

Short Communication

Mini ozonizer for instant production of ozone required for oxidation reactions and its application in the foam flotation–separation technique

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Abstract

The fabrication of a mini ozonizer and its application in the foam flotation–separation technique are described. The system rapidly generates ozone for instant use in small-scale oxidation processes. Its application to the separation of Ce(III) from an alkaline solution is demonstrated.

Keywords: Ozone generation; Oxidation reactions; Foam flotation–separation; Cerium

The use of air oxidation is well established for a number of chemical reactions. Such reactions require a strong stream of air or oxygen and the rate of oxidation can be increased by increasing the flow of air. However, this is possible only up to a limited extent. One way to overcome this limitation could be the use of ozonated air instead of air alone. In this work, a device that can produce ozonated air for instant use in such reactions is

described. Different types of ozonizers have been reported previously [1] but they are usually expensive, complicated to fabricate and difficult to handle. Oxidation reactions are an important part of certain analytical and separation procedures. Foam flotation–separation is a technique in which ozonated air can be used for foam formation and to oxidize the metal ions to be separated to higher valence states. This technique has been used for

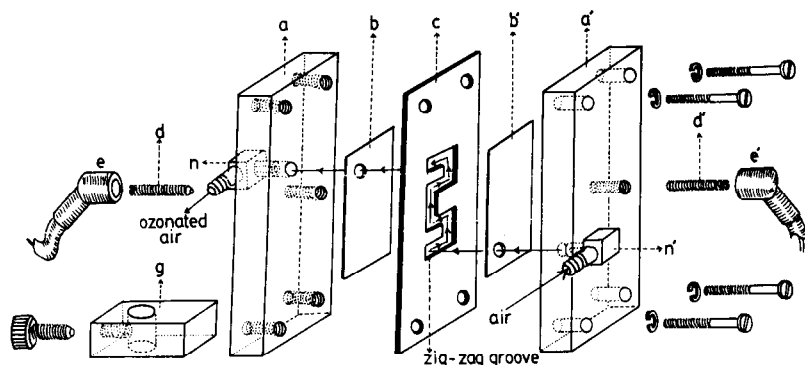


Fig. 1. Exploded view of the mini ozonizer.

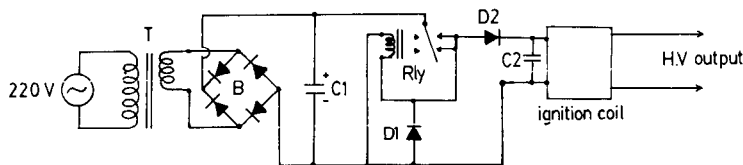


Fig. 2. High-voltage supply. B = Bridge rectifier; C1 = 330 μ F/25 V, C2 = 200 nF/100 V; D1 = 1N4001; D2 = 1N5400; Rly = 6 V four-point relay; T = 6 V/1.5 A.

many years for the separation of a number of materials from their solutions, especially from waste waters. A recent review on foam separation [2] describes the importance of this technique and some versatile applications.

The use of the simple and rapid mini ozonizer developed in this work is illustrated by the separation of Ce(III) as Ce(IV) hydroxide by the foam flotation technique.

Experimental

Construction of the ozonizer. A schematic diagram of the ozonizer is shown in Fig. 1. It consists of a central $12 \times 6 \times 0.6$ cm PTFE sheet (c) with a 0.6 cm wide zig-zag groove. This PTFE sheet is sandwiched between two $6 \times 4 \times 0.1$ cm stainless-steel plate-electrodes (b and b'). Each electrode is etched on the side facing the PTFE sheet (c). The etching was carried out, using a lead cathode as counter electrode, in 14 M sulphuric acid (500 ml) containing chromic oxide (4.0 g). The other side was protected by painting it with a polyvinyl emulsion. The d.c. supply used was 12

V, 40 A for 7 min. This procedure produced a fine grain on the surface required for a multi-discharge action. The sheets b, c and b' are further sandwiched by two $12 \times 6 \times 1.2$ cm Perspex sheets (a and a') with the help of screws. The sheets a and a' are provided with two nozzles (n and n') as inlet and outlet for air and ozonated air. A clamping facility is affixed with the sheet a.

