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Tung-yu Ying[a]; F. Coyne Prenger[a]; Laura A. Worl[b]; Michael D. Johnson[c]; Joseph A. Waynert[a]; Robert M. Wingo[a]

[a] Engineering Sciences and Applications Division, Los Alamos National Laboratories, Los Alamos, New Mexico, USA [b] Nuclear Materials Technologies Division, Los Alamos National Laboratories, Los Alamos, New Mexico, USA [c] Department of Chemistry and Biochemistry, New Mexico State University, Las Cruces, New Mexico, USA

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A Novel Magnetic Separation Technique: Selective Separation of Ultrafine Particles by Magnetophoresis

Tung-yu Ying,[1,*] F. Coyne Prenger,[1] Laura A. Worl,[2]
Michael D. Johnson,[3] Joseph A. Waynert,[1]
and Robert M. Wingo[1]

[1]Los Alamos National Laboratories, Engineering Sciences and
Applications Division, Los Alamos, New Mexico, USA
[2]Los Alamos National Laboratories, Nuclear Materials Technologies
Division, Los Alamos, New Mexico, USA
[3]Department of Chemistry and Biochemistry, New Mexico State
University, Las Cruces, New Mexico, USA

ABSTRACT

The selective and specific extraction of species of interest from local environmental and other sample sources are important for scientific research, industrial processes, and environmental applications. A novel process for selective separation of magnetic particles using magnetophoresis is investigated. The principle of this process is that the direction

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[*]Correspondence: Tung-yu Ying, Los Alamos National Laboratories, Los Alamos, NM 87545, USA; E-mail: tying@lanl.gov.

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and velocity of particle movement in a magnetic field gradient are determined by magnetic, gravitational, and drag forces. By selectively controlling these forces, it is possible to control the mobility of the different species and, therefore, magnetically fractionate mixtures of them into discrete groups. This study demonstrated the selective separation of various species, such as iron (III) oxide and cupric (II) oxide. Experimental results showed that around 70–80% of iron was selectively collected in the upper part of the separation media and around 90% of copper was concentrated in the lower part of the media. A magnetophoresis model was developed in this work to provide a theoretical understanding of this phenomenon and to assist in the design of magnetophoresis experiments.

Key Words: Magnetic separation; Magnetophoresis; Microparticles; Magnetic particles.

INTRODUCTION

Magnetic separation has been shown to be a promising separation process for industrial wastewater treatment and biotechnology applications.[1–4] In conventional high-gradient magnetic separation (HGMS), suspensions flow through a ferromagnetic matrix and subsequently the paramagnetic particles are collected in a capture–no capture scenario. Only a few studies investigated the utilization of HGMS as a selective separation technique.[5,6] In this article, a novel process using magnetophoresis is therefore investigated to selectively separate particles of interest from local environmental and other sample sources.

Magnetophoresis is analogous to electrophoresis,[7] and has been studied by many researchers in biotechnology applications for separation of proteins, DNA, and blood cells.[8–12] Most of the particles or biologic cells examined in those literatures were in micrometer-scale. The magnetophoresis mobility of a particle is determined by its physical properties, such as density and magnetic susceptibility. Because of the difference in magnetophoresis mobility between species, it is possible to magnetically fractionate mixtures of species into discrete groups. The selectivity and specific extraction of identified species of interest are important for industrial processes and environmental applications, especially those involving radiological and biological materials.

Additionally, magnetophoresis is also a nondestructive method for selectively collecting magnetic particles. For example, ion exchange can separate dissolved species in a specific state, which may require pretreatment methods. In magnetophoresis, fractionation of specific particles is conducted without chemically altering the crystal morphology, oxidation state, or speciation of the targeted particles.

In our previous study,[13] we examined the performance of magnetophoresis of various oxide particles, such as hematite (Fe_2O_3), samarium oxide (Sm_2O_3), and copper oxide (CuO) using agarose gels as separation media, which are the preferred media for electrophoresis. Our experimental results showed that oxide particles were able to magnetically migrate through agarose media. The migration distance was, however, limited by the media structure and did not distinguish between test species. In this article, we investigated the selective separation of ultrafine hematite (Fe_2O_3) and copper oxide (CuO) particles using a viscous fluid as separation media. The effects of magnetic field strength, particle size, and magnetic susceptibility on the performance of magnetophoresis were experimentally and theoretically examined. In addition, a force-balance model was developed to provide a theoretical understanding of this phenomenon and to assist in the design of magnetophoresis experiments. Two model parameters, the magnetophoresis mobility factor and the migration velocity, were defined to correlate the effectiveness of the magnetophoresis.

