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Investigations on the State of Water in a Flocculated Filter Cake

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ABSTRACT

The vacuum filtration of hematite slurries and flocculated hematite slurries with anionic, cationic, and nonionic polyacrylamides was investigated. Test results show that the flocculated filter cake generally contains higher residual water than the unflocculated cake. Measurements of flocculants adsorption on a hematite surface, zeta potential, and contact angle of the mineral surface were carried out; the filter cakes were analyzed with a microscope and image analyzer; and the structure of the filter cakes was studied from the viewpoint of fractal theory. The existence state of residual water in flocculated filter cake was discussed. It was pointed out why a flocculated filter cake generally has a higher residual moisture content.

INTRODUCTION

The role played by flocculant filter aids in filtration processes has long been disputed. The key point of the issue is whether flocculants are beneficial to lowering filter cake moisture contents or not. There have been little basic research so far on the existence state of residual water in a flocculated filter cake. Obviously, the optimal application of flocculant filter aids in solid–liquid separation requires a proper understanding of the structure and state of residual water in a flocculated cake.

EXPERIMENTAL

Materials

The materials used in this investigation were hematite taken from the concentrate of the gravity concentration section of Gong Changling concentrator in China and ground in an experimental ball mill for 4 hours, with 65.15% total ferro content and 6.5 μm average particle size.

Flocculants

The flocculants were three kinds of polyacrylamides: nonionic, cationic, and anionic (hydrolytic) polyacrylamides (abbreviated PAM, CPAM, and HPAM). HPAM includes HPAM-1, HPAM-2, and HPAM-3, with molecular weights of about 3.2×10^6, 6.7×10^6, and 9.8×10^6 units, respectively. PAM and CPAM have about 9.8×10^6 and 7.0×10^6 units of molecular weight, respectively.

Experimental Methods

The filtration experiments were carried out with a set of vaccum filtration devices. Samples of 40 g were used, and the concentration of mineral slurries was 50%. The filtration time was 150 seconds. After filtration, the cakes were dried in an oven and then weighed. Finally the cake moisture contents were calculated from the difference between the weight of wet and dry cakes. The zeta potential measurements were made with a single tube electrophoresis instrument. The color change reaction of starch and the triiodo complex on polyacrylamide, and the amount of polyacrylamide adsorbed on the mineral surface were examined with a 721 spectrophotometer (1). The contact angles of the mineral surfaces were determined with a dynamic capillary ascent analysis (2). The structure of the filter cakes was examined with a scanning electron microscope and an image analyzer.

EFFECTS OF FLOCCULANTS ON CAKE MOISTURE CONTENT

Figure 1 shows the effects of pH and flocculants (PAM, CPAM, and HPAM) on the filter cake moisture content. It can be seen that the flocculated cakes all contain higher residual water than the unflocculated cake when the pH values of the slurries were less than 10. The cake flocculated by anionic polyacrylamide contains of the highest moisture content, the moisture content of the cake flocculated by cationic flocculant is the second highest, and was followed by the cake flocculated by nonionic polyacrylamide, which also has slightly higher moisture content than the un-

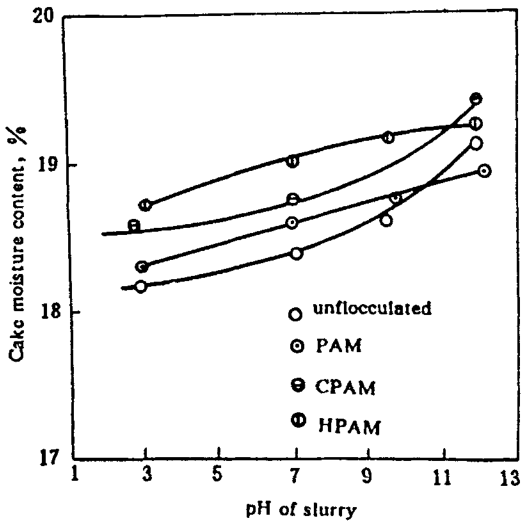

FIG. 1 Effect of pH and flocculants on the filtration of hematites.

