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H. B. Gala^a; R. Kakwani^a; S. H. Chiang^a; J. W. Tierney^a; G. E. Klinzing^a

^a CHEMICAL AND PETROLEUM ENGINEERING DEPARTMENT, UNIVERSITY OF PITTSBURGH, PITTSBURGH, PENNSYLVANIA

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Filtration and Dewatering of Fine Coal

H. B. GALA, R. KAKWANI, S. H. CHIANG, J. W. TIERNEY, and
G. E. KLINZING

CHEMICAL AND PETROLEUM ENGINEERING DEPARTMENT
UNIVERSITY OF PITTSBURGH
PITTSBURGH, PENNSYLVANIA 15261

ABSTRACT

A fundamental study on filtration and dewatering of fine coal is described. Experiments are being conducted in three areas: (1) The microscopic analysis of filter cakes, (2) The measurement of equilibrium desaturation and (3) The determination of filtration and dewatering rates. Preliminary experimental results are presented together with some observations on the microstructure of filter cakes. A three dimensional network model has also been developed and is being used to analyze experimental data.

INTRODUCTION

Filtration is an operation in which undissolved solid particles are separated from liquid by forcing a slurry (suspension of particles in the liquid) through a porous medium. The porous (filter) medium allows the liquid (filtrate) to pass through, but

retains the solids. Initially, particles smaller than the pores of filter medium pass through it, but as the filtration proceeds, the particles retained on the filter medium will form a porous cake which will then capture the smaller particles. This filtration operation is known as the cake filtration. Dewatering is a post-filtration process⁽¹⁾ in which the filtrate is removed from the void space of the filter cake by application of desaturating forces. In many cases, dewatering is achieved by displacing filtrate with air. A very important distinction between the two processes is that while filtration is a single phase flow process, dewatering is a two-phase flow process.

In the past, filtration was not studied from a theoretical viewpoint as other unit operations, such as distillation and absorption. Also, dewatering was not treated as a separate process, but was considered as a part of filtration. Design of a filter unit was largely based on empiricism. Some attempts to understand the fundamental mechanisms underlying filtration have been made in recent years.⁽²⁻¹¹⁾ A review of filtration and dewatering has been prepared by Gala and Chiang.⁽¹²⁾

Dewatering of fine coal (less than 500 μ m) is one of the major problem areas in coal preparation plants. In search of solutions to this problem, a fundamental study of filtration and dewatering operation has been initiated under the sponsorship of the United States Department of Energy. In this

study, a number of variables (as listed in Table 1) will be carefully examined in terms of their effects on dewatering of fine coal slurries.

OBJECTIVES

The overall objective of this study is to seek improved methods of dewatering through a better understanding of the filtration and dewatering process. The investigation is divided into four areas.

Particle and Filter Cake Characterization

The objective of this part is to relate the size distribution of particles in a coal sample to the properties of filter cakes formed from the particles. The structure of a filter cake will depend largely on the packing of the coal particles for a given set of operating conditions. In order to develop a correlation between particle characteristics and the cake structure, micrographic analyses of coal samples (to determine the particle size distribution and particle shape) and filter cakes formed from these samples are performed. Porosity and pore size distribution of filter cakes are related to the coal particle size distribution and particle shape. One such attempt to study the structure of packed bed was made by Ward, et al.⁽¹³⁾ Their results on a sedimented bed showed some definite evidence of particle arrangement in a direction normal to sedimentation. Microscopic analysis of particles

TABLE 1
Variables Involved In Filtration/Dewatering of Fine Coal

Coal	Particle Characteristics	Cake Characteristics	Cake Formation Technique	Operating Parameters
Physical Properties	Particle size Size distribution	Macro-structure Segregation	Direction of Filtration	Concentration of solids in slurry
Chemical Properties	Shape	Gross Defects Micro-structure	Settling of Particles	Driving force -vacuum -Pressure -Centrifugal
Refuse Content	Pore size distribution	Pore size Pore shape	-Natural -Forced Delayed Cake Formation	Cake thickness pH of slurry
	Porosity		Staged Cake Formation	Temperature Chemical Additives

and filter cake is carried out with an optical microscope and image analyzer. A description of this equipment is given in a later section.

