**Experiment 2: Effectiveness of SO₂ Scrubbers**

CH 3600 / ESP 5090: Environmental Chemistry, Plymouth State University


**Introduction:**

Energy production is a major issue in the United States and around the world. In the United States, power generated from coal-fired power supplies nearly 50% of the US power needs—with a majority of these plants located in the northeast and near-midwest.

Generation of power by burning coal has several advantages: coal is relatively inexpensive, quite plentiful and has a high energy content, supplying 20-30 kJ/g of coal burned. However, the environmental impact of burning coal is significant. Due to its complex composition (approximated \( \text{C}_{135}\text{H}_{96}\text{O}_{9}\text{NS} \)), high temperatures, and incomplete oxidation, combustion of coal results in several undesirable by-products: particulate matter (PM), nitrogen oxides (NO\(_x\)) and SO₂.

Once in the atmosphere, these air pollutants have serious ramifications, and research shows that the acidity of precipitation has greatly increased in regions relying heavily on coal for power production. The SO₂ (and NO\(_x\)) gases leaving the smoke stacks at power generation stations react with the water in the atmosphere to create “acid rain”:

\[
\begin{align*}
\text{SO}_2(g) + \text{H}_2\text{O}(l) & \rightarrow \text{H}_2\text{SO}_3(aq) \quad \text{(sulphurous acid)} \\
\text{SO}_2(g) + \text{H}_2\text{O}(l) + \frac{1}{2}\text{O}_2(g) & \rightarrow \text{H}_2\text{SO}_4(aq) \quad \text{(sulphuric acid)} \\
4\text{NO}_2(g) + 2\text{H}_2\text{O}(l) + \text{O}_2(g) & \rightarrow 4\text{HNO}_3(aq) \quad \text{(nitric acid)}
\end{align*}
\]

The addition of these acids to the atmosphere, lakes, streams, and soils can have a dramatic impact on the health of the ecosystem.

To address the issue of acid rain, the Clean Air Act Amendments of 1990 established the Acid Rain Program at the EPA (http://www.epa.gov/airmarkets/progsregs/arp/index.html) and made reduction of NO\(_x\) and SO₂ emissions a national priority. The program’s efforts to reduce SO₂ emissions has been quite successful, utilizing three methods: (1) switching to low-sulfur content coal; (2) removing the sulfur from the coal prior to burning; and (3) scrubbing stack gases to remove the SO₂ before the gases are released to the atmosphere. In this scrubbing process, stack gases are passed through a slurry of CaCO\(_3\) and the following reaction occurs:

\[
\text{2SO}_2(g) + \text{O}_2(g) + 2\text{CaCO}_3(s) \rightarrow 2\text{CaSO}_4(s) + 2\text{CO}_2(g)
\]

This process is highly efficient at removing the SO₂, but unfortunately is very costly, with estimates ranging from $400-600/ton of SO₂ removed.

In this lab, you will test different materials for their effectiveness of scrubbing to remove SO₂ from “simulated” stack gases. The effectiveness of the scrubbers will be analyzed using a pH meter.
Equipment:
- 3 side-arm test tube with stoppers
- 3 pieces of glass tubing
- 4 pieces of Tygon tubing
- 3 rubber stoppers with holes
- 3 ring stands with clamps
- Bunsen burner
- 100 mL beaker
- pH meter
- Distilled water
- ~2 grams sulfur
- Materials for “scrubbing”
  - 0.3 g CaCO$_3$(s)
  - 25 mL 1 M Na$_2$CO$_3$(s)
  - 1 g KOH pellets
  - 25 mL 1M KOH$_{(aq)}$

Safety Considerations:
- Dust from sulfur, calcium carbonate, and calcium sulfate can be irritating to the upper respiratory tract. Avoid breathing any dust by performing all operations in a fume hood.
- KOH and Na$_2$CO$_3$ are highly corrosive, strong bases and should be handled with caution; direct contact with skin and eyes should be avoided.
- As always, appropriate eye protection should be worn at all times.
- Take caution when adjusting rubber tubing and stoppers connected to the glass tubing, as it can break easily and create sharp edges.

Procedure:

General Setup
1. Construct a vacuum valve as shown in the figure from a glass "T", a valve, and the required tubing. Leave the valve in the "open" position.
2. Connect the "vacuum" side to a vacuum aspirator in the hood.

