HIGH EXTRACTION MAGNETIC FILTRATION OF KAOLIN CLAY

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(Received 3 *January* 1976; *and in final form* 3 *February 1976)*

Abstract-A new magnetic separation technique combines high magnetic fields, extreme gradients, and controlled retention time to separate feebly magnetic mineral contaminants from kaolin. Extraction of pyrite, siderite, hematite, iron stained anatase, and mica by magnetic filtration of kaolin significantly upgrades brightness of many sub-marginal deposits and reduces consumption of reagents in processing.

EVOLUTION OF THE PROCESS AND EQUIPMENT

The kaolin development brings together five concepts that make practical large scale use of high extraction magnetic filtration (HEMF). These include retention time, extreme gradients, high intensity fields, electrokinetic dispersion, and efficient design of large magnets. Each of these elements was known and available but never refined and optimized in magnetic separation. The key breakthrough in development of high extraction magnetic filtration was the finding that separations on micrometer and submicrometer kaolin suspensions required very slow transit velocities and maximum exposure to collecting elements to allow capture of paramagnetic particles. To achieve particle capture, the magnetic attraction of a collecting element must overcome the viscous drag of the transporting fluid. Therefore, it is necessary to operate at much lower flow velocities (less than 2 cm/sec.) than had ever been employed in practical separations.

Discriminating control of slurry exposure in the collecting zone of a magnetic filter is best expressed by the concept of retention time. It is the subject of a basic process patent on kaolin, U.S. 3471 011 (Iannicelli, Millman and Stone assigned to J. M. Huber Corp,). All of the surge of activity in high extraction magnetic filtration since 1966 was spawned by this recognition of controlled retention time and the implicit relations of filter path and flow velocity.

The high gradient magnetic separator was patented by S. G. Frantz in 1937 (U.S. 2074085) and has found extensive small scale use in the ceramic industry. This device utilized a canister filled with a matrix of screens fashioned from thin sharp ribbons of 430 magnetic stainless steel. This separator provided a large amount of high gradient collecting surface. The open geometry of this matrix only occupied $10-20\%$ of the magnetized canister volume and allowed $80-90\%$ of the volume to be filled with slurry undergoing separation. A major limitation of the Frantz Ferrofilter® in the processing of kaolin was the relatively low magnetic field of 1.5 kG. In 1955, G. H.

Jones introduced the first high intensity wet separator (British patents 768451 and 767124) which generated fields of up to 20 kG.

PROTOTYPE DEVELOPMENT OF HEMF EQUIPMENT

To exploit the concept of retention time, it was necessary to devise a new magnetic separator combining the high gradients of the Frantz separator and the high intensity fields of the Jones separator. This critical fusion of separator technology was achieved (Iannicelli, 1967) by constructing a hybrid separator utilizing a canister filled with high gradient Frantz screens installed in the field gap of a Jones machine. This hybrid device generated an average field of nearly 10 kG throughout a relatively large volume of 10 gallons. The new hybrid separator furnished a startling increase in efficiency over the previous Frantz and Jones separators.

This led to design of prototype equipment generally resembling a Frantz Ferrofilter®, but modified to generate fields of up to 20 kG. Stainless steel wool had been shown to be more effective on kaolin than coarser ribbon Frantz screens and the prototype incorporated this refinement. This production prototype high extraction magnetic filter (HEMF) consisted of a hollow conductor solenoid surrounding a canister 20 in. dia. and 12 in. high. The solenoid was surrounded by a heavy box-like enclosure which served as a return circuit for magnetic flux generated by the coil. This small filter generated a field of 20 kG and required 300 kW of power. Throughput was about 2 TPH (dry basis). Estimated processing cost was about \$2.00 per ton which made the process of interest only for specialty kaolin applications.

DESCRIPTION OF HEMF EQUIPMENT

HEMF equipment in use consists of a filter canister, typically 84 in. dia. packed with a matrix of compressed magnetic stainless steel wool. The canister is

Fig. 1.

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surrounded by hollow conductor copper coils which energize the entire canister volume to a field of 20 kG. Coils are in turn surrounded by a boxlike enclosure of steel plate which completes the magnetic circuit of the iron bound solenoid. Slurry is pumped through the filter bed by a suitable system of manifolds. Despite the high fields of these magnetic filters, modern magnet design allows them to operate at relatively economic power levels of 400-500 kW. Figure 1 shows an 84 in. separator undergoing tests at the manufacturer's plant.

