STUDIES ON SELECTIVE FLOCCULATION FOR COAL DE-MINERALIZATION

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(Received: October 17, 2000 - Accepted in Revised Form: December 31, 2001)

Abstract Selective flocculation has long been suggested as a method for the treatment of ultra-fine particles typical of which are those produced in a de-sliming process ahead of flotation. In this paper the results of the coal de-mineralization, using synthetic polyacrylamide polymers, are presented. A prescreening test on the suitability of the polymers using Run-Of-Mine (R.O.M.) coal and artificial mixtures of Shirebrook coal and shale the floc settling rates and supernatant clarity of the suspension was carried out and three polymers, Floc Aid 202 (medium anionic), Floc Aid 204 (high anionic) and Polyethylene oxide (P.E.O.) (non- ionic) were employed in subsequent experimental work. The effectiveness of the polymers was established by carrying out settling rate tests using polymers dosages between 0.00025 to 0.0075 g/25g feed and at various suspension pH values. P.E.O. showed significant differences in the flocculation of coal and shale. The results concerning polymers FA 202 and FA 204 are not discussed. Slurry pH, dispersant concentration and polymer dosages were examined and are discussed. Shale entrapment was believed to be due to three reasons. Firstly the mineral particles not fully liberated from the coal structure. This could be infered from the S.E.M. micrographs. Secondly, once coal flocs have been formed, they become larger in size, and on settling collect the much smaller mineral particles, beneath themselves, to the bottom of the container. And thirdly polymers may possess a weak affinity for the shale surface.

Key Words Selective Flocculation, De-sliming, Polymers, Settling Rate

چکیده فلوکولاسیون انتخابی، برای بازیابی ذرّات میکرونی پیشنهاد شده است. در این مقاله با استفاده از پلی‌مرهای مصنوعی، نتایج بازیابی موادّ کربنی (ذرّات زغال سنگ) از موادّ معدنی ارائه شده است. کارآیی پلی‌مرها، هنگامی که مقادیری بین ۰/۰۰۰۲۵-۰/۰۰۷۵ گرم در هر ۲۵ گرم خوراک استفاده شد، به وسیله‌ی سرعت ته نشینی ذرّات زغال و موادّ معدنی مورد ارزیابی قرار گرفت. اکسید پلی‌اتیلن اختلاف قابل ملاحظه‌ای در فلوکولاسیون زغال و موادّ معدنی نشان داد. pH پالپ، غلظت پخش کننده و مقدار پلی‌مرها مورد آزمایش قرار گرفته و بحث شده است. میکرو تصویرها نشان دادند که همراه لخته‌ها مقداری موادّ معدنی نیز وجود دارد که منشأ این موادّ معدنی به این قرار می‌باشد: ۱- آزاد نشدن موادّ معدنی از زغال سنگ (حتّی هنگامی که ذرّات به زیر ۴۵ میکرون خرد شوند)؛ ۲- لخته‌های (زغال سنگ) تشکیل شده در هنگام ته نشینی ذرّات بسیار ریز موادّ معدنی موجود در گلاب را با خود ته‌نشین می‌کنند و ۳- ممکن است پلی‌مر تمایل اندکی به موادّ معدنی داشته باشد.

1. INTRODUCTION

The need for cleaning and recovery of fine and ultra-fine coal fractions, due to coal produced in this way, has the potential for replacing oil by coal-water mixtures, improving overall heat recovery, and to reduce environmental concers about burning uncleaned coal and in waste disposal.

For coal-water mixtures to be suitable substitute for oil, the ash content must be low (less than 2-3% weight). However, de-ashing of coal by physical beneficiation methods is limited by the degree to which the ash producing components are liberated from the coal. In practice this requires ultra-fine grinding of coal. Three general methods are available for the treatment of material less than 500 μm These are:

(i) - Full froth flotation followed by vacuum filtration.

(ii) - De-sliming ahead of flotation

(iii) - After classification by large diameter cyclones, screening of the under flow at about 250 μm.

But Routes ii and iii produce a waste component which contains significant quantities of ultra-fine (i.e.<60 μm) combustible material. Methods available for the ultra-fine particles are:

(i) - Froth flotation

(ii) - Spherical oil agglomeration

(iii) - Selective flocculation.

Y. A. Attia et al. [1] have summarized their effects. In 1953 Haseman [2] by employing a modified starch flocculant separated phosphate rock from clay. Usoni et al. [3] and N. Karapinar et al. [12] who used several reagents, including non-ionic polyacrylamide, were able to carry out the separation of various minerals from quartz on a laboratory scale.

