

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Pressure Drop Trajectory

Barium chloride and sodium sulfate solutions were flowed through 0.02" ID stainless steel capillary of 30 cm (Upchurch Scientific, U-104) and the differential pressure, ΔP , was measured as a function of time. The experimental results were normalized by the initial pressure drop without deposition, ΔP_0 , as described in Experimental Methods section. The result shown in Figure 4.1 shows $(\Delta P - \Delta P_0)$ vs. time for deposition experiment at a total flow rate of 2 mL/min and 25 °C for different runs. It was observed that the pressure drop increases overtime indicating that deposition has occurred inside the capillary.

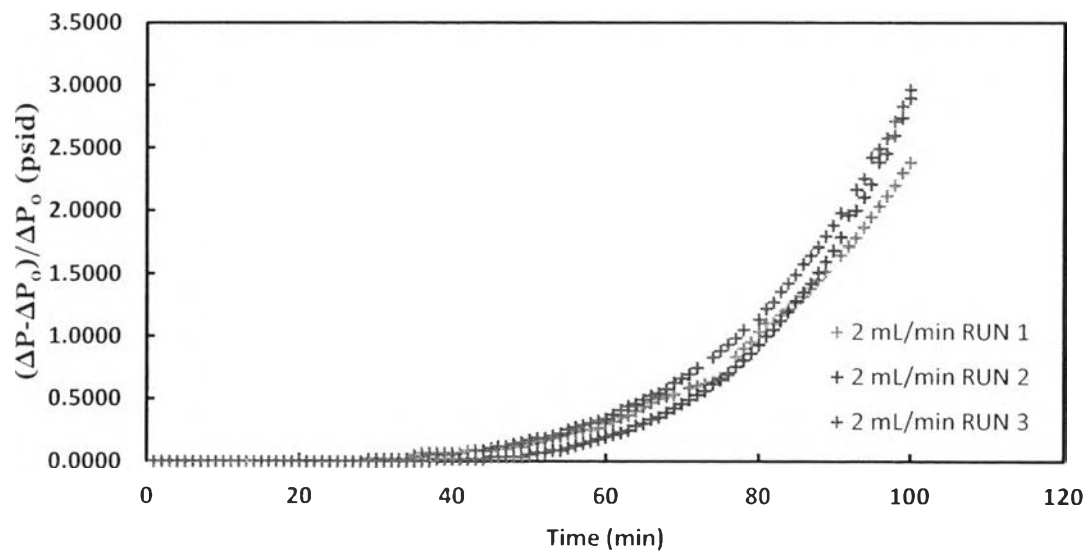


Figure 4.1 Pressure drop vs. time. All runs performed in 0.02" ID capillaries of 30 cm lengths at 25 °C and 2 mL/min.

4.2 Concentration Trajectory

Prior to performing deposition experiments, barium chloride and sodium sulfate solutions were pumped through the feed lines without mixing tee and capillary. One sample was collected to measure the actual concentration of barium ions at inlet. Barium chloride and sodium sulfate solutions were then flowed through 0.02" ID stainless steel capillary of 30 cm (Upchurch Scientific, U-104) and samples are collected overtime as described in the Experimental Methods section. Figure 4.2 shows a plot of barium ion concentrations as a function of time. The solid line at the top of the Figure represents the inlet concentration of barium ions. Although, the inlet concentration was held constant during the experiment, it was observed that barium outlet concentrations decrease as time passes indicating that some of the barium ions have accumulated inside the capillary. This result confirms the result from pressure drop measurement that barium sulfate deposition has occurred in the system.

