Gul Bahar Basim Chapter 4. SURFACE CHEMISTRY ANALYSES

4.1. Introduction

The surface chemistry analyses were performed on various coal samples to detect the changes in the chemistry of the dewatering system in the presence of the novel dewatering aids developed at Virginia Tech. The improvement in the efficiency of the coal dewatering depends on the decrease in the surface tension of the liquid-air interphase or increase in the water contact angle, according to the Laplace equation. To determine the variation of these factors with the usage of the novel reagents, surface tension and contact angle experiments were conducted on the coal samples prepared under the controlled laboratory conditions. Ethylene glycol monooleate (EGMO) was used as a good representative of the novel dewatering aids to perform the surface chemistry analyses. Most of the commonly used dewatering aids either decrease the surface tension (γ) or increase the contact angle (θ); however, the novel reagent was observed to decrease γ and increase θ simultaneously. The increase in the efficiency of dewatering in the presence of the novel dewatering aids may, therefore, be explained on the basis of this fact.

4.2. Experimental Methods and Materials

The surface chemistry of dewatering was studied using two methods. These were the surface tension measurement and the contact angle determination. The tests were conducted both in the absence and the presence of the additives to be able to compare the effects of the dewatering aids in changing the surface chemistry of the system. The novel dewatering aid, EGMO was utilized to determine the significance of using a novel additive in coal dewatering. In addition, the performance of EGMO in increasing the contact angle of coal was compared with the performance of kerosene, which is a very commonly used collector in coal flotation. The Fisher Surface Tensiomat Model 21was used to measure the liquid-air surface tension. This equipment was designed to measure the surface tension using the ring method. The dewatering aid was dissolved in nanopure water at desired concentrations. The prepared solutions were poured into the 50-ml glass cups and the surface tensions were measured by dipping the clean platinum ring into them. To minimize the experimental errors, the measurements were repeated

five times at each concentration and the arithmetic average value of these five results was taken as the surface tension of the solution at that particular concentration.

The surface chemistry analyses were conducted on Sunny Side and Elkview coal samples. The Sunny Side coal was used only for determining the changes in the contact angle in the presence of various novel dewatering aids. The Elkview coal sample, which was received as run of mine coal, was selected to conduct a set of experiments including contact angle and surface tension measurements, the results of which are compared with the results of the filtration tests. To perform the contact angle tests, the proper size coal specimens were hand-picked (~ 1-2 cm² cross sectional area) from a crushed coal sample. The sides of the coal chunks were polished by using 120, 240, 400, 600 grit special silicon carbide grinding papers. To smooth the surfaces that were used to measure the contact angle properly, the larger sides of the samples were further polished by using a polishing cloth. The coal samples were given a rectangular prism shape by this technique and the surface roughness was reduced to minimum. As a result, the total surface area for two samples having similar sizes and shapes became comparable. To clean the prepared coal sample surfaces, they were put into chloroform after polishing for 8 hours. Then they were kept in nanopure water for 24 hours to reach the equilibrium. The first contact angle measurements were conducted on these clean coal samples to detect the base value of the coal surface contact angle in the absence of any reagents.

The contact angle measurements were conducted by using the sesile drop technique utilizing the Rame'-Hart, Model 100 goniometer. One of the polished coal pieces was selected and used for all the tests conducted to complete one set of experiments to keep the consistency. The chosen coal sample was dried by nitrogen blowing after it was taken out of the solution. The droplets were formed on the dried coal sample using a microliter syringe. Nanopure water was used to form the sesile drops. Since the surface of the coal is not homogeneous naturally, five droplets were created on the sample surface and both right and left side contact angles were measured. The arithmetic average of these ten measurements was calculated to get the true contact angle value of the selected coal surface. To determine the change in the contact angle of the coal surface at varying reagent concentrations, the same selected coal sample was weighed and put into a 150-ml glass flask. The flask was filled with nanopure water up to 100-gram

