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High-gradient magnetic separation using ferromagnetic membrane

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ABSTRACT

The magnetic separator with the membrane separating unit made of laser-perforated thick ferromagnetic foil was tested using composite water suspension of magnetic nanoparticles adsorbed on hydroxylapatite microparticles. The average sizes of the particles in the suspension and the magnetic moment of the suspension were measured by dynamic light scattering and electron magnetic resonance correspondingly to evaluate the efficiency of the separation. It was shown experimentally that the separation is effected by the membrane type and the flow rate. Magnetic coarse grains (larger than 1 μm) were captured by the membrane preferably and the magnetic moment of the suspension decreased by 20–25% after the separation. The magnetic field simulation and experimental results demonstrate the higher separation efficiency for thicker membranes.

1. Introduction

Magnetic separation is a commonly used technique in metallurgy, coal industry, for wastewater treatment, etc., to extract and concentrate strongly magnetized particles from a powder or a suspension. High-gradient magnetic separation (HGMS) merged due to the development of conventional magnetic separation technology [1]. HGMS is regarded as a highly productive approach to extract microparticles with the close magnetic susceptibility values.

The most commonly used basic separating unit for the HGMS is a ferromagnetic wire [2] packed variously: as a grid, chaotic tangle or ordered spatial structure with the regular wire arrangement collinear to each other [3,4]. However, there are lot of problems in the analysis of magnetic separation processes and in the description of flow hydrodynamics near the wire separating unit, in particular due to diamagnetic or mechanical capture of particles [5]. This makes separator cleaning difficult and does not allow to extract the captured fraction properly.

Despite these problems the HGMS has been effectively applied to separate various bio-objects like paramagnetic and diamagnetic erythrocytes, red blood cells infected with the malaria parasite, magneto bacteria and magnetic sorbents conjugated with cells, proteins and other biological components [6–8].

Recently the magnetic separators of new designs were developed. Microelectronics technologies are widely employed to create new separators able to capture superfine and weakly magnetized particles. The on-chip made separators based on magnetic field flow fractionation or magnetic chromatography can separate Brownian particles according to their magnetic properties and can be applied for analytics [9–11]. Another construction is a magnetic sifter fabricated with microelectronics techniques can also be used as a separating membrane [12,13]. Hydrodynamics is unambiguous in this type of separator and the separating unit can be easily cleaned comparing to the conventional wire separator. Nevertheless, only thin ferromagnetic sifter can be fabricated by the applied technologies. In this case the hole diameter of the sifter is significantly greater than the sifter thickness, thus the gradient magnetic field does not cover the hole area and the separation is poorly performed.

In this work a new design of the magnetic separator is presented. The thick ferromagnetic membrane would be used as a separating unit. This type of separator allows one to avoid the aforesaid disadvantages and improve the separation.

2. Experiment

The potential of the designed membrane separator (fig. 1) to capture certain particles was tested in the work. The separator consisted of two chambers for retentate and eluate divided with the ferromagnetic membrane (unit 3 in Fig. 1). The membrane was magnetized by the magnetic field of the permanent magnets of $SmCo_5$ (40×40 mm^2 in size and 10 mm in thickness) (unit 1 in Fig. 1). The magnetic field on the magnet surface was 240 kA/m. The membrane was set perpendicular to the magnetic field lines between the permanent magnets and it was equidistant from the

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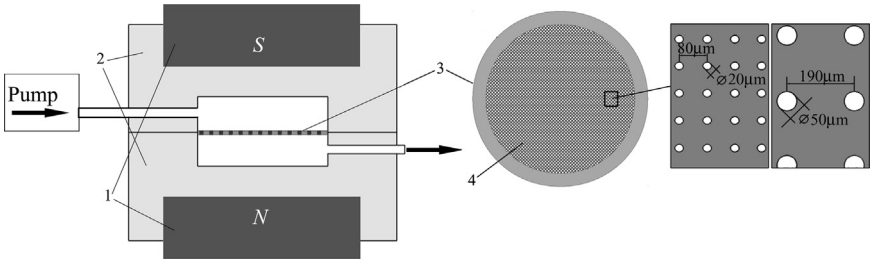

Fig.1. The sectional view of the membrane magnetic separator. 1 are the permanent magnets; 2 is the separator body (PTFE), the arrows point the inlet and outlet; 3 is the ferromagnetic membrane; 4 is the perforated area with holes; 5 is a zoom in the structure of M2 and M4 membranes.

magnet poles at a distance of 10 mm as it is indicated in Fig. 1. The initial suspension was separated as it flowed through the membrane.

