Efficiency of High Gradient Magnetic Separation Applied to Micrometric Magnetic Particles

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This article presents two prototypes of laboratory magnetic separators that generate high gradient magnetic fields. Such a field is created in a separation cell via steel wool. The efficiency of separators was tested on a water suspension containing weakly magnetic Fe₃O₄ nano/micro-particles, prepared in three size fractions in a size range of 60 nm – 10 μm. The separation process was evaluated via optical transmittance of the suspension before and after sequential separation processes. Repeated separations on the same sample exhibit an asymptotic trend that results in the conclusion that it is not possible to trap all solid content. According to the decrease of solid particles concentrations during cyclic separation we set the efficiency of the process. It is maximally 46% for fine fraction, 65% for medium fraction, and 40% for coarse fraction after infinity separation cycles.

Keywords Fe₃O₄ particles; HGMS; magnetic separation

INTRODUCTION

Magnetic separation is a well-established technology (1). In laboratory and biomedical experiments, however, this technique provides a limited tool for separation of nano/micro-particles (2, 3), magnetic cells (4), or blood cells from liquids (5). This limitation is caused by forces that have an opposite direction than magnetic forces.

Generally, the separation condition is that magnetic attraction forces must be larger than the other forces. The result of the process is the separation of input mixture to magnetic and non-magnetic components. The magnetic attraction force depends on the magnetic field gradient. A low gradient magnetic separation can be applied in the most simply way by a permanent magnet located in the side of the test-tube. As such, low-gradient separation principle is used in commercial laboratory low-gradient magnetic separation technologies. This method is suitable for strongly magnetic suspensions. However, in order to separate weakly magnetic materials or paramagnetic materials, a high gradient magnetic separation (HGMS), using a strongly non-homogeneous magnetic field must be created in the vicinity of the magnetic particle (6). Such HGMSeparators are composed of a source of magnetic field and steel wool that is placed into a separation cell. This wool creates a complicated high gradient magnetic field (see Fig. 1) inside of the separation cell (7).

In the works focused on HGMSeparation the physical process is well described (7, 8). On the other hand, very little attention has been paid to other parameters of HGMSeparators such as separation capacity, separation efficiency, size of particles that can be effectively separated out, etc. Theoretical description of these parameters is generally missing in the literature. With the rapid evolution of nanotechnology, these parameters must play a key role in laboratory magnetic separation of nanoparticles. Therefore, the aim of this work is to test magnetic separation efficiency of two self-manufactured HGMSeparator prototypes on weakly magnetic particles, working in dynamic regimes, and to determine some parameters that are important for effective separation of nano/micro-particles.

Theoretical Background (SI Unit System)

In the first approximation, two forces act on the particles in the separator: magnetic \( F_m \) and fluidic resistance \( F_d \). Gravitational force is neglected. We suppose a spherical magnetic particle (paramagnetic or weakly ferromagnetic) having the radius \( R \) in the external field with the magnetic induction \( \mathbf{B} \). The magnetic force \( F_m \) is given by

\[
F_m = V \cdot (\mathbf{M} \cdot \nabla)\mathbf{B}
\]

(1)
where $V = 4/3\pi R^3$ is the volume of particle, $\nabla = \partial/\partial x + j\partial/\partial y + k\partial/\partial z$ is the gradient operator and $M$ is the magnetization, especially below saturation we can write

$$M = \chi \cdot B / \mu_0$$

(2)

where $\chi$ is the effective magnetic susceptibility of particle, $\mu_0$ is the magnetic permeability of free space. Next the magnetic force is

$$F_m = \chi \cdot 4/3 \cdot \pi R^3 \mu_0^{-1} \cdot (B \cdot \nabla)B$$

(3)

When the particle is saturated, $M = M_s$, Eq. (2) must be rewritten as

$$M = M_s \cdot B / |B|$$

(4)

Generally, field gradient is very difficult to determine in cases of HGMS based processes that use the steel wool located in a separation cell. It is difficult to simply express the magnetic force for superparamagnetic particles since they exhibit paramagnetic behavior in a multiparticle amount. Interparticle magnetic interactions are usually taken to be negligible and non-significant for the result of the separation process (9, 10).