The first commercial application of selective flocculation in the world, was reported by Frommer and Colombo [4] of the Bureau of Mines Twin Cities (Minn) Metallurgy Research Center. Using Frommer and Colombo¨s technique iron oxides are selectively flocculated from siliceous slimes as one of the processing steps in the beneficiation of oxidized iron ore at the Tilden Mine on the Marquette range of Michigan¨s upper Peninsula [5].

As far as coal is concerned, Blagov [6] reported that Soviet researchers were attempting to selectively flocculate coal using a polyacrylamide flocculant and sodium pyrophosphate as a dispersant.

Hucko [5] when studying beneficiation of coal by selective flocculation, using an artificial mixtures of Pittsburgh bed coal (2.5 to 3.3% ash) and shale (90% ash), investigated several important parameters, such as type and concentration of dispersant, pH, type and dosage of flocculant, and ionic character of the suspension medium. He reported that selective flocculation of coal/shale suspension is technically feasible, and this was favored under alkaline conditions. Of the flocculants examined, he found that naturally occuring organic colloids such as starches and guars were only moderately selective, but that low molecular weight non-ionic polyacrylamides were more selective. Of the dispersants examined, sodium hexametaphosphate (calgon) was the most effective. The studies also revealed that increasing concentration of ionic species, such as Ca^{+2}, in the suspension medium decreased selectivity.

Blaschke [7] showed that the slimes of a steaming coal containing about 42% ash could be cleaned in one stage by selective flocculation to about 29% ash. Coal slimes containing about 39% ash were selectively flocculated using 3-8 kg/ton sodium salt of carboxymethyl cellulose as dispersant and 0.4-4 kg/ton of an anionic, modified polyacrylamide (Gigtar) as the flocculant. By this method, the ash content was reduced in one stage to about 20% with coal recovery of about 40%.

Using commercial and modified polyacrylamide polymers, some researchers [8,11,13] found, in settling tests with individual suspensions of coal and shale, that anionic flocculants produced higher settling rates for the latter. With cationic flocculants, however, the settling rates for shale were approximately one quarter of those for coal. However, in tests with mixtures of equal amounts of coal and shale on weight basis both types of flocculants produced low-ash coal product. This phenomena was attributed to the preferential adsorption of the flocculants onto the coal surface, the flocculant requirement for adsorption on coal being satisfied prior to significant adsorption on shale.

2. SELECTIVE FLOCCULATION PROCESS DESCRIPTION

The selective flocculation process utilizes the differences in the physical, chemical, and surface properties of various particulate

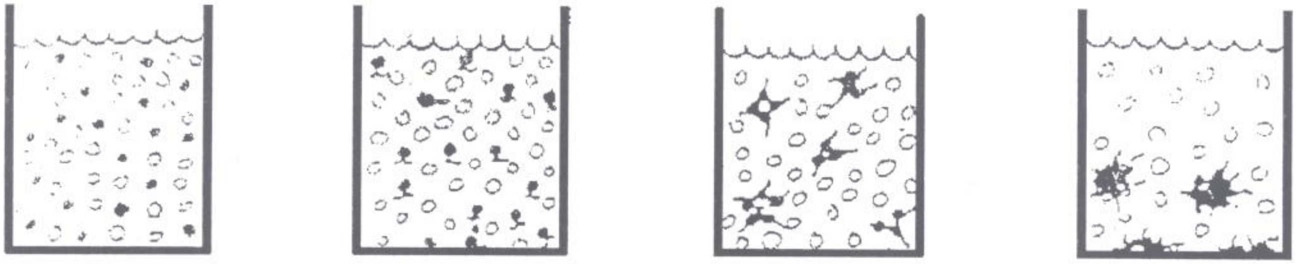


Figure 1. Selective flocculation process for coal beneficiation.

components in a coal suspension. The technique is based on the preferential adsorption of an organic flocculant on the particular solids to be flocculated, leaving the remainder of the particles in suspension.