The ferromagnetic separating unit (unit 3 in Fig. 1) with the diameter of 19 mm was made of the low-coercitivity foil of Permendur alloy (Fe–Co). The holes of 20 or 50 μm in diameter were laser-perforated in the foil of 50 or 100 μm in thickness. The laser equipment MLP2-002 Turbo© (MLP-micromachining) by ESTO (Russia) was used for the precession perforation of the foil. Laser station equipped with Ytterbium fiber lasers (IPG Photonics) were designed for micromachining and marking. The presented membranes were fabricated under following operation mode: laser spot diameter varied from 20 to 50 μm, lasing wavelength was 1.02 μm, impulse frequency and duration were 50 kHz and 0.05 μs correspondingly, laser output power was 20 W and positional resolution was less than 3 μm. The distances between the holes were 80 or 190 μm and the hole densities were 26 or 156 holes/mm^2 correspondingly. The dimensions of the membranes under study are presented in Table 1.

The laser perforation is a novel technology in HGMS devices engineering. Ferromagnetic membranes fabricated with this technique differ from the already known membranes (sifters) fabricated with microelectronics techniques. Using laser perforation we were able to produce regular fine holes in comparatively thick ferromagnetic foil such that the foil thickness was fivefold greater than the hole diameter (aspect ratio). As it would be shown below the aspect ratio value is one of the key factors for the effective HGMS with ferromagnetic membrane.

The particle sizes in the separated suspension were measured by dynamic light scattering (DLS) using Malvern Zetasizer Nano S (UK). The concentration of magnetic phase in suspension before and after separation was determined by electron magnetic resonance (EMR). EMR spectra were measured by X-band spectrometer Bruker EMX 8/2.7 (Germany) at room temperature. Radiowave frequency was 1 mW and modulation amplitude was 1 G. The suspension magnetization proportional to EMR signal intensity was determined by double integration of the experimental spectra. The powder of magnesium oxide containing Mn^{2+} was used as the outer standard.

The suspension of magnetite nanoparticles (MNP) adsorbed on microparticles of hydroxylapatite (HA) was a model composite system for separation tests. The MNP hydrosol was obtained by Massart method [14] by co-precipitation of Fe(II) and Fe(III) in alkaline medium (NH_4OH). The mean diameter of the particles obtained was ~26 nm according to the DLS (Fig. 2 curve 1). These particles are considered as a superparamagnetic. The long-term storage of hydrosol results in particle aggregation to the size of ~50 nm (Fig. 2, curve 2). The MNP concentration was 61(1) mg/ml and volume fraction was 0.012. The initial magnetic hydrosol was mixed with the HA suspension in 1:1000 ratio, thus the resulting MNP concentration was 6.1(5) 10^{-2} mg/ml. The pure HA suspension exposed for 20 min to select supernatant to mix with the MNP hydrosol. The suspension prepared this way kept its stability

Table 1
Parameters of the ferromagnetic membranes under study.

Membrane type	Foil thickness, μm	Hole diameter, μm	Hole density, holes/mm^2
M1	50	20	26
M2	50	20	156
M3	100	20	26
M4	100	50	26

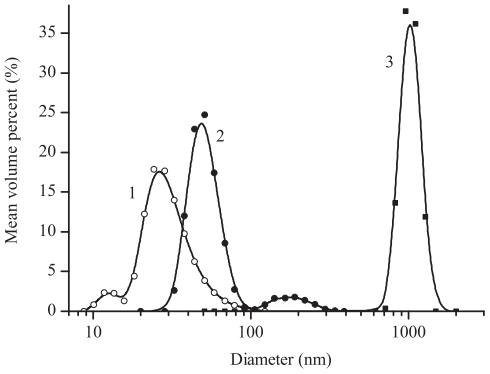

Fig. 2. Mean volume fraction of the particles as a function of their diameter in the hydrosol of the MNP after the synthesis (1) and after long-term storage (2) and in the suspension of the pure HA (3).

during the experiment. The average diameter of the HA grains was 1 μm according to the DLS (Fig. 2. curve 3). The mixture of the MNP and HA was kept for a day to complete the MNP adsorption. The DLS results were represented in terms of mean volume percent.

The resulting suspension introduced into the injector of the piston pump attached to the separator inlet with the tubes (Fig. 1). The flow rate of the suspension can be varied in the range of 40–1 ml/h. Two factions (2 ml each) of the eluate were thieved one after another (*fraction 1* and *fraction 2*) for the following analysis. The retentate of the suspension was also analyzed.

3. Results

Electron magnetic resonance and dynamic light scattering were used for the separation monitoring. The DLS curves of the suspension before (curve 1) and after separation with the flow rate of 20 ml/h (curve 2) and 5 ml/h (curve 3) are presented in Fig. 3. It is seen that after the separation the particles larger than 1 μm were captured by the membrane and their signal disappeared. The volume percentage of the fine particles (size less than 100 nm) increased. These results confirm the efficiency of the membrane to capture coarse magnetic grains.

The flow rate of the suspension affects the separation results. The content of coarse grains of the composite of HA with MNP as