THE EFFECT OF THE CHANNEL HEIGHT ON THE SEPARATION EFFICIENCY OF AN ELECTRICAL FIELD FLOW FRACTIONATION SYSTEM

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ABSTRACT

In this study, the effect of the channel height on the separation efficiency of Electrical Field Flow Fractionation (EFFF) systems was investigated. It has been shown for the first time that if optimum channel height and experimental parameters are selected, baseline separations of nanoparticles (with sizes less than 100nm) can be achieved by Cyclical Electrical Field Flow Fractionation. EFFF channels with four different channel heights were fabricated and separation experiments were made with 15 and 40nm gold nanoparticles. Specifically, baseline separation of 15 and 40nm gold nanoparticles were achieved by using the system with a channel height of 125µm. We believe that by selecting the proper channel height and applying the appropriate voltage waveforms explained in this study, Cyclical Electrical Field Flow Fractionation will be a much more capable method for the fractionation of sub 100nm particles.

KEYWORDS

Ectrical field flow fractionation, nanoparticle separation, nanoparticles, separation, field flow fractionation

INTRODUCTION

Field Flow Fractionation (FFF) is a powerful method for the separation and characterization of macromolecular, colloidal and micron-sized particles [1]. Cyclical EFFF (CyEFFF) is one of the subtechniques of FFF which separates the particles according to their size and electrical mobilities [2]. In CyEFFF, the separation channel is composed of bottom and top electrodes which are separated by a thin spacer. A typical schematic of a CyEFFF system can be seen in Figure 1.

In the literature, EFFF systems have channel heights ranging from 30 to 200µm [3-5]. Until now, there were no studies investigating the different channel heights and their effect on the separation efficiency. In this work, we fabricated channels with 4 different heights and conducted separation experiments with each of them to determine the optimum channel geometry for the separation experiments.

METHODS

An EFFF system (width: 2.4cm, length: 43cm) was fabricated by using graphite electrodes and a Mylar spacer as shown in Figure 2a. Different channel heights were obtained by using spacers of different thicknesses (h=25, 75, 125 and 200µm).

We made separation experiments with a mixture of 15 and 40 nm mean diameter gold nanoparticles. In all experiments, the frequency of the applied square wave voltage was 20Hz and the peak flow velocity in the channel was kept at 4.9mm/s. In each experiment, de-ionized water (18.2 $M\Omega cm^{-1}$) was used as the carrier. In most of the experiments, the electric field inside the channel was kept at 80kVpp/m, but we lowered the electric field for the 200um channel to prevent the electrolytic breakdown of water and bubble formation.

In addition to the separation experiments we also conducted I-V measurements to determine the electrical circuit parameters of the EFFF systems. The electric circuit equivalent of the EFFF system can be seen in Figure 2b. We found all the equivalent circuit components for all of the different EFFF channels.

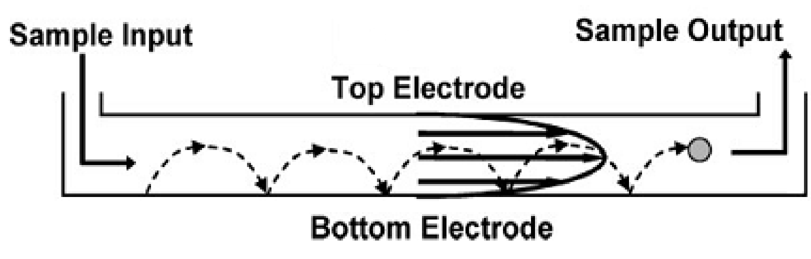

Figure 1. Cyclical EFFF System. The dashed line shows a particle trajectory generated in response to the cyclical field. Oscillating square wave voltages are applied to the electrodes which result in a cyclical electric field inside the channel. As a result of the cyclical electric field, particles move back and forth between the electrodes. Particles with high electrophoretic mobilities move longer distances away from the channel walls and they spend more time in the faster fluid regions. As a consequence, they elute earlier than the lower mobility particles.

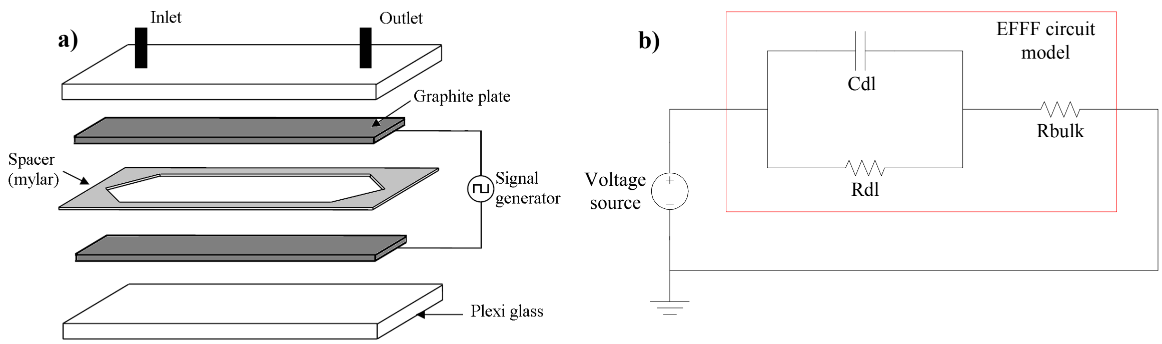

Figure 2. a) EFFF system fabrication b) EFFF electrical circuit model

RESULTS & DISCUSSION

Figure 3a shows the separation results for the 125µm channel. As can be seen from the figure, for the 80% duty cycle condition, we achieved a baseline separation. This is the first baseline separation demonstrated in the Cyclical EFFF literature for sub 100nm nanoparticles. As shown, as the duty cycle of the waveform increased much better separations were achieved. Since earlier CyEFFF related work used 50% duty cycle waveforms, none were capable of fractionating nanoparticles smaller than the size of 100 nanometers.

