ADVANCES IN FILTER AID AND PRECOAT FILTRATION TECHNOLOGY

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ABSTRACT

The use of filter aids and precoat filtration is ubiquitous in a wide number of industries, including chemicals, food processing, pharmaceuticals, mining, municipal (potable) water treatment and waste treatment. World Minerals Inc., the parent company of Celite and Harborlite, and other organizations have recently made major advances in filter aid technology. These advances have now pushed the envelope of performance and purity with a wide range of new filter aid products.

Filter aids are inorganic mineral powders or organic fibrous materials which are used in combination with filtration hardware to enhance filtration performance. Commonly encountered filter aids include diatomite, perlite and cellulose, and some of these materials have been in use for over seventy-five years.

A process trade-off encountered in filter aid technology is between the permeability of the porous media and its turbidity removal properties. Filter aid products are produced in a family of grades over a wide range of permeability ratings. Each filter aid family has a characteristic curve defining this trade-off in either a pressure or vacuum filtration mode. In general, the tightest marine diatomite can remove submicron size turbidity, but as in depth filtration, the practice is to select a filter aid which removes only the size of turbidity necessary to achieve the desired clarity.

World Minerals has developed several new families of filter aid materials capable of shifting the trade-off curves to achieve the same or better level of turbidity removal but with greater permeability compared to established grades. In addition, these advanced filter aids can achieve filtration performance using less weight of filter aid addition, and also greater filtration cycle lengths, in terms of volume processed. The properties of these new filter aids and examples will be presented comparing the performance of these new materials to existing products.

INTRODUCTION TO FILTER AID TECHNOLOGY

One of the most widely used and robust means of microparticulate separation in liquid systems today is highly porous powdered media, often called filter aids. Powdered media provides versatility, high solids loading capacity, high throughput or high clarity (depending upon the filtration objective), ease of scale-up and frequently the lowest cost option in any filtration process. Recent technical advances have resulted in new filter aid products with improved purity and performance, and have stimulated a much greater range of applications where filter aids can be used.

Filter aids are used in conjunction with a variety of filtration equipment to enhance or enable to solid-liquid separations. Typical filtration hardware, in which filter aids might be employed includes:

- ✓ Filter Press and Chamber Filters
- ✓ Horizontal and Vertical Leaf Filters
- ✓ Candle or Tubular Filters
- ✓ Rotary Vacuum Precoat Filters
- ✓ Belt Press Filters
- ✓ Lenticular or Cassette Depth Filters

Selection depends upon the size of the specific application and the filtration objective. For example, in enzyme and antibiotic production, rotary vacuum filters are typically used as a primary filtration step to remove high level of cell mass solids. Given the limited pressure differential, a rotary vacuum filter is well suited for highly permeable filter aids. Filter press and chamber filters are more suited for downstream clarification and polishing filtration.

Integral to each filtration device is a filter septum or media, which can be a depth filter sheet made of paper, fabric or bonded filter aids (see below), or a metallic or plastic woven screen. In static, fixed-bed filtration the unwanted solids collect or accumulate on the filter septum. These solids lack sufficient permeability to permeate fluid and the filtration proceeds very slowly or terminates. The introduction of a dynamic or continuous filter media changes the composition and the permeability of the accumulated cake.

Filter aid as a precoat is applied in advance of filtration on the septum to protect against the penetration of unwanted solids and premature blinding of the media. Filter aid is also used as a bodyfeed to the liquid being filtered. The solids of filter aid and turbidity continuously build up a cake on the surface of the media until the space between adjacent leaves is filled up or the pressure reaches a maximum. The addition of bodyfeed increases the permeability in the accumulating filter cake, restricts solids movement, provides channels for filtrate recovery and extends cycle length. The optimization of bodyfeed addition in the process is a subject found in other reviews. (1, 2)

Filter aid types used in solid-liquid separations include: inorganic mineral powders, which comprises processed diatomaceous earth, known as diatomite; perlite, a vitreous aluminum silicate of volcanic origin; and organic fibrous materials, such as α-cellulose and cotton linter. A mineral filter aid of organic origin, introduced in recent years is rice hull ash. Synthetic silicates made from siliceous filter aids can be used as both adsorbents and filter aids.

Depth filters themselves contain diatomite, perlite and cellulose, plus resins and strength additives. These products, when used without supplemental filter aids, are suited to low solids applications, as they tend to blind quickly when subjected to moderate-to-high level of solids. Their capacity can be increased by the addition of filter aid, as a precoat and as bodyfeed into the unfiltered liquid.

POROUS MEDIA TYPES

Diatomite, perlite and cellulose are the most widely used porous media (filter aids) in dynamic process filtrations, with a high percentage of fine filtration applications using diatomite. Scanning electron micrographs of these porous media types are shown in Figure 1.

