APPENDIX A

VOID VOLUME MEASUREMENT BY THE BREAKTHROUGH METHOD AND CALCULATION OF THE CHANNEL THICKNESS

Void volume (V^0) is defined as the volume of FFF channel in which separation takes place. In this work the void volume was measured by the method given by Giddings et al, (1992) [100].

Silica (<10 μm) was used as the sample probe to determine the breakthrough signal. The measurement of the void volume for each flow rate was obtained by repeated injections (n=5). The different flow rates gave the same void volume, as shown in Figure A1.

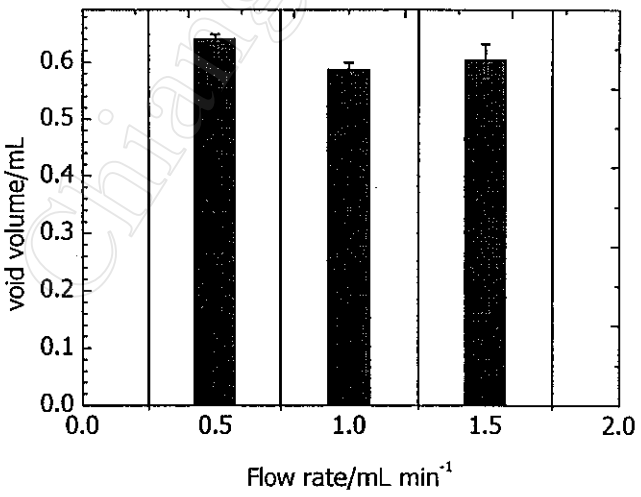

Figure A1 Measured void volumes at the different flow rates

The average void volume can be obtained as 0.610 mL. The post channel void volume of tubing was subtracted from this value to obtain a volume of 0.542 mL.

Calculation of the geometric void volume of the channel can be given by multiply geometric area of the channel by the channel thickness, which was taken as the measured thickness of the overhead transparency (0.100 mm). The geometric void volume was 0.566 mL, a small higher than the measured V^0. Therefore the actual thickness of the channel was then calculated to be 0.095 mm. From the measured void volume and channel area, the channel thickness (w) can be calculated. This yields a value of 0.095 mm for the transparency sheet used to construct the channel.

APPENDIX B

EVALUATION OF THE DIGESTION METHOD OF METAL ANALYSIS OF CLAYS SAMPLE

Evaluation of the digestion method was performed employing certified reference sediment materials and analysis by FAAS. The results obtained by dissolution of two certified reference materials (PACS1 and PACS2) followed by FAAS analysis are shown in Figure B1.

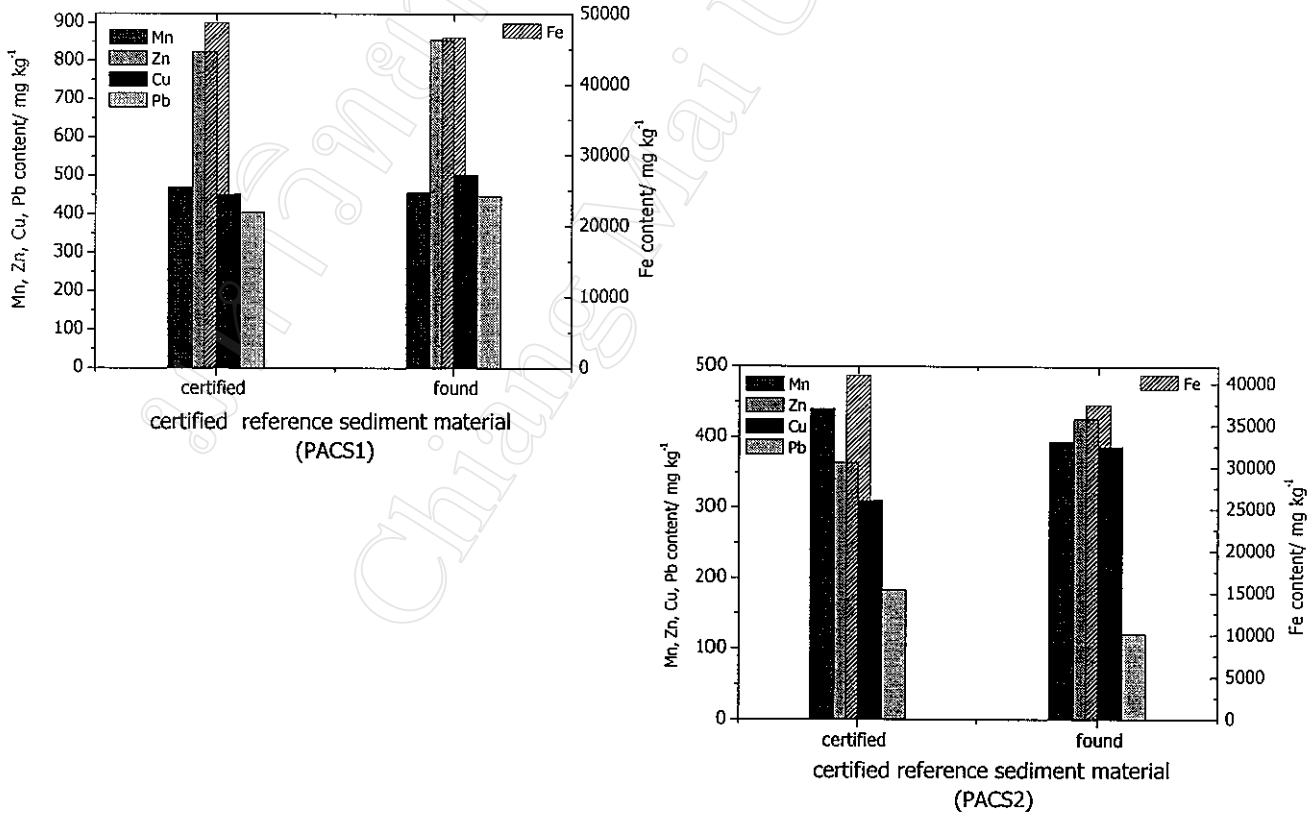

Figure B1 Metal contents in certified reference sediment materials from certified values given and found from digestion method

The metal contents of the clay samples used in this thesis (kaolin clay, Red clay, Ball clay 1 and Ball clay 2) are shown in Figure B2.

