration of 3.365 M, decreasing if the acid concentration is further elevated. Tunç et al. [19] investigated the dissolution kinetics of ulexite in sulfuric acid solutions and determined that the dissolution rate increased with increasing reaction temperature and decreasing particle size, mass concentration and acid concentration, with again no important effect from stirring speed. The dissolution process was reported to be controlled by the diffusion of  $\text{H}_3\text{O}^+$  ions through the product and by-product layer to the mineral surface. In addition to these, there are many studies investigating the dissolution of boron minerals in different aqueous media containing various acids [20-24]. Tunç et al. [25] investigated the conversion kinetics of tincal in acetic acid solutions. They found that conversion rate increased with decreasing particle size and solid-to-liquid ratio and increasing reaction temperature, acid concentration and stirring speed. It has been determined that conversion process is controlled by diffusion through fluid film. Furthermore, Tunç et al. [26] studied the dissolution of colemanite in ammonium sulfate solutions. They found that the conversion rate was increased by decreasing particle size and solid to liquid ratio and by increasing ammonium sulfate concentration and temperature.

Today, one of the processes applied most in boric acid production is the reaction of colemanite with sulfuric acid. Gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) obtained as a by-product in this process, called borogypsum, is separated by filtration. It is a waste which both makes reaction and filtration difficult and causes boron loss. Recently, some researchers worked to produce boric acid from borax. In studies by Mergen et al. [27-29], boric acid production was made from borax solutions with concentrations of 183-245 g/L  $\text{B}_2\text{O}_3$  using  $\text{H}_2\text{SO}_4$ . Sodium sulfate was recovered as a by-product during boric acid fabrication. In this study, boric acid production yield was around 91%, refined boric acid and sodium sulfate crystals

having purities of over 99% were characterized by using X-Ray Diffraction (XRD), Differential Thermal and Thermo Gravimetric Analysis (DTA-TG) and Scanning Electron Microscopy (SEM).

*Nezhad* [30] investigated the reaction of borax crystals with propionic acid to produce boric acid in batch mode and found that the product boric acid precipitated on the solid borax reactant. An increase in the coefficient of variation of feed crystals resulted in an increase in the completion time of the reaction. A sharp interface model with variable bulk fluid concentration of liquid reactant was developed for the process simulation.

In another study, *Nezhad et al.* [31] produced boric acid from borax using oxalic acid. Reaction of borax in solution with oxalic acid crystals exhibited a different behavior, i.e., reactive precipitation process. Crystallization of boric acid took place in the solution phase after complete dissolution of oxalic acid crystals. With a series of experiments by the method of s plane analysis, nucleation and growth rates were evaluated. Relative nucleation kinetics was correlated in terms of the significant process variables.

In another process, tincal is reacted with sulfuric acid and the by-product is sodium sulfate which can be used as a raw but very cheap material. For this reason, a study on the conversion conditions of tincal in nitric acid solutions will be useful as an alternative from the point of view of elimination of the problems encountered in  $H_3BO_3$  production from colemanite or tincal with sulfuric acid.

When tincal is dissolved in nitric acid solutions the products are boric acid and sodium nitrate. Sodium nitrate, being a by-product, is a valuable compound which is used in glass manufacturing, pyrotechnics, medicine, matches, dynamites, manufacturing sodium salts and nitrates, dyes, etc. So, the production of boric acid by this process is more advantageous than by colemanite-sulfuric acid process or tincal-sulfuric acid process.

The more economical industrial means would be established by adopting more productive and adequate production strategies as when tincal is utilized to produce boric acid in Turkey, a country which possesses the greatest tincal reserves worldwide. In the literature, there is no work on the conversion kinetics of tincal in nitric acid solutions. Therefore, this study focused on

the kinetics of tincal-to-boric acid conversion and the determination of the products formed.

