1. Introduction

1.1. The object of research

The object of research is to solve problems of environmental safety by increasing the period of operation of bicarbonate columns between washings. This is achieved by improving the design of the lower part of the bicarbonate column and changing the point of gas injection. The increase in the duration of the columns helps to reduce the amount of liquid waste from the production of soda and is a necessary condition for the greening of the process of obtaining purified sodium bicarbonate.

1.2. Problem description

As a rule, carrying out the processes of heat and mass transfer in the chemical industry is accompanied by significant material and energy costs [1]. Moreover, the effectiveness of these processes is determined by the presence or perfection of the contact devices used and the equipment itself.
as a whole. To date, the most widespread are devices with packed and poppet contact devices [2]. The type of contact device has the greatest influence on hydrodynamics, heat and mass transfer processes, as well as on the dimensions of the apparatus. Also, material and energy costs largely depend on the design of the contact device [3]. So, packed columns, in comparison with disc-shaped ones, have a lower hydraulic resistance per the height of the transfer unit. This is due to the fact that the trays operate in a flooded mode, and the gas passes through the column, overcomes a resistance equal to the pressure of the liquid column on all trays. In tray columns, the gas velocity is limited by an increase in the frequency of liquid removal, which, at high gas flow rates, significantly reduces the efficiency of mass transfer reactions [4]. However, in the production of purified sodium bicarbonate, carbonation of the soda-bicarbonate solution occurs in devices with disc contacts or without them at all.

In this case, the device is 70-80 % full. This is due to the fact that under the conditions of an intensive NaHCO₃ crystallization process, the use of packing will be accompanied by their clogging in a fairly short time. In addition, the use of nozzles excludes the processes of mixing the liquid and the removal of small crystals from the bottom of the columns to the top.

1.3. Suggested solution to the problem

Based on practical and scientific and technical data, the most optimal design of bicarbonate columns is apparatus with a small number of trays [5]. This design leads to a long run of the column due to the fact that in the zone of intense crystallization there are no trays that can get clogged, leading to the stoppage of the columns for washing [6].

So, the author [7] proposed a column design of a slightly different configuration (Fig. 1). Its feature is the presence of circulation and degassing circuits.

![Fig. 1. BIR column design](image)

As can be seen from Fig. 1, the contact elements of the column, in the capacity of which are the interlacing trays (Fig. 2), located only in its upper half.

The use of this type of trays is aimed at limiting back mixing and therefore increasing mass transfer between gas and liquid.

The critical operation in the process of obtaining purified sodium bicarbonate is the NaHCO₃ crystallization. The quality of this process, that is, its kinetics and the size of the crystals obtained, is largely influenced by the intensity of mixing. To obtain large crystals that lend themselves well to filtration, an important condition is to ensure longitudinal mixing of the liquid inside the column and the removal of small crystals of NaHCO₃ (the so-called “seed”) from the bottom of the column to the top to equalize the concentration of salts along the height of the column, also reduces the saturation value.
It is to ensure longitudinal mixing by the author [7] that the use of a circulation loop in the column was proposed.

The circulation loop creates swirling of the liquid flows in the lower part of the column in order to avoid sedimentation of the solid phase and its mixing in the total volume of the liquid. The liquid flowing through the circulation loops is degassed and the gas returns to the column under the first tray.

![Fig. 2. Fragment of a column with cross-precision trays: 1 – ring; 2 – tray; 3 – overflow](image)

This design is due to the fact that it is in the upper part of the column that intensive saturation of the sodium bicarbonate solution occurs. Also, this design prevents clogging of the sludge discharge unit. However, there is no data on the duration of the column flow between washings.

However, in bicarbonate columns, the presence of trays provides not contact of the liquid-gas phases, but uniform dispersion of the gas over the column cross section. Thus, the absence of trays in the lower and middle parts of the column causes a rather uneven distribution of the gas phase; at low gas loads, it significantly reduces the efficiency of the bicarbonate columns [6].

Apparatus with external energy input (apparatus with stirrers, rotary apparatus, apparatus with static mixing devices) have also been widely used in industry for carrying out mass transfer processes in liquid-gas-solid phase systems. In the processes of production of purified sodium bicarbonate, the intensity of stirring plays a very significant role: firstly, it causes the transfer of small crystals to the upper part of the column, where they play the role of crystallization centers; secondly, intensive stirring prevents the deposition of sodium bicarbonate both on the trays and in the bottom of the column [7, 8].

