MASS TRANSFER DURING BORIC ACID DISSOLUTION

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Abstract. The process of mass transfer during the dissolution of boric acid spheres in water was investigated in the temperature range from 293 K to 323 K, and at stirring rotation rates from 1.67 to 6.67 s^{-1}. The study aimed to determine the dependence of the dissolution rate on the stirring rotation rate and water temperature. The analysis of the experimentally obtained results revealed that the most significant factor affecting the intensification of the dissolution process is an increase in the solution temperature. The phenomena of external and internal diffusion during the dissolution of boric acid spheres in water under agitation were also examined. To evaluate the mass transfer process, a generalized criterion equation was used that takes into account all the factors under study. Comparison of experimental and theoretically calculated values showed that the maximum absolute relative error does not exceed 6 %. The results obtained are valuable for further research and potential applications in the chemical industry, pharmacology, and cosmetology.

Keywords: boric acid, dissolution, agitation, diffusion, mass transfer.

1. Introduction

Boric acid is widely used in various industries such as chemical, food, medical, pharmaceutical, nuclear energy, and others. Boron is a chemically scarce element, and the primary producers of boric acid are the USA and Turkey, which possess the largest reserves of boron-containing raw materials. According to literature, the consumption of boron-containing compounds in the world is growing by an average of 2.4 % per year[1].

Industrial production of boric acid is carried out in the form of powder that must be dosed to prepare solutions. To ensure high process productivity, the solution preparation is continuous, and the powdered material must be continuously fed using various volumetric or gravimetric dosing devices. Volumetric dosing devices are easy to manufacture and reliable in operation. However, volumetric dosing of powdered materials is challenging due to their hygroscopic nature, tendency to agglomeration, and, as a result, uncontrolled changes in bulk density. Therefore, to overcome this drawback, it is advisable to granulate powdered materials, which will ensure higher dosing accuracy. However, the dissolution rate of solid materials in a liquid medium depends not only on the intensity of agitation but also on the surface of the solid particles that come into contact with the liquid. Granulated materials have significantly smaller surface areas in contact with the solvent compared to powdered materials. As a result, the study of the dissolution kinetics of granulated boric acid is a relevant task.

In the industry, the dissolution of solid substances is carried out by various methods, such as mechanical stirring in a liquid flow, in a pseudo-fluidized bed of solid particles, air or inert gas bubbling in the solution, the use of screw apparatus, and others. Each of these methods has its advantages and disadvantages, so the specific method is chosen based on the requirements of the technological process. However, in most industrial production cases, mechanical stirring is commonly employed.

Therefore, this study aims to experimentally and theoretically investigate the dissolution kinetics of granulated boric acid (GBA) in distilled water, under different intensities of agitation and at different temperatures.

Research data on various substances present the results of mass transfer coefficient determinations during their dissolution using different intensification methods.

Symak *et al.*[2] provide the results of an experimental study on the dissolution kinetics of potassium sulfate with an average granule diameter of $4 \cdot 10^{-3}$ m in distilled water under pneumatic agitation. They determine the mass transfer coefficient based on experimental investigations and compare it with values theoretically calculated using the theory of locally isotropic turbulence.

Sabadash *et al.*[3] focus on investigating the influence of stirring rotation rate on the mass transfer coefficient. The authors develop a mathematical model of diffusive adsorption for a solid particle and determine the coefficient of internal diffusion. The experimental data are generalized as the Sherwood number dependence on the Reynolds number.

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The processes of dissolution and mass transfer involving chemical reactions are discussed by Frikha *et al.*[4]. Two kinetic models based on heat and mass balances and associated mass transfer in the solid-liquid system with chemical-kinetic processes are presented. The results obtained in this study are correlated with those of other authors. Additionally, the model reflects the change in particle size as a function of time and the dissolution time as a function of agitation intensity. Furthermore, the developed kinetic dissolution model considers the influence of temperature and concentration changes on the dissolution kinetics.

The impact of the gas phase released at the solid surface on the enhancement of the mass transfer coefficient through turbulence in the boundary diffusion layer is demonstrated by Mena et al.[5]. The dissolution of solid substances can be intensified by hydrodynamic cavitation. This work investigates mass transfer during dissolution by the influence of a hydrodynamic cavitation field. The authors suggest that the dissolution intensity for substances dissolving *via* diffusion-controlled and kinetically-controlled mechanisms should be considered. In both cases, the dissolution intensity is determined by the driving force (concentration gradient), particle surface area, and mass transfer coefficient.

