Comparative Evaluation of the Contact Elements Efficiency for Barium Sulfide Solution Carbonization

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Abstract. The article presents the results of an experimental study aimed at obtaining scientifically valid data on the kinetics of absorption of carbon dioxide by a barium sulfide solution and the effect of the design of contact elements (trays) on the mass transfer coefficient in this process. The work was carried out using a laboratory model of the absorber, in which it was possible to install trays of various types. Analysis of literature sources showed that the process of a BaS solution carbonization takes place in two stages, sharply differing in pH. An experimental study of CO_2 absorption kinetics under the conditions of the first stage of the process made it possible to identify the most significant factors influencing its rate. It was also found that the limiting stage of mass transfer is the resistance in the gas phase. The carbonization rate at the second stage is significantly lower than at the first stage and is controlled by the kinetics of the chemical reaction of CO_2 hydration. Mathematical processing of the results of testing models of the cap, sieve, and double-flow trays made it possible to obtain formulas for calculating the mass transfer coefficients for each of them. In the studied range of gas velocities, the mass transfer coefficient on a dual-flow tray was 1.5–2 times lower than on a sieve tray and 2–2.5 times lower than on a cap tray. The data obtained were used in the design of the absorption apparatus.

Keywords: Process innovation · Absorption · Carbon dioxide · Barium Sulfide solution · Mass transfer kinetics · Mass transfer ratio · Cap tray · Sieve tray · Dual flow tray

1 Introduction

One of the leading products in the range of barium compounds is barium carbonate. This substance is used primarily in the glass industry and as raw material for producing many other barium salts.

At present, barium carbonate is obtained from barite concentrate using rather complex technology, including roasting, leaching of barium sulfide, processing it with hydrochloric acid, and precipitation of $BaCO_3$ with sodium carbonate. The advantage of this technology is the high purity of the product obtained. The disadvantages are the use of expensive chemicals, cumbersome equipment, and the high cost of the product.

The search for cheaper methods for obtaining barium carbonate led to the idea of direct carbonization of a barium sulfide solution [1]. Particular difficulties in implementing this technology appear due to insufficient knowledge of the kinetics of the process, which complicates the choice of an appropriate method for organizing mass transfer and the design of the main apparatus, i.e., the absorber.

Barium sulfide carbonation, as it will be shown below, is a complex, multi-stage process. Therefore, it is practical and scientific interest to determine the limiting stages and factors affecting its kinetics. Thus, from the viewpoint of the possibility of creating equipment for implementing the direct carbonization method of barium sulfide solution, it is essential to study this process to obtain scientifically proved data for the design of the absorber.

2   Literature Review

Barium sulfide solution, a strong base salt, and a weak acid are alkaline. The absorption of carbon dioxide by alkaline solutions is widespread in technology [2] and used in numerous food and chemical industries branches.

CO_2 absorption takes place in several stages. The main ones, in terms of the film theory [3, 4], are a diffusion of carbon dioxide from the bulk of the gas to the interface; CO_2 dissolution; diffusion from the interface deep into the liquid; chemical interaction of carbon dioxide with the active component of the liquid. The two last stages mentioned run simultaneously.

In the case of carbon dioxide absorption by barium sulfide solution, the process is complicated due to simultaneous desorption of hydrogen sulfide, which should affect the mass transfer coefficient in the gas phase and pH of the absorbent [5]. Considering the latter factor, the desorption of hydrogen sulfide should also affect the rate of carbon dioxide reaction in the liquid. In addition, the crystallization of barium carbonate is a diffusion process related to crystallization supersaturation [6, 7], and it can affect the kinetics of CO_2 absorption.

The interaction of carbon dioxide with alkaline solutions has been studied in detail. In works [3, 5, 6], it was shown that carbon dioxide in alkaline solutions could enter into the following reactions:

$$CO_2 + OH^- \leftrightarrow HCO_3^- \tag{1}$$

$$CO_2 + H_2O \leftrightarrow HCO_3 \tag{2}$$

The kinetics of carbon dioxide hydration reactions was studied in [3] and [8–10]. The authors obtained equations for calculating the kinetic coefficients of reactions (1), (2). In particular, it was found out that at pH from 8 to 11.5, reaction (2) generally proceeds,

and at pH > 10, the rate of carbon dioxide hydration is mainly determined by reaction (1).