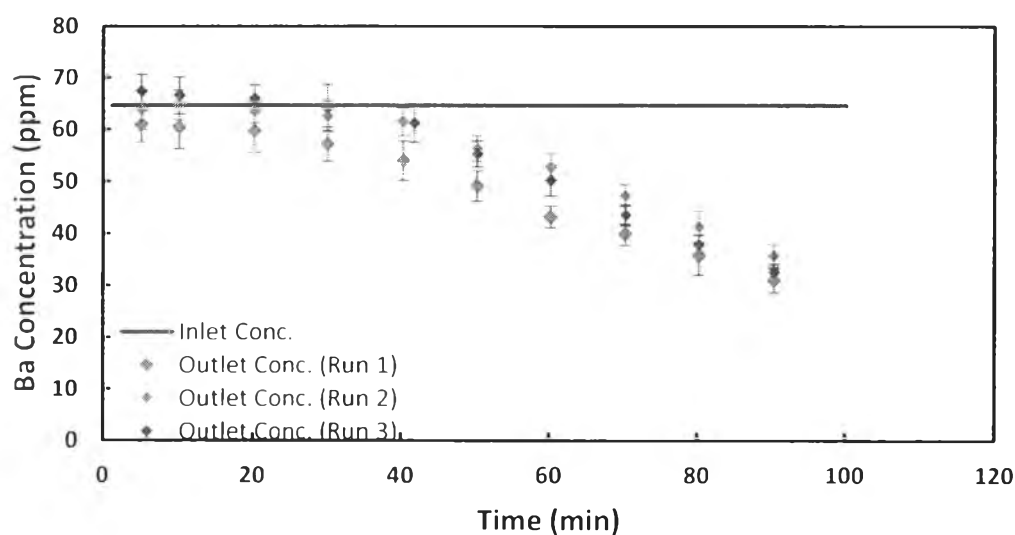


Figure 4.2 Barium ion concentration vs. time for 0.02" ID capillaries of 30 cm at 2 mL/min and 25 °C.

4.3 Capture Efficiency

The capture efficiency is defined as the mass of barium sulfate that deposit compared to the mass of barium sulfate that have passed through the capillary. It can be calculated using equation ().

$$\text{Capture efficiency} = \frac{(C_{Ba^{2+},inlet} - C_{Ba^{2+},outlet})}{C_{Ba^{2+},inlet}} \times 100$$

Figure 4.3 shows the capture efficiency as a function of time for deposition experiments at 2 mL/min and it can be seen that capture efficiency increases as time passes. An observed increase in capture efficiency can be explained by using the idea of surface area in the capillary which will be discussed later on in the Results and Discussion section.

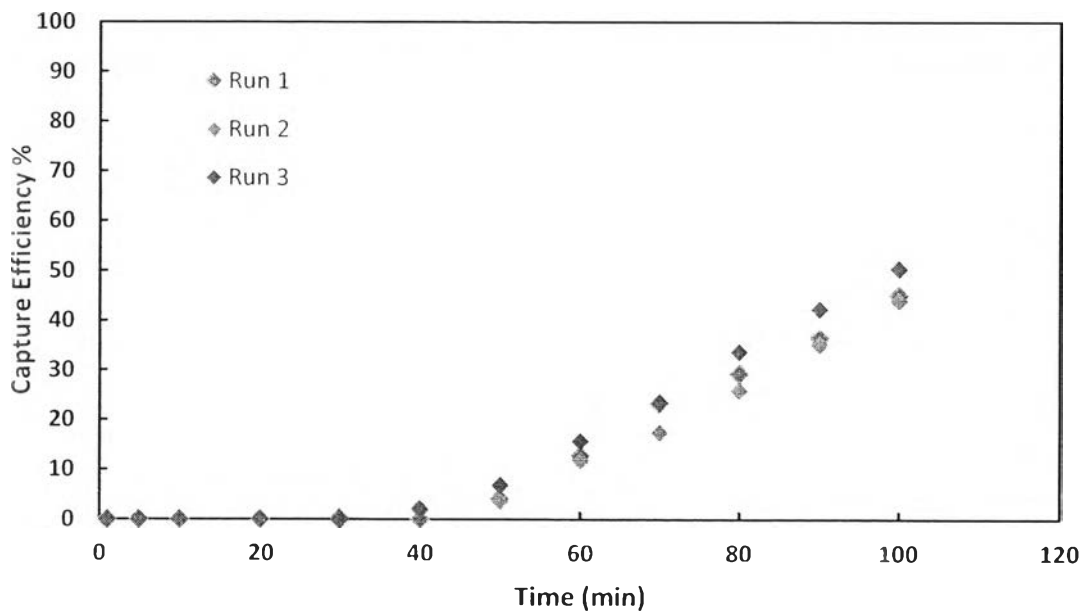


Figure 4.3 Plot between capture efficiency and time. All runs performed in 0.02" ID capillaries of 30 cm lengths at 25 0C and 2 mL/min.