Modelling of Filtration/Dewatering Process

The purpose of a simulation model developed for filtration and dewatering of fine coal particles is for the prediction of the equilibrium and the dynamic properties of a filter cake using characteristic properties of the particles and cake as well as the operating conditions. Filtration involves flow of a single phase through a porous medium; dewatering involves simultaneous flow of two phases. Neither is subject to simple theoretical treatment. Any simulation model used for dewatering must be able to represent a statistical variation in the porous structure. In this work, a three-dimensional network model is being used for the first time to represent the structure of the filter cake.⁽¹⁴⁾ Network models were first used by Fatt.⁽¹⁵⁾ The model, when fully developed, will have the capability of predicting both the equilibrium and the dynamic dewatering properties of the filter cake. In the model, the cake structure is represented by a regular three-dimensional lattice, formed by a set of nodes and bonds. Four different types of lattices, body centered cubic, face centered cubic, tetrahedral and simple cubic, are considered. The study of immiscible displacement in the network is done by Monte

Carlo displacement in the network of size 15x15x15 (3375) nodes has been used successfully to calculate the breakthrough pressure and is now being tested for equilibrium desaturation. Details of the model and results obtained from application of this model are discussed by Riquelme.⁽¹⁴⁾

Equilibrium Measurement

Equilibrium properties are important because they provide the limiting achievable performance of a filtration and dewatering process. The residual moisture content, capillary pressure-saturation curve, and breakthrough pressure of filter cakes are measured. Breakthrough pressure is the pressure at which the non-wetting phase (air in this case) starts to flow continuously through the filter cake. Capillary pressure-saturation data provide an experimental measurement of the relation between equilibrium moisture and the applied pressure difference. This information, together with microscopic data, is required in modelling of dynamic properties of the filter cake, and it also gives a measure of the effect of capillary forces on the retention of water in the filter cake.

Dynamic Measurement

Dynamic measurements are being conducted to obtain rate of filtration and dewatering. The effects of parameters, such as change in driving force

(vacuum), change in slurry concentration, particle size, cake thickness, have been studied. These results, together with equilibrium measurements and micrographic analysis, provide a basis for testing the network model, understanding the underlying mechanism of filtration/dewatering, and predicting performance of filtration units.

EXPERIMENTAL WORK

Micrographic analysis of particles and filter cake is carried out with a Leitz Orthoplan microscope and an Omnicron Alpha Image Analyzer. These equipments are coupled with a HP-85 mini-computer, so that the entire operation can be carried out automatically.

For particle size analysis, a representative sample of the coal (-32 mesh, Pittsburgh-Bruceton Seam) is carefully drawn from the bulk. The coal sample is then mixed with a mounting medium (oil or isopropanol), subjected to ultrasonic vibrations to break up any agglomerates and transferred to a clean glass slide for microscopic examination. Coal particles in several fields of view are counted through the microscope by the image analyzer (as directed by the computer) and analyzed by the computer to obtain a size distribution of the particles. At least 10,000 particles are counted to obtain a statistically representative analysis. A typical size distribution of -32 mesh coal particles (Pittsburgh

coal) obtained in this way is shown in Figure 1. As can be observed, there is a very wide distribution of particle sizes, ranging from a few microns to 500 microns.

Selected filter cakes are consolidated by displacing the residual water with methyl-ethyl-ketone (MEK) and then passing an epoxy resin (Armstrong epoxy) diluted with MEK through the cake. The consolidated cake is cured in an oven at 60 C., cut by an Isomet diamond saw to obtain a predetermined cross-sectional view of the cake, ground on a Buehler Handimat grinder and polished with a rotating wheel. The polished specimen is viewed under the optical microscope directly or photographed using a bellow

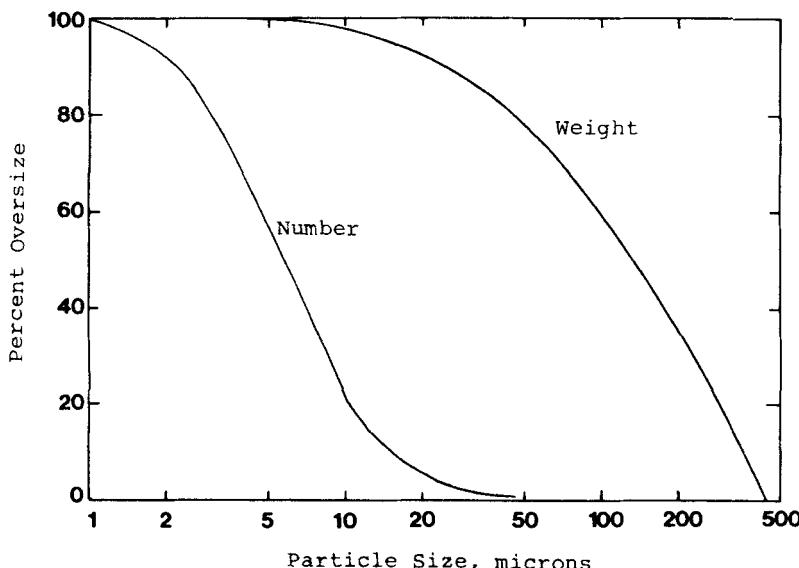


FIGURE 1: Particle Size Distribution of -32 mesh Pittsburgh-Bruceton Seam Coal.