To intensify mass transfer, Gumnitsky *et al.*[6] recommend introducing an inert gas into the solid-liquid system. In the work[7], the inert phase is created by creating a vacuum in the system. The vapor bubbles formed on the solid surface disrupt the boundary diffusion layer, leading to process non-stationarity, which contributes to the increase in kinetic coefficients. All these methods have their advantages and drawbacks. For instance, when using bubbling in the solution, the mass transfer process is often slowed down due to foam formation, which carries away fine solid particles, reducing their participation in the dissolution process.

The above-discussed dissolution processes are related to one or several solid substances with diverse particle size distributions in different solvents and with the use of various intensification methods. In other words, the results presented in the analyzed works are specific to the particular laboratory setup under certain temperature and hydrodynamic conditions, making it impossible to directly apply them to the dissolution of other solid materials under different hydrodynamic conditions.

The review of literature sources shows a wide range of values for the mass transfer coefficient and a discrepancy between values obtained through different methods. Mathematical modeling of mass transfer processes in the solid-liquid system is based on various simplifications and assumptions that affect the accuracy of the obtained results. As stated by Inglezakis *et al.*[8], the dissolution mechanism is not thoroughly studied, which complicates the modeling process.

The mass transfer patterns at low stirring rotation speeds have not been adequately explored. It is essential to consider the particle retention on the vessel bottom to account for their size during mathematical modeling of the process. In this work, the authors measured the apparent mass transfer rate between particles of cation-exchange resin at low stirring rotation frequencies. However, the actual mass transfer rate was not determined for transient regimes, such as stirring start-up and stoppage, as they used the total interfacial area[9].

After analyzing the literature data on the solubility of boric acid, it is evident that there is certain variability in the dissolution coefficients[10]. According to studies by Linke, Perry, and Lange, at a temperature of 273 K, the difference in data is 1.5 %, and at a temperature of 283 K, it is 5.6 %. Given this situation, the investigation of boric acid dissolution kinetics remains a relevant task.

Carletti *et al.*[11] used the electrical resistance method to characterize the dissolution of particles during agitation. Blade rotation speeds lower than the optimal impeller speed were applied. If agitating occurs at a rotation speed lower than that required for initial complete particle suspension, a significant reduction in the energy required for the process can be achieved. The experimental data collected in the study confirm that neither Kolmogorov's theory nor the particle settling velocity theory can be used for accurate prediction of the mass transfer coefficient in devices of different sizes. As a result, the use of dimensionless criteria to describe the mass transfer process is justified.

The determination of the mass transfer coefficient for solid-liquid agitating was performed by Joshi *et al.*[12] using an artificial neural network regressor. This allowed for describing the mass transfer process during agitation based on dimensionless criteria with a relatively low error. The analysis of sources conducted in this study attests to the unquestionable leadership of using dimensionless criteria to describe the dissolution process. The correlation found using the neural network only confirms the relevance of using the Sherwood, Reynolds, and Schmidt criteria.

2. Experimental

2.1. Experimental Setup

The schematic diagram of the experimental setup is shown in Fig. 1. An asynchronous motor 1 with a three-blade impeller 2 stirs distilled water in a thermostable flask 3, in which the granules of GBA 4 were placed. The

temperature of the GBA solution was measured using a thermocouple 5, which was connected to a control device 9. The control device was connected to a heating element 8 and the electric motor 1 to maintain the desired temperature of the GBA solution and the rotation speed of the impeller.
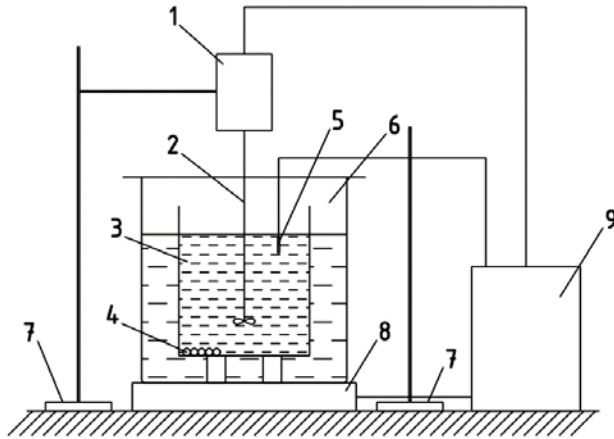