Part 1: Measuring the increase in acidity of rain water by reaction with SO$_2$(g)
1. Using 2 side arm test-tubes, 2 glass pieces of glass tubing and two pieces of tubing, assemble the reaction apparatus, as shown in Figure 1.
2. Using two clamps, attach the test tubes to two ring stands.
3. Attach the "vent" tube to the vacuum valve.
4. Fill a 100 mL beaker with distilled water.
5. Record the pH of the distilled water with a pH meter.
6. Add 25 mL of the distilled water to the second (right) test tube. Be sure that the glass tubing is fully submerged in the water.
7. To the first test tube (left), add 0.1 g of sulfur.
8. Connect everything, turn on the vacuum aspirator, and slowly adjust the vacuum valve until a steady stream of air bubbles is seen in the distilled water. *Be careful not to let it bubble too vigorously!*

9. Use a Bunsen burner to gently heat the sulfur in the test tube.

10. Heat for ~1-2 minutes. Record observations and the exact time of heating in your lab notebook.

11. Extinguish Bunsen burner, open the vacuum valve, and turn off the vacuum aspirator.

12. Use a pH meter to record the pH of the water.

13. Dispose of the water down the sink. Dispose of the sulfur in Tube #1 in a trash can (this may require scraping with a long spatula). Rinse both test tubes thoroughly with distilled water.

**Part 2: Exploration of different reagents as potential SO₂ scrubbers**

1. Adjust the reaction apparatus to contain three side arm test tubes (Figure 2).

2. Add 0.1 g sulfur to Test Tube #1.

3. Fill a 100 mL beaker with distilled water.

4. Record the pH of the distilled water with a pH meter.

5. Add 25 mL distilled water to Test Tube #3. Be sure that the glass tubing is fully submerged in the water.

6. To Test Tube #2 add 1.0 g calcium carbonate and 25 mL distilled water. (Note: with stirring and agitation, the solution will become cloudy, but the calcium carbonate will not dissolve).

7. Attach the apparatus to ring stands.

8. Attach the "vent" tube to the vacuum valve.

9. Connect everything, turn on the vacuum aspirator, and slowly adjust the vacuum valve until a steady stream of air bubbles is seen in the distilled water. *Be careful not to let it bubble too vigorously!* Allow air to blow through for ~2 minutes to agitate the calcium carbonate slurry.

10. Use a Bunsen burner to gently heat the sulfur in the test tube.

11. Heat for ~1-2 minutes. Record observations and the exact time of heating in your lab notebook.

12. Extinguish Bunsen burner, open the vacuum valve, and turn off the vacuum aspirator.

13. Use a pH meter to record the pH of the water in Test Tube #3.

14. Dispose of the calcium carbonate slurry and the water in Tube #3 down the sink. Dispose of the sulfur in Tube #1 in a trash can (this may require scraping with a long spatula). Rinse the test tubes thoroughly with distilled water.

15. Repeat Steps 2-14 three times, replacing the calcium carbonate and water with:
   a) 25 mL 1 M Na₂CO₃,
   b) 1 g KOH(s) (do not add water)
   c) 25 mL 1 M KOH
Analysis

The following should be done in your notebook. You may choose to do this after the lab, but your lab will not be signed off, and your duplicate pages will not be accepted, until it is completed. This analysis should also appear in your formal lab report.

1. Complete the following data table in your lab notebook:

<table>
<thead>
<tr>
<th>Reagent</th>
<th>pH of Distilled water</th>
<th>H$_3$O$^+$ Conc. ([H$_3$O$^+$])</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before</td>
<td>After</td>
</tr>
<tr>
<td>H$_2$O(l)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CaCO$_3$ (slurry)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 M Na$_2$CO$_3$(aq)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>KOH(s)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 M KOH(aq)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2. Calculate the theoretical number of moles of sulfate that could be produced from 0.2 g of sulfur, if 100% of the sulfur is converted to SO$_2$, and 100% of this is absorbed and converted to H$_2$SO$_4$.

3. Assuming that the change in concentration of hydronium ion ([H$_3$O$^+$]) is only from the reaction:

$$\text{H}_2\text{SO}_4 \ (aq) + \text{H}_2\text{O} \rightarrow \text{HSO}_4^- \ (aq) + \text{H}_3\text{O}^+ \ (aq)$$

Calculate the moles of SO$_2$ absorbed in the distilled water in each experiment.

Conclusions

Answers to these questions must be included as part of your Conclusions in your written lab report. Include them in your Conclusions narrative, not as numbered list of questions and answers.

1. Determine which scrubbing agent is the most effective by calculating the percent of SO$_2$ (by moles) absorbed by the scrubbing agents using your answers from questions 2-4 above.

2. If you ran the IC analysis, briefly compare the results from this technique versus the pH measurement.

3. Speculate as to why CaCO$_3$ is the scrubber of choice for industry.