So, to obtain sodium bicarbonate crystals of good quality and sufficient size in bicarbonate columns, a sufficiently long residence time of the liquid in the apparatus is required. This leads to the large dimensions of the bicarbonate columns and, as a consequence, low velocities of the gas phase. The influence of the velocity on the hydrodynamics in the lower part of the column is especially noticeable, where the gas flow rate is also low due to the high pressure. This leads to an uneven distribution of gas over the cross section of the apparatus, poor contact of gas with liquid, rapid overgrowth of the unloading unit, which requires further flushing of the column.

Frequent flushing shutdowns of bicarbonate columns reduce the flow rates of the raw materials and the productivity of the column.

In the “classical” designs of bicarbonate columns, the suspension is removed, as a rule, through a choke in the bottom of the column, in fact, it is not very effective [7, 8]. At the bottom of the column there is a thickening of the suspension and deposits of bicarbonate. The presence of horizontal sections on the pipeline and shut-off equipment in this case leads to clogging and requires steaming of the pipelines.

The second disadvantage of the “classical” designs of bicarbonate columns is the uneven distribution of gas over the cross section, due to low gas loads, which greatly reduce the efficiency.
of the apparatus [9]. A more uniform distribution of gas over the cross section of the column increases the contact surface of the phases and intensifies the absorption of CO$_2$ in the lower part of the column [10]. This solution will allow:

– to increase the mileage of the column between cleanings;
– to increase its productivity by reducing the number of stops for flushing;
– to improve the absorption of CO$_2$ in the lower part of the column and to reduce the loss of;
– improve the quality of crystals.

The aim of this work is to study the influence of the structural characteristics of the lower part of bicarbonate columns on the duration of their operation.

2. Methodology for studying the influence of the design characteristics of the lower part of bicarbonate columns on the duration of the operation period in laboratory conditions

One of the most important parameters characterizing the operation of a bicarbonate (carbonation) column in the production of purified sodium bicarbonate is the length of the run between cleanings. The stoppage of the column for cleaning is caused by overgrowth, as a rule, from the bottom of the column. The reason for this is low gas velocities and uneven gas distribution over the column cross section. So, it was found that the gas flow, leaving from under the distribution cone, is directed upwards and towards the center of the column. As a result, the liquid flow and suspension at the walls during the process are rather inactive and difficult to mix.

To solve the established problem according to the principle of the geometric similarity of the apparatuses, an installation was developed, which emitted the lower section of the bicarbonate column. In the course of the experiments, the design of the installation was modernized and changed. Fig. 3 shows the first sample of the unit with a distribution cone.

![Fig. 3. Laboratory unit for studying the structural and hydrodynamic features of the operation of bicarbonate columns (sample No. 1): 1 – distribution cone; 2 – redistribution baffle](image)

The unit was a structure with a frame made of wood and a transparent part made of polycarbonate. The dimensions of the installation were 1000 mm in width and 1800 mm in
height. In the lower part of the installation, a distribution cone (1) was simulated. A redistribution baffle (2) was installed on top of the cone for a more uniform distribution of gas over the cross section (which was absent on the bicarbonate columns that were in operation at the factories).

Air was supplied to the unit using a household vacuum cleaner. The gas flow rate was controlled using a rotameter. From above, purified sodium bicarbonate was poured into the unit, with a total weight of 10 kg (based on the volume of liquid in the installation). The method of this study was based on the method of visual observation. Thus, the intensity of stirring the suspension and the rate of its sedimentation were analyzed.

3. Results of the study of the influence of the design characteristics of the lower part of bicarbonate columns on the duration of the operation period in laboratory conditions

The purpose of laboratory research was to identify the optimal design of the bicarbonate column suspension unloading unit.

So, during the unit operation (sample No. 1), shown in Fig. 1, almost instant precipitation of sodium bicarbonate was found (Fig. 4).

![Fig. 4. The result of an experimental study in a laboratory unit (sample No. 1)](image)

It was found that the presence of a redistribution baffle (cone) provides a fairly good turbulization of the liquid (Fig. 4), but its presence did not eliminate the uneven distribution of gas over the distribution cone, and as a result, there are sodium bicarbonate deposits.

To solve this problem, the laboratory unit was redesigned: it was decided to dismantle the distribution cone, move the gas supply unit to the center (Fig. 5).

As a result of gas supply in the center of the model under the redistribution cone, the picture of the gas-liquid flow under the cone has changed significantly. Gas (air) that rises in the center captures the slurry flow, forming a circulation loop under the cone (Fig. 6). The liquid (suspension), rising in the center, descends under the walls.

As a result of the research, it was revealed that the second circulation loop is formed above the redistribution cone. The gas flow coming out from under the cone captures the suspension upward, and is less gassed and, therefore, the heavier suspension in the center and at the walls of the apparatus goes down.

