

AESF Research Grant - G2429
Q4 (December), 2023 Quarterly Report

PI: Brian P. Chaplin, University of Illinois at Chicago

Summary:

In this quarter, we focused on deposition of Bi and Sn catalysts to the reactive electrochemical membranes (REMs) and electrochemical oxidation of 6:2-fluorotelomersulfonic acid (6:2-FTS) in a NaClO₄ background electrolyte. Oxidation experiments were performed with bismuth/tin oxide electrodeposited catalysts (BTO/EDT/REM) and BTO catalysts deposited by the incipient wetness method (BTO/IW/REM). An additional BTO electrodeposited catalyst was treated at 550 °C in argon gas (BTO/EDT/Ar/REM). Finally, catalyst stability was investigated during the electrochemical oxidation of 6:2-FTS using a BTO/EDT/REM for a duration of 8 hours at a consistent potential of 4.2 V/SHE. The oxidation experiments were conducted in the crossflow cathode-anode (CF-CA) flow mode. The oxidation experiment with BTO/IW/REM showed >85% 6:2-FTS removal and $41 \pm 3\%$ fluoride yield at 4.2 V/SHE for the NaClO₄ background. The longevity experiment at 4.2 V/SHE showed 6:2-FTS removal of $69.3 \pm 4.5\%$ and a $20.8 \pm 2.5\%$ fluoride yield after 2 hours. The ICP-MS analysis of the longevity experiment indicated significant leaching of Bi within the initial 2-hour period, which affected 6:2-FTS removal.

Results and Discussion

In this quarter, electrochemical oxidation experiments were carried out using a 100 μM 6:2-FTS feed solution in 100 mM NaClO₄. In the CF-CA flow mode, the concentration profiles of 6:2-FTS and fluoride yields from the conducted experiments with BTO/IW/REM were compared with those conducted using BTO/EDT/REM and BTO/EDT/Ar/REM, as illustrated in **Figure 1a** and **Figure 1b**, respectively. The oxidation experiment with BTO/IW/REM, BTO/EDT/REM, and BTO/EDT/Ar/REM showed $41 \pm 3\%$, $36 \pm 1.5\%$, and $38 \pm 5\%$ fluoride yield at 4.2 V/SHE, respectively and >85% 6:2-FTS removal for all prepared REMs. The lower fluoride yield for BTO/EDT/REMs was attributed to the catalyst leaching. However, it is imperative to conduct a detailed analysis of catalyst leaching in the case of BTO/IW/REMs.

As shown in **Figure 2a** and **Figure 2b**, the 6:2-FTS removal reached $69.3 \pm 4.5\%$, with a fluoride yield of $20.8 \pm 2.5\%$ after 2 hours. To analyze Bi and Sn leaching during the experiment, samples were analyzed using inductively coupled plasma mass spectrometry (ICP-MS). The results, depicted in **Figure 2c** and **Figure 2d**, highlight the significant leaching of Bi within the initial 2 hours of the experiment.

Future Work

Future work will focus on the study of the catalyst leaching of BTO/IW/REM to determine the most stable catalyst for the 6:2-FTS oxidation experiment. Additionally, the fluoride balance will be calculated through the analysis of short-chained PFASs in the experiment.