The process of selective flocculation can be considered to be composed of four major sub-processes [7,9]. These, presented conceptually in Figure 1 include:

2.1 Dispersion of coal slurry

2.2 Flocculant adsorption and floc formation

2.3 Floc conditioning

2.4 Separation of flocs from suspension by suitable means.

2.1 Dispersion of Coal Slurry An important aspect of dispersion is that the slurry is adequately agitated. The aims of agitation are two-fold, first to achieve a homogeneous distribution of the fine particles and to break up any loose aggregates that may be present and second to disseminate the reagent quickly to all the particle surfaces. In coal slurries, a dispersant such as calgon when adsorbed on the surface of the fine particles, increases the negative charge of the surface this action in turn, causes electrostatic repulsion among the particles and produces a well dispersed slurry.

2.2 Flocculant Addition and Floc Formation At this stage, the added flocculant first disperses throughout the suspension medium under intense agitation. Then the flocculant adsorbs on the coal surface and coal particles with the adsorbed flocculant collide with other nuclei or single coal particles, leading to the growth of the flocs. The rest of the slurry remains dispersed. However, under various process condition, such as intense mixing, the flocs can break into smaller units, which on further collision with other particles or nuclei, grow to larger flocs.

Among the process parameters that influence this sub-process, the dosage of the selective flocculant has an important effect on the extent of selectivity achieved. Too large a dosage leads to a complete flocculation of the slurry. On the other hand less than the optimum amount results in inadequate flocculation and, therefore, lowers recovery of coal.

2.3 Floc Conditioning The purpose of conditioning is two-fold. First, the flocs must grow and strengthen so that they are able to withstand the mechanical stress imparted by whichever separation technique that follow. Second this must be done in a manner that will minimize the association of undesired mineral matter. Generally, the addition of the flocculant is accompanied by a high level of agitation. Once the flocs are formed the agitation level is lowered.

The quality of flocs formed is related to the quantity of mineral matter that is associated with the flocs which may arise from five sources:

*1. **Entrainment:*** particles that are carried with flocs while settling or moving in suspension.

*2. **Entrapment:*** particles that are trapped within the flocs during their formation or growth Figures 2a and 2b).

*3. **Settling:*** particles that settle independently in the suspension and are collected along with the flocs.

*4. **inclusion:*** particles that are included in the proportion of water that is suspended along with the flocs.

*5. **Inherent:*** non-liberated mineral matter associated with the coal (Figures 3a, 3b, 4a, 4b and 4c).

Since considerable amount of mineral matter associated with the flocs arises due to entrainment and entrapment, re-cleaning the flocs by re-dispersion, releases entrapped mineral matter, and re-flocculation is an effective method to improve quality of the flocs, but some coal may be lost.

2.4 Separation of Flocs from Suspension

The Overall performance of the selective flocculation process is controlled by the floc separation from the suspension. Idealy, the technique used to separate the flocs from the rest of the slurry should be simple, cause minimum break up of flocs, and be highly efficient. The gentle hydrodynamic conditions required for separation makes gravity sedimentation the most common method in use. The gentle hydrodynamic conditions required for separation makes gravity sedimentation the most common method in use. Other effective techniques include elutriation, column flotation and dissolved air flotation.

3. ADVANTAGESOF THE SELECTION FLOCCULATION

Selective flocculation has the following advantages [1] over froth flotation for cleaning coal for coal-water mixture applications:

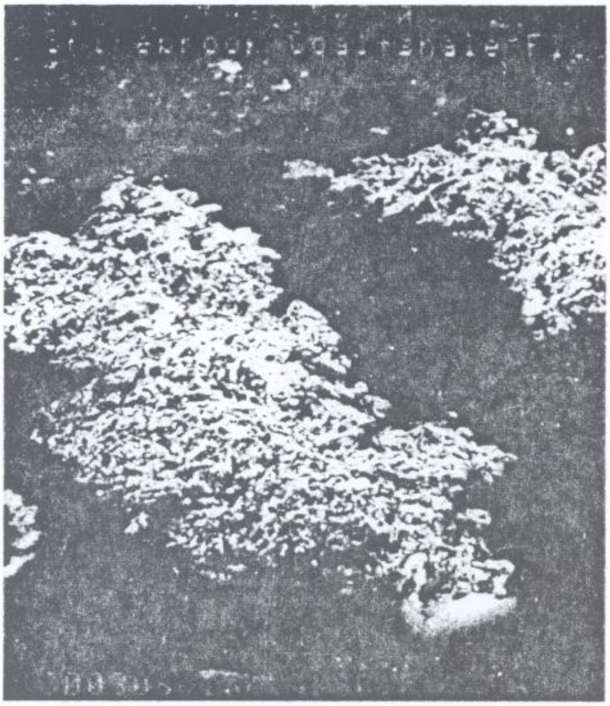
3.1 Effective for Fine and Ultra-Fine Coal This technique is applicable to particles that are too small for cleaning by froth flotation. Although both process perform well for certain particle size ranges, selective flocculation remains effective for both fine and ultra-fine coals.