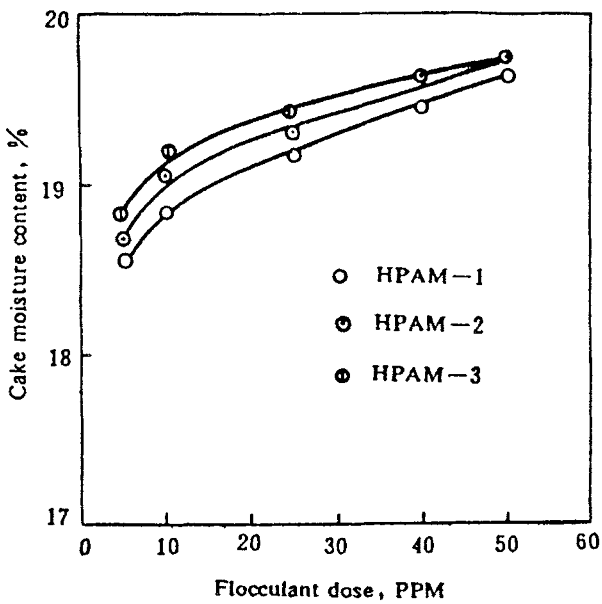

FIG. 2 Effect of flocculant molecular weight and dose on the filtration of hematites.

flocculated cake. However, the use of nonionic polyacrylamide can decrease the cake moisture content when the pH value in hematite slurry is over 11. The effect of the flocculant dose on the cake moisture content is presented in Fig. 2, in which the molecular weight of the flocculants is 3.2×10^6, 6.7×10^6, and 9.8×10^6 units, respectively. The results show that the higher the molecular weight and the dose level, the higher the cake moisture content.

EXISTENCE STATE OF RESIDUAL WATER IN FLOCCULATED FILTER CAKE

Effect of Flocculants on the Cake Structure

The filter cakes were photographed with a Philip SM 505 microscope and then the pictures were analyzed by a Cambridge Quantiment imager analyzer. The results are given in Table 1.

It is seen from Table 1 that the flocculated cakes have a much higher porosity and a bigger pore diameter than an unflocculated cake. At some dose levels, the flocculants of higher molecular weight result in higher porosity and large pore diameter. The higher the flocculant dose level, the higher the porosity and the larger the pore size. The action of flocculant filter aids is to improve the structure of the filter cake; i.e., to enhance the porosity and pore average diameter of the cake. The higher the molecular weight and the dose level of flocculant, the higher the porosity, the larger the pore size of the cake, and the quicker the filtration.

Besides porosity and pore size, the morphological characteristics of pores affected the residual moisture content of the cake. Generally, it is difficult to describe quantitatively the morphology of pores. Nevertheless, fractal theory, using a single fraction or fractal dimension, can represent them. Mandelbrot (3) introduced the concept of "fractal dimension" to

TABLE 1
Results of Cake Structure Analysis

Flocculants	Dose (ppm)	Measured pore count	Porosity (%)	Mean pore diameter (μm)	Maximum pore diameter (μm)
Unflocculated	0	300	47.13	0.826	3.30
HPAM-1	10	126	56.66	1.18	3.90
HPAM-2	10	102	59.33	1.26	4.80
HPAM-3	50	59	66.58	1.55	6.60

characterize self—similar structures. A Koch Island (Fig. 3), which can be regarded as a rugged boundary of a real pore, has the interesting property of having an infinite perimeter enclosing a finite area. While the practical rugged outlines of the cross section of a cake pore may not meet all the requirements needed for the mathematician to describe the curve as fractal, from an operational point of view the roughness of a cake pore boundary can be considered to have statistical self—similarity over a specified range of resolution. For the cake pore, the degree of roughness or irregularity can be given by D_F, a number called the fractal dimension; the higher the D_F value, the rougher the pore. The fractal dimension of the pore boundary was measured by the slit island method; i.e., the area perimeter method (4):

$$\frac{1}{D_F} \log P = \frac{1}{2} \times \log A + C \tag{1}$$

where D_F is the fractal dimension of the boundary of the cake pore section, P and A are the perimeter and area of the pore section, respectively, and C is a constant.

