

2.1. Introduction

The main purpose of conducting the vacuum filtration experiments was to test the novel dewatering aids developed at Virginia Tech (patented by Yoon and Basilio) on fine coal dewatering. In addition, the best operating conditions for the filtration operations were examined using the laboratory scale vacuum filtration technique. The tests were conducted on a large variety of coal samples. In this chapter, the experimental results related to the most important findings on the subject will be presented as a summary. After screening the efficiency of many reagents in filtration, the best performing ones were selected. These reagents decreased the base moisture content of the filter cakes in a range of 5% to 15% points. Among all the reagents tested, Ethylene Glycol Monooleate (EGMO) was found to be the most effective one to decrease the cake moisture. This particular reagent usually gave a 50% moisture reduction compared to the tests conducted in the absence of any dewatering aid. As a result, it was chosen to be used for the experiments, which were conducted to analyze the best operating conditions for the filtration tests.

There are many factors playing an important role on the performance of the vacuum filtration. To improve the dewatering efficiency, these factors must be studied closely. First of all the coal itself is very heterogeneous and as a result, the coal slurry samples show different characteristics. The properties of the sample such as the ash content of the coal, particle size distribution of the slurry and the sensitivity of the coal to oxidation must be determined. The second important factor is related to the selection of the proper dewatering aid. The effective dosage of the additive and the time required to adsorb onto the coal surface (conditioning time) are substantial variables. The operational factors can be classified as the third group, namely, the level of the vacuum pressure, cake thickness, drying cycle time and the slurry temperature

The effects of the listed variables on filtration efficiency were systematically analyzed and the results were reported in this chapter. Furthermore, a statistical analysis was performed to determine the significance of the changes in the operational factors in improving the dewatering of fine coal, quantitatively.

2.2. Sample Collection

Four different coal samples were used for the laboratory filtration tests in this chapter. All of the samples were flotation products from operating coal preparation plants. The coal samples used for the analyses in this work are listed below:

1. 100 mesh x 0 Pond Fines cleaned by flotation at the Middle Fork Preparation Plant, Pittston Coal Management Company, Virginia, U.S.A.
2. 28 mesh x 0 Pittsburgh No.8 coal which is a filter feed at a preparation plant, CONSOL Inc., U.S.A.
3. 28 mesh x 0 Feed to Disc Filters from Elkview Mining Co., British Columbia, Canada.
4. 28 mesh x 0 flotation product, BMCH Mining Company, Australia.

A coal sample slurry was prepared at the laboratory by using the Elkview run of mine coal. This sample was crushed and ground to various particle size distributions to study the effect of particle size distribution on filtration. The same sample was also floated in the laboratory and the filtration tests were conducted on it to compare the response of the floated and non-floated coal slurries to dewatering.

2.3. Dewatering Reagents

The dewatering reagents used in this work were mono unsaturated fatty esters and polysiloxane polymers. Among many reagents tested, the best performing ones were selected to be used in this study. The mainly used two reagents can be listed as follows:

Ethylene Glycol Monooleate (EGMO)

Polymethylhydrosiloxane (PMHS)

2.3.1. Experimental Set-up

Apparatus

Figure 2.1 shows the apparatus used for the laboratory vacuum filtration tests. A 94-mm diameter buchner funnel with a medium porosity (40-60 μm pore size) glass frit was used as the filter. Use of a buchner funnel for filtration test is advantageous over the standard vacuum leaf

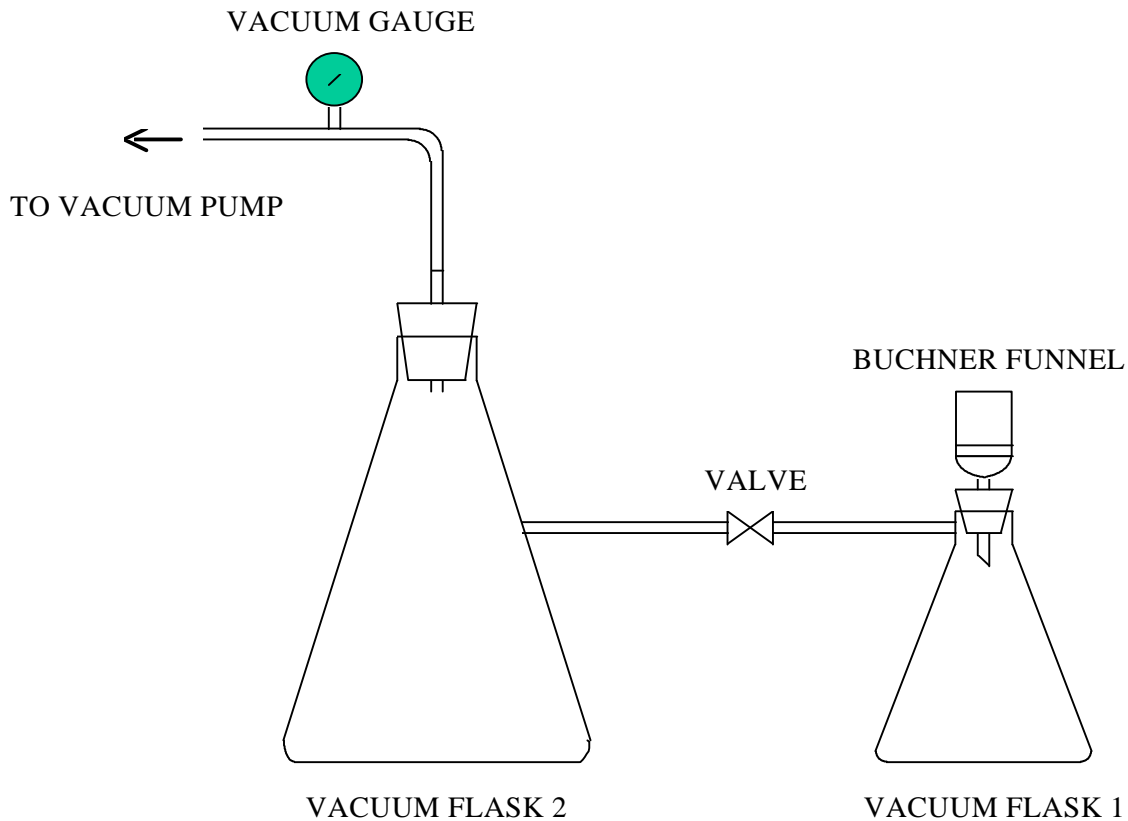


Figure 2.1. Experimental setup for laboratory vacuum filtration tests.

filter test in that the cake thickness can be controlled. The buchner funnel was mounted on a vacuum flask, which in turn was connected to a larger vacuum flask. The larger flask helped to stabilize the vacuum pressure during filtration. The coal slurry placed in the buchner funnel was subjected to a vacuum pressure when the valve between the two vacuum flasks was opened. Most of the filtration tests were conducted at 20-25 inches-Hg of initial vacuum pressure (the pressure measured before the valve was open), which decreased to 17-22 inches Hg at the end of the test.

Procedure

In most cases, coal samples were received in 5-gallon buckets as slurry. The coal sample contained in a bucket was agitated by means of a dynamic mixer to homogenize the slurry. A known volume of the slurry was removed from the bucket using a scoop, while the slurry was being agitated. The slurry was poured into a 250-ml erlenmeyer, to which a known volume of

solution containing dewatering aid was added by means of a Microliter syringe. The flask was subjected to conditioning for a pre-determined time period to allow for the reagent to adsorb on the surface of the coal particles. The coal slurry conditioned in this manner was then transferred to the buchner funnel. Filtration test was commenced when the slurry was subjected to a vacuum pressure by opening the valve between the two flasks. Bulk of the water was quickly removed, and a cake was formed on the glass frit of the buchner funnel. After the cake was formed, the vacuum pressure was kept on for a desired period of time to further drain the water from the cake. After this drying cycle time, approximately 10 grams of the filter cake was removed from the funnel, weighed, and dried for 12 hours at 170 °F. The dry coal sample was weighed again, and the moisture content was calculated from the difference between the dry and wet weights.

2.3.2. Vacuum Filtration Tests Results

The first set of experiments was conducted to screen the efficiency of the novel dewatering aids developed at Virginia Tech. Figure 2.2 shows a comparison between the best performing reagents, EGMO and PMHS. All the tests were conducted on Middle Fork coal sample slurry (100 mesh x 0, 11.29% solid). The vacuum pressure was set to 20 inches-Hg. A known amount of reagent was added in a 100-ml slurry, which was then conditioned for 1 minute by hand shaking to let the reagent adsorb onto the coal surface. This amount of sample slurry gave approximately 0.2 inches thick filter cakes. All the three reagents decreased the cake moisture to sufficiently low values. When no reagent was used, the cake moisture was 28.88% after one minute of drying cycle time. The moisture content of the cake decreased to 14.99% in the presence of 2.5 Lbs./ton of EGMO. At the same dosage and the drying cycle time PMHS, which was a polymer decreased it to 15.37%. There was an approximately 10% points reduction in the moisture contents of the cakes in the presence of the novel dewatering aids. The further increase in the reagent dosages after 2.5 Lbs./ton did not decrease the cake moisture any more. The EGMO was observed to perform slightly better than the others and it was selected to be used to complete the analyses.

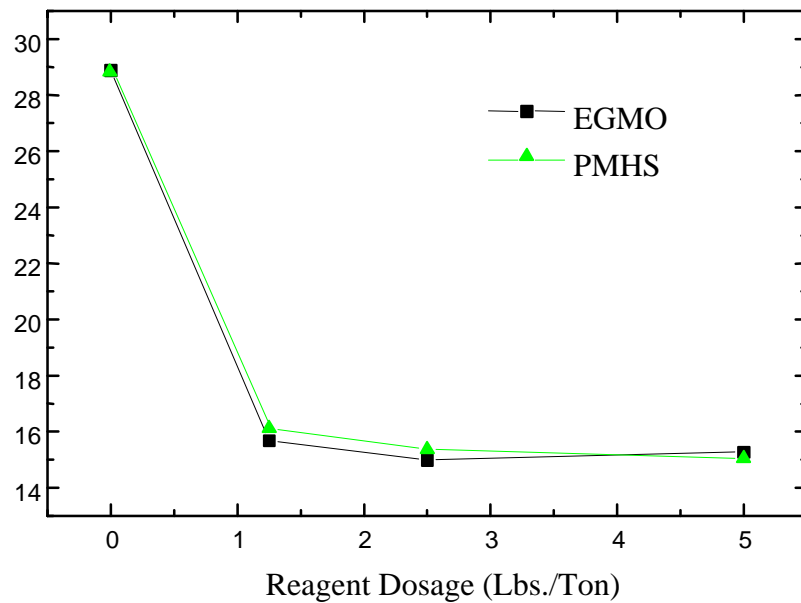


Figure 2.2. Results of the filtration tests conducted on the floated coal sample slurry (100 mesh x 0) from the Middle Fork coal preparation plant as a function of the reagent dosage (EGMO, PMHS). The tests were conducted at 0.2 inches cake thickness and 1 minute drying cycle time.

