EFFECTS OF SURFACE HYDROPHOBICITY AND AIR BUBBLE ENTRAPMENT ON DEWATERING OF FINE COAL PARTICLES

İNCE TANELİ KÖMÜRLERİN SUSUZLANDIRILMASINDA YÜZEY HİDROFOBİSİ VE KABARCIK TUTULMASININ ETKİLERİ

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ABSTRACT

This paper describes the results of experimental works conducted on fine coal samples to find out the role of surface hydrophobicity and air bubble entrapment on dewatering. The test results showed that newly developed reagents increased the contact angle (or hydrophobicity) and decreased the filtrate surface tension, cake formation time and moisture content of the fine particles. When the air bubbles were introduced to the hydrophobic fine coal particles, the cake formation time and moisture content of the filter cake was further reduced, which may be attributed to the fact that micro/nano size air bubbles were entrapped on the surface of the fine particles in a slurry and increased the dewetting characteristics. As a result, this advanced improvement m dewatering of fine particles provides a solution for many coal companies that have had the problems associated with the fine particle dewatering.

ÖZET

Bu çalışma, ince taneli kömürlerin susuzlandırılmasmda yüzey hidrofobisi ve yüzeye kabarcık bağlanmasının rolünün bulunması amacıyla yapılan bir deneysel uygulamanın sonuçlarım vermektedir. Test sonuçlan, yeni geliştirilmiş reaktiflerin malzeme temas açısmı büyüttüğünü, filtrat yüzey gerilimini düşürdüğünü, kek oluşma zamanım azalttığını ve nihayetinde malzeme nem içeriğini azalttığını göstermiştir. İlave olarak sisteme hava verilmesi kek oluşma zamanını ve kekin nem içeriğinin daha da düşmesine neden olmuştur. Bu olay, mikro/nano boyutlu hava kabarcıklarmm yüzeye yapışarak, yüzeyin ıslanmama özelliklerinin geliştirilmesi olarak açıklanabilir. Sonuç olarak, bu geliştirlen teknik kömür şirketlerinin susuzlandırma ile ilgili problemlerine bir çözüm olacaktır.

1. INTRODUCTION

In recent years, production of fine coal has increased with the use of advanced production of surface and underground mining methods. It is estimated that the fine coal represents as much as 20% of total produced coal and most of which has been discarded to refuge ponds because there are no efficient methods (i.e., cleaning and dewatering) for handling the fine coal (Svarovsky, 1991, Asmatulu et.al, 2001a, 2001b). As known, dewatering methods, such as vacuum, pressure, centrifuge, belt press and screen are not effective for the fine particle dewatering. This is because fine coal particles have higher surface area than coarse coal particles, which in turn give a higher moisture content product in the filter cake (Osborne, 1988, Matteson, 1987 and Condie, 1996, 1997).

Almough the costs of fine coal cleaning and dewatering are higher than that of coarse coal, the fine particle processing is an important objective for environmental protection, loss of economic values of fine coal, calorific value improvements, and other conservations. As known, fine coal is generally cleaned by flotation methods (i.e., conventional, column, Jameson cell and jet flotation) at coal preparation plants, so the floated product has more than 70% moisture content, which is inadequate for direct consumption (Couch, 1991, Svarovsky, 1991, Moudgil, et.al, 1989 and Kural, 1994). Therefore, it is necessary to clean and dewater fine particles to obtain better economical and environmental benefits.

In order to solve the problems associated with the fine particle dewatering, several low hydrophile-lipophile balance (HLB) dewatering aids (whose HLB numbers are less than 12) have been developed and successfully tested on the dewatering of the fine coal particles. Experimental studies also showed that when the fine coal particles hydrophobized by the dewatering chemicals were also subjected to air bubbles in a flotation cell, moisture reduction and kinetics of fine coal were further improved. The present study deals with the reducing the moisture content of fine particles and increasing the throughput of current dewatering techniques.