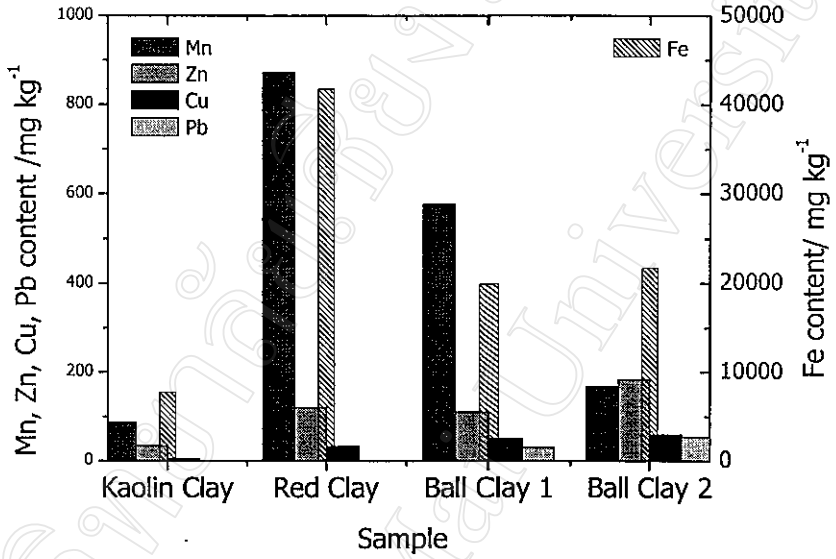

Figure B2 Some metal contents in clay samples

APPENDIX C

PERFORMANCE TEST OF ASYMMETRIC FLOW FIELD-FLOW FRACTIONATION [101-102]

Symmetric flow field-flow fractionation (FlFFF) is now commonly used for separation and characterization of a wide variety of macromolecules and particles including polymers, proteins, cells and sediments. Asymmetric FlFFF is an alternative, which has some advantages associated with employing only one frit. The upper glass channel wall allows the separation channel to be viewed during separation and testing. This allows sample focusing and elution to be monitored using colored samples. It should be also possible for combine an asymmetric FlFFF instrument with flow injection analysis (FIA). The schematic diagram of the channel and separation mechanism for asymmetric FFF is illustrated Figure C1. Test runs with two samples, blue dextran and polystyrene latex beads (43 nm) were performed. By employing the intense color of blue dextran the focusing position of the sample zone can be obtained. The retention of polystyrene latex beads and its focusing time were measured.

In this work, an asymmetric FlFFF system was set up and tested, which was lower in cost and simpler to assemble (using 1 porous ceramic frit) than symmetric FlFFF (comprising 2 porous ceramic frits). The focusing/relaxation of asymmetric FlFFF takes place when the carrier liquid is pumped into both inlet and outlet ends

of the channel. The position of the focused sample zone could be clearly seen using the blue dextran sample.

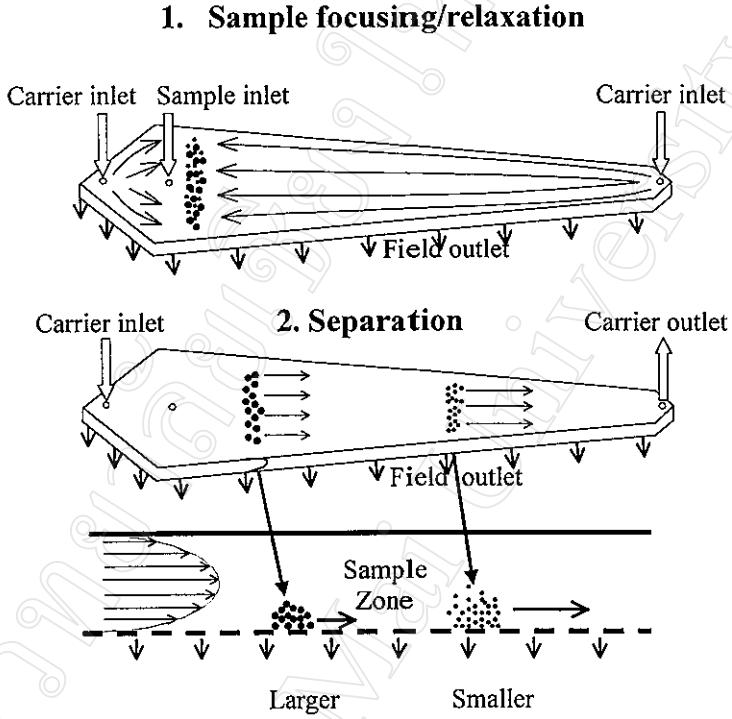

Figure C1 Asymmetric flow field-flow fractionation channel and separation mechanism illustration

The focusing position was 0.5 cm downstream from the sample injection tube. In this channel it was found that the focusing time should be 1 min in order to obtain the minimum overlap of the sample peak with void peak. The retention time of polystyrene latex beads (43 nm) was used to calculate the channel thickness, which was found to be 0.027 cm. Adequate retention and narrow peaks were obtained to all of the polymer and latex particles tested.

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PUBLICATIONS

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