3.2 Reduced Reagent Requirements This process requires less reagent to achieve equivalent cleaning levels. This advantage becomes more pronounced for oxidized or lower rank coal.

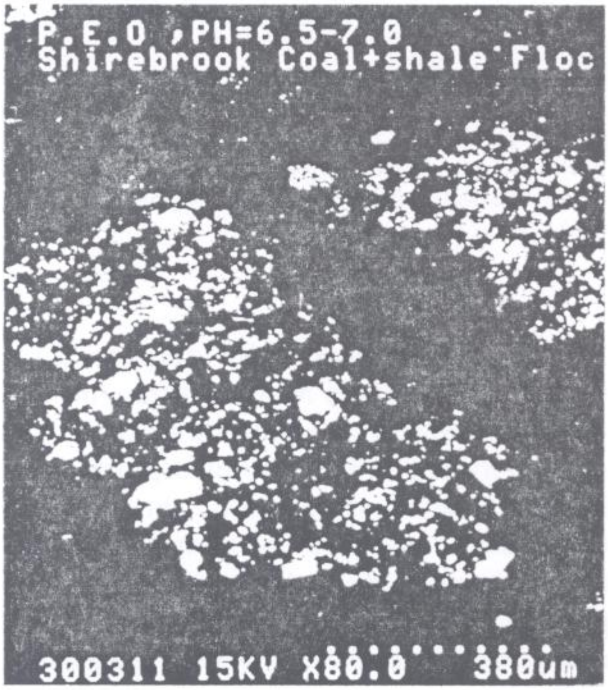
3.3 High Ash Rejection Some of the mineral matter in coal suspensions when cleaned by froth flotation and selective flocculation are entrained with the rising coal-air bubble or with the settling flocs respectively. However, minerals can be released with the conditioning step providing a secondary cleaning mechanism. This results in higher single stage rejection.

3.4 High Single Pass Efficiency Combustible recoveries of 90% have been achieved about 80% mineral matter rejection. Over 95% removal at about 80% mineral matter rejection. Over 95% removal is possible but at somehow lower recoveries.

3.5 Effective Pyrite Removal In forth flotation pyrite is usually floated along with the clean coal. Since pyrite, in selective flocculation, can be dispersed along with the mineral matter, significant removal of inorganic sulfur is possible.



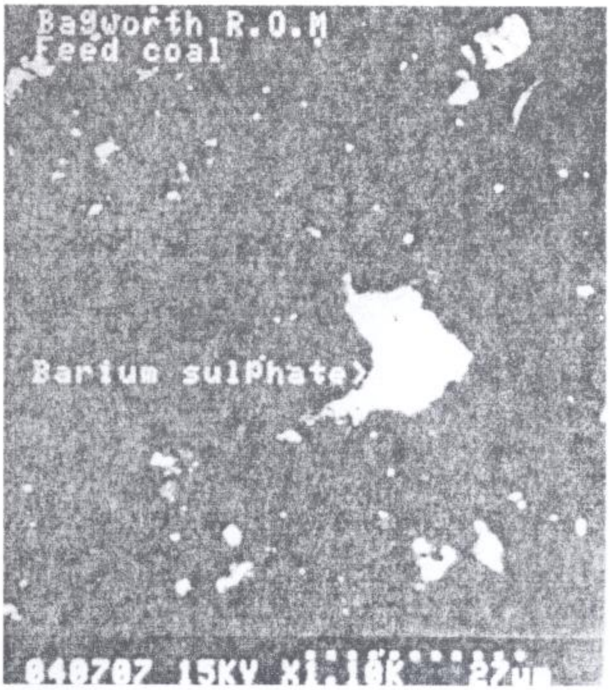
(a)



(b)