Fig. 1. Schematic diagram of the experimental setup:
1 – asynchronous motor, 2 – three-blade impeller,
3 – thermostable glass flask, 4 – granules of boric acid,
5 – thermocouple, 6 – control device, 7 – support,
8 – heating element, 9 – regulating device

2.2. Experimental Methods

Chemically pure boric acid of Turkish origin was used in the study. The experiment took place in a cylindrical vessel with a height of 0.21 m and a diameter of 0.16 m. The temperature was controlled using the Sestos D1s-Vr-220 regulating device and the UNI-T UT-T03 thermocouple, with an accuracy of 0.5 K. To measure

the mass of the spheres, an AD3000 scale was employed, with an accuracy of 0.01 g. The diameter of the spheres was measured using a YT-72300 micrometer, with an accuracy of 0.1×10^{-4} m.

The powder of boric acid was loaded into a die and pressed to form pellets with a diameter of 20 mm using a pressing force of 4 MPa. Before the experiment, the boric acid pellets were placed in a container with a saturated boric acid solution. At certain time intervals, the pellets were removed, the surface moisture was removed with filter paper, and their diameter and mass were measured. The experiment continued until a constant mass was obtained.

For the experimental investigation of the GBA dissolution kinetics, a heating element was activated, and distilled water in the container was heated to the desired temperature. The specified rotation speed of the three-blade impeller was set, and 5 boric acid pellets were loaded simultaneously. Every 180 seconds, the pellets were taken out of the container, surface moisture was removed using filter paper, and the diameter and mass of the pellets were measured. Each experiment was repeated at least three times until stable values were obtained. The change in boric acid concentration was calculated based on the change in mass of the boric acid pellets, and the surface area of the pellets was determined by measuring their diameter. The experiments were conducted at temperatures of 293, 303, 313, and 323 K, and at impeller rotation speeds of 1.67, 3.33, 5, and 6.67 s^{-1}.

The physical characteristics of the pellets and boric acid solution are provided in Tables 1–3.

According to reference data, the saturation concentration of boric acid in distilled water is provided in Table 2.

Table 1. Physical characteristics of boric acid granules

Diameter $d \cdot 10^3$, m	Mass, $M_0 \cdot 10^3$, kg	Porosity, %	Volume, $V \cdot 10^6$, m³	Surface, $F \cdot 10^{-3}$, m²	Molar mass, kg/kmol	Density, kg/m³
20	6	14	4.19	1.256	61.83	1435

Table 2. Saturation concentration of boric acid at different temperatures

T, K	293	303	313	323
Cs, kg/m³	50.04	66.00	87.20	115.40

Table 3. Physical characteristics of the solution

T, K	$\mu \cdot 10^4, Pa \cdot s$	ρ, kg/m³	$D \cdot 10^8$, m²/s
293	10.04	998.20	2.55
303	7.97	995.60	3.32
313	6.58	992.24	4.15
323	5.47	988.00	5.16

2.3. Theoretical Part

The dissolution of solid substances is calculated using a system of differential equations of material balance and the kinetic mass transfer equation.

$$\begin{cases} M_0 - M = W_C \cdot \overline{C_\tau}, & (1) \\ \dfrac{dM}{d\tau} = \beta \cdot F \cdot (C_s - \overline{C_\tau}), & (2) \end{cases}$$

where M_0 is the initial mass of the pellet, kg; M is the current mass of the pellet during dissolution at the time of measurement, kg; $\overline{C_\tau}$ is the average concentration of boric acid in the solution at the time of measurement, kg/m^3; τ is the residence time of the boric acid pellet in the vessel, s; β is the mass transfer coefficient during dissolution, m/s; F is the surface area of the pellet, m^2; W_c is the volume of liquid in the vessel, m^3; C_s is the saturation concentration at the current temperature of the solution, kg/m^3.