camera with high speed Polaroid 52 film and then the photomicrograph is viewed under a macroviewer which is coupled with the Image Analyzer. Figure 2 shows the surface layer near the top section of a filter cake and Figure 4 the pore size distribution obtained for this section. Figure 3 shows a layer of the cake near the bottom section (i.e. near the filter medium) with its pore size distribution as shown in Figure 4. This cake was formed under gravity settling.

An examination of Figures 2 and 3 shows that segregation of particles occurs due to differences in rate of sedimentation. A wide variation in average particle diameter is observed from one layer of the cake to another.

One of the interesting features noted is the presence of air bubbles in the filter cakes formed under vacuum. Figure 5 shows a micrograph of a layer of coal cake close to the filter medium. Air bubbles are clearly visible in this picture as black circular voids. These air bubbles might have been evolved from filtrate or desorbed from the surface of coal particles. Entrapped air bubbles have also been observed by Nemeth and Sirois.⁽¹⁶⁾ The air bubbles are undesirable since they might impede the filtration and dewatering by blocking off micropores and channels through which water would flow otherwise.

Capillary pressure saturation experiments are carried out in standard Ruska cells⁽¹⁷⁾ by measuring

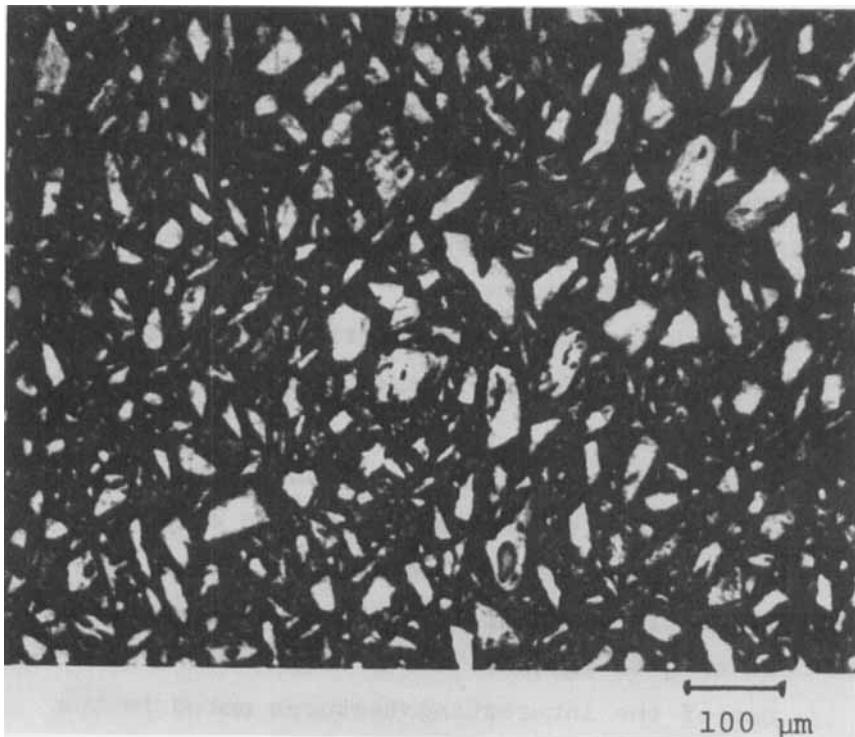


FIGURE 2: Photomicrograph of top layer of a filter cake formed from -32 mesh coal particles (Pittsburgh-Bruceton Seam Coal Mine). Average particle diameter of this layer = $36.6 \mu\text{m}$, Average cake porosity of this layer = 0.54 ± 0.03 .

the volume of water displaced at a known pressure drop across the cake. Cake saturation is calculated by a water balance. Figure 6 shows a typical capillary pressure-saturation curve for coal cakes. Breakthrough pressure through the cake is measured by recording the pressure at which the first air bubble escapes through an inverted cake. It is found