For a single spherical particle, the Stokes’ resistance force is valid

$$F_s = -6\pi \eta R v,$$

(5)

where $\eta$ is the dynamic viscosity of the liquid, $R$ is the particle radius and $v$ is the particle speed related to steel wool or magnets. The magnetic forces pull the suspension particles towards steel wool and the Stokes’ resistance restricts this motion. Steady state, when the magnetic force and the Stokes’ resistance force are equal, sets in almost immediately. The quantity of the separated particles depends on the dimensions of the separation cell and the proportion between the velocity of the particle and fluid, respectively (see Fig. 2).

**Particle Flow Ratio (SI Unit System)**

We have made several assumptions to make a rough estimate of the quality of fraction separation:

1. The velocity of fluid $v_y$ is expressed as the medial value of the drift velocity (velocity profile in the separation cell is neglected),
2. Only cross-coordinate of $F_{mx}$ and $v_x$ is non-zero because it is connected with the separation process,
3. Magnetization $M$ is a linear function of $B$ (weak field), which means that Eq. (2) is valid.

A solid particles flow ratio $\kappa$ defined for a selected point in separation cell is

$$\kappa = I_x / I_y = Cv_x / (Cv_y) = v_x / v_y$$

(6)

See Fig. 2, where $I_x$ and $I_y$ are cross-flow (transversal separation flow) and direct flow of particles and $C$ is the concentration of particles.

Now we can express the coordinate $v_x$ using the Eqs. (3) and (5)

$$v_x = 2\chi \cdot (9\eta \mu_0)^{-1} \cdot R^2 \cdot \left( B_x \cdot \nabla x + B_y \cdot \nabla y + B_z \cdot \nabla z \right) B_x$$

(7)

Considering simple field condition $B_x \partial B_x / \partial x \gg B_y \partial B_x / \partial y$ and $B_z \partial B_x / \partial z$, the transversal velocity is

$$v_x = 2\chi \cdot (9\eta \mu_0)^{-1} \cdot R^2 \cdot B_x \cdot \partial B_x / \partial x$$

(8)

Finally, the flow ratio $\kappa$ is expressed via substitute Eq. (8) into Eq. (6) by equation

$$\kappa = v_x / v_y = 2\chi \cdot (9\eta \mu_0 v_y)^{-1} \cdot R^2 \cdot B_x \cdot \partial B_x / \partial x$$

(9)
This means that the quality of separation linearly depends on $\chi$, $R^2$, $B_x$, and $\partial B_x/\partial x$, and hyperbolically decreases according to $\eta$ and $v_y$.

**METHODS**

**Experimental Arrangement**

The experimental apparatus for magnetic separation includes glass reservoir vessels (1 liter), one of which is placed in Ultrasonic Cleaner VGT 1200, 1.3 liter volume, 60 W, 40 kHz, peristaltic pump of PCD 32 type, max flow-rate 50 mL/min, magnetic separator with a separation cell filled by steel wool that is compressed into the plastic separation cell (Fig. 3). The separation cell volume is approximately 50 mL. The ferromagnetic steel wool has packing density 0.08 g/mL and is composed of wire-strips sized 0.5 × 3 mm. The individual parts are connected by silicone hoses having an inner diameter of 4 mm. A detailed description of the separation process is given in the section titled “Description of the Separation Process.”

**Electromagnetic Separator**

The coil separator (Fig. 4) consists of a wooden stand Pertinax (synthetic resin bonded paper), cylinder with wire winding and a separation cell containing steel wool inside. The coil was wound with a copper wire with the diameter of 0.8 mm. The total number of turns is 1485 and the maximum coil resistance is 4.5 $\Omega$. The separation cell (tube) has 20 mm in diameter. The magnetic field in the central point of the coil was measured by the Hall probe and reached the value of 50 mT at the 3 A electric current (measured without presence of steel wool).