Diatomite

Diatomite is obtained from diatomaceous earth, a sediment greatly enriched in biogenic silica in the form of the siliceous frustules of diatoms, a diverse array of microscopic, single-cell algae of the class Bacillariophyceae. These frustules are sufficiently durable to retain much of their structure through long periods of geologic time and through thermal processing. Diatomite products are characterized by an inherently intricate and highly porous structure composed primarily of silica, along with impurities of alumina, iron oxide, and alkaline earth oxides.

One major advance in diatomite product technology is the development of Celpure™, a highly purified diatomite with enhanced performance. During the manufacture of Celpure the impurities normally present in filter aids are removed thereby yielding a high silica content and ultrapure product. Celpure grades have lighter density than conventional diatomite filter aids. This enables using less filter media (by weight) to match or surpass the performance of an existing filtration process. Figure 2 is a series of scanning electron micrographs showing the surface of a single conventional diatom and Celpure diatoms, revealing that most of the visible impurity particles are absent from the Celpure sample.

Perlite

Perlite is a naturally occurring volcanic glass which thermally expands upon processing. Perlite is chemically a sodium potassium aluminum silicate. After milling, a porous, complicated structure is present, but because its structure is not as intricate (or tortuous) as that of diatomite, perlite is better suited to the separation of coarse microparticulates from liquids having high solids loading. Perlite is lower in density than diatomite and this enables using less filter media (by weight). Perlite and diatomite are useful functional filtration components of depth filter sheets and pads.

Cellulose and Other Organic Media

Cellulose filter media is produced by the sulfite or sulfate processing of hard woods. Cellulose is characterized by its high aspect ratio, which enables it to precoat a septum very easily. It is most often used in that capacity in combination with diatomite. Like perlite, cellulose possesses a less intricate structure than diatomite. Attempts have been made to add structure. These include fibrillating the strands. A new, microcrystalline cellulose material from Germany, Vivapur, has reportedly improved filtration properties compared to regular α-cellulose. Cellulose also has the ability to operate in elevated pH environments above 10, making it frequently used in the chlorine-caustic industry to filter the brine feed to electrolysis membrane separators. Another application of cellulose is in treating machining oils and cutting fluids, to break the emulsion or to trap metal fines. Cellulose can be burned out after the filtration to recover the metal particles.

Other organic media include potato starch particles, cotton linter and polymeric fibers and flakes. These materials can help disperse diatomite in some systems or are specific to certain applications. An unusual mineral filter aid of organic origin is the ash from the combustion of rice hulls. This material has a high silica content and a residual carbon char and has been found to be useful in waste treatment and stabilization of hazardous materials.

GRADE SELECTION AND OPTIMIZATION

Filter aid products come in a family of grades over a wide permeability range. Permeability is a constant used to describe the intrinsic fluid flow of a liquid through a powder media. A thorough discussion of filtration mechanics can be found in numerous reviews. (1, 2, 3)

The selection of the appropriate grade of porous media has traditionally been made using a stochastic approach of numerous lab-scale or pilot-scale trials. While there is no substitute for bench scale studies, there are some guidelines to narrow down the grades to be screened. There are four criteria to consider when selecting an appropriate grade of porous media.

1) Filtrate Clarity

Most processes have a clarity specification that needs to be met or exceeded. Achieving stringent clarity specifications can extend the life of downstream process units or the shelf life or esthetics of the product being filtered. If the liquid being filtered is waste water, then the clarity specification might be set by a permit or regulations. The filtrate clarity achievable is dictated by the grade selected and the nature of the turbidity removed. Once a grade is chosen, its level of usage (bodyfeed addition) combined with the available differential pressure to induce flow will control the volume of unfiltrated feed that can be processed by a given filtration area.

A process trade-off in filter aid grade selection is between the permeability of the porous media and its turbidity removal or retention properties. Each filter aid family has a characteristic curve defining this trade-off in a pressure or vacuum filtration mode. In general, the most retentive filter aid products are diatomite of marine origin. The tightest marine diatomite can remove rigid turbidity below 0.5 μm and deformable turbidity smaller than 0.25 μm.

New families of filter aids shift the curve to enable finer turbidity removal without a sacrifice of permeability compared to commercially available diatomite, such as the widely used Celite® grades.

2) Product Throughput

Product throughput and filtrate clarity are tightly linked when it comes to grade selection. The goal is to select a grade that achieves the desired clarity and maximizes throughput. By selecting a grade that is too fine, the clarity specification can be exceeded but the throughput rate may be extremely low or differential pressure correspondingly too high.

3) Product Yield or Recovery

In many filtration applications, the desired product is filtered with the porous media. At the conclusion of any filtration cycle, the accumulated solids can be washed to maximize product recovery. It is important to confirm that the product of interest does not interact with the filter media (unless it is a desired function of the filter aid to be an adsorbent). Product recovery issues can be improved by selecting a more permeable grade of filter aid. As the porosity of the filter aid increases, the surface area and any associated non-specific interactions decrease.

4) Product Stability and Purity

Careful attention should be paid to both soluble and insoluble metals and impurities when selecting a filter media grade. High concentrations of soluble metals can oxidize various food, chemical and pharmaceutical products, and alter product stability. Even solid state metal oxides can function as sources of catalytic oxidation activity. The use of high purity materials for filtration will help reduce or eliminate product contamination and degradation issues further downstream.

