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High-Gradient Magnetic Field Split-Flow Thin Channel (HGMF-SPLITT) Fractionation of Nanoscale Paramagnetic Particles

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ABSTRACT

High-gradient magnetic field (HGMF) split-flow thin channel (SPLITT) fractionation (HGMF-SPLITT) is a newly developed magnetic fractionation technique for separating submicron and/or nanoscale paramagnetic particulate species in a continuously flowing separator.

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Incorporation of high-gradient magnetic fields, into the split-flow-fractionation separator geometry, increases the magnetic force experienced by a paramagnetic submicron particle relative to current magnetic field-flow-fractionation devices. The application of HGMF-SPLITT fractionation, for selective separation of paramagnetic submicron particulates, was experimentally investigated.

Key Words: Magnetic; Fractionation; Field-flow-fractionation; Paramagnetic; Submicron, High gradient; Separations; Splitt.

INTRODUCTION

The application of high-gradient magnetic fields (HGMF) to split-flow thin channel (SPLITT) fractionation offers the potential to separate paramagnetic particulates from a stream, advancing the traditional approach to magnetic separation and applications of SPLITT. Magnetic separations have been developed and utilized in industrial,[1–3] medical,[4,5] and governmental[6–8] applications as a separation technology. With the advent of high field magnets and advanced matrix materials, high-gradient magnetic separation (HGMS) has extended the range of particle sizes and magnetic susceptibilities that may be addressed via magnetic separations.[9] The production of high magnetic field gradients is crucial to the development of an enhanced magnetic separation system. HGMS capitalizes on the high magnetic field gradients generated from the magnetic induction of small ferromagnetic matrix elements within a magnetized volume. Due to the large magnetic forces generated in HGMS systems, trapping of the magnetic species in the matrix is likely and, therefore, high-gradient magnetic separations are inherently a batch process. However, magnetic separator systems have been designed and implemented for psuedo-continuous processing, usually by operating one or more systems in parallel.

SPLITT-FFF, a subtechnique of field-flow-fractionation (FFF), is a continuous process that separates particles into varied fractions dependent upon the type of applied field. A variety of fields have been utilized that include gravity[10,11], electrical[12], thermal,[13] and magnetic fields.[14–19] Magnetic SPLITT-FFF has been limited to the application of open gradient magnetic fields. Magnetic SPLITT-FFF of ferromagnetic Dynabeads (M-450, 4.5 μm diameter), paramagnetic labeled silica beads, and starches or magnetite (ferromagnetic) impregnated latex spheres have had excellent results for retrieval of the magnetically susceptible material; however, these separations have been limited to large micron-sized paramagnetic particulates, or ferromagnetic submicron particulates principally due to utilization of the weaker applied magnetic forces of the open gradient magnetic systems. There is a need for magnetic SPLITT technology capable of addressing submicron, paramagnetic particles.

High-gradient magnetic field-SPLITT (HGMF-SPLITT) has been theoretically explored as a means to separate submicron paramagnetic particles in nuclear fuel recycling.[20] This theoretical study of thin parallel plate channels with arrays of thin ferromagnetic wires arranged on both walls, and magnetized by a superconducting magnet, suggested that HGMF-SPLITT would be superior to current magnetic SPLITT-FFF systems for particulate separations. This is due to the higher magnetic force achieved with high-gradient magnetic fields. However, no experiments have been carried out for this proposed system.

We have assembled and tested a separation device where cryogenically milled stainless steel wool powder is present to generate high magnetic field gradients in a SPLITT separator. The matrix material locally distorts the magnetic field and generates high field gradients at the surface of the matrix elements. These areas of high magnetic field gradients then become attracting sites for paramagnetic species and are the basis for high-gradient magnetic separation. We describe here the construction and testing of a HGMF-SPLITT separator.