When carbonizing barium sulfide solution, the products of reactions (1) and (2) in an aqueous solution enter into a range of reactions proceeding in the ionic form that is relatively fast, thus, they do not control the process rate.

Barium sulfide carbonation can be described in the following gross equation [11]:

$$BaS + CO_2 = BaCO_3{\downarrow} + H_2S \tag{3}$$

However, the mechanism of this process, in our opinion, should be more complicated since the reactions in solution proceed in the ionic form [5]. Barium sulfide hydrolyses in an aqueous solution:

$$BaS + H_2O \leftrightarrow Ba^{2+} + HS^- + OH^- \tag{4}$$
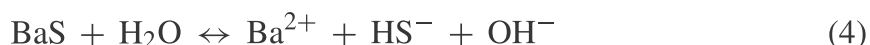
As a result of a reaction (4), an excess of OH^- ions appear in the solution, which determines the alkaline reaction of barium sulfide solution.

The barium ion formed in the process (4) reacts with the carbonate ion as follows:

$$Ba^{2+} + CO_3^{2-} = BaCO_3{\downarrow} \tag{5}$$

The reaction (5) proceeds in the ionic form, and its rate should be sufficiently high, at least higher than reactions (1), (2); nevertheless, the crystallization of the barium carbonate formed over is a heterogeneous process, so there should be some saturation providing crystallization.

Barium sulfide, a salt with a strong base, is almost completely dissociated from the solution. Therefore, at the initial stages of the carbonization process at a molar ratio of $Ba/S^- > 0.5$, the solution has a high pH value [5, 11] due to reaction (4). At subsequent stages, at $Ba/S < 0.5$, i.e., after barium sulfide is wholly converted into hydrosulfide, the pH of the solution reduces rapidly. The hydrosulfide ion formed by reaction (4) adds a proton to the reaction.

$$HS^- + H^+ \leftrightarrow H_2S \tag{6}$$

The rate of this reaction increases with decreasing pH. The formed hydrogen sulfide is desorbed from the solution.

Considering the above analysis of the literature, it can be outlined that the kinetics of the reactions of carbon dioxide hydration and the formation of barium carbonate is related to pH, which was experimentally confirmed in [11]. This relation can become a criterion for whether the chemical reaction is the limiting stage of the absorption process. It should be noted that, among the relatively limited data available in the literature on the kinetics of carbonization of barium sulfide, it was not possible to find information that would make it possible to determine the limiting stages of the process. For example, in [12], the study of kinetics was carried out only at high pH, and in [13], pH control was not carried out, and the decrease in the absorption rate at the end of the experiment was not commented on.

Also, no recommendations were found on choosing a suitable design of a contact element for the industrial implementation of the process under study.

3   Research Methodology

3.1   Laboratory Set

The process study of barium sulfide carbonization was carried out in three stages. In the first preliminary experiments, the dependence of the absorption rate of carbon dioxide to the conversion of barium sulfide into carbonate (carbonization level) was studied. In this part of the work, a bubbling apparatus was used as the main element of the laboratory setup, which was a cylindrical container with a jacket 800 mm high. The inner diameter of the cylinder was 43 mm. The apparatus was equipped with nipples for inlet and outlet of gas, heating water, and sampling.

The second stage aimed to identify factors that significantly affect the process's kinetics and compare the mass transfer characteristics of the cap, sieve, and dual flow trays that were supposed to be used in the designed absorber. The laboratory apparatus used in this part of the study differed from the one described above in its inner diameter, which was 60 mm, and in the presence of a connector in the lower part, into which the models of the studied trays were set up. An externally adjustable overflow was built into the connector.