weight excluding the weight of the container. This system was considered as a slurry with a percent solid content of the mass of the coal chunk. The reagent was added into the flask by means of a microliter syringe, at the calculated dosage in terms of pounds per ton. In the presence of the reagent, the sample was conditioned by hand shaking. The conditioned coal chunk was then taken out of the solution and its surface was dried by blowing nitrogen gas. The solution left in the flask after conditioning was filled into the microliter syringe to form the sesile drops on the coal surface. This method gave the advantage of determining the real contact angle of the coal surface in the slurry. The contact angle measurements were conducted according to the procedure explained previously. After the measurements at one concentration were completed, the coal sample was put into chloroform for half an hour to remove the adsorbed EGMO and clean the surface. Then it was placed in nanopure water for 1 hour to equilibrate again. In order to make sure that the surface was cleaned from the adsorbed reagents, the contact angle of the cleaned surface was measured with nanopure water. These results were compared with the initially determined base contact angle value of the coal, which was measured before treating it with any reagents. When the contact angle values were the same, it was assumed that the surface was clean enough and the same procedures were applied to the coal sample at the following reagent concentrations to determine the contact angle values.

These contact angle measurements were suspected not to reflect the real contact angle change in the system in the presence of the additives due to the very small surface area of the coal chunks relative to the fine size coal particles having the same mass. The adsorption density of the reagent is expected to be different for a sample with a smaller surface area, which changes the response of the system to dewatering due to the changes in the contact angle and surface tension. To make the measurements more reliable, a set of experiment was conducted on 25% solid density coal slurries, which were prepared by using -14 + 28 mesh size crushed coal particles in addition to the polished coal chunk. These samples were treated to detect all the contact angle, surface tension and moisture reduction responses of the coal particles in the dewatering system. The particle size distribution of this slurry samples was still coarse compared to the slurries used for fine coal dewatering. This selection was done on purpose to

prevent the very fine size material stick onto the polished coal surface during conditioning stage and reduce the efficiency of the contact angle measurements.

4.3. Experimental Results and Discussions

The change in the contact angle of the coal surface in the presence of several novel dewatering aids developed at Virginia Tech is shown in Table 4.1. These contact angle measurements were performed on polished Sunny Side coal sample surface using the sessile drop technique. The measurements were conducted both in the absence and the presence of the additives. In the former case, the contact angle of a water drop was measured and in the latter case the contact angle of the pure reagent droplet was measured on the coal surface. The results showed that the surface contact angle values obtained in the presence of the novel dewatering aids were almost two times larger than the base contact angle values measured using pure water droplets. The surface contact angle of the coal increased to 62.2° with EGMO from the 38.9° of the base value. These results indicated that the novel dewatering aids increase the contact angle of the coal surfaces and hence help decreasing the cake moisture content according to the Laplace equation.

Contact Angle With Water	Contact Angle With Reagent
38.9	62.2
44.6	85.7
	Contact Angle With Water 38.9 44.6

Table 4.1. Change in contact angle of the Sunny Side Coal Sample with EGMO and PMHS.



Figure 4.1. Results of the surface tension measurements of the EGMO solutions in water at different concentrations. The tests were conducted at room temperature (~23°C), by Fisher Surface Tensiomat Model 21.

Figure 4.1 shows the results of the surface tension analyses for the solutions prepared at varying EGMO concentrations. To conduct these experiments, EGMO was dissolved in nanopure water at different percentages starting from 1×10^{-6} % up to 100%. At 1×10^{-6} % concentration, the surface tension of the solution was 74.1 dyne/cm, which was very close to the surface tension of the pure water measured by the same equipment (74.2 dyne/cm). As the reagent concentrations started to increase, the surface tension decreased drastically even at very low concentrations. At 1×10^{-3} % concentration the surface tension decreased to 54.56 dyne/cm and at 10^{-1} % concentration it reached to 32.2 dyne/cm. After this concentration, the surface tension values remained almost constant as the reagent dosage was further increased. A value of 30.6 dyne/cm was observed in the presence of 10% EGMO, and the surface tension of the pure reagent was 24.67dyn/cm at 100% concentration.

Figure 4.2 represents the results of the contact angle experiments conducted on the Elkview coal sample. The surfaces of the selected coal samples were prepared according to the



Figure 4.2. Results of the contact angle measurements on polished Elkview coal sample under filtration conditions. The tests were conducted in the presence of Kerosene and EGMO at different dosages. Rame'-Hart Model 100 goniometer was used to perform the measurements.

