Development of Dewatering Aids for Minerals and Coal Fines

by

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The Final Report to United States Department of Energy
for Project Period During January 2001 to January 2004
DOE Award Number, DE-FC26-01NT41053

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Report Issued July 2004

DISCLAIMER

ABSTRACT

MCT has developed a suite of novel dewatering chemicals (or aids) that are designed to cause a decrease in the capillary pressures of the water trapped in a filter cake by i) decreasing the surface tension of water, ii) increasing the contact angles of the particles to be dewatered, and iii) causing the particles to coagulate, all at the same time. The decrease in capillary pressure in turn causes an increase in the rate filtration, an increase in throughput, and a decrease in pressure drop requirement for filtration. The reagents are used frequently as blends of different chemicals in order to bring about the changes in all of the process variables noted above.

The minerals and coal samples tested in the present work included copper sulfide, lead sulfide, zinc sulfide, kaolin clay, talc, and silica. The laboratory-scale test work included studies of reagent types, drying cycle times, cake thickness, slurry temperature, conditioning intensity and time, solid content, and reagent dosages. To better understand the mechanisms involved, fundamental studies were also conducted. These included the measurements of the contact angles of the particles to be dewatered (which are the measures of particle hydrophobicity) and the surface tensions of the filtrates produced from dewatering tests.

The results of the laboratory-scale filtration experiments showed that the use of the novel dewatering aids can reduce the moistures of the filter cake by 30 to 50% over what can be achieved using no dewatering aids. In many cases, such high levels of moisture reductions are sufficient to obviate the needs for thermal drying, which is costly and energy intensive. Furthermore, the use of the novel dewatering aids cause a substantial increase in the kinetics of dewatering, which in turn results in increased throughput. As a result of these technological advantages, the novel dewatering aids have been licensed to Nalco, which is one of the largest mining chemicals companies of the world. At least one mineral company is currently using the technology in full-scale plant operation, which has resulted in the shutdown of a thermal dryer.

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BACKGROUND

In the mining industry, run-of-the-mine (ROM) ores are crushed and pulverized to very fine sizes to liberate (detach) the valuable components from waste rocks. Although ROM coals are rarely pulverized to fine sizes on purpose, a significant amount of fines are produced naturally during shipping and handling. The mineral and coal fines are upgraded before utilization using appropriate methods. One of the most widely used methods of separation is froth flotation, in which air bubbles are introduced to the bottom of a tank containing an aqueous slurry of pulverized ore (or fine coal). The air bubbles selectively pick-up hydrophobic particles, leaving hydrophilic particles behind. The particles to be floated are hydrophobized using appropriate reagents known as *collectors*. For higher rank coals, which are naturally hydrophobic, no collectors may be necessary.

The mineral (or coal) concentrates obtained by flotation are dewatered before they can be further processed or shipped to consumers, while the tailings (or refuse) are discarded with or without extensive dewatering. The dewatering process consists of several steps. In the first step, a slurry is thickened to 35 to 75% solids in a large thickener, while free water is removed from the top and recycled back to the plant. In the second step, the thickened pulp is subjected to a *mechanical dewatering* process, such as filtration or centrifugation, to further remove the water. However, this process is highly inefficient, particularly when the mineral (or coal) particles are fine. In general, the moisture content in the dewatered product increases with decreasing particle size, which indicates that the residual moisture is mostly due to the surface water, i.e., the water molecules that are adhering to the surface. For sulfide mineral concentrates, the

filtered products contain typically 10 to 18% moisture by weight. For coal, the residual moistures are higher (20 to 30% by weight) due to its low specific gravity. Very often, these products need to be further dewatered in a third and the most costly step, i.e., *thermal drying*, which may be an option for high-priced materials. However, it is difficult to justify employing the costly thermal drying step for low-priced commodities such as coal. Even for the high-priced materials, eliminating thermal drying has significant economic and environmental advantages.

PROJECT OBJECTIVE

A committee representing a broad spectrum of the U.S. mining industry developed a document entitled, "The Mining Industry Roadmap for Crosscutting Technologies." It identified two highest R&D priorities in the area of Safe and Efficient Processing. These include:

I. new materials for phase separation in solvent extraction, and
II. improved separation and cleaning technologies, including dewatering,
III. water treatment, and by-product management and utilization.

The roadmap states that "Advances in mineral processing technology have leveled-off, making radical technological breakthroughs necessary for significant advances." It states also that availability of new advanced separation technologies will not only help minimize waste in the mining industry but can also be used for environmental remediation and waste utilization in general.

The objective of the proposed work is to further develop the novel dewatering aids (or chemicals) developed at Virginia Tech and Minerals and Coal Technologies,

Inc. The results obtained in bench-scale filtration experiments showed that the new reagents can reduce the surface moisture up to 50% for many mineral and coal concentrates, depending on the particle size, reagent dosage, conditioning, and other process variables. Commercial deployment of the new dewatering aids will obviate the needs for costly thermal drying, resulting in significant energy savings, waste minimization and increased profitability.

THEORY OF DEWATERING

THERMODYNAMICS

The process of dewatering may be represented by Figure 1 where a water droplet **2** is detached from the surface of a solid **1** and suspended in air **3**. The free energy change ($\Delta G/dA$) associated with this process is given by the following relationship:

$$\frac{dG}{dA} = \gamma_{13} + \gamma_{23} - \gamma_{12}, \qquad [1]$$

where γ_{13}= surface free energy at the solid/air interface

γ_{23} = surface free energy at the water/air interface

γ_{12} = surface free energy at solid/water interface, and

A = area of contact at the solid/water interface.

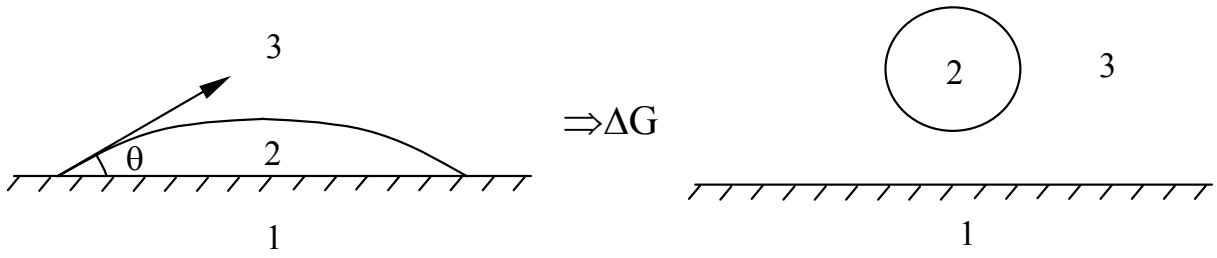

Figure 1 Schematic representation of dewatering; 1, solid, 2, water, 3, air; ΔG, free energy of dewatering.

The equilibrium between the three interfacial energies is given by Young's equation:

$$\gamma_{12} = \gamma_{23}\cos\theta + \gamma_{13}, \qquad [2]$$

where θ is the contact angle.

Substituting Eq. [2] into Eq. [1], one obtains the following relationship:

$$\frac{dG_{dis}}{dA} = \gamma_{23}(1+\cos\theta), \qquad [3]$$

which suggests that dewatering becomes spontaneous, i.e., $dG_{dis}/dA<0$, when $\theta>180^o$. Thus, if a solid can be hydrophobized to the extent that its contact angle exceeds 180^o, dewatering can become spontaneous. However, it is a very difficult task to achieve in reality. Therefore, it is necessary to do work on the system to dewater the solid. The work of dewatering, then becomes:

$$W_d = -\gamma_{23}(1+\cos\theta), \qquad [4]$$

which is negative when $\theta<180^o$, or work must be done on the system. However, one can minimize the work required for dewatering by reducing γ_{23} and by increasing θ.

KINETICS

a) Rate Equation

Mechanical dewatering is described as a process in which water flows through porous media created by a bed of particles. Darcy (1856) was the first to derive the rate equation as follows:

$$\frac{dV}{dt} = K\frac{A}{\eta}\frac{\Delta P}{L}, \qquad [5]$$

in which V is the volume of fluid, t the time, ΔP the pressure drop across the bed, L the bed (cake) thickness, A the cross-sectional area of the cake, η the viscosity of water,

and K is the rate constant known as permeability. Eq. [5] suggests that the rate of dewatering is proportional to the pressure gradient and the cross-sectional area, and is inversely proportional to the viscosity and the cake thickness.

The process of filtration is often related to the flow of liquid through a bundle of capillary tubes. In this case, one can use the Poiseulle's equation (1846):

$$\frac{dV}{dt} = \frac{\pi r^4}{8\eta}\frac{\Delta P}{L} \qquad [6]$$

where r is the radius of the capillary.

By combining Eqs. [5] and [6], Kozney (1927) obtain the following relationship:

$$\frac{dV}{dt} = \frac{A\varepsilon^3}{kS(1-\varepsilon)^2}\frac{\Delta P}{L} \qquad [7]$$

where ε is the cake porosity, which is defined as the volume fraction of the void space in the filter cake, S the specific surface area of the particles per unit volume, and k is a constant, which is referred to as Kozney constant. Theoretically, k should be 2 for an ideal filter cake that is a porous medium made of capillary tubes of radius r. In experiment, k is found to be approximately 5 for filter cakes made of simple monodisperse solids (Carman, 1937). For many industrial filter cakes formed in the presence of flocculants, k is often greater than 5 and can be as large as several thousands (Gray, 1958).

From Eqs. [5] and [7], one can obtain the following relationship:

$$K = \frac{\varepsilon^3}{kS^2(1-\varepsilon)^2} = 1/\alpha \qquad [8]$$

where α is referred to as specific cake resistance. Eq. [8] suggests that cake

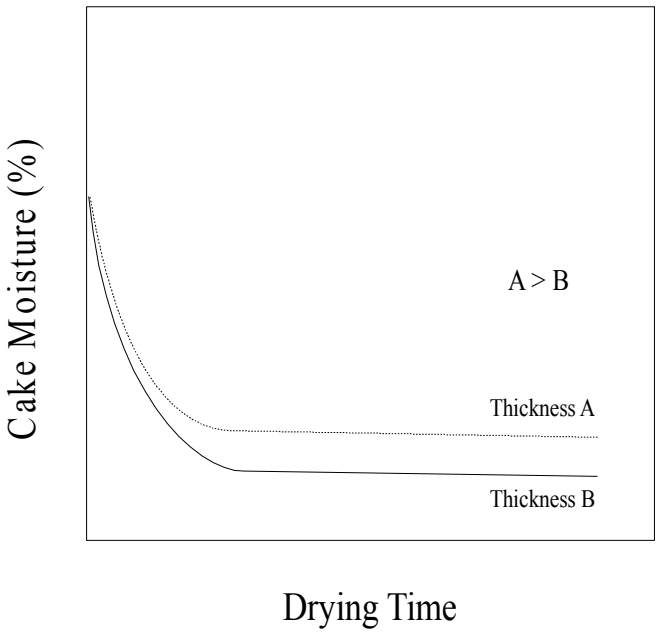

Figure 2 Typical results of dewatering tests, which show that cake moisture decreases rapidly at the beginning and then tapers off with increasing drying cycle time. The latter is due to the difficulty in removing the moisture from the capillaries of smaller radii. The situation gets worse with increasing cake thickness.

permeability decreases with decreasing ε and increasing S, while ε *decreases and* S *increases* with decreasing particle size. Thus, the rate equation derived based on Darcy's law (Eq. [5]) provides an explanation for the difficulty in dewatering fine coal.

Eq. [5] suggests that the flow of filtrate versus time should be linear. However, practically all filtration experiments show parabolic behavior. Figure 2 shows the results of typical batch filtration experiments. The main reason for the discrepancy is that the rate equation based on Darcy's law disregards distribution of pore sizes, *i.e.*, there exists a range of pore sizes (and also shapes) in a porous bed (Gray, 1958). According to the Laplace equation (the implications of which will be discussed in the following section), the water in larger pores will be more easily removed than the water in smaller pores. Thus, the larger pores should determine the initial dewatering rate, while the finer

pores determine the water retention (final cake moisture). Therefore, one can either remove the finer pores by removing the ultrafine particles from a feed or develop a method of removing the water trapped in finer capillaries.

b) Laplace Equation

In order to remove the water from a capillary of radius *r*, it is necessary that the applied pressure (ΔP of Eq. [6] and [7]) be larger than the capillary pressure, *p*:

$$p = \frac{2\gamma_{23}\cos\theta}{r} \qquad [9]$$

which is known as Laplace equation. Here, γ_{23} is the surface tension of water, and θ the water contact angle. One can see that *p* decreases with decreasing γ_{23}, increasing θ, and increasing *r*.

Various chemicals (dewatering aids) are added to coal slurry to control these parameters so that *p* can be reduced. Some of the dewatering aids that are used in industry today are designed to reduce γ_{23} (surface tension of water). Sodium laurylsulfate and sodium dioctylsulfo-succinate are typical examples. Consider a case of filtering fine coal slurry at a vacuum pressure of 22-inches Hg (0.735 atm or 0.745×10^5 Pa). Assume that $\theta = 45^o$ for the coal, which is typical of many high-volatile bituminous coals mined in the U.S., and that no dewatering aids are used, i.e., γ_{23}=72 mN/m. Substituting these numbers into Eq. [9] and solving it for *r*, one obtains *r*=1.4 μm. The water in the capillaries of smaller radius will have pressures higher than the vacuum pressure and, therefore, cannot be removed. When γ_{23} is reduced to 40 mN/m by reagent addition, however, the critical capillary radius at which water can be reduced by vacuum suction is reduced to 0.76 μm.

Various flocculants are used as dewatering aids. Some are organic, e.g., polyacrylamide, while others are inorganic, e.g., alum. The role of these reagents is to increase the effective size of the particles in the filter cake, so that the pore radius, *r,* can be increased. This will greatly decrease the capillary pressure *p* relative to applied vacuum pressure (ΔP) and, hence, increase filtration rate. However, most of the flocculants cause θ to decrease and at the same time create micro-pores within large flocs, both of which contributing to increased final cake moisture. The work done at CONSOL Energy actually showed that flocculants increase the moisture of filter cake (Meenan, 1988).

TECHNICAL APPROACH

The difficulty in removing water from the surface of fine particles may be attributed to the fact that water molecules are held strongly to the surface *via* hydrogen bonding. One can break the bonds and remove the water by subjecting the wet particles to intense heat, high-pressure filters, or high-G centrifuges. However, the use of such brute forces entails high energy costs, maintenance problems, and environmental concerns. A better solution would be to destabilize the surface water by appropriate surface chemical treatment, so that it can be more readily removed by the weaker forces imparted by vacuum, low-pressure filters.

The state of the water adhering to a surface may be best represented by the hydrophobicity (water-hating property). The stronger the hydrophobicity, the weaker the bonds between the water and the surface would become. Therefore, the key to finding appropriate chemical means to destabilize surface water is to increase the

hydrophobicity of the particles to be dewatered. A more traditional measure of surface hydrophobicity is water contact angle (θ of Eq. [9]). In the sessile drop technique, a water droplet is placed on a flat surface of the solid of interest, and the angle at the three-phase contact is measured through the water phase. In general, contact angle increases with increasing hydrophobicity of the surface.

As discussed in the foregoing section, the dewatering aids that are used in industry today can reduce the surface tension (γ_{23}) of water or increase capillary radius (r), but tend to render the surface hydrophilic. Therefore, the approach taken in the present work was to develop dewatering aids that can decrease γ_{23}, increase r, and at the same time increase contact angle θ. The net results of formulating ideal dewatering aids that can meet all of the criteria of the Laplace equation (Eq. [9]) would be a substantial increase in dewatering rate and decrease in final cake moisture.

EXPERIMENTAL

FILTRATION

Most of the dewatering tests were conducted using Buchner funnel-type vacuum filters, and Figure 3 shows the experimental setup. The Buchner filter was mounted on Vacuum Flask 1 (2 liters), which was connected to Vacuum Flask 2 (4 liters) by means of a valve. The second vacuum flask was connected to a vacuum pump (3/4 HP, Cole-Parmer, 220V). The vacuum pressure was controlled by means of a control valve, and monitored by a vacuum gauge.

Majority of the tests were carried our using a 2.5-inch diameter Buchner funnel with a fabric filter, which was purchased from Peterson Filter Company, Salt Lake City,

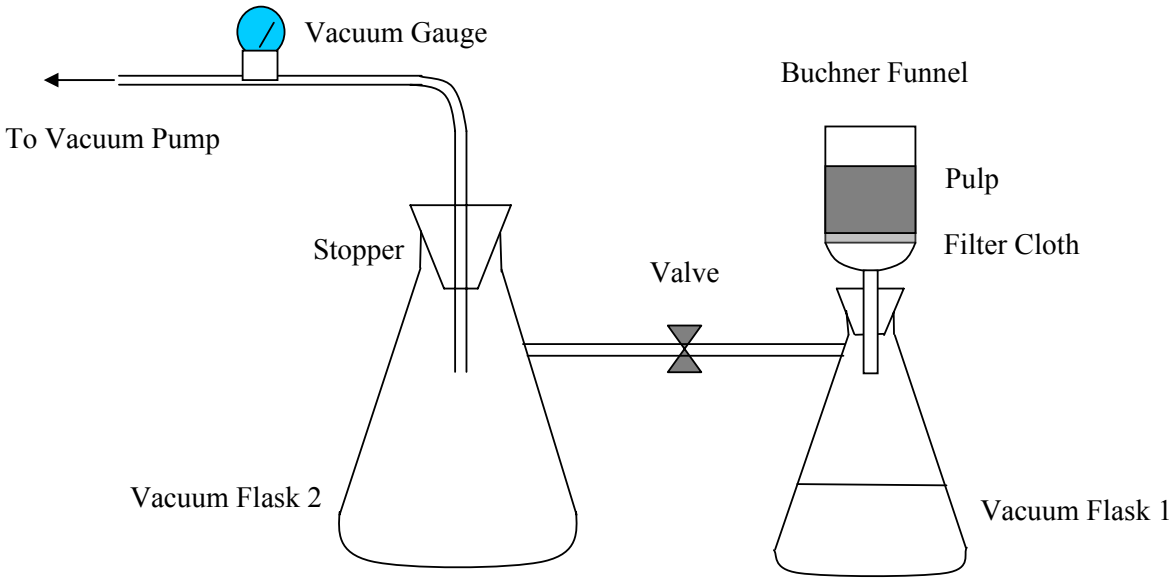

Figure 3 Experimental setup for laboratory vacuum filtration tests.

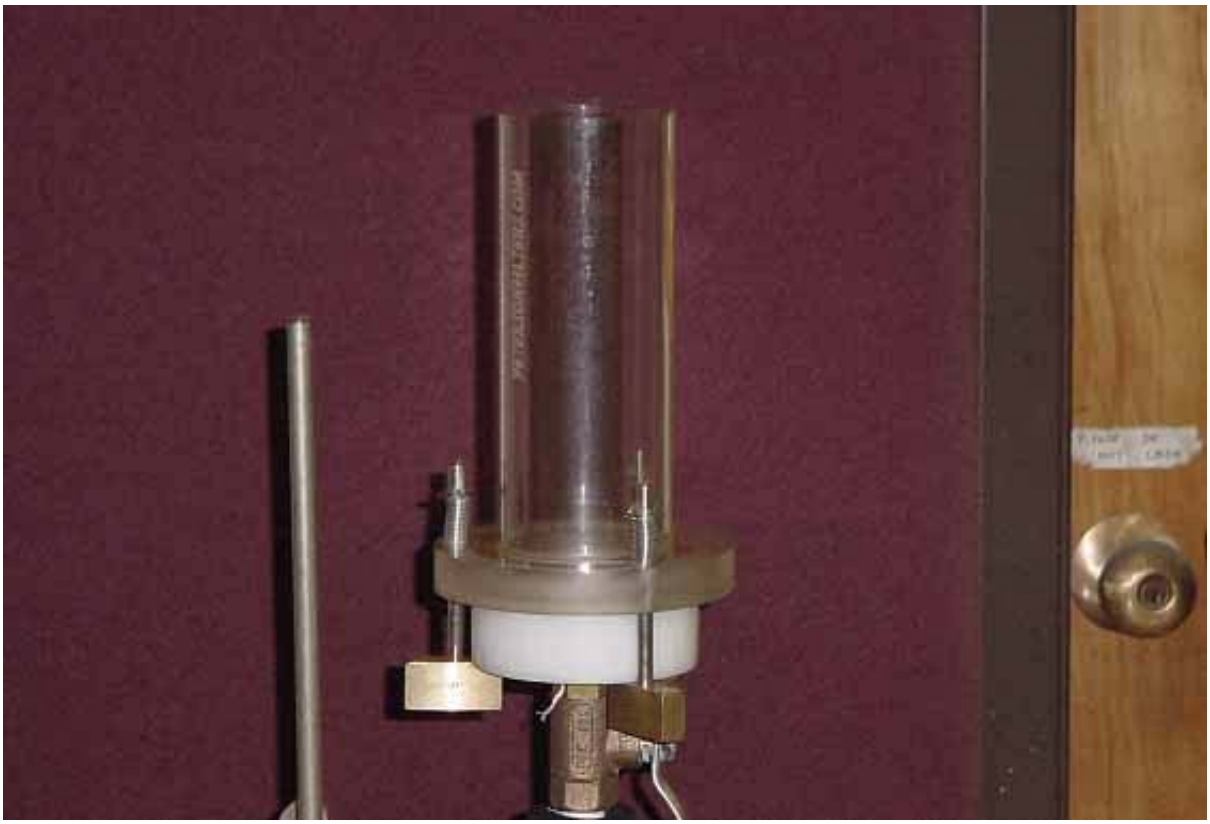

Figure 4 Peterson Buchner filter.

Utah. However, some of the tests were conducted using a 2.5-inch diameter Buchner funnel with a medium porosity glass frit. The Peterson vacuum filter, which is shown in Figure 4, was specially made for MCT. It was constructed such that the filter cake can be easily removed after each experiment. The height of the filter chamber was 5 inches, which made it possible to increase the volume of the slurry to obtain thicker cake if necessary.

Vacuum filtration is limited to a maximum pressure drop (ΔP) of 1 atm (1.01 bar, 29.9 inch Hg, 101.3 KPa). According to Darcy's equation, dewatering rate should increase at higher pressures. Therefore, filtration tests were also conducted using a pressure filter, which is shown schematically in Figure 5.

