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A Method for Determination of Particle Magnetic Susceptibility with Analytical Magnetapheresis

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We recently developed a new method for simple determination of particle magnetic susceptibility using analytical magnetapheresis. This new method does not require laborious calibration plots and trial susceptibility values as do previous analytical magnetapheresis methods. The new method is based on balancing channel flow rates and magnetically induced flow rates for particle deposition in analytical magnetapheresis. The maximal flow rate for complete particle deposition was determined experimentally and set to equal the magnetically induced flow rate for determining particle magnetic susceptibility. This magnetic susceptibility determination generally takes less than 20 min. Several magnetically susceptible and ion-labeled particles were tested using this new method. The carrier magnetic susceptibilities were varied, and erbium ion-labeled particles were studied experimentally, resulting in successful susceptibility determinations of erbium ion-labeled particles and yeasts. The precision of each measurement was generally ∼10%. Experimental determination of particle magnetic susceptibilities differed by less than 10% from reference measurements taken using a superconducting quantum interference device magnetometer. This method can determine minimal susceptibilities on the order of 10^{-9} cgs. The minimum number of erbium labeling ions per particle required for complete deposition of silicas and yeasts was found to be 6.7×10^9. Analytical magnetapheresis shows good potential for use in simple determination of particle magnetic susceptibilities and should become a useful technique.

Magnetic separation has been used in many industrial applications for a long time.[1−3] The use of magnetic separation and related techniques in biotechnology,[4−10] wastewater treatment,[11,12] and other applications[13] has grown rapidly in recent years. Magnetic separation is simple, fast, and selective for magnetically susceptible samples. Magnetic fields using permanent magnets are especially economical and deserve further investigation and development.

Analytical magnetapheresis is a newly developed technique for analyzing magnetically susceptible particles[4] in which these particles in a carrier flow through a separation channel to form deposits under magnetic fields. Front and side views of an analytical magnetapheresis system are shown in Figure 1. The magnetic forces act perpendicularly to the channel flow axis to drive magnetically susceptible particles toward the interpolar gaps. Particles with high magnetic susceptibility (shown as solid circles in the figure) are attracted by the magnetic forces and deposited upon the interpolar gap as they pass along the separation channel. Particles with diamagnetic or low magnetic susceptibility (shown as hollow circles in the figure) are less attracted by the magnetic forces and pass completely through the separation channel. Therefore, particles with different magnetic susceptibilities can be separated by passing them through analytical magnetapheresis separation channels under optimal magnetic fields. A front view of a multichannel analytical magnetapheresis system is shown on the left side of Figure 1. The multichannel setup can simultaneously run magnetapheresis analyses up to eight sample types at a time and is important for comparative experiments. The separation channel is a thin (<0.03 cm), ribbonlike channel with a rectangular shape. The magnetic forces applied to samples can be calculated with good accuracy since the separation channel is unpacked and has a simple geometry. Depositions of magnetically susceptible particles are calculable using the Stokes and force equations for known sample physical parameters and magnetic field strengths. On the other hand, one of the sample physical parameters can be deduced from the percentage of particle depositions and known magnetic field strengths.

Measurements of particle magnetic susceptibility using analytical magnetapheresis have been published.[4] However, most require laborious calibration plots and trial magnetic susceptibilities to obtain particle magnetic susceptibilities. In this study, we devel-