The demand for high purity and high performance filter media has led to the development of a new generation of porous media. These products, introduced under the name Celpure have substantially greater purity than other commercially available diatomite filter aids.

In addition, Celpure's reduced density offers greater filtration capacity with a corresponding reduction in powdered media consumption and reduction in disposal costs. (4) Filtration processes with Celpure grades typically use less media due to a combination of the higher solids loading capacity, lower bulk density and improved flow properties of the media for a given clarity requirement. This improved performance results in longer cycle time lengths and greater liquid being filtered or a reduction in overall processing times compared with conventional grades of diatomite.

SYSTEMATIC METHODS DEVELOPMENT APPROACH TO GRADE SELECTION

As discussed in the previous section, the overall goal of a solid-liquid separation process is to achieve optimum clarity and maximum throughput while minimizing pressure and product losses. This section discusses the clarity and throughput trade-off inherent to filter media. World Minerals R&D has developed a systematic method to characterize various filter media types by retention of particles in a model system.

One of the first attempts at relating the particle retention properties of filter media is to examine the pore size distribution. This is frequently done with depth or static filter media, and was the subject of recent reviews. (5, 6) A popular analytical technique for pore size distribution is based on ASTM Method F 316, which makes use of a bubble-point test method.

The pore size distribution of Celite diatomite porous media can be measured using mercury porosimetry. This method provides the range of effective pore diameters from intraparticulate to interparticulate, but may not adequately quantify what size of turbidity will be retained by the filter media.

A method of measuring particle retention involves lab-scale constant rate filtration experiments with a model suspension system. The test filter used at World Minerals R&D is a single leaf test filter with 20 cm^2 of surface area. A filter paper septum is placed over a wire screen support. The model system used is a 0.5% (wt.) suspension of SAE Fine Dust (Powder Technology Inc., Burnsville, Minn.), which is a heterogeneous, highly dispersed non-deformable particles. The median particle size is 4.16 µm. Particle size analysis is by laser light scattering using a Honeywell Microtrac Xl 00 analyzer. The grade of filter aid being tested is applied as a 3 mm precoat on the filter paper septum. The SAE Fine Dust is suspended with an equal weight suspension of filter media and filtered.

The results are shown on Figure 3 for these three grades of Celite diatomite:

Grade Name	Type	Permeability (Darcy Units)	Median Particle Size (Micrometers)
Celite 577	Fully Calcined	0.2	14.6
Celite Hyflo Super-Cel	Flux Calcined	1.1	22.3
Celite 535	Flux Calcined	3.1	34.3

In addition, these filter aids were examined:

- Celite Fibra-Cel BH-200, a micronized α-cellulose filter aid, which has a permeability of 0.5 Darcy units.

- Harborlite 635 and Harborlite 700, two very fine grades of perlite, which have permeabilities of 0.5 and 0.6 Darcy units.

- Four Celpure grades: Celpure 65, Celpure 100, Celpure 300 and Celpure 1000 (where the number following the name refers to the typical permeability in milliDarcy units).

Figure 3 shows that the less permeable, or tighter, the filter media, the sharper the particle size cut off for a feed turbidity with a broad particle size distribution. As expected, the tightest filter media in this group, Celite 577, provides the greatest retention of turbidity. By taking 99% retention as the basis for the analysis, the particle size of feed turbidity which the filter media can remove has been determined. This is shown on Table 1 for selected Celite diatomite, Fibra-Cel® cellulose, and Harborlite perlite. The 99% particle size retention for Celpure diatomite filter aids is shown on Table 2. For the Celpure grades, the particle size retention for deformable turbidity is also shown. This is based on observations with biological turbidity. It confirms the experience that deformable turbidity can “bridge over” the filter aid particles, which reduces the permeability of the cake, but can tighten up its particle retention.

These results for rigid particle retention are plotted on Figure 4 for the Celite diatomite grade showing the expected trend and trade-off between permeability and particle size retention. The comparison with Celpure diatomite grades on an expanded scale is shown on Figure 5. Celpure has shifted the trade-off curve favorably to achieve greater turbidity removal without a sacrifice in permeability. In addition, Celpure has a greater capacity for turbidity particles and colloids.

CHEMICAL ANALYSIS — COMPARISON OF CELPURE TO ACID WASHED CELITE

Celpure grades are processed in such a way that the non-siliceous impurities are removed, resulting in a porous media with a very high silica content compared to acid washed and conventional diatomite grades. In addition, Celpure products have very low levels of extractable metals, such as iron and aluminum, and correspondingly low electrical conductivities (Table 3).