In each dewatering test, a mineral or coal slurry was agitated in a container by means of mixer to obtain a homogenous slurry. A desired volume of the slurry was then

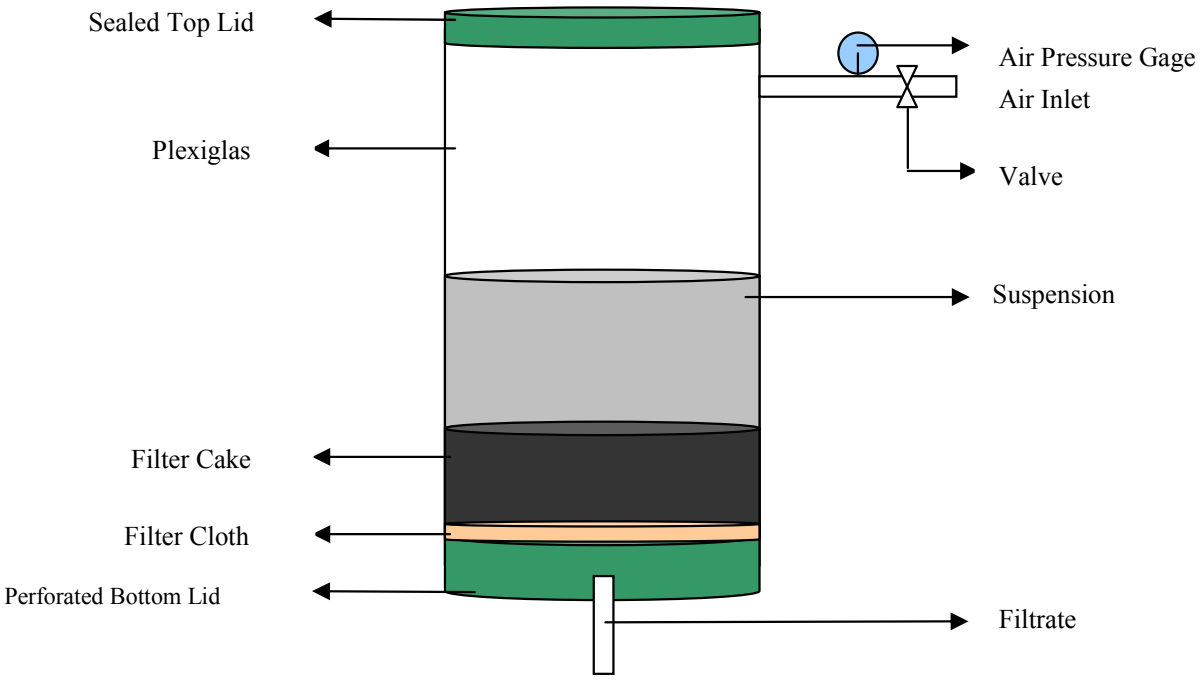

Figure 5 Schematics of the pressure filter.

Figure 6 Pressure filter

removed from the homogenized slurry by means of a measuring cup, and poured into an Erlenmeyer flask. A measured amount of a dewatering aid was then added into the flask by means of a Microliter syringe, and the flask mounted on a mechanical wrist shaker. The mixture of the slurry and the dewatering aid was agitated for a predetermined period of time to ensure adequate contact (or reaction) between the dewatering aid and the surface of the particles in the slurry. This step of the experimental procedure is referred to as “conditioning”. The conditioned slurry was poured into the vacuum or pressure filter. A vacuum or air pressure was applied to initiate filtration experiment. During filtration, cake formation time, initial pressure drop (ΔP), the pressure drop during drainage period, and drying cycle time were recorded. The cake formation time is the time required to remove all visible free water from the

surface of the filter cake and is indicative of dewatering kinetics. The drying cycle is the allotment of time after cake formation in which the pressure difference through the cake was continued for further moisture reduction.

After the filtration, the filter cake was removed from the filter and the mass of wet filter cake was determined. The wet cake was then placed in a conventional oven at 105°C to dry overnight. The weight of the dried filter cake was determined. The moisture content of the filter cake was calculated from the difference between the weights of the wet and dry filter cakes.

CONTACT ANGLE MEASUREMENT

The water contact angles of the coal samples (chunk) were measured using two methods, (1) the sessile drop technique with a Rame-Hart Model 100 Goniometer, (2) the Wilhelmy plate technique with a Sigma-70 Surface Tensio-Meter. For powdered samples, the thin-layer wicking technique was used to measure the contact angles. It should be noted here that the thin-layer wicking technique gives advancing angles as apposed to the equilibrium contact angles obtained using the sessile drop technique. For the sessile drop and Wilhelmy plate techniques, coal samples were polished using different sized sand papers (120, 240, 400 and 600 grits). Final polishing was made using a polishing cloth with a 0.05 μm alumina power. To minimize experimental errors, a small syringe was used to produce sessile drops. For a given coal sample, at least 5 measurements were made and the results were averaged.

For the thin-layer wicking technique, an aqueous suspensions of coal powders (5% solids) was prepared in distilled water. The slurry was agitated by means of a magnetic stirrer in a 250 ml beaker to obtain a homogenous suspension. Approximately

3 ml of the suspension was withdrawn using a plastic syringe and sprayed over a 2.5x7.5 mm glass slide. After the evaporation of water at room temperature (usually one day later), the glass slides coated with powders were dried in an oven at 105°C to remove the residual water. The slides coated with dry powders were stored in a desiccator to prevent the dry samples from adsorbing moisture from air (van Oss, 1992).

A glass slide coated with a dry powder was placed in a beaker containing an organic liquid. The liquid rises along the slide *via* capillary force. The rise velocity was then measured. The capillary rise velocities were measured in several different liquids of known properties (e.g., hexane, heptane, octane and decane). One can determine the mean pore radius *r** and the water contact angle value θ of the fine powders using the Washburn equation (van Oss, 1992):

$$l^2 = \frac{\gamma_{lv} r^* t \cos\theta}{2\eta} \qquad [10]$$

where *l* is the distance traveled by the liquid on the coated slides, *t* is the travel time, γ_{lv} is the liquid surface tension and η is the liquid viscosity.

The samples used for the contact angle measurements were obtained from Elkview Mine in British Columbia, Canada; Middle Fork Coal Preparation Plant, Virginia; Massy Coal Company, West Virginia; and the Pittsburgh Seam coal from Pennsylvania. For each sample, a lump of coal was cut with a diamond saw and polished in the manner described above.

In each contact angle measurement, a polished coal specimen was placed in a beaker containing a measured amount of a screened fine coal (-1.7+0.3 mm). A

selected dewatering aid was then added and agitated for 2 minutes to allow for the reagents to adsorb on the coal surface. The powdered coal was added so that it would be possible to compare the results of the contact angle measurements with the dewatering tests, in which dewatering aids were added in units of lb/ton. The dewatering aids used in the contact angle measurements were Reagent 01DW145, 01DW111, ROE1, and RE1. The reagents were used as 33.3% solution in diesel. After the two minutes of conditioning, the polished coal samples were taken out of the solution and washed with nanopure water before blow-dried in a nitrogen gas stream.

RESULTS AND DISSCUSSION

TASK 1: SURFACE TENSION AND CONTACT ANGLE CONTROL

Subtask 1.1 Developing Novel Dewatering Aids for Mineral Concentrate

The dewatering aids tested in the present work were designed to increase the hydrophobicity (or contact angle θ) of naturally hydrophobic materials such as coal and, hence, decrease the capillary pressure of the water trapped in a filter cake (see Eq. [9]). It was found, however, that the same dewatering aids can also be used for hydrophilic minerals, provided that the minerals are hydrophobized by means of suitable surfactants (or collectors) prior to contacting the dewatering aids.

In this subtask, laboratory-scale dewatering tests were conducted on a range of hydrophilic minerals, which included sphalerite, chalcopyrite, galena, talc, silica (quartz) and kaolin clay. In a given experiment, a hydrophilic mineral is conditioned first with a collector for a given period of time and then conditioned again with a dewatering aid. The collector increases the contact angle from zero to approximately 30 to 60^{o}, which are further increased close to 90^{o} by the dewatering aid. The results obtained in Subtask 1.1 show that the two-stage conditioning technique can improve moisture reduction by 30 to 60% over the case of not using any dewatering aid.

a) Sphalerite

A zinc (sphalerite) concentrate was received from the Garpenburg Mine of Boliden Minerals AB, Sweden. The flotation product was subjected to a series of dewatering tests by following the procedures detailed previously in the Experimental section. However, it was determined that the sample had been superficially oxidized during transportation and storage. Approximately one month of time elapsed since the

sample was taken at the mine. When a sulfide mineral is oxidized, it becomes hydrophilic due to the formation of sulfoxy compounds such as sulfate (SO_4^{2-}) and thiosulfate ($S_2O_3^{2-}$), which readily form hydrogen bonds with water.

To regenerate fresh mineral surfaces, the zinc concentrate sample was wet-ground in a ball mill for 1.5 minutes, and then re-floated using 50-g/ton potassium amyl xanthate (KAX) and 50-g/ton methyl isobutyl carbinol (MIBC) at pH 9.2. The pH was adjusted by lime (CaO). It is believed that the xanthate ions (X^-) displace the hydrophilic sulfoxy compounds and render the surface hydrophobic. The re-floated sample was subjected to filtration tests using a 2.5-inch diameter Buchner filter at 25.2% solids at a 25 inches Hg vacuum pressure. Two minute drying cycle time was employed in each test.

Reagent RA1 was tested as a dewatering aid for the Boliden zinc concentrate. The reagent was used as a 33.3% solution (by volume) in diesel (1 part Reagent RA1 and 2 part diesel by volume). Diesel is used as a carrier solvent that can facilitate the

Table 1 Effects of Using Reagent RA1 for the Dewatering of Garpenburg Zinc Concentrate at 25-inches Hg Vacuum Pressure*

Reagent Dosages (lb/ton)	Moisture Content (%)		
	Cake Thickness (in)		
	0.2	0.4	0.6
0	12.9	14.1	14.7
1	6.1	8.3	9.5
2	5.5	7.2	8.4
3	5.1	7.1	8.3
5	5.0	6.8	8.1

*Test performed with a 2.5 inch diameter Buchner filter; refloated with 50 g/ton KAX and 50 g/ton MIBC; particle size, 0.105 mm x 0; solid content 25.2%; 2 minutes of drying cycle time

adsorption of the dewatering aid on the surface. For each dewatering test, a dosage of reagent was added to the sample slurry and conditioned for 2 minutes before filtering. The test results obtained by varying reagent dosages and cake thickness are given in Table 1. The thickness of the filter cakes was adjusted by varying the volume of slurry placed into the Buchner filter. Reagent dosages shown in the table refer to the active ingredient dosage only. As represented in Table 1, when cake thickness is increased from 0.2 to 0.6 inches, moisture content of the filter cake also increases. This is expected from Darcy's equation (Eq. [5]), which shows that dewatering rate decreased with increasing cake thickness, L.

Nevertheless, the use of the novel dewatering aid substantially increased the moisture reduction at all cake thicknesses tested. At 0.2 inch cake thickness, cake moisture was reduced from 12.9% to 6.1% at 1 lb/ton RA1, which represented a 53% reduction from the control test. Further increase in reagent dosage did not increase the moisture reduction substantially. The moisture reductions at 1 lb/ton were 41.1 and 35.4% at 0.4- and 0.6-inches of cake thicknesses, respectively.

Another set of dewatering tests was conducted using 01DW145 on the Garpenburg zinc concentrate. The reagent was used as a 1:2 mixture with diesel. In each test, the reagent blend was added to the sample slurry contained in an Erlenmeyer flask and conditioned for 2 minutes. The conditioned slurry was then poured into a 2.5-inch diameter Buchner filter, and a vacuum pressure of 25 inches Hg was applied. After a filter cake was formed, the vacuum pressure was applied for another 2 minutes, which is referred as drying cycle time. In this series of tests, the thickness of the filter cake was controlled by changing the volume of the slurry used in filtration tests.

Table 2 Effects of Using 01DW145 for Dewatering Garpenburg Zinc Concentrate at 25 Inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	12.5	13.7	15.0
1	5.4	6.0	8.3
2	4.7	5.4	7.4
3	4.6	5.1	6.9
5	4.3	5.0	6.6

*Refloated using 50 g/ton KAX and 50 g/ton MIBC; partilcle size 0.105 mm x 0; solid content 25.2%; drying cycle time 2 minutes.

Table 2 shows the results obtained using 01DW145 on the Garpenburg zinc concentrate. With regent blend, greater than 50% moisture reductions were obtained even at 0.6 inch cake thickness. For example, the moisture was reduced from 15% to 7.4% at 2 lb/ton, which represented 50.7% reduction. Based on these excellent results, this reagent has been commercialized. The results of the full-scale test work will be presented later in this report.

Table 3 Effects of Using Reagent RE1 in Dewatering Garpenburg Zinc Concentrate at 25 Inches Hg

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	12.2	13.0	14.0
1	8.1	8.7	9.4
2	7.9	8.1	8.5
3	7.0	7.6	8.9
5	6.4	7.8	8.6

*Reflotaed with 50 g/ton KAX and 50 g/ton MIBC; particle size 0.105 mm x 0; solid content 25.2%; drying cycle time 2 minutes.

Table 3 shows the results obtained using Reagent RE1 on the same zinc concentrate. The reagent was used as a 1:2 blend with diesel. The results show that the cake moistures obtained using Reagent RE1 gave 1 to 2% higher moisture contents than those obtained with 01DW145. It appeared that Reagent RE1 created flocs, in which moisture was trapped.

In another set of dewatering tests, Reagent RG1 was used as a dewatering aid for the Garpenburg zinc concentrate. One part of RG1 was mixed with two parts of diesel. The filtration test was conducted on the re-floated zinc concentrate using a 2.5-inch diameter Buchner filter at 25 inches Hg and 2 minutes of drying cycle time. The test results given in Table 4 showed that at 0.4 inch cake thickness, cake moisture was reduced from 12.3 to 8.3% at 2-lb/ton RG1, which represents a 33% moisture reduction.

The improved dewatering results obtained with the Garpenburg zinc concentrate may attributed to the changes in particle hydrophobicity, which in turn caused hydrophobic coagulation of fine particles. Thus, the increase in hydrophobicity controls

Table 4 Effects of Cake Thickness on The Dewatering* of Boliden-Zinc Concentrate Samples Using RG1 at 25 Inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	11.1	12.3	14.1
1	7.5	7.9	10.0
2	7.4	8.3	9.1
3	7.5	7.8	8.5
5	7.0	8.7	8.9

* 2.5 in diameter vacuum filter used; the company sample treated using 50 g/ton KAX and 50 g/ton MIBC; size 0.105 mm x 0; solid content 25.2%; drying cycle time 2 minutes.

two of the three variables of the Laplace equation, i.e., θ and r (capillary radius). The reagent blends tested in the present work also reduces surface tension, γ. Therefore, the dewatering reagents tested in the present work change all three variables of the Laplace equation toward the right direction, which is the reason that the dewatering results shown in this section are superior to the dewatering aids used in industry today. As has already been noted, some of the dewatering aids tested in this subtask have been commercialized.

b) Chalcopyrite

A chalcopyrite ($CuFeS_2$) concentrate was received from the Aitik Mine, Boliden AB, Sweden. Its particle size was 0.150 mm x 0. It was determined that the sample had become superficially oxidized during transportation and storage; therefore, the sample was wet-ground in a ball mill for a very short period of time (1.5 minutes) as a means of regenerating unoxidized and fresh surface. The wet-ground sample was subsequently floated using 50 g/ton KAX and 50 g/ton MIBC. The pH of the flotation pulp was adjusted to 10.5 with lime (CaO). The flotation product was subjected to dewatering

Table 5 Effects of Using Reagent RA1 for Dewatering the Aitik Copper Concentrate at 25 Inches Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	10.2	10.9	12.2
1	5.1	6.5	7.2
2	4.3	6.3	6.6
3	4.1	6.4	6.3
5	3.7	5.8	5.9

* 2.5 in diameter vacuum filter used; the company sample treated using 50 g/ton KAX and 50 g/ton MIBC; size 0.150 mm x 0; solid content 22.3%; drying cycle time 2 minutes.

tests as detailed previously in the Experimental section.

A series of filtration tests were conducted on the wet-ground and re-floated sample. The pulp density of the froth product was 22.3% solids. The filtration tests were conducted using a 2.5-inch diameter Buchner filter at 25 inches Hg vacuum pressure. Two-minutes of drying cycle time was employed for each test.

Reagent RA1 was chosen as a dewatering aid, which was used as a 1:2 mixture with diesel oil. The test results obtained with varying reagent dosages and cake thicknesses are given in Table 5. At 2 lb/ton, the moisture reduction was 46% at 0.6 inches of cake thickness and 58% at 0.2 inches of cake thicknesses. Reagent dosages above 2 lb/ton did not seem to further reduce the cake moisture substantially.

It was shown at Subtask 1.1a that reagent 01DW145 was more effective than other reagents for dewatering zinc concentrate. In Subtask 1.1b, the same reagent was used to dewater copper concentrate sample from Aitik Mine. Table 6 shows the results. At 1 lb/ton 01DW145, cake moisture was reduced from 11.2% (control) to 5.2% at 0.4 inch cake thickness. At 2 lb/ton 01DW145, the moisture was further reduced to 4.6%.

Table 6 Effects of Using 01DW145 for Dewatering Aitik Copper Concentrate at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	9.9	11.2	12.2
1	4.8	5.2	6.4
2	4.3	4.6	5.7
3	3.7	4.2	5.5
5	3.2	3.7	5.2

treated with 50 g/ton KAX and 50 g/ton MIBC; particle size 0.150 mm x 0; 22.3% solids. 2 minutes drying cycle time.

Note, however, that the moisture difference between 1 and 2 lb/ton reagent dosages was less than 1%.

Drying cycle time is one of the more important process variables in fine particle dewatering. Therefore, a series of dewatering tests were conducted by varying drying cycle times. The dewatering aid used for this series was Reagent RE1 as a 1:2 blend with diesel oil. Table 7 shows the test results obtained using a 2.5 inch diameter Buchner filter at 25 inches of Hg vacuum and 1, 2, and 3 minutes of drying cycle times. In control tests, the moisture was reduced from 8.6% to 7.1% as the drying cycle time was increased from 1 minute to 3 minutes. The difference was only 1.5 percentage point. At 2 lb/ton Reagent RE1, on the other hand, the moisture was reduced from 5.2% at 1 minute drying cycle time to 3.0 lb/ton at 3 minutes of drying time. The difference was larger. Thus, the use of Reagent RE1 allowed the drying cycle time to be more effective.

Table 8 shows the effects of slurry temperature on dewatering copper concentrate. The tests were performed using a 1:2 blend of RA1 and diesel. The tests

Table 7 Effect of Using Reagent RE1 for Dewatering a Aitik Copper Concentrate Sample at 25 Inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Drying Cycle Time (min)		
	1 minute	2 minutes	3 minutes
0	8.6	7.8	7.1
1	5.6	3.9	3.5
2	5.2	3.5	3.0
3	4.8	3.5	2.7
5	4.9	3.7	2.9

Refloated with 25 g/ton KAX and 75 g/ton MIBC; particle size 0.150 mm x 0; solid content 21.6%; cake thickness 0.2 in.

Table 8 Effects of Temperature on the Dewatering of Aitik Copper Concentrate Samples Using RA1 at 25 Inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%)		
	Temperature (°C)		
	25	40	60
0	9.0	8.3	8.0
1	5.5	5.1	5.0
2	5.4	4.5	4.1
3	5.1	4.3	4.5
5	5.6	4.1	3.9

Reflotaed with 25 g/ton KAX and 75 g/ton MIBC; size 0.150 mm x 0; solid content 21.6%; cake thickness 0.4 in; drying cycle time 2 minutes.

were conducted at a vacuum pressure of 25 inches Hg, 2 minutes for drying cycle and 0.4 inches of cake thickness by varying the slurry temperature from 25 to 60°C. The results given in Table 8 show that baseline moistures were 9.0, 8.3, and 8% at 25, 40, and 60°C, respectively. At 2 lb/ton RA1, however, the cake moistures were 5.4, 4.5 and 4.1% at 25, 40, and 60°C, respectively. Thus, cake moisture becomes lower at higher temperature, particularly in the presence of Reagent RA1. Increased dewatering at higher temperatures may be attributed to the lower viscosity. As suggested by Poiseuille (Eq: 6) and Darcy Equations (Eq: 5), a lower viscosity is desired for improved kinetics of dewatering.

c) Galena

The novel dewatering aids developed at MCT were also tested for the dewatering of lead (Galena, PbS) concentrate (0.074 mm x 0) from Boliden AB, Sweden. The galena concentrate was received as a thickened slurry, which was oxidized during transportation and storage. To regenerate a fresh hydrophobic surface, the sample was

Table 9 Effects of Using Reagent RA1 for the Dewatering Boliden-Lead Concentrate Sample at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	9.9	11.5	13.1
1	5.3	5.5	7.8
2	4.5	5.2	6.3
3	4.3	5.1	5.9
5	4.0	5.3	5.8

Ball mill ground and reflotaed with 50 g/ton KAX and 50 g/ton MIBC; size 0.075 mm x 0; solid content 26.3%; drying cycle time 2 minutes.

wet-ground for 1.5 minutes and re-floated using 50 g/ton of KAX and 50 g/ton MIBC at pH 9.5 as adjusted with lime. The froth product, which was at 26.3% solids, was used directly without thickening or dilution using a 2.5-inch diameter Buchner funnel filter at 25 inches Hg vacuum pressure and 2-minute drying cycle time. The dewatering aid (Reagent RA1) was used as a 1:2 blend with diesel.

The test results are given in Table 9. At 2 lb/ton RA1, the cake moisture was reduced from 13.1 to 6.3% at 0.6 inches of cake thickness. Lower moistures were obtained at thinner cake. For example, 5.5 and 5.3% moistures were obtained at 0.4 and 0.2 inches cake thicknesses, respectively at a low reagent dosage of 1 lb/ton RA1. At such low levels of moistures, there is no need for thermal drying.

Another series of dewatering tests were conducted on the lead concentrate using 01DW145 as a dewatering aid, with the results shown in Table 10. As shown, a thinner cake produced the best results, which represents a more efficient liquid transport through the thin cake as compared to the thick cake. However, a thick cake (0.6 inches) still yielded a 54% moisture reduction at 3 lbs/ton 01DW145.

Table 10 Effects of Using 01DW145 on the Dewatering of the Boliden Lead Concentrate Samples at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	9.8	11.6	12.2
1	4.2	4.4	7.1
2	3.9	4.2	6.7
3	3.7	4.1	5.6
5	3.6	4.4	5.8

Reground and reflotated with 50 g/ton KAX and 50 g/ton MIBC; size 0.075 mm x 0; solid content 26.3%; drying cycle time 2 minutes.

Table 11 shows the results obtained with Reagent RE1 for the dewatering of the Boliden lead concentrate. With this reagent, moisture reduction was not as good as with the other reagents. At a dosage as high as 5 lb/ton and a relatively thin 0.2 inches cake of thickness, the moisture was reduced from 12.9 to 7.8%, which represents only 39.6% moisture reduction. The relatively poor results may be attributed to the high baseline moisture, which in turn was probably due to possible oxidation. On the other hand, Reagent RE seems to be less sensitive to cake thicknesses than the other reagents.

Table 11 Effects of Using Reagent RE1 for the Dewatering of the Boliden Lead Concentrate Samples at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	12.9	13.0	14.4
1	9.3	10.1	11.2
2	8.3	9.9	10.3
3	8.2	8.0	9.6
5	7.8	8.4	8.8

Reground and reflotated with 50 g/ton KAX and 50 g/ton MIBC; size 0.075 mm x 0; solid content 26.3%; drying cycle time 2 minutes.

Table 12 Effects of Using 01DU133 for the Dewatering of Boliden-Lead Concentrate at 25 inches Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	13.0	13.4	14.8
1	9.4	10.1	12.0
2	8.7	9.1	10.9
3	9.0	9.6	10.3
5	9.2	9.8	10.4

Refloated with 50 g/ton KAX and 50 g/ton MIBC; size 0.075 mm x 0; solid content 26.3%; drying cycle time 2 minutes.

The next series of laboratory dewatering tests were conducted using 01DU133 as a dewatering aid. The nature of this reagent was quite different from 01DW145, which gave excellent results for dewatering the copper and zinc concentrate (Subtask 1.1a and b). The new reagent (01DU133) was used as a 1:2 blend with diesel. Table 12 shows the results obtained using the 2.5-inch diameter vacuum filter at 25 inches of Hg. As shown, the results were not as good as those obtained using 01DW145 (see Table 10). It seems, however, that this reagent is less sensitive to the changes in cake thickness as other dewatering aids. At 5 lb/ton 01DU133, the moisture reductions were 39% at both 0.2 and 0.4 inch cake thicknesses.