The trays under study had the following characteristics. The dual-flow tray was a 1 mm thick stainless steel disc with holes 10 mm in diameter. The free section of the tray was 19.4%. The sieve tray had a similar design, but the hole diameter was 6 mm, and the free section was 17%. The cap tray had one cap. The outer diameter of its cylindrical part was 30 mm. 9 slots 5 mm wide and 10 mm high were cut evenly along its perimeter.

At the preliminary stage of the study, the laboratory setup consisted of a gas mixture preparation unit, an absorber, and an equipment set for measuring and regulating gas flow rates, measuring temperatures, and taking liquid samples for analysis. The setup was supplemented with supply and liquid drainage systems at the second and third stages.

3.2   Experiment Method

At the preliminary stage, barium sulfide carbonization was researched in batch mode. At the beginning of the experiment, the flow rates of carbon dioxide and nitrogen were set by the program of experiments. The gas mixture obtained was fed through a mixer into a laboratory absorber, and then the prepared and previously analyzed absorbent was added to it. During the experiment, liquid and gas samples were periodically taken for analysis.

In the experiments carried out according to the described method, it was found out that after reaching the conversion of sulfide into barium carbonate (further referred to as carbonization level) value of 50%, the rate of carbonation was significantly reduced. Within each of these intervals, the carbonation rate practically did not change. This phenomenon can only be explained by a fundamental change in the mechanism of the process. Therefore, further research was carried out separately for solutions with carbonization levels less than and more than 50%.

The experiments of the second stage were carried out in a flow-through mode. The liquid, heated to a predetermined temperature, was continuously pumped into the absorber and drained into the collector. After the experimental model was established, the values

of the contacting media flow rates were recorded, and gas and liquid samples were taken for analysis before and after the absorber. In addition, the temperature and pressure in the apparatus were recorded.

The primary processing of the experimental results included calculations: gas velocity, the density of irrigation with liquid, absorption rate, its driving force. The mass transfer coefficient was calculated using the well-known formula.

$$Kg = r_{CO_2}/\left(S \cdot \Delta p_{CO_2}\right) \tag{7}$$

where r_{CO_2} is the absorption rate of carbon dioxide, mol/m²s; S is the cross-sectional area of the laboratory absorber, m²; Δp_{CO_2} is a mean logarithmic value of CO_2 partial pressures before and after the absorber, Pa.

4   Results

4.1   Study of the Carbonation of Barium Sulfide Solution at a Carbonation Level of Less Than 50%

Noted in the literature review high absorption rate at a carbonation degree of less than 50% may indicate that the limiting stage is the diffusion of CO_2 from the gas to the interface. Confirmation of this fact would allow rejecting the further study of the carbonization process in this concentration range and using the relations known from the literature to calculate the corresponding equipment. Thus, the task of the studies on this phase was to find the limiting stage of the process. For this purpose, the effect of the carbon dioxide concentration in the gas, barium sulfide in the initial liquid, the gas velocity, the liquid irrigation density, and the temperature on the kinetics of carbon dioxide absorption was researched. The study was carried out in a laboratory apparatus with a dual flow tray set in it.

Mathematical processing of the experimental results by the method of regression analysis showed that a significant effect (the p-level coefficient estimated the significance) on the rate of CO_2 absorption is exerted by: the average partial pressure of carbon dioxide in the gas (the driving force of absorption), gas velocity and temperature. So, the following equation was obtained:

$$\ln r_{CO_2} = 17,8 - 2100T^{-1} + 0.94\ln v + 1.03\ln \Delta p_{CO_2} \tag{8}$$

where v is the gas velocity, m/s;

The accuracy of the obtained equation is characterized by the following values: determination coefficient $R^2 = 0.986$, standard error of equation $\ln r_{CO_2} - 0.073$.

As follows from the above experimental results, the barium sulfide concentration within the limits of the accuracy of the experiments does not affect the absorption rate. It can be explained by the fact that in the considered range of carbonization level, BaS concentration has little effect on the concentration of the hydrogen ion, which determines the rate of CO_2 absorption at high pH values.