4.4 Electron Microscopy Images of Deposits

The deposits shown in Figure 4.4 were generated by flowing barium chloride and sodium sulfate solutions through a 0.02" ID and 30 cm length capillary at 25 °C. The SEM images of the capillary inlet (left) and outlet (right) are shown for deposition experiment at 2 mL/min. The inner capillary wall edge is indicated by dashed circles on the SEM images and the dashed lines separate the capillary into two sides to show the direction of solution flow. The SEM images have been rotated to match their orientation during the deposition experiment. The deposits shown in Figure 5 are non-uniform radially, and the deposit is thicker at the outlet than at the inlet. The image showed that dendritic crystal was observed at the top and bottom of the capillary outlet. The idea of dendrite formation can be used to aid in the understanding an increase in capture efficiency since these crystals have high surface area for barium sulfate to deposit leading to a decrease in barium outlet concentration over time.

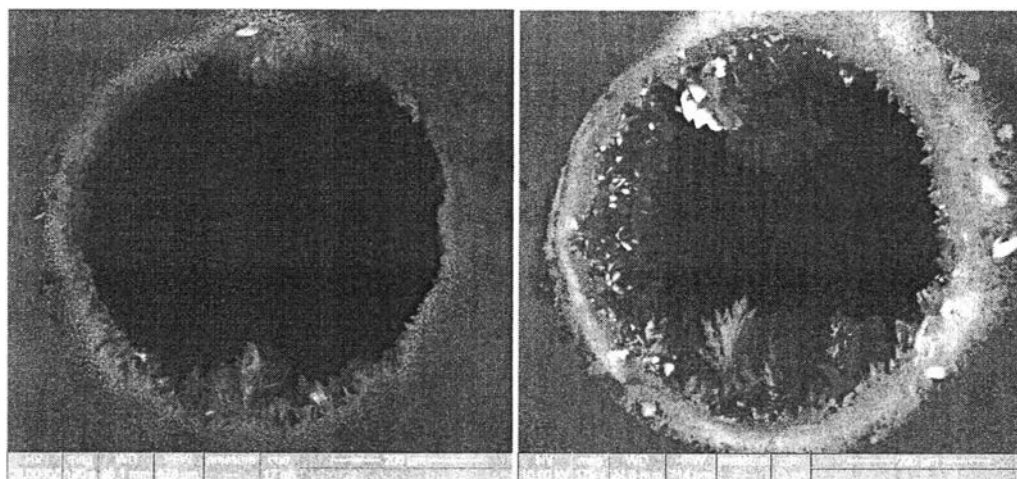


Figure 4.4 SEM images of used capillary inlet (left) and outlet (right) for Run #1. Experiments were performed at 2 mL/min in 0.02" ID and 30 cm length capillaries.

4.5 Detection Time

For quantitative determination of the detection time, t_d , it is defined as the point where an upward trend the pressure is first detected. Or it can be defined as the point where a decrease in barium outlet concentration is observed. The deposition results show that a detection time was observed in both concentration and pressure drop trajectories as indicated by the solid line in Figure and Figure. There are two possible causes for the observed detection time. First, the interaction between barium sulfate particles itself is greater than the interaction between barium sulfate and stainless steel signifying that the rate of first layer formation is slower than the rate of subsequent layer formation. Another possible explanation for the observed detection time is surface area in the capillary. Surface area is considered to be at a minimum value during the first period of the experiment because there is no deposition on the capillary wall, and increases over time due to dendrite formation. The existence of detection time is likely the explanation for the observed dendrite as mentioned previously.