## EXPERIMENTAL SECTION

Tincal ore used in the study was obtained from a region of Kırka, Eskişehir, Turkey. The mineral was manually cleaned of visible impurities, crushed, ground, and sieved by ASTM standard sieves to obtain the nominal particle size fractions of -1500+850, -850+600, -600+425, and -425+300  $\mu\text{m}$ . The ore sample was chemically analyzed and found to contain 34.17%  $B_2O_3$ , 15.13%  $Na_2O$ , 44.95%  $H_2O$ , and 6.75% others.  $B_2O_3$  content was determined by a volumetric method, sodium (Na) by a flame photometer (Sherwood 410). The crystal phase properties and mineralogical structures of the minerals used in the experimental study were determined by X-ray diffraction technique. The analysis was carried out at room temperature on a Philips Panalytical X'Pert Pro brand X-Ray diffractometer, where X-rays were produced in a  $CuK\alpha$  tube of 45 kV and 40 mA. X-ray diffractogram of the sample is given in Fig. 1.

The conversion process was carried out in a 250 mL-spherical glass vessel under atmospheric pressure. The reactor contents were stirred with a mechanical stirrer, with a thermostat to maintain reaction medium at a given temperature. To prevent the loss of the solution by evaporation, a cooling system was attached to the reactor. The parameters investigated and their ranges are given in Table 1.

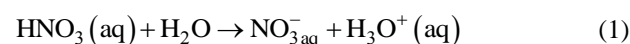
In experiments, first 100 mL of  $HNO_3$  solution was put into the reactor and heated to a desired temperature, and then a known amount of the ore sample was added while the solution was stirred. At the end of a given reaction period, the stirring was terminated, and the content was filtered. The  $B_2O_3$  content of the filtrate was determined by a potentiometric method reported by Nemodruk and Karalova [4]. The fraction of the mineral passing into the solution was calculated as follows:

$$X_{B_2O_3} = \frac{\text{conversion amount of } B_2O_3}{\text{total amount of } B_2O_3 \text{ in sample}}$$

## RESULTS AND DISCUSSION

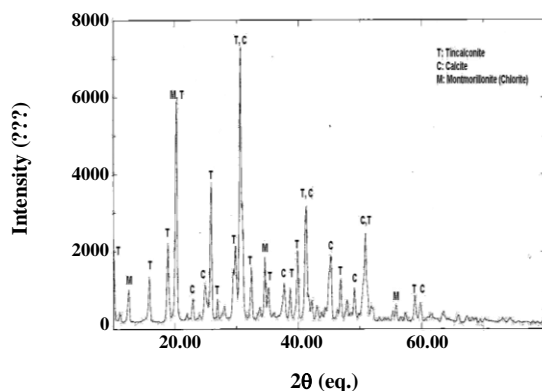
### Conversion reactions

$HNO_3$ , a strong acid, ionizes in an aqueous medium as

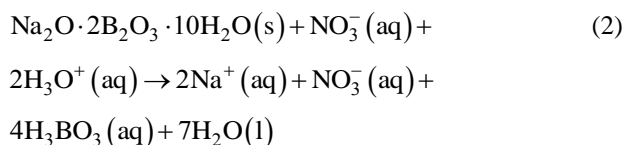


**Table 1: Parameters used in the experiments and their ranges.**

Parameters	Values
Particle diameter, $\mu\text{m}$	-1500+850*, -850+600, -600+425, -425+300
Acid concentration, $\text{mol.L}^{-1}$	0.5, 1, 2*, 4
Mass concentration, $\text{g.mL}^{-1}$	0.04, 0.07, 0.10*
Stirring speed, $\text{s}^{-1}$	20.93, 41.86*, 62.80
Reaction temperature, $^{\circ}\text{C}$	30*, 40, 50, 60

**Fig. 1: X-ray diffractogram of original borax mineral.**

When tincal is added to such a solution, the following reaction takes place in the medium



### Effects of Parameters

The effects of various parameters on the conversion process were investigated using the values for each parameter listed in Table 1. The values of the parameters other than the one being tested for its effect on the dissolution in an experiment were kept constant and these constant values for each parameter are marked with a superscript asterisk in Table 1.

The dissolution rate increased with decreasing particle size (Fig. 2) as the specific surface area of the sample and the dispersion degree of the suspension increased. The variation of the conversion rate between various solid-to-liquid ratios is seen in Fig. 3. The figure shows that the decrease in the amount of solid per amount of reagent

in the reaction mixture has an increasing effect on the conversion rate.

Fig. 4 shows that increasing reaction temperature increased the conversion rate, as expected from the exponential dependence of the rate constant in the Arrhenius equation. The experimental results for the effect of stirring speed on the conversion process are shown in Fig. 5. The stirring speed appears to have less effect on the conversion rate.