The difference $C_s - \overline{C_\tau}$ represents the driving force of the dissolution process.

By integrating Eq. (2) over the time interval from 0 to τ, we obtain the time dependence of the mass transfer coefficient.

$$\beta = \frac{M_0 - M}{F \cdot (C_s - \overline{C_\tau}) \cdot \tau}. \quad (3)$$

The surface area of the pellet, $\overline{F}$, decreases during dissolution, therefore, the following average value of this quantity was used for calculations:

$$\overline{F} = \frac{\pi \cdot (d_i^2 + d_{i+1}^2)}{2}, \quad (4)$$

where d_i is the pellet diameter at time τ, i is the number of samples ($i = 1, 2, 3, \dots n$).

The average value of boric acid concentration $\overline{C_\tau}$ in the solution was determined using the following equation:

$$\overline{C_\tau} = \frac{C_{i+1} + C_i}{2}, \quad (5)$$

where C_i is a current concentration of boric acid in the solution ($C_0 = 0, \tau = 0$), kg/m^3.

The concentration of boric acid in the solution during the time interval τ was determined from Eq. (6):

$$C_\tau = \frac{M_0 - M}{W}. \quad (6)$$

In mass transfer theory, kinetic, hydrodynamic, and geometric variables are represented by dimensionless groups. In dimensionless groups, the mass transfer coefficient β is expressed in the form of the Sherwood number.

$$Sh = \frac{\overline{\beta} \cdot \overline{d}}{D}, \quad (7)$$

where $\overline{\beta}$ is the average value of the mass transfer coefficient, $\overline{d}$ is the average diameter of the boric acid pellet, m; D is the diffusion coefficient of boric acid in distilled water, m^2/s.

The Sherwood number is a function of several parameters, with the main ones being the Reynolds number (Re), Schmidt number (Sc), and Geometrical simplex (Γ).

$$Sh = f(Re, Sc, \Gamma).$$

The empirical equation for determining the Sherwood number for the investigated system gas a view:

$$Sh = A \cdot Re^x \cdot Sc^y \cdot \Gamma, \quad (8)$$

where A is the proportionality constant, determined on the basis of experimental data.

The Reynolds number, which characterizes the hydrodynamic situation of the system, is defined as:

$$Re = \frac{\rho \cdot n \cdot d_m^2}{\mu}, \quad (9)$$

where ρ is the liquid density, kg/m^3; n is the rotational speed of the stirring, s^{-1}; d_m^2 is the stirring diameter of the, m; μ is dynamic viscosity of the liquid, Pa·s.

The Schmidt number, which characterizes the physical parameters of the system, is calculated as:

$$Sc = \frac{\nu}{D}, \quad (10)$$

where ν is the kinematic viscosity of the solution, m^2/s.

The value of the geometric simplex is determined by the ratio of the particle diameter to the apparatus diameter:

$$\Gamma = \frac{d_к}{D_a}, \quad (11)$$

where $d_к$ is the diameter of the boric acid pellet, m; D_a is the diameter of the apparatus, m.

Considering that the physical parameters of the solution vary within a narrow range, according to the recommendations of many authors[13], we assume that the exponent near the Schmidt number is 0.33. Thus, the empirical equation takes the form:

$$Sh = A \cdot Re^x \cdot Sc^{0,33} \cdot \Gamma. \quad (12)$$

The diffusion coefficient was estimated using the Wilke-Chang method from the following equation[14]:

$$D = 7,4 \cdot 10^{-8} \cdot \left[\frac{(\phi \cdot M_p)^{0,5} \cdot T}{\mu_p \cdot V_1^{0,6}} \right], \quad (13)$$

where D is the mutual diffusion coefficient at very low concentration of the solute, m^2/s; M_p is the solvent molecular weight; T is the temperature, K; μ_p is the solvent viscosity, mPa·s; V_1 is the molar volume of the solution at the boiling temperature, m^3/mol; ϕ is the "association parameter" of the solvent (for water ϕ =2.6).

3. Results and Discussion

The results of the experimental studies on the change in the diameter of boron acid granules are presented in Fig. 2. As a result of the dissolution of the granulated boron acid, the diameter of the granules decreases. Therefore, to determine the mass transfer surface area, the average surface area of the granules was calculated during the experimental investigations at different temperatures and stirring speeds using Eq. (4). The results of these investigations are shown in Fig. 2.