The effect of temperature on the absorption rate was also insignificant. In total, this indicates that the resistance to the absorption process is due to diffusion, i.e., mass transfer, and a chemical reaction is not the limiting stage of the process.

From Eq. (8), in particular, it follows that the rate of CO_2 absorption is proportional to the driving force of the process, which makes it possible to calculate the kinetics of absorption using the classical two-film model.

Thus, the data obtained allow us to assert that when a barium sulfide solution absorbs carbon dioxide with a carbonization degree of less than 50%, mass transfer in the gas phase is the limiting stage.

4.2   Study of Barium Sulfide Carbonization at Carbonization Level More Than 50%

Barium sulfide solution with a carbonization level of more than 50% does not contain barium sulfide since the latter is wholly converted into barium hydrosulfide. Therefore, we will consider the carbonization of the Ba(HS)2 solution further.

At this stage of the work, the effect of CO_2 concentration in the gas, barium concentration in the initial liquid (in terms of sulphide), the gas velocity, the density of liquid irrigation and temperature, on the kinetics of carbon dioxide absorption was studied.

In experiments with a dual flow tray, the direct proportionality between the absorption rate and the mean logarithmic concentration of carbon dioxide in the gas was experimentally confirmed. This made it possible to further characterize the absorption kinetics in terms of the mass transfer coefficient.

In the same experiments, it was found that the rate of carbon dioxide absorption is lower than this indicator has at carbonation level less than 50%, and significantly. Since the conditions of all diffusion stages of the process did not change, such a significant decrease in the absorption rate can only be explained by changes in the mechanism of chemical reactions caused by the pH decrease of an absorbent. For example, the control over the rate of CO_2 binding is transferred from reaction (1) to reaction (2).

In experiments with the same contact element, the effect of barium concentration in the solution on the absorption rate was evaluated. Under the following conditions, the experiments were carried out: volume fraction of CO_2 in gas is 40%; gas velocity is 0.7 m/s; irrigation density is 0.015 m^3/m^2s; temperature is 50 °C. The mass fraction of barium in the absorbent in terms of BaS varied from 5 to 9%.

The analysis of the experimental results did not show significant differences in the value of the mass transfer coefficient about barium concentration in the solution. Based on it, with an accuracy admissible in engineering calculations, it can be assumed that the effect of Ba(HS)$_2$ concentration on the absorption rate can be neglected. This means that the process chemistry at the consideration stage of carbonization is determined by reaction (2), i.e., by CO_2 hydration. Since the water concentration in the solution varies insignificantly with a change in barium hydrosulfide concentration, the latter does not affect the carbonization rate.

The study of the effect of the other factors (gas velocity, irrigation density, and temperature) was combined with the data obtained to select an appropriate contact element for carbonization. The experiments were carried out under the following conditions: CO_2 volume fraction in gas is 40%; mass fraction of barium in terms of sulfide in solution is 7%. The parameters studied varied in the following ranges: gas velocity is from 0.2 to 1.2 m/s; irrigation density is from 0.004 to 0.04 m^3/m^2s; temperature is from 20 to 80 °C; overflow height is from 0 to 20 cm.

To facilitate the calculation of the absorber, the experimental data obtained have been summarized in the form of the following empirical equation for calculating the mass transfer coefficient

$$\frac{K_g}{K_{g,0}} = 10^{a_1 \frac{t}{t_0}} \left(a_2 + a_3 \frac{h}{h_0} \right) \left(\frac{v}{v_0} \right)^{a_4} \left(\frac{l}{l_0} \right)^{a_5} \tag{9}$$

where $a_1 \ldots a_5$ are the coefficients of the equation; h is overflow height, cm; $h_0 = 10$ cm, t is temperature, °C; $t_0 = 50$ °C, v is gas velocity, m/s; $v_0 = 0.7$ m/s, l is irrigation density, $m^3/m^2 s$; $l_0 = 0.015$ $m^3/m^2 s$; $K_{g,0}$ is the mass transfer coefficient of the corresponding tray at $h = h_0$, $t = t_0$, $v = v_0$ and $l = l_0$, mol/$m^2 s$ Pa.