The air enters the zig-zag groove through one nozzle, via the path shown in Fig. 1, where it is converted to ozone as a result of an electric discharge between the electrodes when the supply is on. The ozonated air thus produced leaves through the other nozzle, which is connected to the flotation column. Sheets a and a' are provided with studs d and d' and probes e and e' for connection to the high tension.

The high-tension supply circuit is shown in Fig. 2. The ignition coil switching mechanism for the production of high tension is similar to that of a common electric bell. An electric discharge of about 5 kV is obtained, which is suitable for ozone formation. Maximum efficiency of the ozonizer

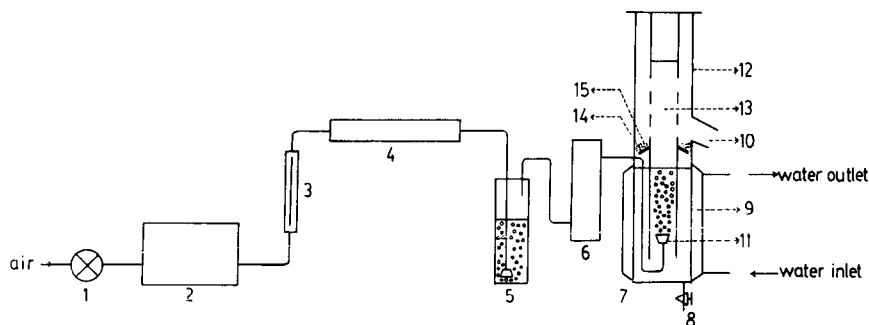


Fig. 3. Schematic diagram of flotation system. 1, Air filter; 2, aquarium air pump; 3, flow meter; 4, ascarite column; 5, humidifier; 6, mini ozonizer; 7, flotation assembly; 8, drain; 9, thermostated water jacket (14 cm \times 6.3 cm i.d.); 10, foam discharge port; 11, fritted glass sparger; 12, container column; 13, main flotation column; 14, perforated glass collar; 15, glass-wool bed.

was achieved by passing dry air at a flow-rate of 18 ml min^{-1} . The efficiency was measured by carrying out iodimetric titrations of ozone [3].

Flotation-separation experiment. A schematic diagram of the experimental set-up is shown in Fig. 3. The flotation assembly consists of two columns, the main column ($48 \times 3.2 \text{ cm i.d.}$) and the container column ($51 \times 5.2 \text{ cm i.d.}$). The lower portion of the container column is provided with a jacket (14 cm) for circulation of thermostated water. This assembly is connected to the ozonizer via a glass tube as shown in Fig. 3. The open end of the glass tube is provided with a fritted glass sparger. The test solution (about 250 ml) is transferred to the container column. Air is pumped through a filter at a flow-rate of 20 ml min^{-1} to an ascarite column to remove acidic gases, especially carbon dioxide, after which it passes through a humidifier and enters the ozonizer. The ozonated air formed is led into the flotation column.

The test solution used in this study was prepared from analytical-reagent grade chemicals. A solution containing $200 \mu\text{g ml}^{-1}$ cerium(III) nitrate and a solution containing $300 \mu\text{g ml}^{-1}$ sodium laurate were prepared in doubly distilled, de-ionized water. The two solutions (120 ml of each) were mixed and the pH of the mixture was adjusted to 9 with 0.5 M NaOH. ^{144}Ce (Amersham) was added to the above solution as a tracer. Test samples (1 ml each) were taken from the drain port after an interval of 10 min and counted for radioactivity using a Ludlum Model 261 single-channel analyser with a Model 243 well-type detector.

Results and discussion

Performance of the ozonizer. From the iodimetric titration curve (Fig. 4) of the ozonated air produced by the ozonizer it was found that the maximum conversion was obtained at a flow-rate of 18 ml min^{-1} , and for safety a flow-rate of 20 ml min^{-1} was adopted in the separation study. The rate of ozone generation decreased after extended use of the electrodes. A similar effect was observed with an increased moisture content in air. The efficiency can be restored by cleaning and drying the electrodes.