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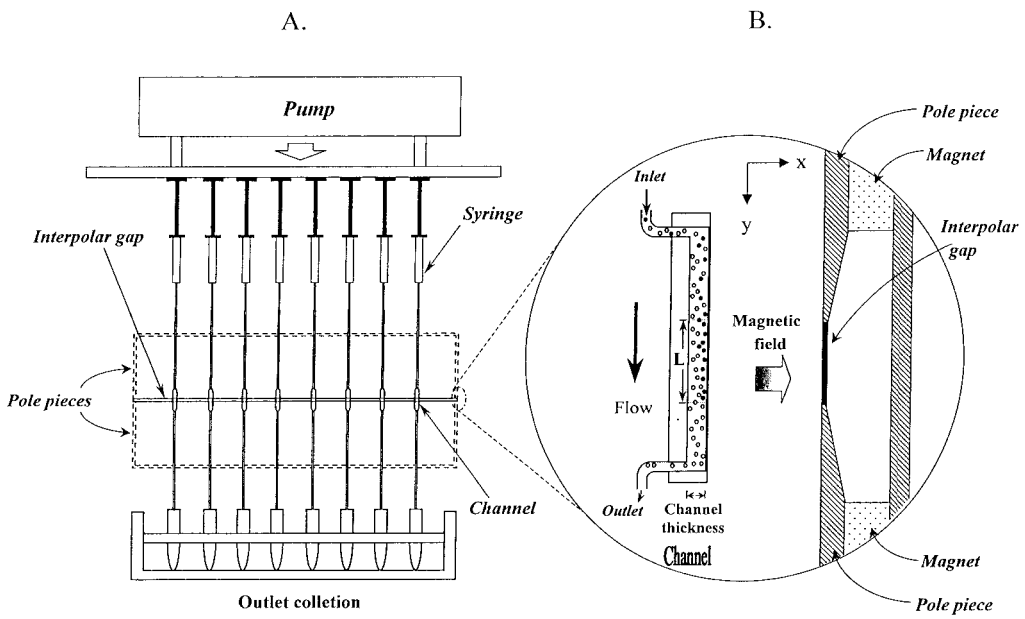

Figure 1. Front and side views of analytical magnetapheresis setup with multiple channels: (A) front view; (B) side view.

oped a method for determining particle magnetic susceptibility based on balancing channel flow rates and magnetically induced flow rates for deposition in analytical magnetapheresis. Several magnetically susceptible particles, ion-labeled yeasts, and silica particles were studied using this method.

THEORY

Particles in the channel are subject to three transport processes. The first is the flow that carries the particles moving along the channel axis at linear flow velocity v. The second is the field-induced migration that moves the particles perpendicular to the flow axis at velocity U. The third is Brownian motion that is negligible for micrometer-sized particles. We confine our discussion to micrometer-sized particle samples in this study. Therefore, the balance between the first and the second transport processes determines whether particles are deposited on the interpolar gap. Magnetic depositions occur only when the magnetically field-induced velocity is large and not exceeded by the flow velocity. Here, we assume the channel stream to be divided into a series of thin laminae with thickness equal to dx. The time needed for a particle with lateral velocity U to cross a single lamina is

$$\mathrm{d}t = \mathrm{d}x/U \tag{1}$$

The particle is transported along the stream for a distance of dy in the same time period, where

$$\mathrm{d}y = v\ \mathrm{d}t = v(\mathrm{d}x/U) \tag{2}$$

The volumetric flow rate passing along the lamina is given by

$$\mathrm{d}\dot{V} = bv\ \mathrm{d}x \tag{3}$$

where b is the channel breadth.

Substitution of eq 3 into eq 2 yields

$$\mathrm{d}y = \mathrm{d}\dot{V}/bU \tag{4}$$

This equation shows that the incremental distance dy gained along the flow axis is linear to the volumetric flow rate d$\dot{V}$ across which the particles have simultaneously migrated. Clearly, the summation of all dy under the magnetic field yields the length of the interpolar gap width L, as shown in Figure 1. At this distance, particles migrate to their final positions, which determine whether they are deposited on the gap. This depends on the volumetric flow rate elements crossed during the transport process, which is obtained by summing up all small d$\dot{V}$ to give $\Delta\dot{V}$:

$$\Delta\dot{V} = bLU \tag{5}$$

where $\Delta\dot{V}$ is the field-induced flow rate, L is the interpolar gap width, b is the channel breadth, and U is the field-induced velocity.