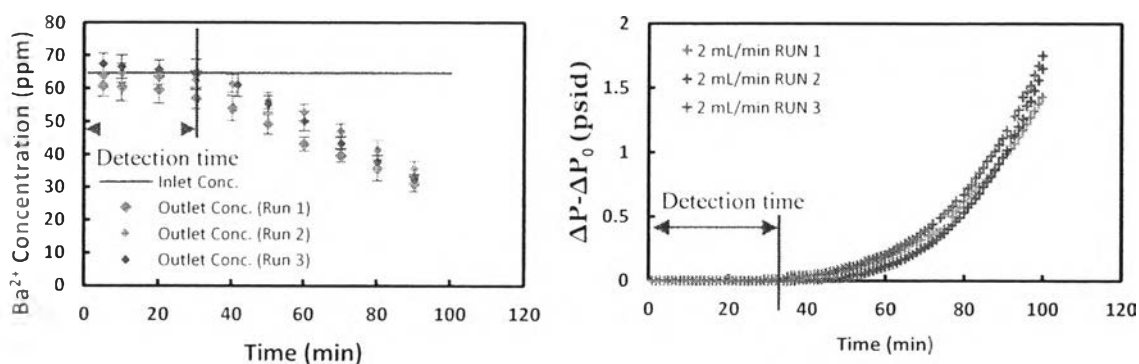


Figure 4.5 Detection time observed in concentration and pressure drop trajectories.

4.6 Deposition Profile

Deposition experiments were performed in 0.02" ID stainless steel capillaries of 10 cm, 20 cm, and 30 cm length (Upchurch Scientific, U-102, U-103, and U-104 respectively) at a total flow rate of 2 and 8 mL/min and the differential pressure of each capillary length was measured as a function of time. The pressure drop in each section can be calculated by decoupling the pressure drop profile into three sections (0-10, 10-20, and 20-30 cm) using equations below

$$\Delta P_{1^{st}section} = \Delta P_1$$

$$\Delta P_{2^{nd}section} = \Delta P_2 - \Delta P_1$$

$$\Delta P_{3^{rd}section} = \Delta P_3 - \Delta P_2$$

where ΔP_1 = pressure drop for 10 cm capillary

ΔP_2 = pressure drop for 20 cm capillary

ΔP_3 = pressure drop for 30 cm capillary

As you can see from Figure 4.6 the pressure in the second section is the highest and higher than in the first and third section respectively. It indicates that deposits mostly formed in the length between 10 and 20 cm. This can be confirmed by the results from concentration measurement. Figure 4.7 shows a plot of normalized concentration as a function of time and it can be seen that as the capillary length is increased the concentration of barium ions decrease. The observed decrease in concentration is caused by the deposition on the capillary wall as most of the barium ions deposit and residence time as bulk aggregation starts occurring. Moreover, the concentration trajectory is very similar at 20 and 30 cm but different from 10 cm indicating that most of the deposits occur in the second section. SEM images of used capillaries were taken to confirm these results. Figure 4.8 shows the capillary inlet and capillary outlet for 10 and 20 cm length capillary. It can be clearly seen that the deposits mostly occur in the second section as can be seen from the SEM image of capillary outlet for 20 cm.