MODEL DEVELOPMENT

The operating principle of magnetophoresis is that the direction and velocity of particle movement in a magnetic field gradient are determined by magnetic (F_m), gravitational (F_g), and drag (F_d) forces. By selectively controlling these forces, it is possible to control the mobility of the different species and, therefore, magnetically fractionate mixtures of them into discrete groups. If the analysis is limited to the motion of small particles (<75 μm), the inertial force can be neglected and the force balance equation is written as

$$\vec{F}_m + \vec{F}_g + \vec{F}_d = 0 \tag{1}$$

where (1)

$$\vec{F}_m = \frac{4}{3\mu_0}\pi b^3 \chi |B| \nabla \left|\vec{B}\right| \tag{2a}$$

$$\vec{F}_g = \frac{4}{3}\pi b^3 \left(\rho_p - \rho_f\right)\vec{g} \tag{2b}$$

$$\vec{F}_d = 6\pi\eta b\vec{U} \tag{2c}$$

In Eqs. (2a)–(2c), b is the particle radius (m); χ is the difference between the volume magnetic susceptibility of the particle and fluid; B is magnetic induction (Tesla); μ_0 ($=4\pi \times 10^{-7}$ H/m) is the permeability of free space; ρ_p and ρ_f are the density of the particle and separation media, respectively

$(\mathrm{Kg/m^3})$; η is the dynamic viscosity (Kg/m-s); and U is the particle migration velocity (m/s).

By inserting Eqs. (2a)–(2c) into Eq. (1), the migration velocity can be obtained,

$$\vec{U} = \frac{2b^2\chi|B|\nabla\left|\vec{B}\right|}{9\eta\mu_0} + \frac{2b^2\left(\rho_p - \rho_f\right)\vec{g}}{9\eta} \tag{3}$$

Eq. (3) considers the magnetic field and field gradient generated by the magnet only, and the interactions between particles are not included. The first term in Eq. (3) is the magnetic velocity due to the applied magnetic field gradient and the second term is the settling velocity due to gravity. Eq. (3) shows that the migration velocity is strongly dependent on particle size. For the case where the magnetic field gradient and the body force are in opposite directions, as in the experiments conducted in this article, the magnetic force is in the opposite direction of the gravitational force. If the magnetic force is greater than the gravitational force, the particles will migrate upwards (against gravity), and vice versa.

The magnetophoresis mobility factor, Y, is defined as the ratio of magnetic to gravitational forces acting on a particle or

$$Y = \frac{\vec{F}_m}{\vec{F}_g} = \frac{\chi|B|\nabla\left|\vec{B}\right|}{\mu_0\left(\rho_p - \rho_f\right)\vec{g}} \tag{4}$$

The product of $B\nabla B$ in Eq. (4) is not a constant, and its value is determined by its position in a magnetic field gradient. Three operating regimes in the magnetophoresis process is, therefore, defined by Eq. (4): (i) the regime where the magnetic force is the dominant force ($Y > 1$) and particles will migrate upwards; (ii) the regime where magnetic and gravitational forces are nearly equal ($Y = 1$) and particles will remain stationary; and (iii) the regime where the gravitational force is the dominant force ($Y < 1$) and particles will move downwards. In addition, the greater the absolute value of Y, the further the particles are able to move away from their original position in a given amount of time. It should be noted that the magnetophoresis mobility factor (Y) is only dependent on the properties of a species, such as magnetic susceptibility and density, and the product of $B\nabla B$, but independent of particle size. It means regardless of its size, a particle is able to reach the point where magnetic and gravitational forces are equal ($Y = 1$), while the time needed to reach that point is strongly dependent on the particle size.