For particles to be transported across a lamina of flow rate $\dot{V}_i$ and deposited on the interpolar gap under magnetic driving force, the magnetically induced flow rate (bLU_m) must be larger than or equal to $\dot{V}_i$, as the following equation shows:

$$bLU_\mathrm{m} \geq \dot{V}_i \tag{6}$$

where U_m is the magnetically induced velocity. The U_m can be calculated using[13,14]

$$U_\mathrm{m} = \Delta\chi\Delta H^2 d/48\eta \tag{7}$$

where $\Delta\chi = \chi_\mathrm{p} - \chi_\mathrm{c}$, χ_p and χ_c are the respective magnetic susceptibilities of particles and carriers, η is the fluid viscosity, d is the spherical particle diameter or the effective spherical particle diameter, and ΔH is the drop in magnetic field strength.

Channel flow rates were increased progressively to maximum under complete particle deposition conditions. The maximal channel flow rate was set equal to the magnetically induced flow

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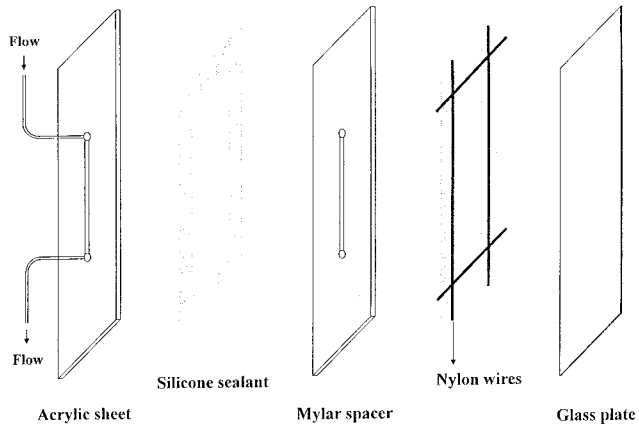

Figure 2. Diagram of analytical magnetapheresis channel components.

rate to determine particle magnetic susceptibilities, expressed in the following equation.

$$bLU_m = \dot{V}_{max} \tag{8}$$

Substitution of eq 7 into eq 8 yields

$$\Delta\chi = 48\eta\dot{V}_{max}/\Delta H^2 bLd \tag{9}$$

Therefore, particle magnetic susceptibilities were obtained from known carrier magnetic susceptibilities and given the remaining experimental parameters (b, L, $\dot{V}_{max}$, ΔH^2, η, d) in the study.

EXPERIMENTAL SECTION

The channel length, breadth, and thickness used were 1.0, 0.1, and 0.025 cm, respectively. The calculated void volume was 0.0025 mL. The injection volume was 0.02 mL. The channel components from the top to the bottom consisted of a plastic sheet, Mylar spacer, Nylon wires, and glass plate. The channel consisted of one layer of cutout Mylar. Nylon wires with a thickness of 3 μm were placed between the spacer and a glass plate for easy opening of the channel after magnetapheresis. The layers were then sandwiched together with silicone sealant to make a better seal between the plastic sheet and the glass plate, which served as the channel walls. The bottom plate, made of thin (150 μm) glass was used for particle depositions. A diagram of the separation channel is shown in Figure 2.

A permanent magnet assembly consisting of one pair of rare earth magnets (neodymium−iron−boron, Nd−Fe−B) was used to build magnetic fields. The magnets were connected by soft iron pole pieces, which conducted the magnetic flux lines to the interpolar gap. Nd−Fe−B magnets are characterized by a maximum energy product of 3.00×10^7 G·Oe, and ours were obtained from the Super Electronics Co., (Taipei, Taiwan). A gap width of 5 mm was used for all experiments, and the saturation field B_0 was 1.22×10^4 G. The gap width and channel breadth corresponded with the deposition boundary. The gap length was 10 cm. Magnetic field measurements were made using a gaussmeter with a Hall effect probe (model Gauss MG-7D, Walker Scientific Inc., Worcester, MA) with adjustable microstages. Adjustable microstages were important for determining magnetic field strengths, which varied exponentially with distances close to the interpolar gap. The probe measured magnetic flux perpendicular to a sensing area with a diameter of 6.94 mm. The combined magnets and pole pieces had a geometrical size of 17.5 cm × 10 cm × 6.0 cm and weighed 5.5 kg. The distances between channel and magnetic gap were optimized for various particles at various flow rates and magnetic susceptibilities. Mylar of various thicknesses was used to adjust the separation distances for different magnetic field strengths.