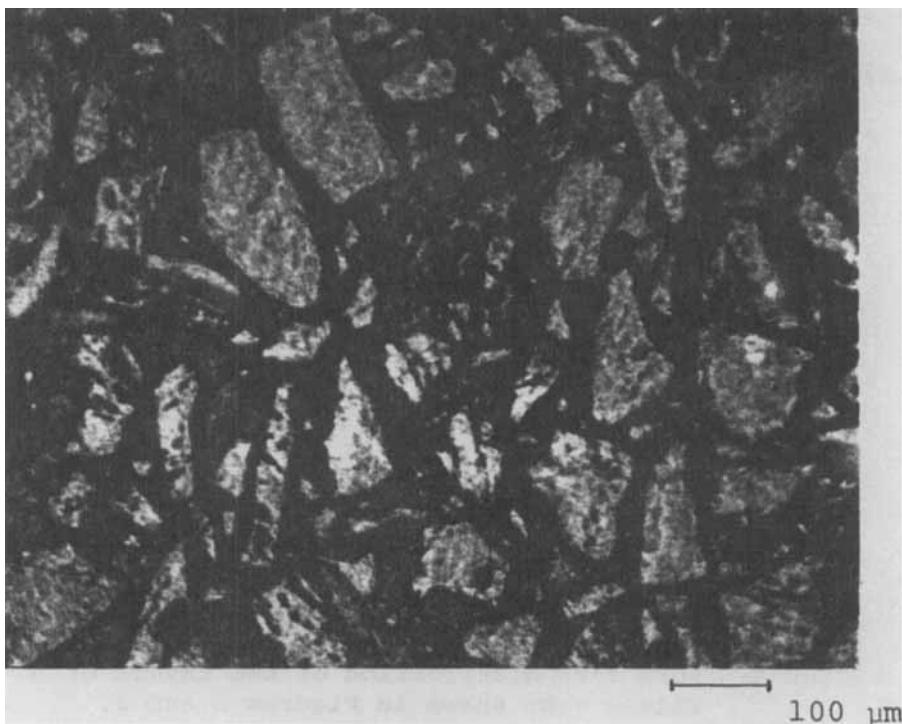


FIGURE 3: Photomicrograph of bottom layer of a filter cake formed from -32 mesh coal particles (Pittsburgh-Bruceton Seam Coal Mine). Average particle diameter of this layer = $57.1 \mu\text{m}$. Average cake porosity = 0.49 ± 0.03 .

that air breakthrough occurred in -32 mesh coal cakes at about 6 to 7 kPa pressure and is independent of the cake thickness.

For dynamic measurements, a filtration unit made of a rectangular Plexiglas box and a Plexiglas cylinder is used. This unit, as shown in Figure 7, contains a load cell and transducer system (both ob-

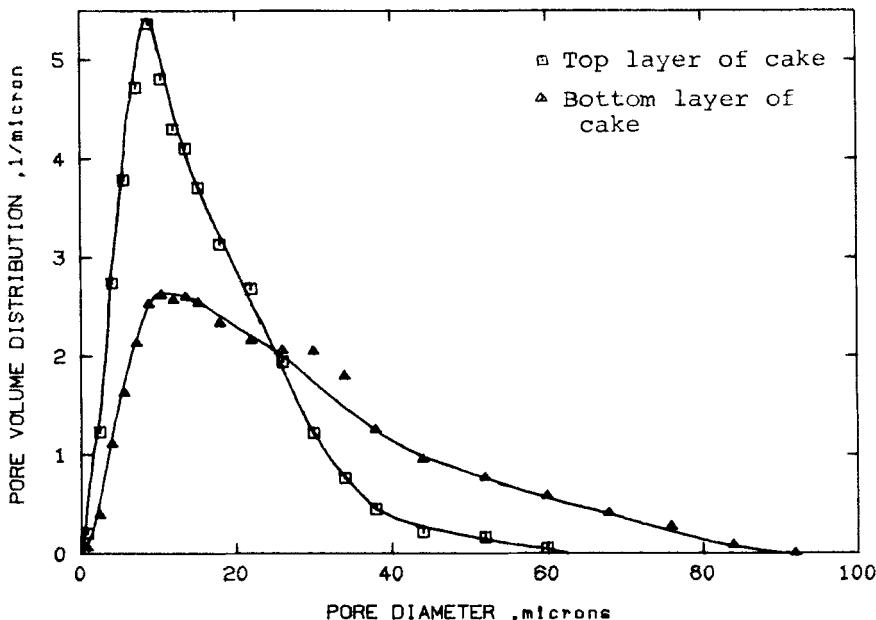


FIGURE 4: Pore size distribution of two layers of a filter cake shown in Figures 2 and 3. Y axis shows % volume occupied by pores in a size interval divided by the size interval.

tained from Gould-Statham Company). A container for collecting the filtrate passing through the filter cake is placed on the load cell. An electrical signal is generated for a change in the weight of the container. The electrical signal is recorded to provide a continuous record of weight of filtrate vs. time. From these records, filtration and dewaterring rate data are obtained.