The dimensionless number Y can be also used as an indication of separation effectiveness by magnetophoresis. For example, Table 1 lists the Y values of several oxide species. By arbitrarily choosing the Y value of hematite

Table 1. Magnetophoresis mobility factor, Y, of various oxides.

Oxide name	Density (g/cm^3)	Magnetic susceptibility ($\times 10^{-6}$)	$Y\,(=F_m/F_g)$[a]
Er_2O_3	8.64	20,979.1	4.22
Mn_2O_3	4.50	5,050.59	2.22
Fe_2O_3	5.24	2,760	1.00
Co_2O_3	6.07	2,337.75	0.71
Eu_2O_3	7.42	2,676.03	0.64
PrO_2	6.82	956.6	0.25
NiO	6.67	740.46	0.20
UO_2	10.96	1,203.7	0.19
CuO	6.40	264	0.08
PuO_2	11.46	383.68	0.06
CeO_2	7.13	13.54	0.006

[a] The Y value of Fe_2O_3 is chosen as 1.00. A reference value of $(B\nabla B)$ in Eq. 4 can be obtained and used for all the other Y values.

as one, a reference value for $(B\nabla B)$ in Eq. (4) can be obtained, and used for all the other Y values. As shown in Table 1, the Y of copper oxide is approximately 10 times less than the value of hematite. Therefore, it is expected that an effective separation between these two species can be achieved by magnetophoresis. On the other hand, the difference of the Y values between hematite and cobalt oxide (Co_2O_3) is small, and the separation of these two species would be more difficult.

Furthermore, the force balance model developed in this work can be used to help design magnetophoresis experiments, such as determining the magnetic field strength, the sample placement, and separation media characteristics. Figure 1(a) shows the lower quadrant of the magnetic field profile inside the superconducting solenoid magnet on the axis. As shown in Fig. 1, the magnetic field is very uniform in the center part of the coil, and starts decreasing away from the midplane to form a magnetic field gradient. The magnetic force, however, not only depends on magnetic field strength B, but on the product of $(B\nabla B)$ as shown in Eq. (2a). Figure 1(b) shows the magnetic force profile inside the lower quadrant of the magnet. It is clear that the magnetic force reaches its maximum about 100 mm from the magnet midplane.

To achieve an effective separation between two species, it is necessary to find a location, where one species will migrate upward and the other downward. In the case of separation of hematite and copper oxide, as shown in Fig. 1(b), the region where the magnetic force dominates for