MATERIALS AND METHODS

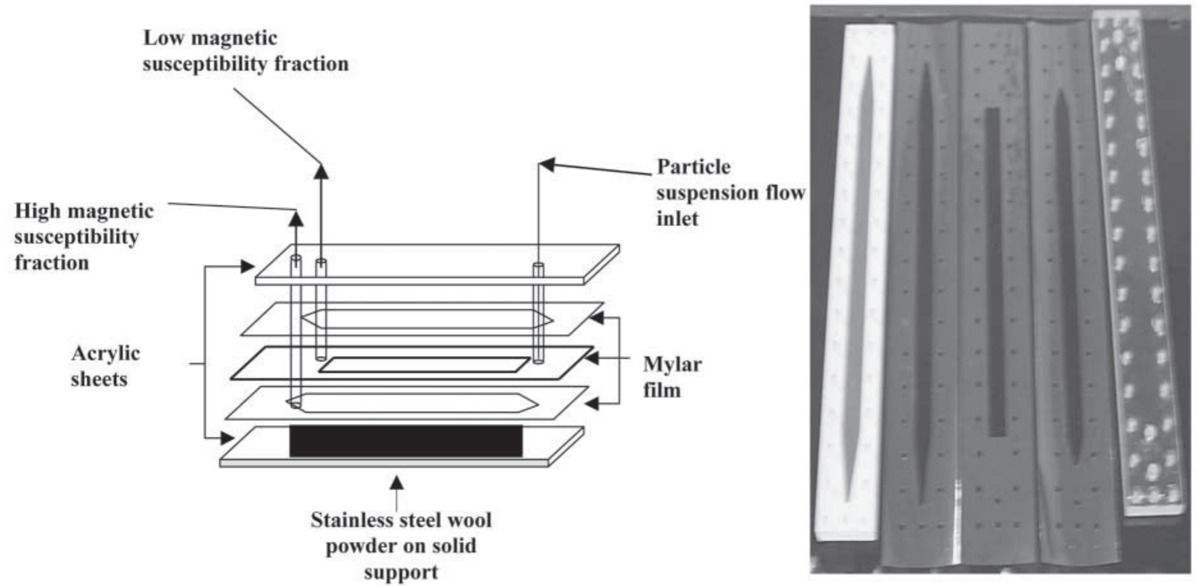
A low-temperature superconducting magnet with a 3-in. warm bore and an 8 Tesla maximum field strength (Cryomagnetics Inc., Oak Ridge, TN) was used for these experiments. Solutions were pumped through the separator by a Pharmacia P-3 six-roller peristaltic pump (Sweden). The matrix material, shaved stainless steel wool, was obtained from MEMTEC Inc. (Deland, FL). The stainless steel wool was cryogenically milled using a SPEX Certiprep 6800 freezer/mill (Metuchen, NJ) to generate a powder, which was wet-sieved (40 μm sieve) to remove any remaining large fibers. Double-sided, pressure sensitive, Mylar tape was used as a fixative and solid support for the powdered ferromagnetic matrix. Mylar sheets (0.003, 0.002, and 0.001-in thickness) were obtained from Plastics Supply Co. (Albuquerque, NM) and used to define the flow channel dimensions. A Permatex spray adhesive 118DA (Solon, OH) obtained from National Automotive Parts Association (Los Alamos, NM) was used to join and seal the varied Mylar sheets. Acrylic plates (0.5 in. depth, 12 in. length, 2 in. width) purchased from Plastics Supply Co. (Albuquerque, NM) were used to confine the flow channel and "sandwich" the Mylar sheets. Aerosolized Teflon mold release #6075, purchased from Crown (Gardnerville, NV) was used to cover the powdered matrix material.