To ensure that there is no seepage of water around the periphery of the coal cake during filtration measurements, the cake is formed in a rubber

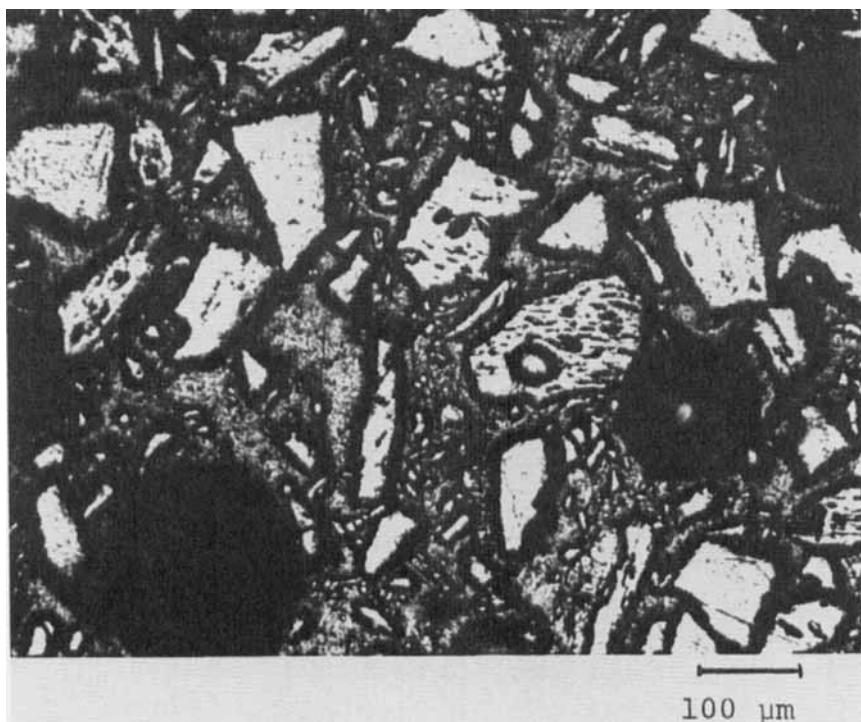


FIGURE 5: Photomicrograph of a section of filter cake near the filter medium showing the presence of air bubbles. (The cake was formed under vacuum.) Black circles in the picture are air bubbles.

sleeve mounted inside the Plexiglas cylinder. The sleeve sticks to the inside surface of the cylinder on application of vacuum and it expands and holds the coal cake firmly when vacuum is released.

Experiments are carried out by mixing a known amount of coal with distilled water to form a slurry. The slurry is then filtered through a Whatmann #1 filter paper in the cylinder, by application of

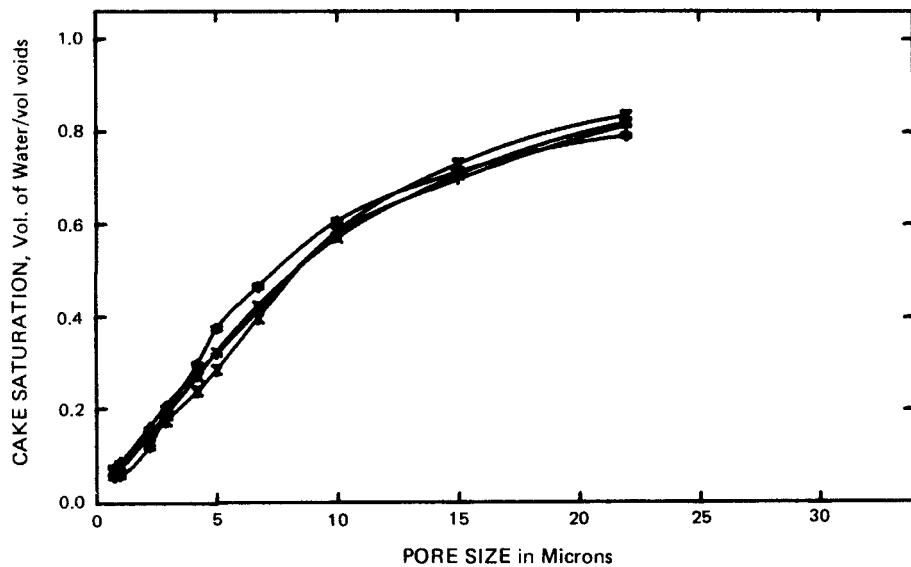


FIGURE 6: Capillary pressure-saturation curve for four coal cakes (formed by gravity settling from -32 mesh coal samples).

vacuum. The weight of filtrate collected is recorded as a function of time. Figure 8 shows a typical filtration curve and Figure 9 shows the results obtained by analyzing several filtration/dewatering curves obtained by varying the vacuum. The specific cake resistance and filter medium resistance are calculated by application of the parabolic filtration equation to the filtration data.⁽¹⁹⁾ As is observed in the figure, vacuum has little effect on cake resistance. On the other hand, the moisture content of cake decreases with an increase in driving force (vacuum) as expected.