Figure 2.3 shows the relationship between the slurry pH and the cake moisture content. The pH values of the 100-ml Middle Fork sample slurries were changed in the range of pH 7.6 (natural pH of the slurry) and pH 11 using NaOH. The drying cycle time was 3 minutes and the cakes were ~0.15 inches thick. The vacuum pressure was set to 20 inches-Hg for all the conducted tests. EGMO was used to compare the effect of pH in the presence of the dewatering aid. The dosage of the reagent was kept constant at 1.25 Lbs./ton and it was added into the slurry after the pH was adjusted. The slurries were conditioned for one minute by hand shaking to let the reagent adsorb onto the coal surface. The conditioning was done even on the control tests in the absence of the dewatering aid to keep the consistency. For the base experiments, without any additives the modification in pH was observed to change the final moisture content of the filter cake. At the natural pH of the slurry (pH 7.6), the cake moisture was 23.80%. As the pH value increased, the cake moisture increased up to 28.6% at pH 9 and then a plateau was reached. The variation in the moisture content of the filter cake for the base experiments can be explained on

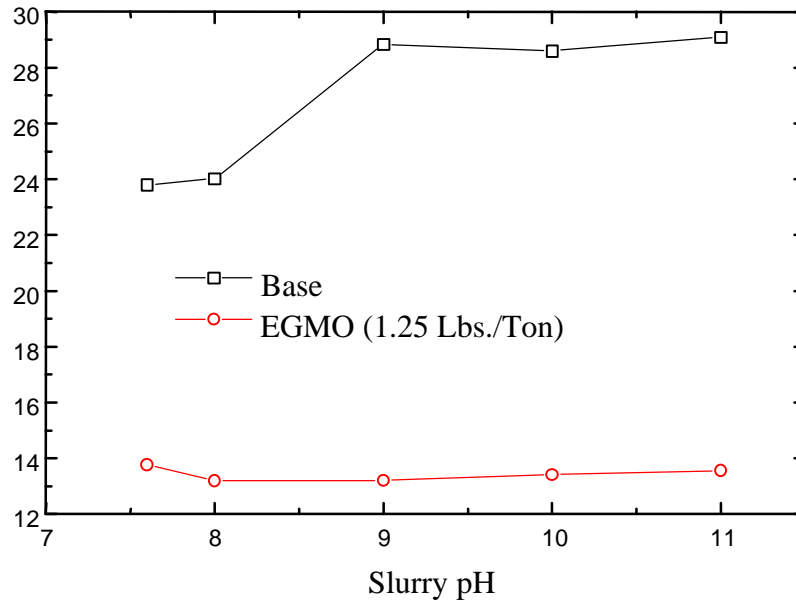


Figure 2.3. Results of the filtration tests conducted on the floatation products (100 mesh x 0) from the Middle Fork coal preparation plant as a function of the slurry pH with and without the dewatering aid (1.25 Lbs./ton of EGMO). All tests were conducted at 3 minutes of drying cycle time and 0.15 inches cake thickness.

the basis of the change in the surface charge of the coal. The coal particles are negatively charged in water due to the tendency of the OH^- ions to go onto the surface. As the pH of the slurry increases, the surface charge of the coal increases as a result of the increase in the amount of the OH^- ions in the solution. Thus, the negative charge of the coal surfaces increase with increasing pH. At high pH values all the particles tend to repel each other and disperse since they are all strongly charged. The dispersion of the particles decreases the effective radius of the capillaries formed in the filter cake structure and also more surface area of the coal is exposed to water. These result in an increase in the cake moisture content. However, in the presence of 1.25 Lbs/ton of active EGMO, the change in the slurry pH had no effect on the final cake moisture content and it remained almost constant at a value of 13%. This observation implies that the novel additive adsorbs onto the surface sufficiently and eliminates the effect of the surface charges.

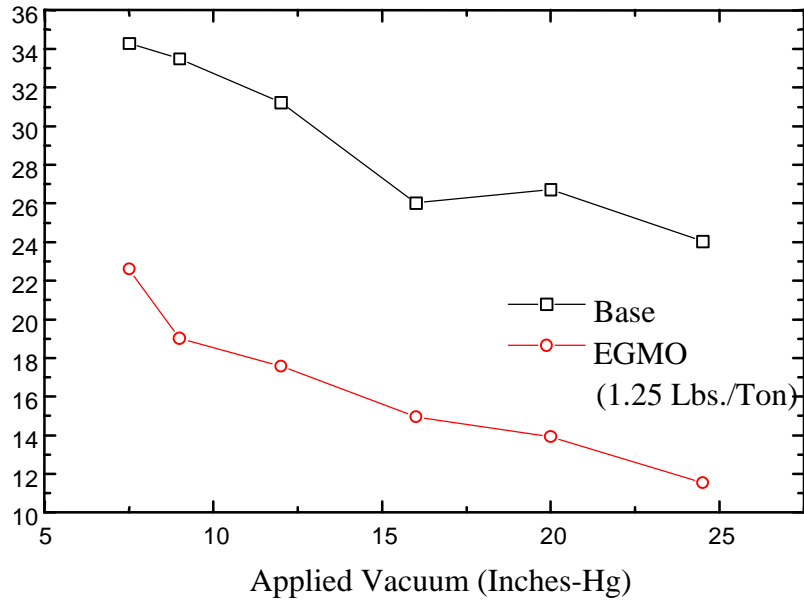


Figure 2.4. Results of the filtration tests conducted on the floatation products (100 mesh x 0) from the Middle Fork coal preparation plant as a function of the applied vacuum with and without the dewatering aid (1.25 lbs/ton of EGMO). All tests were conducted at 3 minutes of drying cycle time and 0.15 inches of cake thickness.

The level of the applied vacuum pressure is also a very important factor modifying the efficiency of the vacuum filtration. Figure 2.4 illustrates the change in the moisture content of the filter cake of the Middle Fork coal sample slurry, as a function of the applied vacuum pressure. The drying cycle time was set to 3 minutes for all the tests. The moisture content of the cake decreased as the vacuum pressure increased regardless of whether the dewatering aid was used or not. The base moisture content of the 0.15 inches thick filter cake at a very low, 7.5 inches-Hg of vacuum pressure was 34.29%. In the presence of 1.25 Lbs/ton of EGMO, the cake moisture decreased to 22.61% at this pressure level. As the vacuum pressure increased, the moisture content of the cakes for both the base tests and the tests in the presence of the EGMO decreased almost parallel. The cake moistures were about 10 to 12% lower in the presence of the dewatering aid. Finally, at 24.5 inches-Hg vacuum pressure, the base moisture content of the sample decreased to 24.04% and with the addition of the EGMO, a cake moisture content of 11.54% was reached.

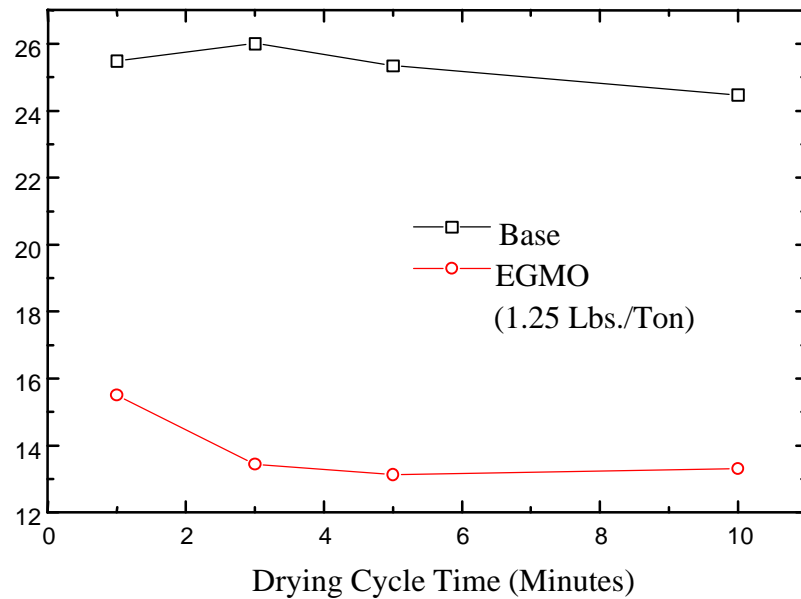


Figure 2.5. Effects of drying cycle time on the cake moisture. Filtration tests were conducted on a Microcel flotation product from the Middle Fork coal preparation plant with and without dewatering aid (1.25 lbs/ton of EGMO). The cake thickness was approximately 0.1 inches.

Figure 2.5 shows the results of the filtration tests conducted on the Middle Fork coal sample as a function of the drying cycle time. The tests were performed with and without dewatering aid (EGMO) for comparison. The cake thickness was approximately 0.1 inches for all the tests. The tests conducted using 1.25 lbs/ton of EGMO produced filter cakes with approximately 10% lower moisture contents than without using the dewatering aid. That was consistent with the results of the previous experiments. After 1-minute drying cycle time, the cake moisture values were 25.4% and 15.5% in the absence and presence of the dewatering aid, respectively. When the drying cycle time was increased to 5 minutes, the moisture content of the cake was further reduced to 13.1% with the addition of EGMO. Increase in drying cycle time beyond 5 minutes did not make a significant difference in reducing the cake moisture. In the absence of dewatering aid, however, the drying cycle time did not show any notable change.

