Iodine Ionic-Molecular Flotation in a Lab-made Device
Installation and Preliminary Tests

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Separation by ion flotation was rarely approached, mainly for recovering the heavy metal cations. Installation simplicity as well as low power consumption needed in order to achieve the desired separation degree are the main advantages of the method, and these considerations lead to a relatively low total cost of implementation for such systems. This paper presents a study for iodine regenerative separation by ionic flotation, in a lab-made device, from dilute aqueous synthetic solutions (20-120 ppm), which properly simulates the sea, probe or mine water. The study of iodine concentration by flotation accompanied by iodide ion oxidation to molecular iodine aimed to determine the optimal oxidant amount, Cl2 and H2O2 revealed that the best results are obtained when the amount of oxidant has concentrations similar to the iodide ion in the solution subject for processing.

Keywords: ionic-molecular flotation, flotation device, iodide separation, oxidized species separation

Sea, probe and/or mine waters, have a complex composition containing a great variety of components as soluble and insoluble ionic, molecular and colloidal species with variable concentrations, which are slowing the separation, removal or recovery [1-4]. Such aqueous systems require the application of available separation methods that allow the separation and/or recovery of useful components [5-7]. Among these methods, extensively have been studied: ion exchange [8,9], adsorption [10,11], reverse osmosis, ultrafiltration [12-15] and liquid membranes [16,17].

Separation by ion flotation was rarely approached, being applied mainly for recovering the heavy metal cations [18-20]. This paper presents a study for iodine regenerative separation by ionic flotation, in a lab-made device, from dilute aqueous synthetic solutions (20-120 ppm I-), which properly simulates the sea, probe or mine water.

The aim is to achieve regenerative separation of iodine as iodide ion (I-) or tri-iodide (I3-) from aqueous solutions using ionic flotation as separation method using as collectors cationic surfactants and as coligands alkyl normal alcohols and polyethylene glycols.

**Experimental part**
The flotation installation description and operation
Basics flotation installation construction

Plant simplicity as well as low power consumption needed in order to achieve the desired separation degree are the main advantages of the method, and these considerations lead to a relatively low total cost of implementation for such systems [21-30].

In the construction of such installation could be used both steel and plastics. Because using steel would greatly increase the cost of implementation it was chosen plastic material, namely polymethylmethacrylate - organic glass (table 1). This transparent plastic material with good mechanical and chemical strengths was widely used in various fields. The table 1 shows the most important properties of the material under consideration.

<table>
<thead>
<tr>
<th>Color</th>
<th>transparent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow resistance</td>
<td>min. 45 MPa</td>
</tr>
<tr>
<td>Elasticity module</td>
<td>min. 1380 MPa</td>
</tr>
<tr>
<td>Workability</td>
<td>good</td>
</tr>
<tr>
<td>Resistance to chemical agents</td>
<td>yes</td>
</tr>
<tr>
<td>Resistance to weather</td>
<td>yes</td>
</tr>
<tr>
<td>Good usage in the range</td>
<td>-30° - +90° C</td>
</tr>
<tr>
<td>Hardness</td>
<td>100-170 MPa</td>
</tr>
<tr>
<td>Optical properties</td>
<td>very good</td>
</tr>
<tr>
<td>Density</td>
<td>1.2 kg/dm³</td>
</tr>
<tr>
<td>Electrical isolator</td>
<td>even in wet conditions</td>
</tr>
<tr>
<td>Moisture absorption</td>
<td>reduced</td>
</tr>
<tr>
<td>Dimensional stability</td>
<td>yes</td>
</tr>
<tr>
<td>Glass transition temperature</td>
<td>105° C</td>
</tr>
<tr>
<td>Decomposition temperature</td>
<td>300° C</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>55-75 MPa</td>
</tr>
<tr>
<td>Elongation at break</td>
<td>2-7%</td>
</tr>
</tbody>
</table>

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Table 1
MAIN CHARACTERISTICS OF POLYMETHYL-METHACRYLATE
The easiest flotation installation may consist of:
- flotation column,
- column feeding tank,
- vessel for collecting foam.

In addition to these main components we can also find:
- dosing pump of the solution in the column,
- compressor, connected to the column air distributor,
- adjuster for fluid level in the column.