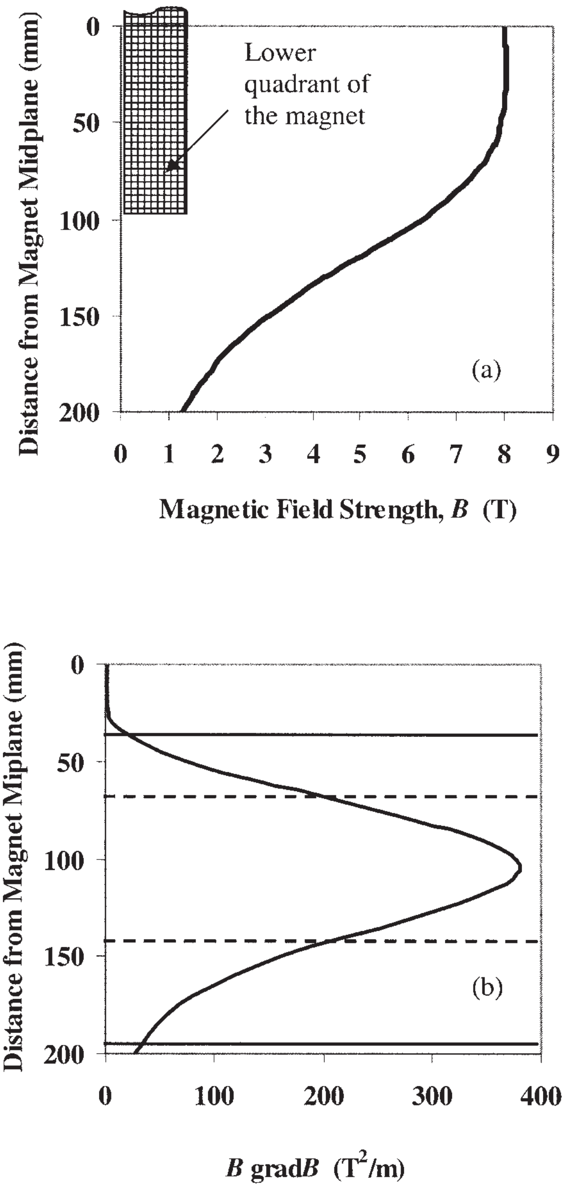

Figure 1. (a) Magnetic field profile of the lower quadrant magnet. (b) Magnetic force profile of the lower quadrant magnet. The region where the magnetic force dominates for hematite is indicated by the solid horizontal lines and for copper oxide by the dashed lines.

hematite is indicated by the solid horizontal lines and for copper oxide by the dashed lines. Therefore, in order to achieve an effective separation, the sample mixture needs to be placed outside the dash lines and inside the solid lines.

Two orientations of magnetophoresis experiments were examined. The first one is the top-down orientation, in which the species of higher mobility factor will stay on top, and the other will move down due to gravity. The other is bottom-up orientation, where the species of higher mobility factor will migrate up due to magnetic force, and the other will stay on the bottom. In both cases, the sample placement in the magnet chamber and the magnetic field strength were determined by the model.

MATERIALS AND METHODS

Magnetophoresis experiments were conducted with two types of particles, hematite (Fe_2O_3) and copper oxide (CuO) (CERAC, Milwaukee, WI). Particle size measurements of these two oxides were determined by a Horiba LA 900 particle size analyzer (Kyoto, Japan). The mean diameters for hematite and copper oxide were 0.41 (± 0.32) μm and 0.77 (± 0.37) μm, respectively. Pure glycerol (Sigma-Aldrich, Milwaukee, WI) was diluted with deionized water to various concentrations and used as separation media. The pH of both particle suspensions and dilute glycerol solutions was adjusted to approximately 11 by NaOH (1M) solution.

The experimental setup is shown in Fig. 2. The dilute glycerol solution was first introduced into a 2-cm ID column to form a 50-mm long separation media. Then approximately 1-mL sample mixture was introduced at either the top or bottom of the separation media, depending on the predicted migration for the experiment. The column was then attached to a hanger and placed inside the magnetic coil. The low temperature, superconducting solenoid magnet (Cryomagnetics Inc., Oak Ridge, TN) used in this work generated a maximum magnetic field between 0.1 and 8 Tesla. The applied field, sample placement, glycerol solution concentration, and experiment time were determined from the magnetophoresis model.

For each experiment, photos were taken at the end of the experiment to give a visual comparison with the model predictions. After the experiment, the column was immediately immersed into liquid nitrogen to freeze the separation media and thus immobilize the particulates. The frozen media was then cut into several fractions along the axis and digested for ICP analysis. A Thermo Jarrell Ash ICP-OES (Franklin, MA) was used to determine the metals distribution within the separation media.

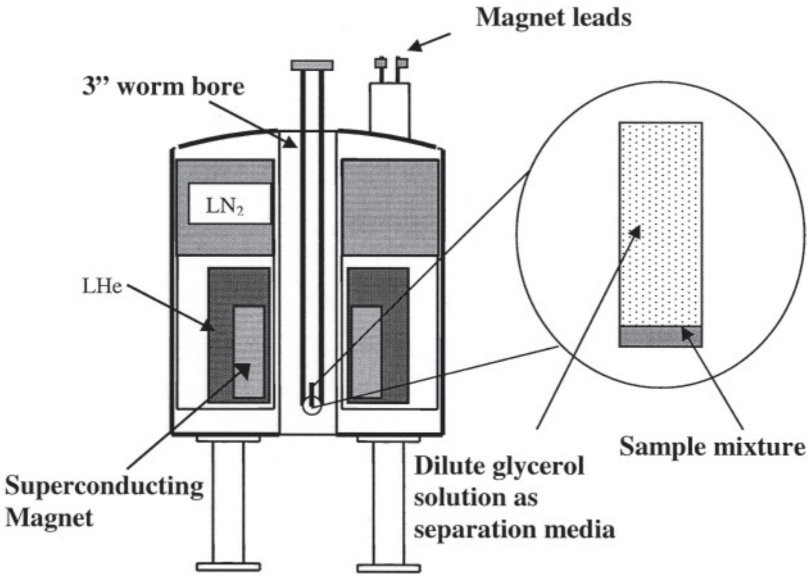

Figure 2. Schematic of the experimental apparatus.