2. THEORY

Thermodynamics: The process of the fine coal dewatering may be shown in Figure 1 at which the contact area of a water droplet (2) decreases on the surface of solid (1) and orients through to air (3). The free energy change per unit area of the surface (AG/dA) can be represented as follow (Adamson, 1997, Meyers, 1988 and Yoon et.al 1997):
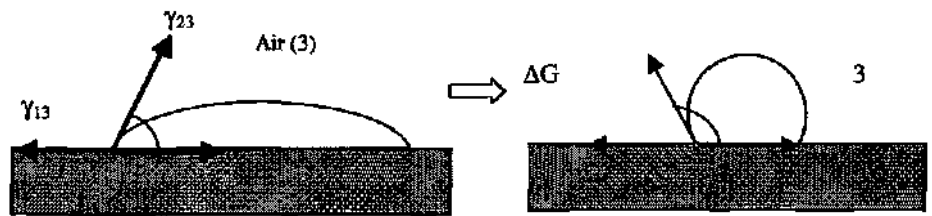

Figure 1. Schematic representation of water displacement from the surface of a coal sample.

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

where γ_{13} is surface free energy of the solid/air interface, γ_{23} is surface free energy of the water/air interface, γ_{12} is surface free energy of the solid/water interface and A is the contact area of solid/water interface. The equilibrium between the three interfacial energies can be expressed by Young's equation (derived in 1805) associated with the contact angle 0 (Adamson, 1997 and Meyers, 1988):

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

Substituting Eq. [2] into Eq. [1], the following relationship can be obtained.

$$\frac{dG}{dA} = \gamma_{23} (1 + \cos\theta)$$ [3]

which suggests that the dewatering can be spontaneous when $\Delta G/dA$ is lower than 0, or contact angle is higher than 180°, which cannot be met in actual dewatering conditions. Thus, one can decrease the work on dewatering (W_d) by adding hydrophobizing chemicals as given in the following equation (Adamson, 1997, Leja, 1982 and Singh, 1997):

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

which expresses that the work of adhesion on the fine particle dewatering can be minimized by decreasing water/air surface tension and increasing contact angle as much as possible (or decreasing contact area of the water droplet on the solid) (Adamson, 1997, Meyers, 1988 and Leja, 1982):

Kinetics: Dewatering of fine particles can be described as a process associated with the flow of water through the porous media created by a bed of particles on the filter media. Darcy derived the rate equation of the dewatering process in 1856 (Kozney, 1927, Svarovsky, 1991, Leonard, 1991):

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

in which V is the volume of fluid, t is the filtration time, ΔP is the pressure drop across the filter cake, L is the cake thickness, A is the cross-sectional area of the cake, η is the viscosity of water, and K is the rate constant known as permeability. The Darcy equation shows that the rate of dewatering is proportional to the applied pressure and the filter area, and is inversely proportional to the viscosity of the process water (Kozney, 1927 and Carman, 1937).

The process of filtration is often related to the flow of liquid through a bundle of capillary tubes in a filter cake. In this case, one can use the Poiseulle's equation derived in 1846 (Svarovsky, 1991):

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

where r is the radius of the capillary.

By combining Eqs. [5] and [6], Kozney obtained the following relationship (Kozney, 1927 and Svarovsky, 1991):

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

where e is the cake porosity, which is dependent on the volume fraction of the void space in the filter cake, S is the specific surface area of the particles per unit volume, and k is a constant which is referred to as Kozney constant. It was reported that k value should be 2 for an ideal filter cake that is a porous medium made of capillary tubes of radius r. However, the test results showed that k was 5 for filter cakes made of monodispersed fine particles. In the industrial application, k was found greater man 5 and could be as large as several thousands in the presence of flocculants (Svarovsky, 1991 and Leonard, 1991).

From Eqs. [5] and [7], the following equation can be received:

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

where a is specific cake resistance of the filter cake. According to this equation, cake permeability decreases with decreasing E and S. Hence, the dewatering rate equation based on Darcy's law gives an explanation about how difficult dewatering fine coal is in the plants.