For this analysis we consider a maximum volume of 2.5 L that can be subject to the separation.

Due to the presence of foam, the total volume of the column will be more than 2.5 L and at sizing the column this aspect will be taken into account.

Thus, at an inner diameter of 84 mm and a 4 L capacity of the column, will result a height of 720 mm. For column feeding tank, a 2.5 L capacity can be considered, and in the case of the vessel for collecting foam, the capacity will be double, 5 L, this being necessary in order to collect the foam bubbles, which will be broken by various methods.

Table 2 presents the main quotas for the installation components.

The figure 1 shows a simplified diagram of the flotation installation.

Flotation installation construction stages

The separation column construction

The first step in the construction of such installation, consists in choosing the appropriate material for the separation type we want to achieve, as well as processing it in order to obtain the desired column size.

Choosing as raw material (see the properties from table 1), polymethylmethacrylate - transparent pipe, a pipe of 2000 mm length (purchased from Mons Medius), inner diameter of 84 mm and outer diameter of 90 mm, mode form.

The maximum capacity of the column will be 4 L, resulting 720 mm height of the column. After cutting the desired height of the column, the cylinder was processing. This will require an entry for liquid supply from the tank, an exit which will connect the column with the liquid adjuster and second exit toward the foam collecting vessel (at the top of the column) – mounting three couplings for corresponding tube connecting, and threads at both ends of the cylinder for the two caps. The figure 2 presents the separation column scheme.

For processing the cylinder, the two column caps are manufactured, the upper one made of polystyrene and the lower one of high density polyethylene. The upper cap made of polystyrene transparent material, which will allow viewing the top of the column and the lower cap made of high-density polyethylene material that can be easily processed in order to connect the compressor to the air distributor.

For coupling the compressor to column air distributor in the lower cap, connecting and cuping pipes will be mounted. Compulsory, on the path from the compressor to the column entry it is required to mount a tap.

At the lower cap it will be also mounted a coupling with a check valve that allows the injection of surfactant at the appropriate moment.

Before the final installation of the column the air distributor was chosen. For an effective mass transfer, the air nozzles must have a diameter as small as possible 0.1 – 0.3 mm. Initially at the beginning, a distributor of metal material was tested. However, due to the nozzles sizes it was placed a distributor with polypropylene frame and nozzles diameter of 0.2 mm on steel sieve support. Since the distributor diameter was larger than the diameter of the nozzle mounted in the cap, in order to make the connection between these components a polypropylene reduction was used.

### Table 2

<table>
<thead>
<tr>
<th>Component</th>
<th>Height (mm)</th>
<th>Inner diameter (mm)</th>
<th>Wall thickness (mm)</th>
<th>Tolerance (+/- mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Separation column</td>
<td>720</td>
<td>84</td>
<td>3</td>
<td>0.2</td>
</tr>
<tr>
<td>Column feeding tank</td>
<td>270</td>
<td>-</td>
<td>3</td>
<td>-</td>
</tr>
<tr>
<td>Vessel for collecting foam</td>
<td>345</td>
<td>-</td>
<td>3</td>
<td>-</td>
</tr>
</tbody>
</table>

Fig. 1. The simplified diagram of the flotation installation

Fig. 2. The separation column scheme
After choosing the distributor, this was mounted in the lower cap.

The choice of the containers
For the two containers of the installation, column feed tank (capacity 2.5 L) and foam collecting vessel (capacity 5 L) were chosen two glass containers with glass stopper and tap purchased from MultiLab Bucharest. The table 3 presents the recipients model and their characteristics.

The choice of pump and compressor
For mixture dosing into the column it was used a MTA Kutesz 5187A peristaltic pump model shown in the figure 8, with high dosing precision and low flow.
A 100 L capacity vessel was chosen for air circulation to the distributor (Bamax compressor).

Assembly of flotation installation
For mounting the three recipients of the installation - the flotation column, column supply tank and foam collecting vessel, a metal frame from copper tube was made on which those recipients were fixed by means of polyamide clamps able to support the components.

The metal frame was fixed on a wooden board, fitted with wheels for an easier handling. After fixing the recipients, column level adjustor, peristaltic pump and compressor will be also fixed.

The connection between the tank and the column was made with a polyethylene hose which passed through the peristaltic pump and follows the shape of the metal frame, by help of polyamide straps.