The effect of  $\text{HNO}_3$  concentration on the conversion process was investigated using acid concentrations given in Table 1 under the standard experimental conditions. The experimental results presented in Fig. 6 show that the increase of acid concentration increases the dissolution rate. This behavior can be explained by the formation of  $\text{NO}_3^-$  and  $\text{H}_3\text{O}^+$  ions via the dissociation of  $\text{HNO}_3$  in water, as at high acid concentrations the amount of  $\text{H}_3\text{O}^+$  per volume is higher than that at low concentrations, and diffusion of  $\text{H}_3\text{O}^+$  ions to the mineral at high concentrations is faster.

$\text{NaNO}_3$  occurring in the reaction medium dissolves completely and does not affect the diffusion to the mineral surface of  $\text{H}_3\text{O}^+$  ions.

### Kinetics

A kinetic analysis was conducted using the data related with conversion for this process. The ideal models in a heterogeneous reaction system in the form of  $A_{\text{fluid}} + bB_{\text{solid}} \rightarrow \text{Products}$

have been derived taking into account the fluid and solid concentrations under steady state conditions and it has been stated that the reaction rate can be controlled by one of the three ideal models expressed as fluid film diffusion, product film diffusion, and surface chemical reaction for these kinds of systems.

In the literature, mathematical equations are given for models expressing non-catalytic reaction of fluid-solid

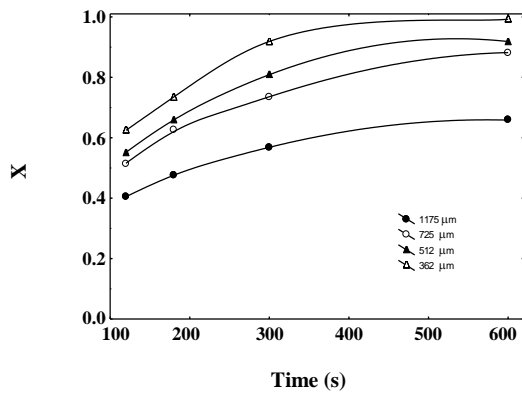


Fig. 2: Effect of particle size on conversion.

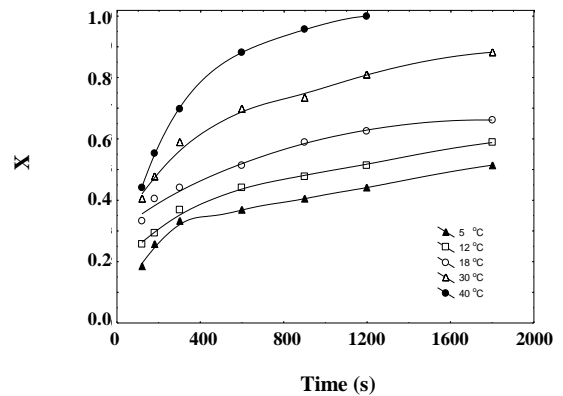


Fig. 4: Effect of reaction temperature on conversion.

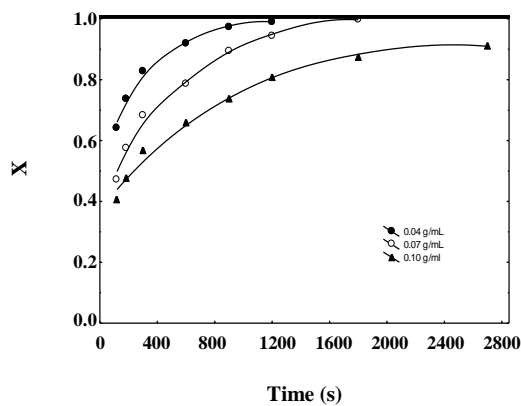


Fig. 3: Effect of solid-to-liquid ratio on conversion.