The coefficients of Eq. (9), the characteristics of its accuracy, and the values of $K_{g,0}$ for each of the studied trays, calculated using the nonlinear evaluation program, are given in Table 1.

Table 1. Coefficients and the accuracy characteristics of Eq. (9).

Type of tray	Coefficients					$K_{g,0}$	Residual RMS	Coefficient of determinization
	a_1	a_2	a_3	a_4	a_5			
Dual flow	−0.042	1.12	0	0.72	0.58	4.8	0.178	0.935
Sieve	0.074	0.33	0.51	0.33	0.17	9.5	0.085	0.955
Cap	0.068	0.57	0.27	0.47	0.21	14.8	0.063	0.964

The value of coefficient a4 can estimate the effect of gas velocity on absorption kinetics. Thus, its highest value for a dual-flow tray indicates that with an increase in the gas velocity, the absorption rate increases to a greater extent than other tested contact devices. On cap and sieve trays, the increase in the mass transfer coefficient with an increase in the gas velocity is approximately the same. These features are explained by the fact that the height of the foam layer and, consequently, the interface on the dual-flow tray is related to the gas velocity to a much greater extent than in the overflow trays. In the latter, the height of the foam is mainly determined by the height of the overflow.

According to the value of coefficient a5, the irrigation density's effect on the mass transfer coefficient is less than the gas velocity. The most significant change in the mass transfer coefficient with an increase in the irrigation density is observed on the dual flow tray. As in the previous case, the relation of the height of the foam layer on the tray to the irrigation density.

Since the carbon dioxide absorption at carbonization level of the absorbent more than 50% is presumably controlled by the chemical reaction in the liquid, the estimation of the dependence of the rate of carbonization on temperature was based on the consideration of its influence on the coefficient of mass transfer in a liquid. According to the theory of absorption, in the case under consideration the mass transfer coefficient related to liquid (K_l) can be calculated using the experimentally found values of the mass transfer coefficient related to gas, using the ratio $K_l = H_{CO_2} K_g$ where H_{CO_2} is Henry's coefficient.

In Fig. 1, the graphs of the relations of K_l calculated by the method described above to the temperature at the gas velocity 0.7 m/s; irrigation density 0.015 m^3/m^{2}s, and overflow height 10 cm are shown.
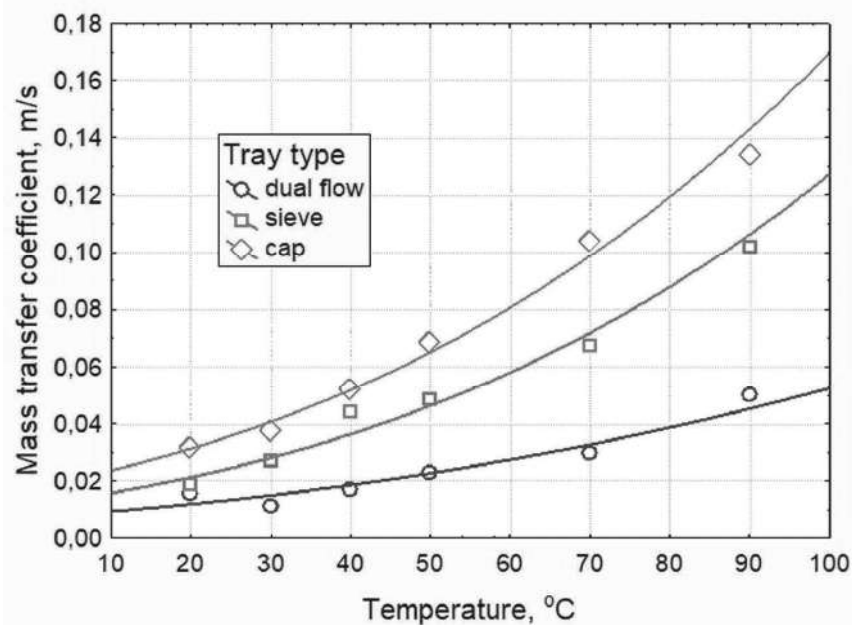

Fig. 1. The mass transfer coefficient refers to the liquid, to the temperature for the dual flow tray - 1, sieve tray - 2, and cap tray - 3.