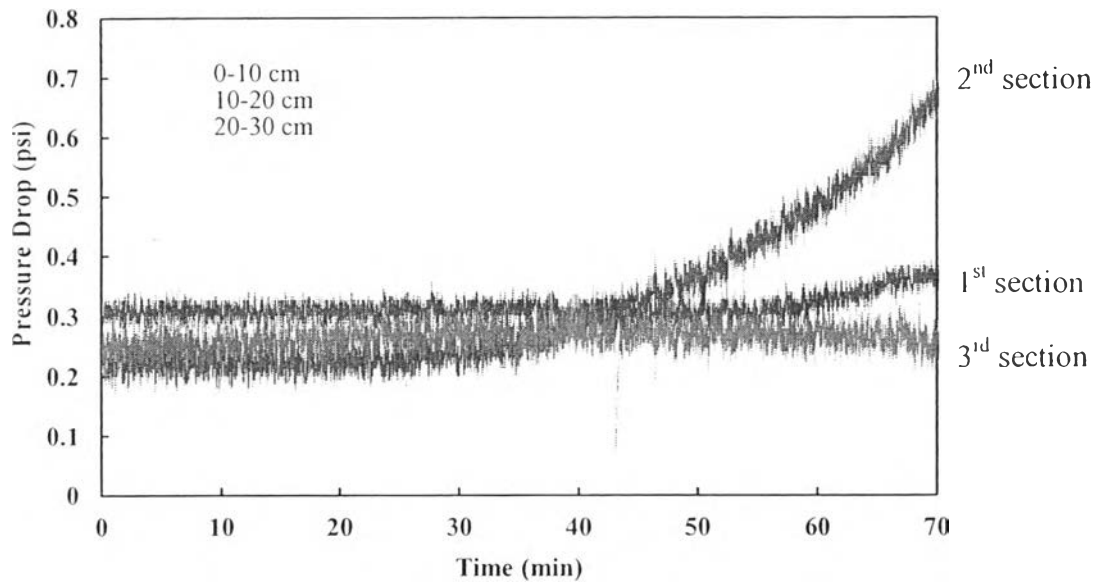


Figure 4.6 Pressure drop vs. Time for each capillary section for deposition experiments performed at 2 mL/min.

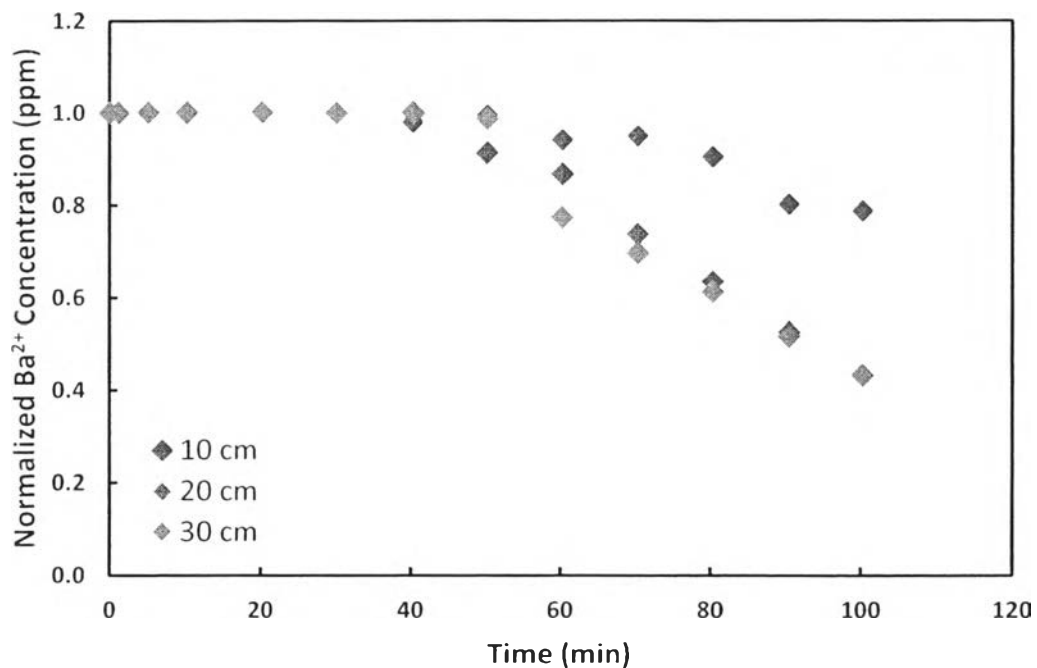


Figure 4.7 Normalized barium concentration vs. Time for different capillary lengths.