![FIG. 3. The experimental set-up of separation process.](image3.png)

![FIG. 4. Scheme of electromagnetic (coil) separator function. The magnetic particles are captured in the steel wool and the non-magnetic (white) particles flow out of the separator.](image4.png)
Separator with Permanent Magnets

The separator (Fig. 5) was equipped with two permanent magnets placed opposite each other in a cylindrical case made of nylon. NdFeB magnets with dimensions $38 \times 20 \times 15$ mm, remanence 1.38 T and coercive force of 923 kA/m were used. The magnetic field without the steel wool reached 300 mT in the central point of separation cell.

Description of the Separation Process

Separation processes using both separators function as follows: Prepared suspension, approximately 1 liter, was brought into the separator using the peristaltic pump after its homogenization by ultrasound in Ultrasonic Cleaner, while the same flow rate of 40 mL/min was maintained for the whole period of separation. The mixture further continued through the separation cell filled by steel wool to the collection vessel, where the separation process ended. The separation process for the same sample was taking place in five identical separation cycles. The steel wool was changed after these five separation steps, while its mass was maintained. The degree of separation was evaluated via optical density of suspension immediately after separations.

Material

The $\text{Fe}_2\text{O}_3$ powder was used for the measurement. The material was provided by Sigma Aldrich Company, product number 310050. The composition of this powder determined via X-ray diffraction is 96% of hematite and 4% of maghemite. Such material was selected for its significantly weak magnetic fraction, since hematite is weakly ferromagnetic and maghemite is ferrimagnetic material under room temperature. The magnetization of powder was performed using the Vibrating Sample Magnetometer (VSM, EV9 Microsense), Fig. 6. The saturation magnetization reached 2 A.m$^2$/kg and initial susceptibility was approximately $10^{-5}$ m$^3$/kg. In the case of ferrimagnetic magnetite, these parameters are presented to be in the order 100 times stronger (11).

The powder suspension was prepared using distilled water. In the first step the suspension was undergone to sedimentation. This method is based on the speed of particle sedimentation according to size. The next step was the ultrasound cavitation disintegration that lies in the periodic compression and release of the fluid, while cavitation bubbles are created, primarily on the particles. Collapse of the bubbles on the particles then leads to creation of local temperature and pressure extremes that result in destruction of the particles (12, 13). Three $\text{Fe}_2\text{O}_3$ fractions were prepared—coarse, medium, and fine. Their size distributions are presented in Fig. 7 and were determined by employing the dynamic light scattering method by a Malvern Zetasizer NanoZS (type ZEN3600). The distribution of coarse fraction was out of measurement limits of this optic device and has mode of approximately 5 μm, based on the knowledge of initial particles size distribution.

Evaluation of Separation Processes

The evaporation and photometry techniques were selected for the evaluation of the separation processes.

The evaporation method provides information about mass concentration $C$ of particles in suspension and is based on the measurement of dry powder weight after fluid evaporation, from 50 mL, $C = (\text{mass of solid powder})/(\text{volume of suspension})$ in mg/mL. While the method does not depend on coagulation and sedimentation of particles, only the densest samples were determined using this method in the range of calibration measurement. Other samples were excluded from such
RESULTS AND DISCUSSION

Results

The results of magnetic separation on Fe₂O₃ powder are presented in Fig. 8. It is apparent that devices separated the magnetic component from the suspension after each separation cycle, which shows an asymptotic trend in ideal cases. In several cases the asymptotic trend was not observed, which was caused by unknown process errors. Those non-asymptotic processes are designated as w.t. in Fig. 8 and are taken to be invalid in subsequent theoretical computations. In spite of the fact that measurement repetitions A, B, C, D, E were done using the same initial suspension sources (coarse, medium and fine), we found differences in T₀ values and subsequently in the individual separation processes. For this reason these repetitions could not be averaged for the subsequent data processing.