The analysis of the results from the experimental investigations of the changes in the surface area of boron acid granules, as shown in Fig. 2, indicates that both the astirring frequency and the temperature of the distilled water affect the intensity of the average surface area reduction of the granules. For instance, after 1600 seconds, at a stirring frequency of 1.67 s^{-1} and a temperature of 293 K, the average surface area of the granules decreases from 1.35×10^{-3} m^2 to 1.145×10^{-3} m^2, which is a reduction of 1.18 times, and at a temperature of 323 K, it reduces to 0.66×10^{-3} m^2, which is a reduction of 2.05 times.
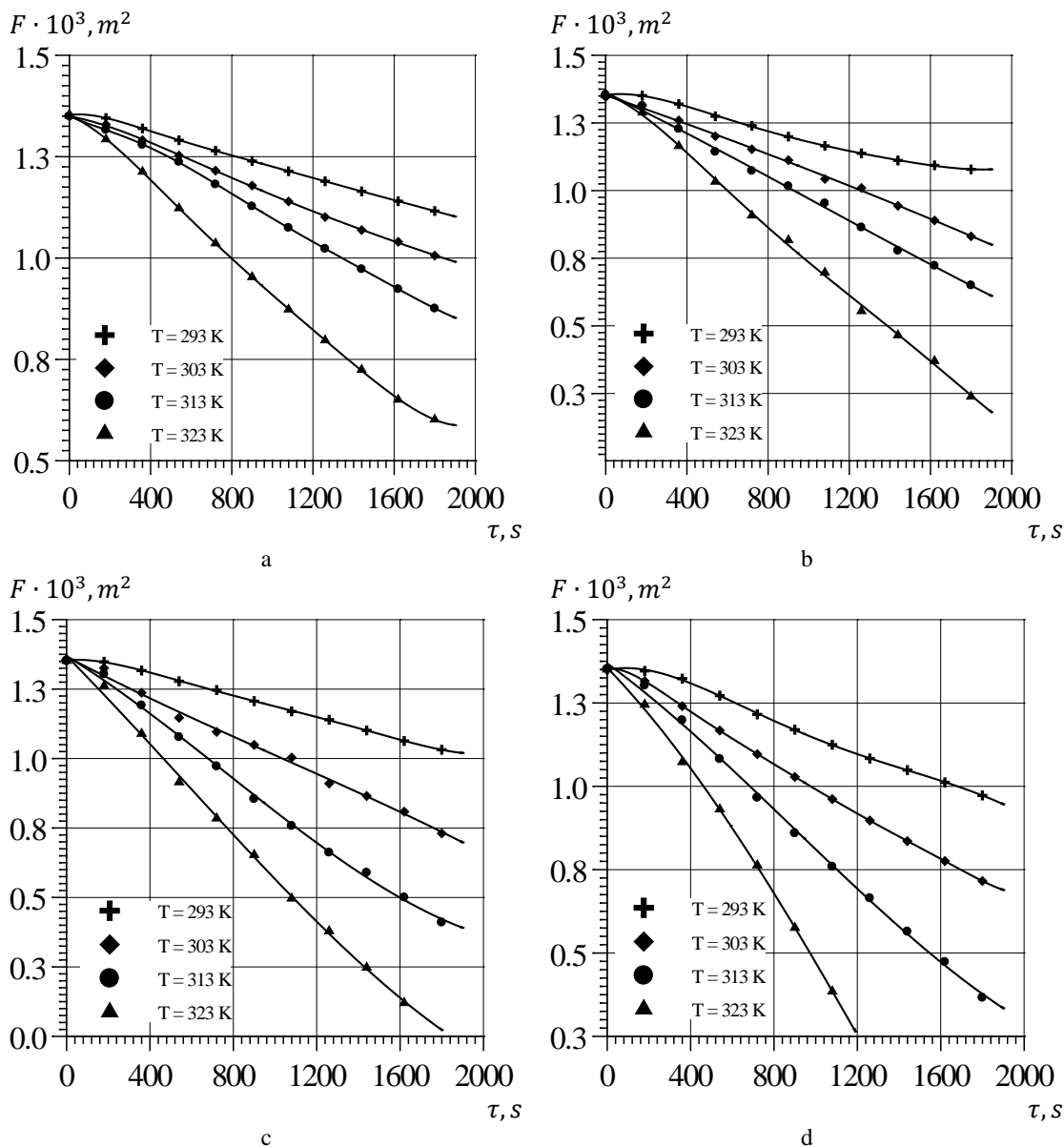