It is assumed that the filter cake consists of a bundle of capillary tubes with different radiuses. In order to remove the water from the capillary tube of radius r, the applied pressure should be larger than the capillary pressure, p (Adamson, 1997, Meyers, 1988 and Yoon et.al 1997):

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

which is well-known as *Laplace* equation in which ¥& is the surface tension of water/air interface, and 6 is the water contact angle on a solid surface. According to the Laplace equation, p decreases with decreasing ^3, increasing 9 and r, which can be beneficial for a dewatering of the fine particles.

3. EXPERIMENTAL

3.1 Sample

Several different coal samples including Virginia, Pittsburgh and British Columbia-Canada were used for the dewatering tests in the presence and absence of dewatering aids and air bubbles. The samples were dense medium coal (DMC) products, which were crushed, ball mill ground and used for the dewatering tests. Usually, the solid content of coal fed to an air pressure filter was approximately 17%. In order to avoid to surface oxidation of the clean coal, every other week new samples were prepared for the tests.

3.2 Test Procedure

Laboratory Set-Up: The air pressure filter was designed and constructed for this study. It was made of a Plexiglas cylinder with dimensions of 7 cm in diameter and 25 cm in height. As shown

in Figure 2, the bottom of the pressure filter was made of perforated Plexiglas frit, on which a fabric filter medium was placed. The top of the filter was also covered with a Plexiglas lid. Compressed air was injected to the top portion of the pressure filter so that the slurry introduced to the chamber was subjected to the desired pressures, which were varied in the range of 50 to 200 kPa. A known volume of a slurry sample was agitated into a conventional Denver flotation cell of 1000 ml capacity at 1600-rpm speed and then was poured into the filter before the drying cycle time period started. In each experiment, the cake thickness, air pressure, and cake formation time were recorded. After the tests were performed, a part of a representative filter cake was removed, weighed, and dried in an oven overnight. The sample was weighed again after the drying, and the moisture content was calculated from the difference between the dry and wet weights.

In the air bubble effect tests, the slurry was poured into the same conventional Denver flotation machine with the 1000 ml cell. At this time, when a dewatering aid was added to the cell by means of a plastic syringe, 2 minutes of air (generated by the flotation unit) agitation at 1600 rpm was given to the slurry to be able to adsorb the nano/micro size bubbles on the hydrophobic (or hydrophobized) particle surfaces.
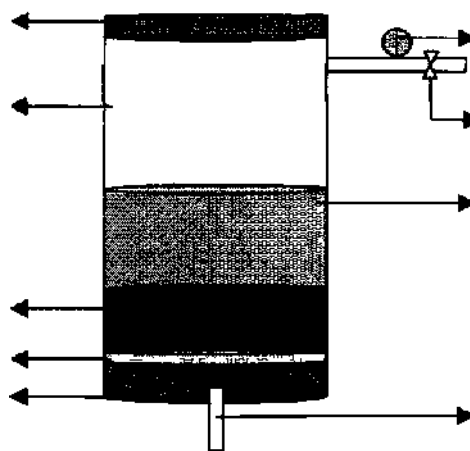

Figure 2. A schematic representation of the 7 cm in diameter pressure filter.

Contact Angle and Surface Tension Measurements: The tests were performed using a Rame Hart Mode! 100 Goniometer and Sigma70 technique. In the previous measurements, the sessile liquid drop technique was chosen to measure the advancing water contact angle (6a) on the solid samples. Before the tests, different coal samples were polished using different sized sand papers (120, 240, 400 and 600 grit) and then a polishing cloth was used with a 0.05 um alumina micro polisher in order to obtain a smooth surface. The coal surfaces were then used to determine the contact angle improvements. To be able to eliminate experimental errors, a small 5 ml size syringe with a needle was utilized for the tests. Also, surface tensions of the filtrate liquids received at the end of each tests were measured using a ring method of the Sigma70 measurement technique.