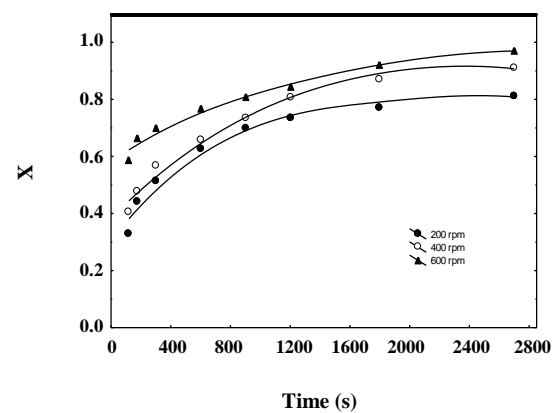


Fig. 5: Effect of stirring speed on conversion.

particles, which are considered to have no change in fluid concentration[32]. A pseudo homogenous model for the cases which fluid reactant concentration changed with time was used by Karagölge *et al.* [33]. Here, this model was used by modification as follows for Reaction (2): conversion rate can be written as

$$\frac{dX_{\text{tincal}}}{dt} = k(1 - X_{\text{tincal}})C_{\text{HNO}_3} \quad (3)$$

According to stoichiometry of Reaction (2), relationship between moles of reactants can be expressed as follows:

$$n_{\text{HNO}_3} \cong 2n_{\text{tincal}} \quad (4)$$

and also,

$$X_{\text{HNO}_3} = \frac{C_{0,\text{HNO}_3} - C_{\text{HNO}_3}}{C_{0,\text{HNO}_3}} = \frac{n_{\text{HNO}_3}/V}{n_{0,\text{HNO}_3}/V} = \frac{n_{\text{HNO}_3}}{n_{0,\text{HNO}_3}} \quad (5)$$

$$X_{\text{tincal}} = \frac{n_{\text{tincal}}}{n_{0,\text{tincal}}} \quad (6)$$

From Equation 5

$$C_{\text{HNO}_3} = C_{0,\text{HNO}_3}(1 - X_{\text{HNO}_3}) \quad (7)$$

and by combining Equations (5), (6) and (7), the following equation is obtained

$$X_{\text{HNO}_3} = \frac{2n_{0,\text{tincal}}}{n_{0,\text{HNO}_3}} X_{\text{tincal}} \quad (8)$$

Then, by substituting this result in Equation 3

$$\frac{dX_{\text{tincal}}}{dt} = k(1 - X_{\text{tincal}}) \left( 1 - \frac{2n_{0,\text{tincal}}}{n_{0,\text{HNO}_3}} X_{\text{tincal}} \right) C_{0,\text{HNO}_3} \quad (9)$$

$$\text{If } p = \frac{2n_{0,\text{tincal}}}{n_{0,\text{HNO}_3}},$$

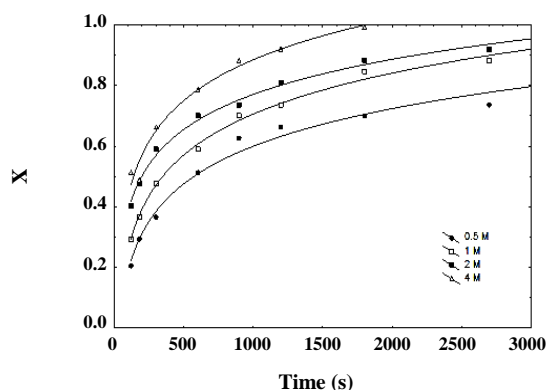


Fig. 6: Effect of acid concentration on conversion.

$$\frac{dX_{\text{tincal}}}{dt} = k(1 - X_{\text{tincal}})(1 - pX_{\text{tincal}}) C_{0,\text{HNO}_3} \quad (10)$$

is obtained. When this equation is integrated, the following equations are found:

$$\frac{1}{1-p} \ln \left( \frac{1-pX_{\text{tincal}}}{1-X_{\text{tincal}}} \right) = kC_{0,\text{HNO}_3} t$$

from  $pX_{\text{tincal}} = X_{\text{HNO}_3}$   
and

$$p = \frac{2n_{0,\text{tincal}}}{n_{0,\text{HNO}_3}} = \frac{2C_{0,\text{tincal}}}{C_{0,\text{HNO}_3}}$$

$$\frac{1}{1-p} \ln \left( \frac{1-X_{\text{HNO}_3}}{1-X_{\text{tincal}}} \right) = kC_{0,\text{HNO}_3} t \quad (11)$$

$$\ln \left( \frac{1-X_{\text{HNO}_3}}{1-X_{\text{tincal}}} \right) = (C_{0,\text{HNO}_3} - 2C_{0,\text{tincal}}) kt \quad (12)$$

$$X_{\text{tincal}} = 1 - \frac{1-X_{\text{HNO}_3}}{\exp \left[ (C_{0,\text{HNO}_3} - 2C_{0,\text{tincal}}) kt \right]} \quad (13)$$