The following steps were used to operate magnetapheresis: (1) Magnetapheresis channels were held steadily on the interpolar gap of pole pieces, as shown in Figure 1. (2) Collection tubes were put at the end of channel outlets. (3) Syringes with fixed volume of sample solutions were put into microsyringe pump. (4) Microsyringe pump were turned on for magnetapheresis.

Reference magnetic susceptibility measurements were made using an MPMS5 model superconducting quantum interference device (SQUID) magnetometer from Quantum Design (San Diego, CA). SQUID magnetic field strengths used for susceptibility measurements ranged from 1.0×10^4 to 2.0×10^4 G. The cgs system and volume magnetic susceptibility, χ, are used throughout this study for convenient calculation unless otherwise indicated.

Particle verifications and countings were done under light microscopy (Olympus BX-50, Tokyo, Japan). At least 200 particles were used for injection, and a 3% error rate was assumed in complete-deposition particle counting experiments. A multichannel microsyringe pump (model KDS 220, KD Scientific Inc., Boston, MA) was used to deliver samples at controllable flow rates through the separation channel under magnetic fields. A hemacytometer under the microscope was used to count particles and to calculate particle concentrations at the channel inlet and outlet. Phosphate-buffered solutions with pH of 7.02 and viscosities η equal to 1.0×10^{-2} g cm^{-1} s^{-1} were used as carriers in this study. Molybdenum particles (<2 μm), and iron nitrate were purchased from Sigma Chemical Co. (St. Louis, MO). Silica particles (<9 μm) were from Hypersil (Cheshire, England). Erbium chloride was obtained from Strem Chemicals Co. (Newsburyport, MA). Dynabeads M-450 of size 4.5 μm were obtained from Dynal Inc., (Lake Success, NY). Copper(II) oxides (<7 μm), chromium(III) oxide (1−2 μm), tungsten(IV) sulfide (1−2 μm), titanium oxide (<3 μm), and iron oxide (<7 μm) particles were from Aldrich Chemicals Co. (St. Louis, MO). Yeasts were from a bakery store in a nearby market. Particle size distributions were determined using an Analysette 22 model laser particle sizer from Fritsch GmbH (Idar-Obstein, Germany).

Erbium ion labels were prepared by mixing 1 mL of 1 mM labeling ions with a 9-mL solution containing ~9.0×10^6 particles for 1 h and shaking every 15 min unless otherwise specified. Labeling ion concentrations were changed systematically for silica and yeast samples in ion-labeled experiments. All ion-labeled particles were washed three times with buffer solution before use to remove unlabeled ions.

RESULTS AND DISCUSSIONS

The new channel assembly described in the Experimental Section provides a better seal capacity and easier bottom-plate disassembly (without breaking the glass plate) after magnetapheresis than earlier assembly.[4] Silicone sealant provides a better seal and can be loosened easily by twisting the embedded Nylon

Table 1. Determined Magnetic Susceptibilities of Fe_2O_3 and Dynabead Particles at Various Distances from Analytical Magnetapheresis[a]

particle	distance between channel and magnetic gap (nm)	ΔH^2 (g/cm^2·s^2))	flow rate $\dot{V}_{max}$ (μL/min)	$\Delta\chi \pm$ SD[b] ($n = 5$)
Fe_2O_3	45.15	89.0	9.8 ± 0.6	0.027 ± 0.004
	55.15	45.6	5.0 ± 0.5	0.027 ± 0.004
	65.15	25.4	2.8 ± 0.2	0.027 ± 0.004
	75.15	15.0	1.7 ± 0.2	0.028 ± 0.005
	85.15	9.2	1.0 ± 0.1	0.026 ± 0.004
Dynabeads	25.15	376.5	22.0 ± 0.5	0.021 ± 0.001
	35.15	135.4	8.0 ± 0.6	0.021 ± 0.002
	45.15	60.8	3.5 ± 0.4	0.020 ± 0.002
	55.15	31.2	1.8 ± 0.2	0.021 ± 0.002
	65.15	17.4	1.0 ± 0.1	0.020 ± 0.002

[a] Susceptibility from SQUID measurement: Fe_2O_3, 0.029 ± 0.001; Dynabeads, 0.020 ± 0.001. [b]SD, standard deviation.