4. RESULTS AND DISCUSSIONS

4.1 Virginia Coal Sample

Table 1 shows the results obtained on a bituminous coal sample received from Virginia. The coal sample was a DMC that was crushed and ball-mill ground to -0.5 mm. Ash, sulfur and solid contents of the sample were 8.9%, 1.03% and 17.7%, respectively. A 7 cm in diameter air pressure filter was used at an air pressure of 100 kPa, 1.5 cm cake thickness and 2 minutes of drying cycle time. The tests were conducted using a dewatering aid (code name Reagent QX141) with and without air bubble. The sample was conditioned for 2 minutes (optimum time) for the reagent adsorption in the cell. Following experimental parameters were evaluated m this study.

Contact Angle: Contact angle measurements were conducted on the DMC samples using sessile drop technique. Small chunks of the coal samples (rectangular 2x10x10 mm) were polished and put into the screened slurry sample (0.6 mm x 0.038) to condition with the chemicals. For each region coal, five measurements were taken and then the measurements were averaged to avoid experimental errors. It was expected that the values of the contact angle were improved by increasing the amount of the reagent. It is recognized that increasing the hydrophobicity (or contact angle) of the coal particle can be due to the surfactant molecule adsorption on the solid surface. At 0 kg/ton and 4 kg/ton reagent additions, the contact angle of the coal surface was improved from 51° to 91°. Shown in Table 1 are the equilibrium contact angles of the Virginia coal samples.

Table 1. Effects of surface hydrophobicity and air bubbles on the dewatering of Virginia coal samples* (-0.5 mm) using Reagent QX141 at 100 kPa air pressure.

Reagent Dosages (kg/ton)	Contact Angle (Degree)	Filtrate Surface Tension (mN/m)	Cake Formation Time (sec)		Moisture Content (%wt)	
			w/o Air	w/Air	w/o Air	w/Air
0	51	71	53	50	23.7	22.2
0.5	76	66	21	16	17.6	14.1
1	S3	61	16	13	15.8	12.3
2	89	58	15	11	14.2	10.8
4	91	51	14	9	14.9	10.3

* 7 cm in diameter air pressure filter; the sample crushed, ground and used; 2 minutes of air agitation;
2 minutes of conditioning time, particle size 0.5x 0 mm; cake thickness 1.5 cm; solid content 17.7%.

According to the Laplace equation, the pressure in the capdlary tubes should be zero if the contact angle is 90°. This means that water İn capillary tubes in the filter cake can be spontaneously removed without applying air/vacuum pressure. In other words, the water molecules under this condition cannot stay in the capillary tubes. Thus, the contact angle obtained using 4 kg/ton Reagent QX141 would reduce a great deal of capillary pressure in the coal cake. In all of the dewatering tests, it was observed that the applied pressures on the cake were decreased due to the hydrophobicity improvements of the particles.

Filtrate Surface Tension: Filtrate samples were taken at the end of the drying cycle time, and then the surface tension of the process liquid was measured using a ring method of a Sigma70 device. In the absence of die dewatering aid, me surface tension of the filtrate was 71 mN/m, which was very close to pure water surface tension (72 mN/m). As the reagent concentration was increased, die surface tension of the liquid was decreased to lower levels. With a 4 kg/ton reagent addition, the surface tension of die liquid was dropped to 51 mN/m. Note that the surface tension of die Reagent QX141 is around 26 mN/m and less Üıan 0.05% of the reagent decrease the filtrate surface tension to this level. Thus, it may be concluded that most of the surfactant molecules adsorbed on the fine particles.

Cake Formation Time: This dewatering period was ended when a bulk of the water was passed through the fdter cake surface. At die end of this period, die drying cycle time starts for a couple of minutes, depending on the particle size and die types of dewatering machines (Svarovsky, 1991). Several experiments were conducted on the fine coal samples to determine the cake formation times. The test result confirmed that in the presence of die reagents, the cake formation time was substantially decreased. For example, the cake formation time of 0 kg/ton and 4 kg/ton reagents were 53 sec and 14 sec, respectively. However, in the presence of air agitation to tiie slurry die cake formation times were 50 sec to 9 sec, which may be attributed to die fact mat nano/micro size air bubbles were entrapped on tiie fine particles and improved die dewatering kinetics. Hence, the cake formation time of die reagent tests was approximately five times lower tiian diat of die base case. This indicates that die change in die contact angle, surface tension, air bubble entrapped and die otiier cake parameters strongly affect die cake formation time.