It may be of interest to note here that in addition to being able to achieve high moisture reductions, the dewatering aids tested in the present work also increased the kinetics of dewatering. In general, cake formation time is 2 to 3 times shorter than the case of not using any dewatering aid. Increased kinetics may not necessarily give rise to lower cake moistures. However, the increased kinetics invariably results in higher cake thicknesses and, hence, higher throughput. Being able to increase throughput is

an important aspect of commercializing a new dewatering aid. The main reason for the increased kinetics bestowed by the dewatering aids tested in the present work is that the reagent blends can cause i) increase in contact angle, ii) decrease in surface tension and iii) increase in capillary radius, all at the same time.

d) Talc

Talc ($Mg_3Si_4O_{10}(OH)_2$), a naturally hydrophobic mineral is utilized for a variety of applications including but not limited to paper coating (removal of sticky materials from wood pulp), ceramics, polymer composites, paint, etc (Yordon, 2002). The talc sample employed for dewatering tests was acquired from Luzenac – America. The sample was floated by adding 75 g/ton MIBC as a frother at neutral pH before filtration tests were conducted. The solid content of the flotation concentrate (150 x 0 μm) was 25.3%. The dewatering tests were carried out in the same manner as previously described in the experimental section.

Table 13 shows dewatering results obtained by using RA1 as the dewatering aid.

Table 13 Effects of Using RA1 for the Dewatering of Luzenac Talc Samples at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	24.9	25.8	26.7
1	15.3	17.7	20.9
2	14.4	14.6	17.9
3	12.6	14.5	16.5
5	11.9	13.6	17.6

* 2.5 in diameter vacuum filter used; the sample treated using 75 g/ton MIBC; size 0.150 mm x 0; solid content 25.3%; drying cycle time 2 minutes.

RA1 was mixed with diesel in a 1:2 volume ratio of active to solvent. Greater than 50% moisture reductions are observed for 0.2 and 0.4 inches cake thickness with addition of 5 lb/ton RA1. A 0.6-inch cake thickness and 5lb/ton dosage of RA1 decreased moisture from 26.7 to 17.6% at the same dewatering conditions, as well.

Reagent 01DW145 was also tested as a dewatering aid on the Luzenac talc sample. The test results shown in Table 14 show an increase in baseline moisture (24.4, 25.6, and 26.4%) with increasing cake thickness (0.2, 0.4, and 0.6 inches). The addition of reagent 01DW145 to the slurry, decreases cake moistures by approximately 12% for 0.2 and 0.4 inch cakes and 7% moisture for the thickest cake of 0.6 inches. For example, with a dosage of 3 lb/ton 01DW145, the resultant cake moistures were lowered to 13.8, 15.0 and 18.9% at 0.2, 0.4 and 0.6 inches of cake thickness, respectively. Further increase in the reagent dosage beyond 3lb/ton suggests diminishing returns which confirm that for this sample 3 lb/ton of the reagent can be sufficient.

Table 15 shows the effects of using reagent RE1 as the dewatering aid for the

Table 14 Effects of Using 01DW145 for the Dewatering* of Luzenac Talc Samples at 25 Inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	24.4	25.6	26.4
1	18.2	19.1	22.1
2	14.7	15.9	19.9
3	13.8	15.0	18.9
5	12.3	13.7	18.6

* 2.5 in diameter vacuum filter used; the sample treated using 75 g/ton MIBC; size 0.150 mm x 0; solid content 25.3%; drying cycle time 2 minutes.

same Luzenac – America talc sample when cake thickness is varied. The reagent, RE1, was mixed to a 1:2 volume ratio with diesel and added to the talc concentrate. Test results shown in Table 15 demonstrate that increasing cake thickness will also increase moisture. For example moisture results for 3lb/ton treatment of RE1 will produce filter cakes with 14.3, 17.9, and 19.9% at 0.2, 0.4, and 0.6 inches of cake thickness. These test results correspond to the previous talc dewatering results.

Results for the effects of using RG1 as a dewatering aid on the Luzenac talc flotation concentrate are given in Table 16. A 1:2 volume ratio of active to solvent was used for RG1 and diesel. At 3 lb/ton of RG1, moisture was reduced from 26 to 17.6% at 0.4 cake thickness, which approximately represents a 32% moisture reduction. However, a reagent dosage of 3lb/ton combined with a cake thickness of 0.6 inches produced final moisture of 20.1%, a decrease of approximately 25% when compared to the baseline test.

Table 15 Effects of Using RE1 for the Dewatering* of Luzenac Talc Samples at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	23.8	25.1	26.1
1	15.2	19.0	21.7
2	15.0	17.3	21.2
3	14.3	17.9	19.9
5	13.9	16.5	19.8

* 2.5 in diameter vacuum filter used; the sample treated using 75 g/ton MIBC; size 0.150 mm x 0; solid content 25.3%; drying cycle time 2 minutes.

Table 16 Effects of Using RG1 for the Dewatering* of Luzenac Talc Samples at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	24.2	26.0	26.9
1	16.8	19.9	23.0
2	16.2	18.2	21.8
3	15.7	17.6	20.1
5	14.6	17.0	19.9

* 2.5 in diameter vacuum filter used; the sample treated using 75 g/ton MIBC; size 0.150 mm x 0; solid content 25.3%; drying cycle time 2 minutes.

01DU133 was chosen as a dewatering aid to test its effectiveness in removing water from the Luzenac talc flotation concentrate. The results, shown in Table 17, report the effects of combining varied 01DU133 dosage and increased cake thickness. The active reagent, 01DU133, was mixed with diesel to a 1:2 volume ratio. As detailed in Table 17, moisture reductions when a 5 lb/ton dosage of 01DU133 was used are 43,

Table 17 Effects of Using 01DU133 for the Dewatering* of Luzenac Talc Samples at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Cake Thickness (in)		
	0.2	0.4	0.6
0	24.2	25.2	26.5
1	18.7	20.0	23.5
2	16.3	19.6	21.5
3	15.4	18.4	20.4
5	13.8	16.3	19.7

* 2.5 in diameter vacuum filter used; the sample treated using 75 g/ton MIBC; size 0.150 mm x 0; solid content 25.3%; drying cycle time 2 minutes.

35, and 26% at 0.2, 0.4 and 0.6 inches of cake thickness, respectively when compared to the baseline moistures.

In the last set of talc dewatering tests, RY2 was employed as a dewatering aid. Reagent RY2 was combined with diesel in a volume ratio of 1:2 active to solvent for this test. Test results given in Table 18 show moisture reductions to be between 50 and 38% at a dosage of 3 lb/ton RY2. For example, a 3lb/ton RY2 dosage and 0.4 inch filter cake yields the moisture of 14.7%, which corresponds to a 45% moisture reduction when compared to the baseline test. Additionally, it is important to note that RY2 is an edible product and therefore safe for talc used for medical purposes, however, more study will need to be conducted to find a more suitable (edible) solvent for RY2 than diesel which is not edible.

The effects of MCT developed dewatering aids used on Luzenac- America talc

Table 18 Effects of Using RY2 for the Dewatering* of Luzenac Talc (0.15 mm x 0) Samples at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)		
	Cake Thickness (inch)		
	0.2	0.4	0.6
0	25.2	26.9	28.4
1	14.2	17.3	18.9
3	12.6	14.7	17.5
5	11.8	13.4	16.3

* 2.5 in diameter vacuum filter used; the sample treated using 75 g/ton MIBC; size 0.150 mm x 0; solid content 25.3%; drying cycle time 2 minutes.

show significant moisture reductions for all reagents. Moisture reductions span from 53 to 23% moisture reductions for the entire suite of dewatering aids and cake thicknesses. The reagent providing the best moisture reductions was RY2 overall when comparing final moisture percentages with baseline results and using a 3lb/ton total reagent dosage. However, a comparison of overall final moistures by weight percent, RY2, 01DW145, and RA1 are approximately equal in dewatering effectiveness for all cake thicknesses when adding a 3lb/ton reagent dosage. The advantage of using the MCT developed dewatering aids is the decreased need of costly thermal drying to lower talc filter cake moistures to acceptable industry levels.

e) Silica

Silica is a naturally hydrophilic material that adsorbs surface water easily. To test for the efficiency of MCT-developed reagents to remove water from fine particle silica, a series of dewatering tests were conducted with a silica sample (74 μm x 0) obtained from Tennessee. Dewatering tests conducted with the Tennessee silica gave information on the effects of combining flotation with dewatering aids. The silica sample was prepared and tested in the same manner as described in the Experimental section.

In the dewatering tests conducted with Sphalerite, Chalcopyrite, Galena, and Talc, the addition of hydrophobizing surfactants are designed to further enhances the hydrophobicity of fine mineral particles, that are either naturally hydrophobic or have been hydrophobized during flotation, which is crucial for spontaneous removal of surface free water. However, as mentioned previously silica is a hydrophilic mineral. Until now, in all of dewatering tests described in this report the MCT reagents have been used to further increase the hydrophobicity oof minerals to be dewatered. The

purpose of this current test series is to determine if the addition of MCT developed reagents can change a mineral surface, in this case silica, from a hydrophilic to a more hydrophobic surface.

A two series of tests was conducted with the silica sample from Tennessee. The first series of tests was to characterize the effects of using 01DW145 alone as a dewatering aid. The results of these tests are given in Table 19. Baseline tests provide a silica filter cake with 21.2% moisture by weigh and a corresponding cake formation time of 114 seconds. At 2 lbs/ton 01DW145, cake moisture was reduced to 16.7% and cake formation time reduced to 107 seconds. Further reagent addition (greater than 2lb/ton) shows an increase in cake moistures (Table 19). The results in Table 19 suggested that the surface properties of the silica particles should be further altered for

Table 19 Treatment Effects on Silica Dewatering* Conducted on Tennessee-USA Sample (74 μmX0) Using 01DW145 Dissolved 33.3% in Diesel at 25 Inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	without Amine Flotation		with Amine Flotation	
	Moisture Content (%wt)	Cake Formation Time (sec)	Moisture Content (%wt)	Cake Formation Time (sec)
0	21.2	114	15.3	20
0.5	19.2	110	7.4	15
1	17.8	105	6.2	12
2	16.7	107	5.8	11
3	17.4	117	6.4	12
5	18.2	121	6.7	12

* 2.5-inch diameter vacuum filter; 2 minutes drying cycle time; sample floated using 200 g/ton Dodecylamine at pH 9.5; and cake thickness 0.45 inches.

higher moisture reduction. To change the surface characteristics (hydrophobicity) of the fine particle silica, the sample was treated with 200 g/ton lime, and then floated with 200 g/ton dodecylamine, 100 g/ton MIBC at a pH of 9.5. This treatment is referred to as the first hydrophobization step. As shown in Table 19 under the "with Amine Flotation" heading, the lime and amine combination reduced cake moisture from 21.2 to 15.3%, and reduced cake formation time by 94 seconds. When the novel dewatering aid was added to the flotation product as a second hydrophobization step, the moisture was further reduced to 5.8% with a 2 lb/ton 01DW145 dosage. Additionally, the cake formation time was also lowered by 8 seconds.

A similar set of tests was conducted with the same silica sample but using Reagent 01DW111 blended with diesel blend at a 1:2 ratio. As shown in Table 20, a 2

Table 20 Treatment Effects on Silica Dewatering* Conducted on Tennessee-USA Sample (74 μm X 0) Using 01DW111 Dissolved 33.3% in Diesel at 25 Inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	without Amine Flotation		with Amine Flotation	
	Moisture Content (%wt)	Cake Formation Time (sec)	Moisture Content (%wt)	Cake Formation Time (sec)
0	21.2	104	15.3	21
0.5	20.8	101	9.0	16
1	20.6	98	7.1	12
2	19.7	97	6.2	11
3	20.3	113	6.0	10
5	20.8	118	7.4	13

* 2.5-inch diameter vacuum filter; 2 minute drying cycle time; sample floated using 200 g/ton Dodecylamine at pH 9.5; and cake thickness 0.45 inches.

lb/ton dosage of 01DW111 decreased cake moisture from 21.2% to 19.7%, this is 3% higher cake moisture when compared with 01DW145 at the same dosage. However, the cake moisture was significantly reduced to 6.2% when the sample was pre-treated by amine flotation prior to dewatering. The decreased moistures are possibly the result of hydrophobicity enhancement of the fine silica particles due to the two-step hydrophobization process. Further decrease of moisture in the silica filter cakes is not needed because of handling, storage and health problems created by dry silica dust.

Additional dewatering tests were performed using a fine quartz powder obtained from Fisher Chemical Scientific. The tests provide evidence that the MCT developed surfactants work well when the particles are reasonably hydrophobic. The use of the surfactant in a manner described in the earlier sections further enhances the hydrophobicity of the particles, which is essential for spontaneous dewatering of fine

Table 21 Treatment Effects on Silica Dewatering* Conducted on a Fisher Silica flour (38 μm x 0) Using 01DU133 Dissolved 33.3% in Diesel at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	without Amine Flotation		with Amine Flotation	
	Moisture Content (%wt)	Cake Formation Time (sec)	Moisture Content (%wt)	Cake Formation Time (sec)
0	26.4	161	19.2	26
0.5	26.0	158	12.1	20
1	25.3	157	9.7	19
2	24.4	158	8.9	18
3	24.7	170	8.9	18
5	24.6	183	9.2	19

* 2.5-inch diameter vacuum filter; 2 minute drying cycle time; Fisher sample floated using 200 g/ton Dodecylamine at pH 9.5; and cake thickness 0.45 inches.

particles. The hydrophobicity of the silica flotation product is not strong enough to support spontaneous moisture removal. To better demonstrate this point, a series of dewatering tests were conducted on the 38 μm x 0 size silica sample from the Fischer Scientific Company. The silica sample was dewatered in the same manner as described in the Experimental section and tests were conducted similar to that of the Tennessee silica sample with a cake thickness of 0.45 inches at 25 inches of Hg vacuum.

The reagent 01DU133 was used as the dewatering aid after dissolved in diesel at a 1:2 volume ratio. Table 21 presents baseline cake moisture of 26.4% with a formation time of 161 seconds. The addition of 2lbs/ton 01DU133 reduced the cake moisture to 24.4% and decreased the formation time to 158 seconds. The moisture reduction obtained in this test was not as effective as those obtained in the previous examples. The use of dodecylamine in flotation, as the first hydrophobizing step, decreased cake moisture to 19.2% and cake formation time to 26 seconds. When the dewatering aid, 01DU133, was added to the flotation product as a second hydrophobization reagent, the moisture was further reduced to 8.9% at a 2 lb/ton dosage; this corresponds to an overall moisture decrease of 66% when compared to baseline results. Additionally, the cake formation time was further decreased to 18 seconds. The previous tests show that low HLB numbered surfactants (dewatering aids) work more effectively when naturally hydrophilic particles are initially hydrophobized with a high HLB surfactant like amine used in these examples.

f) Clay

Kaolin is a naturally hydrophilic clay mineral and therefore very difficult to dewater. In the kaolin industry, fine clay is dewatered using drum and high-pressure filters, which yields a product with 40 to 60% moisture content in the filter cake. In addition, part of the moisture in the filter cake is spray dried using a natural gas flame to obtain a 70 to 90% solid content (30 to 10% moisture) product before marketing. The spray drying is costly and not environmentally benign, but it is the only method used to produce relatively dry clay (Tiller, 1996; Basilio, 1997; Yoon, 1992). Filtration tests were conducted on an East Georgia kaolin clay sample, the size of which was 90% finer than 2 μm. Initially, the kaolin clay was bleached and pretreated using sodium hydrosulfite, alum and NALCO 9765 chemicals at pH 2.8 (sulfuric acid added). The clay sample was then conditioned using a 1000 g/ton dodecylammonium hydrochloride and 120 g/ton MIBC at pH 9.3 (lime added) to make the surface slightly hydrophobic - called first

Table 22 Dewatering Results* of the East Georgia Kaolin Clay Sample* Using 01DW145 (dissolved 33.3% in diesel) and ARY16 Spray at 150 kPa Air Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DW145	01DW145 + ARY16 Spray
0	43.1	41.2
10	38.6	36.5
20	36.9	34.6
30	35.7	33.3
50	33.3	30.8

*2.5 in diameter air pressure filter used; the clay sample was bleached and pretreated using sodium hydrosulfite, alum and NALCO 9765 chemicals at pH 2.8 (sulfuric acid). This sample was then conditioned with 1000 g/ton of Dodecyl Amine at pH 9.2 (lime); particle size 2 μm x 0; solid content of sample 5%; cake thickness 0.1 inch; conditioning time 5 minutes; drying cycle time 3 minutes.

hydrophobization step. Afterwards, the treated sample was subjected to pressure filtration tests.

The test results obtained using 01DW145 and ethanol spray is given in Table 22. The baseline moisture is 43.1% when using pressure filtration with no reagent added. At a 30 lb/ton 01DW145 (dissolved in diesel at a 1:2 ratio), the moisture content of the cake was reduced to 35.7%. Additionally, when 3-5 lb/ton of ethanol was sprayed on the cake, the cake moisture decreased further to 33.3%. These results suggest that the dewatering methods may be able to eliminate the use of spray dryers in the clay industry or at least decrease the overall dewatering cost. With further optimization of the process, the reagent consumption can be reduced to significantly lower levels for the clay dewatering.

The next set of tests was carried out on the same treated kaolin samples using

Table 23 Dewatering Results* of the East Georgia Kaolin Clay Sample* Using 01DU133 (dissolved 33.3% in diesel) and ARY16 Spray at 150 kPa Air Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DU133	01DU133+ ARY16 Spray
0	43.7	41.6
10	37.6	35.1
20	35.6	32.9
30	32.8	30.1
50	31.6	29.6

*2.5 in diameter air pressure filter used; the clay sample was bleached and pretreated using sodium hydrosulfite, alum and NALCO 9765 chemicals at pH 2.8 (sulfuric acid). This sample was then conditioned with 1000 g/ton of Dodecyl Amine at pH 9.2 (lime); particle size 2 μm x 0; solid content of sample 5%; cake thickness 0.1 inch; conditioning time 5 minutes; drying cycle time 3 minutes.

01DU133 as a dewatering aid. The results, given in Table 23, show that the moisture reduction of 01DU133 is very identical to that of 01DW145. This means that when clay particles are initially hydrophobized by amine type collector at appropriate conditions, those particles are vulnerable to be further hydrophobized by newly developed MCT chemicals. This may be the main reason of achieving higher moisture reduction for kaolin clay sample.

Some of the dewatering aids are highly purified reagents used for food, pharmacy or cosmetics, so the costs of these chemicals can be prohibitive. For this reason, low-cost dewatering aids were developed by MCT to decrease the overall reagent cost. 01DW111 is a natural low cost reagent and used for dewatering of the East Georgia Kaolin Clay with ethanol spray. Table 24 shows the results of the air

Table 24 Dewatering Results* of the East Georgia Kaolin Clay Sample* Using Reagent 01DW111 (dissolved 33.3% in diesel) and ARY16 Spray at 150 kPa Air Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DW111	01DW111 + ARY16 Spray
0	43.6	40.9
10	39.2	37.1
20	37.6	35.0
30	36.1	33.7
50	34.4	31.0

*2.5 in diameter air pressure filter used; the clay sample was bleached and pretreated using sodium hydrosulfite, alum and NALCO 9765 chemicals at pH 2.8 (sulfuric acid). This sample was then conditioned with 1000 g/ton of Dodecyl Amine at pH 9.2 (lime); particle size 2 μm x 0; solid content of sample 5%; cake thickness 0.1 inch; conditioning time 5 minutes; drying cycle time 3 minutes.

Table 25 Dewatering Results* of the East Georgia Kaolin Clay Sample* Using Reagent 01DW110 (dissolved 33.3% in diesel) and ARY16 Spray at 150 KPa Air Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DW110	01DW110 + ARY16 Spray
0	43.2	41.5
10	39.7	38.3
20	38.1	37.2
30	38.9	36.8
50	37.2	36.7

*2.5 in diameter air pressure filter used; the clay sample was bleached and pretreated using sodium hydrosulfite, alum and NALCO 9765 chemicals at pH 2.8 (sulfuric acid). This sample was then conditioned with 1000 g/ton of Dodecyl Amine at pH 9.2 (lime); particle size 2 μm x 0; solid content of sample 5%; cake thickness 0.1 inch; conditioning time 5 minutes; drying cycle time 3 minutes.

pressure filter tests conducted using 01DW111 with and without ethanol spray. The results show that the performance of this low cost dewatering aid is as good as the more costly reagents used in the previous tests.

01DW110 is an environmentally safe new generation of hydrophobizing chemical obtained form different naturally occurring compounds. In this set of test, this chemical was utilized to dewater the East Georgia Kaolin Clay, which was bleached and treated using appropriate chemicals and conditions. The test results given in Table 25 show that in the presence of 50 lb/ton of 01DW110 and 3-5 lb/ton of ethanol spray, moisture content of filter cake was reduced form 43.2% to 36.7%. It is apparent that this moisture difference between base and reagent addition will reduce the dewatering cost and environmental concerns of conventionally used thermal dryers for clay industry.

Subtask 1.2 Contact Angle Measurements

The contact angle of water on a flat surface is indicative to the energy needed to remove the water from that surface. As the contact angle of water on a coal or mineral surface increases (figure 1), the energy input required to remove the free surface water decreases (Equations 2 and 3). This process becomes spontaneous at a contact angle of 180º. The purpose of this subtask is to test the effects of using reagents and solvents to change the water contact angle over coal surfaces.

a) Sessile Drop and Wilhelmy Plate Technique

Two methods were employed for the measurement of the contact angle on a chunk coal surface. i.e., the sessile drop technique and Wilhelmy plate technique as mentioned previously in the Experimental section. The water contact angles were measured on coal samples obtained from Elkview, Middle Fork and Massey coal companies. Samples were prepared in the manner explained previously in the Experimental section under Contact Angle Measurement.

Table 26 gives the contact angle measurements of different coal samples. Without reagents, Elkview, Middle Fork and Pittsburgh coal samples gave 32°, 42° and 21° contact angle values, respectively. The addition of 1 lb/ton 01DW145 increased the contact angles to 60°, 70° and 59°, respectively. When 5 lbs/ton dosage of 01DW145 was added to the samples, the contact angles increased to 90°, 92° and 88°. The other reagents (ROE1, 01DW111, and RE1) produced similar contact angles for the same coal samples.

Table 26 Effects of Reagents[1] Dosages on the Contact Angle of Coal Samples* Using Sessile Drop Technique

Reagent Dosage (lb/ton)	Contact Angle (°)					
	Elkview Coal		Middle Fork Coal		Pittsburgh Coal	
	01DW145	ROE1	01DW145	01DW111	01DW145	RE1
0	32	32	42	42	21	21
1	60	68	70	68	59	56
2	76	80	79	75	72	76
3	85	87	86	83	83	84
5	90	92	92	88	88	90

[1]The reagents dissolved 33.3% in diesel. *one side of coal samples polished using 120, 240, 400 and 600 grit sand papers. The reagents were added to slurry including the coal powder sample (-1.7 + 0.3 mm) and polished coal chunks to measure the contact angles.

In the next series of tests, dewatering test was conducted with Pittsburgh coal samples. At the meanwhile, the contact angle of the coal sample and the surface tension of the filtrate were measured. Kerosene, a conventional solvent widely used in coal industry, was utilized to compare the results with the newly developed dewatering aids such as RG1, 01DW111 and RY5. The dense medium coal sample was crushed, ground and floated using l lb/ton of kerosene and 100 g/ton of MIBC prior to dewatering tests. Tables 27, 28 and 29 show contact angle results measured on a Pittsburgh coal sample, along with the results of the Buchner filter tests conducted on the same coal samples and the surface tensions of the filtrates (process waters).