Paramagnetic iron oxide, hematite (Fe_2O_3), was purchased from CERAC (Milwaukee, WI). Specific size distributions of Fe_2O_3 were obtained by sedimentation and decantation procedures relying upon the relationship between settling velocity and particle diameter described in Stokes Law. Particle size distributions were acquired with a Horiba LA 900 (Kyoto, Japan) particle size analyzer, in the centrifugal mode, and verified against latex sphere particle size standards from Duke Scientific (Palo Alta, CA). Particulate suspensions in nanopure water adjusted to pH 10 were sonicated in a Branson sonicator (Shelton, CT) for 10 min prior to being pumped through the separator. Sodium hydroxide (Fisher Scientific, Pittsburgh, PA) was used for pH adjustment. Trace metal nitric acid (Fisher Scientific, Pittsburgh, PA) was used for digestion of the hematite. Metal concentrations were acquired on a Jarrell Ash ICP-OES (Franklin, MA) following the hot plate digestion of experimental samples. ICP standards (SPEX Certiprep, Metuchen, NJ) were purchased from Fisher Scientific.

An exploded-view schematic of the HGMF-SPLITT device is illustrated in Fig. 1 along with a digital image of the primary components prior to assembly. Several separator devices were fabricated in this laboratory with channel depths of 0.007- and 0.005-in. Assembly of the separator has been described previously[19]. Stainless steel wool powder was applied to the separation channel to generate the high-gradient matrix material within the separator.

The separator assembly was operated in an upflow configuration oriented vertically, and feed suspensions introduced from the bottom. Feed suspensions were pumped to the inlet port of the separator via tubing routed through the peristaltic pump. For experiments conducted with an applied field, the splitter was suspended in the warm bore of the superconducting magnet such that the separation channel was located in a region of homogenous magnetic field. For experiments conducted without an applied field, the splitter was secured in a vertical position on a laboratory benchtop using a ringstand with clamps.

Nanopure water was pumped through the splitter assembly via a peristaltic pump, and outlet flows were transferred to two graduated cylinders while flow volumes were periodically collected. The two outlets were labeled high gradient and low gradient. Samples collected from the flow exiting below the splitting plane were labeled high-gradient outlet, and flow exiting from above the splitting plane was labeled low-gradient outlet, as shown in Fig. 2. Superficial velocities (cm/s) along the separation channel were calculated from total measured flow rates. Feed solutions of hematite particle suspensions, with a nominal particle size distribution of 460 nm (+/−300 nm), were processed through the splitter assembly. Following processing of the hematite suspension, a flush sample was acquired by flowing nanopure water through the splitter assembly, removed from the warm bore of the magnet, at superficial velocities approximately 10 times greater than processing velocities.



Left-right: acrylic plate with stainless steel wool on solid support; Mylar sheets to define the flow channel; acrylic plate with outlet ports.

Figure 1. An exploded view (left) of high-gradient magnetic field thin channel split flow fractionator assembly and image of primary components (right) prior to final assembly used in experiments.