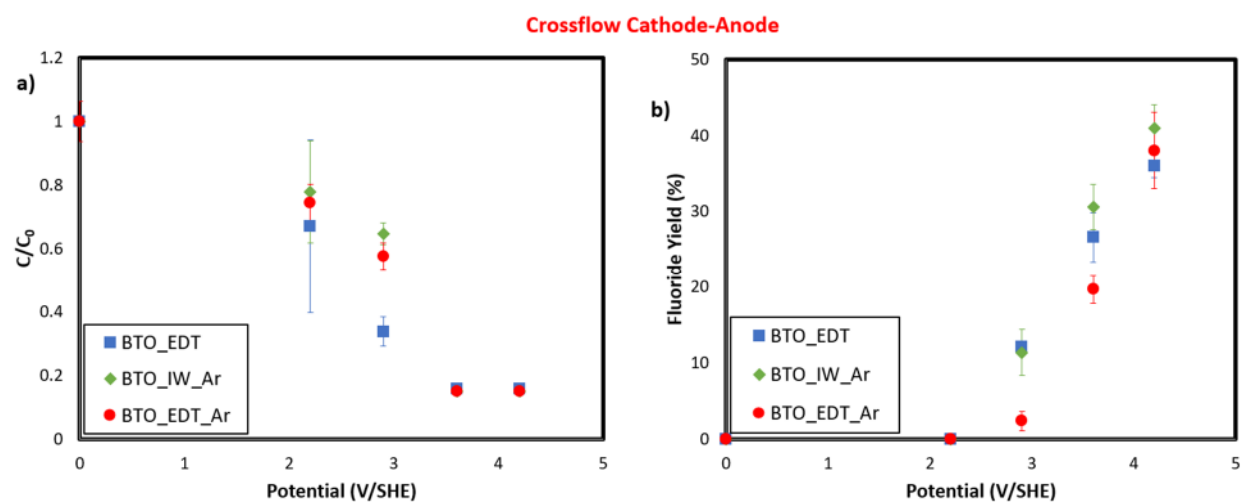


Figure 1: (a) 6:2 FTS concentration profile, (b) fluoride yield for the 100 μM 6:2 FTS oxidation experiments using BTO/EDT/REM, BTO/IW/REM, and BTO/EDT/Ar/REM at different potentials and at a constant flux of 240 LMH in a CF-CA flow mode of the flow-through reactor in NaClO_4 feed solutions.

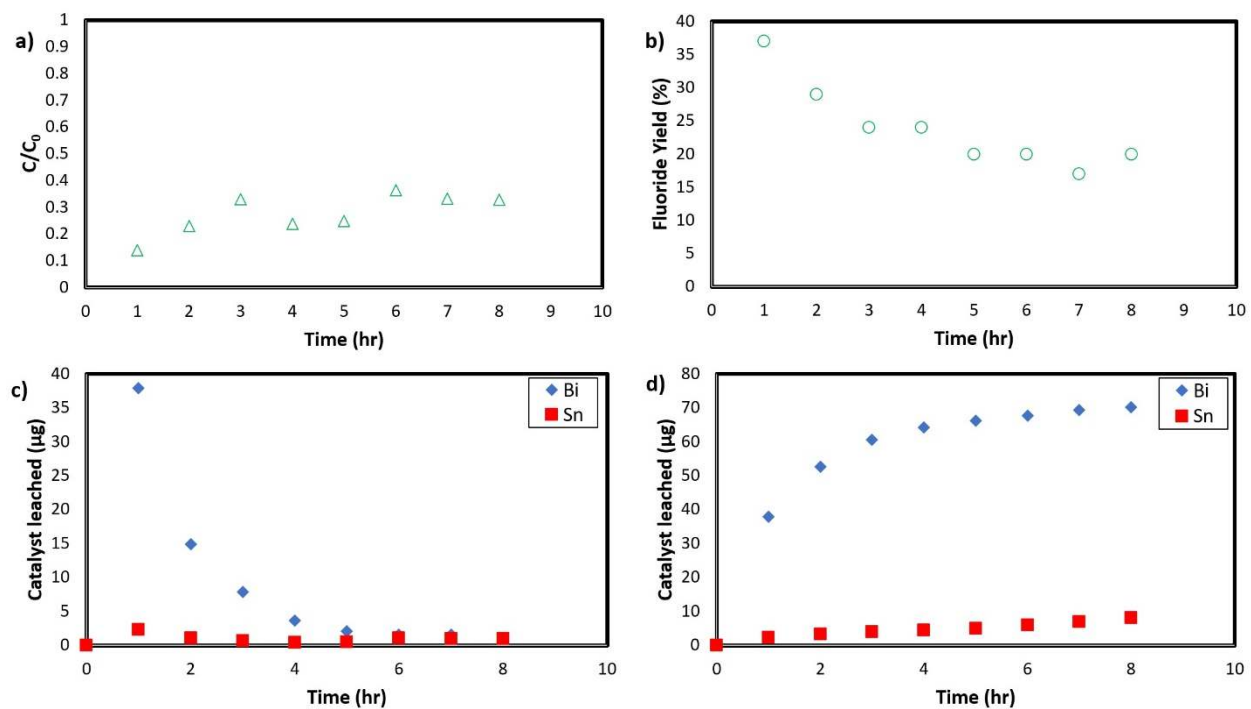


Figure 2: (a) 6:2 FTS Concentration profile, (b) fluoride yield with time in NaClO_4 electrolytes at 4.2 V/SHE using BTO/EDT/REM. (c) catalyst leached vs time, (d) cumulative catalyst leached vs time for the longevity experiment.