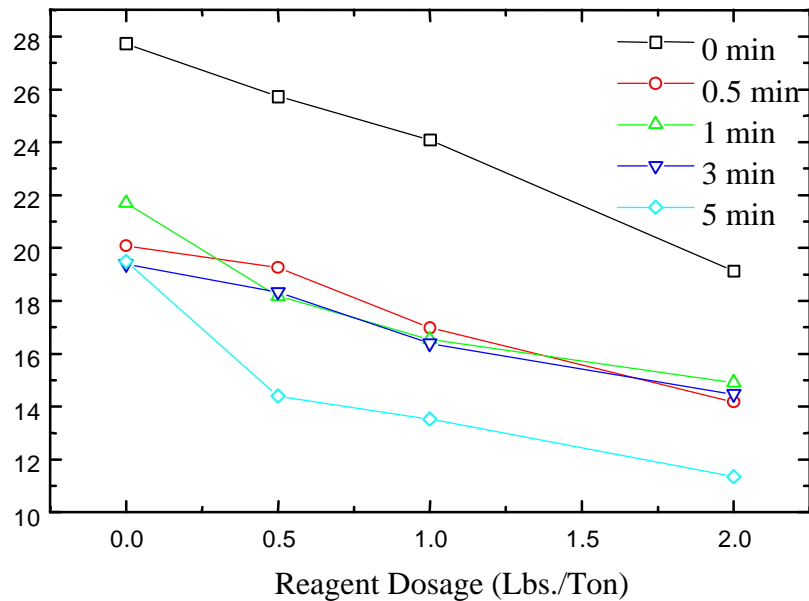


Figure 2.6. Effects of drying cycle time on cake moisture content as a function of the reagent dosage (EGMO). Filtration tests were conducted on Middle Fork coal sample (100 mesh x 0). The cake thickness was 0.2 inches for all the tests.

A more detailed set of experiments was conducted to determine the effect of drying cycle time on cake moisture by using Middle Fork sample slurry. Figure 2.6 shows the changes in moisture content of the filter cakes as a function of the reagent dosage (EGMO) at different drying cycle times. The vacuum pressure was set to 25 inches-Hg and the cake thickness was kept at 0.2 inches. The test results were consistent with the previous observations in that the longer the drying cycle time was, the lower the moisture content became. It is desirable, however, to use as short a drying cycle time as possible due to the economical concerns. When the disc filters are used, usually 1 to 2-minute of drying cycle times are employed. Relatively longer drying cycle times can be used when the horizontal belt filters are utilized.

An important indication of Figure 2.6 was that when the vacuum was cut as soon as the cake was formed (no drying cycle time), the cake moisture continued to decrease with the increasing dosage of the EGMO. The cake moisture obtained after drying cycle times of 0.5 to 3 minutes was about the same at all the used levels of the reagent. When the drying cycle time was extended to 5 minutes, the cake moisture was further reduced. These results suggest the

following; during the initial stage of dewatering, capillary water was removed quickly. At a moderate drying cycle time, funicular water was removed and at considerably longer drying cycle time periods, pendular water began to be removed.

Figure 2.7 shows the effects of cake thickness on cake moisture. A Middle Fork coal sample was used for the tests in the absence and the presence of the dewatering aid (1.25 Lbs./ton of EGMO). The vacuum pressure was set to 20 inches-Hg and the drying cycle time was fixed to 5 minutes in all experiments. The cake thickness was varied by changing the volume of the slurry in the range of 100 to 400 ml. As the cake thickness increased from 0.1 to 0.4 inches, the cake moisture increased from 13.1 to 20.5%, respectively. Thus, the advantage of using the dewatering aid diminished considerably at higher cake thicknesses. However, the cake moisture.

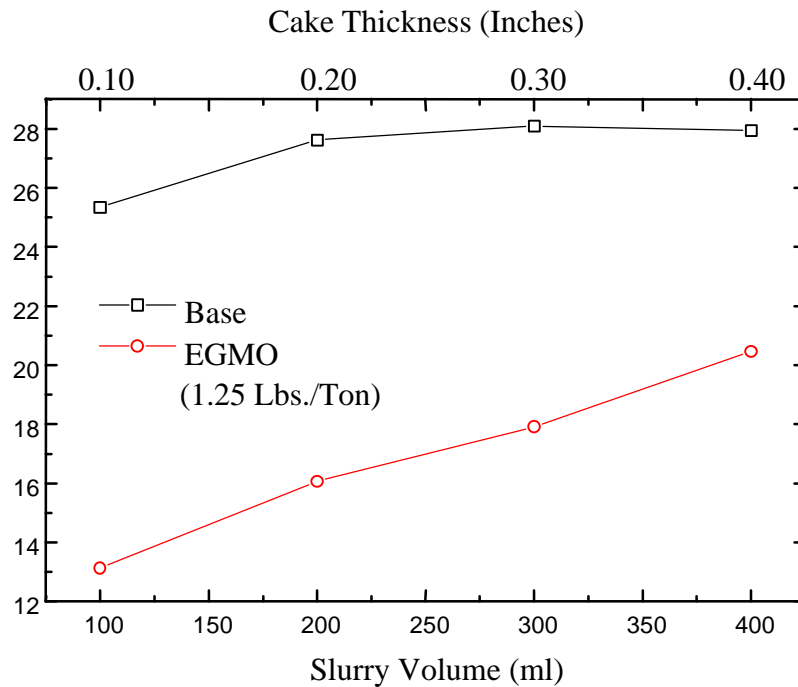


Figure 2.7. Effects of the change in cake thickness on cake moisture content. Filtration tests were conducted on the flotation products (100 mesh x 0) obtained from the Middle Fork preparation plant with and without dewatering aid (1.25 Lbs./ton of EGMO). All tests were conducted at 5 minutes of drying cycle time.

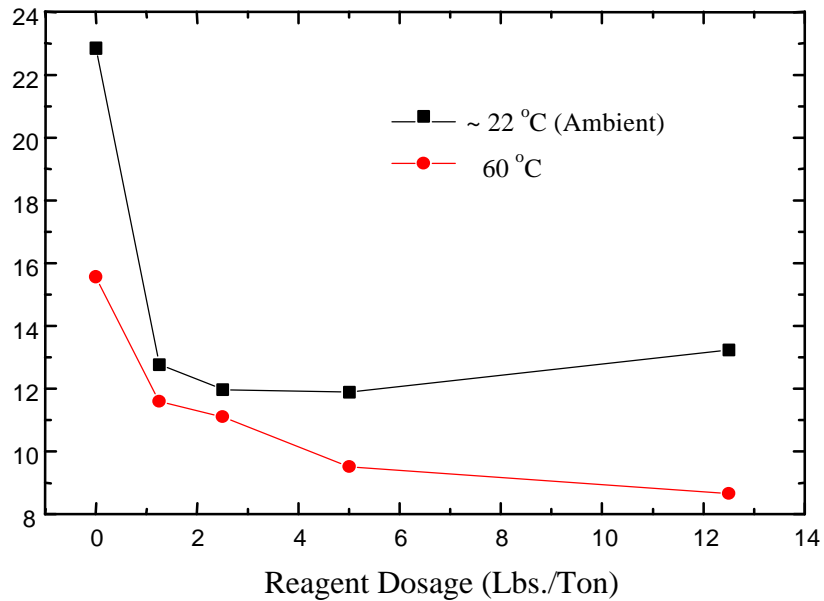


Figure 2.8. Results of the filtration tests conducted on the flotation products from the Middle Fork coal preparation plant as a function of reagent dosage (EGMO). The tests were conducted at 0.2 inches of cake thickness and at two different temperatures.

of the control tests also increased with increasing cake thickness. Therefore, the advantage of using EGMO was evident even at higher cake thicknesses. At 0.4 inches of cake thickness, for example, the cake moisture obtained with 1.25 Lbs./ton of EGMO was approximately 8% lower than the control tests. This difference may further increase at higher dosages of EGMO.

Figure 2.8 shows the results of the filtration tests conducted on the flotation products from Middle Fork coal preparation plant (10.23% solid) using varying amounts of EGMO. The best conditions determined in the previous tests were applied in this set of experiments. All of the tests were conducted using 200 ml of sample slurry, which gave approximately 0.2 inches of cake thickness. The samples were conditioned for two minutes by hand shaking. The drying cycle time was set to 5 minutes and the vacuum pressure was adjusted to 25 inches-Hg. At ambient temperature, the cake moisture obtained without using dewatering aid was 22.9%, which decreased further with increased reagent dosage, at 5 Lbs./ton of EGMO, the cake moisture became as low as 11.9%. However, there were no significant benefits of increasing reagent additions beyond 2.5 lb/ton when the coal slurry was filtered at the ambient temperature.

Also as shown in Figure 2.7 is the effect of temperature on cake moisture. The sample slurries, contained in 250-ml erlenmeyers, were heated to 60° in a water bath prior to filtration. As shown, the cake moisture decreases substantially at the higher temperature. When no dewatering aids were used, the cake moisture was only 15.6%, which was lower than the value obtained (22.9%) at the ambient temperature. The cake moisture decreased further down as EGMO was added to aid the filtration. It is interesting that the cake moisture continued to decrease with increasing reagent dosage without reaching a plateau at elevated temperature, which is different from what was observed at the ambient temperature. The beneficial effects of filtering fine coal slurry at an elevated temperature may be attributed to the reduction in the viscosity of the water trapped in the capillary formed between the particles. Apparently, there is a correlation between using EGMO and doing filtration at an elevated temperature.

In order to determine the effect of slurry temperature on the cake moisture, the EGMO was tested on a Middle Fork coal sample at three different slurry temperatures and cake thicknesses. For this set of experiments the vacuum pressure was set to 28 inches-Hg and a 5 minutes of drying cycle time was employed. The solid content of the coal slurry was 24.4 %, and 50, 100 and 200 ml of sample slurries were used to form 0.1, 0.2 and 0.4 inches thick cakes.

Cake Thickness (inches)	0.1" (50 ml sample slurry)		0.2" (100 ml sample slurry)		0.4" (200 ml sample slurry)	
	Base	EGMO (2 Lbs./ton)	Base	EGMO (2 Lbs./ton)	Base	EGMO (2 Lbs./ton)
23	13.10	4.90	17.80	11.74	18.69	18.21
60	13.36	3.50	14.37	9.27	15.46	13.08
80	13.90	2.12	14.51	7.30	15.87	13.99

Table 2.1. Results of the filtration tests conducted on Middle Fork coal sample (100 mesh x 0) using EGMO (2 lbs/ton). The tests were run at three different temperatures and cake thicknesses. The vacuum pressure was set to 28 inches-Hg and the drying cycle time was 5 minutes.

For the 0.1 inches thick cake, the base moisture content of 13.10% was obtained at the ambient temperature (23°C), which decreased to 4.90% at 2 Lbs./ton of EGMO addition. The moisture values of the control tests did not change significantly at elevated temperatures. In the presence of the dewatering aid, however, at 80°C the moisture was reduced to as low as 2.12% for 0.1 inches thick cake. As the cake thickness increased, the beneficial effect of heating the slurry diminished. At 0.1 inches of cake thickness, the moisture content decreased by 57%, while the cake moisture decreased from 4.9% at 23°C to 2.12% at 80°C. For 0.2 inches thick cake the reduction was 38% and for 0.4 inches thick cake it was only 28%. These observations indicated the decline in the effect of high slurry temperature at high cake thicknesses. As a solution, the dosage of the novel additive EGMO can be increased to reduce the moisture content of the thick cakes further down at high temperatures.