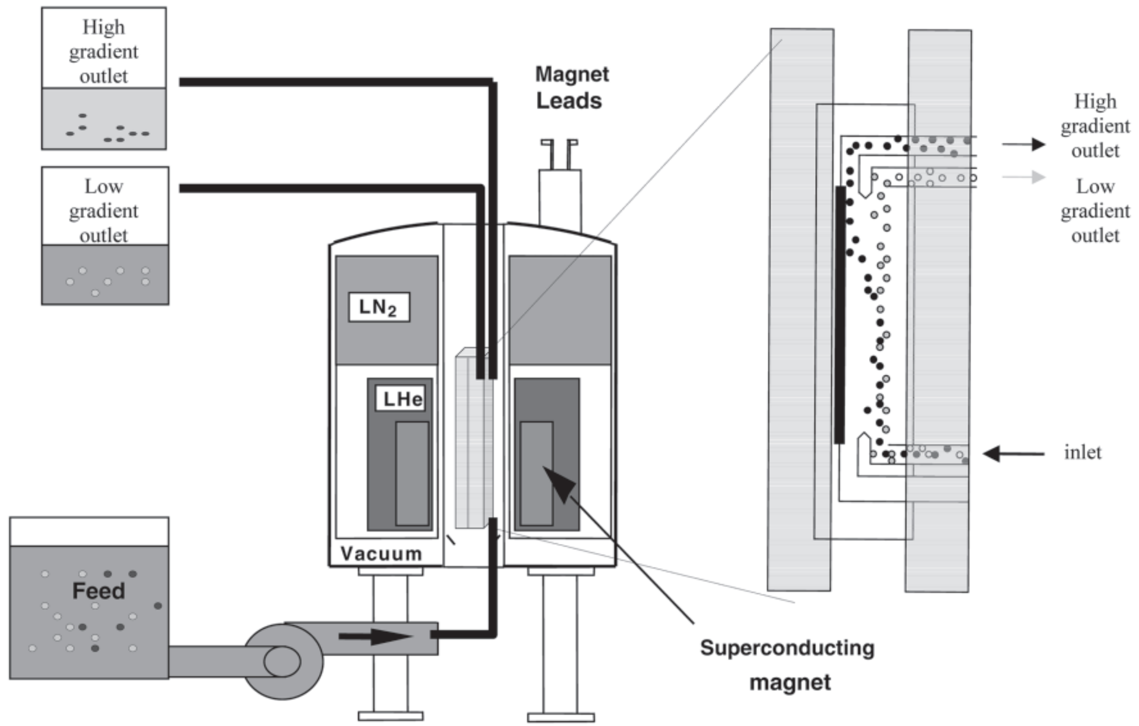


Figure 2. Experimental set-up with separator assembly suspended in warm bore of magnet showing high magnetic susceptibility particles exiting outlet port associated with stainless steel wool powder monolayer and low magnetic susceptibility particles exiting port away from matrix.

Experimental samples (feed, high gradient, low gradient, and flush) were collected and analyzed by ICP-AES.

RESULTS AND DISCUSSION

The primary accomplishment of this work is the inclusion of a high-gradient magnetic matrix material into the geometry of a SPLITT flow-fractionator device. The potential to efficiently separate submicron weakly magnetic particles in a discrete fashion would provide advancement in traditional HGMS processing devices. Two modes of operation are envisioned. One approach is a continuous flow device with the magnetic particles attracted toward the HGMS matrix and exclusively diverted to the high-gradient outlet. A second approach would be a capture device effectively retaining all paramagnetic particles on the HGMS matrix material. This approach would offer advantages in forensic applications where minute samples are available and capture of individual particles is critical.

In a continuous flow–no capture design, conceptually, the best results that we would expect to achieve with the current configuration are demonstrated in Fig. 3. This is an idealized figure and the data is not experimentally acquired. Our channel geometry is such that the splitting plane is centered in the vertical

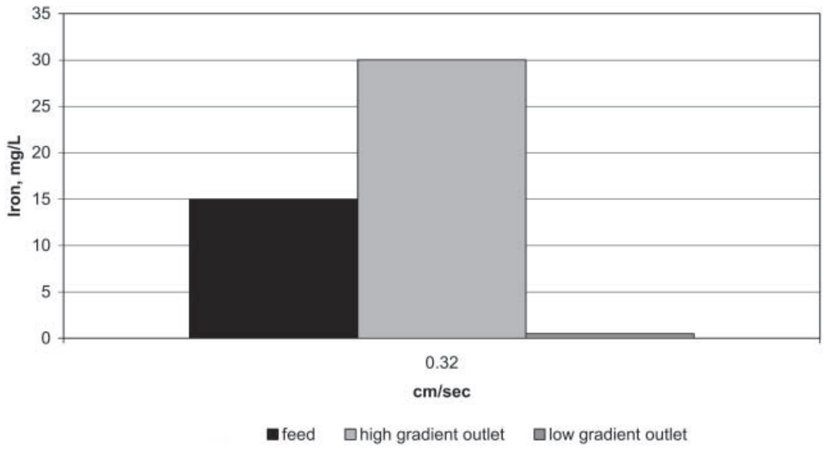


Figure 3. Conceptual best-case results of a continuous flow device where the concentration of the high-gradient outlet would be double the concentration of feed. The results are based on a separator with an applied field of 7 Tesla, 0.007-in. channel depth, 45 μm stainless steel wool powder matrix material, and 460 nm Fe_2O_3 particles in feed solution.