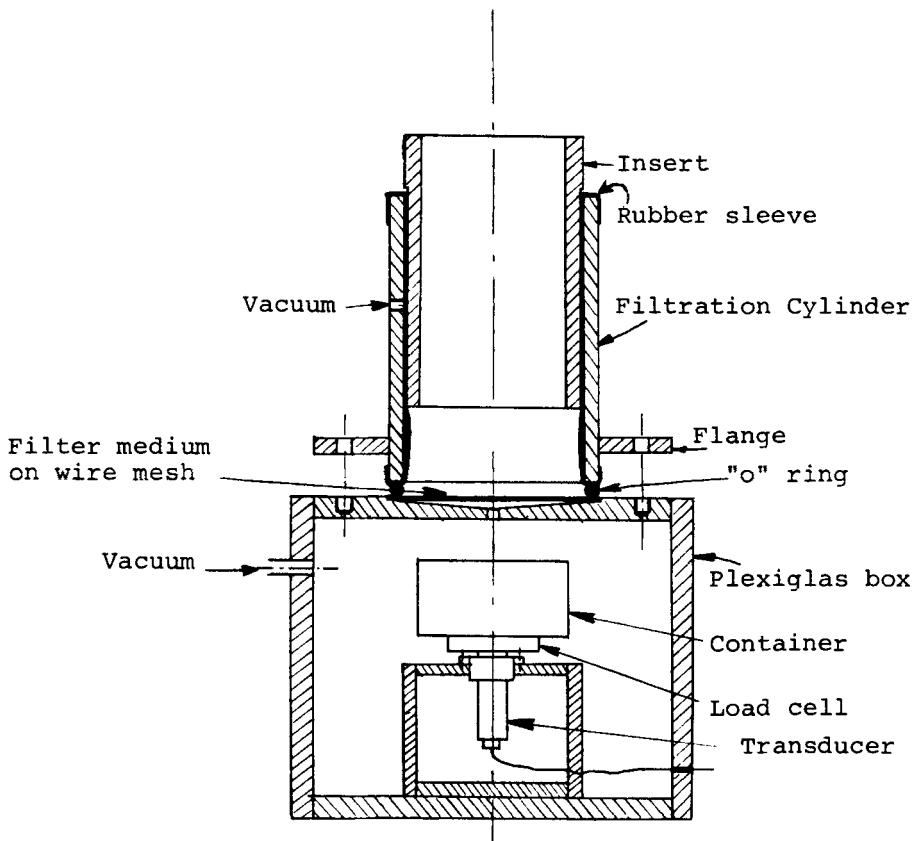


FIGURE 7: The filtration unit, showing the rubber sleeve arrangement and the weighing assembly.

OBSERVATIONS AND CONCLUSIONS

Micrographic analysis of fine coal filter cakes reveals detailed structural features which have not been observed before. For cakes formed by gravity settling, significant variations in average particle size and pore size distribution are noted at various

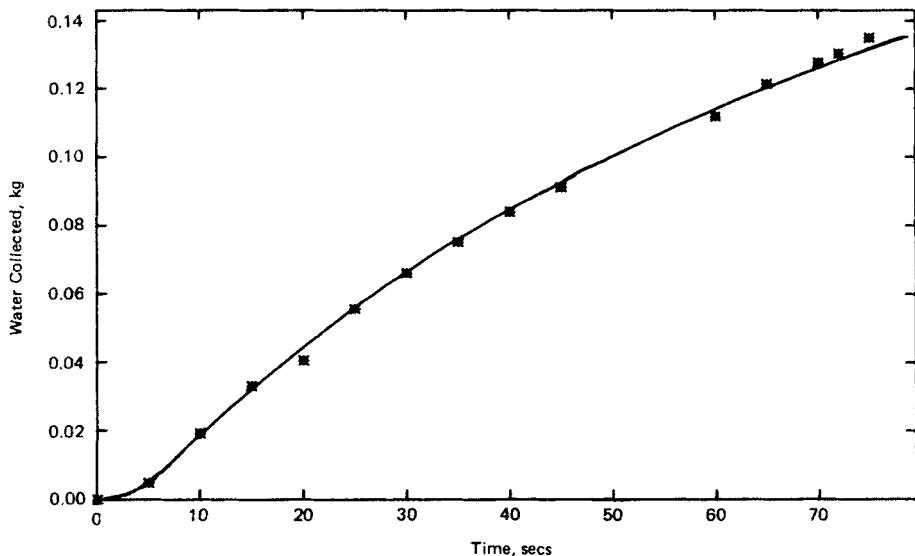


FIGURE 8: A typical filtration curve.