Since the separation process depends on many parameters, an appropriate physical function describing the repetitive process is hardly possible to derive. To understand a separation process more precisely we decided to describe it via a mathematical equation. Thus, we created appropriate asymptotic function expressing transmittance T(%) of light via suspension in form

\[ T = T_0 + b[1 - \exp(-k \cdot N)] \]  

(10)

where \( T_0 \) is the initial transmittance, \( b \) (%) and \( k \) (-) are the constants and \( N \) (-) is number of separation cycles applied. The separation limit \( T_\infty = T_0 + b \) expresses transmittance after an infinite number of separation cycles. The function represented by Eq. (10) provides a good theoretical fit to experimental points since in several cases the sum of square deviations \( r^2 \rightarrow 1 \), using Matlab software.

The parameters \( T_\infty \), evaluated base of measurements, are displayed in the figure legends. While the parameter \( T_0 \) was optically determined to be 0–30% in cases of coarse and medium fraction, in the case of fine fraction \( T_0 > 60\% \). The reason for these results is in low initial concentration of particles in suspensions with fine fraction. Additionally, we expected that coarse fraction should have a higher value of \( T_\infty \) since the separation quality coefficient is according to Eq. (9) proportional to particles size \( R^3 \). However, the general trend is opposite (see Table 1). The fine fractions have (ostensibly) higher separation limits. The separation trends displayed in Fig. 8 for fine fraction do not have an asymptotic, but rather chaotic trend. In our opinion, such results could be explained via limited wool surface area of the separators, i.e., during the separation of coarse and medium fraction the wool surface is quickly covered by strongly concentrated suspensions. Additionally, the fine fraction probably was not really separated, since the fraction was prepared above the particle-size separation limit of the separator, which must be limited by Brownian force (see below).

In Table 2, the separation ranges after five cycles are presented. It is apparent that separation ranges \( T_\infty \) are significantly higher in cases of coarse and medium fractions than in cases of fine fraction. It is not reliably possible to compare the efficiency between both types of separators.

Solid Concentrations in Suspensions

Real concentration of solid matter in suspension can be estimated using the well-known Lambert-Beer law that defines transmittance as \( T(\%) = 100 \cdot 10^{-\alpha C} \) where \( \alpha \) (mL·mg⁻¹) is the...
FIG. 8. Results of the magnetic separations evaluated using the transmittance of green light (500 nm) through the suspension containing magnetic Fe₂O₃ particles. The symbols A, B, C, D, E designate names of the same samples (repetitions of measurement), and the abbreviation w.t. denotes “without asymptotic trend”. Flow rate \( q = 40 \) mL/min. The parameter \( T_\infty \) (%) is presented in the legend for asymptotic separation cycles.

| Limit transmittance \( T_\infty \) after an infinite number of separation cycles evaluated using Eq. (10) and relation \( T_\infty = T_0 + b \), base of five experimental separation cycles |
|-----------------------------------------------|
| Sample | PM-coarse | C-coarse | PM-medium | C-medium | PM-fine | C-fine |
|--------|------------|----------|-----------|----------|---------|--------|
| A      | 51         | 52       | 57        | 59       | 90      | w.t.   |
| B      | 45         | w.t.     | 65        | 82       | 69      | w.t.   |
| C      | w.t.       | w.t.     | 73        | 84       | w.t.    | w.t.   |
| D      | 70         | w.t.     |           |          |         |        |
| E      |            |          |           |          | 91      |        |

*Note*: The abbreviation w.t. denotes “without asymptotic trend”, PM denotes separator with permanent magnets and C denotes the separator with coil.
coefficient depending on optical properties of dispersed material, length of absorption layer and wavelength of light, and $C$ (mg/mL) is the concentration of matter in suspension. Using this law, the concentration after $N$-th separation cycles is

$$C_N = -\alpha^{-1} \cdot \log(T_N/100)$$ (11)

and depends on transmittance $T_N$ of suspension after $N$-th separation cycle.

Equation (11) provides the simplest approximation of $C$ since the parameter $\alpha$ depends on particle size distribution.