dimension of our separation channel. With this configuration, the volume processed in our channel would be split in half with one portion exiting the high-gradient outlet while the other portion exits the low-gradient outlet. Complete particle accountability or 100% mass balance is obtained because all particles pass through the separator exiting to the high-gradient or low-gradient outlets. As particles flow through the separator channel, the magnetic forces from the HGMS matrix material are large enough to attract all of the paramagnetic particles toward the supported matrix and below the outlet-splitting plane. In this mode of continuous-flow operation, we would expect all of the paramagnetic particles to be within the high-gradient outlet sample. These assumptions would result in doubling the concentration of material within the high-gradient sample relative to the initial feed concentration.

In contrast to the conceptual "best-case" results of Fig. 3, in our experiments we have observed particle retention within the separator device, which requires a flushing procedure for particulate collection. Both mechanical trapping and magnetic trapping are observed during a separation experiment. We consider the particulates captured on the bottom surface of the separation channel as being magnetically captured due to the high magnetic forces generated by the stainless steel powder matrix. Particle retention within the separator, not due to magnetic attractive forces, we consider as being mechanically entrapped. Most of the material captured within the separator, either due to magnetic forces or mechanical entrainment, may be recovered in a flushing procedure.

We performed a series of mechanical and magnetic trapping tests to determine the extent of trapping within the separator with and without an applied field. Figure 4 details the results of a 0.007-in. channel depth separator processing 460 nm hematite with no applied magnetic field. At 0.70 cm/s superficial velocities, there is very little mechanical retention within the split-flow fractionation device and essentially no difference in the concentration of hematite exiting either of the outlets. Figure 5 details the performance of the same separator and superficial velocity but in the presence of a 6 T magnetic field. Application of a 6 Tesla magnetic field increases the magnitude of material retained within the separator, relative to no applied field. The difference between the amount of material captured with, and without, an applied magnetic field is considered magnetic capture.

If particulates are magnetically captured along the surface of the separation channel, then it is reasonable to assume that their trajectories were such that they had crossed the vertical mid-plane of the separation channel and would have exited the high-gradient outlet had capture not occurred. If this assumption is valid then subtracting the mechanical capture from the magnetic capture and adding this value to the high-gradient outlet results in a concentration greater than the feed. Simply, magnetically captured hematite is

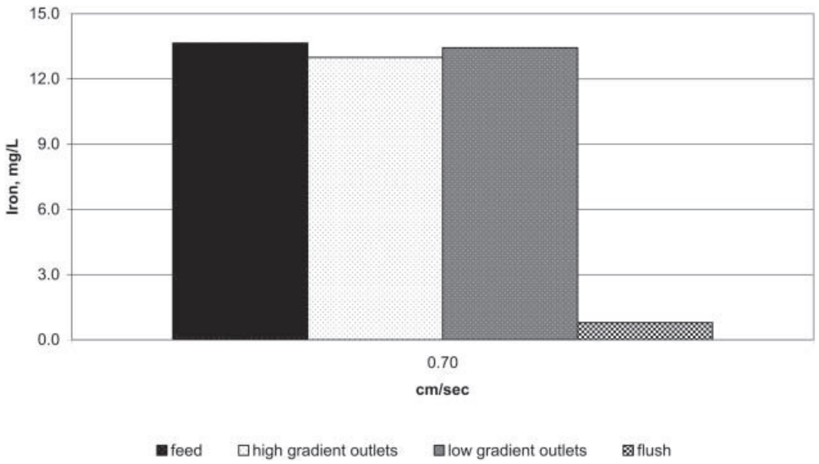


Figure 4. Hematite, nominally 460 nm, processed with no magnetic field present. A 97% mass balance was obtained and 2.9% of the particles were captured at 0.69 cm/sec superficial velocity.

assumed to contribute to the high-gradient outlet iron concentrations and is labeled as "flush-compensated high-gradient outlet." This concentration effect is conceptually the continuous flow approach for particle separation as described previously.