procedure explained in the Experimental Methods and Materials section. The tests were performed using kerosene and EGMO to compare the effect of these reagents in increasing the contact angle of the coal surface. The weights of the coal chunks were 2.25 gram and 2.4 gram for the tests conducted with kerosene and EGMO, respectively. The adsorption densities of the reagents on the sample surfaces were assumed to be identical depending on the sample specimens having almost the same weight and similar shapes. Kerosene is one of the most common collectors used for the coal flotation. However, the experimental results showed that the increase in the contact angle of the coal surface was higher in the presence of the novel dewatering aid EGMO, compared to kerosene. The base contact angle value of the coal piece that was used to perform the tests with kerosene was 28.02° and the base contact angle of the other coal piece was 29.64°. These values were considered as close enough to make a comparison between the test results obtained with kerosene and EGMO. In the presence of kerosene, the contact angle of the coal surface started to increase significantly only after 3 Lbs./ton of reagent addition. The contact angle value was 39.85° at this dosage, which increased

slightly and reached 42° at 5 Lbs./ton dosage. On the other hand, in the presence of EGMO, the contact angle immediately increased to 42.39° at 1 Lb./ton dosage and continued to increase up to 46.78° at 5 Lbs./ton EGMO addition.

To determine the change in the surface chemistry of the dewatering system in the presence of the novel dewatering aid EGMO, surface tension, contact angle and vacuum filtration tests were conducted on the Elkview run of mine coal sample. A coal specimen was prepared by polishing the surface as explained previously. In addition, the -14 + 28 mesh (-1190 +600 µm) size fraction of the crushed Elkview run of mine sample was separated by screening. The mean particle size of this sample was 747.5 µm. A 25-gram of it was weighed including the coal chunk and a 25% solids (by weight) content coal slurry was prepared by adding 75-ml nanopure water in a 150-ml flask. The coal particles with a coarse particle size distribution was preferred to prepare the slurry on purpose to prevent the very fine size material stick onto the coal chunk that was kept in the solution to measure the contact angle. The coal slurry was first conditioned under the flotation conditions to make the tests results comparable with the dewatering tests conducted on the floated sample slurries received from the operating coal preparation plants. To simulate the flotation conditions, the slurry was conditioned for 3 minutes by hand shaking with 400-g/ton kerosene addition. After that, MIBC was added into the slurry at 100-g/ton dosage as the frother. The slurry was conditioned for one more minute. This sample was assumed to represent the base conditions because the regular coal slurry samples that were used to conduct the dewatering tests were sent as floated clean products from the coal preparation plants. The first tests were conducted on this sample. Then the sample was cleaned in chloroform and the same treatment was applied again to make the surface reflect the properties of the floated coal. The usage the same coal specimen at all the tests of a set of experiments gave the advantage of keeping the surface properties consistent. To conduct the tests in the presence of the dewatering aid, EGMO was added into the slurry at calculated dosages that was at the flotation conditions. Then it was conditioned for one more minute by hand shaking. At the end of the conditioning, the coal specimen was used to measure the change in the contact angle and the vacuum filtration tests were conducted on the remaining part of the

coal slurry. The filtrates collected during the vacuum filtration tests were used to observe the change in the surface tension of the solution after the coal particles were treated in it.

Figure 4.3 shows the results of the contact angle measurements obtained on the Elkview coal sample at flotation conditions. The conditioned coal chunk was taken out of the solution and dried by nitrogen blowing. The contact angle value that was measured at the base condition was 49.92°. It is important to note that the base slurry sample was conditioned with kerosene as a collector and MIBC as a frother to represent the floated slurry sample. There was a significant increase in the contact angle of the base sample compared to the results obtained with the Elkview coal in the absence of any reagents. As it was illustrated in Figure 4.2 the contact angle of the untreated coal was approximately 28-29°. The addition of EGMO into the slurry after treating it with the flotation reagents further increased the contact angle to 59.25° at 1 Lbs./ton dosage. It continued to increase up to 70.62° value at 3 Lbs./ton of EGMO addition.



Figure 4.3. Results of the contact angle tests conducted on the Elkview coal sample in the presence of EGMO. The coal sample was treated at flotation conditions first and then the reagent was added into the slurry as the dewatering aid.



Figure 4.4. Results of the vacuum filtration tests organized on the Elkview coal sample slurry. The tests were conducted in the presence of the novel dewatering aid EGMO at varying concentrations. The vacuum pressure was set to 25 inches-Hg and the drying cycle time was 2 minutes. The cake thickness was approximately 0.25 inches for all the tests.