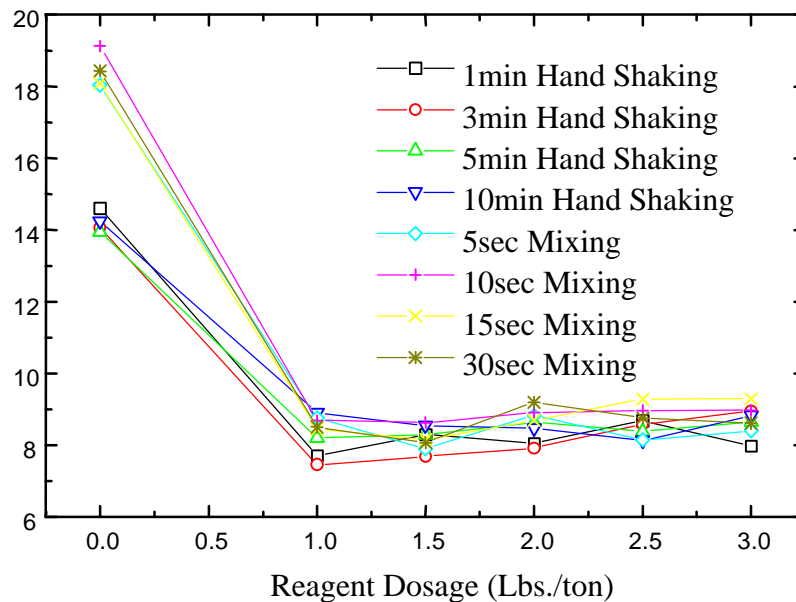


Figure 2.9. Effect of the conditioning method and time on moisture content at the varying dosages of EGMO. Tests were conducted on Middle Fork coal sample at 0.4 inches of cake thickness. The drying cycle time was 3 minutes.

Figure 2.9 represents the results of the tests conducted on the Middle Fork coal sample as a function of the reagent dosage for different conditioning times and methods. Two different methods of conditioning were tested. These included hand-shaking and mixing (by means of a blender). In each test, 100-ml coal slurries with 36.05% solid content were used. The time periods for hand-shaking were selected as 1, 3, 5 and 10 minutes. For the mixing method, shorter time periods i.e., 5, 10, 15 and 30 seconds were chosen to make the results comparable with the hand shaking. EGMO was used as the dewatering aid. The drying cycle time was 3 minutes and the cake thickness was ~0.4 inches in all the conducted tests. The base moisture contents of the samples conditioned by hand-shaking varied between 13.5- 14.5%. The control tests gave about 4-5% higher cake moisture for the samples conditioned by mixing. This increase observed in the moisture content of the results of the control tests was due to the change in the particle size distribution of the slurry as a result of the particle breakage during the high shear blending operation. However, in the presence of EGMO, the moisture content of both the mixed and the hand conditioned samples decreased sharply. At 1lbs/ton of reagent addition, a plateau was reached in all cases. For the hand conditioned samples the cake moisture contents of about 7-8% were reached and the values obtained with the mixed samples were 1-2% higher. These results indicated that the mixing method changed the particle size distribution of the sample and caused a decrease in the particle size. As a result, the final moisture contents of the samples conditioned with mixing went up. This indicates that the hand-shaking method is better than the mixing technique and even 1 minute conditioning time was sufficient enough.

Figure 2.10 shows a set of test results obtained with the flotation product (28 mesh x 0) from the Elkview Mining Company using EGMO. The tests were conducted both at the ambient temperature (~22°C) and at 60°C using 200 ml of coal slurry. The cake thicknesses measured after the drying cycle time were in the neighborhood of 0.2 inches. The sample slurries were conditioned for 1 minute by hand shaking and the drying cycle time was kept at 2 minutes to make the results comparable with the industrial applications. When the tests were conducted without using the dewatering aid, the cake moistures were 20.8 and 18.9% at 22 and 60°C, respectively. With the addition of EGMO, the cake moisture decreased substantially. The

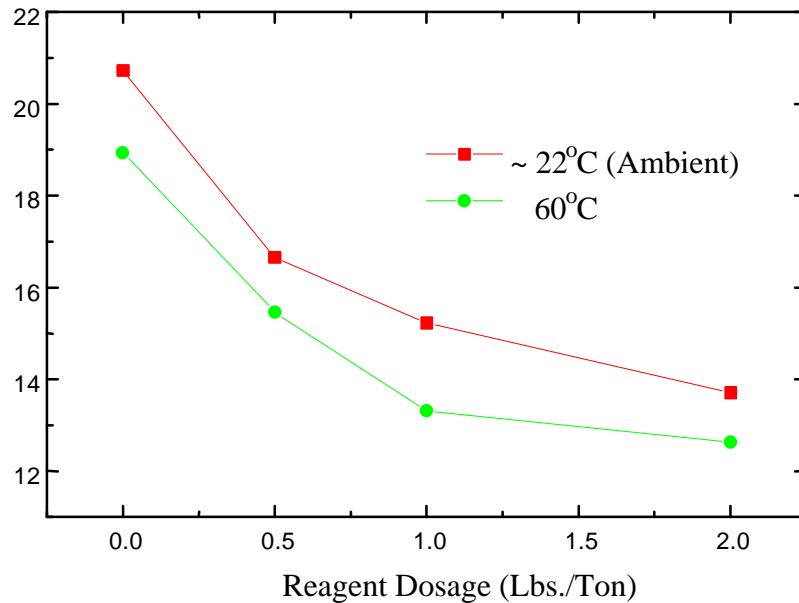


Figure 2.10. Results of the filtration tests conducted on the flotation products (28 mesh x 0) from Elkview Mining Company as a function of reagent dosage (EGMO). The tests were conducted at 0.2 inches of cake thickness and at two different temperatures.

moisture reduction increased with the increasing reagent dosage. At 2 lb/ton, the cake moisture decreased to 13.71% at 22 °C. At the elevated temperature, it was further reduced to 12.63% at the same reagent dosage. It is possible that the moisture reduction reaches a plateau at higher reagent dosages but the reagent dosages used in this set of experiment were kept low to be in the range of the industrial applicability.

In addition to reducing the final cake moisture content of the filter cakes, EGMO was observed to be capable of improving the kinetics of dewatering as well. This was observed from the results of the tests conducted on the Elkview coal sample that were shown in Figure 2.10. When no reagent was used, the cake formation time was 20 seconds at the ambient temperature and in the presence of 2 Lbs./ton of EGMO, it decreased to 7 seconds. At 60 °C, the cake formation time was 7 seconds without the reagent addition and then it reduced further down to 5 seconds at each level of the reagent addition.

Reagent Dosage (Lbs./ton)	Cake Formation Time (sec)	Product Moisture (% weight)
0	25	24.37
0.5	20	21.24
1	18	19.48
2	15	16.64

Table 2.2. Results of the filtration experiments conducted on the oxidized Elkview coal sample (28 mesh x 0). The tests were conducted at 0.2 inches of cake thickness and at 2 minutes drying cycle time.

The novel dewatering aid, EGMO was observed to be sensitive to the oxidation of the coal sample. Table 2.2 shows the results obtained after aging the Elview coal sample for four weeks at the ambient temperature before conducting the filtration tests. Although EGMO was able to reduce the cake moisture extensively below the level that can be achieved without the addition of the dewatering aid, the final cake moisture content was not as low as the values obtained when the tests were conducted soon after the sample had been received. In addition, the cake formation times increased compared to the results taken on the fresh sample. It is likely that coal particles are superficially oxidized during the process of aging, which may be detrimental to the adsorption of the dewatering aid used in the present work.

Figure 2.11 represents the effect of EGMO on moisture reduction at two different cake thicknesses and slurry temperatures. The tests were conducted on a BMCH Australian coal sample (100 x 0 mesh, 25% solid). The vacuum pressure was set to 25 inches-Hg, drying cycle time was kept at 2 minutes and all the samples were conditioned by hand shaking for 1 minute. The cake thicknesses were fairly high compared to the previous experiments conducted on the other coal samples. Figure 2.11-a shows the results of the tests conducted with 100-ml sample slurries, which gave approximately 0.25 inches thick cakes. The base moisture contents were 25.46% and 23.61% at 22°C and 60°C slurry temperatures, respectively. In the presence of 3 Lbs./ton of EGMO, these values reduced to 14.63% and 11.66%. When the same tests were performed with 0.5 inches thick cakes by using 200-ml slurries, moisture contents increased as expected. It is seen in Figure 2.11-b that, the base moisture contents raised to 28.37% and

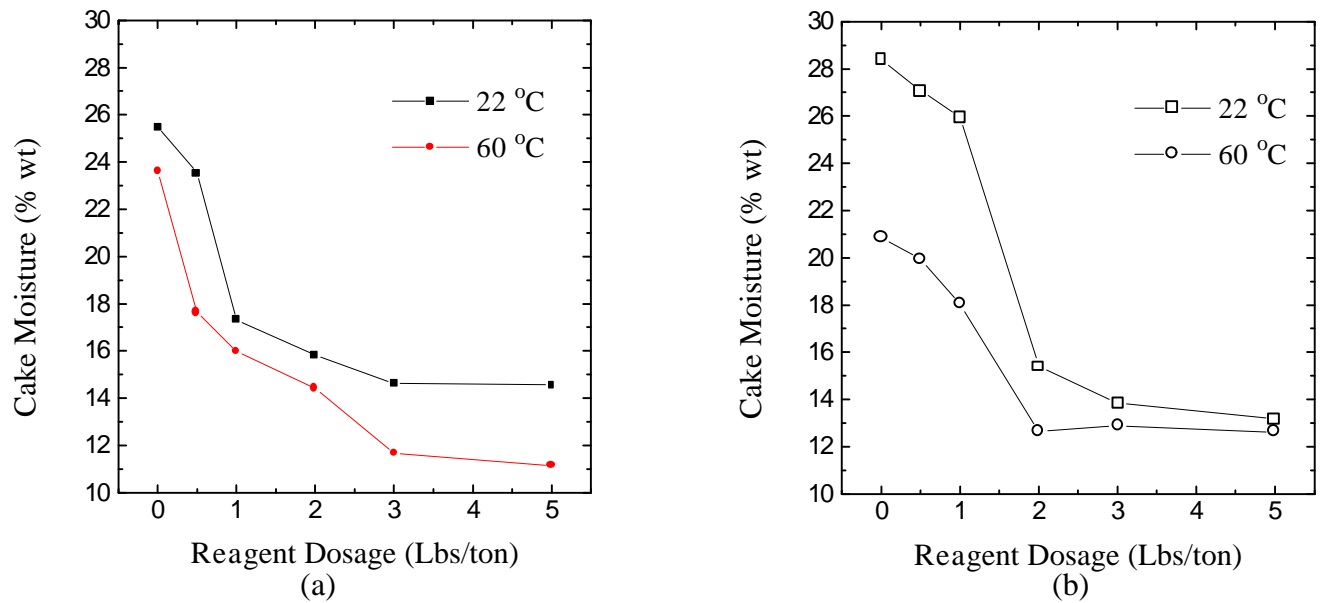


Figure 2.11. Results of filtration tests conducted on the flotation product of BMCH Australian coal sample as a function of reagent dosage (EGMO) at ambient and elevated temperatures and different cake thicknesses. Cake thickness was 0.25 inches for the test results represented in Figure 11-a and 0.55 inches for the ones in Figure 11-b. The tests were conducted at 2 minutes of drying cycle time.