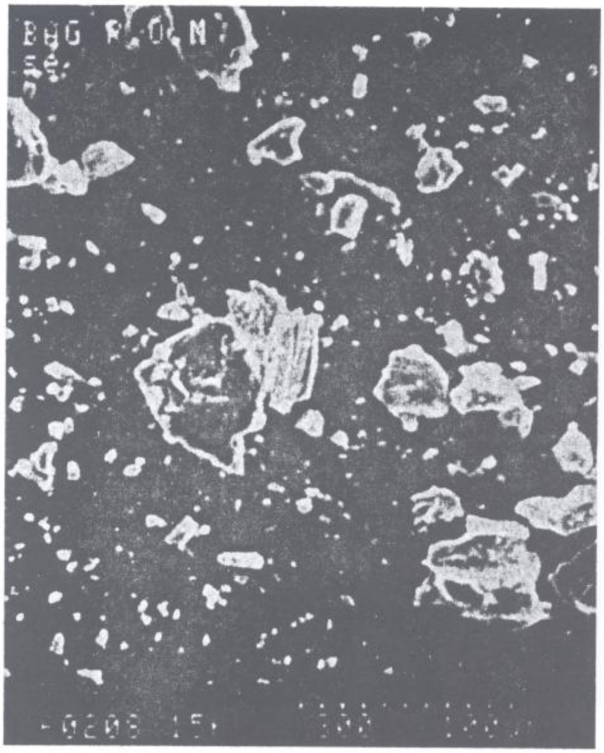
Figure 2. (a) SEM micrograph of the -45 μm Shirebrook coal/shale artificial mixture subjected to P.E.O. and (b) SEM micrograph of the -45 μm Shirebrook coal/shale artificial mixture subjected to P.E.O. using Backscattered Electron Detector. The bright areas are the minerals entrapped within the flocs.

(a)



(b)

Figure 3. (a) SEM micrograph of the -45 μm fraction of Bagworth R.O.M. coal (ash= 21.0 % wt) and (b) SEM micrograph of the -45 μm fraction of Bagworth R.O.M. coal (ash= 21.0 % wt) using a Backscattered Electron Detector.



(a)

(b)

(c)

Figure 4. (a) SEM micrograph of the -45μm fraction Bagworth R.O.M coal, (b) Magnified SEM micrograph of the -45 μm fraction Bagworth R.O.M coal and (c) Using Backscattered Electron Detector to show the unliberated mineral particle from the coal matrix (shown as bright area).

3.6 Clean Coal Easily Re-dispersed for Coal-Water Mixture Manufacture In froth flotation collectors are added to make the coal surface hydrophobic to allow capture by air bubbles. In selective flocculation process since the addition of selective flocculant or dispersant does not increase the hydrophobicity of the coal particle, the clean coal can be more readily re-dispersed for coal-water mixture preparation.

4. EXPERIMENTALS

4.1 Samples Preparation The coals and shales were obtained from the Licestershire Colliery, England, spread over a clean surface

TABLE 1. Ash Content (%wt) of Selectively Flocculated Bagworth Coal (Feed Ash = 49.0 %wt) at Various Polymer Dosages and pH Values.

Polymer Type	Polymer Dosage (g/25 Feed)	pH					
		2.0	4.0	6.0	8.0	10.0	11.0
		Ash Content (%wt)					
FA 202	0.01	43.2	41.5	41.3	40.4	42.0	44.5
(Medium	0.05	41.0	39.1	42.0	38.0	39.5	41.0
Anionic)	0.1	42.0	41.0	38.3	41.4	41.4	41.9
FA 204	0.01	39.0	42.0	39.0	40.7	41.3	45.5
(High	0.05	38.8	38.6	41.1	39.4	39.9	41.4
Anionic)	0.10	41.2	40.4	39.6	40.4	42.4	40.1
P.E.O	0.01	39.0	41.0	38.0	40.0	39.3	43.2
(Non-Ionic)	0.05	37.9	39.1	40.2	38.2	38.0	38.4
	0.10	41.0	40.3	41.0	40.8	42.0	43.4

of material and allowed to dry , over 24 hours, at room temperature. The samples, using a laboratory cone crusher, were crushed to less than 5 mm in diameter. The crushed samples were then milled in a Kek Mill three times, before feeding them into a locker "Rotex" triple deck screen by means of vibrating hopper. Adequate -45 μm size fractions for the experimental purposes were obtaind . In order to minimize any possible oxidation effects the collected samples were put inside resealable polythene bags and left in a dark and dry place .

4.2 Samples Ash Analysis The ash contents (% wt) of the -45 μm fraction and the sediments were measured according to BS 1016: part 3: 1973. It is assumed that any bag will be a representative sample of the whole with regard to any particular coal or shale sample.