Figure 4.4 shows the results of the vacuum filtration experiments conducted on the Elkview coal sample slurries that were prepared previously. The conditioned samples were subjected to filtration by setting the vacuum pressure to 25 inches-Hg. The cake thicknesses were approximately 0.25 inches for all the tests and the drying cycle time was set to 2 minutes. The base moisture content of the sample was 10.73%, which decreased to 7.49% in the presence of 1 Lbs./ton of EGMO. The moisture contents obtained with this coal sample were relatively low due to the coarse particle size distribution of the slurry. There was an obvious reduction in the cake moisture content with increasing contact angle values which was clear from the comparison between the results of the coate in the cake moisture by increasing contact angle values is the results of the Figure 2.12 presented in Chapter 2. In that figure the change in the moisture contents of the floated and non-floated coal samples in the presence of EGMO were compared. The coal sample was the same particular Elkview coal sample that was crushed and screened to

minus 28-mesh size. The moisture contents of the filter cakes obtained with the floated coal slurry sample were about 4% lower than the results obtained with the non-floated sample slurry at varying dosages of EGMO. It was observed from the contact angle measurements conducted on the floated and non-floated coal samples that, the contact angle values obtained in the presence of EGMO were 20-25 degrees higher for the floated sample. That difference can be noticed obviously from the comparison of the results illustrated in Figure 4.1 and Figure 4.2. The consistency of the data presented in this work indicated the importance of the increase in the contact angle on decreasing the cake moisture content.



Figure 4.5. Comparison of the surface tension results of the solution prepared with EGMO in the absence of the coal particles and the surface tension of filtrates collected from the vacuum filtration tests. The increase in the surface tension of the filtrates indicates the adsorption of the reagent onto the coal surface during the conditioning.

Figure 4.5 illustrates the results of the surface tension measurements of the solutions prepared at varying concentrations of EGMO in the absence and the presence of the coal particles as a solid phase in the system. To make a comparison, the filtrates of the vacuum filtration experiments conducted on the Elkview coal sample were collected. The novel dewatering aid, EGMO was used for the filtration experiments at 1, 2 and 3 Lbs./ton dosages. The slurry had 25% solid content by weight with 75-ml water content. The percentage concentration of the reagent in the 100-ml slurry was calculated as the ratio of the volume of the EGMO added into the slurry in terms of ml, to the volume of water in the slurry. According to this calculation the added reagent concentrations at all the dosages that were used for the filtration tests were less than 1%. The surface tension measurements conducted on the filtrates of the slurries gave quite high values with respect to the results of the tests conducted in the absence of the coal particles in the system. This implies that the reagent was transferred onto the coal surface and adsorbed effectively. As a result, less amount of reagent was left in the solution and the liquid surface tension value increased. The surface tension of the filtrate of the base test was 73.0 dyne/cm, which was very close to the surface tension of water. It can be concluded that the entire kerosene and MIBC were adsorbed onto the coal particles in the slurry at the used dosages to create the flotation conditions. However, when the dewatering aid was added into the slurry the surface tensions of the filtrates were started to decrease. The reduction in the surface tension indicated that some of the reagent was left in the solution. At high dosages of the dewatering aid in the system, the excessive amount of the reagent left in the solution helped decreasing the liquid surface tension. Although there were some deviations in the surface tension values of the filtrates, the results of these tests were beneficial to show that the EGMO adsorbed onto the coal surface. This adsorption created an increase in the surface contact angle and the excessive amount left in the solution helped decreasing the liquid surface tension. Both of these factors are very favorable to decrease the cake moisture content and improve the dewatering efficiency. As a result, the good performance of the novel dewatering aids in fine coal dewatering can be explained based on their combined effect on increasing θ and decreasing γ , which improves the surface chemistry of the system significantly.

4.4. Conclusions

- 1. The novel dewatering aids decreased the liquid-air surface tension of the coal slurry effectively at reasonably low concentrations. In the presence of the solid phase in the system the additive was transferred and adsorbed onto the coal surface and the surface tension value of the liquid phase increased. However, the excessive amount of the reagent that was not adsorbed but left in the solution was still effective in decreasing the surface tension of the slurry up to some extent.
- 2. The adsorption of the reagent onto the coal surface increased the solid-liquid contact angle. The increase in the contact angle was larger in the presence of the novel dewatering aid EGMO compared to kerosene, which is a commonly used collector in coal flotation. The best improvement in the contact angle of the coal surface was observed in the presence of the flotation reagents in the system with the novel dewatering aid. The pre-treatment of the coal surfaces with flotation is very critical in terms of the efficiency of the dewatering. The filter cakes were observed to have lower moisture contents when they were floated and had fresh surfaces, which was supported by the results of the contact angle tests showing a significant increase in the contact angle for the floated coal samples.
- 3. The novel dewatering aids were effective both in decreasing the surface tension of the liquidair interphase and increasing the contact angle of the solid-liquid interphase. This effect of the novel reagents improved the dewatering efficiency of fine coal and as a result, low moisture content filter cakes were produced in the presence of these reagents in the vacuum filtration and the centrifugation applications.