APPLICATIONS DATA – CLARIFICATION OF HIGH PURITY LIQUID WITH CELPURE

Removal of Activated Carbon from a High Purity Liquid

The performance of Celpure has been compared to acid washed Celite grades in the removal of activated carbon from a high purity liquid. This test case also illustrated the advantages of a gradient precoat application. Bodyfeed was not needed in this study. The case was conducted in a production, 20,000-L batch size using Celpure 65 and 1000 or Celpure 300 and 1000. The existing process uses acid washed Celite Standard Super-Cel and acid washed Celite Hyflo Super-Cel. The absence of soluble iron is very important to the stability of this product.

The gradient consisted of an initial precoat layer (3 mm) of Celpure 65 or 300, followed by a top layer of Celpure 1000. This approach is similar to practices in fixed depth filters of placing the larger pores on the top layer and the finer pores on the bottom layer of the filter. (7)

These results are given in Table 4 and show that Celpure not only has lower extractable iron, but also allows improvements in the filtration process: greater clarity, greater throughput and reduced usage.

CONCLUSIONS

Rigid particle studies have shown that conventional diatomite grades are capable of 99% retention of particulate turbidity below 0.5 μm. The retention of deformable biological turbidity is even finer, going into the colloidal particulate region. It is the intricate structure of diatomite, particularly the enhanced properties of Celpure that accounts for this submicron filtration ability. Celpure grades have improved the performance of conventional diatomite by achieving the same level of particulate retention with a 50% increase in permeability. Celpure is a major advance in porous media technology.

When designing screening studies, rigid particle cut-off curves are useful for narrowing down the grades to consider. An analysis and understanding of the particle size distribution and nature of the turbidity to be removed is still paramount to zeroing in on the porous media to choose. These techniques plus major product advances offer the filtration practitioner new tools to achieve the objectives of a solid-liquid separation process.

ACKNOWLEDGMENTS

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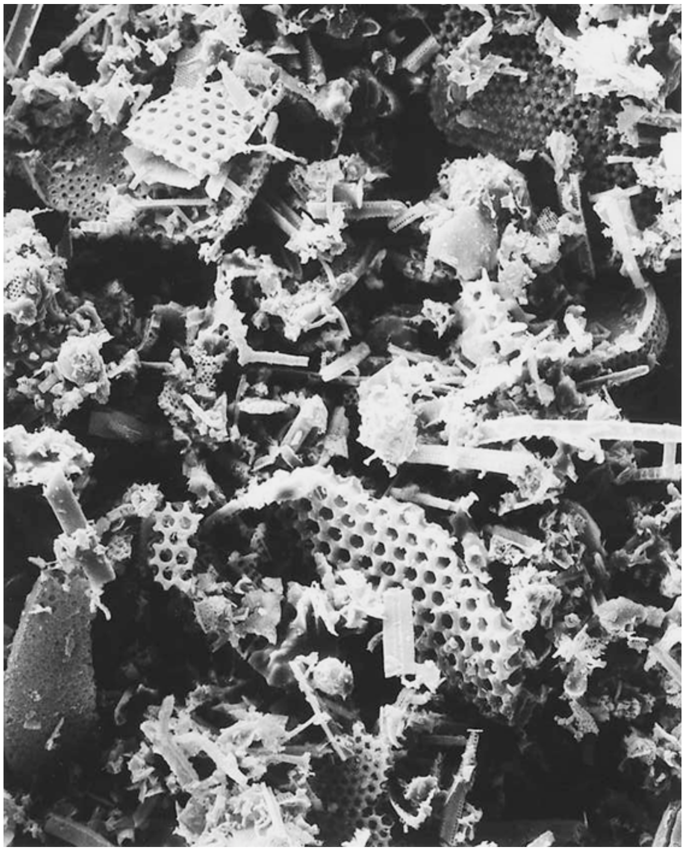
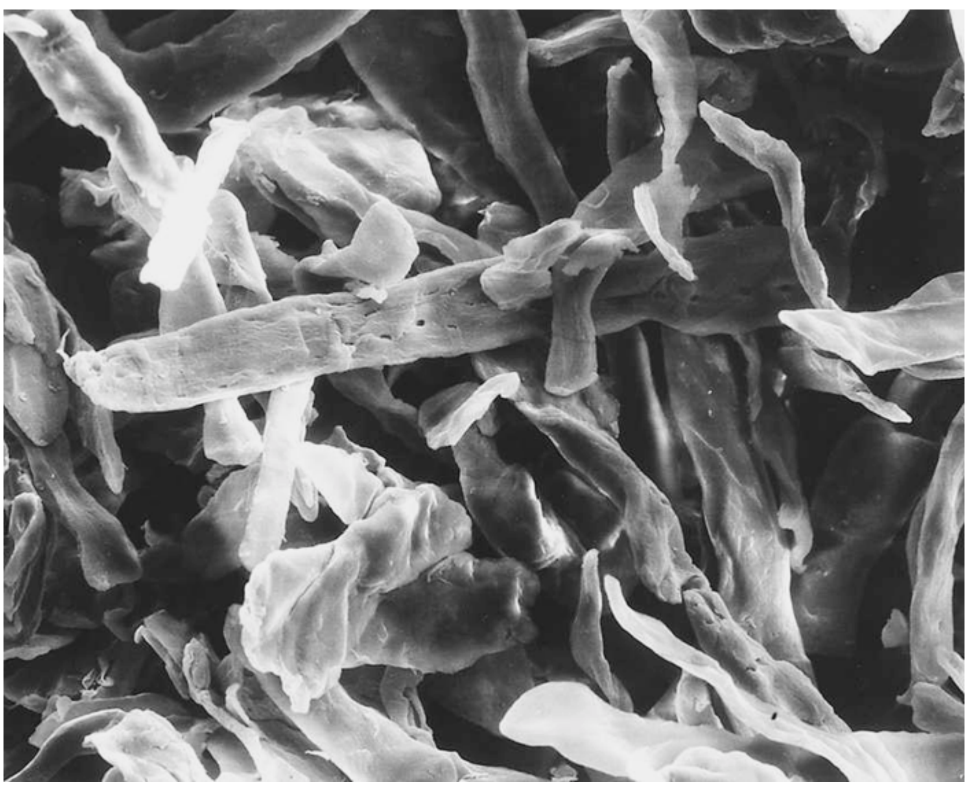