Solids: -32 mesh coal particles
Slurry concentration of solid: 0.25
kg/kg water
Applied pressure gradient: 40 kPa

layers along the direction of filtration. Similar variations are also observed in cakes formed under vacuum. These variations should be included in any model for the filtration/dewatering process. The network model used here can include the effect of particle segregation on the dewatering process. Figure 10 shows a plot of node saturation versus number based pore size for an equilibrium desaturation calculation using the network model.⁽¹⁴⁾ These are generalized properties and cannot be compared directly with experimental results such as shown in Figure 6, but the similarity of the curves is evident.

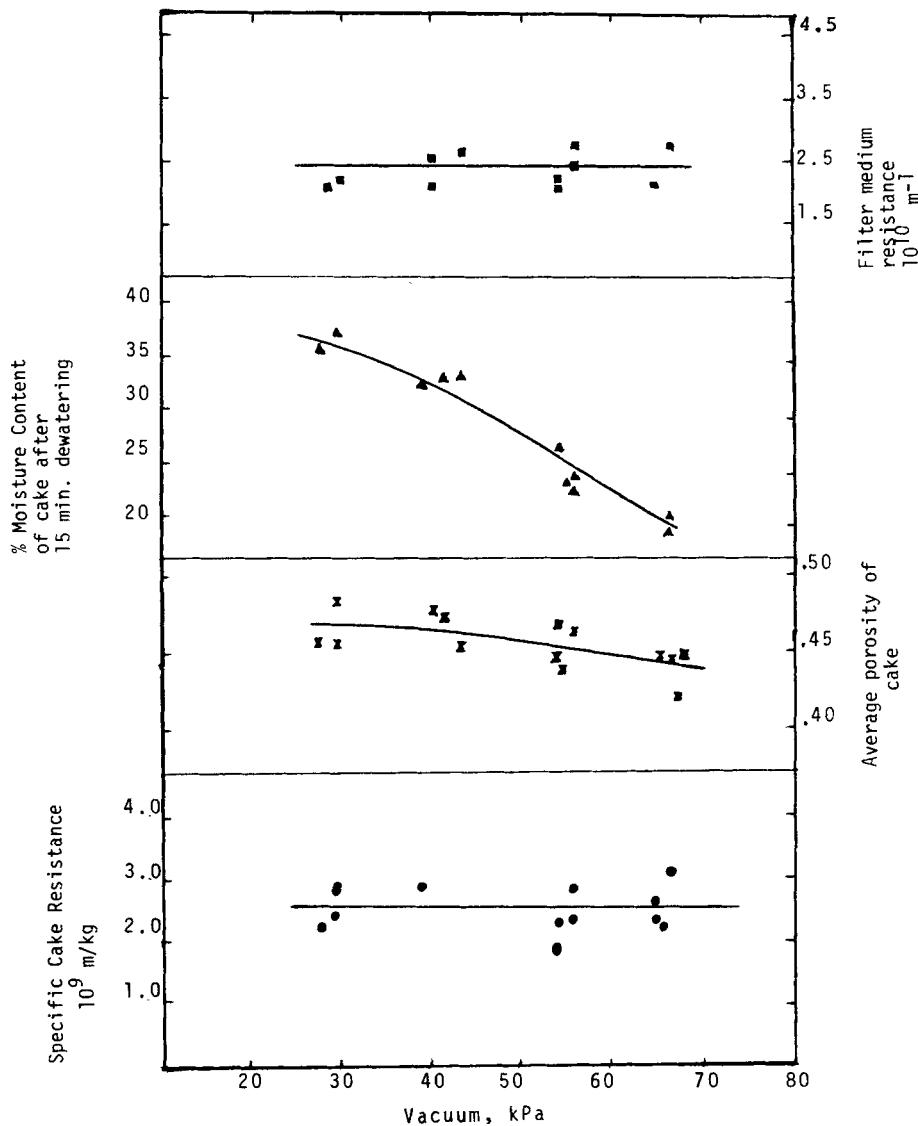


FIGURE 9: Effect of vacuum on properties of a coal cake.