Table 27 Effects of Reagent RG1 and Kerosene on Contact Angle, Filtrate Surface Tension, Cake Formation Time and Cake Moisture of a Pittsburgh Coal Sample

Reagent Type	Reagent Dosages (lbs/ton)	Contact Angle (Degree)	Filtrate Surface Tension (μN/m)	Cake Formation Time (sec)	Moisture Content (%wt)
None	0	12	71	61	28.2
Kerosene	1	44	69	50	24.9
RG1	1	73	65	24	18.4
	2	86	62	15	16.2
	3	89	63	13	15.4
	5	91	56	12	15.5

*2.5 in diameter Buchner filter used; dense medium coal sample crushed, ground and floated using 1 lb/ton kerosene and 100 g/ton MIBC; RG1 dissolved 33.3% in diesel; solid content of sample 18.2%; vacuum pressure 25 inches of Hg; cake thickness 0.5 inch; particle size -0.5 mm; conditioning time 2 minutes; drying cycle time 2 minutes.

Results show that the reagent additions provides a substantial increase in contact angle and a decrease in surface tension, both of which are conducive to the improvement of dewatering fine particles. With a 5 lb/ton addition of reagent RG1,

Table 28 Effects of Kerosene and 01DW111 on Contact Angle of a Pittsburgh Coal Sample, Filtrate Surface Tension, Cake Formation Time and Cake Moisture*

Reagent Type	Reagent Dosages (lb./ton)	Contact Angle (Degree)	Filtrate Surface Tension (μN/m)	Cake Formation Time (sec)	Moisture Content (%wt)
None	0	16	71.4	36	28.7
Kerosene	1	42	70.2	24	25.3
01DW111	1	72	65.4	16	17.6
	2	81	65.7	13	16.1
	3	87	60.4	12	15.2
	5	91	59.0	11	14.7

*2.5 in diameter Buchner filter used; dense medium coal sample crushed, ground and floated using 1 lb/ton kerosene and 100 g/ton MIBC; 01DW111 dissolved 33.3% in diesel; 18.2% solids; vacuum pressure 25 inches of Hg; cake thickness 0.5 inch; particle size 0.5 mm; conditioning time 2 minutes; drying cycle time 2 minutes.

Table 29 Effects of Kerosene and RY5 on Contact Angle of a Pittsburgh Coal Sample, Filtrate Surface Tension, Cake Formation Time and Cake Moisture*

Reagent Type	Reagent Dosages (lbs/ton)	Contact Angle (Degree)	Filtrate Surface Tension (μN/m)	Cake Formation Time (sec)	Moisture Content (%wt)
None	0	13	71.4	34	27.4
Kerosene	1	48	70.1	27	25.4
RY5	1	69	65.9	16	19.6
	2	75	64.1	13	17.0
	3	87	59.8	12	16.5
	5	88	58.7	12	16.6

*2.5 in diameter Buchner filter used; dense medium coal sample crushed, ground and floated using 1 lb/ton kerosene and 100 g/ton MIBC; RY5 dissolved 33.3% in diesel; solid content of sample 18.2%; vacuum pressure 25 inches of Hg; cake thickness 0.5 inch; particle size 0.5 mm; conditioning time 2 minutes; drying cycle time 2 minutes.

01DW111 and RY5, the contact angles increased from around 10° to greater than 90°, also, moistures were decreased from approximately 28% to 15.5, 14.7 and 16.6%, respectively. The results of this test give proof to the effectiveness of MCT developed dewatering aids to enhance the hydrophobicity of coal particles. According to the Laplace equation (Eq: 5), capillary pressure is negative for contact angles above 90° (Adamson, 1997; Myers, 1988; Leja, 1982). The theory behind the Laplace equation was observed in this series of tests as a significant decrease in applied pressure during the tests. Additionally, cake formation times were reduced approximately three to five times when reagents were added.

It is also important to note that a 1 lb/ton kerosene addition reduced moistures from approximately 28 to 25%, which is less effective than using the newly developed

MCT Reagents dissolved in diesel. Increased dosage of kerosene did not improve moisture reduction beyond 5%. Additionally, when kerosene dosage was significantly increased to high concentrations, moisture content increased as a result of water trapped within the flocs of coal created by the high reagent dosage.

b) Thin Layer Wicking Technique

The thin layer wicking tests were conducted on the fine silica and coal samples by immersing the glass slides (coated with fine particles) vertically into 5 mm depth of nonpolar wicking liquids (alkenes) in 150 ml beaker. The coated slides were sealed inside a conditioning beaker for an hour to introduce the vapor of the wicking liquids to the silica particles before the tests (van Oss, 1992). When the prepared slides were immersed into the nonpolar low energy liquids, the liquid traveled rapidly across the glass surface. For higher energy liquids or hydrophobic particles, this time can take more than half an hour. During the tests, the travel distance (l^2) was recorded as a function of time (t) and plotted to determine the mean pore radii r^* and contact angle θ using the Washburn equation. For each coal and silica powders, the plot of $2\eta l^2/t$ vs. γ_{lv} the alkenes should be a straight line with a slope of r^*. Initially, the contact angle θ was assumed to be zero (i.e., cos θ = 1) because of the low surface tension and apolarity of the wicking liquids, such as hexane, heptane, octane and decane. When the mean pore radius was determined, the contact angle values of high surface tension liquids on a solid substrate, such as water, formamide, ethylene glycol, ethylene iodide and glycerol can be calculated using the Washburn equation. Table 30 shows the surface tension components of selected liquids at 20 °C (Adamson, 1997; Myers, 1988; Leja, 1982; van Oss, 1992 and 1994).

Table 30 Surface Tension Components (in mN/m) and Viscosities (in poise) of the Selected Wicking Liquids at 20 °C

Liquids	γ_l	γ_l^{LW}	γ_l^{AB}	γ_l^+	γ_l^-	η
Hexane	18.40	18.40	0	0	0	0.00326
Heptane	20.3	20.3	0	0	0	0.00409
Octane	21.6	21.6	0	0	0	0.00542
Decane	23.8	23.8	0	0	0	0.00907
Methylene iodide	50.8	50.8	0	0	0	0.02800
Ethylene Glycol	48.0	29.0	19.0	1.92	47.0	0.19900
Glycerol	64.0	34.0	30.0	3.92	57.4	14.9000
Formamide	58.0	39.0	19.0	2.28	39.6	0.04550
Water	72.8	21.8	51.0	25.5	25.5	0.01000

In the thin layer wicking experiments, linear plots of l^2 vs. t with the coal and silica powders were obtained using hexane, heptane, octane and decane as seen in Figures 7 and 8, correspondingly. These were all apolar low energy liquids that completely covered the particles deposited on the glass slides, representing $\cos\theta=1$ (contact angle of 0°). Using the straight line slope for each liquid, the values of the mean pore radii r^* were determined to be 7.33×10^{-4} for the coal (Figure 9) and 7.12×10^{-5} cm for the silica (Figure 10) from the slopes ($2\eta l^2/t$ vs. γ_{lv}). After the mean pore radius was determined, the same wetting tests were performed with water on the silica sample in the presence and absence of 01DW145. In these tests, again linear plots (l^2 vs. t) of water were determined, and then the water contact angles were calculated from the slopes for coal (Figure 11) and silica (Figure 12) samples. The water contact angles obtained using the Washburn equation are given in Table 31.

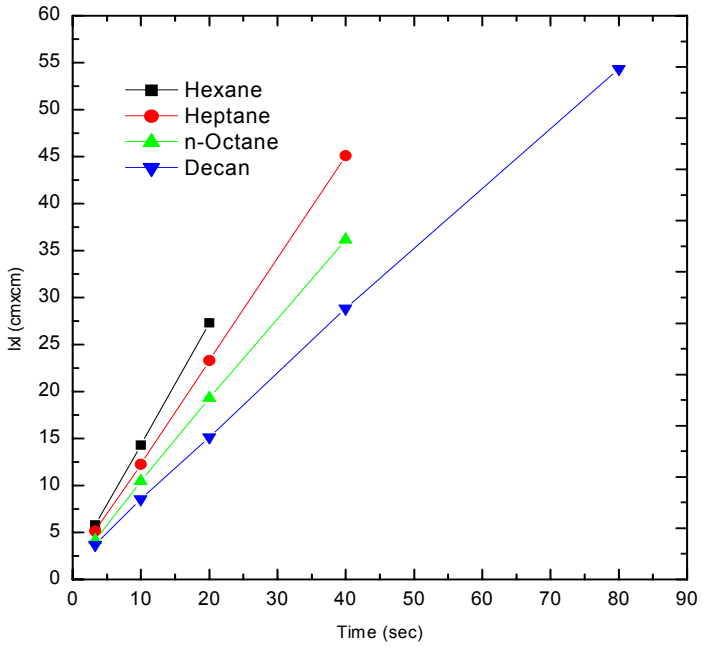

Figure 7 Wetting of Moss 3 Virginia dense medium coal sample (-150 micron) with low energy liquids in the thin layer wicking experiments

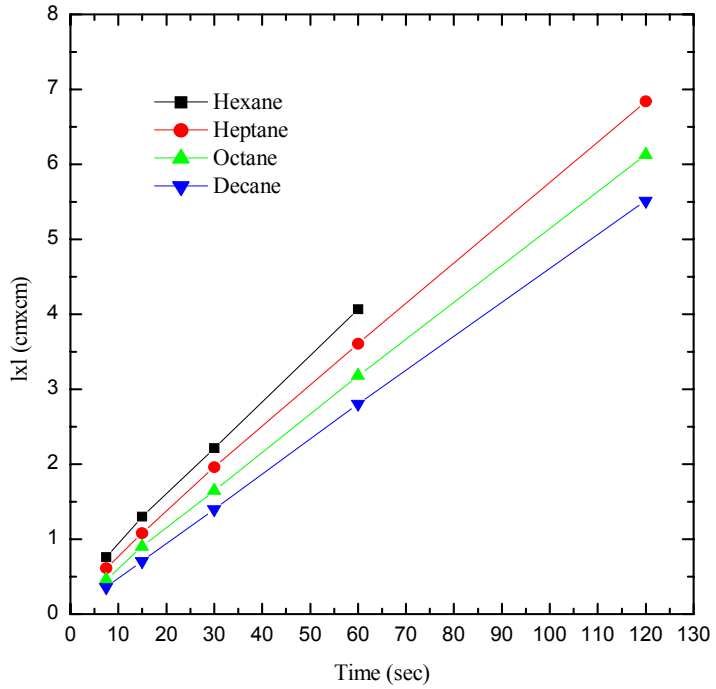

Figure 8 Wetting of the Fisher silica powder (-63 micron) with low energy liquids in the thin layer wicking experiments conducted on glass slides at 20°C.

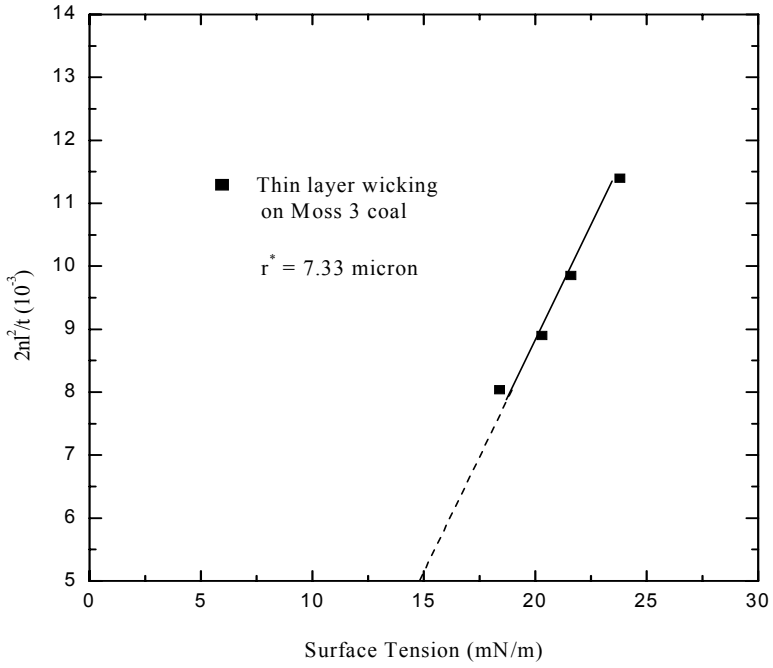

Figure 9 The plot of $2nl^2/t$ vs. surface tension of liquids obtained from thin layer wicking on Moss 3 – Virginia dense medium coal sample (-150 micron) with low energy liquids.

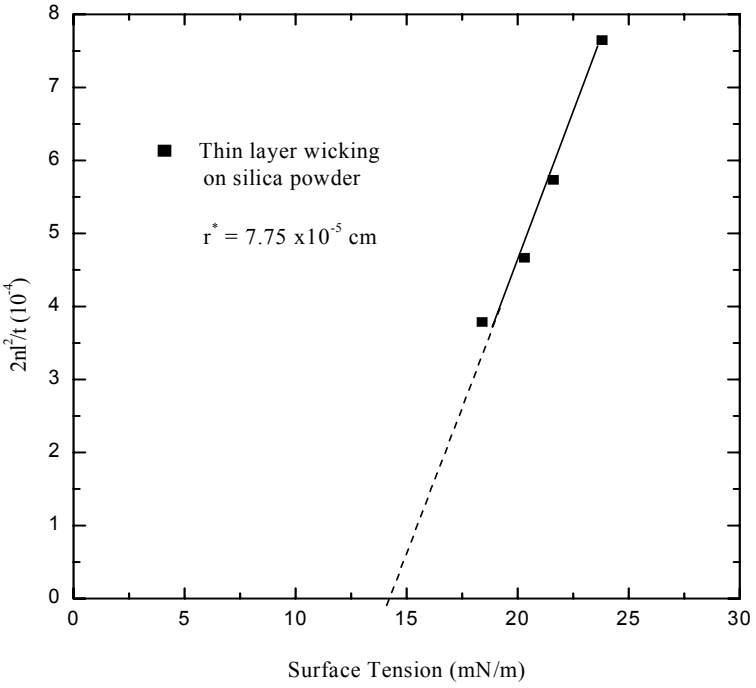

Figure 10 The plot of $2nl^2/t$ vs. surface tension of liquids obtained from the thin layer wicking on the Fisher silica powder (-63 micron) with low energy liquids.

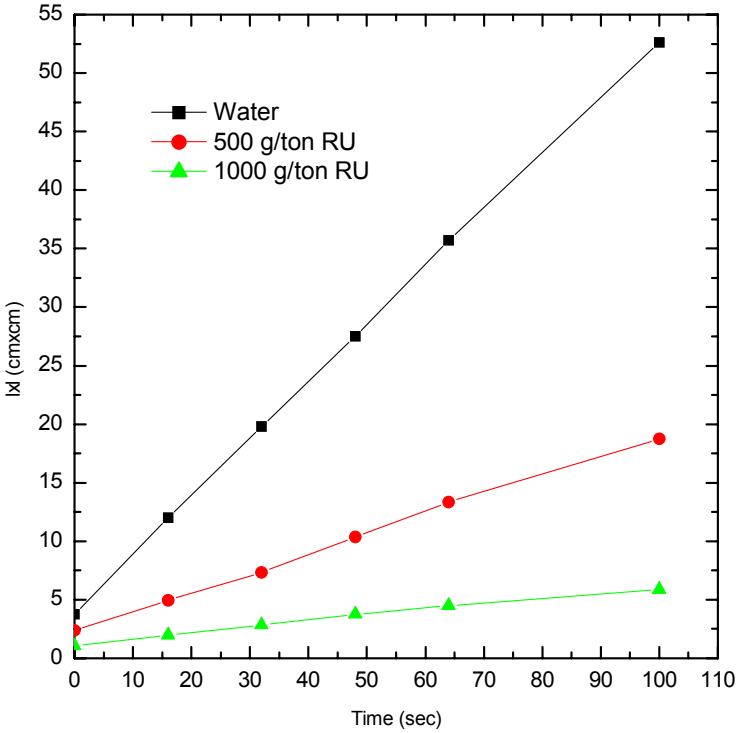

Figure 11 Wetting of Moss 3 – Virginia dense medium coal sample (-150 micron) with water in the presence and absence of 01DW145 in the thin layer wicking experiments conducted on a 2.5x7.5 cm glass slide at 20°C.

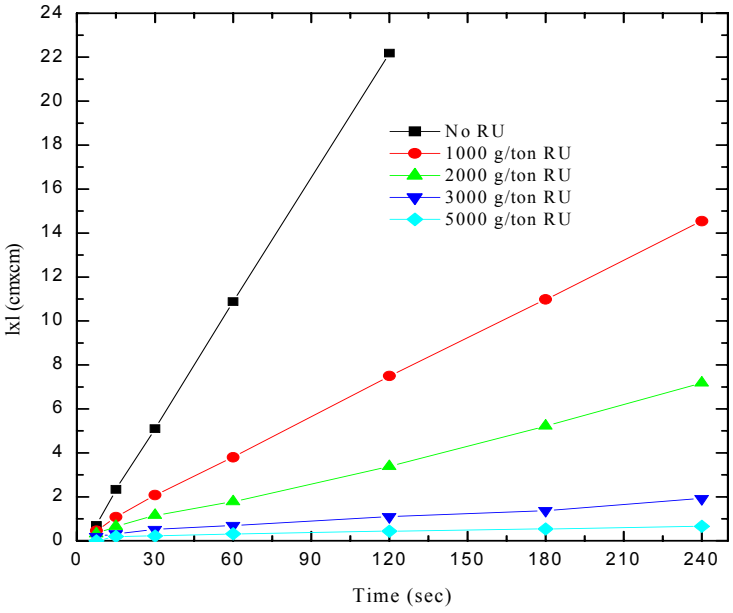

Figure 12 Wetting of the Fisher silica powder (-63 micron) with water in the presence or absence of 01DW145 in the thin layer wicking experiments conducted on glass slides at 20°C.

Table 31 Contact Angle Values as a Function of Reagent Dosage on the Coal and Silica Powders Using Thin Layer Wicking Method

01DW145 dosages (kg/ton)	Water Contact Angles (degrees)	
	Coal	Silica
0	78.7	42.6
1	87.2	75.8
2	89.6	82.5
3	> 90	87.0
5	> 90	89.4

Results in Table 31 show that the contact angle of the untreated silica sample was 42.6° but 78.7° for the untreated coal sample. It was observed in this test that water traveled faster on the silica-coated slide than on the coal coated slide. However, when 01DW145 was added to the silica suspension at a pH of 9.5, the slope of the lines gradually decreased as the increase of reagents dosage. At a 5 kg/ton dose of 01DW145, the line was nearly parallel to the x-axis, and the water contact angle increased from 42.6° to 89.4° correspondently. This indicated that when the reagent dosage was gradually increased, the silica particles became increasingly hydrophobic, which will facilitate the dewatering of the silica slurry. For instance, an increase in contact angle of fine silica particles from 44 to 89°, the energy required for dewatering will be reduced by approximately 41 times according to equation 5. Additionally, hydrophobic coagulation at the higher contact angles increases the pore radii and improves the kinetics of dewatering.

The coal sample from Moss III – Virginia gave 78.7° without any reagent

additions, which indicated that the coal sample was already naturally hydrophobic. Note that this coal sample does not need first hydrophobization step. When a 2 kg/ton 01DW145 was used, the contact angle of the coal particles increased from the baseline angle to approximately 90°. Further increase in reagent dosage did not increase the contact angle, representing the upper limits of the Washburn Equation.

The purpose of the present study is to increase the contact angle to improve the dewatering efficiency of fine coal and mineral particles. The enhanced hydrophobicity of coal and mineral particles will weaken the hydrogen bonds between the water molecules and the surfaces of coal and mineral particles, resulting in more efficient water removal. More efficient water removal from particulate materials, will allow for use of more economical mechanical dewatering devices, such as screens, vacuum filters, pressure filters, and centrifuges.

Subtask 1.3 Reagent Syntheses

a) Modification of Natural Reagents

Previous tests have shown that synthetic dewatering aids are superior to the natural dewatering aids for water removal. In this present work, natural reagents will be modified to change the molecular structure of the reagents. As known, these natural products mostly have one head group with two or three tail groups. The purpose of this subtask is to modify these natural products so that they have more desirable characteristics for dewatering fine particle. Transesterification is a solution of overcoming the disadvantage of natural products by simply breaking the double or triple bonds of the naturally occurring reagents so that hydrophobic monolayer on the surface of fine particles can be enhanced. In this process, lipid molecules react with an alcohol

in the presence of a catalyst such as H^+ or OH^- ions. To accelerate the reaction rate, the reaction may be carried out at a slightly elevated temperature (40 to 80^oC). The reaction products, containing fatty esters and glycerol, may be used without purification to minimize reagent costs.

In the first set of tests, ROE1 was transesterified by mixing with butanol at an approximate molar ratio of 1:3, and agitated on a hot plate for one hour after adding acetic acid (2% by volume) to the mixture. The reaction product was then used as a dewatering aid without purification. A series of vacuum filtration tests were conducted with Middle Fork coal samples using butanol, diesel, pure ROE1, esterified ROE1, pure ROE1/diesel blend (1:2 by volume), and esterified ROE1 (dissolved in diesel at 1:2 ratio). The dense medium coal sample form Middle Fork was crushed, ground, and floated with 1 lb/ton of kerosene and 100 g/ton MIBC. All tests were conducted using a 2.5 inch diameter Buchner funnel at 16.3% solid content, 2 min drying cycle time, 0.41 inch cake thickness and 25 inch Hg vacuum pressure.

In Table 32, it is shown that at 6 lb/ton of kerosene, diesel, pure ROE1, esterified ROE1, pure ROE1 and esterified ROE1 additions, the cake moisture contents were decreased form 21.5% to 18.9, 18.4, 15.4, 14.5, 13.6 and 12.2%, respectively. The reagent dosages given in the table represent bulk dosages (reagent + solvent) of the reagents. Table 32 also shows that a further increase beyond 6lb/ton of reagent did not significantly improve the moisture reductions. The results given in Table 32 clearly show that the modified ROE1 (dissolved in diesel) gave considerably better results than the naturally occurring compounds without modifications and the pure solvents (butanol or diesel). The greater moisture reductions are the result of the closed pack monolayer

Table 32 Effects of Using Kerosene, Diesel, ROE1 and Esterified ROE1 on Dewatering of Middle Fork Coal Sample* (0.6 mm X 0) at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Cake Moisture (% wt)					
	Kerosene	diesel	Pure ROE1	Esterified ROE1	Pure ROE1 (33.3% in diesel)	Esterified ROE1 (33.3% in diesel)
0	21.5	21.5	21.5	21.5	21.5	21.5
1	19.3	17.6	17.1	17.4	16.6	15.8
3	18.9	18.5	16.0	15.9	15.4	13.3
6	18.9	18.4	15.4	14.5	13.6	12.2
9	19.0	18.7	15.0	15.0	13.5	11.3
15	19.9	17.5	14.9	14.9	12.6	10.2

* 2.5 inch diameter Buchaner funnel used; cake thickness 0.41 in.; drying cycle time 2 minutes; solid content 16.3%; the dens medium sample crushed, ground and floated using 1 lb/ton kerosene and 100 g/ton MIBC. 1 The ROE1 esterified by using 1 mole of ROE1, 3 mole butanol and 2% by volume acetic acid on a hot plate.

formation on the solid surface and the solubility improvements by esterification.