Thus, it was experimentally proved that the circulation loop, due to the presence of a redistribution cone and the supply of gas to the center of the apparatus, prevent the precipitation of sodium bicarbonate, and therefore the period of operation of the bicarbonate column without stopping for washing can be extended. However, this method of supplying gas can lead to its slipping into the sludge discharge pipe. Therefore, under the created conditions, it will be most favorable to remove the suspension from the space above the redistribution cone.
4. Results of the study of the influence of the design characteristics of the lower part of bicarbonate columns on the duration of the operation period in industrial conditions

Initially, to solve the problem with clogging of the bottom of the column, it was impossible for the bicarbonate slurry to exit from the column, a fairly simple solution was proposed that did not require significant reconstruction of the bicarbonate column and significant capital investments. The proposal was to lead the discharge pipe inside the distribution cone (Fig. 7).
Fig. 7. The proposed scheme for the design of the suspension discharge pipe

However, it should be noted that the proposed measures did not lead to a significant positive effect. So, after a certain time of operation of the columns, an increase in the hydraulic resistance of the column was observed, as a result, as it was found, clogging of the lower part, which made it impossible for gas to pass, as evidenced by Fig. 8.

Fig. 8. Photo of the state of the lower part of the bicarbonate column after the implementation of the recommendations at one of the soda plants

Based on the results of laboratory studies, it was recommended to reconstruct the lower part of the bicarbonate column A and change the number of contact elements represented by mesh trays. As a rule, three zones can be conventionally distinguished in terms of height in a column:
- zone of addition of soda solution according to NaHCO₃ (top of the apparatus)
- zone of the beginning of crystallization of NaHCO₃ (middle part)
- crystal growth zone (bottom of the column).

With the complete absence of contact elements in the column, longitudinal mixing of the liquid takes place, which reduces the efficiency of the carbonization process. For example, it is undesirable for small crystals to enter the bottom of the column, which will complicate the discharge
of the sediment. Carrying out small crystals from the middle to the upper zone is also undesirable under certain conditions, since this leads to their dissolution, which in turn contributes to a decrease in crystallization centers.

The results of the study in an industrial environment are presented in Table 1.

<table>
<thead>
<tr>
<th>Number of trays in the column, pcs.</th>
<th>No. of tray</th>
<th>The duration of the column operation, days</th>
</tr>
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<tbody>
<tr>
<td>Before reconstruction</td>
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<td></td>
</tr>
<tr>
<td>7</td>
<td>1–7</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>1–6</td>
<td>7–8</td>
</tr>
<tr>
<td>5</td>
<td>2–6</td>
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<td>2.5.6</td>
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<td>1.5.6</td>
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<td>2</td>
<td>2–6</td>
<td>9–10</td>
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<tr>
<td>after reconstruction</td>
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<tr>
<td>2</td>
<td>3.6</td>
<td>17</td>
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<tr>
<td>2</td>
<td>1.5</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>2.6</td>
<td>16–18</td>
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</tbody>
</table>

Note: tray numbering starts at the bottom; the distance between the plates is 3500 mm

Based on the production data presented in Table 1, it was found that the most optimal configuration of the arrangement of the trays in the column was the latter (with trays # 2 and # 6).

5. Discussion

As a result of the modernization of the design of the lower part of the industrial bicarbonate column, it was possible to increase the duration of its operation between stops for washing by 1.5–2 times, and to reduce the formation of liquid waste (Table 1). In addition, the optimal number and arrangement of contact elements (mesh trays) in the column was determined.

It should be noted that to date there are no data on similar studies aimed at optimizing the design characteristics of bicarbonate columns.

Among the limitations of research, it should be noted that it is impossible to reproduce the laboratory model in real dimensions, which could affect the error of the experiments. However, industrial studies have confirmed the effectiveness of the proposed design solutions and confirmed the hypothesis established during laboratory experiments.

Among the prospects for further research is improving the accuracy of the theoretical study of the problem, which was solved, as well as modeling the hydrodynamic conditions of the operation of bicarbonate columns and finding ways to further improve, ecologize and optimize the process.

6. Conclusions

1. It was found that the circulation loop and gas supply to the center of the apparatus prevent the precipitation of sodium bicarbonate, and therefore increase the duration of the bicarbonate column operation without stopping for washing. Such optimization of the design features of carbonization columns for the production of purified sodium bicarbonate is of significant importance both for the technical and economic indicators of soda plants and for the environment. After all, increasing the flow of the columns between flushing stops significantly reduces the formation of liquid waste.

2. On the basis of the performed research, the optimal design of the unit for unloading the sludge of bicarbonate columns for the production of purified sodium bicarbonate was developed, as a result, it was confirmed in industrial conditions.
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References