RESULTS AND DISCUSSION

The analytical model was used to evaluate the effects of magnetic field strength and particle size on the separation efficiency. Visual comparisons between photo images of experiment results and model predictions are also presented. Experimental results for separation of hematite and copper oxide particles by magnetophoresis in both a top-down and bottom-up orientation under various magnetic field strengths are presented. Finally, the effect of particle agglomeration on separation performance is discussed.

Model Results

The effects of magnetic field strength on the separation efficiency of hematite and copper oxide particles are shown in Fig. 3. To have an effective separation, the sample location was selected, such that one species migrated towards the magnet midplane (migration velocity > 0), while the other species migrated away from the midplane (migration velocity < 0). As shown in Fig. 3(a), at a magnetic field strength of 7-T, the migration velocity of 1.0 μm copper oxide particles in the region between 95 and 115 mm from

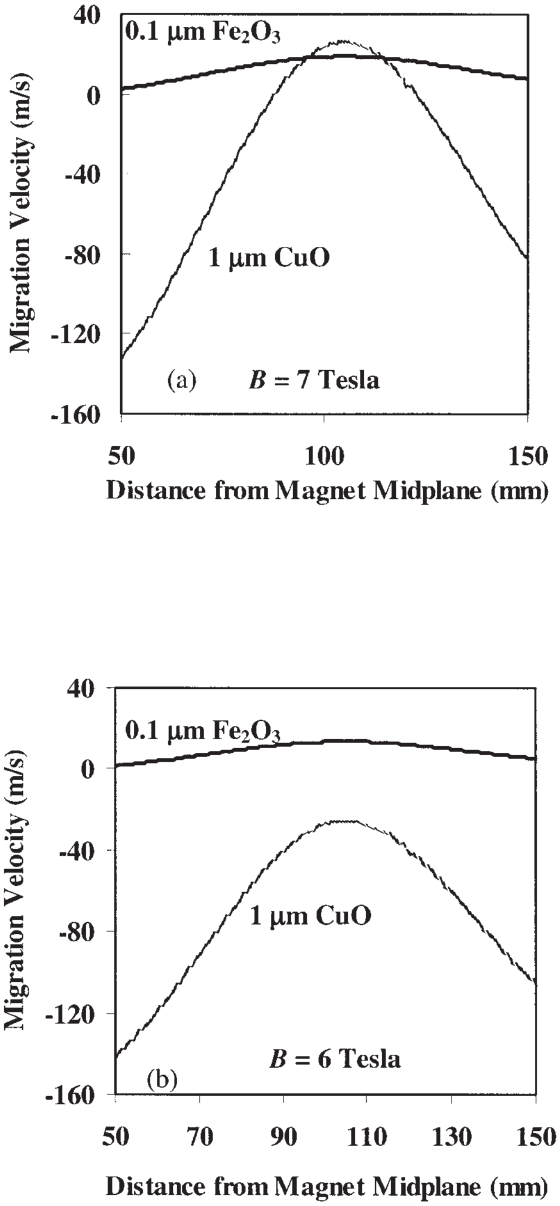

Figure 3. The effect of magnetic field strength on the separation efficiency of hematite and copper oxide particles: (a) magnetic field strength = 7 T; (b) magnetic field strength = 6 T.

the midplane is not only positive, meaning the particles will move toward the magnet midplane along with the hematite particles, but their velocity is also greater than that of 0.1 μm hematite particles. By lowering the magnetic field strength to 6-T, as shown in Fig. 3(b), the migration velocity of copper oxide is always negative, meaning the particles will move away from the magnet midplane and in a direction opposite to the hematite. It can be concluded from Fig. 3 that higher magnetic field strengths do not mean better separation performance. Instead, the magnetic field strength needs to be tailored to achieve migration in opposite directions for the species.