Table 1 details experimental results and conditions for separation experiments conducted with two separators having 0.007- and 0.005-in. channel depths (175 and 125 μm), varied superficial velocities, and applied magnetic fields. Flush-compensated high-gradient outlet, in mg/L, was calculated by adding the mass of iron from magnetic capture (flush) to the mass of iron intrinsically present in the high-gradient outlet sample. Simply, the mass of iron (as hematite) that was magnetically captured on the matrix, and subsequently released in a flushing procedure, is added to the mass of iron that exited the high-gradient outlet during processing and the concentration of iron is recalculated and entitled "Flush compensated high-gradient outlet, mg/L" in Table 1. For example, the 0.007-in. channel depth and 0.69 cm/s superficial velocity operated at 6T had approximately 0.008 mg of iron captured on the matrix from 18.2 mL of feed processed. This mass of iron is added to 0.049 mg of iron that originally exited the high-gradient outlet (with a high-gradient outlet sample volume of 10.3 mL) and effectively increased the concentration from 4.9 mg/L to 5.5 mg/L. The result of adding magnetically captured material to the high-gradient outlet concentration, and generating a "flush compensated" high-gradient outlet, is apparent when

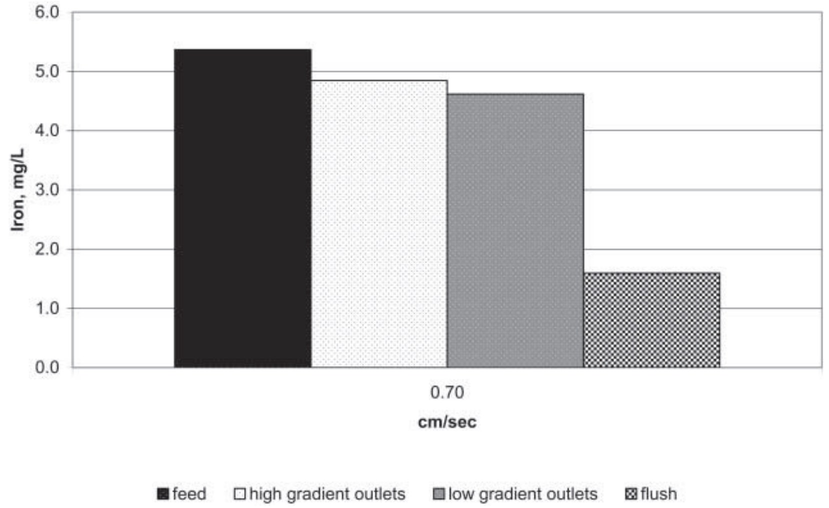


Figure 5. Hematite, nominally 460 nm, processed at a 6 Tesla magnetic field is retained within the separator (8.5% capture). A 97% mass balance was obtained at 0.70 cm/sec superficial velocity.

comparing these values with and without an applied field. When applying a magnetic field, and compensating for magnetic capture, each of the experiments conducted with an applied field had a "flush compensated" high-gradient outlet concentration greater than the feed concentration. In comparison, when processing material without an applied field, there is not an accompanying increase in concentration. This comparison holds true for each no-field experiment except for the 0.005-in. channel depth and lowest superficial velocity (0.16 cm/s). In this case, the very small channel depth (125 μm $\sim$ 0.005 in.) and very slow superficial velocity (0.16 cm/s) results in significant physical capture of the hematite particles.