20.85% at 22°C and 60°C slurry temperatures. A plateau was reached at both temperatures with the 3 lbs/ton of EGMO, which is similar to the results obtained with the thin filter cakes and the moisture contents decreased to 13.85% and 12.88%. These results indicate that the reagent usage could decrease the cake moisture contents to sufficiently low values even for the thick cakes. However, it was obvious that the synergetic effect of the elevated temperature and the reagent usage on moisture content reduction was also affected by the characteristics of the coal sample.

Figure 2.12 shows a comparison between the responses of the floated and non-floated Elkview coal sample slurries to dewatering. These tests were conducted to determine the effect of flotation on the efficiency of filtration. During the flotation stage, the coal particles are treated with the collectors and frothers. These reagents adsorb onto the particle surfaces and increase the hydrophobicity of the coal. Furthermore, the excessive amounts of the reagents which are

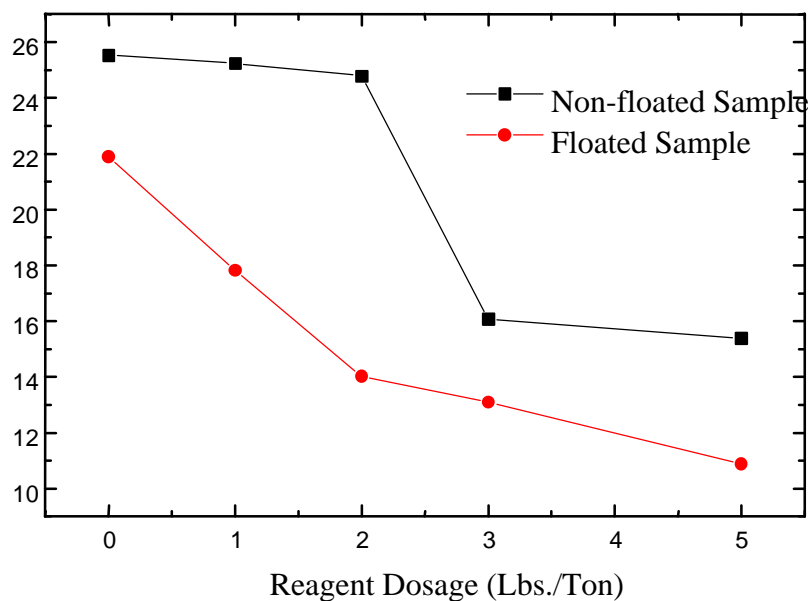


Figure 2.12. Effects on flotation on reduction of the moisture content of the filter cake. Filtration tests were conducted on the flotation product of Elkview run of mine coal sample as a function of reagent dosage (EGMO) on floated and non-floated sample slurries. Drying cycle time was 2 minutes and the cakes were 0.2 inches thick.

left in the slurry after the flotation are usually helpful at the filtration stage. The coal sample used in these tests was received as run-of mine coal from Elkview Coal Company. It was first crushed with jaw and roll crushers. In the laboratory, the + 28 mesh size fractions of the crushed sample was removed by screening. A slurry was prepared using 28 mesh x 0 (minus 600 μm) size fraction coal. Then it was mixed in a 5-gallon bucket for several hours and waited for two days to let the coal surface get completely wet. When the filtration tests were conducted on this sample, the cake moisture content was 25.53% without any reagent addition. The cake thickness was approximately 0.2 inches and the drying cycle time was 2 minutes in all the conducted tests. At low dosages of EGMO, the moisture content remained almost the same as the base value. In the presence of 3 Lbs./ton EGMO, a sharp decrease was observed and the moisture content reduced to 15.39%. Only at the relatively high dosages of the dewatering aid the unfloated sample started to respond to the presence of the additive. This irregular reduction was suspected

to be due to the oxidation of the sample during crushing stage and the absence of the flotation reagents in the slurry

As a second step, the slurry was floated with 400-g/ton kerosene (collector) and 100 g/ton of MIBC (frother). Flotation process refreshed the surfaces of the oxidized coal particles. When the same filtration tests were conducted on this floated sample slurry, the base moisture content decreased down to 21.89% and a moisture content value of 10.88% was reached in the presence of 5 lbs/ton of EGMO. The results of these tests indicated that, flotation plays a very important role on the filtration efficiency. The effective dosage of the dewatering aids required for reducing the moisture content of the floated samples were relatively lower, since the coal surface was pre-treated during the flotation.

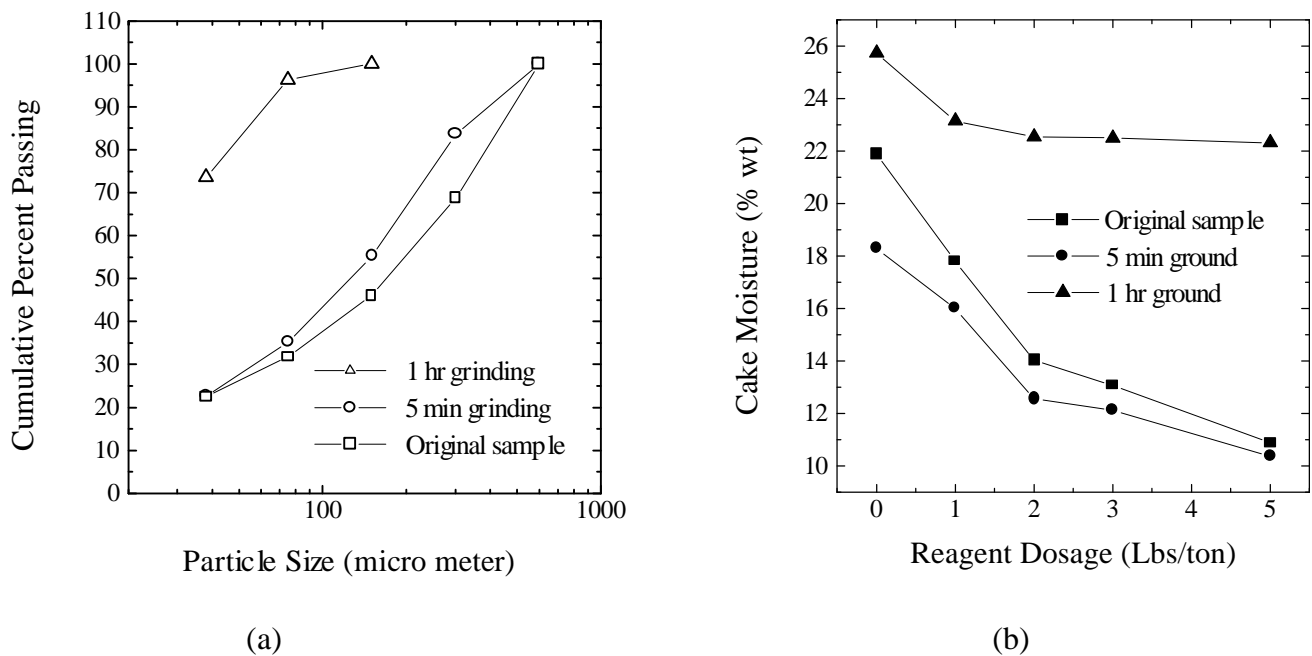


Figure 2.13. Effect of particle size distribution on moisture content reduction of the filter cakes. Figure 13-a shows the particle size distribution of the sample after different periods of grinding. Figure 13-b represents the results of the filtration tests on these samples. Filtration tests were conducted on the flotation product of Elkview coal sample as a function of the reagent dosage (EGMO). Cake thickness was 0.2 inches and the drying cycle time was 2 minutes in all the tests.

Figure 2.13-a and b illustrate the change in moisture content of the Elkview coal sample as a function of the particle size distribution of the sample slurry. The oxidized Elkview coal sample was ground at different time periods to prepare a fresh surface and then floated with 400 g/ton kerosene and 100 g/ton MIBC as in the previous case to pre-treat the particles. Three different samples were used to conduct these experiments. The first one was the floated minus 28 mesh fraction of the crushed Elkview coal sample that was used in the previous set of experiments. The other samples were prepared by grinding the crushed run of mine coal for 5 minutes and 1-hour time periods. Figure 2.13-a shows the particle size distributions of these three coal samples. After 5 minutes grinding, the size distribution of the sample was almost same with the original sample. However, since fresh particle surfaces were created by grinding the sample, a better moisture reduction was observed for the 5 minutes ground sample as shown in Figure 1.13-b. The base moisture contents of the original sample and the 5 minutes ground sample were 21.89% and 18.29%, respectively. At 5 Lbs./ton of EGMO addition, these values decreased to 10.88% and 10.37%. On the other hand, for the 1-hour ground sample the top particle size was only 75 micron and the base moisture content was 25.73%. This value reduced to 21.58% in the presence of 2 lbs/ton of EGMO and no further reductions were observed by increasing the reagent dosage. That was most probably due to the formation of very compact cake structure. The reduction in the particle size causes the formation of very small size capillaries between the coal particles in the filter cake structure and as a result the filter cake resistance increases. The filter medium resistance also goes up due to the blinding of the filter media. Meanwhile, the total particle size exposed to water increases with the decreasing particle size. In summary, these results indicated that if the sample is ground for a very long time period, the particle size gets too small which is detrimental to dewatering due to the reasons explained above.