Table 2. Determined Magnetic Susceptibilities of Various Particles from Analytical Magnetapheresis

particle	distance between channel and magnetic gap (mm)	size (μm)	ΔH^2 (g/(cm^2·s^2))	$\dot{V}_{max}$ (μL/min)	$(\Delta\chi \pm$ SD)[a] $\times 10^3$ ($n = 5$)	SQUID $(\Delta\chi \pm$ SD)[a] $\times 10^3$ ($n = 10$)
Dynabead	35.15	4.50 ± 0.05	135.30	8.0 ± 0.6	21 ± 2	20 ± 1
Fe_2O_3	65.15	6.59 ± 0.79	25.40	2.8 ± 0.2	27 ± 4	29 ± 1
Fe	90.15	3.76 ± 0.49	4.17	1.4 ± 0.1	143 ± 21	146 ± 3
Cr_2O_3	0.15	1.56 ± 0.50	58016	7.22 ± 0.08	0.13 ± 0.04	0.14 ± 0.01
CuO	0.15	6.21 ± 0.63	230754	21.1 ± 0.2	0.024 ± 0.002	0.022 ± 0.001
Mo	0.15	1.16 ± 0.35	43144	0.43 ± 0.06	0.014 ± 0.005	0.012 ± 0.001
WS_2	0.15	1.59 ± 0.54	59132	0.59 ± 0.01	0.010 ± 0.003	0.009 ± 0.001
TiO_2	0.15	2.92 ± 0.63	108568	0.040 ± 0.001	0.00020 ± 0.00004	0.00023 ± 0.00001

[a] SD, standard deviation.

wires at both ends after magnetapheresis. This improved channel assembly allowed experimental results of analytical magnetapheresis to be very consistent from run to run.

A new method for determining particle magnetic susceptibility was evaluated using several magnetic particles and erbium ion-labeled particles. Particles with high magnetic susceptibilities, such as Fe_2O_3 and Dynabeads, were used to test this new method at various magnetic field strengths, as shown in Table 1. Susceptibilities determined through analytical magnetapheresis at various magnetic field strengths were quite consistent and within 8% of reference SQUID measurements. The relative standard deviation (RSD) of total $\Delta\chi$ measurements were 8.8 and 15.5% for Dynabeads and Fe_2O_3, respectively. The 95% confidence limits of total $\Delta\chi$ measurements were 0.027 ± 0.005 and 0.021 ± 0.002 for Fe_2O_3 and Dynabeads, respectively. The standard errors of means on symbol $\Delta\chi$ measurements were 0.002 and 0.01 for Fe_2O_3 and Dynabeads, respectively. Several particles with various magnetic susceptibilities were examined using this new method. The determined susceptibilities of these magnetic particles were with an average of 9.1% different from reference SQUID measurements, as shown in Table 2. Particles with narrow size distribution showed smaller standard deviations in determined susceptibilities than particles with wider size distribution. The mean RSD of total $\Delta\chi$ measurements were 5.7 and 15% for SQUID and magnetapheresis, respectively. The 95% confidence limits of total symbol $\Delta\chi$ measurements were mean ± 0.72 SD and mean ± 1.24 SD for SQUID and magnetapheresis, respectively. The magnetic susceptibilities of erbium ion-labeled silicas at various labeling concentrations for complete deposition were also determined with this new method, as shown in Table 3. The mean RSD of total symbol $\Delta\chi$ measurements were 8.0%. The 95% confidence limits of total $\Delta\chi$ measurements were mean ± 1.24 SD for each labeling ion concentration. Labeling ion concentrations were varied systematically for susceptibility determination. Table 3 shows that the saturation of labeling ion concentration is 0.20 M for 9.0×10^6 particles and that the minimum susceptibility that can be determined using this method is $\sim 5.7 \times 10^{-9}$. The minimum number of erbium labeling ions per particle required for complete deposition of silicas was 6.7×10^9, as calculated by dividing 1.0×10^{-7} M labeling ion by 9.0×10^6 particles. All ions were assumed to be attached to particle surfaces. The determined susceptibilities of erbium ion-labeled yeasts at various labeling concentrations are shown in Table 4. The mean RSD of total symbol $\Delta\chi$ measurements were 9.0%. The 95% confidence limits of total $\Delta\chi$ measurements were mean ± 1.24 SD for each labeling ion concentration. The saturation of labeling ion concentration was 0.10 M for 9.0×10^6 particles, and the minimum susceptibility that can be determined from this method is $\sim 2.7 \times 10^{-9}$. The minimum number of erbium labeling ions per particle required for complete deposition of yeasts was 6.7×10^9, the same as that for silicas. The results shown in Tables 3 and 4 are very consistent for all labeling ion concentrations. The minimum susceptibility that can be determined from this new method is on the order of 10^{-9} cgs.