In the next set of tests (Table 33), RY10 was used as a dewatering aid. RY10 was mixed with ethanol at an approximate molar ratio of 1:3. After mixing, acetic acid (approximately 2% by volume) was added and was then agitated on a hot plate for one hour. The product, containing RA3, RA7, RA4, and glycerol, was used as a dewatering aid without purification in vacuum filtration tests. Additionally, ethanol, diesel and pure RY10 were also tested to compare the esterification effects on fine coal dewatering. The tests were conducted on the SGS Australian coal sample, which had been pulverized and ground in the same manner as mentioned above. The test results are given in Table 33. As the results demonstrate, the use of the modified RY10 reduced the cake moistures substantially. The performance was much better than that obtained form unmodified RY10.

Further testing used RY2 with and without transesterification. RY2 was esterified by the same methods as described above. The tests were conducted on a dense medium coal sample shipped from the Meadow River coal preparation plant. It was pulverized, wet-ground in a ball mill, and floated using 1 lb/ton kerosene and 0.2 lb/ton MIBC. In each experiment, one part of the modified RY2 was dissolved in two parts of diesel by volume. The test results are given in Table 34. Baseline tests produced filter cakes with 22.6% moisture. When a 1 lb/ton RY2 or 1 lb/ton esterified RY2 was added to the sample slurry, the moisture contents of the cakes was decreased to 16.3% and 13.5%, correspondingly. A 5 lb/ton addition of the reagents further reduced cake moistures to 13.3% and 9.9%. Results in Table 34 show that lower cake moistures can be obtained when using esterified RY2 as a dewatering aid.

The present studies suggest that esterified reagents are more effective reagents

Table 33 Effects of Using Ethanol, Diesel, RY10 and Esterified RY10 on Dewatering of SGS-Australia Coal Sample* (0.5 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Cake Moisture (% wt)					
	Ethanol	Diesel	Pure RY10	Esterified RY10	Pure RY10 (33.3% in Diesel)	Esterified RY10 (33.3% in Diesel)
0	22.4	22.4	22.4	22.4	22.4	22.4
1	20.5	19.9	19.0	17.7	17.1	16.4
3	19.1	18.1	16.0	15.8	15.2	13.4
6	18.6	17.9	15.4	15.6	13.8	11.5
9	18.7	18.7	15.3	15.0	12.8	10.4
15	18.4	19.0	14.9	15.3	12.2	9.9

* 2.5 inch diameter Buchaner funnel used; cake thickness 0.42 in.; drying cycle time 2 minutes; solid content 16.4%; the coarse coal crushed, ground and floated using 1 lb/ton kerosene and 100 g/ton MIBC. 1 The RY10 esterified by using 1 mole of RY10, 3 mole ethanol and 2% by volume acetic acid on a hot plate.

Table 34 Effect of RY2 And Esterified RY2 on Dewatering of Meadow River Coal Sample* (0.5 mm X 0) at 25 Inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	RY2	Esterified RY2
0	22.6	22.6
1	16.3	13.5
2	14.4	11.0
3	13.8	10.6
5	13.3	9.9

* 2.5-inch diameter Buchaner vacuum filter; 2 minute drying cycle time; cake thickness 0.4 in.; dense medium sample floated using 1 lb/ton kerosene and 100 g/ton MIBC; and solid content of sample 16.9%. [1] The processed RY2 obtained using 1 mole RY2, 3 moles ethanol and 2% by volume acetic acid on a hot plate. The reagents dissolved 33.3% in diesel before use.

and work very well with coal. To confirm this efficiency, a series of dewatering tests was performed on the Middle Fork coal samples using esterified RR1 and RG1. Tables 35 and 36 show the dewatering results obtained using the esterified RR1 and RG1 reagents that were blended with diesel at a volume ratio of 1:2. Results in Tables 35 and 36 show that additional 1 to 3% cake moisture reduction can be achieved when using the esterified reagents. This suggests that the esterified reagents are more effective dewatering aids for use in dewatering fine coal.

In the last set of esterification tests, RY15, RY10 and RY16 were esterified using the same procedures as explained as above. The test results are shown in Tables 37 and 38. The results show RY15 offered the best dewatering results, which may be due to the selectivity of this reagent for the absorption on the coal surface.

The use of modified and unmodified reagents presents a clear difference for the reduction of cake moistures. The test results prove the hypothesis that the modification

(breaking the surfactant molecules into small molecules) of natural products can significantly improve effectiveness of fine particle dewatering. Additionally, use of acetic acid in the process of modification possibly changes the acid-base chemistry of the modified reagents, thus improves dewatering results. However, a more detailed study should be conducted on the modifications of the reagents to investigate the change in the molecular structures and the characteristics.

Table 35 Effect of RR1 and Esterified RR1 on Dewatering of Middle Fork Coal Sample* (0.5 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	RR1	Esterified RR1
0	19.3	19.3
1	13.3	10.7
2	12.2	11.3
3	12.7	10.6
5	11.3	10.3

* 2.5-inch diameter Buchaner vacuum filter; 2 minute drying cycle time; cake thickness 0.4-0.5 in.; dens medium sample floated using 1 lb/ton kerosene and 100 g/ton MIBC; and solid content of sample 16.1%. [1] The processed RR1 obtained using 1 mole RR1, 3 moles ARY16 and 5% by volume acetic acid on a hot plate. The reagents dissolved 33.3% in diesel before use.

Table 36 Effect of RG1 and Esterified RG1 on Dewatering of Middle Fork Coal Sample* (0.5 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	RG1	Esterified RG1
0	20.0	20.0
1	14.5	11.2
2	12.8	10.5
3	12.1	10.1
5	13.1	10.0

* 2.5-inch diameter Buchaner vacuum filter; 2 minute drying cycle time; cake thickness 0.4-0.5 in.; dens medium sample floated using 1 lb/ton kerosene and 100 g/ton MIBC; and solid content of sample 16.1%. [1] The esterified RG1 obtained using 1 mole RG1, 3 mole ARY16 and 5% by volume acetic acid. RG1 and esterified RG1 dissolved 33.3% in diesel before use.

Table 37 Effects of Esterified1 RY15 and RY10 on Dewatering of Middle Fork Coal Sample* (1 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	RY15	RY10
0	19.7	19.7
1	13.3	12.4
2	11.4	11.2
3	10.4	9.4
5	9.8	9.0
7	8.0	7.9

* 2.5-inch diameter Buchaner vacuum filter; 2 minute drying cycle time; cake thickness 0.41 in.; dens medium sample floated using 1 lb/ton kerosene and 100 g/ton MIBC; and solid content of sample 15.9%. [1] The esterified reagents obtained using 1 mole reagent, 3 mole butonal and 5% by volume acetic acid. The products dissolved 33.3% in diesel before use.

Table 38 Effects of Esterified1 RY16 and RY10 on Dewatering of Middle Fork Coal Sample* (0.6 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	RY16	RY10
0	25.1	25.1
1	19.8	17.3
2	17.2	15.9
3	15.6	15.7
5	15.0	14.3

* 2 minute drying cycle time; cake thickness 0.41 in.; dens medium sample floated using 1 lb/ton kerosene and 100 g/ton MIBC; solid content of sample 15.9%. [1] The esterified reagents obtained using 1 mole active ingredient, 3 mole butanol and 2% by volume acetic acid. The products dissolved 33.3% in diesel before use.

b) Synthesis of RW Reagents

Eight series of tests were conducted to assess the effectiveness of RW type of reagents for dewatering coal. All reagents were dissolved to 33.3% active in diesel, unless otherwise noted. The coal sample of dense medium products was taken from Moss #3 Plant, Pittston Middlefork Mine. All samples were pulverized, ground, and floated with a 300 g/ton dose of kerosene and 150 g/ton dose of MIBC. The floated coal slurry containing 17 to 18% solid were used for dewatering test under the conditions: 20 inches Hg vacuum pressure, 2 minutes dry cycle time, 5 minutes conditioning time, and 100 g slurry added to a flask for conditioning.

Figures 13 and Figure 14 present initial comparative moisture content results for a series of lab-grade reagents at 3 lbs/ton dosage. RW3 and RW8 were identified as the promising reagents when compared with 01DU133, which was used as a standard

reagent for the screening tests. Similarly, Figure 15 shows the test results on a series of commercial-grade reagents. RW16, RW20 and RW21 gave better results than 01DU133. These promising reagents are subjected to further testing to determine the optimal dosage for coal dewatering.

Results given in figures 16 through 20 suggest that RW3 and RW21 are more effective dewatering aids than 01DU133 at all dosages. RW16, RW20 and RW25 are superior dewatering aids to 01DU133 when using dosages of 3lbs/ton or more. 01DU133 is a synthetic RW-type reagent, and therefore more expensive than RW3 and all other reagents tested in this work, which are naturally derived. There is a great potential for using these new reagents for coal dewatering.

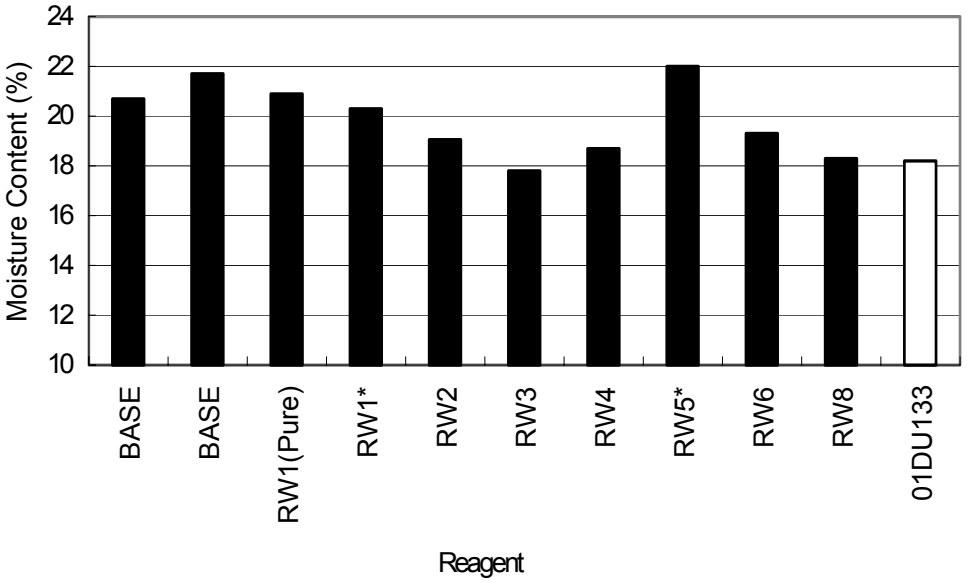

Figure 13 Moisture content results for dewatering tests using 3 lb/ton dosage of laboratory reagents with Middlefork coal (gringing 3 minutes). *dissolved to 33.3% active in butanol; other reagents dissolved to 33.3% active in diesel.

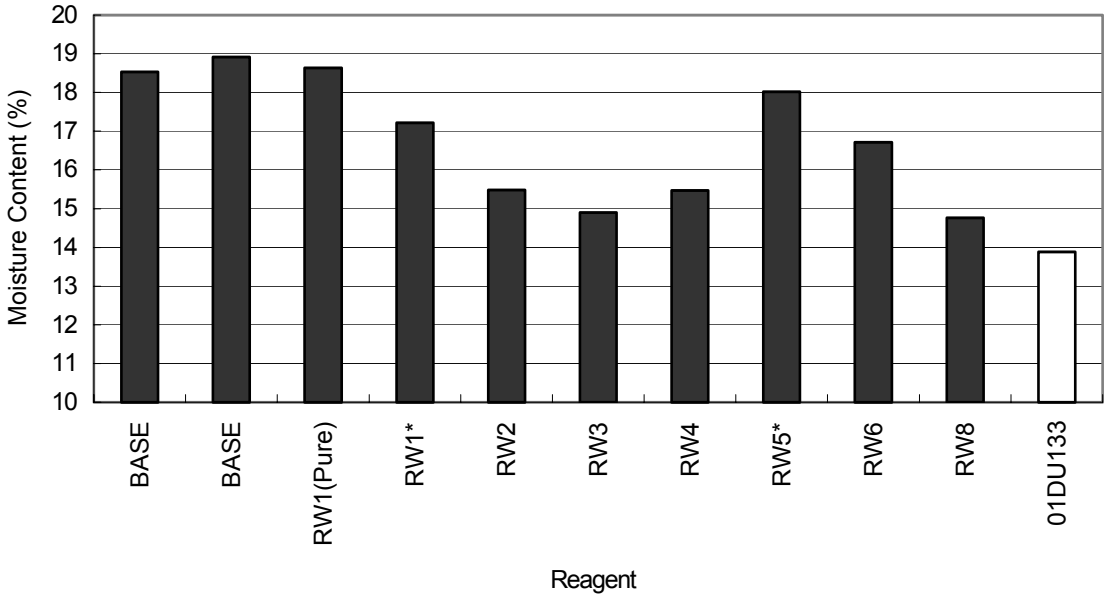

Figure 14 Moisture content results for dewatering tests using 3 lb/ton dosage of laboratory reagents with Middlefork coal (gringing 5 minutes). *dissolved to 33.3% active in butanol; other reagents dissolved to 33.3% active in diesel.

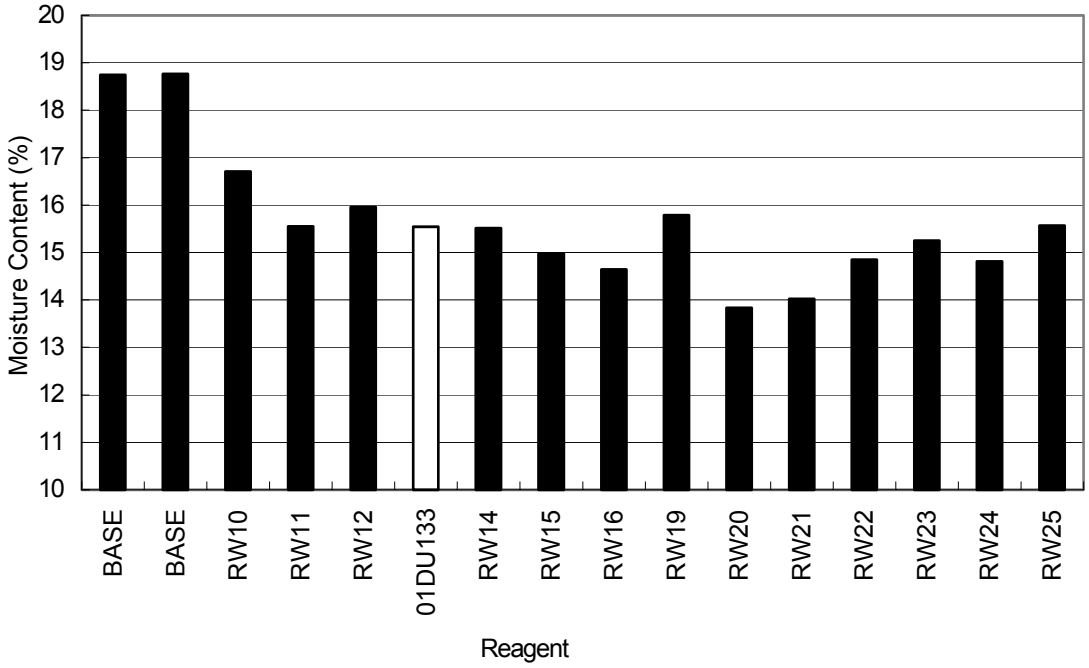

Figure 15 Moisture content results for dewatering test series III using a 3lb/ton dosage of commercial reagents with Middlefork coal (grinding 5 minutes). Reagents dissolved to 33.3% active in diesel.

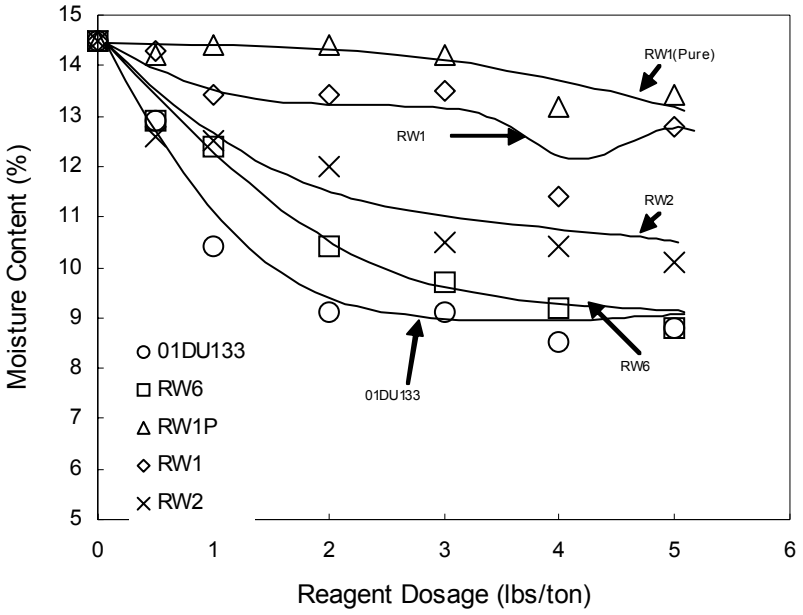

Figure 16 Plot of results showing moisture content vs. reagent dosage (lbs/ton) for dewatering tests using RW1, RW1 (pure), RW2, RW6, and 01DU133 as dewatering aids on Middlefork coal.

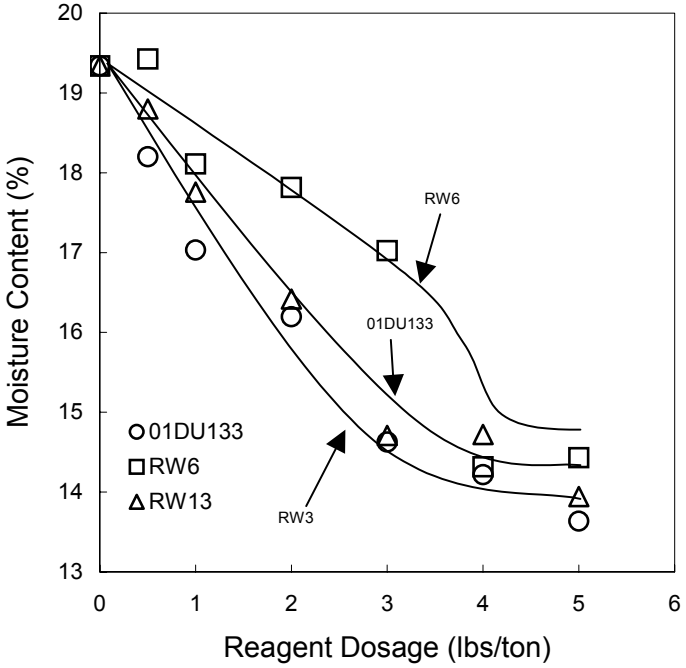

Figure 17 Plot of results showing moisture content vs. reagent dosage (lbs/ton) for dewatering tests using RW3, RW6, and 01DU133 as dewatering aids on Middlefork coal.

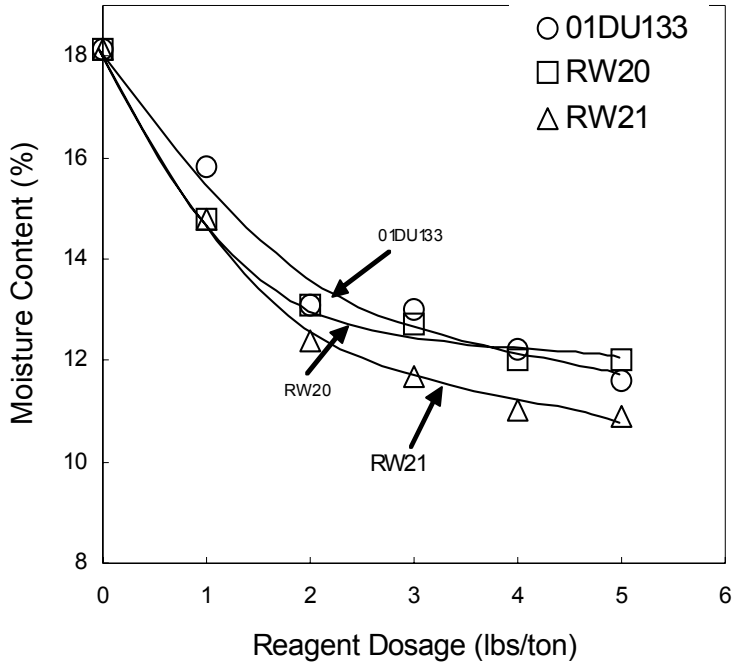

Figure 18 Plot of results showing moisture content vs. reagent dosage (lbs/ton) for dewatering test series VII using 01DU133, RW20, and RW21 as dewatering aids on Middlefork coal.

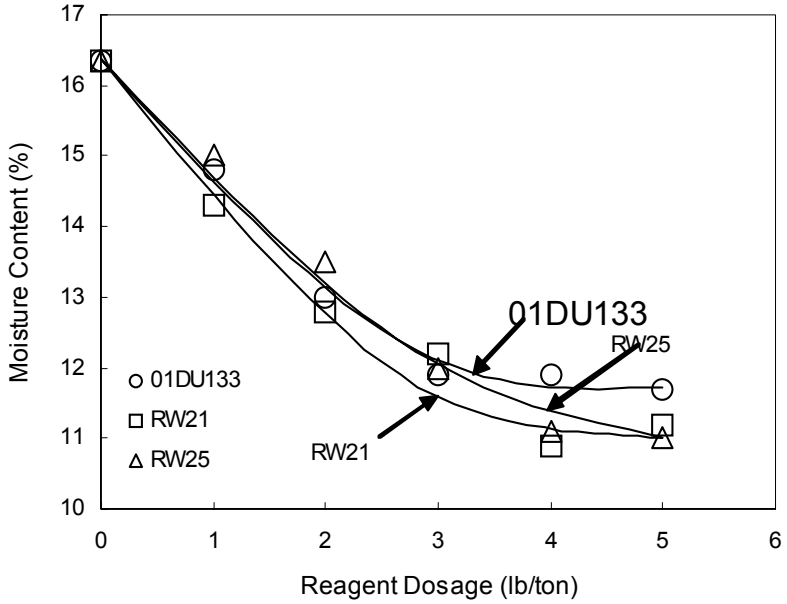

Figure 19 Plot of results showing moisture content vs. reagent dosage (lbs/ton) for dewatering tests using 01DU133, RW21, and RW25 as dewatering aids on Middlefork coal (grinding 2.5 mintues).