DESCRIPTION OF PROCESS

HEMF units are operated in the manner of depth filters on a batch or cyclic basis. The filter bed matrix is magnetized and slurry pumped through to yield a filtered or nonmagnetic product. When the matrix is saturated, water is fed into the filter at a comparable rate to displace the nonmagnetic product. The magnet is then de-energized and the bed flushed with high velocity water to remove magnetic contaminants. Processing of the product is resumed after re-energizing the magnet. Flow velocity can be varied from 40 in./min-lO in./min, corresponding to a slurry retention time of $30-120$ sec in a magnetic field. Filtration rate at 30 sec retention corresponds to 24 gallons/ft²/ min.

UTILIZATION IN KAOLIN

HEMF equipment is employed to brighten kaolin by extraction of submicron feebly magnetic contaminants. Removal of only $1-2\%$ of discolored ferruginous contaminants, based on original kaolin, typically brightens kaolin 2-4 General Electric Brightness units. In extreme cases, brightness improvement may be as low as 1 or as much as 30 GE units, depending on the nature of the mineral contaminants in the kaolin.

These improvements can be utilized to (a) mine lower quality crude kaolin, thereby extending reserves (b) reduce the consumption of leaching chemicals (c) produce super brightness clays (d) allow use of hard media to delaminate kaolin without discoloration (e) produce clay products having superior end use performance in ceramics, glass and catalysts because of reduced mineral contaminants.

EXPERIMENTAL PROCEDURE

A series of kaolin samples was dispersed in water \aleph with 0.2% tetrasodium pyrophosphate to furnish deflocculated slurries having a solids content $20-30\%$. Each slurry was subjected to high extraction magnetic filtration on a PEM 5 in. pilot plant magnetic separator. Separation tests were carried out at 20 kG using a canister 2.5 cm dia. \times 50 cm high containing 6% by volume of "medium" 430 magnetic stainless steel wool.

Each slurry was pumped vertically through the magnetized canister at controlled rates designed to give retention times ranging from 30 to 240 sec. The feed phase with slurry lasted 6 min during which a nonmagnetic product was collected from the canister. At the end of this period, the canister was rinsed with water at the same flow rate as that at which the slurry was processed. Upon completion of the rinse phase, the magnet was de-energized and high velocity flush water was introduced to recover the magnetic fraction trapped in the canister. Following the flush phase, the cleansed canister was returned to service for another complete test cycle. The non magnetic product from each test was filtered, dried and tested for brightness by standard TAPPI methods. Chemical analysis for Fe₂O₃, TiO₂, K₂O were conducted by X-ray fluorescence.

RESULTS AND DISCUSSION

Responses of submarginal kaolin from lone, California to magnetic separation are shown in Figs. 2-6. As a result of HEMF processing, it is possible to convert all of these clays from unusable submarginal deposits to usable kaolin reserves.

Figure 2 shows the response of a submarginal kaolin to HEMF at 20 kG , with 0-2 min retention time. The original GE brightness of this clay was about 73. Crude selection normally requires a minimum brightness of 78-79 in commercial kaolin processing. After 30 sec retention, the brightness of this sample rose to 79. This was accompanied by a decrease in iron content from 0.58 to 0.47% and a dramatic reduction in $TiO₂$ content from 1.9 to 1.2%. After 2 min retention, brightness of this clay was 82 and th final $TiO₂$ analysis was 0.8%, or less than half of the original.

Figure 3 shows a submarginal kaolin of 75 brightness with an unusually high $TiO₂$ value of 3.36. After

Fig. 3.

30 sec magnetic retention, brightness reached 78 and the TiO₂ content was reduced to a value of 2.70% . After 2 min retention, the brightness of the clay was about 82, allowing it to qualify for use as a paper filler without further processing. An interesting byproduct of this treatment was reduction of K_2O from 0.30 to 0.22% and finally to 0.19%. This represents a 37% decrease in mica content and is a very disirable improvement in the fired properties of this clay in ceramic outlets.

Figure 4 shows the unusual and spectacular response of a very discolored kaolin which would never be considered a useful reserve prior to the advent of High Extraction Magnetic Filtration. After one minute retention time, brightness rose from 49 to 70. $Fe₂O₃$ content decreased from 2.3 to 0.7% and TiO₂ content decreased from 1.9 to 0.8%. After 4 min retention, this kaolin achieved a remarkable brightness of 79 and $Fe₂O₃$ and $TiO₂$ contents of 0.5 and 0.4%. $K₂O$ content decreased from 0.3 to 0.18% after 4 min, indicating significant removal of mica. As a result of magnetic treatment, an utterly unusable kaolin was converted to filler brightness. Subsequent chemical leaching of this whole fraction clay resulted in an 89 brightness which would put this clay in the premium brightness range.