Figure 2.14 compares the efficiency of EGMO and flocculants as dewatering aids for filtration. The tests were conducted on the Middle Fork coal sample at 0.2 inches cake thickness and 2 minutes drying cycle time. Two types of flocculants were selected, including Magnafloc 1011 and starch. Magnafloc 1011 is a anionic synthetic polymer with high molecular weight and

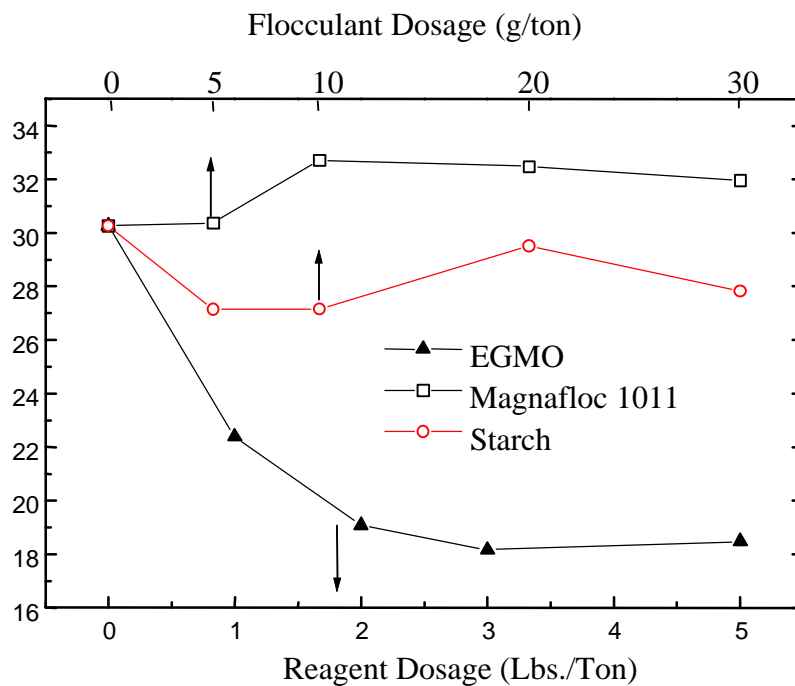


Figure 2.14. Results of filtration experiments conducted on Middle Fork coal sample as a function of EGMO, Magnafloc 1011 & starch dosage. The efficiency of the flocculants in reducing the moisture content was compared with the efficiency of the EGMO. The cake thickness was 0.20 inches and the drying cycle time was 2 minutes for all tests.

starch is a very well known natural flocculant. The base moisture content of the cake was 30.26 % in the absence of any dewatering aid. In the presence of Magnafloc 1011, this value increased to 32.72% at a dosage of 10 g/ton and then remained in the same level for the further increased dosages. This was most probably due to the formation of trapped water between the flocculated coal particles in the filter cake. Similarly, addition of the starch, which is a weaker flocculant, decreased the moisture content only by 2% at low dosages (5-10 g/ton) and then an increase was observed at the higher dosages. These results indicated that the flocculant usage was inefficient in filtration. On the other hand, the novel dewatering aid EGMO decreased the cake moisture by ~11% at a dosage of 5 Lbs./ton and a cake moisture content of 18.47% was reached.

Table 2.3 shows the results of the filtration tests conducted on the flotation product from CONSOL, Inc., which was a 28 mesh x 0 Pittsburgh # 8 seam coal. Each test was conducted by using 200 ml of coal slurry, which gave approximately 0.2 inches of cake thickness. As shown,

Reagent Dosage (Lbs./ton)	Cake Formation Time (sec)	Product Moisture (% weight)
0	61	25.57
0.5	48	23.96
1	36	22.42
2	36	21.92

Table 2.3. Results Obtained Using EGMO on the Pittsburgh Coal Sample (28 mesh x 0). The cake thickness was 0.2 inches and the drying cycle time was set to 2 minutes.

the cake moisture was lower than that was obtained without dewatering aid by 4% only in the presence of 2 Lbs./ton of EGMO. This result was very poor compared to the performance of this dewatering aid on the other coal samples. The poor results obtained with the sample may be attributed to the possible contamination of the coal sample by the flocculants during the plant operation.

2.3.3. Statistical Analyses

The statistical analyses were performed on the BMCH coal sample (100 mesh x 0, 25% solid), using the Design Expert software. Four main parameters were studied. These included the temperature and volume of the sample slurry (which changed the cake thickness), reagent dosage and the drying cycle time. EGMO was used as the dewatering aid since it was determined to be a good performing reagent in the previous tests. Three application levels were chosen for each variable as the upper limit, lower limit and the medium value. The slurry temperature was changed in the range of 22 (Ambient) to 60°C. The minimum and the maximum amounts of the slurry volume were determined as 100 and 200 ml and the medium value was 150 ml in this range. The cake thicknesses were 0.25 inches for 100 ml, 0.35 inches for 150 ml and 0.55 inches for the 200-ml sample slurries. The third factor was the reagent dosage and EGMO was used at 1, 2 and 3 Lbs./ton of additions. According to the previous observations, this dosage range was observed to be effective on reducing the cake moisture. The drying cycle time periods, which was determined as a fourth factor, were selected as 1, 2 and 5 minutes. Table 2.4 shows the selected ranges of the studied factors.

FACTORS		Minimum Level	Medium Level	Maximum Level
A	Temperature (°C)	22	41	60
B	Slurry Volume (ml)	100	150	200
C	Reagent Dosage (Lbs./ton)	1	2	3
D	Drying Cycle Time (min)	1	2	5

Table 2.4. The application ranges of the selected factors for statistical analyses.

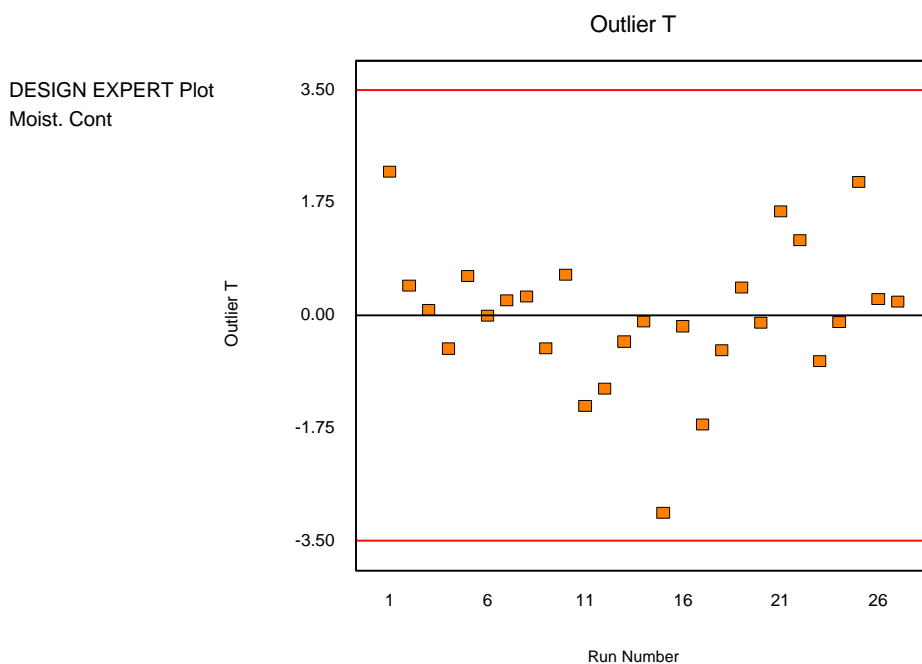


Figure 2.15. The outlier values for the linear model.

The Design Expert program gave 27 tests conditions to be conducted to perform the statistical analyses. Appendix I lists the organization of the tests and the test results in the run order. The Box-Behnken method was chosen as the response surface design type. The program gave three different solution models according to the test results: linear, quadratic and cubic. The cubic model gave more than one solution (aliased) so it was neglected. Between the linear

and the quadratic models, the linear model was observed to fit better to the experimental results. Although the R-square (0.3812) value of the linear model, which estimates the fitness of the model, was lower than the quadratic model (0.5795), the quadratic model was observed to give unrealistic results. The linear model has also the advantage of representing the direct relationship between the selected factors and the cake moisture reduction. Besides, it gave a more suitable outlier as represented in Figure 2.15. The outlier values determined by the model are the normalized deviation of the experimental results from the predicted value in the program. The range illustrated in Figure 2.15 for the linear model is approximately between 2.3 and -3 and the values below ± 3.5 are considered as good fit.

The reason for the deviation between the predicted and the experimental results was suspected to be due to the oxidation of the coal sample during the time passed to complete the experiments. The results of the control tests also supported this. In a period of one week, that was spent to complete the tests, three control experiments were conducted at the medium levels of the each variable range. The tests were repeated at 41°C slurry temperature with 150-ml sample slurry and in the presence of 2 Lbs./ton of EGMO. The drying cycle time was kept at 2 minutes. The results of the control tests (run numbers, 4, 7 and 26) showed the increase in the moisture content by time. The cake moisture was 16.3% for the first control test and then it went up to 19.43% and 19.50% for the following repetitions as a result of the oxidation of the sample.

Based on the linear model, the relationship between the moisture content reduction and the selected filtration variables was expressed as in equation 2.1. This equation is in the coded factors and shows the relative effect of the each variable on changing the cake moisture. Equation 2.2 represents the same relationship in the actual values.

$$\begin{aligned}
 \text{Cake moisture (\%)} &= 18.18 && [2.1] \\
 &- 0.54 \times \text{Temperature} \\
 &+ 4.25 \times \text{Slurry volume} \\
 &- 1.04 \times \text{Reagent Dosage} \\
 &- 0.53 \times \text{Drying Cycle Time}
 \end{aligned}$$

$$\begin{aligned}
 \text{Cake moisture (\%)} &= 9.47 && [2.2] \\
 &- 2.85 \times 10^{-2} \times \text{Temperature}
 \end{aligned}$$

$$\begin{aligned}
&+ 8.5 \cdot 10^{-2} \times \text{Slurry volume} \\
&- 1.04 \times \text{Reagent Dosage} \\
&- 0.26 \times \text{Drying Cycle Time}
\end{aligned}$$

According to equation 2.1, the volume of the sample slurry (cake thickness) has the most dominant effect on changing the cake moisture content. As the slurry volume increases, the cake moisture also increases by 4.25 times of it. The reagent dosage is the second important factor on moisture content reduction. The temperature and the drying cycle time have almost the same effect and they both help decreasing the cake moisture at the increasing levels.