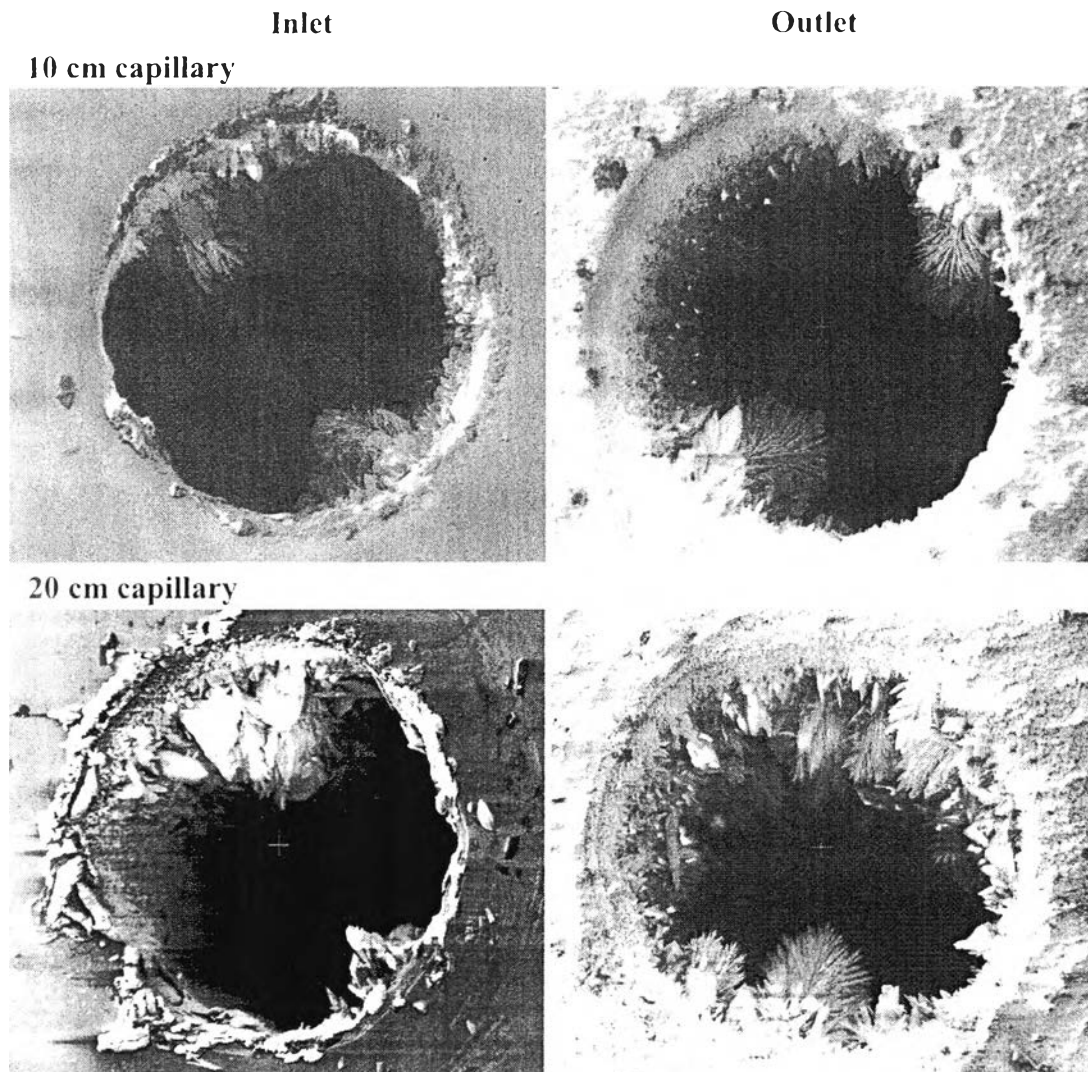


Figure 4.8 SEM images of capillary inlet and outlet for the barium sulfate generated in 0.02" ID capillaries with 10, 20, and 30 cm length. Most of the deposits were observed at 20 cm capillary outlet.

4.7 Deposition Profile as a Function of Flow Rate

Deposition experiments were carried out by following the same procedures as discussed earlier but the total flow rate was changed to 8 mL/min. The experimental results shown in Figure 4.9 show the differential pressure as a function of time and it was observed that the pressure drop in the first section is the highest indicating that most of the deposits occur in the first section. The upward trends for the second and third section were observed after 30 and 40 minutes respectively suggesting that the deposition starts occurring at time approximately equal to 30 and 40 minutes. The results also suggests that the deposition rate in the third section is the highest after that time as can be seen from the slop of the graph. The results is in good agreement with the concentration trajectory shown in Figure 4.10 that there was no significant difference in normalized barium ion concentrations for all capillary lengths until it reached approximately 35 min. SEM images of capillary inlet and outlet for each capillary length are shown in Figure 4.11.