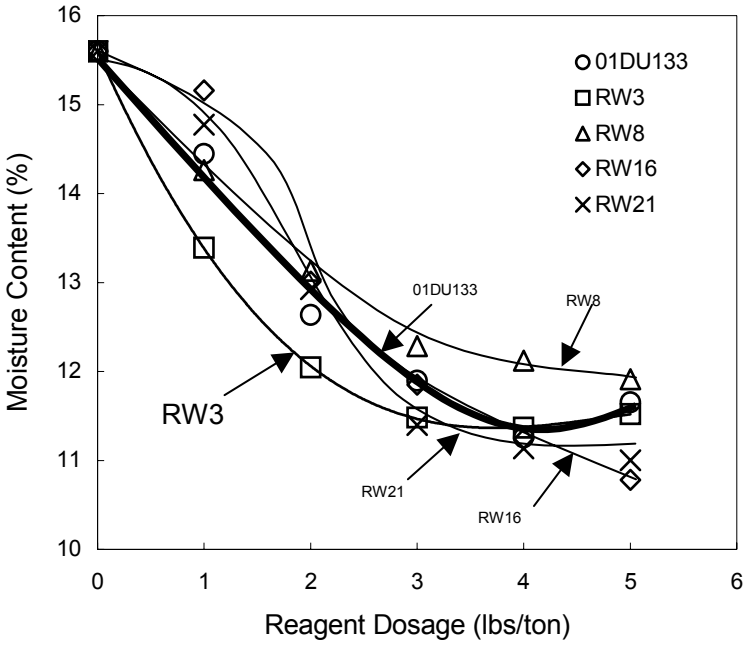

Figure 20 Plot of results showing moisture content vs. reagent dosage (lbs/ton) for dewatering tests using RW3, RW8, RW16, RW21, and 01DU133 (bold Line) as dewatering aids on Middlefork coal (grinding 2.5 minutes)

Subtask 1.4 Developing Carrier Agents

Dewatering aids tested in the present project are mostly insoluble in water. Previous work has determined that, carrier solvents are keys for the desirable dewatering performance of these reagents. Thus, it is necessary to dissolve the dewatering aids in an appropriate solvent for better reagent adhesion to the fine particle surfaces. In this subtask, multiple carrier solvents are tested for effectiveness of dewatering fine coal particles. This subtask will determine the optimum solvent for these reagents that is both inexpensive and readily available in the market.

In the first two sets of tests, 01DW110, an environmentally friendly product, was utilized as a solvent combined with and without diesel for dissolving reagents 01DU133 and 01DW145. Tables 39 and 40 show the dewatering results conducted on Bailey–Pittsburgh coal samples using 01DU133 and 01DW145 that were dissolved in 01DW110 and diesel. The coal slurry was diluted to 18.3% solids by adding fresh water. Reagent dosages shown in the tables refer to the active ingredients only.

In the base tests, the cake moisture content was 30.7% (given in Table 39). At 3 lb/ton of 01DU133 that was dissolved in 01DW110 (33.3% active), moisture content was reduced from 30.7 to 18.6%. When 01DU133 was dissolved in 01DW110 and diesel together, the moisture content became 16.1%. However, the moisture content of the filter cake went down to 15.4% when diesel alone was used as the solvent, which

Table 39 Dewatering Results of Bailey-CONSOL Filter Feed Coal Samples (-0.6 mm) Using 01DU133 Dissolved 33.3% in 01DW110, Diesel and Combination of Them (1:1:1) at 25 Inches of Hg Vacuum Pressure

Reagent Dosages (lbs/ton)	Moisture Content (%wt)		
	01DU133+01DW110	01DU133+ 01DW110 +Diesel	01DU133+ Diesel
0	30.7	30.7	30.7
1	19.4	18.9	17.7
2	19.0	17.8	16.8
3	18.6	16.1	15.4
5	20.2	17.3	16.5

*2.5 in diameter new Buchner filter used; the filter feed sample diluted to 18.3% by adding fresh water; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minute.

Table 40 Dewatering Results of Bailey-CONSOL Filter Feed Coal Samples (28 Mesh X 0) Using 01DW145 Dissolved 33.3% in 01DW110, 01DW145+01DW110+Diesel (1:1:1) and 01DW145 +Diesel at 25 Inches of Hg Vacuum Pressure

Reagent Dosages (lbs/ton)	Moisture Content (%wt)		
	01DW145 + 01DW110	01DW145 + 01DW110 + Diesel	01DW145 + Diesel
0	31.1	31.1	31.1
1	21.9	20.0	19.0
2	21.2	18.2	17.1
3	20.9	16.6	15.9
5	22.0	16.8	14.6

*2.5 in diameter new Buchner filter used; the filter feed sample diluted to 18.3% by adding fresh water; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minute.

represents approximately 50% moisture reductions. In addition to these, it is found that at 3 lb/ton of reagent addition cake formation times were decreased from 51 sec to 12 sec, which will greatly increase the throughput of the filters in plants. Similar dewatering tests results were obtained by using 01DW145 dissolved in 01DW110 and diesel as seen in Table 40.

Another test investigated the effects of napthol and diesel as carrier solvents for the dewatering aid 01DW145. Table 41 shows effects of reagent dosage and solvent type on the dewatering of Pittsburgh dense medium coal. The ratio between reagent 01DW145 and solvents was 1:2 by volume. Moisture reductions for both solvents are very similar for dosages of 2lbs/ton and greater. The slight difference between the carrier solvents may be due to the selectivity or chain length of the solvent.

Table 41 Effects of Solvent on Dewatering of Pittsburgh Dense Medium Coal Sample (0.42 mm X 0) Using 01DW145 at 25 Inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DW145 (33.3% in Napthol)	01DW145 (33.3%in Diesel)
0	16.8	16.8
1	14.4	13.6
2	11.8	11.3
3	9.6	8.8
5	8.8	8.1

*2.5 in diameter new Buchner filter used; the dense medium coal sample crushed, ground and floated by using kerosene and MIBC; solid content of sample 17.8%; cake thickness 0.4 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

Table 42 shows the effects of the carrier agents ARY15, diesel, and their mixture (1:1). ROE1 was dissolved in these solvents at 1:2 ratio by volume. Dewatering results obtained with Virginia Crews coal samples are given in Table 42 and show that diesel was the more effective solvent than ARY15. The cake moisture was reduced from 21.5% to 9.6% at 5 lbs/ton dose of ROE1.

Table 42 Effects of Solvents on Dewatering* of Virginia Crews Coal Samples (0.6 mm X 0) Using ROE1 at 20 Inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)		
	ARY15	ARY15+ diesel (1:1)	Diesel
0	21.5	21.5	21.5
1	16.3	16.6	16.6
2	15.2	13.2	13.0
3	14.0	11.9	10.2
5	13.2	10.9	9.6

*2.5 in diameter Buchner filter used; the sample was floated using 100 g/ton kerosene and 120 g/ton MIBC; solid content of sample 17.8; cake thickness 0.4 in.; conditioning time 2 minutes; drying cycle time 2 minutes; ROE1 dissolved 33.3% in the solvents.

Table 43 Solvent Effects on Dewatering Results of Pittsburgh Coal Sample (0.42 mm X 0) Using 01DW145 (1:2 In Solvent) at 25 Inches of Hg Vacuum Pressure

Reagent Addition (lb/ton)	Moisture Content (% wt.)			
	01DW145 1:2 in Butanol	01DW145 1:2 in ARY15	01DW145 1:2 in Montanol	01DW145 1:2 in Diesel
0	29.4	29.4	29.4	29.4
1	23.2	23.1	23.8	22.21
2	20.2	18.7	18.5	18.0
3	19.0	17.9	17.1	16.2
5	18.3	16.4	15.8	14.6

*2.5 in diameter Buchner filter used; solid content 17.4%; conditioning time 2 minutes; drying cycle time 2 minutes; cake thickness 0.4 in.

Table 43 shows the comparison of dewatering performance when butanol, ARY15, montanol, and diesel were used as the solvents of reagent 01DW145. As shown in Table 43, diesel once again produced the lowest cake moisture (14.6%) when 5lb/ton 01DW145 used.

Table 44 Solvent Effects on Dewatering of Pittsburgh Dense Medium Coal Sample (0.42 mm x 0) Using 01DW145 80 Dissolved 33.3% in ARY15 and ARY14 at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DW145 (33.3% in ARY15)	01DW145 (33.3%in ARY14)
0	28.5	28.5
1	23.1	23.4
2	18.4	18.2
3	17.9	17.2
5	17.3	16.8

*2.5 in diameter new Buchner filter used; the dense medium coal sample crushed, ground and floated by using kerosene and MIBC; solid content of sample 17.2%; cake thickness 0.4 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

In the last set of the tests, 01DW145 was dissolved in ARY15 and montanol to a 1:2 volume ratio. The effects of using ARY15 and montanol as dewatering aid solvents on dewatering performance are shown in Table 44. Overall, both solvents were effective for water removal although diesel was also better than ARY15.

Subtask 1.5 Developing Regent Blends

It was found that using a mixture of pure reagents and naturally occurring reagents showed synergistic effects on dewatering of fine coal particles. Different coal samples require different reagent blends due to the variance in surface characteristics of coal samples. The purpose of this subtask is to develop and test different reagent blends for their effectiveness in water removal from the surfaces of coals that have a diverse surface properties.

Table 45 shows the effects using RY2 and 01DW145 separately and their 1:1 blend with vacuum filtration tests on a Middle Fork coal sample. All reagents were

Table 45 Effects of Using a RY2-01DW145 Blend for the Filtration of a Middle Fork Bituminous Coal Sample (0.6 mm X 0) at 150 KPa Air Pressure

Reagent Addition (lb/ton)	Moisture Content (% wt)		
	RY2	01DW145	Combination
0	25.7	25.7	25.7
1	16.2	13.4	13.0
2	14.2	10.3	10.4
3	12.0	9.5	9.3
5	11.7	9.0	8.7

*2.5 in diameter Buchner filter used; the filter feed sample diluted to 16.2% by adding fresh water; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

dissolved to a 1:2 mixture in diesel. The coal sample, a filter feed/flotation product (0.6 mm x 0), was diluted to 16.2% solids by adding fresh water. Results in Table 45 show that all reagents effectively reduce cake moistures. However, 01DW145 and 1:1 blend of both reagents produced the best moisture reductions. The cake moisture was reduced from 25.7% to 11.7%, 9.0% and 8.7% at 5 lbs/ton of RY2, 01DW145, and the 1:1 blend, respectively. RY2 is a cheap, natural product. Therefore, the combined use of RY2 and 01DW145 can result in better dewatering performance at lower cost.

Another series of test compared the effects of blending RY5 and 01DW145, results of which are shown in Table 46. Similarly, RY5, 01DW145 were tested individually and as a 1:1 blend. The dewatering aids were dissolved to a 1:2 ratio in diesel. At 3 lb/ton RY5, 01DW145 and their 1:1 blend, the moisture contents were reduced from 26.1% to 13.5%, 10.2% and 10.4%, respectively. Further increase in dosages did not further reduce the cake moisture contents.

Table 47 shows the results obtained on a bituminous coal sample from Middle Fork - Virginia. The coal sample was a 0.6 mm x 0 size flotation product received as a

Table 46 Effects of Using a RY5-01DW145 Blend for the Filtration of a Middle Fork–Virginia Bituminous Coal Sample (0.6 mm X 0) at 150 kPa Air Pressure

Reagent Addition (lb/ton)	Moisture Content (% wt)		
	RY5	01DW145	Combination
0	26.1	26.1	26.1
1	17.3	14.2	15.3
2	14.9	11.3	12.4
3	13.5	10.2	10.4
5	13.2	9.6	9.2

*2.5 in diameter Buchner filter used; the filter feed sample diluted to 16.2% by adding fresh water; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

Table 47 Effects of Using a 01DW111 - 01DU133 Blend for the Filtration of a Middle Fork Bituminous Coal Sample (0.6 mm x 0) at 150 kPa Air Pressure

Reagent Addition (lb/ton)	Moisture Content (% wt)		
	01DW111	01DU133	Combination
0	27.8	27.8	27.8
1	22.8	18.7	19.2
2	19.9	15.2	16.3
3	18.3	13.5	13.7
5	19.2	12.2	11.6

*2.5 in diameter Buchner filter used; the filter feed sample diluted to 16.2% by adding fresh water; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

slurry. The sample was diluted to 16.2% solids by adding fresh water. The dewatering tests were conducted with using 01DW111, 01DU133 separately and as a 1:1 blend. A 5 lb/ton dose of the reagent blend reduced filter cake moistures form 27.8% to 11.6%, which is a greater moisture reduction than using 01DU133 or 01DW111 individually.

A similar dewatering test was conducted using RY3 and 01DU133 separately

Table 48 Effects of Using a RY3-01DU133 Blend for the Filtration of a Middle Fork-Virginia Bituminous Coal Sample (0.6 mm X 0) at 150 KPa Air Pressure

Reagent Addition (lb/ton)	Moisture Content (% wt)		
	RY3	01DU133	Combination
0	25.7	25.7	25.7
1	16.6	12.1	13.3
2	14.7	9.2	10.1
3	13.8	8.6	8.5
5	12.7	8.5	8.3

*2.5 in diameter Buchner filter used; the filter feed sample diluted to 16.2% by adding fresh water; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

and as a 1:1 ratio blend. Table 48 results show the moisture reduction of 68% when 5 lbs/ton RY3-01DU133 blend was used. The combined use of these two reagents is more effective for coal dewatering than using either of them alone.

Table 49 shows results for a test that assessed the combined use of dewatering aids RY12 and 01DW145. They are mixed at 1:1 ratio by volume and then dissolved in diesel at 33.3% concentration. Results in Table 49 show that 01DW145 and a 1:1 blend of RY12 and 01DW145 were almost equally effective. With a dosage of 3lbs/ton, the cake moisture was decreased from 16.2% to 10.4, 9.8, and 9.2% for RY12, RY12+01DW145, and 01DW145 respectively.

Results shown in Table 50 present the test result of RY13 and 01DW145. RY13 and 01DW145 reduced the cake moisture from a baseline of 16.2% to 9.4 and 7.8%, respectively, when they were used alone at a 3lb/ton reagent dosage. The moisture reduction for 01DW145 is over 50%. However, when they were used together as a 1:1 blend, the cake moisture was reduced to 8.6%. Results given in Table 50 shows the

Table 49 Effects of Reagent Blends on Dewatering* of Virginia Crews Coal Samples (0.6 mm X 0) at 25 Inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)		
	RY12	RY12+01DW145 (1:1)	01DW145
0	16.2	16.2	16.2
1	12.1	11.6	11.8
2	11.6	10.2	10.0
3	10.4	9.8	9.2
5	10.0	9.2	8.7

*2.5 in diameter Buchner filter used; the sample was floated using 100 g/ton kerosene and 120 g/ton MIBC; solid content of sample 17.8; cake thickness 0.42 in.; conditioning time 2 minutes; drying cycle time 2 minutes; the reagents dissolved 33.3% in diesel.

Table 50 Effects of Reagent Blends on Dewatering* of Virginia Crews Coal Samples (0.6 mm X 0) at 25 Inches of Hg Vacuum

Reagent Dosage (lb/ton)	Moisture Content (%wt)		
	RY13	RY13+01DW145 (1:1)	01DW145
0	16.2	16.2	16.2
1	11.9	11.6	10.7
2	10.2	9.3	9.1
3	9.4	8.6	8.1
5	9.4	8.6	7.8

*2.5 in diameter Buchner filter used; the sample was floated using 100 g/ton kerosene and 120 g/ton MIBC; solid content of sample 17.8; cake thickness 0.42 in.; conditioning time 2 minutes; drying cycle time 2 minutes; the reagents dissolved 33.3% in the solvents.

blend of RY13 and 01DW145 did not improve the dewatering performance of the reagent 01DW145.

Another test was conducted to test the effectiveness of combining the dewatering aids RY14 and 01DU133 (mixed at 1:1 ratio). Results in Table 51 show that 01DU133

Table 51 Effects of Reagent Blends on Dewatering* of Virginia Crews Coal Samples (0.6 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)		
	RY14	RY14+01DU133 (1:1)	01DU133
0	16.8	16.8	16.8
1	13.5	12.7	11.5
2	10.3	9.1	9.2
3	9.8	8.4	8.3
5	9.0	8.4	8.0

*2.5 in diameter Buchner filter used; the sample was floated using 100 g/ton kerosene and 120 g/ton MIBC; solid content of sample 17.8; cake thickness 0.42 in.; conditioning time 2 minutes; drying cycle time 2 minutes; the reagents dissolved 33.3% in diesel.

Table 52 Dewatering Results* of Virginia Crews Coal Sample (0.6 mm x 0) Using Reagents 01DU145 and 01DU133 Dissolved 33.3% in Diesel at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	01DW145	01DU133
0	19.4	19.4
1	15.4	15.7
2	14.2	14.4
3	12.1	12.9
5	10.7	11.4

*2.5 in diameter Buchner filter used; the sample was floated using 100 g/ton kerosene and 120 g/ton MIBC; solid content of sample 17.8; cake thickness 0.42 in.; conditioning time 2 minutes; drying cycle time 2 minutes; the reagents dissolved 33.3% in diesel.

and the 1:1 blend mixture of RY14 and 01DU133 are almost equally effective. With a dosage of 3lbs/ton, the cake moistures were reduced from 16.2% to 9.4, 8.6, and 8.1% for RY14, RY14+01DU133, and 01DU133, respectively.

Further testing with Virginia Crews coal compared the effectiveness of 01DW145 and 01DU133 as dewatering aids. Reagents 01DW145 and 01DU133 were dissolved to 33.3% in diesel and then tested on Virginia Crews 0.6mm x 0 coal. Test results, shown in Table 52 show that 01DW145 and 01DU133 are approximately similar in dewatering performance for this coal sample. The baseline moisture is 2.6% higher that that shown in Table 51. This is due to the surface oxidation over time while tests were conducted. For example, tests with results shown in Table 51 were completed before tests shown in Table 52 were started.

Table 53 Dewatering Results* of Virginia Crews Coal Sample (0.6 mm x 0) Using Reagents 01DW111 and 01DW145 Dissolved 33.3% in Diesel at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	01DW111	01DW145
0	14.9	14.9
1	11.7	11.2
2	10.0	9.8
3	9.4	8.8
5	8.7	8.1

*2.5 in diameter Buchner filter used; the sample was floated using 100 g/ton kerosene and 120 g/ton MIBC; solid content of sample 17.8; cake thickness 0.42 in.; conditioning time 2 minutes; drying cycle time 2 minutes; the reagents dissolved 33.3% in diesel.

Results shown in Table 53 compare the effectiveness of dewatering aids 01DW111 and 01DW145. Each reagent was tested with 0.6mm x 0 coal from Virginia Crews. The results show that 01DW145 is just slightly more effective than 01DW111 for moisture reductions. A 3lb/ton dosage of 01DW111 and 01DW145 produces cake moisture of 9.4% and 8.8%, respectively.

Table 54 Comparison of the Reagents (33.3% in diesel) Conducted on West Virginia Fine Coal Sample* (0.5 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Addition (lb/ton)	Moisture Content (% wt.)			
	Diesel	RR1	RR1A	01DU133
0	22.60	22.60	22.60	22.60
1	20.77	18.46	18.32	13.65
2	19.82	14.16	15.17	11.24
3	18.95	14.06	14.68	10.88
5	16.68	13.45	13.94	10.62

*2.5 in diameter Buchner filter used; solid content 17.3%; conditioning time 2 minutes; drying cycle time 2 minutes; Diesel used 3 times more to compare the other reagent volumes

Table 55 Dewatering Results of Buchanan Filter Feed Coal Sample* (0.6 mm x 0) Using Reagents 01DW145 and 01DU133 dissolved 33.3% in Diesel at 20 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	01DW145	01DU133
0	20.6	20.6
1	13.9	13.6
2	13.1	12.8
3	12.5	12.5
5	12.3	12.0

*2.5 in diameter Buchner filter used; solid content of sample 23.2%; cake thickness 0.6 in.; conditioning time 2 minutes; drying cycle time 2 minutes.

A comparison test between diesel, RR1, RR1A, and 01DU133 shows 01DU133 to be the superior dewatering aid on West Virginia fine coal, according to results shown in Table 54. 1 lb/ton of 01DU133 reduced the cake moisture by approximately 9%, and a further 3% decrease was obtained at 5 lbs/ton dosage.

Test results given in Table 55 correlate strongly with previous tests between 01DW145 and 01DU133. Results for both reagents are very similar for all reagent dosages. Table 55 suggests that regardless of which reagent is used, a moisture reduction of 8% can be achieved with the addition of either reagent. This allows for cost to be the determining factor for reagent choice and therefore, more economically beneficial.

Table 56 shows the dewatering results obtained from Buchanan filter feed coal samples (0.6 mm x 0) with two types of reagent pretreatment: RE1 and RA1, which are dissolved in diesel at the ratio 1:2. The cake moisture reduction was indeed very close for the two reagents at the same total dosage.

Table 56 Dewatering Results of Buchanan Filter Feed Coal Sample* (0.6 mm x 0) Using Reagents RA1 and RE1 Dissolved 33.3% in Diesel at 20 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	RE1	RA1
0	19.0	19.0
1	14.1	13.2
2	12.9	12.0
3	12.5	11.8
5	13.0	12.2

*2.5 in diameter Buchner filter used; solid content of sample 23.2%; cake thickness 0.6 in.; conditioning time 2 minutes; drying cycle time of 2 minutes.

Test results shown in Table 57 compare the dewatering effectiveness of ROE3 and ROE2 for dewatering of Buchanan filter feed coal (0.6mm x 0). Both reagents were dissolved in diesel at 1:2 ratio. As shown in Table 57, the moisture results are almost the same for both reagents at the same dosage. A 3lb/ton of reagent lowers cake moisture by approximately 9% from the base. Table 58 gives test results of the comparison between ROE1, ROE2, ROE3, and 01DW145. For these tests, all

Table 57 Dewatering Results of Buchanan Filter Feed Coal Sample* (0.6 mm x 0) Using Reagents ROE2 and ROE3 Dissolved 33.3% in Diesel at 20 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	ROE3	ROE2
0	21.1	21.1
1	13.6	13.9
2	12.2	12.6
3	11.5	12.3
5	11.7	11.9

*2.5 in diameter Buchner filter used; solid content of sample diluted from 23.2 to 16.9% adding plant water; cake thickness 0.45 in.; conditioning time 2 minutes; drying cycle time 2 minutes.

Table 58 Dewatering Results of Consol Buchanan Filter Feed Coal Sample (0.6 mm x 0) Using Reagents ROE1, ROE2, ROE3 and 01DW145 (33.3% in diesel) at 20 inches of Hg Vacuum Pressure

Reagent Addition (lb./ton)	Moisture Content (% wt.)			
	ROE1	ROE2	ROE3	01DW145
0	19.2	19.2	19.2	19.2
1	12.1	12.1	12.0	11.7
2	11.2	11.0	11.3	11.2
3	11.2	11.3	10.4	10.5
5	11.4	11.5	10.8	10.6

*2.5 in diameter Buchner filter used; solid content 17.4%; conditioning time 2 minutes; drying cycle time 2 minutes; the first three reagents esterified using ARY16 and acetic acid on a hotplate.

dewatering aids were dissolved in diesel at 1:2 ratio. All these reagents are almost equally effective for the dewatering performance on this coal sample.

Most of the dewatering tests performed in the foregoing section were conducted mostly on the bituminous coal samples. Another bituminous coal sample obtained form Massy–West Virginia was tested for dewatering tests. The filter feed sample was diluted

Table 59 Effects of Using a 01DW111-RG1 Blend for the Filtration of a Massy-West Virginia Coal Sample (0.6 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Addition (lb/ton)	Moisture Content (% wt)		
	01DW111	RG1	Combination
0	24.4	24.4	24.4
1	19.3	19.8	19.3
2	18.7	15.8	15.6
3	17.2	13.4	12.1
5	17.6	12.3	11.1

*2.5 in diameter Buchner filter used; the filter feed sample diluted to 16.9% by adding fresh water; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

using fresh water. The dewatering tests were carried out using 01DW111, RG1 and blend of these two reagents. As shown in Table 59, at 5lb/ton 01DW111, RG1 and blend of these two, moisture contents of the cakes were reduced from 24.4% to 17.6%, 12.3% and 11.1%, respectively. This is apparent evidence that the combination of two reagents performed better than 01DW111 and RG1 itself.

In the last set of blending tests, the same Massy – West Virginia filter feed coal sample was used for dewatering tests. In these tests, 01DW111, RA1 and combination of two were used to compare the blending effect. The test results were given in Table 60. The moisture reduction of the blend reagent was as high as RA1 alone. It was also seen that the kinetics of using the reagents were 3 to 5 times higher as compared to the baseline tests. The tests results indicted that the use of a blending reagent is a solution to decreasing the reagent cost for industrial applications.