Moisture Content: Moisture content is major concern for a filter cake before further drying it in thermal dryers. According to this finding, it is assumed tiiat the moisture reduction can also be dependent on die contact angle (or hydrophobicity) improvements on the fine particles. As previously stated, in the presence of die Reagent QX141, the contact angle was increased from 51" to 91°. Therefore, this should reduce die breakthrough pressure, cake formation time and dewettability of the fine particles, and increase me moisture reduction in die filter cake. In die base study, the product moisture was 23.7% at 100 kPa air pressure; in contrast, when a 4 kg/ton reagent was added to the same slurry, it was reduced to 14.9% where the contact angle was 91° at die solid/liquid/air interface.

In addition to the surface hydrophobization, as die slurry was agitated at 1600 rpm speed of the Denver flotation cell with air bubbles, 3-4% lower moisture content of filter cake was received at tiie same conditions. The reason is mat when die slurry was mixed witii air bubbles and dien formed on the filter media, diese bubbles could replace the water film adhering between die fine particles and make die cake dryer. Consequently, this result may provide that air agitation can help lower the moisture content of hydrophobized fine particles, which may be due to the microscopic size air bubble entrapment on the fine particles.

4.2 Pittsburgh Coal Sample

The tests were conducted using a 7 cm in diameter air pressure filter at a 100 kPa air pressure with a 2 minute drying cycle time and 1.5 cm cake tiiickness. Ash and sulfur contents of the

sample were 8.3% and 1.17%, respectively. The other test parameters were the same as the previous test. Reagent QX142 used in the present work was nonionic surfactants with less than 10 HUB number. The reagent was insoluble in water; therefore, they were used as solutions in appropriate solvents. For one part of the dewatering aid, two parts of solvents were added to make the solution. The test results given in Table 2 were obtained on a Pittsburgh dense medium coal sample, which was pulverized to -1.0 mm and then used for the tests.

It is seen that the low HLB surfactant constitutes an important group of dewatering aids for fine coal dewatering. For instance, at a 4 kg/ton reagent, contact angle of the coal sample was increased from 45° to 89° which represents twice as much as the base case; meanwhile, surface tension of the filtrate was decreased from 71 mN/m to 54 mN/m. Besides, when the same amount of reagent was added to the pulp, cake formation times were significantly reduced from 48 sec to 14 sec. Further improvement İn cake formation time was also seen when air bubbles were incorporated to the hydrophobic particles.

The moisture content was the main objective of the test work. As seen, when the reagent dosages were increased from 0 kg/ton to 4 kg/ton, the moisture contents of the filter cakes were gradually decreased. For example, the moisture was reduced from 18.7 to 10.7% at 4 kg/ton of reagent addition, which represents approximately 43% reduction. However, at the same dewatering condition if air bubbles were given to the pulp during the conditioning time, this reduction was enhanced up to 58% comparing to the base tests obtained without air addition to the pulp. The test results indicted that the use of the dewatering aid and air bubbles decreased the cake moisture contents below 8% levels for the fine coal sample, which in turn could replace the thermal dryers used in preparation plants.

Table 2. Effects of surface hydrophobicity and air bubbles on the dewatering of Pittsburgh coal samples* (-1 mm) using Reagent QX142 at 100 kPa air pressure.

Reagent Dosages (kg/ton)	Contact Angle (Degree)	Filtrate Surface Tension (mN/m)	Cake Formation Time (sec)		Moisture Content (%wt)	
			w/o Air	w/Air	w/o Air	w/Air
0	45	71	48	47	18.7	16.3
0.5	72	67	22	19	13.1	10.0
1	79	63	17	15	12.0	8.6
2	84	60	16	12	10.8	8.1
4	89	54	14	10	10.7	7.8

* 7 cm in diameter air pressure filter; the sample crushed, ground and used; 2 minutes of air agitation; 2 minutes of conditioning tune, particle size 1 mm x 0; cake thickness 1.5 cm; solid content 17.7%.