In Table 1, mass balance (%) is determined by measuring the concentration of iron present in experimental samples (high-gradient outlet, low-gradient outlet, feed, flush), calculating the mass of iron in the experimental samples and dividing by the mass of iron processed through the separator. Material accountability in our system was typically in the $>$90% range. The capture percent is the mass of iron, from the flush sample, divided by the mass of iron processed through the separator obtained from the feed concentration and volume of feed processed.

The experimental run that approaches the conceptual best-case for continuous flow/no capture mode is for the 0.005-in. channel depth at the lowest

Table 1. Experimental results and conditions for two HGMF-SPLITT channel geometries.

Field, channel depth, and superficial velocity, cm/s	Feed, mg/L	[Iron] high-gradient outlet, mg/L	[Iron] low-gradient outlet, mg/L	Feed captured, %	[Iron] in flush compensated high-gradient outlet, mg/L	Mass balance, %	Volume of hematite suspensions processed, mL
6 Tesla							
0.007-in. depth							
0.69 cm/s	5.4	4.9	4.6	8.5	5.5	97	18.2
0.32 cm/s	6.1	4.8	4.4	14.7	6.6	90	22.6
0.12 cm/s	6.1	4.2	3.9	37.1	8.7	103	22.8
4 Tesla							
0.005-in. depth							
0.50 cm/s	12.1	9.0	6.8	22.0	13.5	90	18.8
0.45 cm/s	14.3	11.5	7.4	48.9	21.9	109	13.8
0.19 cm/s	15.1	5.3	4.2	82.0	32.0	113	22.3
No field							
0.007-in. depth							
0.69 cm/s	13.6	12.3	13.4	2.9	13.0	96	20.6
0.32 cm/s	13.6	11.2	12.9	2.7	12.9	91	22.3
0.12 cm/s	8.8	6.9	7.6	14.0	9.4	97	23.1
No field							
0.005-in. depth							
0.75 cm/s	13.2	10.6	8.2	5.9	11.6	81	18.7
0.38 cm/s	12.3	11.0	12.5	5.7	11.8	97	13.2
0.16 cm/s	13.1	8.3	7.6	29.0	15.5	90	24.1

superficial velocity used, 0.19 cm/s, while applying a magnetic field of 4 Tesla. An initial concentration of 15.1 mg/L was approximately doubled, to 32 mg/L, in the high-gradient outlet when compensating for capture due to the magnetic forces within the separator. An examination of the data for the two separators used to process submicron hematite with and without an applied magnetic field reveals that the presence of an applied magnetic field increases the amount of hematite magnetically captured. Further, a decrease in the superficial velocity within the separation channel also increases the amount of hematite captured.

The capture that occurs at lower superficial velocities is due to an increase in both magnetic capture and mechanical trapping. The decrease in superficial velocities allows for longer particulate residence times in the separator. The smaller particles have lower magnetically induced velocities due to smaller magnetic volumes, relative to larger particles, and increased residence times allow these particles more time to approach the active surface and be magnetically retained. Further, if the particles are magnetically captured, the decreased superficial velocity also results in decreased viscous drag forces, which minimizes shearing of the particles off of the active surface. The smaller shear force also contributes to increased physical trapping of the particulates on the face of the splitting plane, or other exposed physical trapping surfaces.

Figure 6 shows the capture percent (from Table 1) and illustrates that a greater percentage of hematite is captured with reduced separator channel depths, when applying a magnetic field, relative to no magnetic field, or when reducing the superficial velocities within the separation channel. For example, when processing submicron hematite through the 0.007-in. separator at 0.12 cm/s, 37% of the feed is captured when in the presence of a 6 Tesla applied field vs. 14% of the feed captured without an applied field. Similarly, for the 0.005-in. separator at 0.19 cm/s, 82% of the feed was captured with an applied field, whereas only 29% captured occurs without an applied field. The difference between the percent of feed captured is not as significant when superficial velocities are increased. For the 0.007-in. separator at 0.69 cm/s, an applied magnetic field captures 8.5% of the feed relative to 2.9% capture without an applied field. This trend continues with the 0.005-in. separator. The decrease in channel depth benefits separator performance by initially locating the separable particle closer to the magnetically active surface. Decreasing the superficial velocities allows the separable particles more time to be attracted to the active surface. The combined effect of decreasing channel depths and superficial velocities is a larger percentage of the particles being magnetically captured by the matrix material.

