An Inexpensive Magnetic Mineral Separator for Fine-Grained Sediment

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Introduction

This report describes a system designed to separate magnetic particles from fine-grained sediment or pulverized rock. The device is fashioned crudely after the separator described by Petersen and others (1986), but it differs in its ability to process large (about 2-20 g) samples and in its use of a simple magnet. Excluding the peristaltic pump, the separator is inexpensive to make—our version cost about $500 in 1996, including commercial glass-blowing and machine-shop costs. Less than one hour is required to process a sample, including sample preparation, set up, separation, and clean up. The separator is effective in removing magnetic particles with diameters less than 1 μm to more than 100 μm from clay- and silt-dominated sediments. It does not work well for sand-size particles, many of which bypass the magnet in the large-volume lower reservoir. The separator has not yet been tested for its effectiveness in concentrating bacteriogenic magnetic particles such as magnetosomes from magnetotactic bacteria.

The efficacy of separation varies with factors such as abundance, size, and mineralogy of magnetic particles. We have not systematically monitored the efficacy of separation, such as by comparing magnetic susceptibility and isothermal remanent magnetization (IRM) values of bulk samples before and after separation. However, high efficiency is indicated by values of $(IRM_{bulk}-IRM_{nonmag})/IRM_{bulk}$ being >0.9 for most measured samples.

The separator is not intended for coarse-grained materials, and it may not successfully separate sand-sized magnetic grains. Furthermore, pebbles or other large debris may block the bottom exit tube. If this occurs, a vacuum may develop in the lower reservoir, pulling the magnet bar into the reservoir and likely producing a geyser from the small hole in the top cap. Placing the system on a tray is recommended to contain any resultant flooding.

Magnetic particles can be removed from coarse sediment by a different process. We insert a strong cylindrical magnet into a thin-walled plastic sandwich bag or suitable (but usually more expensive) substitute. By repeatedly stirring a sediment slurry in a beaker around the covered magnet, we capture magnetic grains and transfer them to a second beaker of clean water. Repeating this process from the second beaker to a third beaker further purifies the separate. When working with sand, this simple separation technique is preferable to using the separator.

System Components

The system requires a specially made lower reservoir tightly covered by a top cap. The top cap holds a magnet, encased in a removable sleeve, that extends down into the reservoir (Figures 1 and 2). Sediment slurry drains from the bottom of the lower reservoir to a peristaltic pump through tubing, which in turn connects to a 500-ml upper reservoir (Figures 3 and 4). (See Appendix for detailed description of components.) Slurry pumped to the upper reservoir drains via tubing into the lower reservoir. As the slurry circulates through the system, the magnet in the lower reservoir captures magnetic grains in the sediment.

Operating Procedures

Sample disaggregation is critical for effective separation. After using a mortar and pestle if necessary to gently disaggregate the sample, place the sample in a beaker of distilled water. Add surfactant to the slurry (to decrease sediment clumping) and place the beaker in an ultrasonic bath.

Before the separation, the following system conditions must be met: (1) The unit is clean. (2) The pump is off and in forward position. (3) The pump tubing is attached to the lower reservoir and passes through the pump. (4) The pump tubing is inserted into the top of the upper reservoir. (5) The standard flexible tubing connects the upper and lower reservoirs; vacuum grease eases insertion of the tubing into the inclined intake of the lower reservoir. (6) A thin layer of vacuum grease is spread on the o-rings of the magnet casing and on the o-ring that seals the gap between the lower reservoir and top cap. (7) The top cap is off, placed upside down on the table or tray, with the magnet and casing inserted.

The separation proceeds with the following steps: (1) Stir the slurry while pouring it into the lower reservoir, filling the reservoir to a level just above the top of the inclined intake tube (so that most of the thinly-covered end of the magnet will be immersed when the cap is in place – add distilled water if necessary). (2) Cover the lower reservoir with the top cap, using pinch clamp jaws to compress the pieces tightly together. (3) Pour any remaining slurry through a cone funnel into the top of the upper reservoir and/or add distilled water until the upper reservoir is about half full. (4) Start the pump, monitoring the system closely for blockage or leaks. (5) After the operator has confirmed that the system is working properly, the system may be operated unattended. Allow the system to operate for about 30 minutes, checking occasionally for problems or varying flow rates. Although generally not necessary, the operator can monitor separation progress by placing a strong cylindrical magnet on another stand so that the end contacts the glass wall of the lower reservoir beneath the intake, near the bottom of the vessel. In the early stages of separation, magnetic grains are commonly seen against the vessel wall when quickly removing this magnet. When separation is complete, no such grains will be found.

