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PO Box 1225 · 40637 Meerbusch – Germany
phone: +49 (0) 2132 93 57 60
fax: +49 (0) 2132 93 57 62
e-mail: Info@Filtech.de
web: www.Filtech.de

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SELECTIVE SEPARATION OF MAGNETIC PARTICLES BY MAGNETIC FIELD ENHANCED CENTRIFUGATION

Dipl.-Ing. Johannes Lindner*, Dipl.-Ing. Katharina Wagner,

Prof. Dr.-Ing. Hermann Nirschl

Institute of Mechanical Process Engineering (MVM),
Karlsruhe Institute of Technology (KIT)
Email: johannes.lindner@kit.edu

ABSTRACT

Magnetic filtration achieves high separation efficiencies. Magnetic Field Enhanced Centrifugation (MEC) is a method to clean a magnetic filter continuously, allowing the continuous separation of magnetizable particles out of a suspension.
A possible use is the separation of proteins out of a biosuspension using carrier particles. The presentation shows the selective separation of magnetizable particles out of a biosuspension. Theoretic background is the separation of magnetic filtration for the target particles and centrifugational separation for contaminants.

KEYWORDS

Magnetic Filtration; Centrifugation; selective Bioseparation; High Gradient Magnetic Separation

1. Introduction

Magnetic Field Enhanced Centrifugation (MEC) is a hybrid process which combines the advantages of magnetic filtration and centrifugation: the high separation efficiency of magnetic filtration and the continuous operation of centrifugation. Magnetic Filtration is a process for the separation of any kind of magnetic particles out of a suspension. Examples for applications are the removal of swarf out of gear oil or waste water treatment. Yet as centrifugation is a rather expensive task, cost-intensive applications are a more promising application. One possibility is the use in biotechnology.

Downstream processing in biotechnology is the most cost-intensive part in the production of bioproducts like proteins, a high number of purification steps reduces efficiency. An alternative approach consists of the use of surface functionalised particles with magnetic core. The use of these particles is established for analytic purposes. Nevertheless due to high particle costs and the lack of high efficiency separating devices, they are not yet used for downstream processing. The particle costs depend on the complexity of the production process and the particle quantities produced. Hence it seems possible to upscale particle production to overcome the limits.

For separation, High Gradient Magnetic Separation is well-established yet limited to small amounts of fluid. The necessity to stop the process for cleaning the wires avoids the use of this device for large quantities. In Magnetic Field Enhanced Centrifugation a magnetic filter is cleaned continuously by centrifugation, allowing the separation of larger quantities of particles out of fluid. Hence MEC allows the separation of large volumina of product.

The principle of Magnetic Centrifugation

In Magnetic Field Enhanced Centrifugation (MEC) a magnetic filter is placed inside of a centrifuge as shown in Diagram 1.1. Both turn at a specific rotational speed. A surrounding watercooled electromagnet induces a magnetic field on the wires. These create locally high field gradients, collecting and agglomerating particles on their surface. Big particle heaps detach by centrifugal forces from the wires and are gathered at the centrifuge wall.

This can be realised in an automatic process.

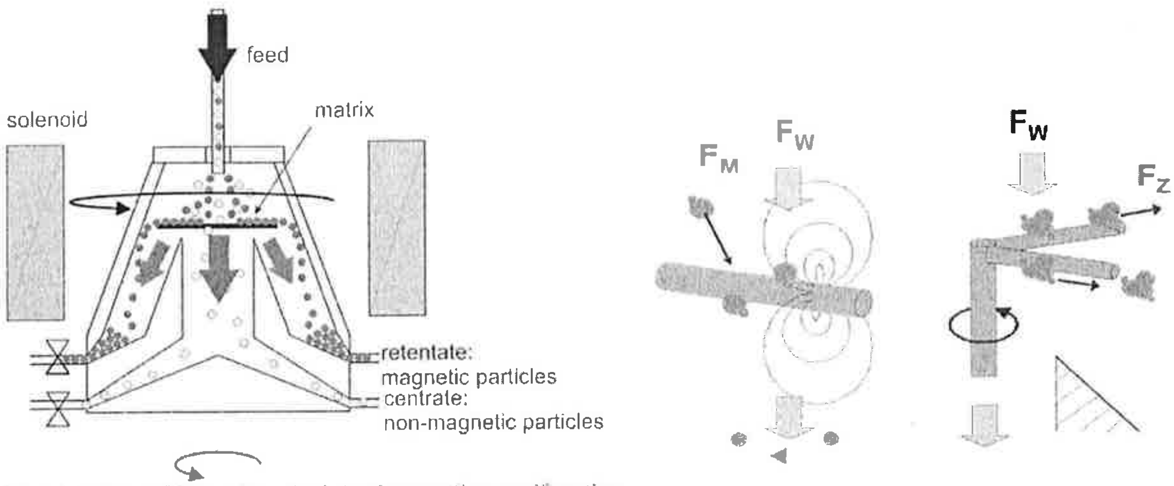

Diagram 1.1 and 1.2: The principle of magnetic centrifugation [1]