It is seen that the most important element of improving the dewatering of fine particles is the hydrophobicity enhancement of particles in the second step. According

Table 60 Effects of Using a 01DW111-RA1 Blend for the Filtration of a Massy-West Virginia Coal Sample (0.6 mm x 0) at 25 inches of Hg Vacuum Pressure

Reagent Addition (lb/ton)	Moisture Content (% wt)		
	01DW111	RA1	Combination
0	24.1	24.1	24.1
1	20.0	17.2	17.8
2	18.0	16.2	16.7
3	16.3	14.6	14.2
5	16.5	13.5	13.4

*2.5 in diameter Buchner filter used; the filter feed sample diluted to 16.9% by adding fresh water; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

to Eq. [5], a relatively small increment in hydrophobicity can bring a large decrease in capillary pressure (p) and, hence, a large decrease in cake moisture. The initial hydrophobization step obtained during the flotation step (or naturally hydrophobic surface) may be neglected for high moisture reductions from the filter cake. However, second step will substantially give higher moisture reduction because of the increase in the contact angle (or hydrophobicity) of the coal surface.

In addition, the increased hydrophobicity of the fine particles causes fine particles to coagulate, which is referred to as hydrophobic coagulation. The phenomena causes an increase in *r* and, hence, a decrease in capillary pressure *p*. Also, the dewatering aids cause a decrease in surface tension γ_{23} of the liquid. The carrier solvent used in these reagents can also decrease the surface tension. As a result, the approach taken in the present work was designed to meet all three of the criteria (contact angle, surface tension and capillary radii) for the improved dewatering performance.

Subtask 1.6 Effects of Conditioning

a) Coal Samples

Reagent conditioning and agitation intensity greatly play important role in fine particle dewatering. Bourgeois (1995) reported that proper agitation of slurry could easily increase the rate of filtration three times when using flocculants. However, excessively long conditioning times reduce filtration rates. To find out the effect of conditioning time, a series of dewatering tests were conducted on a sample of filter feed coal (28 mesh x 0) from Australia that was diluted to a solid content of 16.3% with Blacksburg tap water. The tests used a dosage of 3 lbs/ton 01DW145 and were

Table 61 Effects of Conditioning on Dewatering* of Australia Filter Feed Coal Samples (28 mesh X 0) Using 01DW145 at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Conditioning Times (sec)	Moisture Content (%wt)	
		Shaker (weak)	Blender (Strong)
Base	0	22.3	22.3
3 lb/ton 01DW145	0	18.3	18.2
	30	16.6	14.2
	60	14.9	13.0
	120	13.8	12.6

*2.5 in diameter new Buchner filter used; the filter feed sample diluted to from 24.7% to 16.3% by adding fresh water; cake thickness 0.5 inch; drying cycle time 2 minutes.

conditioned for different lengths of time and varying intensity; the results of which are shown in table 61.

As seen from Table 61, the moisture contents of the base tests were 22.3% for both the shaker and blender (3000 rpm). When a dosage of 3 lbs/ton 01DW145 was added without conditioning, the moisture content decreased to approximately 18%. With increased conditioning time, moisture content continually decreased down to 14.9% and 13.0% for weak and strong conditioning at 1 minutes of conditioning time, respectively. An approximately 2% difference in moisture was observed between the shaker and blender, possibly because of the increased adsorption efficiency of the reagent at high conditioning intensity.

Tests of drying cycle time were conducted on filter feed coal sample (28 mesh x 0) from Buchanan Coal in Virginia to determine the optimum dry cycle time and reagent dosage when using 01DW145 as the dewatering aid. For comparison, 1 and 2 minute drying cycle times were applied to the slurry samples. Results of the drying cycle times

are given in Table 62. Results show the moisture reduction to be best when drying cycle times were extended from 1 to 2 minutes regardless of reagent dosage.

In previous tests, we have been using MCT chemicals after dissolved in appropriate carrier solvents to improve the absorption effects on the fine particles. In the next set of tests, however, 01DW145 was emulsified using 99.6% nanopure water and 0.4% 01DW145, and then the emulsified reagents were compared with pure 01DW145 and 01DW145 dissolved in diesel. The comparison tests were conducted on Buchanan filter feed coal samples (28 mesh x 0) with a Buchner funnel filter. Reagents were conditioned in a blender at 3000 rpm speed. As shown in Table 63, at 3000 g/ton of pure 01DW145, emulsified 01DW145 and 01DW145 solution (dissolved 33.3% in diesel); the cake moisture contents were decreased from 22.1% to 15.3%, 14.4% and 13.1%, respectively. This indicates that at high speed conditioning pure reagent and emulsified reagent can also work in the absence of diesel addition, which can decrease the reagent cost to the consumers.

Table 62 Effects of Drying Cycle Time on Dewatering* of Buchanan Filter Feed Coal Samples (28 mesh x 0) Using 01DW145 at 20 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	1 Minute	2 Minutes
0	20.4	17.9
1	14.9	13.2
2	14.1	12.3
3	13.5	12.3
5	13.3	12.1

*2.5 in diameter new Buchner filter used; solid content 23.1%; cake thickness 0.5 inch; 2 minutes conditioning time.

Table 63 Effects of High Speed Conditioning of Pure 01DW145, Emulsified 01DW145 and 01DW145 Dissolved 33.3% in Diesel Conducted on Buchanan Filter Feed Coal Sample (28 mesh x 0) at 20 inches of Hg Vacuum Pressure

Reagent Addition (g/ton)	Moisture Content (% wt)		
	Pure 01DW145	Emulsified 01DW145	01DW145 (33.3% in diesle)
0	22.1	22.1	22.1
500	18.3	17.4	17.9
1000	17.9	16.7	17.1
2000	17.2	15.9	14.5
3000	15.3	14.4	13.1

*2.5 in diameter new Buchner filter used; the filter feed sample diluted to 16.2% by adding fresh water; cake thickness 0.5 inch; blender conditioning time 2 minutes; drying cycle time 2 minute; emulsification of 01DW145 was made of using 99.6% water and 0.4% reagent.

Similar tests were conducted with reagent RA1, and the test results are given in Table 64. It is seen that high-speed conditionings help improve the dewatering efficiency of pure RA1 and emulsified RA1. Thus, this means that high speed conditioning is one of the main options for better dewatering of fine particles in plant operation.

In the last set of high-speed agitation tests, 01DW145 was dissolved in diesel at concentration of 33.3% and 50.0%. The test results given in Table 65 showed that at high speed conditioning dewatering results for both treatments were very identical to each other. This indicates that high speed conditioning can decrease the amount of career solvents that we have been using in the present project.

Table 64 Effects of High Speed Conditioning of Pure RA1, Emulsified RA1 and RA1 Dissolved 33.3% in Diesel Conducted on Buchanan Filter Feed Coal Sample (28 mesh X 0) at 20 Inches of Hg Vacuum Pressure

Reagent Addition (g/ton)	Moisture Content (% wt)		
	Pure RA1	Emulsified RA1	RA1 (33.3% in Diesel)
0	22.0	22.0	22.0
500	17.8	16.9	17.1
1000	16.8	16.1	16.4
2000	16.2	15.4	14.3
3000	15.1	14.1	13.4

*2.5 in diameter new Buchner filter used; the filter feed sample diluted to 16.2% by adding fresh water; cake thickness 0.5 inch; blender conditioning time 2 minutes; drying cycle time 2 minute; emulsification of RA1 was made of using 99.6% water and 0.4% reagent.

Table 65 Effects of High Speed Conditioning Time of 01DW145 Dissolved 33.3% and 50.0% in Diesel Conducted on Buchanan Filter Feed Coal Samples* (28 mesh X 0) at 20 Inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DW145 33.3% in Diesel	01DW145 50.0% in Diesel
0	18.6	18.6
1	13.1	12.4
2	11.7	11.5
3	10.7	10.8
5	11.1	10.9

*2.5 in diameter new Buchner filter used; solid content 17.24%; cake thickness 0.5 inch; 2 minutes conditioning time; 2 minutes drying cycle time.

b) Mineral Samples

The copper sample (0.150 mm x 0) used for dewatering tests was a flotation product received from Sweden. It was determined that the sample was superficially oxidized during shipment. To obtain a fresh surface, the sample was wet-ground in a ball mill and re-floated using 50g/ton KAX and 50 g/ton MIBC at pH 10.5 (adjusted with lime). The flotation product containing 25.2% solid was subjected to vacuum filtration tests at 25-inches of Hg and 2 minutes of drying cycle time. RA1 was chosen as a dewatering aid for the copper (chalcopyrite) concentrate sample. The test results obtained at different slurry temperatures (20, 40, and 60°C) are given in Table 8.

As seen, the moisture contents of the base tests were 9.0, 8.3 and 8.0% at 20, 40, and 60°C, respectively. At 3 lb/ton of RA1, the moisture contents were decreased to 5.1, 4.3, and 4.0%. Also, reagent additions above 3 lb/ton did not significantly improve the moisture reduction, which may be the existence of the pendular moisture levels in the cake. It was also seen that dewatering kinetics were improved by 2-3 times at higher temperature. This may be because of the lower viscosity of the process water at higher temperature. Consequently, this is clear evidence that slurry temperature is one of the important parameters to decrease the moisture content of the filter cakes.

In the next set of tests, RE1 was dissolved to 33.3% in diesel and used for dewatering of the same copper concentrates. The dewatering tests were conducted at different slurry temperatures. The test results are given in Table 66. It is shown that the results are similar to those obtained in the previous tests. As a result, it can be concluded that thermal driers may be eliminated to achieve such low moisture contents of filter cakes in the plants.

Table 66 Effects of Temperatures on the Dewatering* of Boliden-Copper Concentrate Using RE1 Dissolved to 33.3% in Diesel at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb/ton)	Moisture Content (%) Temperature (°C)		
	20	40	60
0	10.1	8.2	7.9
1	5.7	5.5	5.5
2	5.1	4.2	4.0
3	5.0	4.1	4.0
5	4.8	4.3	4.1

* 2.5 in diameter vacuum filter used; the company sample treated using 50 g/ton KAX and 50 g/ton MIBC; cake thickness 0.4 in.; particle size 0.150 mm x 0; solid content 25.2%; drying cycle time 2 minutes.

Subtask 1.7 Effects of Reagent Spray

From the Laplace equation (Eq. 9), it is understood that decreasing surface tension is a useful option for decreasing capillary pressure and therefore, would be an effective method for enhancing dewatering kinetics and moisture reduction efficiency. In filtration, the bulk of the water in the feed stream is easily removed at the beginning of a filtration process. However, the trapped water in the filter cake capillaries is difficult to remove. It is known that the surface tensions of water and ARY16 are 72.8 mN/m and 22.8 mN/m at 20^{o}C, respectively. Therefore, the spray of ARY16 to the filter cake should help to further lower the surface tension of the water remaining in filter cake and therefore result in reduced cake moistures. Therefore, more effective moisture reductions could be achieved by adding surface tension lowering reagents during the drying cycle time. Water trapped in filter cake capillaries would then be removed more efficiently during this process. For this reason, a series of tests were conducted to

demonstrate the effects of adding different surface tension lowering reagents to the filter cake by aerosol spray at the end of the cake formation time (also the beginning of the drying cycle).

An East Georgia kaolin clay sample was used in the dewatering tests, of which 90% of the sample was smaller than 2 μm. Initially, the kaolin clay was bleached and pretreated using sodium hydrosulfite, alum, and NALCO 9765 at a pH of 2.8 (sulfuric acid added). The clay sample was then conditioned with 1000 g/ton dodecylammonium hydrochloride and 120 g/ton MIBC at a pH of 9.3 (adjusted with lime addition) to induce a slight surface hydrophobicity, referred to as the first hydrophobization step. Next, the bleached samples were conditioned with a dosage of 01DW145 for 5 minutes and then subjected to pressure filtration using a 2.5-inch diameter pressure filter at 150 kPa, with a cake thickness of 0.1 inches, 3 minutes of drying cycle time, and tested for dewatering enhancement by adding ARY16 spray at the end of the cake formation. The test results

Table 67 Dewatering Results of the East Georgia Kaolin Clay Sample* Using 01DW145 (Dissolved 33.3% in Diesel) and ARY16 Spray at 150 KPa Air Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DW145	01DW145 + ARY16 Spray
0	39.7	37.2
10	36.3	34.1
20	33.1	30.9
30	32.1	29.8
50	31.3	29.7

*2.5 in diameter air pressure filter used; the clay sample was bleached using sodium hydrosulfite at pH 2.8 (sulfuric acid); alum was added as coagulant; the sample was then conditioned with 1500 g/ton of dodecyl amine; particle size 2 μm x 0; solid content of sample 6.2%; cake thickness 0.1 inch; conditioning time 5 minutes; drying cycle time 3 minutes.

obtained using 01DW145 and ARY16 spray are given in Table 67. Approximately 4 lb/ton of ARY16 was manually sprayed on the cake surface at the beginning of drying cycle time.

As shown in table 67, the spray technique further reduced the cake moisture significantly. For example, without ARY16 spray, the moisture content was 39.7%. With 4 lb/ton ARY16 sprayed onto the cake surface, the moisture was reduced to 37.2%. However, with 30 lb/ton 01DW145 added and 4 lb/ton of ARY16 sprayed, the moisture content was decreased down to 29.8%. Furthermore, a dosage increase beyond 30 lbs/ton of 01DW145 did not significantly decrease the cake moisture.

In the second series of tests, the clay sample was conditioned and tested in the same manner as the first series; however, 01DU133 was used as the dewatering aid instead. Table 68 presents the results obtained by spraying approximately 4 lb/ton of ARY16 at the beginning of the 2-minute drying cycle time.

Table 68 Dewatering Results* of the East Georgia Kaolin Clay Using 01DU133 (Dissolved 33.3% in diesel) and ARY16 Spray at 150 KPa Air Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	01DU133	01DU133+ ARY16 Spray
0	39.7	37.6
10	35.0	33.2
20	33.2	31.2
30	32.0	29.9
50	30.4	29.7

*2.5 in diameter air pressure filter used; the clay sample was bleached using sodium hydrosulfite at pH 2.9 (sulfuric acid); alum was added as coagulant; the sample was then conditioned with 1500 g/ton of dodecyl amine; particle size 2 μm x 0; solid content of sample 6.2%; cake thickness 0.1 inch; conditioning time 5 minutes; drying cycle time 3 minutes.

Results given in Table 68 nearly replicate test results shown in Table 67. The results suggest that a combination of dewatering aid and ARY16 spray will produce a promising moisture reduction for clay samples.

TASK 2: CONTROL OF CAPILLARY RADII

a) Effects of Electrolyte Additions

Capillary pressure can be reduced by i) decreasing surface tension (γ), ii) increasing capillary radius (r), and iii) increasing contact angle (θ). As discussed earlier, the primary role of the formulated reagents is to increase the contact angle of particles to render the particle surface more hydrophobic. However, the novel dewatering aids also decrease surface tension and increase capillary radius. Evidence for this effect is manifested by a decrease in vacuum pressure whenever a novel dewatering aid is added to the system. The vacuum pressure decrease is a result of hydrophobic coagulation that occurs between the particles with higher contact angle. As the contact angle increases, the hydrophobic coagulation of fine particles also increases, so do the capillary radius. Thus, novel dewatering aids can increase the contact angle, decrease the surface tension, and increase the particle size (or capillary radius) simultaneously. These effects are conducive to a decrease in capillary pressure.

In addition, particle size can be enlarged by adding coagulants and flocculants, which will further decrease capillary pressure. Therefore, a series of Buchner filter tests were conducted using 01DU133 combined with inorganic electrolytes. Table 69 shows the test results performed on Pittsburgh dense medium coal samples (0.5 mm x 0) in the presence of Al^{3+} ions. In each experiment, the coal sample was conditioned with an

Table 69 Effects of Aluminum Ions on Dewatering of Pittsburgh Dense Medium Coal Samples* (28 mesh x 0) Using 01DU133 at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	01DU133	30 g/ton Al Ions+ 01DU133
0	21.4	19.8
1	17.4	14.1
2	15.1	12.4
3	13.7	11.4
5	11.9	11.3

*2.5 in diameter new Buchner filter used; the dense medium coal sample crushed and ground to –0.5 mm; Solid content 16.2%; cake thickness 0.5 inch; conditioning 2 min; drying cycle time 2 minutes.

electrolyte for 5 minutes and then conditioned with 01DU133 (dissolved in diesel at 1:2 ratio) for 2 minutes.

The results show that the use of the electrolytes substantially decrease the amount of the novel dewatering aid required for a better dewatering. For example, a 3-lb/ton dosage of reagent reduced the moisture content from 21.4 to 13.7%. However, addition of 30 g/ton Al^{3+} to the sample further decreased the cake moisture to 11.4%.

Table 70 Dewatering Results of Alabama Coal Sample (325 mesh x 0) Using 01DW145 and 500 g/ton Al ions at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	01DW145	500 g/ton Al ion + 01DW145
0	40.3	39.7
1	38.5	37.4
2	37.4	36.3
3	35.9	35.9

*2.5 in Buchner filter used; the filter feed sample floated using 100 g/ton MIBC; solid content 17.2%; cake thickness 0.5 inch; conditioning time 3 minutes; drying cycle time 2 minutes.

This indicates that adding electrolytes to the slurry is a beneficial method of reducing reagent consumption. The improved dewatering effectiveness of adding electrolytes is due to fine particle enlargement when zeta potential of the particles has been changed by electrolytes. Additionally, a slight increase in dewatering kinetics was observed with electrolyte addition.

Table 70 shows dewatering results for varying the dosage of 01DW145 with 500 g/ton $AlCl_3$. The tests were conducted on a – 325 mesh bituminous coal from Alabama. The coal sample was first floated by using 100 g/ton MIBC. Owing to the small particle size of the sample, this is the most difficult coal sample to dewater in this study. The results suggest that a 2 lb/ton dosage of 01DW145 alone will decrease the moisture content from 40.3 to 37.4%, however, a 2 lb/ton dosage of 01DW145 in combination with 500 g/ton of $AlCl_3$ will decrease the moisture content a further 1% to 36.3%.

b) Effects of Vibration

The main role of the dewatering aid is to liberate the water molecules adhering to fine particles surfaces by increasing the hydrophobicity of the particle surface. However, the reagents do not aid in transporting the liberated water through a filter cake. In this study, the filter cake was subjected to a mechanical vibration during dewatering, as a means of assisting the transportation (removal) of the liberated water through the cake. The tests were conducted on a bituminous coal from the Elkview Mine in Canada. The coal sample was first floated using 1 lb/ton kerosene and 100 g/ton MIBC before the dewatering tests. Then, the floatation product was conditioned with a 2 lb/ton dosage of 01DU133 mixed with diesel at a 1:2 ratio. Table 71 presents the effects of mechanical vibration and varied drying cycle time.

Table 71 Effects of Vibration on the Dewatering* of Elkview-Canada Coal Sample (28 mesh x 0) Using 01DU133 at 25 inches of Hg Vacuum Pressure

Drying Cycle Time (min)	Cake Moisture (% wt.)			
	without 01DU133		with 2 lb/ton 01DU133	
	without Vibration	with Vibration	without Vibration	with Vibration
1	28.4	24.3	17.8	12.8
3	27.2	22.6	15.3	10.5
5	26.7	21.0	14.8	9.0
7	26.3	20.2	14.4	8.1
9	26.1	19.4	14.0	7.6

* 2.5 inch Buchner filter; dense medium sample floated using 1 lb/ton kerosene and 100 g/ton MIBC; 2 min conditioning; 0.5 in cake thickness; 17% solid content.

Table 71 suggests that, without adding 01DU133, moisture content of the filter cakes was 28.4% and 24.3% without and with vibration at 1 minute drying cycle time, respectively. When 2 lbs/ton of 01DU133 was added to the same slurry samples, the moisture content decreased to 17.8% and 12.8%. Further moisture reduction was observed with increased drying cycle time up to 9 minutes, resulting in cake moisture of 14.0% and 7.6% without and with added vibration, respectively. Thus, results suggest that vibration during dewatering can further decrease the cake moisture.

In the next series of tests, coal samples from West Virginia were dewatered with and without mechanical vibration. This coal samples were floated, conditioned, and dewatered in the same manner as the Elkview samples, but with 01DW145 as the dewatering aid instead of 01DU133. The results of these tests are given in Table 72; and approximately replicate the moisture reduction trend observed on the Elkview coal sample.

Effects of vibration on ores other than coal were also tested in the next series of

Table 72 Effects of Vibration on the Dewatering* of West Virginia Coal Sample (28 mesh x 0) Using 01DW145 at 25 inches of Hg Vacuum Pressure

Drying Cycle Time (min)	Cake Moisture (% wt.)			
	without 01DW145		with 2 lb/ton 01DW145	
	without Vibration	with Vibration	without Vibration	with Vibration
1	25.5	19.2	26.4	21.7
3	15.2	10.3	17.7	12.1
5	12.3	8.5	16.5	10.3
7	12.2	6.4	15.6	9.2
9	11.5	5.5	15.2	8.5

* 2.5 inch Buchner filter; dense medium sample floated using 1 lb/ton kerosene and 100 g/ton MIBC; 2 min conditioning; 0.5 in cake thickness; 17.4% solid content.

tests with copper samples (0.150 mm x 0) received from Sweden. The copper sample was refreshed by wet grinding in a ball mill and re-floated using 50 g/ton KAX and 50 g/ton MIBC at a pH of 10.5 (adjusted using lime). The resulting flotation product had a solids content of 25.4% and was dewatered with vacuum filtration using RA1 and RE1 as the dewatering aids at 25-inches of Hg and varied drying cycle time. The results of these tests are given in Tables 73 and 74. The baseline moisture was approximately 10% without vibration or reagent addition. With a reagent dosage of 2 lbs/ton and a drying cycle time of 9 minutes combined with vibration, the moisture content was decreased to below 3%. Note, results not shown in Tables 73 and 74 suggest diminishing results for, reagent additions greater than 3 lb/ton, probably because of pendular moisture levels in the cake. The results are comparable with the dewatering results in plant where a dry cake was produced using a high-pressure filter and thermal drying.

Consequently, it was further demonstrated that dewatering effectiveness was

Table 73 Effects of Vibration on the Dewatering* of Sweden Copper Samples (0.105 mm x 0) Using RA1 at 25 inches of Hg Vacuum Pressure

Drying Cycle Time (min)	Cake Moisture (% wt.)			
	Without RA1		with 2 lb/ton RA1	
	Without Vibration	with Vibration	Without Vibration	with Vibration
3	10.3	7.6	6.2	4.2
5	10.3	7.3	5.3	3.7
7	9.0	7.0	4.4	3.5
9	8.4	6.9	4.5	2.6

* 2.5 in diameter vacuum filter used; the company sample treated using 50 g/ton KAX and 50 g/ton MIBC; size 0.150 mm x 0; cake thickness 0.5 in.; solid content 25.4%; drying cycle time 2 minutes.