4.3 Methed of Settling Rates Measurements At first Bagworth coal (feed ash=49% wt) at various flocculant dosages and pH was tried. The results are presented in Table 1. From the results it is obvious that the selective flocculation of Bagworth R.O.M. coal does not produce satisfactory results. To get a better result, Shirebrook coal (10 %wt. ash) and shale (82 %wt. ash) were obtained. In order to have a better and more clear understanding of the coal and the shale particles behavior in a suspension and the reduction of the particles in either coal or shale species towards polymer adsorption, settling rates of individual coal and shale samples were, therefore, determined as follows:

In settling rates measurements, 450 cm^3 of 5.55 % prepared suspension, of either

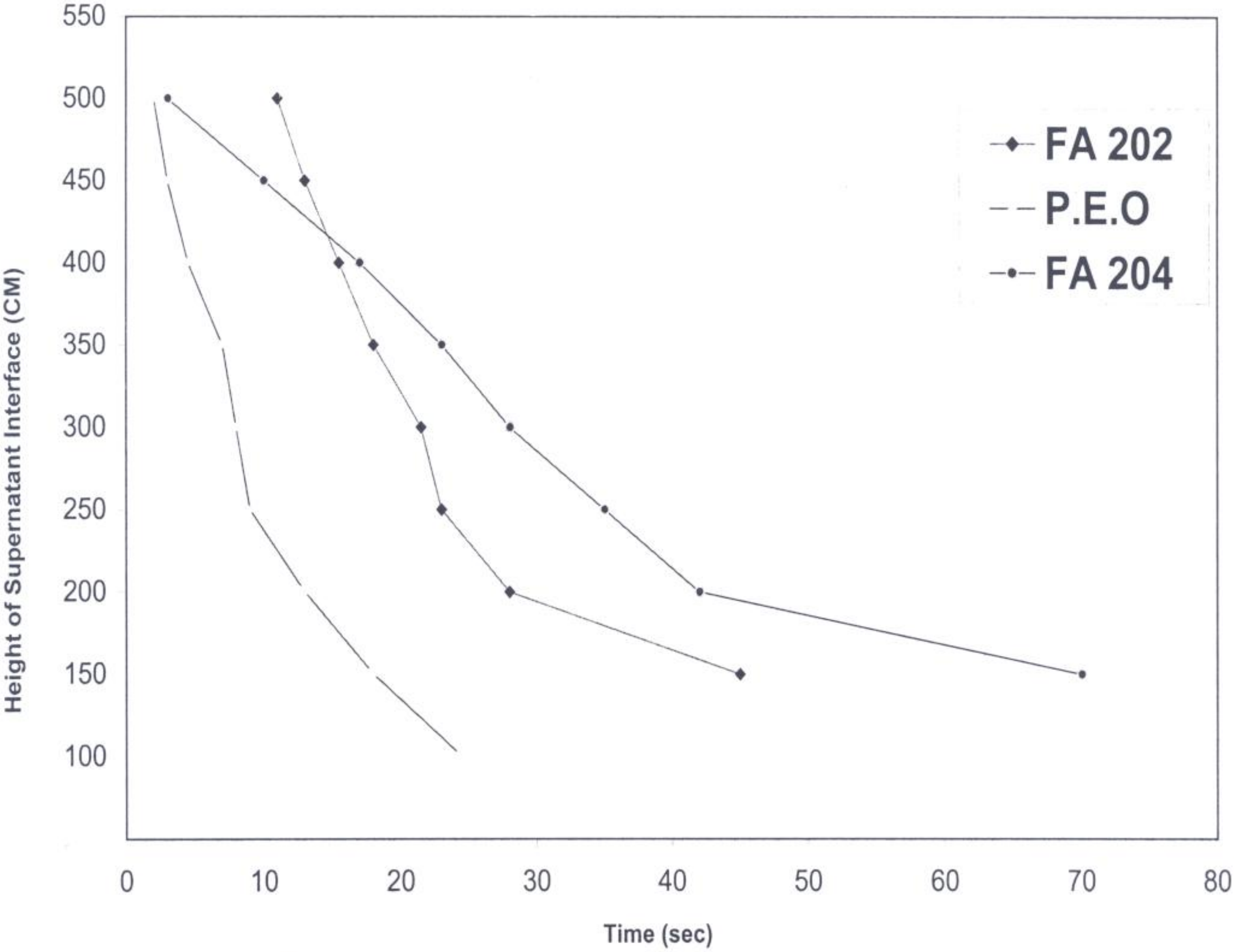


Figure 5. Settling test for 5 % suspension of Shirebrook coal treated with 0.0075 g flocculant / 25 g coal at pH = 10, for various flocculants.