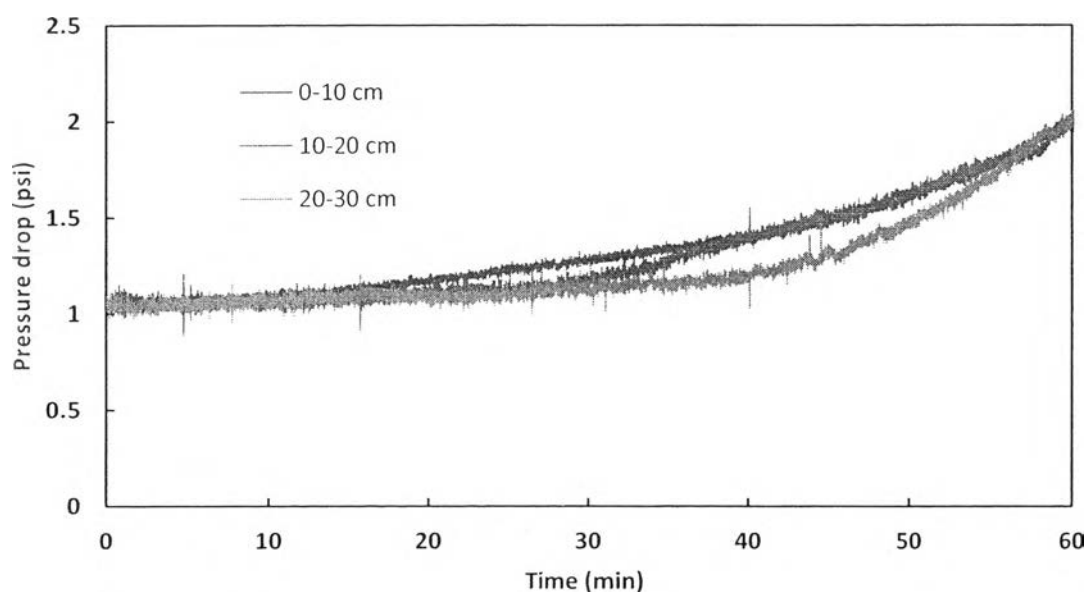


Figure 4.9 Pressure drop vs. Time for each capillary section for deposition experiments performed at 8 mL/min.

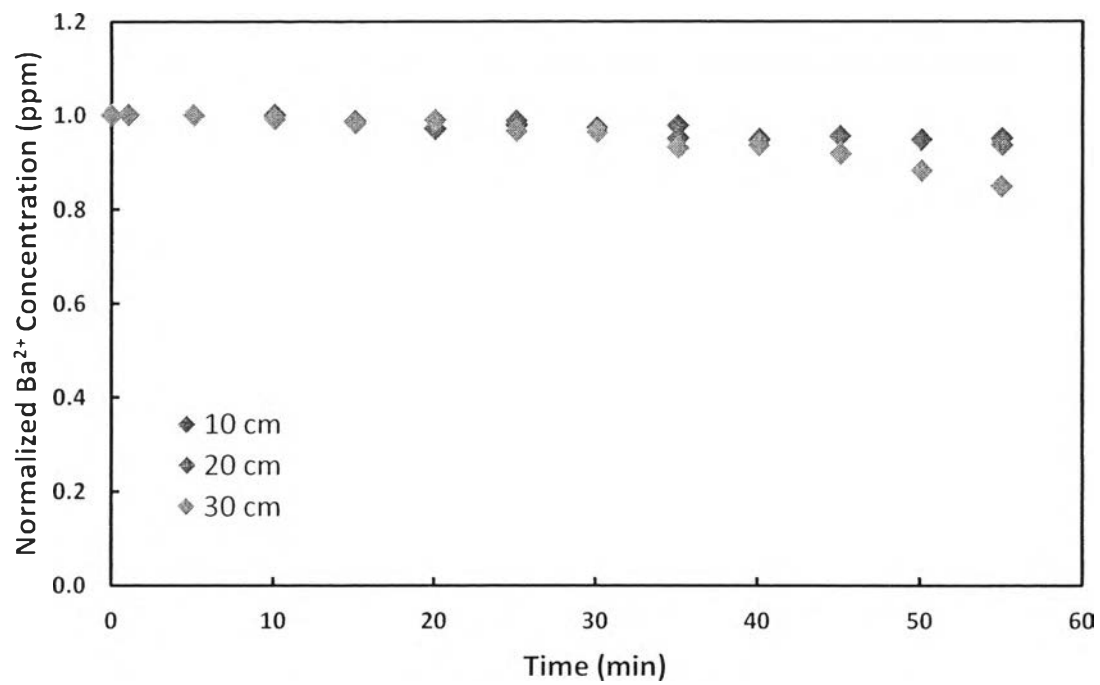


Figure 4.10 Normalized barium concentration vs. Time for different capillary lengths.