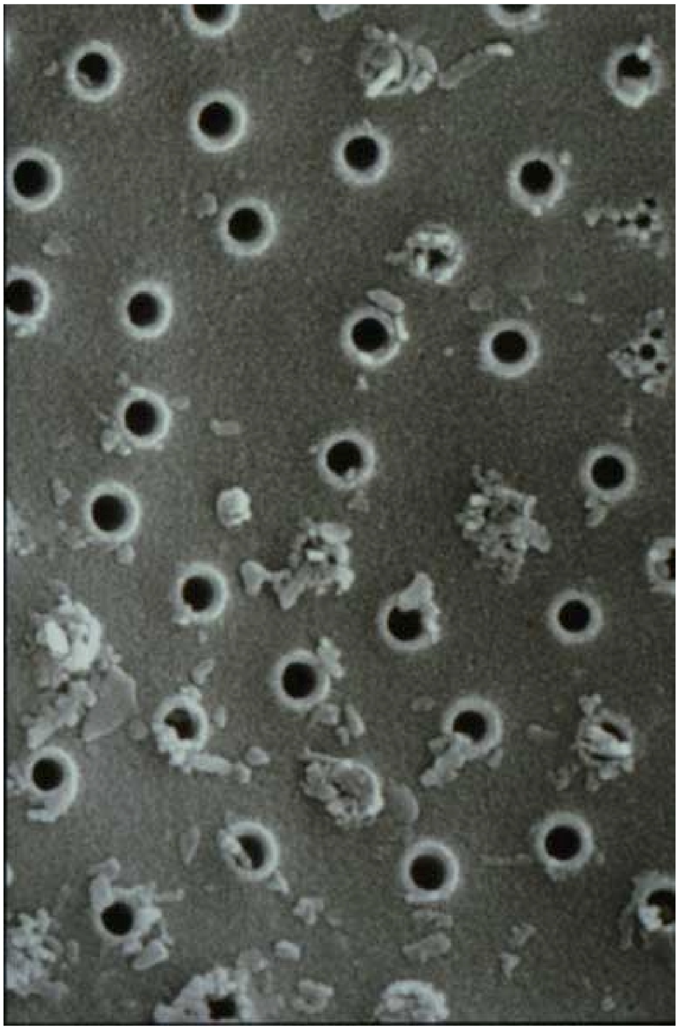
MARINE DIATOMITE

EXPANDED MILLED PERLITE

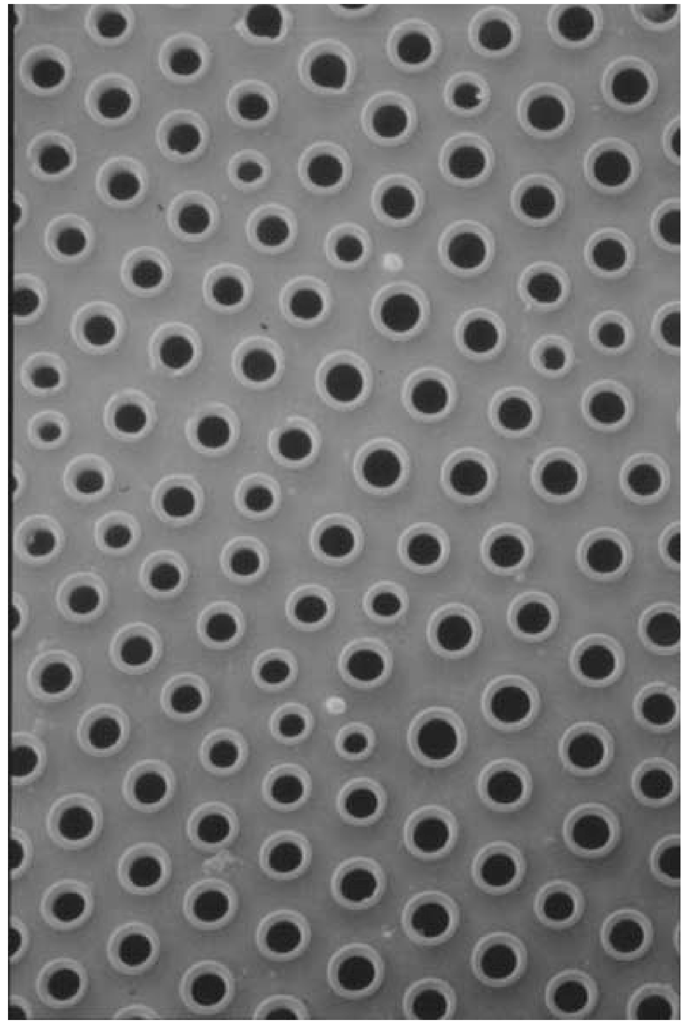


CELLULOSE

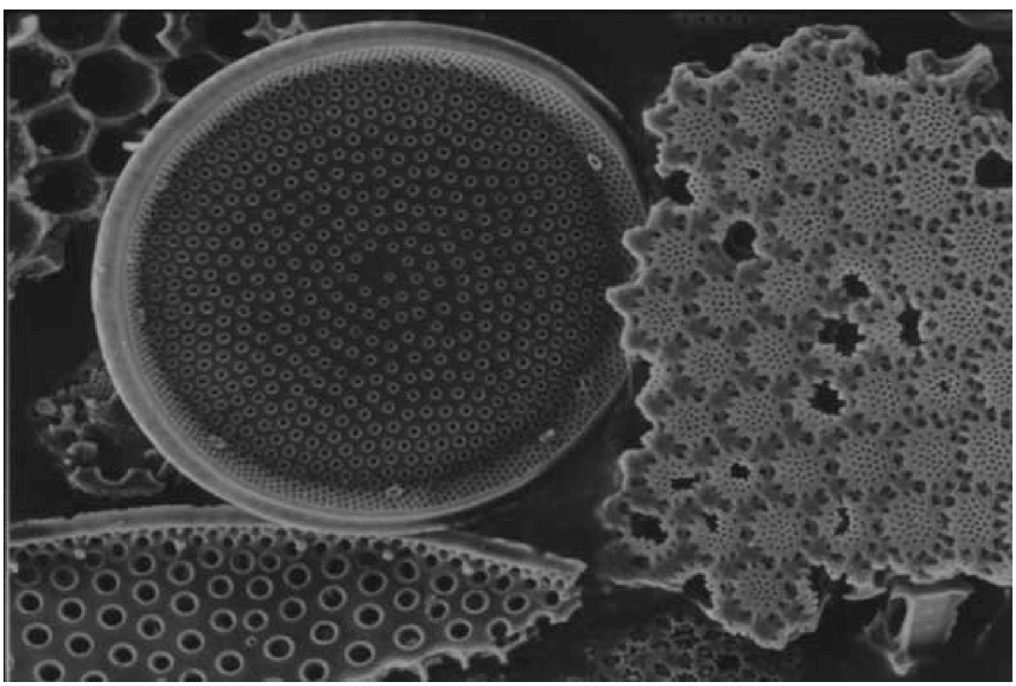
Figure 1. Scanning Electron Micrographs of Porous Media.



CELITE
SURFACE OF SINGLE DIATOM



CELPURE
SURFACE OF SINGLE DIATOM



CELPURE

Figure 2. Scanning Electron Micrographs of Conventional Diatomite and Celpure™

Table 1.

Particle Size Retention (99%) of Selected Celite® and Harborlite® Filter Aids

FILTER AID	SIZE RETAINED
CELITE CALCINED DIATOMITE GRADES:	
CELITE 577	1.0 µm
CELITE 521 (ACID WASHED)	1.2
CELITE STANDARD SUPER-CEL®	1.2
CELITE 512	1.2
CELITE FLUX CALCINED DIATOMITE GRADES:	
CELITE HYFLO SUPER-CEL (ACID WASHED)	1.6
CELITE HYFLO SUPER-CEL	2.0
CELITE 503	2.3
CELITE 535	3.3
CELITE CELLULOSE GRADE:	
FIBRA-CEL® BH-200	7.5
HARBORLITE PERLITE GRADES:	
HARBORLITE 635	8.5
HARBORLITE 700	6.0

Table 2.

Particle Size Retention Results for Celpure™ Grades

CELPURE FILTER AID GRADES

	Rigid	Deformable
CELPURE 65	<0.5 µm	0.3 µm
CELPURE 100	<0.5	0.4
CELPURE 300	0.75	0.6
CELPURE 1000	1.9	1.0

Table 3.