Shirebrook coal (Rank 702) or shale was transfered into the graduated perspex cylinders (dimensions: 4.5 cm diameter, and 40 cm height, graduated every 10 cm^3 up to 500 cm^3). The required volume of the standard polymer solution (0.05 % concentration) was accurately measured, by a pre-rinsed glass pipette, into a 200 cm^3 volumetric flask and made up to 50 cm^3 mark with distilled water. The suspensions and the polymer solutions had identical pH readings in any one experiment. The diluted polymer solution was added to the top, and to the middle of the coal suspension bringing the final volume to 500 cm^3 (i.e. a final 5 % wt. solid concentration reached). The contact of the polymer solution with glass was kept to a minimum as polyacrylamides tend to be adsorbed onto glass surfaces. By means of a glass stopper, the top of the cylinder was sealed before manually inverting end-over-end six times to ensure complete mixing of the polymer solution and the coal suspension. The cylinder was then immediately placed onto a flat surface and a stop-watch simultaneously started. The results are graphically shown in Figures 5 and 6 respectively.

A lamp and scale arrangement was used to note the movement of the floc boundary [10]. The settling of the floc boundary through successive 10 cm^3 graduation on the

TABLE 2. Settling Rates (cm/sec) of Shirebrook Shale Treated with P.E.O. at Different Polymer Dosages and pH.

P.E.O Dosage (g / 25 g feed)	pH					
	2.0	4.0	6.0	8.0	10.0	11.0
0.00025	0.01	0.009	-	-	-	-
0.00125	0.07	0.07	0.05	0.05	-	-
0.0025	2.03	1.63	0.07	0.12	0.08	0.07
0.0075	3.25	3.20	0.95	0.81	0.45	0.78

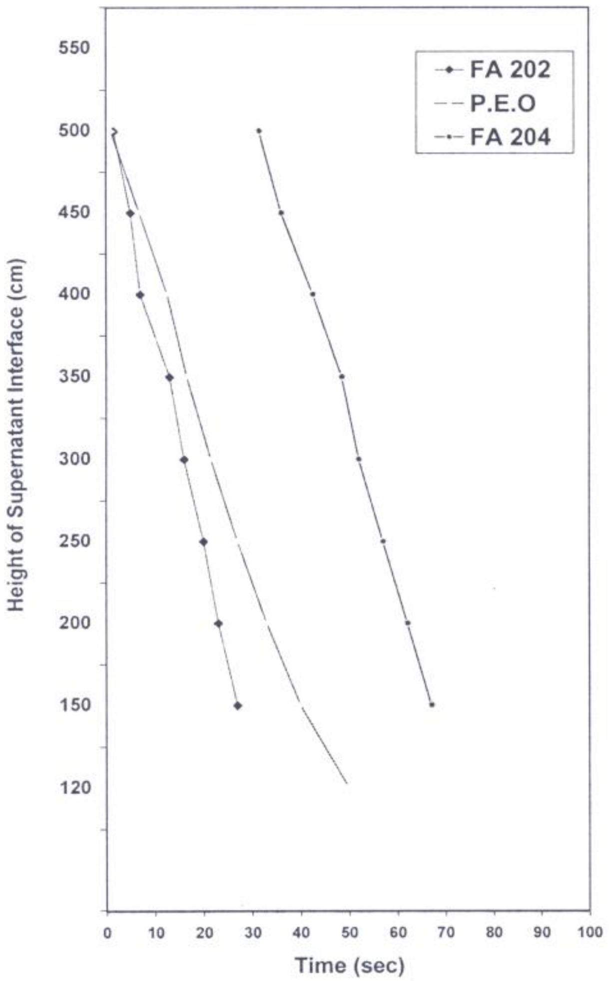


Figure 6. Settling test for 5 % suspension of Shirebrook coal treated with 0.0075 g flocculant / 25 g coal at pH = 10, for various flocculants.

cylinder was followed by eye. The time at the instant at which the floc boundary reached the graduation was recorded. For relatively fast settling rates the time at every 20 or 30 cm^3 graduation was recorded.

The settling rates (cm/sec) were calculated by dividing the distance between 450 cm^3 and 300 cm^3 graduation (8.133 cm) by the time (seconds) taken for the floc boundary to fall between the marks. Plots of the curves show these points to fall well within the linear portion of the curves.