Equation (1) shows that if the pores in cakes have the same area section, the smaller the perimeter of the pore section, i.e., the smaller the fractal dimension of pore, the tidier the pore, and the smoother the pore inside wall.

The fractal dimension values of a set of cakes are listed in Table 2. The fractal dimension values of the flocculated cakes are greater than those of the unflocculated cakes, and the greater the flocculant molecular weight

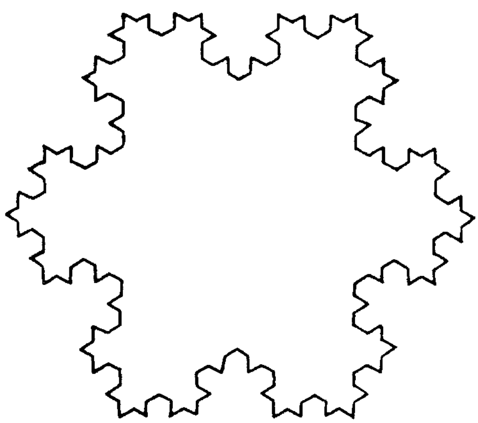

FIG. 3 A model for pore roughness of a Koch Island (D_F = 1.2618).

TABLE 2
Fractal Values of a Set of Cakes

Flocculants	HPAM-1	HPAM-3	HPAM-3	Unflocculated
Dose (ppm)	5	5	50	0
D_F	1.243	1.266	1.296	1.219
R (interrelation coefficient)	0.98100	0.98400	0.98701	0.98921

and the addition, the greater the fractal dimension value. The study shows that the macroscopic cake structure can be improved by flocculants, but the microscopic structure may be worsened and affect cake dewatering adversely. That is, flocculants can aggregate particulates and make cakes have higher porosity, larger pore sizes, and more permeability cakes amenable to rapid filtration, but flocculated cakes generally have more untidy pores and a rougher pore boundary, which is not beneficial to cake dewatering or to decreases in the residual water in the filter cakes. This is one of the major causes of higher residual moisture content in a flocculated cake than in an unflocculated one.

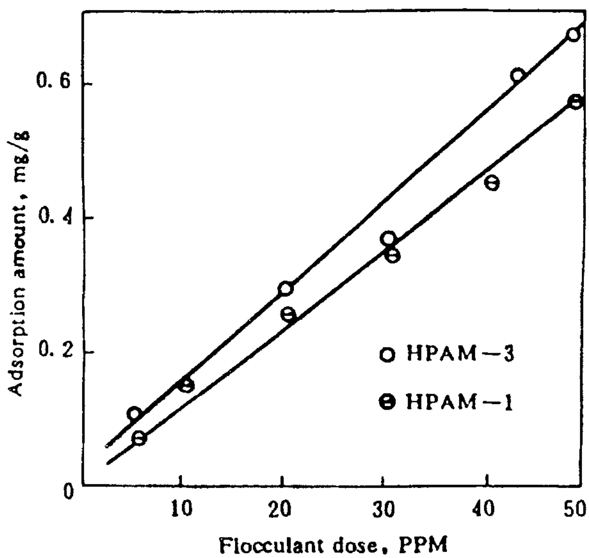

FIG. 4 Flocculant adsorption amount vs dose.

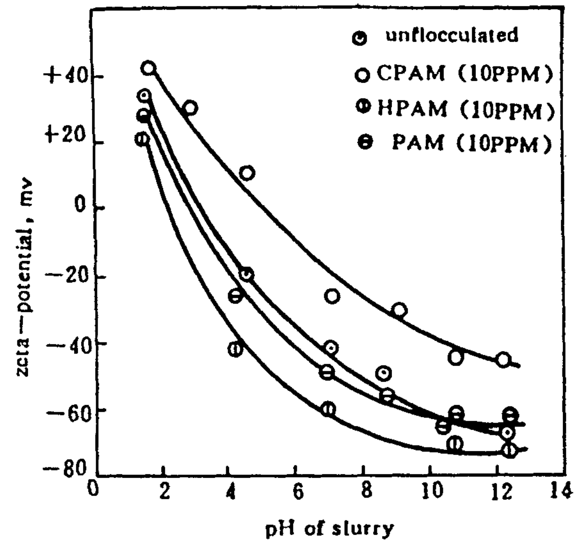

FIG. 5 Zeta potential vs pH for different flocculants.