Determined magnetic susceptibilities of various particles in carriers with different susceptibilities are shown in Table 5. The effects of reduced $\Delta\chi$ values (relative magnetic susceptibility changes between particles and carrier) on particle susceptibility determinations are clear. Particles with high magnetic susceptibilities (Fe, Fe_2O_3, Dynabeads) showed little change in determined susceptibilities from differing carrier susceptibility (reduced $\Delta\chi$)

Table 3. Determined Magnetic Susceptibilities of Er^{3+}-Labeled Silicas at Various Labeling Concentrations from Analytical Magnetapheresis

	ion concn (M)	size (μm)	ΔH^2 (g/(cm^2·s^2))	$\dot{V}_{max}$ (mL/min)	($\Delta\chi \pm$ SD)[a] $\times 10^6$ ($n = 5$)
	0.25			2.20 ± 0.10	1557 ± 107
	0.2			2.20 ± 0.10	1557 ± 107
	0.15			1.80 ± 0.20	1274 ± 156
	0.1			1.20 ± 0.10	850 ± 83
silicas	1.0×10^{-2}	7.8 ± 0.4	289 750	0.72 ± 0.03	510 ± 34
	1.0×10^{-3}			0.56 ± 0.02	396 ± 25
	1.0×10^{-4}			0.26 ± 0.01	184 ± 12
	1.0×10^{-5}			0.11 ± 0.01	78 ± 8
	1.0×10^{-6}			0.0065 ± 0.0002	4.60 ± 0.28
	1.0×10^{-7}			0.000008 ± 0.000001	0.006 ± 0.001
	1.0×10^{-8}			0.000 ± 0.000	0.00 ± 0.00

[a] SD, standard deviation.

Table 4. Determined Magnetic Susceptibilities of Er^{3+}-Labeled Yeasts at Various Labeling Concentrations from Analytical Magnetapheresis

	ion concn (M)	size (μm)	ΔH^2 (g/(cm^2·s^2))	$\dot{V}_{max}$ (mL/min)	($\Delta\chi \pm$ SD)[a] $\times 10^6$ ($n = 5$)
	0.15			0.24 ± 0.01	646 ± 55
	1.0×10^{-1}			0.24 ± 0.01	646 ± 55
	1.0×10^{-2}			0.102 ± 0.002	274 ± 21
	1.0×10^{-3}			0.035 ± 0.002	94 ± 9
yeasts	1.0×10^{-4}	4.0 ± 0.3	148 694	0.009 ± 0.001	24 ± 3
	1.0×10^{-5}			0.0012 ± 0.0001	3.2 ± 0.4
	1.0×10^{-6}			0.0000018 ± 0.0000001	0.0048 ± 0.0005
	1.0×10^{-7}			0.0000010 ± 0.0000001	0.0027 ± 0.0003
	1.0×10^{-8}			0.00 ± 0.00	0 ± 0

[a] SD, standard deviation.