Magnetic forces decrease quadratically over the distance, but are huge at small distances, hence a fine filter has high separation efficiencies, while non-magnetic particles are not affected of the magnetic forces. Hence the achievable selectivity is very high. Yet in MEC there is additionally the centrifugational force that separates particles due to a density difference to the fluid. This affects as well magnetic particles, which usually have a high density due to their magnetic core, as non-magnetic particles. Hence the efficiency and selectivity of the process completely depends on the density of contaminants. Nevertheless only eliminating major contamination is sufficient in the process, as in subsequent washing steps small amounts of contamination can be removed easily.

2. Theoretic background

The force on a magnetic body in an inhomogenous magnetic field is given by the magnetization of the body M_P, its volume V_P, the magnetic field constant μ_0 and the gradient of the magnetic field strength H [4]:

$$\overrightarrow{F_m} = M_P V_P \mu_0 \nabla \mathrm{H} \tag{1}$$

The magnetisation M can be calculated as a function of the magnetic background field H_0 from the susceptibility κ_m, which is a function of the material parameter κ_{im} and a geometry constant D_m being 1/2 for a cylinder and 1/3 for a sphere. κ_{im} is for magnetite 500 at low field strength and 0,61 at 1T, for steel it is 2000 respectively 2,1 and above for very pure iron. κ_{im} >>1 is valid as long as saturation magnetisation is not reached [4]:

$$M = \kappa_m H_0 = \frac{\kappa_{im}}{1 + D_m \kappa_{im}} H_0 \xrightarrow{for\ \kappa_{im} \gg 1} \frac{H_0}{D_m} \tag{2}$$

In contrary to para- or diamagnetic materials, the magnetisation of ferromagnetic materials reaches a maximum, the saturation magnetisation. Usually the velocity of a magnetic particle close to a magnetic cylinder v_m is given as a function of $\Delta\kappa * H_0$ instead of M_D, based on the definition of κ [4]:

$$\kappa = \frac{dM}{dH} \tag{3}$$

For ferromagnetic particles κ is not constant but a function of the magnetic field H_0. M is almost constant once saturation magnetization is reached. Hence in the special but important case of reached saturation magnetisation of ferromagnetic particles a constant is replaced by two variables. Therefore a form is preferable that uses the magnetisation of particle M_p and wire M_D, which can be replaced by either the formulae mentioned above or the saturation magnetization.

By introducing the analytic solution of a magnetic cylinder as shown by Straton [2] and calculating the laminar flow resistance of a sphere, the flow velocity resulting relative to the fluid at any point is deduced in cylindrical coordinates r and φ as:

$$\overrightarrow{v_m} = \frac{2}{9} \frac{b^2 \mu_0 M_P M_D}{\eta} \frac{a^2}{r^3} \alpha \left[\frac{\left(\frac{a^2}{r^2}\alpha + \cos(2\varphi)\right)\overrightarrow{e_r} - (\sin(2\varphi))\overrightarrow{e_\varphi}}{\sqrt{1 + \frac{a^4}{r^4}\alpha^2 + 2\frac{a^2}{r^2}\alpha\cos(2\varphi)}} \right] with\ \alpha = \frac{\mu_p - \mu_f}{\mu_p + \mu_f} \tag{4}$$

In this case b is the particle radius, a the wire radius, η the viscosity and μ_P and μ_f the permittivity of particle respectively fluid. At $y = \sqrt{x^2 + a^2\alpha}$ is the change between repulsing and attracting forces. By setting $\overrightarrow{v_m} = \overrightarrow{v_0}$ there is one point (r_0, φ_0) where fluid and magnetic velocity are in balance and hence a particle does not move.