Figure 1 demonstrates that the temperature dependence of the mass transfer coefficient related to the liquid has an exponential character. This form of dependence is more typical for the rate of a chemical reaction than for the diffusion coefficient.

The relation of the absorption kinetics to the overflow height is the most significant factor affecting the determination of the stage that limits the absorption rate. In the experiments, the overflow height varied from 0 to 20 cm with a step of 2 cm. We used an external overflow. Its height was not equal to the height of the foam. It characterizes the height of the layer of light liquid on the tray. The absorbent temperature was 50 °C, and the gas velocity and irrigation density were the same as the experiments evaluating the temperature effect.

As Eq. (9) shows, the mass transfer coefficient increases linearly with the overflow height. The kinetics of a chemical reaction is proportional to the volume of the liquid in which it flows. Therefore, the presence of a linear relation of the mass transfer coefficient on the overflow height and the effect of temperature on it proves the assumption that the process chemistry limits the absorption rate.

5   Conclusions

The article presents the results of an experimental study to obtain data on the kinetics of carbon dioxide absorption by a barium sulfide solution and the choice of contact elements type (trays) for the absorber design.

The literature review has shown that two stages are significantly different in pH in barium sulfide carbonization. The first occurs when less than 50% of BaS is converted to $BaCO_3$ and pH is more than 11, and the second – is when more than 50% of BaS is converted, and pH is from 7.5 to 9. It was also found that the kinetics of CO_2 hydration under the conditions of the first and second stages is controlled by different reactions. This made it possible to assume that the absorption rate of carbon dioxide at various stages will also differ.

Our study of carbonation kinetics confirmed this assumption. It was shown that only the partial pressure of CO2 in the gas, gas velocity, and temperature, has a significant effect on the absorption rate at the first stage. Any considerable effect of BaS concentration in the absorbent and the irrigation density was not found. The results obtained indicate that the absorption process is limited by resistance in the gas.

The carbonization rate at the second stage of the process, as experiments showed, is controlled by the kinetics of the chemical reaction of CO_2 hydration and is substantially lower than at the first stage of the process. The concentration of barium hydrosulfide (the reaction product at the first stage) in the absorbent has no significant effect on the kinetics of the process.

A further study of the BaS solution's carbonization was carried out to select an appropriate contact element for the equipment used in the process. Cap, sieve, and dual flow trays were tested, and the absorption rate of carbon dioxide was evaluated, characterized by the mass transfer coefficient.

As a result of an experimental study, equations were obtained for calculating the mass transfer coefficient about the temperature, gas velocity, and irrigation density for all types of trays mentioned.

Comparison of the obtained experimental data showed that the highest absorption intensity is achieved on the cap tray. The sieve plate turned out to be somewhat worse. On the dual flow tray, the absorption rate was the lowest. It was approximately two times lower than on a sieve tray and 2.5 times lower than on a cap tray. However, this does not characterize the dual flow tray as inappropriate for use in this process. The technical capabilities of conducting laboratory experiments did not allow us to achieve gas velocity in a laboratory absorber exceeding 1.2 m/s. At the same time, dual-flow trays in soda ash production operate at gas velocities of 2.5 m/s and higher. Extrapolating the relation of the mass transfer coefficient to the gas velocity, it can be shown that at high gas velocities, the indices of all the considered trays approach each other.

Thus, it can be concluded that all the trays studied are appropriate for carrying out the carbonization process of barium hydrosulfide solution. The selection of a specific type from considered contact elements for designing an absorption tower can be based primarily on design approaches that consider the designer's experience, the experience of operating particular devices, and the possibility of modifying the existing equipment for carrying out this process.

The work results were transferred to a specialized organization to design a pilot plant for BaS solution carbonization.

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