Table 5. Determined Magnetic Susceptibilities of Various Particles in Carriers with Different Magnetic Susceptibilities from Analytical Magnetapheresis[a]

			buffer solution		$MnSO_4$ solution	
particle[b]	size (μm)	ΔH^2 (g/(cm^2·s^2))	$\dot{V}_{max}$ (μL/min)	($\Delta\chi \pm$ SD) $\times 10^3$ ($n = 5$)	$\dot{V}_{max}$ (μL/min)	($\Delta\chi \pm$ SD $\times 10^3$ ($n = 5$)
Dynabead[a]	4.50 ± 0.05	135.30	8.0 ± 1.0	21 ± 3	8.0 ± 1.0	21 ± 3
Fe_2O_3[b]	6.59 ± 0.79	25.40	2.8 ± 0.2	27 ± 4	2.8 ± 0.2	27 ± 4
Fe[c]	3.76 ± 0.49	4.17	1.4 ± 0.1	143 ± 21	1.4 ± 0.1	143 ± 21
Cr_2O_3[d]	1.56 ± 0.50	58016	7.22 ± 0.08	0.13 ± 0.04	4.40 ± 0.08	0.078 ± 0.025
CuO[d]	6.21 ± 0.63	230754	21.1 ± 0.2	0.024 ± 0.002		
Mo[d]	1.16 ± 0.35	43144	0.43 ± 0.06	0.014 ± 0.005		
WS_2[d]	1.59 ± 0.54	59132	0.59 ± 0.001	0.010 ± 0.003		
TiO_2[d]	2.92 ± 0.63	108568	0.040 ± 0.001	0.00020 ± 0.00004		

[a] Susceptibility from SQUID measurement: $MnSO_4$ solution, 4.17×10^{-5}; buffer solution, -4.0×10^{-8}. [b] Distance between channel and magnetic gap (mm): a = 3.15, b = 65.15, c = 90.15, and d = 0.15.

effects. Determined chromium oxide (Cr_2O_3) susceptibility changed more markedly than that of other particles because its susceptibility was close to the $\Delta\chi$ values. The differences in determined particle magnetic susceptibilities of Cr_2O_3 in different carriers are consistent with the carrier susceptibility difference. Particles with magnetic susceptibilities smaller than that of the $MnSO_4$ carrier cannot be determined using this new method. The results in Table 5 indicate the consistency of this new method for particle susceptibility determination.

Extreme temperature could affect the susceptibility measurement. Susceptibility values were quite constant at room-temperature ranges, as in our experiments. The flow rate stability was very important to the susceptibility determination in this experiment. Our flow rate varied from 1 to 11% with an average of 7% variations. Sample concentration larger than 1.0×10^6 mL^{-1} could cause sample interaction problems in this study.

Experimental running times of analytical magnetapheresis were short and the whole measurement generally took less than 20 min once magnetic field strengths were known. Magnetic field strengths only needed to be measured once for fixed gap widths. The minimal magnetic susceptibility we could determine was ~1.0×10^{-9} cgs. Particles with higher magnetic susceptibilities would have higher maximal flow rates for complete depositions and, thus, greater speed in determining particle susceptibilities.

The new method does not require laborious calibration plots and trial susceptibility values to determine particle magnetic susceptibility as do previous methods in analytical magnetapheresis. This new method can also determine lower values of particle magnetic susceptibility. Multichannel analytical magnetapheresis setups are preferred for comparative experiments and multisample particle susceptibility determination. This new method for determining particle magnetic susceptibility will certainly not replace or compete with standard reference methods, such as the SQUID measurement, in terms of accuracy, measuring ranges, controllable temperatures, and field strengths. However, the instrumentation and running costs of SQUID are vastly higher than that of analytical magnetapheresis. This new method of analytical magnetapheresis should be very attractive and promising to provide a simple, rapid, and economical method for obtaining particle magnetic susceptibilities.

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