Fig. 2. Change in the average surface area of granules during dissolution.
a – 1.67 s^{-1}; b – 3.33 s^{-1}; c – 5 s^{-1}; d – 6.67 s^{-1}

At a stirring frequency of 5 s^{-1} and a temperature of 293 K, after 1600 seconds, the average surface area of the granules reduces from 1.35×10^{-3} m^2 to 0.48×10^{-3} m^2, which is a reduction of 2.8 times, and at a temperature of 323 K, it reduces to 0.14×10^{-3} m^2, which is a reduction of 9.64 times. Therefore, as observed in Fig. 3, the concentration of the boron acid solution increases accordingly. Moreover, at a stirring frequency of 1.67 s^{-1} and an increase in water temperature from 293 K to 323 K over 1600 seconds, the dissolution intensity increases by 3.36 times, whereas at a temperature of 323 K and a

change in stirring frequency from 1.67 s^{-1} to 6.67 s^{-1}, it only increases by 1.74 times.

To generalize the experimental data, it is appropriate to present them in dimensionless complexes. Such representation of the experimental results will enable their application for predicting the kinetics of boron acid dissolution in production volumes. Mass transfer coefficients were calculated based on the experimental data using Eq. (3), and the calculation results are presented in Table 4.
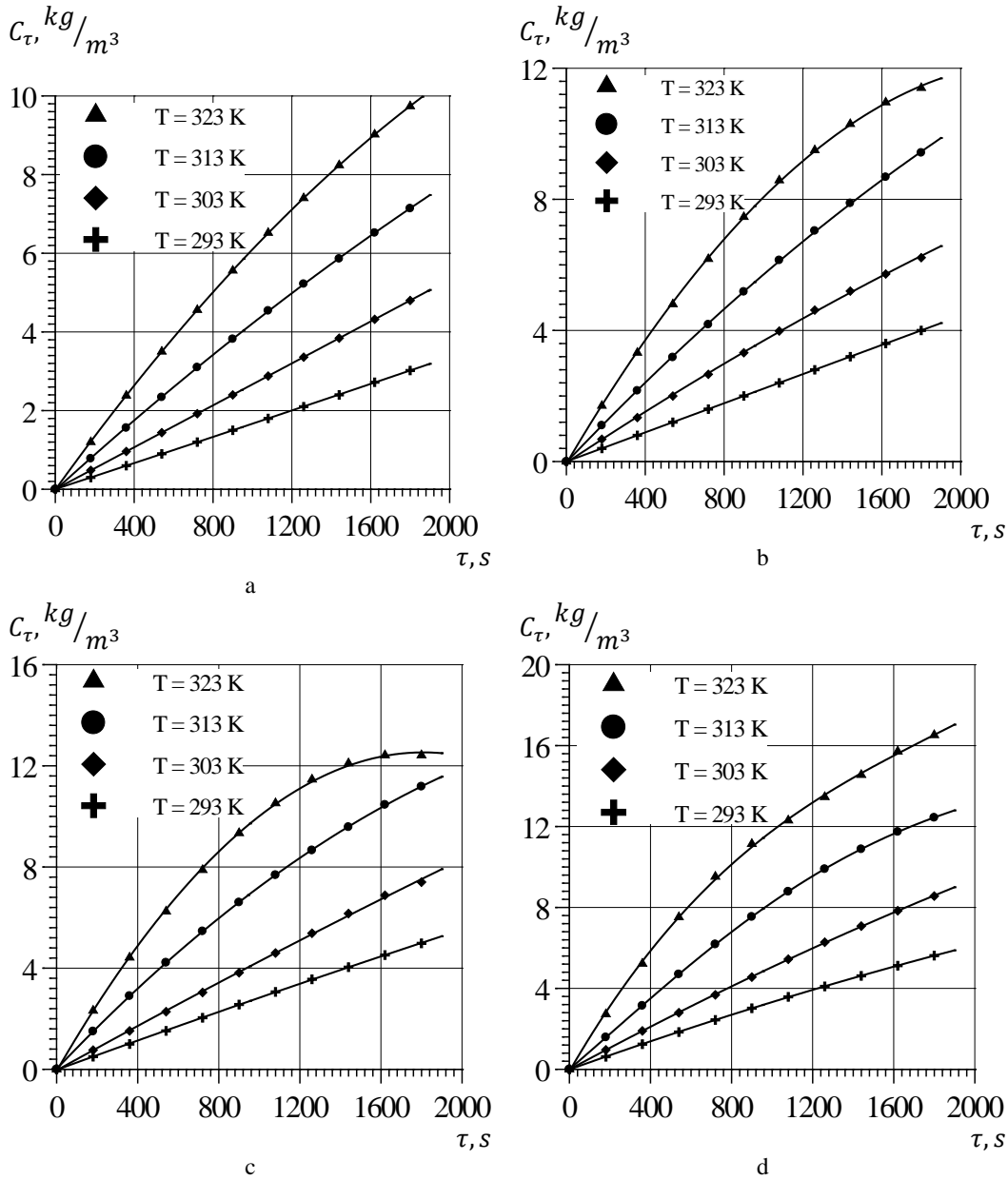