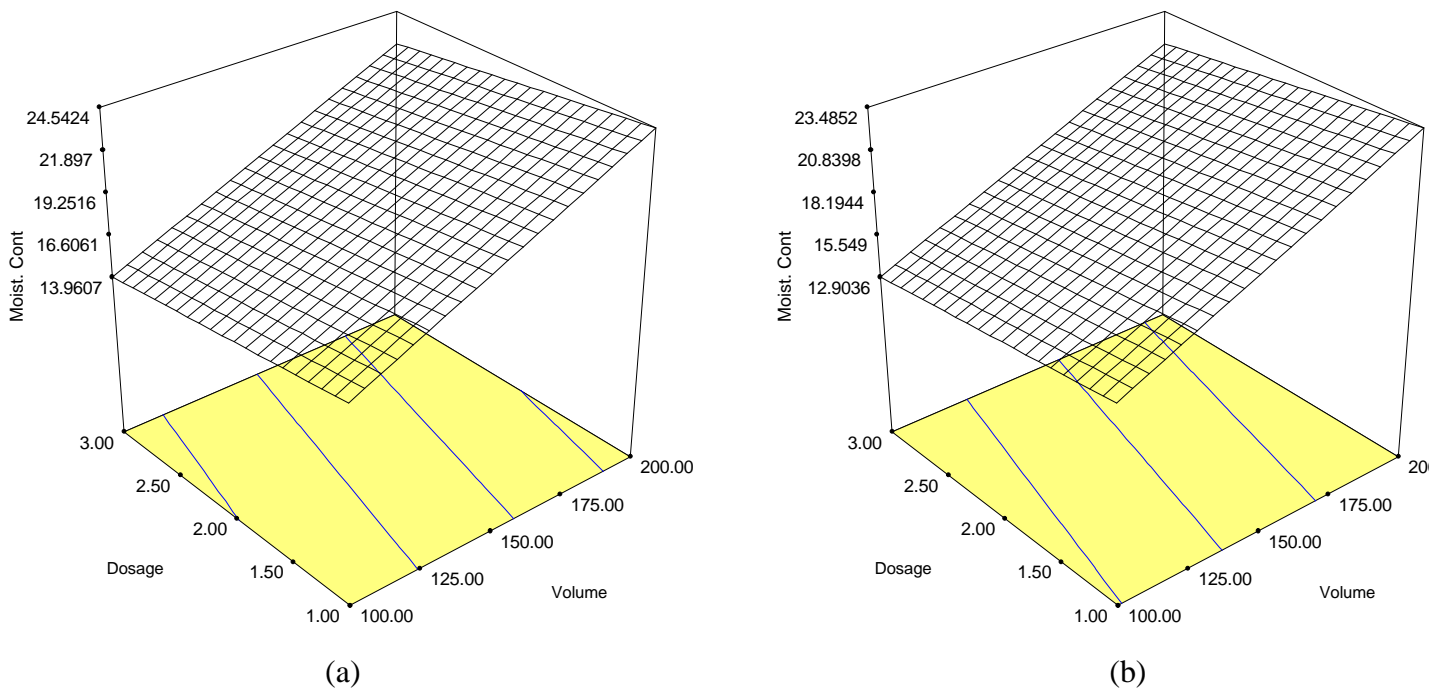


Figure 2.16. Change in the moisture content of the BMCH Australian coal sample as a function of the dosage of the dewatering aid (EGMO) and volume of the sample slurry according to the Design Expert. The plots were taken for 22°C slurry temperature and drying cycle times of 1minute (a) and 5 minutes (b).

Figure 2.16 illustrates the three dimensional view of the reagent dosage-slurry volume relationship based on the linear model. The response surface was selected as the moisture content. The model predicted that the moisture content of the filter cake increases with the increasing slurry volume and the decreasing reagent dosage. For 1 minute drying cycle time, the moisture content of the cake was determined by Design Expert as 13.96 % at 3 Lbs./ton reagent addition using 100 ml sample slurry. This value increased to 24.54% with 1 Lb./ton of reagent and 200 ml sample slurry volume as seen in Figure 2.16-a. For 5 minutes drying cycle time, the moisture contents were predicted to decrease only by 1% points more compared to the 1 minute dried cakes at each level as illustrated in Figure 2.16 (b). The result of the program gave 12.90% cake moisture with 3 Lbs./ton EGMO at 100-ml volume and 23.49% moisture with 1 Lbs./ton EGMO at 200 ml after 5 minutes drying cycle time. These results highlighted the weak effect of drying cycle time on reducing the cake moisture content compared to the effect of the slurry volume and reagent dosage.

The slurry temperature was estimated to have almost the same effect with the drying cycle time on cake moisture reduction as expressed in equation 2.1. Figure 2.17-a and b show the three dimensional reagent dosage-slurry volume plots at elevated temperature with 1 minute and 5 minutes drying cycle time periods, respectively. The results indicated that the moisture content reduction was slightly decreased by increasing the slurry temperature. When the slurry temperature was increased to 60°C (maximum level), only about 1% reduction in the moisture contents were observed compared to the values at 22°C both for 1 minute and 5 minutes drying cycle times. After 1 minute drying, the cake moisture content was predicted to be 12.88% (13.96% at 22°C) with 3 Lbs./ton EGMO addition for a 100 ml sample slurry. This predicted value decreased to 11.82% (12.90% at 22°C) with 5 minutes drying cycle time under the same conditions. According to these results the effect of the drying cycle time also remained the same at the elevated temperatures. Similarly, a 1% reduction was predicted at 60°C between 12.88% and 11.82% with 1 minute and 5-minute drying cycle time periods as observed at 22°C.

Although the linear model was chosen to identify the direct relationship between the cake moisture and the selected variables, it was found to be beneficial to use the quadratic model to

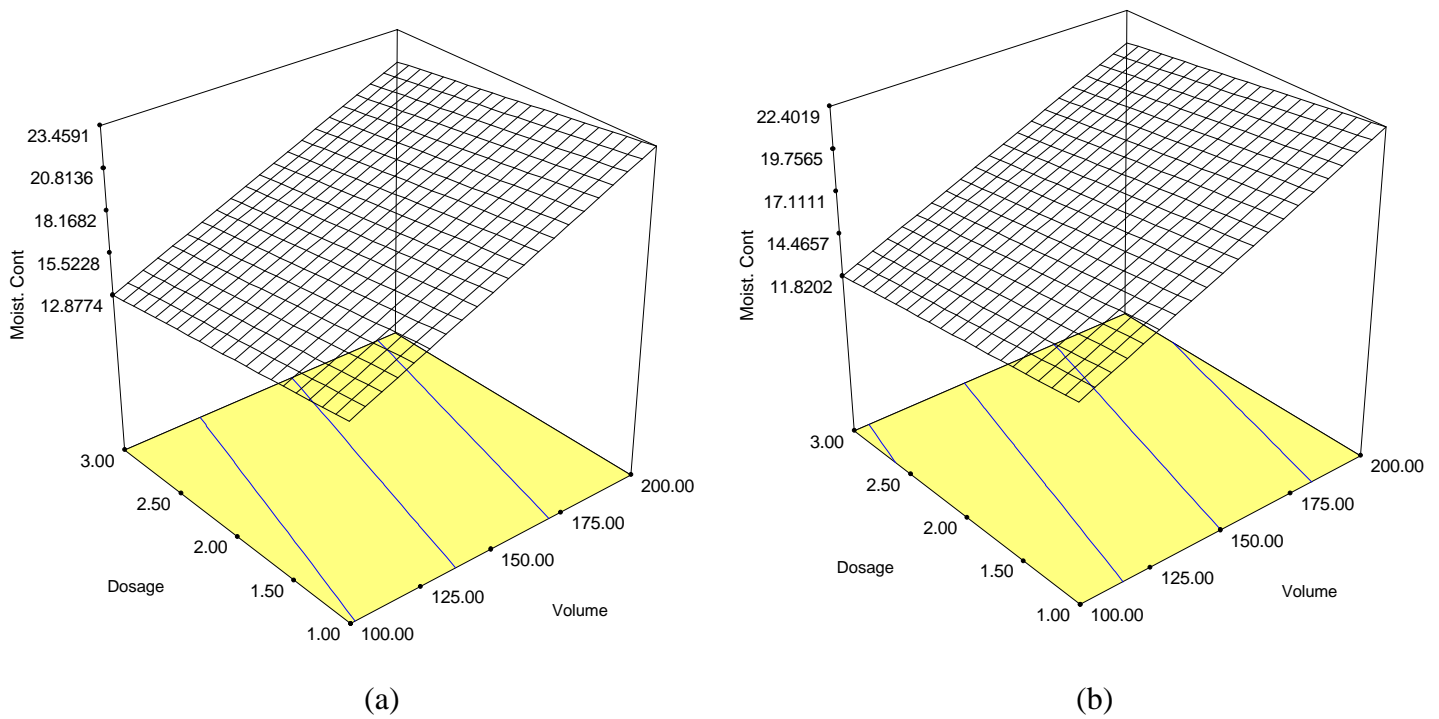


Figure 2.17. Change in the moisture content of the BMCH Australian coal sample as a function of the dosage of the dewatering aid (EGMO) and volume of the sample slurry according to Design Expert. The plots were taken for 60°C slurry temperature and drying cycle times of 1minute (a) and 5 minutes (b).

determine the combined effects of these factors. Equation 2.3 shows the moisture content relationship in coded factors determined by Design Expert based on the quadratic model. Some of the terms in the equation did not agree with the general expectations of the vacuum filtration, but the combined effects of the slurry temperature and reagent dosage (A&C) and the reagent dosage and drying cycle time (C&D) were observed to be making sense and important. They both were found to make a combined effect on decreasing the cake moisture content when they are combined. If both the slurry temperature and the reagent dosage are increased at the same time, the cake moisture decreases further down than it is expected to decrease based on the individual effect of each factor separately. The moisture content of the cake is predicted to decrease 1.29 times of the multiplication of these factors. Similarly, if both the reagent dosage and the drying cycle time are increased, a reduction of 3.42 times of their multiplication in moisture content is expected which is quite an important effect.

$$\text{Moisture Content (\%)} = \text{[2.3]}$$

$$\begin{aligned}
& 18.34 - 0.54 \times A + 5.11 \times B - 2.18 \times C - 0.41 \times D \\
& + 1.42 \times A^2 - 1.05 \times B^2 + 0.05 \times C^2 - 0.54 \times D^2 \\
& + 1.01 \times AB - 1.29 \times AC + 0.02 \times AD - 0.38 \times BC + 2.58 \times BD - 3.42 \times CD
\end{aligned}$$

Figure 2.18 shows the drying cycle time-reagent dosage plots of Design Expert at 22°C (a) and 60°C (b) by using the quadratic model. It can be seen that the cake moisture is expected to decrease to 7.70% at ambient temperature with 100 ml of sample slurry when the highest levels of the reagent dosage and the drying cycle time are used. If the temperature is increased to 60°C under the same conditions (Figure 2.18-b), a further decrease is estimated in moisture content down to 2.07%. These values show the combined effect of increasing reagent dosage and drying cycle time on reducing the cake moisture content. The third factor, temperature, has