The connection between the distributor and the compressor was made with pressure-resistant hose, on the route which compulsion mounted a tap. At this point, it was checked that the control valve mounted on the lower cap was closed. The column liquid adjustor used during the operation bringing the column liquid level at the desired level.

Operation and operating parameters
After loading the tank with sample solution the peristaltic pump could be started and set-up at the desired flow rate. After reaching the required liquid level in the column (at least until a complete cover of the air distributor) the air compressor will be started. The surfactant injection will be done acting the control valve and injecting the solution through it, followed by its rapid closure. Then the tap situated on the air distributor route opens. The air bubbles released generates foam which climbs up in the column and will be collected in the foam vessel, subsequently broken by different methods.

The separation can be performed and analyzed varying various parameters: a) feed rate, set by operating the peristaltic pump control button; b) volume; c)surfactant type and concentration; d) partial blocking of the distributor one or two clamps could be used to section the propylene tube; e) sampling time from the foam collecting vessel.

Preliminary tests for molecular iodine flotation in the ionic-molecular-flotation installation
The experimental work for molecular iodine concentration involves iodide ion oxidation from feed solution [28-31].

The purpose of this operation is to increase the iodine separation efficiency from poor sources, decreasing the reagent consumption and simplification the iodine recovery from foam.

In order to determine iodine separation efficiency from feed solution we started from the previous results [31-35], which allowed establishing the following parameters
- feed solution concentration 20-100 ppm iodine;
- surfactant concentration, octadecyl pyridinium, (ODP), 10^{-4} M;
- coligand PEG 4000, 10^{-5} M;
- air flow through the column 0.8 L/min;
- amount of solution processed 1000 mL.

The study of iodine concentration by flotation accompanied by iodide ion oxidation to molecular iodine aimed to determine the optimal oxidant amount, Cl_{2} and H_{2}O_{2} (fig. 3 -5).

The separation efficiency of the iodine from feeding solutions with 20, 60 and 100 ppm iodide depended on the amount of oxidant added in the flotation column.

The best results were obtained when the amount of oxidant had concentrations similar to the iodide ion in solution subject to processing.

The behavior of the two oxidants is different, especially, after reaching the maximum efficiency.

<table>
<thead>
<tr>
<th>Recipient</th>
<th>Capacity (mL)</th>
<th>Spout diameter (mm)</th>
<th>Outlet (mm)</th>
<th>Height (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Supply tank</td>
<td>2500</td>
<td>48</td>
<td>30</td>
<td>270</td>
</tr>
<tr>
<td>Collecting vessel</td>
<td>5000</td>
<td>58</td>
<td>32</td>
<td>345</td>
</tr>
</tbody>
</table>

Table 3
RECIPIENTS MODEL AND THEIR CHARACTERISTICS

Fig. 3. The iodine separation efficiency variation according to oxidant concentration for a feeding solution with 20 ppm iodine

![Fig. 3](image)

Fig. 4. The iodine separation efficiency variation according to oxidant concentration for a feeding solution with 60 ppm iodine

![Fig. 4](image)
The addition of hydrogen peroxide in excess was unfavorable for separation while it was less affected by gaseous chlorine excess.

We recommend the use of gaseous chlorine as oxidizing agent for iodine separation from poor sources by flotation. Such oxidizing agent is also easily dispensed and handled which is another advantage for its use.

Conclusions

The study of iodine recuperative separation by means of ionic flotation is based on three arguments: reduced consumption of materials, reduced investment and successful separation process applied for dilute solutions. The working installation is relatively simple, versatile and does not require special precautions because the gas (air) introduction system consists of a compressor connected to the system lens.

The performed study on the iodine concentration by ionic flotation accompanied by iodide ion oxidation to molecular iodine and the determination of the optimal oxidant amount, Cl₂ and H₂O₂, revealed that the best results were obtained when the amount of oxidant had concentrations similar to the iodide ion in solution subject to processing.

The behaviour of the two oxidants was different, if the addition of hydrogen peroxide in excess was unfavorable for separation or by contrary the gaseous chlorine excess did not affect the separation process.

It is recommended to use as oxidizing agent the gaseous chlorine for iodine separation from poor sources by ionic flotation.

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References