GREENING AN EXPERIMENT FOR ENVIRONMENTAL CHEMISTRY: H₂S CAPTURE WITH ELEMENTAL RECOVERY

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ABSTRACT

An experiment is presented to demonstrate the production, capture, and electrochemical remediation of H_2S in a closed syringe-based system. Final products are its pure constituent elements. This experiment is suitable for an environmental chemistry laboratory and it can be completed in 3-4 h. [African Journal of Chemical Education—AJCE 9(3), November 2019]

INTRODUCTION

There are very few processes in which a pollutant is captured and transformed into its pure, constitutive elements. One such a case involves H_2S , a dreadful highly toxic, corrosive, and pestilent gas that brings about childhood memories of rotten eggs. A member of the so-called reduced sulfur gases [1], its decomposition can be achieved by electrochemical, photochemical, plasma, solar, and microwave methods [2]. Among these, its electrochemical remediation - the object of the present experiment - has been reviewed elsewhere [3]. This normally involves its reactive absorption and S²⁻ oxidation [4], followed by the oxidant's regeneration. Oxidants tested include Fe(III) [5] and I_3^- [6]. Fe(III) can oxidize S²⁻ either alone or as a Fe(III) chelate [1,7,8]. Another electrochemical alternative involves S²⁻ oxidation to SO₄²⁻ [9].

We developed an earlier version of an experiment to demonstrate H₂S capture by its reaction with I₃⁻ as the oxidizer, whereby colloidal S is produced [6]. The resulting Γ ions are then sent to the anolyte of an electrochemical cell to achieve triiodide ion regeneration, while hydrogen gas is produced at the cathode. The global result is the decomposition of H₂S into its elements in a very pure form, i.e., H₂ + S that represents a splendid resource recovery [8]. However, this experiment suffered from a major drawback: it was designed to be performed in a beaker, with the concomitant need for a fume hood due to the toxic nature of the pollutant. In the present experiment we substantially decreased this requirement by performing all the necessary steps in closed plastic syringes with the aid of three-way stopcock valves.



Figure 1. Three-way stopcock valve.

EXPERIMENTAL

Materials required

Three 10-mL syringes, three 5-mL syringes, seven three-way stopcock valves, one Millipore filter (45 microns or similar), one universal stand, one 3-finger clamp, one 9-V battery, one set of alligator clips, two unsharpened syringe needles, 10-cm silicone hose, 1 small plastic cup, powdered pyrite, 10-mL of a 0.25 M KI, 0.05 M I₂, and 0.05 M HCl solution (called *triiodide solution*), 5-mL of 6 M HCl.



Figure 2. Experimental set-up. 1) Ground pyrite storage, 2) HCl solution, 3) H₂S collection and storage, 4) I_3^- solution, 5) Millipore filter, 6) electrochemical cell, and 7) regenerated I_3^- solution.

Procedure

Put together a set of syringes according to the diagram shown in Figure 2. Place 1 mL of 6 M HCl in a 5-mL syringe. Place 0.3 mg of powdered pyrite in a small plastic cap. Fit this cap into a 10-mL syringe. Place 3 mL of the triiodide solution in a 5-mL syringe. Connect two needles to a 10-mL syringe; these will work as electrodes. Gradually add the HCl solution to the syringe containing pyrite by opening the corresponding three-way stopcock valve. H₂S begins to form. Transfer the H₂S gas produced into an empty syringe. Pay attention to the timely opening and closing of the keys before

and after use. Convey the gas into the syringe containing the triiodide solution. This solution begins to change color from yellow to milky white (see Figure 3).



Figure 3. A close-up of the oxidation cell.

When the solution is essentially whitish, pass it through the filter into the electrochemical cell (i.e., the syringe that has the needles connected). Connect two alligator clips to a 9-volt battery and to the needles (see Figure 4). Allow the electrolysis to occur until the color of the solution changes from clear transparent to dark brown (see Figure 5). Disconnect the battery. The brown solution contains the regenerated iodine (as triiodide ions). Transfer this solution to a 5-mL syringe. The system is now ready for a new cycle.



Figure 4. A close-up of the electrochemical cell.



Figure 5. Oxidant regeneration inside the electrochemical cell.

RESULTS AND CONCLUSION

The schematic reactions are the following. Notes: All species are assumed aqueous, unless otherwise specified; also, the sulfur produced may result in different forms.

a)	In solution:	
H ₂ S(g)	$+ I_3^- \rightarrow S(s) + 3I^- + 2H^+$	(1)
b)	At the anode:	
$3I^{-} \rightarrow I$	$a_3 + 2e^{-1}$	(2)
c)	At the cathode:	
$2H^{+} + 2$	$2e^{-} \rightarrow H_2(g)$	(3)
d)	Global:	
$H_2S(g)$	\to H ₂ (g) + S(s)	(4)

Therefore, H₂ and colloidal sulfur are produced and can be tested in traditional ways. The electrochemical route to pollutant remediation is an exciting alternative for the treatment of a variety of pollutants.

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