In the range of our experiment, the parameter $\alpha$ is estimated based on calibration photometry measurement of specially prepared samples, not via magnetic separation, having various concentrations (see Fig. 9). The concentrations in the initial and densest 50 mL suspension samples were determined via evaporation method, $C_0(\text{fine}) = (0.0170 \pm 0.0011)$ mg/mL, $C_0(\text{medium}) = (0.199 \pm 0.0011)$ mg/mL, $C_0(\text{coarse}) = (0.223 \pm 0.0011)$ mg/mL. Subsequently, the initial samples were diluted in four steps by the same volume as previous samples had had to concentrations $C_0/2$, $C_0/3$, $C_0/4$, and $C_0/5$ and passed for photometry. These concentrations

| Sample | PM-coarse | C-coarse | PM-medium | C-medium | PM-fine | C-fine |
|--------|-----------|----------|-----------|----------|---------|--------|
| A      | 34        | 41       | 40        | 40       | 9       | -4     |
| B      | 32        | 69       | 44        | 54       | 6       | 1      |
| C      | 65        | 76       | 45        | 47       | -1      | -2     |
| D      | 56        | 14       |           |          |         |        |
| E      |           |          |           |          | 8       |        |

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![FIG. 9. Calibration transmittance measurements on Fe$_2$O$_3$ suspensions using 500 nm light. In case of fine fraction there are only four points in the fitting since the transmittance in case of the initial concentration is zero. The fitting equation is $C = -\log(T/100) + \beta$. The values of the parameters are $\alpha_{\text{fine}} = (21.5 \pm 2.4)$ mL/mg, $\beta_{\text{fine}} = (0.0025 \pm 0.0008)$ mg/mL, $r^2_{\text{fine}} = 0.986$, $\alpha_{\text{medium}} = (13.9 \pm 1.0)$ mL/mg, $\beta_{\text{medium}} = (0.0291 \pm 0.0064)$ mg/mL, $r^2_{\text{medium}} = 0.994$, $\alpha_{\text{coarse}} = (12.9 \pm 2.0)$ mL/mg, $\beta_{\text{coarse}} = (0.0414 \pm 0.0061)$ mg/mL, $r^2_{\text{coarse}} = 0.990$, using standard errors.](image-url)
do not correspond to samples having transmittance \( T_0 \) in main separation measurements in Fig. 8. Fitting the relation between \( C \) and \( \log(T/100) \) in equation \( C = -\log(T/100)/\alpha + \beta \), one can find the parameters \( \alpha \) and \( \beta \) and subsequently estimate the real concentrations of suspensions from main separation measurements. The parameter \( \beta \) calibrates the photometry device for the real concentration of suspension when \( T = 100\% \). Finally, the separation efficiency per single or several separation steps \( \eta_n \) (\%), between cycles \( N \) and \( N + n \), where \( n = 0, 1, 2, \ldots \infty \) is the count of other separation steps, is derived to be

\[
\eta_n = -100 \cdot \Delta C/C_N
= 100 \cdot \alpha^{-1} \cdot \log(T_{N+n}/T_N)/C_N
\]  

valid in interval 0–100\%, where change of the concentration is

\[
\Delta C = C_{N+n} - C_N = \alpha^{-1} \cdot \log(T_{N+n}/T_N).
\]

Using these last equations, for example, medium fraction sample B separated in five cycles using an electromagnetic separator having measured transmittances \( T_0 = 17\% \), \( T_5 = 71\% \), and \( T_\infty = 82\% \), estimated concentrations and efficiency are \( C_0 = (0.0888 \pm 0.0083) \text{ mg/mL} \), \( C_5 = (0.0406 \pm 0.0065) \text{ mg/mL} \), \( \Delta C = (-0.0481 \pm 0.0106) \text{ mg/mL} \), and \( \eta_5 = (54 \pm 13)\% \). Its concentration after infinite separation cycles is \( C_\infty = (0.0358 \pm 0.0064) \text{ mg/mL} \) and \( \eta_\infty = (60 \pm 13)\% \), taking into account calibration parameters \( \alpha \) and \( \beta \) valid for medium fraction and uncertainty of transmittance 0.577\%. The efficiency after infinite separation cycles is shown in Table 3. In spite of the fact that we measured and processed the data carefully, standard confidence intervals are large.