The migration history of hematite particles of various sizes calculated by the magnetophoresis model is shown in Fig. 4(a). It shows that the migration velocity is strongly dependent on particle size, although smaller particles will eventually move to the same final location as the larger particles because the magnetophoresis capability is independent of particle size. However, the time scale required for smaller particles to move to the final location is very large and is not practical. The magnetophoresis model was used to determine the time scale that produced a reasonable migration distance. As shown in Fig. 4(b), for example, a particle location based on size was obtained from

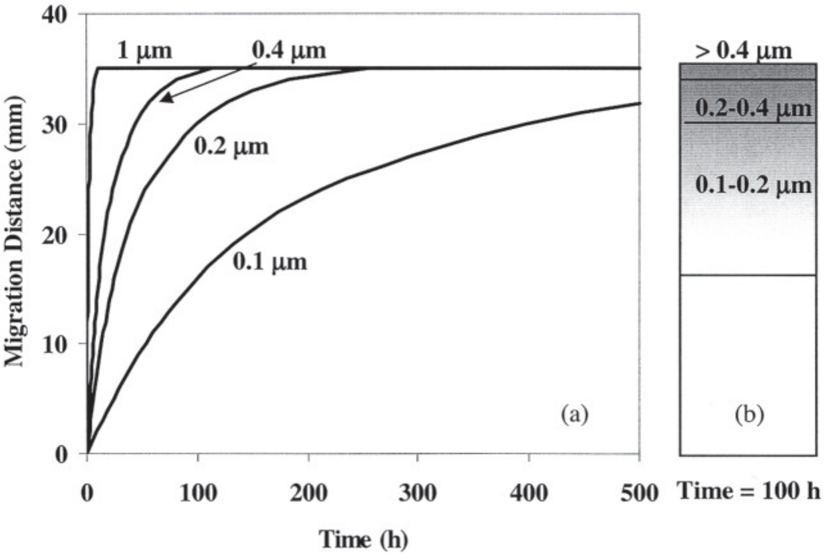

Figure 4. The effect of particle size on the effectiveness of magentophoresis: (a) the migration history of hematite particles of various sizes; (b) size distribution of hematite particles within a separation media after 100 h. Magnetic field strength = 5.5 T at the starting position of hematite; glycerol concentration = 30% by wt.

the model after 100 h, and the 0.1 μm hematite particles were predicted to migrate 15 mm from their original location. Compared to the length of the separation media (50 mm) used in the experiments, this separation distance was adequate to distinguish the two species.

Experimental Results

A comparison between experimental and model results is shown in Fig. 5 for a bottom-up magnetophoresis experiment of hematite particles. The photo images in Fig. 5(a) and (b) were taken at initial and final (time = 41 h) stages of the experiment, respectively, and the particle location based on size calculated by the model is shown in Fig. 5(c). Compared with Fig. 5(a), Fig. 5(b) shows that the color is much darker in the Top-1 section of the media, indicating a large amount of hematite particles moved to this region. The color becomes lighter in the Top-2 section, indicating smaller particles did not have time to migrate to the top. The colors in the middle and bottom sections in Fig. 5(b) both are much lighter than the two top sections, meaning only small