Likewise, the mathematical relationship between rate constant ( $k$ ) and the parameters was found statistically by using computer software as follows.

$$k = 1.53(S/L)^{-0.66} (D)^{-0.70} (SS)^{0.52} \exp(37.493/RT) \quad (14)$$

As a result, kinetics equation defining the conversion process can be expressed as

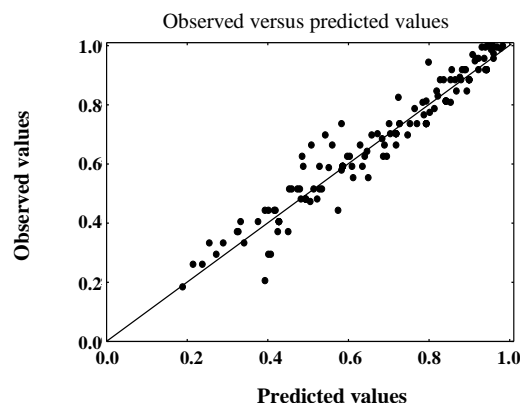


Fig. 7: The experimental and theoretical dissolution fraction data for all the parameters.

$$X_{\text{tincal}} = 1 - \frac{1 - X_{\text{HNO}_3}}{\exp \left[ (C_{0,\text{HNO}_3} - 2C_{0,\text{tincal}}) 1.53(S/L)^{-0.66} (D)^{0.70} (SS)^{0.52} (t)^{0.56} \exp(37.493/RT) \right]} \quad (15)$$

When the experimental values of  $X$  versus the values found from the model above were plotted, it was seen that the model was harmonious to experimental data. It is seen in Fig. 7 in which a plot of observed values versus predicted values is given that the model determined fits to experimental data. Equation 15 and Figs. 1-6 shows that nitric acid concentration and temperature have the most important effect on dissolution. Comparison of this process with the colemanite-sulfuric acid method which is in use presently shows that this process is more advantageous with respect to the following points: A solid waste called borogypsum forms in sulfuric acid-colemanite process. This waste makes filtration difficult and causes boron loss. But, no solid waste exists in the proposed process. There is only a very small amount of solid waste due to impurities in tincal ore, and it may be separated by filtration. By-product of the proposed process is sodium nitrate in solution. As the solid waste in the solution is separated by filtration and boric acid is separated by crystallization and filtration, the remaining sodium nitrate in solution may be obtained and is an important raw material.

## CONCLUSIONS

Conversion of tincal was studied in the range of 0.5-4 mol/L nitric acid concentrations. It was observed that the conversion rate increased with decreasing particle size and solid-to-liquid ratio and with increasing temperature and acid concentration, but it was less

affected by stirring speed. The effective parameters on the conversion rate are reaction temperature, particle size, solid-to-liquid ratio, and acid concentration. The activation energy is  $37.5 \text{ kJ.mol}^{-1}$

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### Nomenclature

X	Fractional conversion
t	Time, min
t*	Time for complete conversion of a single solid particle, min
b	Stoichiometric coefficient of B (solid) reacting with each mole of A, fluid
D	Particle size, mm
T	Reaction temperature, °C
S/L	Solid-to-liquid ratio, g/mL
E	Activation energy, kJ/mol
k	Reaction rate constant
C	Concentration, M
SS	Stirring speed, rpm
n	Mol of reactant
V	Solution volume, $\text{cm}^3$
$k_g$	Mass transfer coefficient for film diffusion control, cm/s
$\rho_B$	Molar density, $\text{mol/cm}^3$
R	Particle radius, cm
$k_s$	First reaction rate constant based on unit surface, cm/s
$D_e$	Effective diffusion coefficient, $\text{cm}^2/\text{s}$

### Indices

Af	Fluid A
B	Solid reactant
0	Initial

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