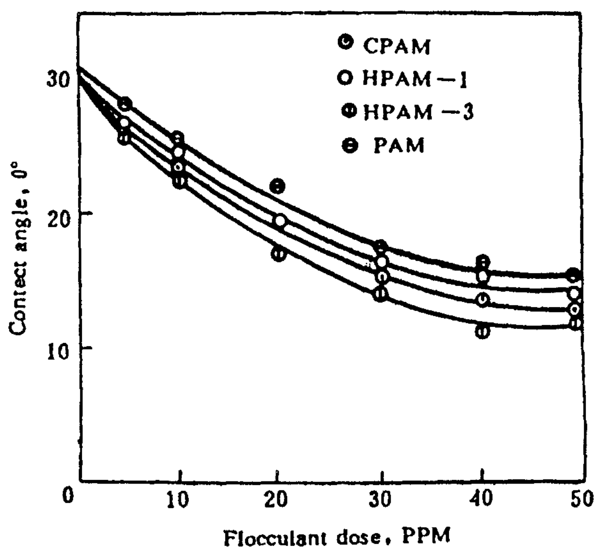

FIG. 6 Contact angle vs concentration for different flocculants.

Effect of Flocculants on Mineral Surface Characteristics

The adsorption of flocculant on the hematite surface as a function of flocculant dose and molecular weight is shown in Fig. 4 where the amount of adsorbed flocculant on the mineral surface rises with the increase of flocculant dose, and the higher the flocculant molecular weight, the higher the adsorption.

The relationship of the zeta potential to the pH for various flocculants is given in Fig. 5 where it can be seen that the adsorption of polyacylamides on hematite surface changes the zeta potential of the mineral surface. Cationic flocculant raises the zeta potential, while anionic and nonionic flocculants reduce the zeta potential of the hematite surface.

Figure 6 shows the effect of various polyacrylamides on the contact angle between a hematite surface and water. These polyacrylamides can render a mineral surface more hydrophilic. The effect of nonionic polyacrylamide on the contact angle of hematite surface is the least, cationic polyacrylamide is immediate, and anionic polyacrylamide has the greatest effect. In addition, the higher the molecular weight and the dose of flocculant, the lower the contact angle.

DISCUSSION

The use of flocculants in the vacuum filtration of mineral slurries can raise the filtration rate, but in most cases flocculated cakes contain more residual water than unflocculated cakes. It has been suggested that this is due to intrafloccular water locked within the flocculated structure for a long time (5), and the higher the molecular weight and the dose, the larger the floc volume and therefore the more water contained in the flocs.

During the dewatering process of mineral slurries, the water retained within the filter cake exists principally in three forms (6): 1) surface water, including surface adhesion water and interparticle-bound water, the former refers to the water film covering on the particle surface, the latter exists in the capillaries or interstitial voids between particles in contact; 2) capillary water, held within the capillaries or voids of the individual particles; and 3) chemically bound water, combined with a particle, such as in crystal water, etc. Clearly, vacuum filtration can only affect the removal of surface water and some capillary water.

Flocculants can aggregate fine particles but can hardly reduce the contact area between solid and water. Therefore, surface adhesion water in the cake is not decreased by flocculant addition. Furthermore, the interparticle bound water in a flocculated cake is more difficult to removed due to the more untidy pore structure and the rougher walls which can make it easier to retain residual water.