Particle size: -32 mesh

Slurry concentration of solids: 0.25
kg/kg water

Cake thickness: 3 cms.

Cake diameter: 4.8 cms.

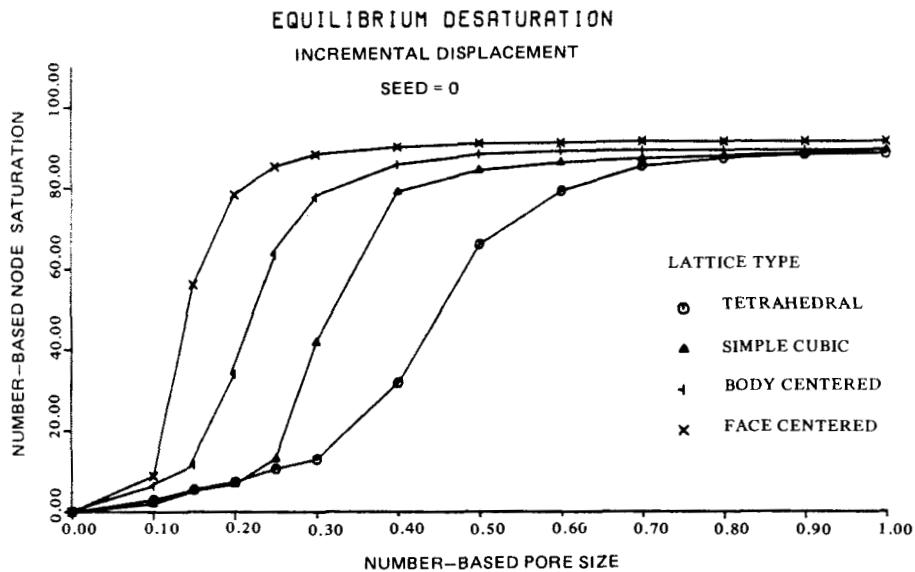


FIGURE 10: Capillary pressure-saturation curve in terms of generalized properties obtained from (three dimensional) network model.

The generalized property curves can be converted to actual pore size and moisture content in filter cakes by assigning volumes to the nodes and/or bonds of the lattice network used for the calculation. The assigned volumes are obtained directly from the micrographic analysis of filter cakes.

For each of the equilibrium and dynamic experiments performed, a new sample of fine coal particles was used. Considering the difficulty in obtaining similar samples of fine particles and other experimental uncertainties, the variation in the data as observed from Figure 6 and Figure 9 is quite reasonable. It was observed that the use of rubber

sleeves in the filtration cylinder helped to improve the reproducibility of experimental runs carried out under similar conditions.

Particle size has a significant effect on the filtration and dewatering rate of coal particles. It was observed⁽¹⁸⁾ that the cakes formed from -200 mesh coal particles were much more difficult to dewater than the cakes formed from -32 mesh coal particles. In contrast to this, the cakes formed from -100 + 200 mesh coal cakes have the largest pore diameter followed by -32 mesh coal cakes. As would be expected, -200 mesh coal cakes have the smallest pore diameter. Table 2 shows the values for pore diameters for several filter cakes. The large filtration rate in -100 + 200 mesh coal cakes was attributed⁽¹⁸⁾ to the absence of small particles (-200 mesh particles) in these cakes.

Figures 3 and 4 show two sections of a filter cake. The porosities of these two sections as determined by the image analyzer were found to be nearly the same.⁽¹⁹⁾ It was found that under these conditions the simple parabolic equation can be applied to analyze the filtration data. The results shown in Figure 9 were obtained from such an analysis. From these results, it can be concluded that filter cakes formed from fine coal particles are incompressible in the pressure range of 20 to 70 kPa.

These preliminary experimental results and theoretical analysis have revealed a number of important features of fine particle cake filtration. A clear

TABLE 2

Particle and Pore Size Distribution of Coal Cakes¹

Bulk Coal Size	Avg. Particle Size, μm	Avg. Pore Size, μm
-32 mesh	36.6 ² 57.1 ³	13.5 ² 22.9 ³
-100 + 200 mesh	45.8	17.5
-200 mesh	21.8	8.4

1. All cakes were formed under 20 cm Hg vacuum.
2. At the top layer of the cake surface.
3. At the bottom layer of the cake cross-section near the cake filter medium interface.

understanding of the micro-structure and its role in determining the dynamic and equilibrium behavior of filter cakes is a prerequisite in the future development and improvement of the filtration/dewatering process.

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