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For φ=0 and by setting a=r the term in parentheses cancels and for a fluid with low permittivity α tends to 1. Different approaches have been developed to calculate the capturing radius of a magnetic wire from v_m using this form of the equation, one of these being the equation derived by Gerber and Birss [3]:

$$Rc_a \cong \frac{3}{4} \cdot \sqrt{3} \cdot \left(\left|\frac{v_m}{v_0}\right|\right)^{\frac{1}{3}} \cdot \left[1 - \frac{2}{3} \cdot \left(\frac{v_m}{v_0}\right)^{-\frac{2}{3}}\right] \tag{5}$$

Hence it is possible to calculate the separation efficiency of classic magnetic filtration as well as magnetic centrifugation as shown in [1]. The total separation efficiency E_{total} is calculated out of the separation efficiency of every single stage $E_{s,i}$, the latter being determined by calculating the area that is covered by the wires A_c and the total are A_{total}:

$$E_{total} = 1 - \prod_{i=1}^{N} \left(1 - E_{s,i}\right) \quad \text{With} \quad E_S = \frac{A_c}{A_{total}} \tag{6}$$

Important parameters for the separation can be deduced form (4) and (5):

$$E \sim \left(\frac{H_0^2 * b^2 * \tau * a^2}{r^3 * \eta}\right)^n \tag{7}$$

In this case n is a parameter between 1/3 and 1 depending on the proportion of v_m/v_0. The magnetic background field H_0 only has got an influence as long as the saturation magnetization of particle and wire is not reached. The influence of the particle radius b is quadratic, yet increasing the particle size results in low specific surface, reducing the efficiency of carrier particles. In case of separation of unwanted particles out of media, the size of target contaminants cannot be influenced. The residence time τ depends on the process volume and hence magnet volume, which is the most important source of investment cost. The throughput has got a reciprocal influence on τ, resulting in a limit to the throughput by a decrease of the efficiency. The viscosity η depends on the mother liquid the resource is to be separated from and usually cannot be influenced. Hence while several parameters influence the separation efficiency, it is not possible to set most of these parameters. The only possibility left is to reduce the distance of the particle to the wire r and hence to use fine filters.

3. Methods and Materials

The suspension for testing is in the first part based on purified water, containing 2 g/l of coated beads with super-paramagnetic core. The mean particle size is about 1 to 5 µm. The centrifuge is custom-made. The water-cooled electromagnet used for separation has been produced by Steinert. The current used is up to 110 A resulting in a magnetic field of 0.5 T in the center of the magnet.

The centrate is continuously discharged from the centrifuge, while the retentate is kept batch-wise in the centrifuge. The concentration has been determined gravimetrically. The separation efficiency is calculated as the ratio of centrate and feed concentration.

$$E_i = 1 - \frac{c_{i,effluent}}{c_{i,feed}} \tag{8}$$

The feed for the second part is soy protein, taken from a process stream of Solae Denmark without further treatment. Antifoaming agent has been used in the tests.

Experimental separation

Separation out of water

While the separation of particles out of water is relatively easy compared to several other fluids, it is however an important task. Water treatment is a possible use of the magnetic particle separation. Different influences on the separation have been investigated such as the number of wires, the wire shape and the matrix diameter. As a consequence a specific matrix design has been kept. As Diagram 2 shows, the separation efficiency rises for high field intensity. As shown in the same diagram, different wire shapes and a different number of wires lead to higher separation as well.

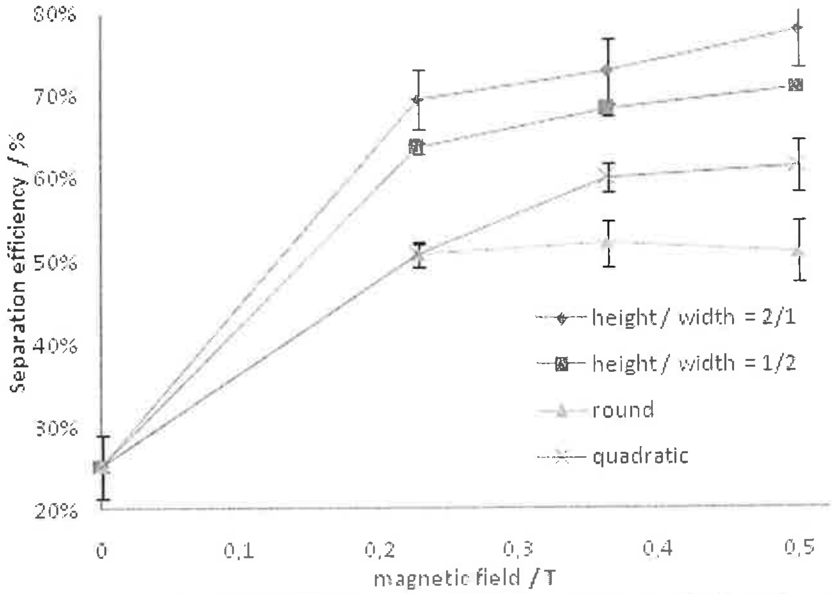

Diagram 2: The separation efficiency out of water at different matrix designs [1]