Figure 3b, shows the UV fractograms for individual injections of 15 and 40 nm gold nanoparticles, and a mixture. As can be seen in the figure, the first peak in the bottom fractogram corresponds to the 15nm gold nanoparticles and the second peak in the fractogram corresponds to the 40nm gold.

For each experiment, resolutions of the separations were calculated according to the below equation:

$$R = \frac{2(t_2 - t_1)}{pw_2 - pw_1}$$

R: resolution; t_1 & t_2: peak center times; pw_1 & pw_2: peak width durations

Highest resolution results were obtained for h=125µm as shown in Table 1. For the 25um channel, no separation was observed. The reason for that was the electrical shortening of the top and bottom electrodes at the 25µm separation distance. In general, the best separation results were obtained for the 125µm channel, 75 and 200µm channels were less effective.

Electric circuit parameters found for each EFFF channel were tabulated in Table 4. As expected, 75µm channel has the highest capacitance. Rbulk corresponding to the 125µm channel was found to be smallest among the others

CONCLUSION

By this study, it has been shown that the selection of the proper channel height has a crucial importance to achieve separations of the nanoparticles. In addition, baseline separation of the sub 50nm particles was achieved for the first time with the Cyclical EFFF method. This separation became possible by selecting the optimum channel height and by applying the optimum voltage waveforms with high duty cycles. In general, by the help of the methods explained in this study, Cyclical Electrical Field Flow Fractionation now carries a great potential in the fractionation of nanoparticles with sizes even smaller than 50nm.

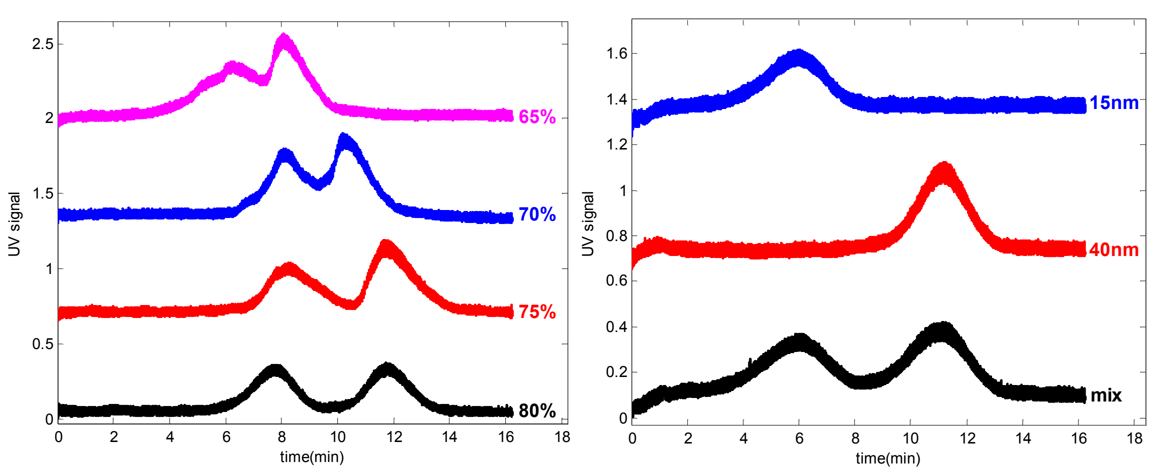

Figure 3. a) UV fractograms for the separation of 15 and 40 nm gold particles (channel height= 125 um) f=20Hz, flow=4.9mm/s, E-field=80kV/m b) Fractograms corresponding to the injections of only 15 and 40 nm gold nanoparticles and their mixture. Experimental conditions were same as part a, and duty cycle is 80%.

Table 1. Separation resolution results for channel height of 125µm.

h=125µm	*Electrical field(kVpp/m)*	*80*	*80*	*80*	*80*
	Duty cycle	*65%*	*70%*	*75%*	*80%*
	Resolution	*0.26*	*0.40*	*0.69*	*0.84*

Table 2. Separation resolution results for channel height of 200µm. (For the 200µm channel, bubble formation observed at 80kVpp/m E-field and we lowered the E-field magnitude to get rid of the bubbles.

h=200µm	*Electrical field(kVpp/m)*	*80*	*80*	*50*	*50*
	Duty cycle	*75%*	*80%*	*75%*	*80%*
	Resolution	*Bubble*	*Bubble*	*0.61*	*Bubble*

Table 3. Separation resolution results for channel height of 75µm.

h=75µm	*Electrical field(kVpp/m)*	*80*	*80*	*133*	*133*
	Duty cycle	*80%*	*90%*	*75%*	*80%*
	Resolution	*0.31*	*0.49*	*0.25*	*bubble*

Table 4. EFFF electrical circuit parameters

Channel Height	Rdl (double layer resistance)	Cdl (double layer capacitance)	Rbulk (bulk resistance)
h=75µm	184 Ohm	5 mF	41 Ohm
h=125µm	206 Ohm	1.8 mF	23 Ohm
h=200µm	1033 Ohm	0.5 mF	51 Ohm

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