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MAGNETIC SEPARATION RESEARCH: A SURVEY OF SOME ACTIVITIES IN THE NETHERLANDS

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<u>Abstract</u> A survey of magnetic separation research at FDO Engineering consultants, the University of Nijmegen, and the Energy Research Foundation (ECN) will be given.

FDO efforts were primarily on coal cleaning using the high gradient magnetic separation (HGMS) technique. The Institute for Materials of the University of Nijmegen performs fundamental studies on magnetic flocculation. A continuous flow magnetic separator is briefly described. The Energy Research Foundation has built and operates an HGMS-system with superconducting magnets. Auxiliary equipment enables the execution of well controlled HGMS pilot scale tests. Intensive contacts and cooperation exist between scientists of the 3 research organisations, and with other industries

which are interested in magnetic separation applications.

1. HGMS STUDIES PERFORMED BY FDO

FDO Engineering Consultants is a subsidiary of Vmf-Stork. Vmf-Stork has over 14,000 employees of which 5,000 are based outside the Netherlands; annual sales are over 2,000 million Dutch Guilders; activities include the design, construction and assembly of machinery, production lines and complete plants for among others the food processing, the textile and paperprinting, and

suspended solids and micro-organisms from waste water, removal of oil from water, removal of heavy metals from waste water, recovery of immobilized enzymes, anaerobic treatment of industrial waste water, and production of drinking water ³⁾.

The application of HGMS in the production of drinking water was considered to be the most promising application. Subsequent laboratory scale experiments performed by FDO and TNO showed that with standard HGMS filters iron removal efficiencies of 76 to 95% can be obtained for subsoil water, flocculated surface water, and back flushing water of sand filters.

FDO commissioned the University of Delft to explore the application of HGMS in the chemical process industry. The recovery of catalyst particles was indicated as a promising application 4.

Further laboratory scale experimental work has been performed on micronized coal water mixtures, fly ash ⁵⁾, dust from sinter plants, basic oxygen furnace dust, and wolframite ore.

1.2. Coal cleaning by HGMS

Results of HGMS coal cleaning experiments on a number of German coal types have been published in Refs. 6, 7, 8, and 9.

The Netherlands Government commissioned FDO, through the Energy Research Foundation ECN-BEOP, to evaluate the applicability of HGMS for sulphur and mineral matter reduction in coal. This HGMS-project was part of the Netherlands National Coal Research Programme 10,11. The goal of the programme is to eliminate barriers to the re-introduction of coal in the Netherlands as a main source of energy.

The coal-cleaning programme within the coal research programme covered the HGMS process, the electrostatic separation process, and the microbiological desulphurization process.

Four German coal types were studied by FDO and partners.

A laboratory scale cyclic HGMS system was built which was able to process coal-water slurries. Coal sample size was 50-500

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grams, slurry concentration was coal:water= 1:4 (weight), slurry velocities in the HGMS filter 0.4 - 28 cm/s, magnetic field strength 0 - 8 Tesla (occasionally fields up to 14 Tesla were used). Bitter magnet facilities of the High Field Magnet Laboratory of the University of Nijmegen were used. Coal samples, pulverized to 100% < 75 μ m, were supplied by the Dutch Bank for Coal Specimen, SBN. Mineral matter composition and particle size distribution analysis of the coal was performed by the Energy Research Foundation. Magnetic properties were determined using magnetometer equipment of the Solid State Physics Department of the University of Nijmegen.

Mineral matter reductions of 60 to 75% and pyritic sulphur reductions of 40 to 95% were obtained at Btu-recoveries of 70 to 90%.

It was shown that diamagnetic mineral matter particles are removed from coal by HGMS by virtue of the presence of conglomerates of para- and diamagnetic matter particles.

The magnetic characterization revealed that the paramagnetic susceptiblity of pyritic particles in the 4 coal types was 5, 40, 46, and 93 10^{-6} emu/gr. Nevertheless, mineral matter particle size distribution analysis revealed that even the weakly paramagnetic pyrite particles of micronsize range are to some extent removed by HGMS.

It is therefore concluded that incomplete liberation of pyrite and mineral matter in coal is the most important factor for incomplete removal of these substances in HGMS coal cleaning. Quantitative measurement of the degree of liberation of pyrite in the four German coal types will be performed in the near future.