Flocculants have little influence on filtrate surface tension but can exert a great effect on the zeta potential of the particle surface, and thus the states of the electrical double layer and the hydrated layer are changed. Generally, material slurries are neutral or alkaline in practical filtration processes so that most particles carry a negative surface charge. Cationic flocculants can lower the zeta potential of the particle surface with negative charges, compress the electrical double layer, and tend to thin the hydrated film. The function of anionic flocculants is contrary to that described above. Therefore, in the case of solid surface electrical properties, cakes which have been flocculated by cationic and nonionic flocculants must have less surface water than those flocculated by anionic flocculants. This conclusion has been drawn from the results of the filtration experiments (see Fig. 1). In the practice of filtering mineral products, the use of anionic flocculants often results in higher filter cake moisture content than the use of other flocculants.

Generally, the higher the molecular weight and the dose, the larger the floc size and the higher the flocculated cake moisture content. Therefore, it is believed that intrafloccular water locked within the floc structure produces a higher residual moisture content than those of the tightly packed unflocculated cakes. As we know, flocculants, both ionic and nonionic, are water-soluble polymers and are strongly hydrophilic. If a particle surface adsorbs flocculants, it will become more hydrophilic. As shown in Figs. 4–6, the higher the molecular weight and the dose level, the greater the flocculant adsorption on the surface; the greater the adsorption, the smaller the contact angle and the more hydrophilic the particle surface. The hydrophilicity of anionic flocculants is the strongest, cationic second, and nonionic the weakest. We have discovered from a series of studies that the more hydrophilic the mineral surface under the action of flocculants, the higher the cake moisture content. Thus, there is reason to think that one of the major reasons why flocculated cakes usually contain a higher residual moisture content than unflocculated cakes is the stronger hydrophilicity of the particle surface as flocculants are adsorbed on it.

As the hydrophilicity of the particle surface is enhanced, its hydrated layer will be thickened. The state of water molecules in a hydrated layer is different from that of free water molecules; water molecules in a hydrated layer are gathered on the particle surface in an orderly or structured manner and are difficult to removed by mechanical forces. The thickness of a hydrated layer varies from 10^{-7} to 10^{-3} cm depending on the particle surface hydrophilicity (7). The ratio of the water within a hydrated layer to the cake residual moisture content is very high and is enhanced by the reduction of particle size and increase in the specific surface area of particles. For example, consider a material with a specific surface area of 5.1 m^2/g (used in the above experiments). Suppose that the hydrated layer has a thickness of 200 Å; then the cake should contains 10.2% surface water. Theoretically, if the hydrated layer thickness is increased by 20 Å by the adsorption of flocculants on the material surface, the cake moisture content would be increased by 1.02%. Thus, it can be inferred that a flocculated cake usually contains a higher residual moisture content than an unflocculated cake for two reasons: the enhancement of hydrated layer thickness by use of flocculants and the variation of the microscopic structure of flocculated cakes. In summary, the overall concept of water entrapped within a floc structure is too general and does not contribute to an understanding of the all-important aspects of the problem.

Under some conditions, if the cake structure is improved by the use of flocculants so drainage of capillary water and interparticle-bound water is improved with no more than small changes in the hydrophilicity of the mineral surface, it will be possible to lower the cake moisture content and reduce the filtration cycle time by adding the selected flocculants. For example, if the material surface itself is very hydrophilic, or if the pH value of slurry is very high, a decrease in the cake moisture can be expected by using cationic and nonionic polymers under some conditions.

CONCLUSIONS

The use of flocculants can improve the macroscopic structure of a filter cake and enhance the filtration rate, but it seldom lowers the residual moisture content of the cake. Conversely, flocculants produce cakes with higher moisture contents than unflocculated cakes. This results from two major causes. One cause is the change in the fractal dimension of the cake pore, leading to more untidy pores and a rougher pore wall and consequently more water retained at the points of contact between particles. The other cause is a more hydrophilic particle surface from the adsorption of flocculants on the surface, resulting in a thicker hydrated layer. The higher the molecular weight and the dose level of flocculants, and the stronger the hydrophilicity of the flocculant polar group, the more remarkable the effects mentioned above.

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