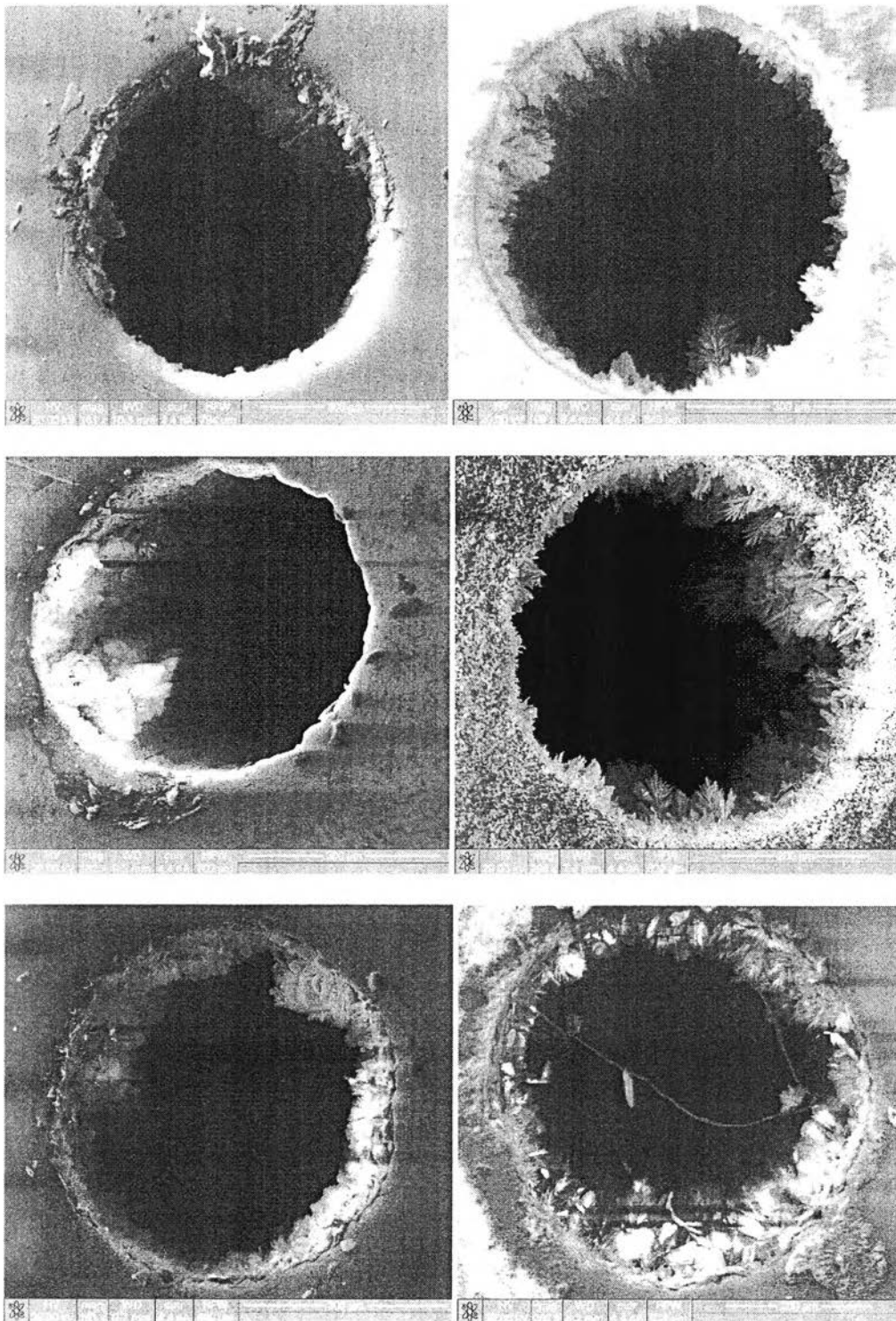


Figure 4.11 SEM images of capillary inlet and outlet for the barium sulfate generated in 0.02" ID capillaries with 10, 20, and 30 cm length.