Chapter 5. CONCLUSIONS

The results of this investigation are summarized as follows:

- 1. The novel dewatering aids developed at Virginia Tech increased the efficiency of the fine coal dewatering. The moisture contents of the coal cakes were decreased approximately 50% in the presence of the novel additives compared to the results of the control tests. It was also observed that the performance of the reagent is strongly affected by the characteristics of the coal sample, such as the particle size distribution, ash content and surface oxidation.
- 2. The vacuum filtration tests showed that the efficiency of vacuum filtration increases with increasing reagent dosage, drying cycle time and slurry temperature and decreases with the increasing cake thickness (which varies depending on the volume of the slurry). The statistical analyses also indicated that the thickness of the cake plays the most significant role in moisture content reduction. There is a correlation between the reagent dosage, slurry temperature and the drying cycle time. If all are increased simultaneously the cake moisture decreases very extensively. The moisture contents of the filter cakes were decreased to as low as 2 to 5% for the thin cakes at high slurry temperature (60°C), reasonably long drying cycle time (2-5 minutes) and about 2 to 5 Lbs/ton of reagent addition.
- 3. The novel dewatering aids improved the efficiency of the centrifuge filtration as well. Using the centrifuges, the moisture contents of the fairly thick cakes (0.5 to 3 inches) were decreased successfully. However the significance of the reagent usage diminished at very high centrifugal force levels. This can be attributed to the excellent dewatering capabilities of the centrifuges at high G force level even without using any reagents.
- 4. The surface analyses conducted in the presence of the novel dewatering aid EGMO showed that the surface tension of the slurry decreased while the contact angle of the coal surface increased in the presence of this additive in the dewatering system. This observation clearly explains the reason of high performance of the novel additives in increasing the dewatering efficiency.
- 5. The utilization of the novel dewatering aids developed at Virginia Tech is believed to be an effective solution to the problems in fine coal dewatering. As a future work, these reagents are planned to be tested in pilot plant applications.

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APPENDIX I.

STATISTICAL ANALYSES EXPERIMENTAL RESULTS

	Slurry Temperature	Slurry Volume	Reagent Dosage	Drying Cycle Time	Cake Moisture
Run Number	(°C)	(<i>ml</i>)	(Lbs/ton)	(min)	(%wt)
1	22	150	3	2	25.60
2	60	150	3	2	18.63
3	22	150	1	2	20.34
4	41	150	2	2	16.30
5	60	200	2	2	24.47
6	22	200	2	2	23.24
7	41	150	2	2	19.43
8	41	150	1	5	19.78
9	41	100	3	2	11.21
10	60	150	2	5	19.44
11	41	150	3	5	11.59
12	41	100	2	5	9.28
13	41	100	2	1	12.87
14	22	100	2	2	14.38
15	41	200	3	2	11.95
16	41	200	2	1	22.30
17	41	150	1	1	13.52
18	60	100	2	2	11.59
19	60	150	2	1	19.85
20	41	200	1	2	23.30
21	41	100	1	2	21.06
22	41	200	2	5	26.13
23	22	150	2	1	16.49
24	60	150	1	2	18.55
25	41	150	3	1	25.08
26	41	150	2	2	19.50
27	22	150	2	5	18.98

Gul Bahar Basim was born on May 4, 1973 in Ankara, Turkey. She graduated from Karsiyaka Gazi High School in Izmir, Turkey in 1990. She then enrolled at Middle East Technical University to study Mining Engineering, where she was graduated with a Bachelors of Science degree in July, 1995. During her undergraduate career she specialized on minerals processing.

In January 1996, she enrolled in graduate school at Virginia Polytechnic Institute and State University to pursue a Master of Science Degree in Mining and Minerals Engineering. She concentrated on surface chemistry and completed her research dealing with the dewatering of fine coal with her graduate advisor Dr. Roe-Hoan Yoon.