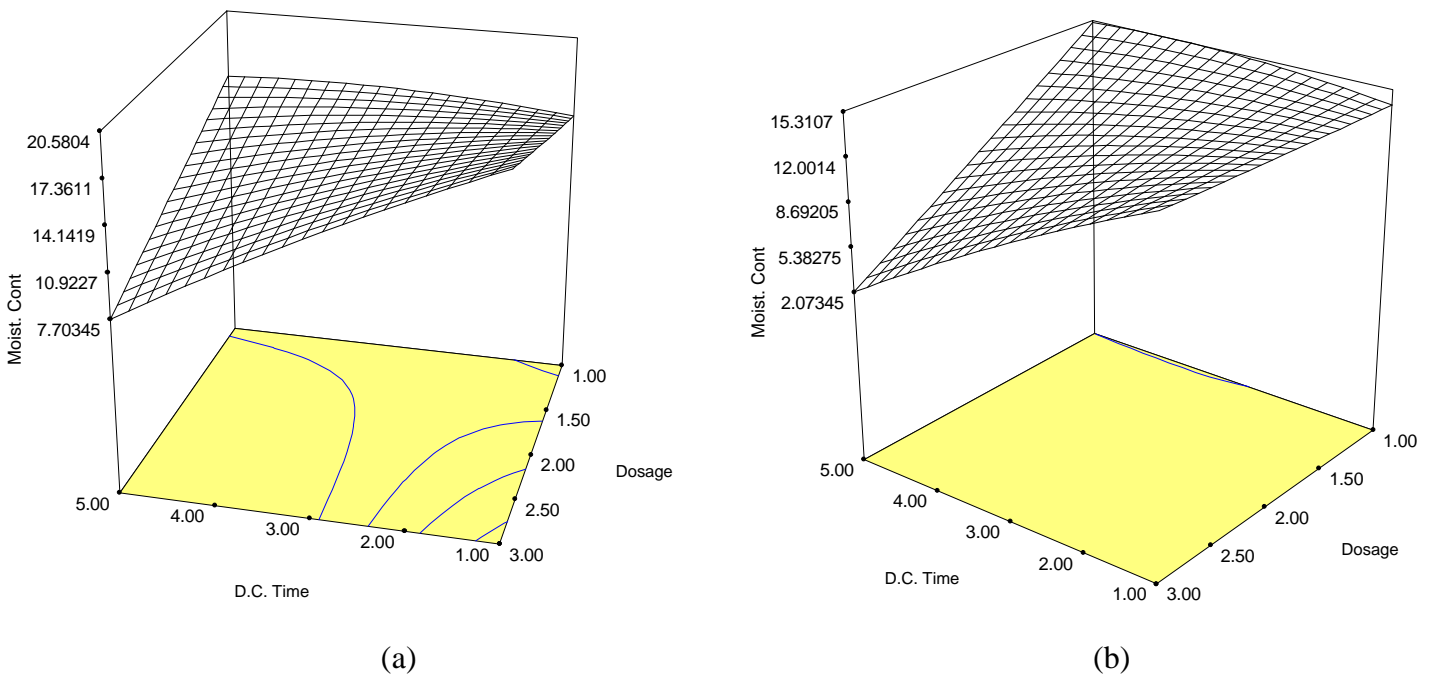


Figure 2.18. Change in the moisture content of the BMCH Australian coal sample as a function of the dosage of the dewatering aid (EGMO) and drying cycle time based on the Design Expert. The plots were taken for 100-ml slurry volume and slurry temperatures of 22°C (a) and 60°C (b).

also a correlation combined with the reagent dosage. This can be explained on the basis of the increasing solubility of the reagent at the elevated temperatures. According to this observation, a correlation also exists between the temperature, slurry volume and the drying cycle time. The extremely low moisture content value (2.07%), that is expected to be reached at 60°C with 3 Lbs./ton of EGMO addition and at 5 minutes drying cycle time for a 100 ml slurry indicates that clearly. In summary, decreasing the volume of the slurry (cake thickness) and increasing the all other three factors at the same time is the requirement to reach the low cake moisture content values.

2.4. Conclusions

1. The novel dewatering aids developed at Virginia Tech were found to be effective in decreasing the moisture content of the fine coal slurries. The reagents decreased the cake moisture of a Middle Fork coal slurry (100 mesh x 0) by 10-14% points. Two reagents, Polymethylhydrosiloxane (polymer) and Ethylene Glycol Monooleate were used as the novel dewatering additives, which were patented by Yoon and Basilio. Among all the reagents tried on the coal sample, EGMO was determined to perform slightly better and it was used to complete the rest of the study.
2. The increase in the pH of the coal slurry (pH 7.6 to pH 11) increased the moisture content of the cakes filtered without using the dewatering aid on a Middle Fork coal sample. This was due to the increase in the surface charge of the coal by increased pH. However, in the presence of the EGMO, the moisture content remained almost constant at all the pH values. This implies that the novel dewatering aid adsorbs onto the coal surface efficiently and decreases the surface charge. That helps to reduce the moisture content of the coal cakes.
3. On a Middle Fork coal sample slurry, the increase in the vacuum pressure decreased the cake moisture regardless of whether the dewatering aids were used or not. Although there were a parallel decrease in the moisture content of the base experiments and the tests conducted with the dewatering aid, in the presence of the EGMO the moisture content values were approximately 12% lower compared to the results of the control tests.
4. Increase in the drying cycle time, helped to decrease the moisture content of a 0.1 inches thick cake in the presence of the dewatering aid (EGMO) on a Middle Fork coal sample,

while no significant decrease was observed for the base experiment results conducted under the same conditions. However, after 3 to 5 minutes of drying cycle time a plateau was reached and the cake moisture remained almost constant even with the reagent addition. During the initial stages of dewatering, the capillary water was removed sufficiently. For a 0.2 inches thick filter cake, the water was observed to be in the funicular state between 0.5 to 3 minutes drying cycle time periods and the pendular water was started to reduce after 5 minutes drying. The moisture content of the cake was observed to be about 8% and 12% points lower at the funicular and the pendular states compared to the initial values, respectively. These results indicated that a reasonable reduction in the moisture content of the filter cakes is possible even at short periods of drying cycle time.

5. An increase in the cake moisture was detected with the increasing cake thickness both in the absence and the presence of the dewatering aid on a Middle Fork coal sample. The relative effect of the novel dewatering aid on reducing the moisture content diminished at the increased cake thicknesses. For a 0.1-inch thick cake there was 12% difference in the moisture contents of the cakes produced with and without the usage of the dewatering aid. This difference reduced as the cake thickness increased. For the 0.4 inches thick cake there was only 8% points difference between the results of the tests conducted in the absence and the presence of the dewatering aid.
6. At the elevated temperatures, the moisture content of the filter cake was observed to decrease further down. The moisture content reduction continued as the reagent dosages increased without reaching a plateau. This was due to the decrease in the viscosity of the capillary water at the elevated temperatures. However, as the cake thickness increased, the effect of temperature on reducing the cake moisture became less significant.
7. The hand-conditioning method was noticed to be more suitable for conditioning than the mixing based on the results obtained with a Middle Fork coal sample. The mixing of the coal slurry by means of a blender decreased the original particle size distribution of the sample and caused a serious increase in the final cake moisture content. However, the slurry particle size distribution did not change when the hand-conditioning was applied on the coal samples. Without using any reagents, the test results obtained by mixing was around 4 to 5% points

higher than the cake moistures obtained by hand-shaking under the same conditions. When the dewatering aid was used, the difference became less significant. In the presence of EGMO it was only 1 to 2%. A 1minute conditioning time by hand shaking method was observed to be sufficient enough for the reagent adsorption on the surface of the coal particles.

8. On a 28 mesh x 0 Elkview coal sample effects of dewatering aid was also studied. The tests were conducted at 0.2 inches of cake thickness at 22°C and 60°C. The drying cycle time was kept at 2 minutes and the EGMO was used at the dosages up to 2 Lbs./ton to fit the results to the industrial applications. Under these reasonable test conditions, the cake moisture decreased about 8% points indicating the industrial applicability of the process.
9. The dewatering aid helped improving the kinetics of the vacuum filtration as well. In the presence of the reagent, cake formation time was shorter than the tests conducted in the absence of it. At the elevated temperatures, a further decrease in the cake formation time was observed for the tests conducted on Elkview coal sample.
10. The novel dewatering aids were sensitive to the oxidation of the coal sample. The efficiency of the filtration decreased as the coal became oxidized. The cake formation time was also longer for the oxidized coal slurries. It can be concluded that because of the oxidation, the reagent could not adsorb onto the coal surface properly. The harmful effect of oxidation can be minimized by grinding the coal sample, which creates fresh surfaces.
11. Flotation process was determined to have a crucial effect on the filtration efficiency. Since the coal surface is pretreated during the flotation application, the response of the floated particles to filtration was quite better compared to the non-floated slurries. Floating the slurry also helps to reduce the detrimental effect of oxidation on the coal surface. The surface treatment during the flotation increases the hydrophobicity of the coal as a result of the adsorption of the reagents onto the coal surface.
12. The effect of particle size distribution on cake moisture reduction was studied on an Elkview run of mine sample. Three different slurries were prepared by crushing and grinding this coal. First sample was a minus 28-mesh fraction of the crushed coal. The other two were prepared by 5 minutes and 1hour grinding. The 5 minutes ground sample gave the lowest

moisture content. That was due to the elimination of the surface oxidation of the particles by grinding and increase in the hydrophobicity as a result of the flotation. The finest coal sample prepared by 1 hour grinding had the highest moisture content and lowest decrease in the cake moisture compared to the base value (~3%). That was because of the increasing coal surface area by decreasing particle size and the reduced capillary sizes in the cake structure. It is quite obvious that the size distribution of the coal sample plays an important role on the efficiency of filtration.

13. The performances of the natural and the synthetic flocculants were compared with the novel dewatering aid EGMO in terms of the capability to reduce the cake moisture. Although the flocculants were used at lower dosages compared to the novel dewatering aid, they were observed to increase the moisture content of the filter cakes. That was due to the trapped water between the flocculated particles.
14. The statistical analyses performed on the BMCH coal sample indicated that the cake thickness plays the major role on determining the moisture content of the filter cakes. The reagent dosage, slurry temperature and drying cycle time were determined to have less dominant effects. However, a correlation was determined based on the statistical results between the reagent dosage, drying cycle time and the slurry temperature. The best results in reducing the cake moisture can be obtained by decreasing the cake thickness (slurry volume), and increasing the reagent dosage, drying cycle time and the slurry temperature which was also concluded from the results of the regular filtration tests.