Table 74 Effects of Vibration on the Dewatering* of Sweden Copper Samples (0.105 mm x 0) Using RE1 at 25 inches of Hg Vacuum Pressure

Drying Cycle Time (min)	Cake Moisture (% wt.)			
	without RE1		with 2 lb/ton RE1	
	without Vibration	with Vibration	without Vibration	with Vibration
3	10.2	7.9	5.6	5.14
5	9.9	7.3	5.6	4.4
7	9.2	6.9	5.0	3.3
9	8.5	6.2	4.3	2.9

* 2.5 in diameter vacuum filter used; the company sample treated using 50 g/ton KAX and 50 g/ton MIBC; size 0.150 mm x 0; cake thickness 0.5 in.; solid content 25.4%; drying cycle time 2 minutes.

dependent upon the particle size, particle hydrophobicity, surface tension of the water within capillary tubes, and vibration during dewatering. Test results show a more significant decrease of the cake moisture when all the methods were used in combination, than when using just one method. In these tests, the parameters of the

Laplace equation were varied to achieve lower filter cake moistures. To date, there has been no information or results of combining these dewatering parameters in a single unit. In addition, the combination of the parameters could also produce higher dewatering kinetics, when compared to conventional methods already in use. Overall, this process can potentially eliminate thermal dryers used in processing plants, therefore decrease cost, and minimize environmental impact.

c) Effects of Desliming

As discussed previously, fine particles hold more surface water because of the greater specific surface area, and according to Laplace (Eq: 9), decrease the pore size within the filter cake, resulting in the reduction of the capillary radii and possible blockage of filtration paths. The objective of this series of tests is to investigate the effect of desliming fine particles on dewatering.

Table 75 shows the dewatering results obtained using 01DW111 on Australia filter feed coal samples. The effect of desliming was tested only using a portion of the

Table 75 Desliming Effects on Dewatering of Australia Filter Feed Coal Samples Using 01DW111 at 20 inches of Hg Vacuum Pressure

Reagent Dosage (lobs/ton)	Moisture Content (%wt)	
	Reagent Addition After Desliming	Reagent Addition Before Desliming
0	21.8	21.8
1	14.6	15.7
2	12.2	13.3
3	10.6	12.2

*2.5 in diameter new Buchner filter used; the filter feed sample diluted to 16.2% by adding fresh water; 50% of –325 removed by screening; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minutes.

filter feed coal that had been deslimed by removing 50% of -325 mesh particles while another sample was tested as received. The results showed a further 1~2% moisture decrease due to desliming.

Table 76 shows more evidence on the improvement of dewatering efficiency by desliming. The moisture could be reduced to a significantly low level as more fine particles were removed from the Australia filter feed coal samples. The cake moisture was reduced from 25.3% to 16% when 5 lbs/t 01DW111 was used as dewatering aid, however, at the same dosage, the cake moisture was reduced from 12.5% to 5.4% when 100% of -325 mesh fine particles were removed.

Another test was conducted using Bailey-CONSOL dense medium coal samples, which were crushed, wet-ground and sized before the dewatering test. Three samples with different particle size distributions (1 mm x 0, 0.5 mm x 0 and 0.1 mm x 0) were prepared in such a manner and subjected to dewatering test using 01DW145 (33.3% dissolved in diesel) as dewatering aids. Test results are presented in table 77. As expected, the lowest cake moisture was obtained from the coarse sample (1 mm x 0). When 3 lbs/t reagents was used, the cake moisture was reduced from 18.4% to 9.2% for the coarse sample (1 mm x 0), representing a 50% moisture reduction, but for the fine sample (0.1 mm x 0), the moisture reduction was only 32.7% with the cake moisture reduced to 16.7% from the baseline 24.8%.

The data in Table 78 shows further improvement of moisture reduction when 50% of -38 μm size fraction was removed from each of three samples discussed above. For instance, the cake moisture was reduced from 20.8% to 8.1% for the finest sample (0.1 mm x 0) due to desliming.

Table 76 Effects of Desliming on Dewatering of Australia Filter Feed Coal Samples (28 mesh X 0) Using Reagent 01DW111 at 20 Inches of Hg Vacuum Pressure and 0.5 Cake Thickness

Reagent Dosage (lobs/ton)	Moisture Content (%wt)				
	-28 mesh	25% of –325 mesh removed	50% of –325 mesh removed	75% of –325 mesh removed	100% of –325 mesh removed
0	25.3	22.5	18.9	15.0	12.5
1	19.2	15.8	10.7	8.1	6.1
2	17.0	14.1	8.7	6.3	5.6
3	15.7	12.6	8.1	6.1	5.0
5	16.0	13.5	8.5	6.3	5.4

*2.5 in diameter new Buchner filter used; solid content of sample reduced to 17.6% by adding fresh water; the fine particles were removed from the filter feed by screening; conditioning time 2 minutes; drying cycle time 2 minutes.

Table 77 Effects of Particle Size on Dewatering* of Bailey-CONSOL Dense Medium Coal Samples Using 01DW145 Dissolved 33.3% in Diesel at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb./ton)	Moisture Content (%wt)		
	1 mm x 0	0.5 mm x 0	0.1 mm x 0
0	18.4	20.9	24.8
1	11.1	13.5	19.3
2	9.5	11.7	17.1
3	9.2	10.9	16.7
5	10.0	11.2	15.8

*2.5 in diameter new Buchner filter used; the coal samples crushed, wet-ground and sized; solid contents of samples 18%; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minute.

Table 78 Effects of Desliming on Dewatering* of Bailey-CONSOL Dense Medium Coal Samples Using 01DW145 Dissolved 33.3% in Diesel at 25 inches of Hg Vacuum Pressure

Reagent Dosages (lb./ton)	Moisture Content (%wt)		
	Deslimed - 1 mm	Deslimed -0.5 mm	Deslimed -0.1 mm
0	15.1	17.8	20.8
1	8.2	9.3	12.6
2	6.0	6.6	9.2
3	5.3	5.2	8.4
5	5.7	5.6	8.1

*2.5 in diameter new Buchner filter used; the coal samples crushed, wet-ground and sized; in the desliming, 50% of – 38 micron size fraction removed from the samples; solid contents of samples 18%; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minute.

d) Effects of Cake Pressing

Similar to vibration, cake pressing may assist the transportation of the liberated water in the cake. As shown in Table 79, the cake moisture may be further reduced by 2~6% if the cake was pressed after 1 minute of drying cycle time. The total drying cycle time was 2 minutes. The test was conducted on an extremely fine Alabama coal samples (-325 mesh), which was first floated using 100 g/ton kerosene and 100 g/ton MIBC, and then filtered using 01DW111 as a dewatering aids. When 5 lbs/ton reagents were used, the cake moisture was reduced to 36.1% without cake pressing, but down to 30.3% if cake pressing was applied in the midway of the drying cycle.

Table 79 Cake Pressing Effects on Dewatering of Alabama Coal Samples (-325 mesh) Using Reagent 01DW111 at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	without Cake Pressing	with Cake Pressing
0	40.1	38.4
1	38.8	35.1
2	37.7	33.6
3	37.0	31.6
5	36.1	30.3

*2.5 in diameter new Buchner filter used; the fine coal sample floated using 100 g/ton kerosene and 100 g/ton MIBC; solid content 10.7%; cake thickness 0.45 inch; conditioning time 2 minutes; drying cycle time 2 minutes; the cake was pressed after 1 minute of drying cycle time.

Table 80 gives another example of cake pressing effects on dewatering. The test was conducted on an RAPOCA composite coal sample (-28 mesh) using 01DW145 as a dewatering aids at 25 inches of Hg vacuum pressure. The cake moisture was reduced from 25.8% to 17.4% with 5 lbs/ton 01DW145 used. On contrast, the cake moisture

Table 80 Cake Pressing Effects on Dewatering of RAPOCA Composite Coal Samples (-28 mesh) Using 01DW145 at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	without Cake Pressing	with Cake Pressing
0	25.8	24.0
1	21.1	18.2
2	19.2	17.0
3	18.1	15.0
5	17.4	14.1

*2.5 in diameter new Buchner filter used; the fine coal sample floated using 200 g/ton kerosene and 100 g/ton MIBC; solid content 16.7%; cake thickness 0.5 inch; conditioning time 2 minutes; drying cycle time 2 minutes; the cake was pressed after 1 minute of drying cycle time.

decreased from 24% to 14.1% under the same dosage of reagents if the cake was pressed after 1 minutes of drying cycle time.

e) Effects of New Additional Reagents

Most dewatering aids developed in the present project are not water soluble. In order to improve the dispersion of the reagent in the feed slurry, the reagent was mechanically emulsified before it was added to the slurry. A milky reagent emulsion was obtained by mixing 0.4 parts of the reagent and 99.6 parts of water at very high speed of agitation for 2 hours.

Table 81 shows the dewatering results obtained from Buchanan filter feed coal samples (0.6 mm x 0) with two types of reagent pretreatment: pure 01DW145 emulsified in water as described above; and 01DW145 dissolved in diesel at the ratio 1:2. The cake moisture reduction was indeed very close for two treatments of the reagent at the same active dosage as shown in Table 81. However, if diesel, also a dewatering aid to

Table 81 Emulsion Effect of 01DW145 on Dewatering* of Buchanan Filter Feed Coal Samples (0.6 mm x 0) at 20 inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	moisture Content (%wt)	
	Pure 01DW145 Emulsified	01DW145 33.3% in Diesel
0	20.8	20.8
1	15.3	14.5
2	13.5	13.7
3	13.2	13.3
5	14.8	13.1

*2.5 in diameter Buchner filter used; the filter feed coal sample tested; solid content of sample 16.2%; cake thickness 0.5 in.; conditioning time 2 minutes; drying cycle time 2 minutes; emulsification of 01DW145 was made of using 99.6% nanopure water and 0.4% the reagent after 2 hrs of mixing.

Table 82 Emulsion Effect of RA1 on Dewatering* of Buchanan Filter Feed Coal Samples (0.6 mm x 0) at 20 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	Pure RA1 Emulsified	RA1 33.3% in Diesel
0	20.5	20.5
1	15.6	15.2
2	14.6	14.0
3	13.5	13.4
5	14.1	12.9

*2.5 in diameter Buchner filter used; the filter feed coal sample tested; solid content of sample 16.2%; cake thickness 0.5 in.; conditioning time 2 minutes; drying cycle time 2 minutes; emulsification of RA1 was made of using 99.6% nanopure water and 0.4% the reagent after 2 hrs of mixing.

some extent, was taken into consideration, the emulsified reagent may be more effective in term of total reagent dosage for achieving certain moisture reduction.

Table 82 shows the similar results obtained using RA1 as a dewatering aid on the same coal samples. Reagent-solvent blend somewhat outperformed the reagent-water emulsion under the same active dosage of the reagent. However, Table 83 shows that pure 01DW145 gave the higher moisture reduction than 01DW145 emulsion, indicating inadequate evidence to verify the effectiveness of reagent emulsification. Table 84 reveals the similar results obtained using RA1. The cake moisture was reduced to 15.1%, 14.1% and 13.4% for RA1 emulsion, pure RA1 and 33.3% RA1 dissolved in diesel, respectively.

Table 83 Effects of High Speed Conditioning of Pure 01DW145, Emulsified 01DW145 and 01DW145 33.3% in Diesel on Buchanan-NALCO Filter Feed Coal Sample (0.6 mm X 0) at 20 Inches of Hg Vacuum Pressure

Reagent Dosage (g/ton)	Moisture Content (%wt)		
	Pure 01DW145 Emulsified	Pure 01DW145	01DW145 33.3% in Diesel
0	20.1	20.1	20.1
500	18.3	17.4	17.9
1000	17.9	16.7	17.1
2000	17.2	15.9	14.5
3000	15.3	14.4	13.1

*2.5 in diameter Buchner filter used; the filter feed coal sample tested; solid content of sample 16.2%; cake thickness 0.5 in.; conditioning time 2 minutes; drying cycle time 2 minutes; emulsification of 01DW145 was made of using 99.6% nanopure water and 0.4% the reagent after 2 hrs of mixing.

Table 85 shows the dewatering results on Bailey dense medium coal sample (0.6 mm x 0) at 20 inches of Hg vacuum pressure. The reagents used in this test include diesel (generally used as a solvent in the present work), pure ROE1, EOE1 dissolved in diesel at the ratio 1:2, and esterified ROE1 that was also dissolved in diesel at the ratio 1:2. The moisture reduction was 27.1%, 30.9%, 42.6% and 47.9% for these reagents or reagent blends in the order described above. The optimal reagent blend is esterified ROE1 that dissolved in diesel.

Table 84 Effects of High Speed Conditioning of Pure RA1, Emulsified RA1 and RA1 33.3% in Diesel on Buchanan-NALCO Filter Feed Coal Sample (0.6 mm X 0) at 20 inches of Hg Vacuum Pressure

Reagent Dosage (g/ton)	Moisture Content (%wt)		
	Pure RA1 Emulsified	Pure RA1	RA1 33.3% in Diesel
0	20.0	20.0	20.0
500	17.8	17.0	17.1
1000	16.8	16.1	16.4
2000	16.2	15.4	14.4
3000	15.1	14.1	13.4

*2.5 in diameter Buchner filter used; the filter feed coal sample tested; solid content of sample 16.2%; cake thickness 0.5 in.; conditioning time 2 minutes; drying cycle time 2 minutes; emulsification of RA1 was made of using 99.6% nanopure water and 0.4% the reagent after 2 hrs of mixing.

Table 85 Dewatering Results of Diesel, ROE1 and Esterified ROE1 on Bailey Dense Medium Coal Sample (0.6 mm x 0) at 20 inches of Hg Vacuum Pressure

Reagent Addition (lb./ton)	Moisture Content (% wt.)			
	Diesel	Pure ROE1	ROE1 (33.3% in Diesel)	Esterified ROE1 (33.3% in Diesel)
0	18.8	18.8	18.8	18.8
1	16.9	16.1	14.6	14.0
2	15.1	14.1	12.4	11.8
3	14.7	13.3	11.3	10.6
5	13.7	13.0	10.8	9.8

*2.5 in diameter Buchner filter used; solid content 16.7%; conditioning time 2 minutes; drying cycle time 2 minutes; cake thickness 0.4in.

f) Effects of EDTA Use

EDTA (ethylenediaminetetraacetic acid) is a chelating agent commonly used in industry to remove divalent metal ions. EDTA was added to filter feed coal (28mesh x 0)

Table 86 Dewatering Results of EDTA Conducted on Bailey-CONSOL Filter Feed Coal Sample (28 mesh x 0) at 25 inches of Hg Vacuum Pressure

EDTA Dosages (mg/lt.)	Moisture Content (%)
0	24.6
25	24.0
50	21.8
75	21.6
150	22.4
300	23.6

*2.5 in diameter new Buchner filter used; the filter feed sample diluted from 33.4% to16.3% by adding fresh water; cake thickness 0.5 inch; EDTA conditioning time 2 minutes; drying cycle time 2 minutes.

Table 87 Results of the Filtration Tests Conducted on a Bailey – CONSOL Filter Feed Coal Sample (28 mesh x 0) Using 75 mg/lt EDTA and 01DU133 at 25 inches of Hg Vacuum Pressure

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	01DU133	75 mg/lt EDTA + 01DU133
0	25.0	21.6
1	17.0	16.1
2	16.7	14.6
3	15.1	13.7
5	13.6	11.8

*2.5 in diameter new Buchner filter used; the filter feed sample diluted from 33.4% to 16.3% by adding fresh water; cake thickness 0.5 inch; EDTA conditioning 5; 01DU133 conditioning time 2 minutes; drying cycle time 2 minutes.

from Bailey-Consol. Test results of EDTA addition are shown in Table 86. As seen in Table 86, a very low dosage (75 mg/liter) of EDTA could reduce 1.5% cake moistures. Higher dose of EDTA did not cause further moisture reduction. In the next set of tests, EDTA was added together with reagent 01DW133. Results of these tests are shown in Table 87. It is seen that addition of EDTA could produce 1 to 2% lower moisture of filter cake.

g) Effect of Pressure

As noted in the Laplace equation (Eq.5), the pressure must be significant enough to overcome the surface tension of water trapped within filter cake capillaries. The addition of dewatering aids helps to lower the vacuum pressure needed for dewatering to occur. However, it must be larger than the capillary pressure.

Table 88 demonstrates the effect of vacuum pressure on the cake moisture of filter feed coal from Buchanan. Results in Table 88 suggest that vacuum pressure is a key parameter for the dewatering of fine coal. A vacuum pressure of 12 inches Hg is

Table 88 Effects of Vacuum Pressure on Dewatering of Buchanan Filter Feed Coal Sample* (0.6 mm x 0) Using 01DU133 at 0.5 inch Cake Thickness

Reagent Dosage (lb/ton)	Moisture Content (%wt)	
	12 inches of Hg	24 inches of Hg
0	24.5	18.8
1	18.4	12.5
2	16.6	11.1
3	15.2	10.3
5	14.7	9.9

*2.5 in diameter Buchner filter used; solid content of sample 17.3%; cake thickness 0.5 in.; conditioning time 2 minutes; drying cycle time 2 minutes.

sufficient to remove a portion of the interstitial water from the filter cake (15.2%) at a 3lb/ton dosage of 01DU133, but the cake moisture was reduced to 10.3% at 24 inches Hg vacuum produce,

h) Effect of Oxidation

As coal is mined from the active face, it begins to oxidize, resulting in a decrease of its natural hydrophobicity. MCT developed dewatering aid, RA1, was tested for effectiveness in compensating for coal oxidation. The filter feed coal sample from Buchanan was tested while fresh and after two weeks of aging. Results of these tests are given in Table 89. Baseline tests show 4.5% higher moisture due to coal oxidation (aging). However, with a total dosage of 5lb/ton RA1, the moisture results are quite similar, 10.5 and 10.8% for fresh and aged samples, respectively. The similarity in the results at higher dosage shows that the reagent can compensate for surface oxidation by increasing the surface hydrophobicity.

Table 89 Effects of Sample Ageing on Dewatering* of Buchanan – NALCO Filter Feed Coal Samples (-0.6 mm) Using RA1 (50% In diesel) at 20 Inches of Hg Vacuum Pressure

Reagent Dosage (lbs/ton)	Moisture Content (%wt)	
	Fresh Filter Feed	Two Weeks Old
0	16.9	21.4
1	12.9	14.8
2	11.7	12.7
3	10.5	11.4
5	10.5	10.8

*2.5 in diameter new Buchner filter used; solid content reduced to 16.0% by adding fresh water; cake thickness 0.5 inch; 2 minutes conditioning time; 2 minutes drying cycle time.

SUMMARY AND CONCLUSION

Several novel chemicals have been developed at MCT and tested on different fine mineral samples (e.g., copper, lead and zinc concentrates, talc, silica and kaolin clay) using Buchner vacuum filter and air pressure filters. The objectives of the dewatering tests were to verify the efficiency of novel dewatering aids on different hydrophobic and hydrophilic minerals.

The experimental studies conducted on copper, lead and zinc sulfide concentrates showed that MCT reagents could reduce the total moisture content of the filter cake by more than 50%, depending on the sample and dewatering conditions. It was also seen that some of the chemicals (RA1, 01DU145 and 01DU133) seemed to be slightly better than the other reagents, which may be attributed to the selectivity of these reagents on the sulfide minerals. In most of the tests, it is shown that approximately 2 lb/ton of the reagent is sufficient for obtaining satisfactory results.

Another tests conducted on Luzenac- America talc sample revealed that the use of novel dewatering aids reduced the cake moisture content by more than 12% depending on cake thickness, reagent dosage and drying time. Even at larger cake thickness, the moisture reduction was closer to 10% absolute, which means that these reagents were also effective on the thick cake dewatering applications.

The hydrophobization tests were also conducted on silica samples. When the novel dewatering aids were used directly, moisture reductions of less then 4% were obtained. When the silica samples were treated first by a cationic surfactant, followed by a novel dewatering aid, more substantial moisture reduction were achieved. The resultant filter cake would not need any further dewatering or thermal drying processes.

The two-step hydrophobization process employed for silica was also tested for the dewatering of the kaolin clay samples received from Georgia. The clay samples were hydrophobized first by dodecyl amine at pH 9.2, and then treated with the novel dewatering aids. The test results showed that kaolin can be dewatered mechanically, and thus obviate the need for thermal drying.

The beneficial effects of using MCT reagents can be attributed to the increased dewatering kinetics. A set kinetics tests showed that the rate of filtration can be increased by more than 4 times as compared to the baseline tests. The increase in

kinetics should result in increased throughput, which is a added benefit of using the novel dewatering aids.

Several novel dewatering chemicals were tested on different fine coal samples using Buchner vacuum filter and air pressure filter. The test results showed that the moisture contents of the filter cake were more than 10% lower than the base case depending on the test conditions. This may be attributed to the surface hydrophobization of the particles as shown by the increase in water contact angle. The contact angle measurements conducted in the present work showed that the addition of novel dewatering aids significantly increased the hydrophobicity of fine particles.

Modification of naturally occurring reagents by a suitable esterification process can also be used as low-cost dewatering aids. It was found that the use of these reagents require appropriate solvents to achieve maximum efficiency.

The present investigation also included the studies of the effects of conditioning (agitation time, speed and temperature), reagent spray, and control of capillary radii (electrolytic coagulants and mechanical vibration) on the dewatering performance of the novel dewatering aids. Results show high intensity conditioning, reagent spray, addition of coagulants and mechanical vibration can further improve the moisture reduction.

Some of the reagents have been tested in full-scale operation. As a result of the successful trial, Boliden Mineral AB in Sweden is using the reagents commercially. The net results of using the MCT reagents is that costly thermal drier has been eliminated, while at the same time the throughput of the vacuum filter was increased.

REFERENCES

Adamson, A. W. and Gast, A. P. “Physical Chemistry of Surfaces – sixth addition,” John Wiley & Sons, Inc, New York, 1997.

Basilio, C. I. “Recent Advances in the Processing of Kaolin,” Thiele Kaolin Company, special presentation at Virginia Tech, 1997.

Bourgeois, Proc. 7th Australian Coal Preparation Conference, pp.177, 1995.

Carman, P.C., “Fluid Flow through Granular Beds,” Trans. Ins. Chem. Eng., Vol. 15., pp. 150-166, 1937.

Darcy, H.P.G. “Les Fontains Publique de la Ville de Dijon,’ Victor Dalmont, Paris, 1856.

Gray, V.R., “The Dewatering of Fine Coal,” J. Inst. Fuel, Vol. 31, p.96-108, 1958.

Kozney, J. “Uber Kapillare Leiling des Wassers in Boden,” Wein Akad. Wiss. Sitz. Berichte, Vol. 136 (IIa), pp.271-306, 1927.

Leja, J., “Surface Chemistry of Forth Flotation`, University of British Columbia, Vancouver- Canada, 1982.

Meenan, “Proceedings of the Industrial Practice of Fine Coal Processing”, Society of Mining Engineering, 1988, p.223-229.

Myers, D., Surfactant Science and Technology, VCH Publisher, 1988.

Tiller, F.M. “Unifying the Theory of Thickening, Filtration and Centrifugation,” University of Houston, Texas-USA, 1996.

van Oss, C.J., Geise, R.F., Li, Z., Murphy, K., Norris, J., Chaudhury, M.K. and Good, R.J., “Determination of Contact Angles and Pore Sizes of Porous Media by Column and Thin Layer Wicking,” J. Adhesion Sci. Technol. Vol. 6, No.4, 413-428, 1992.

van Oss, C.J. Interfacial Forces in Aqueous Media. Marcel Dekker, inc., New York., 1994.

Yoon, R.-H., Nagaraj, D.R., Wang, S.S. and Hilderband, T.M., “Beneficiation of Kaolin Clay by Froth Flotation using Hydroxamate Collectors,” Minerals Engineering, Vol. 5, Nos.3-5, pp. 457-467, 1992.

Yordon, J.L., Kortmeyer, J., Yildirim, I. And Yoon. R.H. “Studies of the Interactions Between Talc and Stickies During Paper Recycling,” SME Annual Meeting, Feb. 25-27, Phoenix, Arizona, 2002.