4.3 British Columbia Coal Sample

The dense medium coal sample received from the operation plant located in British Columbia-Canada was crushed and ball mill ground to -0.6 mm before the tests. It was found that ash, sulfur and solid contents of sample were 7.2%, 0.87% and 17.3%, respectively. The dewatering

aid - code named as a Reagent QX143 was a low HUB hydrophobizing agent and used in the tests after dissolving in an appropriate solvent. The results obtained using the air pressure filter at 100 kPa air pressure, 2 minute drying cycle time and 1.5 cm cake thickness was given in Table 3.

Table 3. Effects of surface hydrophobicity and air bubbles on the dewatering of British Columbia coal samples* (-0.6 mm) using Reagent QX143 at 100 kPa air pressure.

Reagent Dosages (kg/ton)	Contact Angle (Degree)	Filtrate Surface Tension (mN/m)	Cake Formation Time (sec)		Moisture Content (%wt)	
			w/o Air	w/Air	w/o Air	w/ Air
0	48	71	51	47	20.7	18.8
0.5	75	66	20	18	15.2	13.2
1	81	60	16	13	13.4	10.7
2	86	58	14	11	12.5	9.6
4	90	53	13	9	12.3	9.0

* 7 cm in diameter air pressure filter; the sample crushed, ground and used; 2 minutes of air agitation; 2 minutes of conditioning time, particle size 0.6 mmxO; cake thickness 1.5 cm; solid content 17.3%.

The second, third and forth columns of Table 3 were associated with contact angle, filtrate surface tension and cake formation time, which are important parameters in obtaining lower moisture content of a filter cake. It is seen that increasing reagent dosages from 0 kg/ton to 4 kg/ton also increased contact angles from 48° to 90° and decreased filtrate surface tensions (i.e., from 71 mN/m to 53 mN/m) and cake formation times (i.e., from 51 sec to 13 sec). It was found in the present study that the cake formation time was also dependent on the entrapped air bubbles on the fine coal particles. For instance, the cake formation time of the slurry was 3-4 seconds lower than the previous cake formation time received without air bubble addition.

Table 3 also shows a comparison of moisture contents in the presence and absence of air bubble addition on the hydrophobic fine coal particles. All the tests conducted on the British Columbia coal sample showed that hydrophobicity enhancement on the fine particles significantly decreased the moisture content of the filter cake. However, hydrophobicity plus air bubble attachment on the fine particles gave approximately 3% lower moisture content of filter cake as compared to the former test These two parameters can also affect the hydrophobic coagulation where the fine particles can come together and act as larger particles, helping lower the overall moisture content It is obvious that the moisture content below 10% can help solve the fine coal uewatering in the preparation plants where thermal dryers are extensively used.