Typical Bulk and Extractable Chemical Properties of Celpure™ 300 Compared to Acid Washed Celite® 521

Bulk Chemistry by X-Ray Fluorescence

Component	**Celpure 300**	**Celite 521**
SiO_2	98.65%	91.62%
Al_2O_3	0.60	4.20
Fe_2O_3	0.27	1.39
$Na_2O + K_2O$	0.24	1.33
Soluble Aluminum (mg Al/kg), pH 4.5	<3	8
Soluble Iron (mg Fe/kg), pH 4.5	<3	5
Conductivity in H_2O (µS-cm)	<12.5	20

Table 4.

Application Results – Clarification of a High Purity Liquid

	Existing Process	Celpure 65/1000	Celpure 300/1000
Product Clarity (Optical Density)	8	3	7
Filter Aid Usage (Relative Amounts)	1	0.8	0.6
Volume Processed in 120 Minutes (L)	14,000	16,000	21,000
Soluble Iron, µg/L	8.4		5.6
Soluble Iron from Filter Aid, µg/L	2.8		0.0

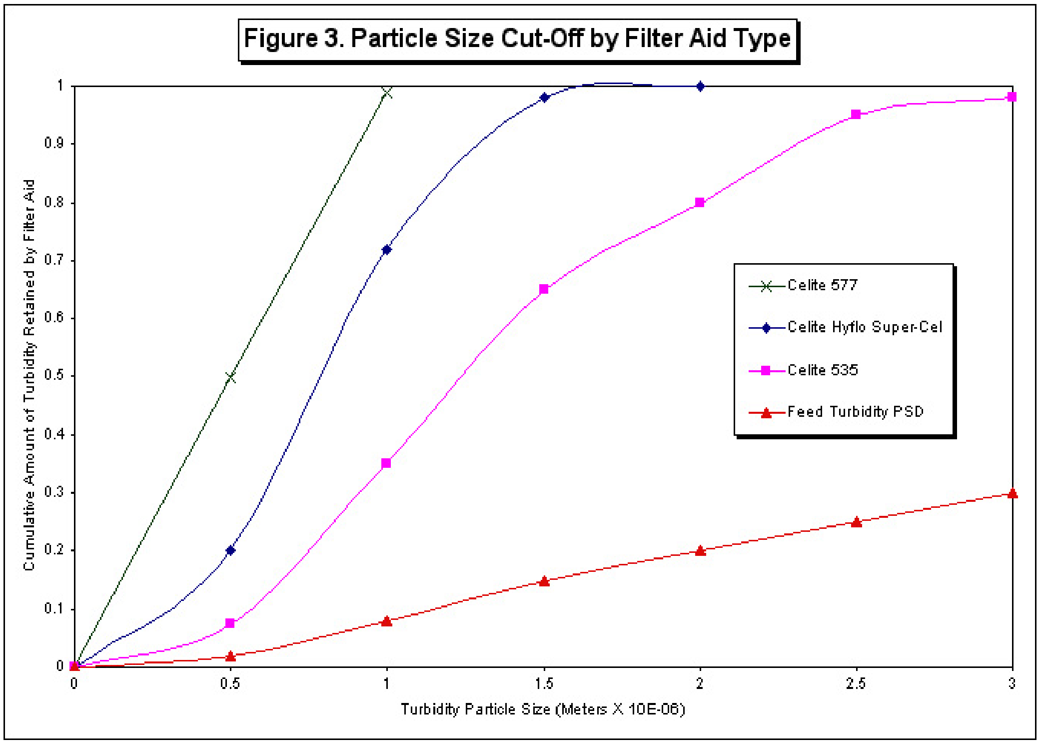


Figure 3. Particle Size Cut-Off by Filter Aid Type
Cumulative Amount of Turbidity Retained by Filter Aid
Turbidity Particle Size (Meters X 10E-06)
Celite 577
Celite Hyflo Super-Cel
Celite 535
Feed Turbidity PSD