Fig. 3. Kinetics of Boron Acid Dissolution: a – 1.67 s^{-1}; b – 3.33 s^{-1}; c – 5 s^{-1}; d – 6.67 s^{-1}

Table 4. Averaged values of mass transfer coefficients and diameters of boron acid granules depending on temperature and stirring frequency for 1800 s

n, s^{-1} \ T, K	$\beta \cdot 10^5, m/s$				$d_{\text{к}} \cdot 10^2, \text{m}$			
	293	303	313	323	293	303	313	323
1.67	1.40	1.73	2.08	2.56	1.95	1.92	1.87	1.72
3.33	1.90	2.40	3.10	3.85	1.93	1.85	1.77	1.64
5	2.33	3.18	4.47	4.84	1.94	1.75	1.60	1.71
6.67	2.70	3.55	5.06	6.21	1.91	1.80	1.59	1.6

Table 5. Values of coefficients of internal diffusion of boron acid and Schmidt numbers

T, K	$D, \cdot 10^8 \text{ m}^2/\text{s}$	Sc
293	2.55	39.30
303	3.22	24.10
313	4.15	15.96
323	5.16	10.73

The relationship between the mass transfer coefficients $\frac{\beta_{T+10}}{\beta_T}$ indicates its decrease within the range of 1.20–1.31, indicating that the process is in the diffusion-controlled region within the specified interval of the study.

The calculated values of the coefficients of internal diffusion of boron acid from the granule pores into the solution and the Schmidt numbers depending on the solution temperature are presented in Table 5.

To determine the unknown coefficients A and x in Eq. (12), the experimental data were represented as a graphical relationship $Sh/Sc = f(Re)$ in logarithmic coordinates (Fig. 4, *a*).

By approximating the results of the experimental studies with a power function, we obtained the calculated values of the coefficient *A* and *x* for the investigated temperatures of the boron acid solution

$$Sh = 4.92 \cdot 10^{-1} \cdot Re^{0,5} \cdot Sc^{0.33} \cdot \frac{d_{\text{к}}}{D_a}. \qquad (14)$$