5. MODEL DEVELOPMENT

The final moisture content remained in a filter cake varies depending on the particle size, applied pressure, drying cycle time, structure of filter cake, properties of solid and liquid, solid/liquid interfacial tension, temperature, etc (Svarovsky, 1991, Matteson, 1987 and Leonard, 1991). In addition, it was found out in this study that the efficiency of dewatering could be strongly related to the surface modification of the fine particles. In other words, the surface hydrophobicity and air bubble entrapment could also affect the dewatering of fine particles. It is assumed that when the particles are hydrophobic enough in water, microscopic bubbles can attach on the particle surface and make them nonwettable. According to this explanation, the process of the fine coal dewatering may be shown in Figure 3 depending on the surface hydrophobicity and air bubble entrapments.
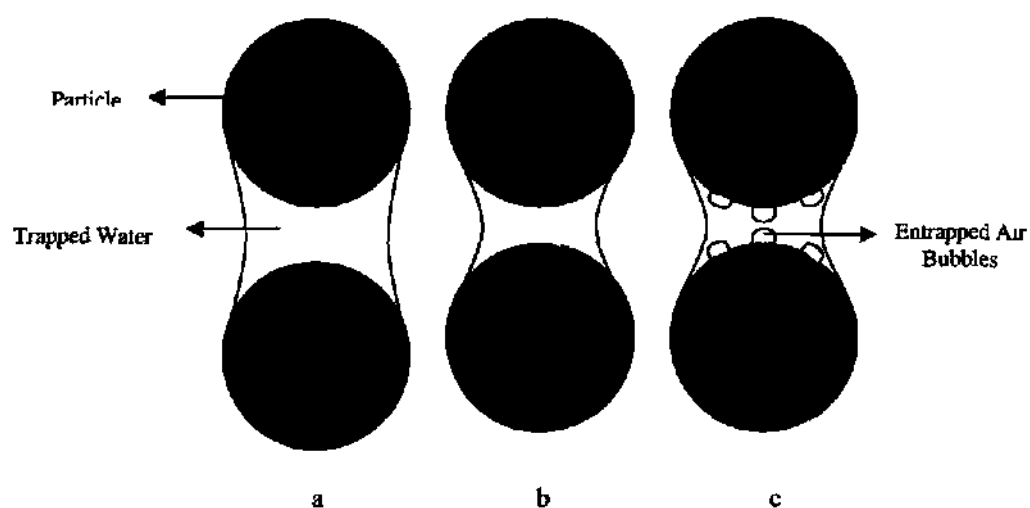

Figure 3. Schematic illustration of a possible dewatering process with a) two spherical particles, b) hydrophobized particles and c) hydrophibized particles with air bubble entrapment

As seen, unmodified two particles (Figure 3 a) are in capillary state with a film of trapped water, which is obtai"ned after the pressure is applied on the filter cake. This situation is called equilibrium saturation with a larger meniscus of water between the particles, which may give higher moisture content of filter cake. In the second case (Figure 3 b), this meniscus becomes smaller than the previous one, which may be due to the fact that the hydrophobized fine particles are less wettable in the slurry and give lower moisture content because hydrophobic particles does not appreciate the water molecules on the surface. Finally, the third case (Figure 3 c) may be both the hydrophobicity and air bubble entrapment effects on the filter cake. It is seen in this model that micro/nano size air bubbles gather in between two particles and decrease the total moisture content of two particles. This may be a reason of the reducing cake formation time and overall moisture content of the filter cake.

6. CONCLUSIONS

According to the Laplace equation, achieving the lower cake moistures from fine coal particles during filtration is based on the surface tension lowenng, capillary radius enlargement, and contact angle improvements. The capillary radius enlargement can also be dependent on the hydrophobic coagulation. Consequently, the dewatering chemicals probably control all these parameters and give such a high moisture reductions.

In addition, the dewatering kinetics of the sample was also increased approximately 4 times higher than the base case because of the surface hydrophobicity of the coal particles. For example, the base tests and 4 kg/ton reagent tests gave around 50 seconds and 10 seconds cake formation times, respectively. This means that the capacity of the filtration machines can be gradually increased in the preparation plants.

More interesting results were received when the coal slurry was agitated with air bubbles in a floatation cell. In other words, higher kinetics and lower moisture content can be obtained with air bubble entrapment on the hydrophobized fine particles. The reason İs that if the particles are hydrophobized with the dewatering aids (see the contact angle improvements), micro/nano size fine bubbles can attach onto the hydrophobic particles and make them nonwettable in the pulp. According to the model developed in this study, this can give an advantage in lowering the moisture content of the filter cakes. Overall, using hydrophobization and air bubble entrapment procedures, throughput of the filters can be improved and dewatering costs and environmental concerns of the thermal dryers can be reduced for the industry.

More detailed studies are necessary to détermine the effects of hydrophobicity and air bubble entrapment on the processing of filtration, flotation, coagulation, flocculation, selective flocculation, sedimentation and other wet methods of fine coal, mineral and colloidal particles.

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