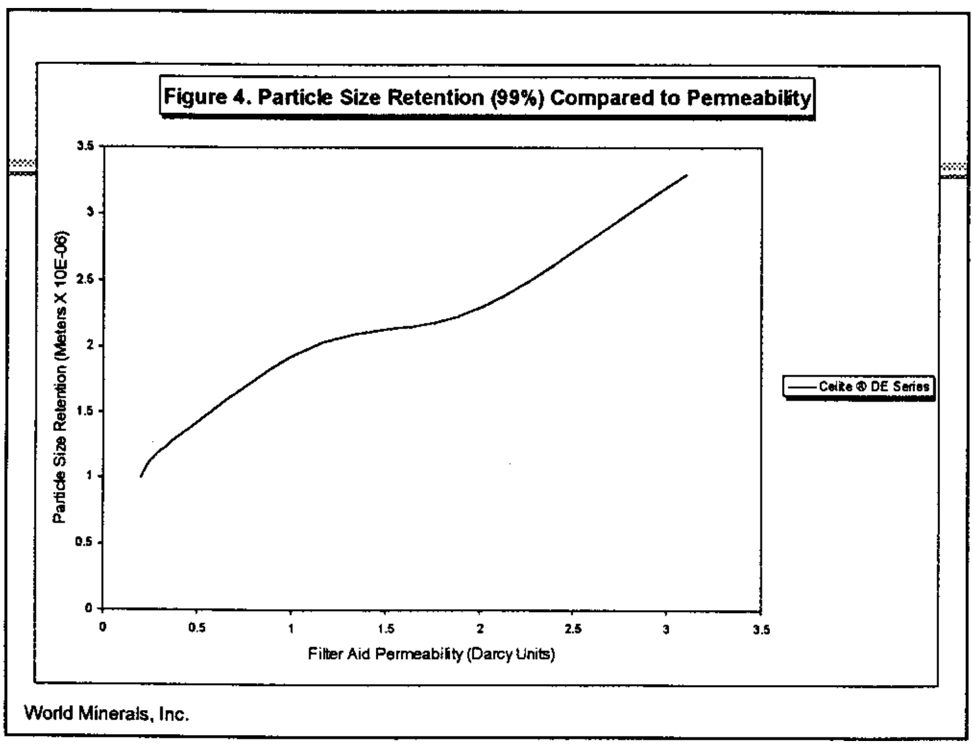


Figure 4. Particle Size Retention (99%) Compared to Permeability
Particle Size Retention (Meters X 10E-06)
Filter Aid Permeability (Darcy Units)
Celite ® DE Series
World Minerals, Inc.

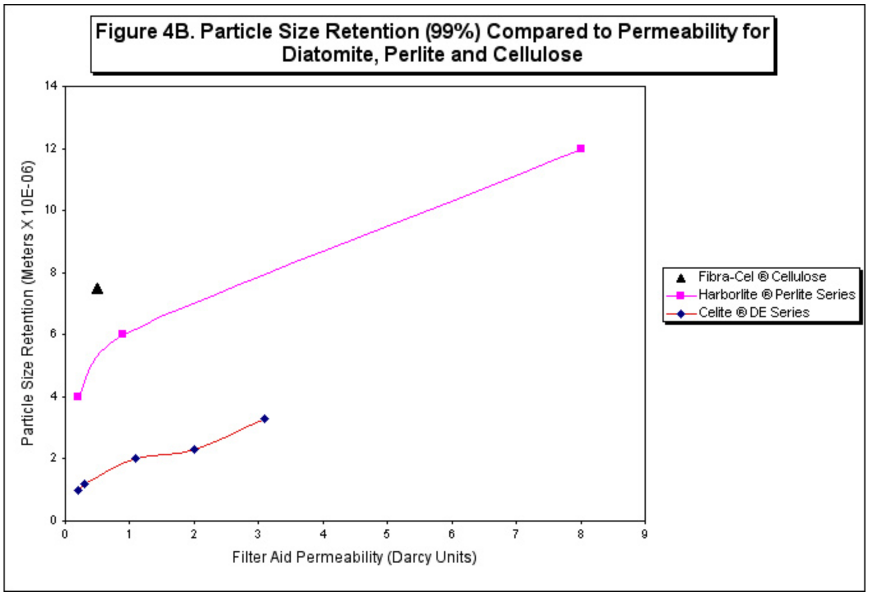


Figure 4B. Particle Size Retention (99%) Compared to Permeability for Diatomite, Perlite and Cellulose

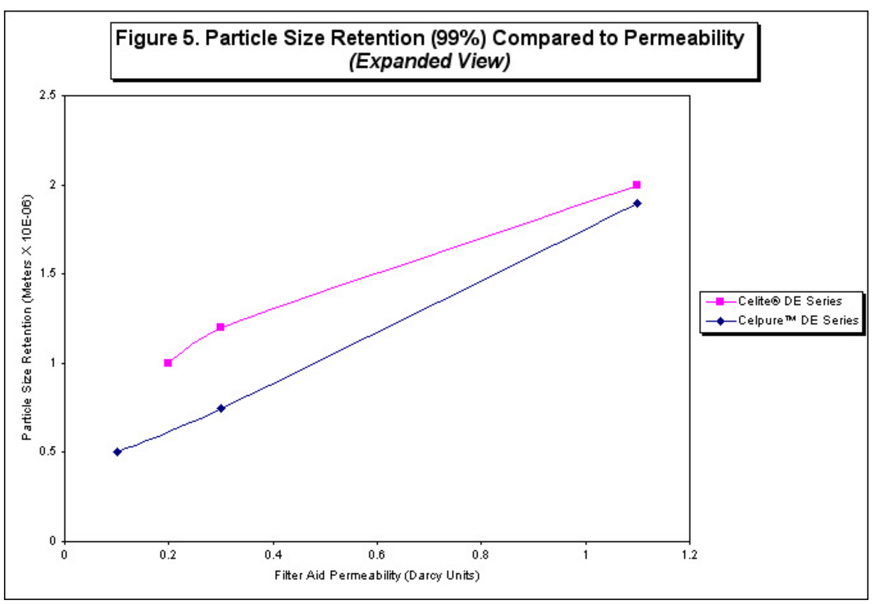


Figure 5. Particle Size Retention (99%) Compared to Permeability *(Expanded View)*