Grade (quality with respect to pyritic sulphur and mineral matter content) versus Btu-recovery relationships were determined for the coals. An important aspect is that the Btu-recovery increases if larger slurry velocities are used, see Fig. 1. Moreover, it was



FIGURE 1 Pyritic sulphur reduction versus Btu-recovery for Pattberg coal at slurry velocities of 1.7, 4.25, and 8.5 cm/s.



FIGURE 2 Ash reduction versus B/v for 4 German coal types.

found that the ash and pyritic sulphur reductions depend on the ratio of B/v (B magnetic field strength, v slurry transport velocity), see Fig. 2.

Therefore this result points to the use of high magnetic fields in HGMS systems if high Btu-recoveries are requested in conjunction with high pyritic sulphur and mineral matter reductions.

1.3. HGMS of fly ash

Results of HGMS fly ash upgrading experiments on 5 fly ashes originating from Dutch pulverized coal fired utility boilers have been presented in Ref. 12.

The Netherlands Government commissioned FDO, through ECN-BEOP, to evaluate the applicability of HGMS for fly ash upgrading. This project is part of the Netherlands National Research Programme on Coal.

HGMS processing of fly ash (dry, suspended in air) at low magnetic fields ($\sim 1-2$ Tesla) results in the production of a magnetic fraction which is enriched in iron. Some enrichment of the volatile elements Sb and As in the mags has been found and is thought to be due to the mags fraction having a smaller average particle size than the original fly ash.

For one fly ash type, which was produced at a higher firing temperature than the other 4 fly ash types, only 10 wt% could be collected in the magnetic fraction even at fields up to 8 Tesla. Apparently the magnetic part (the iron compounds) of this fly ash is associated with only 10% of the fly ash. Moreover, SEM-analysis revealed that the ash particles were not spherical in shape.

The non-magnetic fraction (tails) showed an increase in the loss on ignition value. It is indeed expected that unburnt or partially burnt coal particles (diamagnetic particles) are collected in the tails. For one particular fly ash with 11% loss on ignition, a tails fraction was obtained with a 26% loss on ignition value. The mags fraction had a loss on ignition value of 8%. The cost of upgrading of fly ash with dry HGMS was estimated b be Dfl. 9/ton of fly ash (\$3/ton) at a fly ash concentration in ir of 1 kg/m³. Fly ash concentrations of fly ash in the laboratory cale dry HGMS experiments were 0.2 kg/m³.



FIGURE 3 HGMS-filter section situated in the 6 cm bore of the Bittermagnet Of the High Field Magnet Laboratory of the University of Nijmegen.

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2. MAGNETIC SEPARATION RESEARCH PERFORMED BY THE RESEARCH INSTI-TUTE FOR MATERIALS, UNIVERSITY OF NIJMEGEN

In 1980 a research project on magnetic separation was started at the University of Nijmegen. This project, supported by the Netherlands Foundation for Technical Research (S.T.W.) is taking advantage of the technical facilities at the Faculty of Science and especially at the High Field Magnet Laboratory, where three 15 Tesla Bittermagnets, one 20 Tesla Bittermagnet and one 25 Tesla hybrid magnet (a second, 32 Tesla hybrid magnet is on its way) are available to scientists from all over the world. Bittermagnets have shown to be very useful in laboratory-scale experiments on e.g. highfield-high-velocity HGMS, Continuous Flow Magnetic Separation (CFMS), or in more fundamental studies as on magnetically induced flocculation; i.e. in experiments in which high magnetic fields are needed for relatively short periods of time.

Apart from the actual research by the Nijmegen group, of which a review is given in the following, a whole series of smallscale high gradient magnetic separation tests have been carried out for and together with several Dutch companies and industries from abroad on materials as edible oil, plating sludges, fly ash from electrical power plants, furnace dust from steel factories, coal, and sludge from the Rotterdam harbour.