Separation out of a biostream

The separation of magnetic particles out of water is a relatively easy task compared to the separation out of several complex media. The processing of some media is, depending on the medium, more complicated than the separation out of water. For specific media, contamination of the environment and vice versa of the medium from the environment has to be avoided. Some media are prone to foaming or tend to form bridges, have high viscosities and other shortcomings. One medium chosen for testing is a stream of soy protein. Foaming is suppressed by using antifoaming agents. Contamination is only in the final process a serious problem but not in first tests on the process performance.

In the tests separating particles out of a soy protein stream, a high percentage of particles has been separated by centrifugation. Diagram 3 shows the separation at different parameters: at low throughput (rectangle curve) a high amount of particles is separated already by centrifugation.

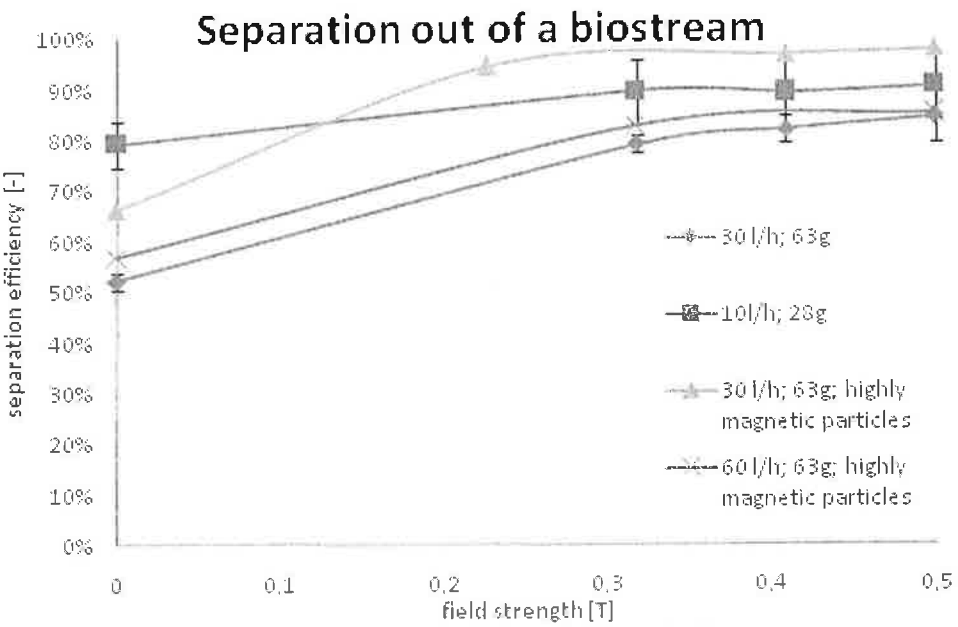

Diagram 3: Separation of magnetic particles out of a biostream

The centrifugational force is 28 times the acceleration of gravity. A throughput of 10 l/h is not interesting in an industrial process. By increasing the throughput to 30 l/h the separation reduces, despite increasing the centrifugational velocity and using different wire matrices. Another particle kind having a higher filling degree of magnetite shows better separation. Due to the higher amount of magnetite, the weight of the particles is higher and hence the separation by centrifugation at 0 T is higher as well. Although the separation by centrifugation seems to work well, at high throughputs as in industrial processes it is hardly possible to achieve the same separation efficiencies as by magnetic filtration.

A fine and optimised matrix achieves 98 % separation efficiency of highly magnetic particles at 110 A at a throughput of 30 l/h and 1500 rpm even in a lab centrifuge. Possibilities in a larger production centrifuge are superior, as well in terms of throughput as in terms of separation efficiency.

A washing step is necessary to eliminate rests of soy from the particles. As one of the particle kinds tested is slightly hydrophobic, soy constituents of about 18 % of the particle mass after drying agglomerate to this particle kind. While this is not selective separation, this effect can be used for the separation in waste water treatment and different applications for the separation of drug or hormone residues or heavy metals.

Conclusion

The separation of magnetic particles out of biostreams, in this case out of a soy stream, has been investigated using MEC. The separation of 98% of the particles out of soy has been performed at 30 l/h in a lab MEC. Washing steps are necessary to clean the magnetic particles of contaminants other than those agglomerated to the particles. While the selectivity of the process is limited, only a small fraction of the mother liquid finally contaminates the separated particles.

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