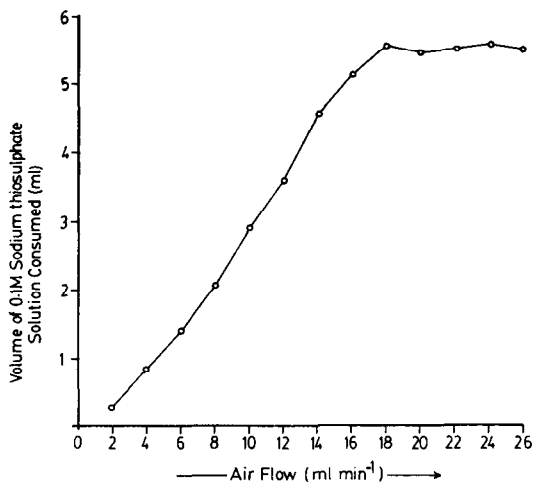


Fig. 4. Iodimetric titration curve.

Separation of cerium. Optimization of experimental conditions was achieved by studying the effect of pH (Fig. 5) and temperature (Table 1) on the recovery of Ce(III) from aqueous solution. This indicated a pH of 9 and a temperature of 40°C to be optimum. For the separation of $100 \mu\text{g}$ of Ce(III) in 1 l of solution no interference was caused by 20 mg l^{-1} of Na^+ , K^+ , Ca^{2+} , Mg^{2+} , Cl^- , NO_3^- , SO_4^{2-} ; 10 mg l^{-1} of Eu^{3+} , Gd^{3+} , Yb^{3+} ; 2 mg l^{-1} of Sr^{2+} ; 0.1 mg of Fe^{3+} , Mn^{2+} , Al^{3+} .

A substantial removal of Ce(III) by precipitation as cerium(IV) hydroxide followed by flotation with sodium laurate was obtained by the use of ozonated air. The recovery was $> 94\%$ compared with about 50% with the use of normal air at

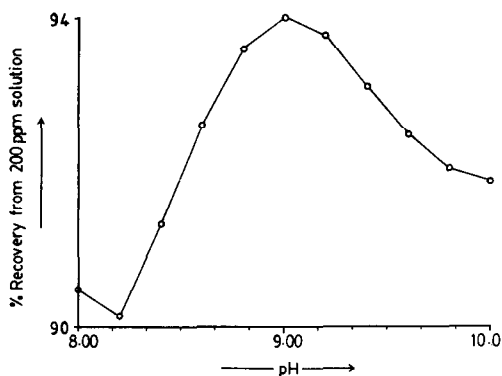


Fig. 5. Effect of pH on recovery. $\text{ppm} = \mu\text{g ml}^{-1}$.

TABLE 1

Effect of temperature on the recovery of Ce(III) by the use of ozonated air at pH 9

Temperature (°C)	Recovery (%)	Temperature (°C)	Recovery (%)
25	90.4	40	94.2
30	91.1	45	94.0
35	92.5	50	92.3

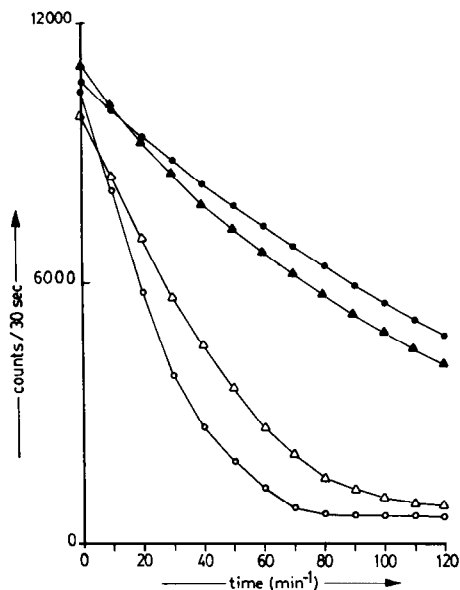


Fig. 6. Separation of cerium. ○, Ozonated air at 40°C; ●, normal air at 40°C; △, ozonated air at 25°C; ▲, normal air at 25°C.

40°C after 120 min (Fig. 6). The procedure appears to be useful for analytical purposes without introducing the risk of contamination. In addition, the flotation technique associated with ozonizer can be employed in industrial processes for the recovery of cerium and other materials.

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