Extract the magnet and magnetic grains using the following procedures: (1) Stop the pump. (2) Move the end of the pump tubing from the upper reservoir into a large (1000 ml) beaker and start the pump. (3) Drain the system completely, then stop the pump. (4) Unscrew pinch clamp and remove it and the top cap. (5) Hold the top cap/magnet over a petri dish, with a wash bottle of distilled water nearby. With the top cap in one hand, insert a small, weak magnet into the 5 mm opening at the top of the cap, contact the top of the magnet through the opening, and pull up gently but quickly. Magnetic grains will cling to the Teflon™ casing. (6) Holding both the top cap and the small weak magnet in one hand, with the other hand use a wash bottle to gently wash the magnetic grains from the bottom of the magnet casing. (7) After the first wash, pull the magnet and casing from the top cap and remove the magnet completely. Wash any remaining grains into the dish.

Use the following steps to dry the grains and transfer them to a small sample vial: (1) Decant excess water from the sample, holding a strong circular magnet to the bottom of the dish while decanting. (2) Dry the sample with a brief acetone wash. Hold the magnet to the bottom of the dish while pouring off acetone. (2) To transfer dry grains to a sample vial or microscope slide, we use small (12 x 1 cm), commercially available "magnetic tweezers" – a magnet plunger in a thin-walled aluminum case. Weighing paper, cut like a scoop, works well to scrape stubborn grains from the petri dish.

Reference

Petersen, N., Von Dobonek, T., and Vali, H., 1986, Fossil bacterial magnetite in deep-sea sediments from the South Atlantic Ocean: Nature, v. 320, p. 611-615.

Appendix:
Comments on Components

Upper reservoir. Pear-shaped separatory funnel, 500 ml, with Rotaflo™ stopcock. We modified a commercially available funnel so that the stopcock faced up instead of down (Figure 4).

Lower reservoir. Made by commercial glass blower (Figures 1, 2, and 3). The upper, flanged part is a standard #50 flat o-ring joint, having an outside diameter (OD) of ~80 mm. A standard o-ring should come with this piece (~66 mm OD; ~3.4 mm thick). The main body, the inclined inlet, and the bottom exit tube were added. We experimented with the angle (about 15^o above horizontal) of the inlet. If the angle is too steep, too much sediment will bypass the magnet; if too shallow, sediment builds up in the inlet. Sediment will accumulate in the inlet behind the tubing in any case under low flow rates, so the operator may need to vary the flow rate a few times during the separation to flush this area.

Magnet and magnet casing. We machined the Teflon™ casing of a 3" x 1/2" (7.6 x 1.3 cm) round magnetic stirring bar (Figure 1). (English units are given where necessary to describe a standard size for components.) The top was cut off so that the used length is about 60 mm long; the magnet will thus be above the open top and is easily inserted and removed from the casing. The lower portion of the casing was machined down to a diameter of about 9 mm to reduce wall thickness and generate a stronger field over the area to which the magnetic minerals will be attracted. Because the machining created circular grooves, we hand-polished the machined end of the casing using lapidary grits. Two circular excavations were made on the upper half of the casing to seat two o-rings, which are ~13 mm OD and ~1.7 mm thick.

Top cap. Machined from transparent plastic (polymethyl methacrylate; e.g., Lucite™) (Figure 1). The magnet and casing fit into the central cavity so that the thinly covered part of the magnet extends beyond the cap into the reservoir. The o-rings hold the covered magnet firmly in place.

Pump. We use a variable flow, peristaltic pump. Our particular pump was chosen for the option of doing separations in fluids other than water, for its sensitive control on flow rates, and for its forward/reverse flow direction control. Some types of sediment may clog at the bottom of the lower reservoir before starting the separation; in such cases, the operator will need to flush the exit of the lower reservoir by pumping gently upward toward the magnet (i.e., running the pump in reverse).

Pump tubing. Connects lower and upper reservoirs via pump (Figure 4). Our separator uses 1/4" (0.64 cm) inner diameter (ID), 1/16" (0.16 cm) wall silicone rubber tubing.

Other tubing. Connects upper reservoir to lower one with short length (Figure 4). 1/2” (1.3 cm) OD, 1/16” (0.16 cm) wall standard laboratory flexible tubing.

Pinch clamp for o-ring joint. Holds cap securely on top of lower reservoir (Figure 3). With spring-closed forked jaws and screw-locking device. Size 18/8 fits o-ring joint at top of lower reservoir.

Stand and various clamps. For organizing the system vertically (Figure 4).

Surfactant for sample disaggregation. We use a general purpose, non-corrosive cleaner available from laboratory-products suppliers.

Figure 1. Lower reservoir, top cap, and magnet with casing, drawn to scale.

Figure 2. Assembled magnetic-mineral separator (comprising the lower reservoir, top cap with magnet and casing, and tubing), drawn to scale.

Figure 3. Photograph of magnetic-mineral separator, assembled on stand with clamps.

Figure 4. Photograph of the entire system, including upper reservoir, separator, pump, and tubing.