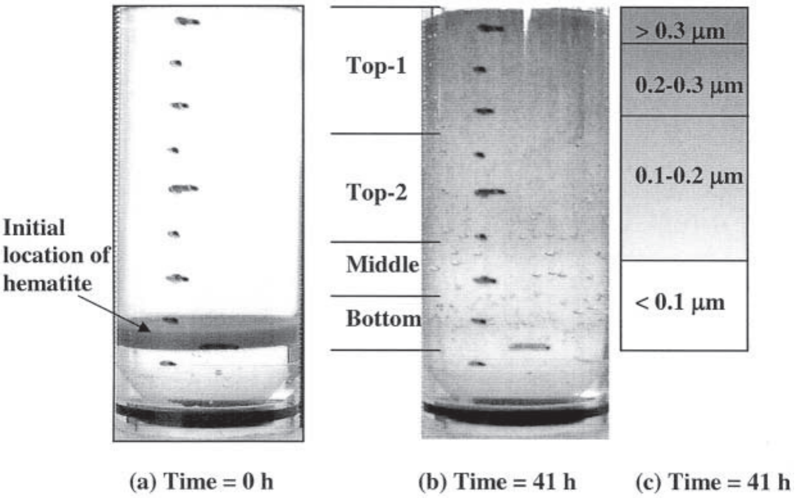

Figure 5. Comparison between experimental results and model predictions for magnetophoresis of hematite particles: (a) photo image taken at the initial stage of the experiment; (b) photo image taken at the final stage of the experiment (time = 41 h); (c) model prediction of particle size distribution within a separation media after 41 h. Magnetic field strength = 4.7 T at the starting position of hematite; glycerol concentration = 30 % by wt.

amounts of hematite particles were presented in these regions. It was observed that some particles were stuck on the wall of the column at the starting location as shown in the bottom section in Fig. 5(b). It maybe due to the interactions between the column surface and the particles. In general, the model prediction (Fig. 5(c)) agrees well with the experimental observations (Fig. 5(b)).

At the conclusion of the experiment the separation media was removed from the magnet, and was frozen and cut into four fractions for ICP analysis. The ICP results are shown in Table 2 (Exp. 1). In Table 2, the ICP results are presented in terms of distribution percentage, which is the weight of metal in each section divided by the total weight of metal in all sections. It shows that 76.13% of iron (52.27% in Top-1 section and 23.86 % in Top-2 section) was in the region as the model predicted. The rest of the iron in the middle and bottom sections was likely particle sizes smaller than 0.1 μm, as well as material that interacted with the column wall.

Table 2 also lists the ICP results of separation of hematite and copper oxide particles by magnetophoresis under various operating conditions. It should be noted that the value of magnetic field strength reported in Table 2 is the local magnetic field strength at the initial location of the sample mixture. Exp. 2 in Table 2 was conducted at the same operating conditions as Exp. 1. The initial sample placement was chosen at the position where copper oxide particles are dominated by gravity and will not migrate upward. The results in Exp. 2 show a similar iron distribution as in Exp. 1. A higher percentage of iron was, however, found in the middle and bottom sections compared with Exp. 1. An interesting result is observed in the distribution for copper (Exp. 2), in which 27.18% copper was found in the Top-1, Top-2, and Middle sections. From the given experimental conditions, the gravitational force was greater than the magnetic force on copper oxide particles in the initial placement (the bottom section). It was expected that all the copper oxide particles would remain on the bottom. The unexpected distribution of copper in the upper media was possibly due to the magnetic or chemical agglomeration between hematite and copper oxide particles.

When copper oxide particles agglomerate with hematite particles, the magnetic susceptibility and the mobility factor of the aggregate are between those of hematite and copper oxide. The aggregates are then distributed in the separation domain depending on the form of the aggregates. For example, when a hematite particle of larger size agglomerates with a copper oxide particle of smaller size, the in-situ formed aggregate may be able to migrate to the upper part of the domain. On the other hand, an aggregate with a larger portion of copper oxide may stay on the bottom or move to the middle part of the domain. This is the reason that in Exp. 2 (Table 2) more iron was left in the middle and bottom sections while some of the copper oxide moved to the upper part of the domain.

Table 2. Results of ICP analysis for magnetophoresis experiments.

	Operating conditions				Results of ICP analysis (%)[a]			
Exp. no.	Orientation	Magnetic field (T)[b]	Glycerol solutions (% by wt.)	Samples	Top-1	Top-2	Middle	Bottom
1	Bottom-up	4.7	30	Fe_2O_3	52.27	23.86	11.36	12.50
2	Bottom-up	4.7	30	Fe_2O_3	43.33	21.67	15.00	20.00
				CuO	4.35	7.61	15.22	72.82
3	Top-down	7.0	12.5	Fe_2O_3	51.28	—	2.56	46.16
				CuO	6.52	—	2.17	91.31
4	Top-down	2.3	20	Fe_2O_3	68.49	10.27	14.38	6.86
				CuO	3.11	4.28	18.68	73.93

[a] The results of ICP analysis are presented in terms of metal distribution percentage, which is the weight of metal in one fraction divided by the total weight of metal in all fractions.