The current performance of the HGMF-SPLITT has not yet realized a significant enrichment for the paramagnetic particle-feed suspension when

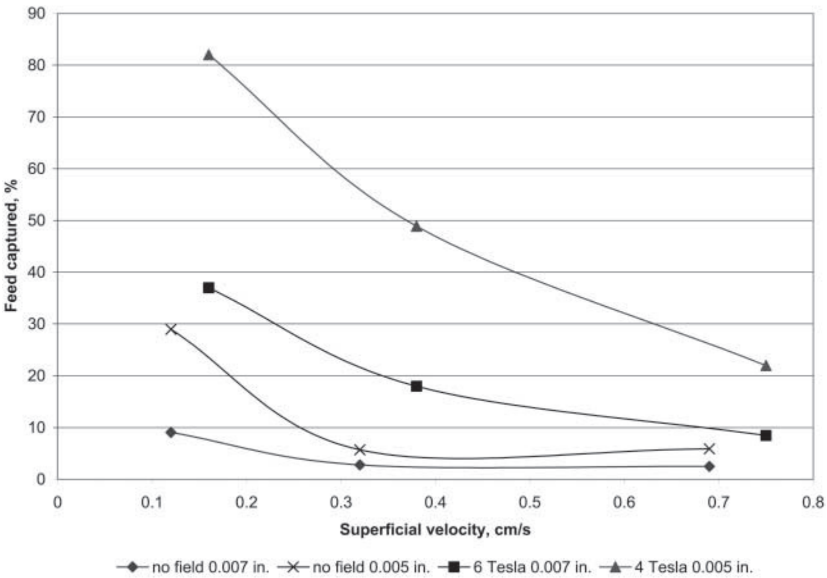


Figure 6. The effect of superficial velocity, channel depth, and applied magnetic fields to the capture of hematite particles.

operated as a continuous flow separator. There are some factors in operation and assembly of the separation device that may allow for improved continuous-flow performance. To optimize the system toward a continuous-flow/no-capture device, we have examined several variables including the uniformity and type of the magnetic matrix material, the type of pumping system, modification of the splitting plane edge, and further reduction of the channel depth. Improving the size uniformity of the matrix material (stainless steel powder), decreasing the nominal size of the matrix material, pulseless sample introduction, and further decreasing the channel depths should improve the overall performance of the HGMF-SPLITT. These optimization variables will offer a system with distinct advantages for both SPLITT and HGMS methodologies for particle separation.

The system presented here has some operational issues related to application as a continuously flowing high-gradient magnetic separator and further investigation is warranted to overcome these difficulties. The high magnetic field gradients generated by the steel wool powder result in magnetic forces great enough to influence the trajectory of a submicron paramagnetic particle in a SPLITT separator. Unfortunately, these forces are so large that capture of the particle onto the active matrix results. This, in effect, negates the continuous flow aspect of the SPLITT geometry and generates a typical

HGMS device. If attempts are made to decrease the magnetic force such that capture does not occur, it appears that the trajectory of the paramagnetic particle is not influenced enough and concentration of paramagnetic particles in the high-gradient outlet does not result. Further research into fine-tuning of matrix properties, channel geometries, sample introduction, and fluid-flow characteristics may allow for realization of a high-gradient, magnetic field, split-flow separator.

In summary, we have shown that the presence of a high-gradient magnetic matrix material in a SPLITT-type separator increases the range of particle sizes and magnetic susceptibilities addressable by magnetic SPLITT technology. The application of strong magnetic fields, low superficial velocities, and decreasing channel depths increases the amount of paramagnetic material retained within the separator.

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