Figure 5 shows the dramatic reduction of $TiO₂$ in a sideritic clay. The original $TiO₂$ content of 2.38% decreased to 1.1% and finally to 0.71% after 2 min retention. G.E. brightness increased 12 points from 62 to 74. Potassium oxide content of this clay was reduced from 0.21% to 0.14% after 2 min retention. Figure 6 shows the response of a gray lignitic kaolin (containing pyrite) to magnetic separation. Although the brightness increase was only 14 points over a retention span of 4 min and the final product submarginal with respect to brightness, the results are nevertheless significant. The $Fe₂O₃$ content decreased from 2% to 0.5% and the TiO₂ reduction was parallel. K₂O content was halved from 0.28 to 0.15%.

The most significant aspect of this experiment was the striking reduction in the sulfur content of this clay. The initial sulfur value was 0.72% and the final sulfur content after four minutes retention was 0.08%. This represents a 90% reduction in the sulfur content of this clay through the nearly complete removal of micron and submicron pyrite by High Extraction

Fig. 7.

in that it demonstrates removal of pyrite in a very difficult fine particle size range. This and other results indicated that magnetic separation of pyrite from coal is feasible and that High Extraction Magnetic Filtration of coal is a viable method of coal desulfurization.

Figure 7 shows the response of a good quality Georgia kaolin $(80\%$ minus two microns) as a result of HEMF processing. Even though the $Fe₂O₃$ content was reduced only 10% and the TiO₂ content only 25%, the brightness improvement of 2-3 points is commercially significant. A low retention time of 30 sec was sufficient ro raise unbleached brightness. from 84.0 to 86.5. Normally, this would require treatment of this kaolin with from 4 to 81b of zinc or sodium dithionite leaching chemical per ton of clay. Magnetic separation allows this brightness level to be reached without leaching reagents. In practice, 1-2Ibs. of leaching chemical may be used merely to

improve shade of kaolin. In any case there would be a saving of leaching reagent ranging from 2 to 71b per ton of clay. HEMF treatment of kaolin under these conditions costs less than 2 Ib of dithionite reagent.

At a 2.0 min retention time, brightness of this clay reached the 88 range. This can be converted to an 89 brightness after leaching, which allows the clay to meet premium or super brightness specifications and command a significant increase in price.

Removal of iron stained anatase, siderite, and pyrite as shown in Figs. 2-7, as well as removal of hematite, tourmaline, and other ferruginous minerals from kaolin has enormous significance to the beneficiation of other industrial minerals ranging from asbestos to zircon. Mineral slimes extracted by HEMF from kaolin include iron stained anatase and quartz, hematite, mica, tourmaline, siderite and pyrite. Depletion of iron values in clay varies from 10 to 90% of original iron content. Bulk magnetic susceptibility of magnetic fractions is as low as 35×10^{-6} emu/cc, and iron content of magnetic fractions is usually less than 5% (expressed as $Fe₂O₃$).

Figures 2–6 show HEMF response of kaolin containing relatively high amounts of $Fe₂O₃$ and $TiO₂$ while Fig. 7 shows the effect on a kaolin with a much lower $Fe₂O₃$ and TiO₂ content. In all of these cases, iron stained anatase is the principal mineral contaminant extracted by HEMF.

ECONOMICS

Economics of magnetic separation on large (84 in.) HEMF equipment are extremely attractive compared to that of small prototype units. This is due to efficient design coupled with the fact that the capacity of HEMF separators increases with the square of the diameter while the amount of copper conductor increases linearly with the diameter. Operating costs for an 84 in. unit are estimated in Table 1. Magnetic Separation in kaolin processing is a comparatively low cost and versatile technique. By variation of retention

time, it is possible to achieve differing degrees of magnetic extraction at acceptable variations in costs. The HEMF process is not only low in operating cost, but also furnishes high yield (98-99%) of kaolin product based on feed.

CONCLUSIONS

High extraction magnetic filtration is a versatile separation technique which is especially well suited for investigative studies on clay minerals. It is also a practical economic process which has been thoroughly proven in large scale commercial practice since its development by the kaolin industry of Georgia.

The concept of retention time which resulted from the early work on kaolin is still the most critical single parameter in use of high extraction magnetic filtration both in kaolin and other areas. Retention

time has the added practical significance in that it directly fixes throughput of any given magnetic filter volume without reference to geometric considerations of flow velocity and canister length.

The kaolin separation problem represents the most difficult application of magnetic separation ever commercialized. Up to now, the only industrial use of the process is in the kaolin industry where it originated. However, the technology and experience gained from kaolin processing can now be applied advantageously to beneficiation of other industrial minerals, concentration of metallic ores, desulfurization of coal and clean up of effluent streams.

Acknowledgement-The author is indebted to Mr. N. Halaka and Dr. J. Negrych as well as to the Interpace Corporation for their assistance and their permission to publish the data on lone kaolin samples.