### Importance of Parameters

#### Equation 9

Separation ratio \( \kappa \), defined via Eq. (9), is proportional to \( B, \partial B/\partial x, \chi, \) and \( R^2 \), and inversely proportional to \( \eta \) and \( v \). The parameter \( v \) is flow rate \( q \) divided to hose cross-section area. The effect of \( B \) depends on magnetic field generated by magnets/coil. The effect of \( \partial B/\partial x \) depends on packing density of steel wool and its magnetic properties. The effect of material susceptibility can be tested using different materials and the effect of viscosity using different liquids. The effect of particle size \( R \) is hard to verify since it is difficult to prepare a monodispersion, but our results are partially discussed in the result section. Finally, we tested the effect of a flow rate of liquid (see Fig. 10). In accordance with expectations, slower flow of suspension results in more effective separation.

#### Equation 10

The magnetic force acting on a single particle must overcome Stokes’ viscous force, and, in a limited case, the Brownian force as well. This Brownian force may be estimated by the term \( k_B T/R (2) \). From equilibrium condition between magnetic and Brownian force the size of the particles that cannot be separated may be estimated. The physical meaning of parameters presented in Eq. (10) could be considered in this way: In case of hypothetical separator having infinite capacity and wool surface area (see below), the parameter \( T_\infty = T_0 + b \) probably depends only on Boltzmann factor \( \exp[-wR^3/(k_BT)] \), where the term \( wR^3 \) represents potential magnetic energy of a particle in the vicinity of steel wool, where \( w \) (J/m³) depends

![FIG. 10. Effect of flow rate \( q \) on medium fraction of Fe₂O₃ during magnetic separation. It was tested on a separator based on permanent magnets.](image)

### Table 3

| Sample | PM-coarse | C-coarse | PM-medium | C-medium | PM-fine | C-fine |
|--------|-----------|----------|-----------|----------|---------|--------|
| A      | 38 ± 13   | 40 ± 13  | 46 ± 13   | 47 ± 13  | 46 ± 18 | w.t.   |
| B      | 38 ± 13   | w.t.     | 51 ± 13   | 60 ± 13  | 18 ± 15 | w.t.   |
| C      | w.t.      | w.t.     | 47 ± 14   | 57 ± 14  | w.t.    | w.t.   |
| D      |           |          | 65 ± 13   |          |         | w.t.   |
| E      |           |          |          |          | 32 ± 21 |        |
Fe$_2$O$_3$ particles, in dynamic regimes. Using a new theoretical approach to evaluation of separation efficiency via optical transmittance $T(\%)$ of suspension we discovered that separation process exhibits an asymptotic trend. The asymptotic equation, $T = T_0 + b(1 - \exp(-kN))$, constructed in this work, provides good information about the parameters of the separation process. This asymptoticity is caused mainly by the wool surface area capacity of separators and size separation limits of particles connected with forces acting on them. The separation without magnetic field exhibits a certain level of filtration effect whose origin is unclear, but it is a result of inner magnetic and adhesion forces. The separation efficiency depends on the flow-rate of the suspension, slower flow of suspension results in a better separation effect. The transmittance value can be recomputed to real concentration of solid powder matter in suspension using Lambert-Beer law. In spite of the fact that this law is used mainly for solutions, for our Fe$_2$O$_3$ nano/micro particles dispersed in water it appears to be the simplest but useful relation between concentration and transmittance. Based on this theory we estimate that maximal efficiency after infinity separation cycles on fine fraction reaches $\eta_\infty = (46 \pm 18) \%$, on medium fraction reaches $\eta_\infty = (65 \pm 13) \%$ and on the coarse fraction $\eta_\infty = (40 \pm 13) \%$. The efficiency in case of fine fraction is influenced by the negative fact that samples were prepared on/above the level of size separation limits of Fe$_2$O$_3$ solid particles.

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