[b] The magnetic field strength reported in Table 2 was the local field at the starting location of the sample mixture.

The agglomeration can be divided into two categories: (i) chemical agglomeration; and (ii) magnetic agglomeration. Chemical agglomeration is mainly due to the van der Waals and electrostatic forces between particles. In the present work, the pH of sample suspensions and separation media were both adjusted to 11. With this pH, the surface charges of hematite and copper oxide particles are both negative, and the chance of chemical agglomeration is minimized. It is, however, very difficult to prevent particles from magnetic agglomeration when inside a magnetic field.

Tsouris et al.[14,15] discussed the magnetic flocculation of paramagnetic particles inside a magnetic field. Their model and experimental results showed that the possibility of particle agglomeration is largely enhanced by applying an external magnetic field. The energy barrier between two particles, which prevents particles from agglomeration, is lowered by the magnetic potential, and thus the possibility of particle flocculation is increased. To minimize the magnetic agglomeration, a lower magnetic field is required.

Exps. 3 ($B = 7$-T) and 4 ($B = 2.3$-T) in Table 2 demonstrate the effect of magnetic field strength on the particle agglomeration. Both experiments were top-down orientations and the placement of the sample mixture was chosen at the position where hematite would remain in the Top-1 section and copper oxide particles would move down by gravity. The ICP results in Exp. 3, Table 2, show that 51.28% of the iron stayed in the Top-1 section while 93.49% of the copper moved to the lower part of the separation domain. In the case of lower magnetic field strength (Exp. 4, $B = 2.3$-T), 68.49% of the iron was found in the Top-1 section while 96.89% of the copper was in the rest of the domain. A 17.21% increase of iron in the Top-1 section was achieved by lowering the magnetic field strength from 7.0 to 2.3 T. Furthermore, in the Bottom sections of these experiments, the iron percentage was lowered from 46.16% (Exp. 3) to 6.86% (Exp. 4) due to the decrease in magnetic field strength.

By lowering the magnetic field strength, not only is the magnetic potential decreased, but the collision frequency between hematite and copper oxide particles is also decreased due to the increase in migration velocity of copper oxide particles. As shown in Eq. (3), the migration velocity is the difference between magnetic velocity and settling velocity. The magnetic velocity decreases with magnetic field strength, and since settling velocity is a constant, the migration velocity for copper oxide increases. The sooner copper oxide particles leave the original placement, the less chance they will have to collide or agglomerate with hematite particles.

Comparing the metals distribution in the separation media between bottom-up (Exp. 2) and top-down (Exp. 4) experiments in Table 2, the results show that 66% and 78.76% of the iron were selectively collected in the upper part of the separation domain (Top-1 and Top-2) for Exp. 2 and Exp. 4, respectively, and 88.04% and 92.61% of the copper were collected in the lower part of the domain (Middle and Bottom) for Exp. 2 and Exp. 4, respectively. Although the top-down orientation (Exp. 4) showed a better separation efficiency, the magnetic agglomeration in this case was considered lower than that in Exp. 3 due to the lower magnetic field strength at the starting location. Further study on the effect of orientation on magnetophoresis effectiveness is needed.

CONCLUSION

Selective separation of magnetic sub-micron particles by magnetophoresis was demonstrated in this study. Magnetic species are separated in a magnetic field gradient due to differences in their magnetophoresis mobility. Experiments were conducted using a superconducting solenoid magnet and dilute glycerol solutions as separation media for the separation of hematite and copper oxide particles. Experiment parameters, such as magnetic field strength, media concentration, and sample placement, were determined by a magnetophoresis model developed for the purpose.

Selective separation of hematite and copper oxide particles by magnetophoresis was examined in a top-down and bottom-up orientation. A good correlation between analytical model and experimental results was obtained. Experimental results showed that approximately 70–80% of the iron was selectively collected in the upper part of the separation domain, and approximately 90% of the copper was concentrated in the lower part of the domain. Both the experimental and model results showed that a higher magnetic field strength did not result in better separation. In addition, the magnetic agglomeration increased with increasing magnetic field strength with a corresponding decrease in separation efficiency. An interaction between the wall surface of the separation column and the sample particles was observed and further study on the column materials is needed to minimize this effect.

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