It has been shown experimentally $^{13,14,15)}$ that suspensions of paramagnetic particles with intermediate magnetic susceptibilities can be destabilized by a magnetic field. For magnetically induced flocculation to occur the potential energy due to magnetic dipole-dipole interaction has to be sufficiently high to eliminate the energy barrier, created by a repulsive electrical double layer interaction and an attractive London-van der Waals interaction. Magnetic flocculation is therefore closely related to colloid stability and can be used as an alternative method to study this subject. Even diamagnetic particles can be made to flocculate under the influence of a magnetic field $^{16)}$. It is, however,



FIGURE 4 Survey of the magnet-hall of the High Field Magnet Laboratory of the University of Nijmegen

necessary to increase their net magnetic susceptibility, with respect to the surrounding fluid, by dispersing them in a solution of a paramagnetic salt. In this way the isoelectric point of the particles, in the presence of specific adsorption of ions, could be determined from measurements of the threshold field for flocculation. In addition, the process of magnetic flocculation of both colloidal and relatively coarse $(1 - 40 \ \mu m)$ particles has been described theoretically in cooperation with M.R. Parker of the University of Salford ^{17,18,19}. Here, explicit attention is paid to flocculation between dissimilar particles, the formation of aggregates other than doublets, and the kinetics of magnetic flocculation in comparison with the classical theory of rapid coagulation. The aspects of magnetic flocculation in colloids with particle sizes much smaller than 1 μ m are presently being investigated by means of light scattering and transmission experiments in high magnetic fields.

Sedimentation measurements on suspensions of paramagnetic particles have shown that the settling rate can be enhanced by more than one order of magnitude by applying a strong and inhomogeneous magnetic field. This can be utilized to separate particles on the basis of differences in their magnetically enhanced settling velocity by letting them settle in a fluid stream directed upwards (Continuous Flow Magnetic Separation). The particles with the highest settling rates (mags) are able to overcome the hydrodynamic forces of the fluid and can be collected at a certain place, while less magnetic particles are carried away by the fluid. Several experiments have been carried out to demonstrate the effect of this separation technique for both test slurries of fine particles ²⁰⁾ and slurries with many components and with a broad particle size distribution, like fly ash and paramagnetic ores ²¹⁾. Although the magnetic forces involved in CFMS are several orders of magnitude smaller than in HGMS, the separation is very effective, while the continuity of the process

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is a great advantage. For application in industrial processes, however, CFMS is probably more suited to treat mineral slurries prior to the actual processing stage.

In addition to the study on magnetic flocculation in colloids with ultra fine paramagnetic particles, experiments are carried out to investigate the characteristics of particle capture in high gradient magnetic separation of ultra fine colloids. For such small particles it can be expected that diffusion due to Brownian motion influences the capture process. Preliminary experiments have indicated that empirical laws, describing the capture of relatively coarse particles by a filter matrix, have to be corrected for the case of ultra fine particles.

3. MAGNETIC SEPARATION AT THE ENERGY RESEARCH FOUNDATION ECN

For about one year an HGMS-test facility is in operation at ECN; the parameters of this facility are such that pilot-scale experiments can be conducted.

The facility has been built to carry out application directed research as support to the activities of Dutch industries in this field.

Heart of the facility is a superconducting magnet of 4.5 Tesla, which has been constructed in the workshops of ECN a few years ago and has been used since then for stability measurements on superconductors.

Figure 5 gives an impression of the HGMS test rig. The helium dewar in which the magnet has been placed has a free bore of 149 mm; the length of the magnet is 400 mm and the length of the dewar amounts to 1450 mm.

From the beginning on there has been the intention to keep the system mobile in order to run it, when necessary, under real process conditions at the place where the process is located; furthermore the facility is used exclusively for magnetic separation enabling to meet the requirements of the experiments in a flexible





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FIGURE 6 "Malvern" particle sizer for in situ measurements

Faraday-method (constant H $\frac{dH}{dx}$). For these measurements a Varian 9-inch water-cooled magnet is available; the magnetic force exerted on the sample is measured by a Sartorius 2004 MP 6 microbalance.

In cooperation with Dutch industry a programme has been started for searching new applications in the field of magnetic separation. A lot of industries handling large volumes (coal, fertilizer, drinking water etc.) and/or strongly magnetizable materials (steel works) have been approached in order to inform them on the potentialities of magnetic separation. Testing of a great deal of samples originating from these industries is underway at this moment.

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