A comparison of the calculated Sherwood numbers based on experimental data with the theoretically calculated values based on Eq. (14) is presented in Fig. 4. As shown in Fig. 5, the maximum absolute value of the relative error does not exceed 6 %, which is considered acceptable for practical calculations of technological parameters for dissolving granulated boric acid in chemical industry facilities.
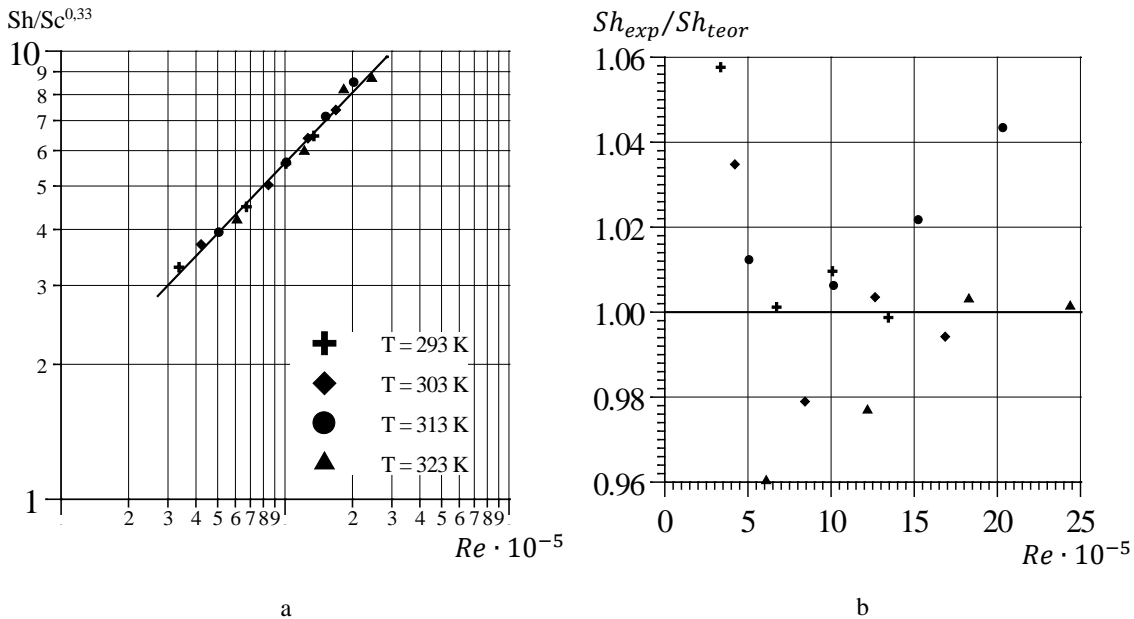

a

b

Fig. 4 consists of two subplots: *a* shows the graphical dependence of $\frac{Sh}{Sc} 0.33 = f(Re)$ based on experimental data, and *b* represents the ratio of the calculated Sherwood numbers from experimental data to the theoretically calculated ones based on Eq. (14) as a function of Reynolds number $Sh_{exp}/Sh_{teor} = f(Re)$

4. Conclusions

The dissolution of boric acid in distilled water was investigated under various temperatures and stirring speeds. The results showed that the driving force of the dissolution process depended on both the stirring speed and the temperature of the distilled water. The data analysis revealed that an increase in the stirring speed and the solution temperature led to a higher intensity of boric acid dissolution. However, the temperature had a more significant impact on intensifying the dissolution process compared to the stirring speed, though it did not shift the process from diffusion-controlled to kinetic-controlled. The mass transfer process was evaluated using a generalized dimensionless equation, which considered all the investigated factors. The experimental values were compared with the theoretically calculated ones, and it was found that the maximum absolute value of the relative error did not exceed 6 %. These results indicate that the obtained data are sufficiently accurate for practical calculations of technological parameters in the dissolution of granulated boric acid in the chemical industry.

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МАСООБМІН ПІД ЧАС РОЗЧИНЕННЯ БОРНОЇ КИСЛОТИ

Анотація. *Досліджено процес масообміну під час розчинення кульок борної кислоти у воді за температури 293–323 К та за частоти обертання мішалки від 1,67 до 6,67 с⁻¹. Метою дослідження було визначення залежності інтенсивності розчинення від частоти обертання мішалки та температури води. Аналіз експериментально отриманих результатів показав, що найбільш значущим фактором. який впливає на інтенсифікацію процесу розчинення, є підвищення температури розчину. Досліджено також явища зовнішньої та внутрішньої дифузії в процесі розчинення кульок борної кислоти у воді під час перемішування. Для оцінки процесу масопереносу використано узагальнене критеріальне рівняння, яке враховує всі досліджувані фактори. Порівняння експериментальних і теоретично розрахованих значень показало, що абсолютна відносна похибка не перевищує 6 %. Отримані результати цінні для подальших досліджень і потенційного застосування в хімічній промисловості, фармакології та косметології.*

Ключові слова: *борна кислота, розчинення, інтенсифікація, дифузія, масообмін.*