From the figures it is obvious that P.E.O. comparatively could produce better results and ,therefore, was further exploited.

The results, when using P.E.O. only, are given in Tables 2 and 3 and are graphically shown in Figures 7 and 8 respectively.

Looking at the results and the relevant graphs in Tables 2 and 3 and Figures 7 and 8 respectively the areas in which the maximum differences in the settling rates occured were identified. Based on this results an artificial 50/50 wt/wt mixture of Shirebrook coal and Shale was floculated. (agitation speed before and after polymer addition was 3400 and 340 r.p.m respectively). The results are given in Table 4 and shown graphically in Figure 9.

TABLE 3. Settling Rates (cm/sec) of Shirebrook Coal Treated with P.E.O. at Different Polymer Dosages and pH.

P.E.O. dosage (g / 25 g feed)	pH					
	2.0	4.0	6.0	8.0	10.0	11.0
0.00025	0.19	0.12	0.17	0.22	0.22	0.29
0.00125	0.45	0.58	1.02	0.81	0.80	2.03
0.0025	1.02	1.16	1.16	1.16	1.36	1.30
0.0075	1.36	1.81	1.81	2.71	2.03	2.03

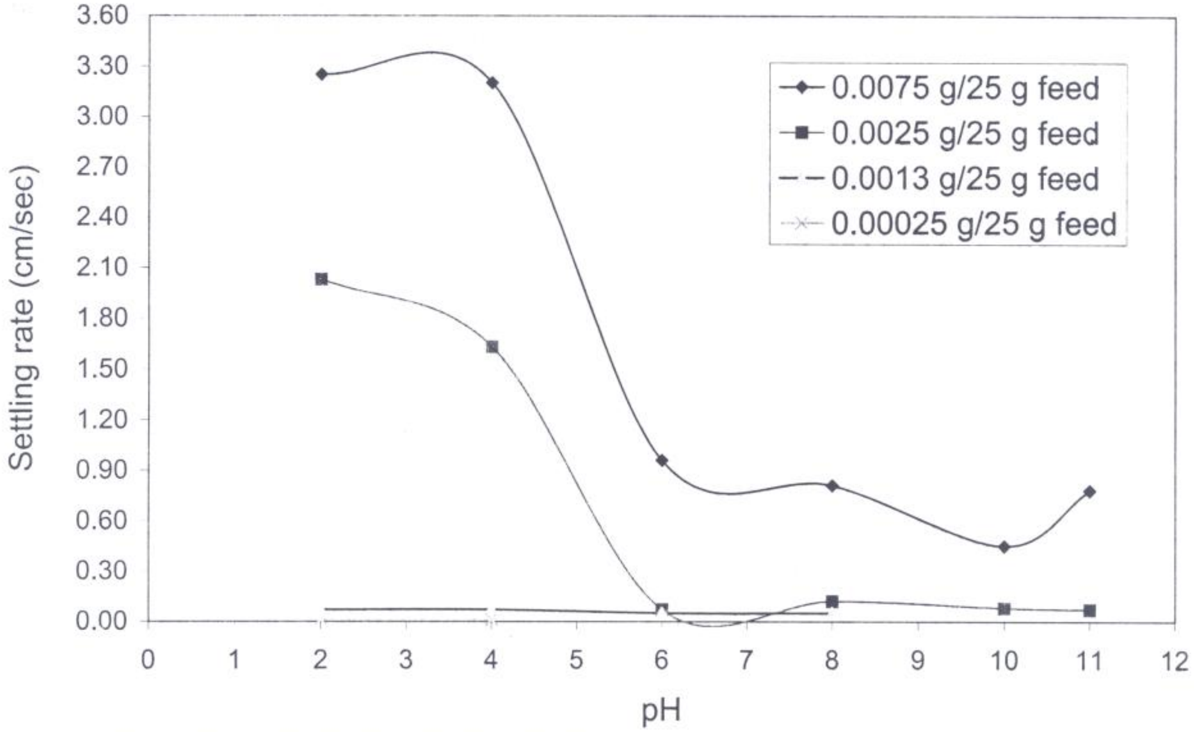


Figure 7. Settling rate (cm/sec) of Shirebrook shale treated with P.E.O. at different dosage and pH.

5. DISCUSSION

Current flocculation theory suggests that it is possible to alter the charge on the particles within suspension. Assume that two population of particles, for example coal and shale, exist within a suspension. If the coal particles are assumed to carry virtually no electrostatic

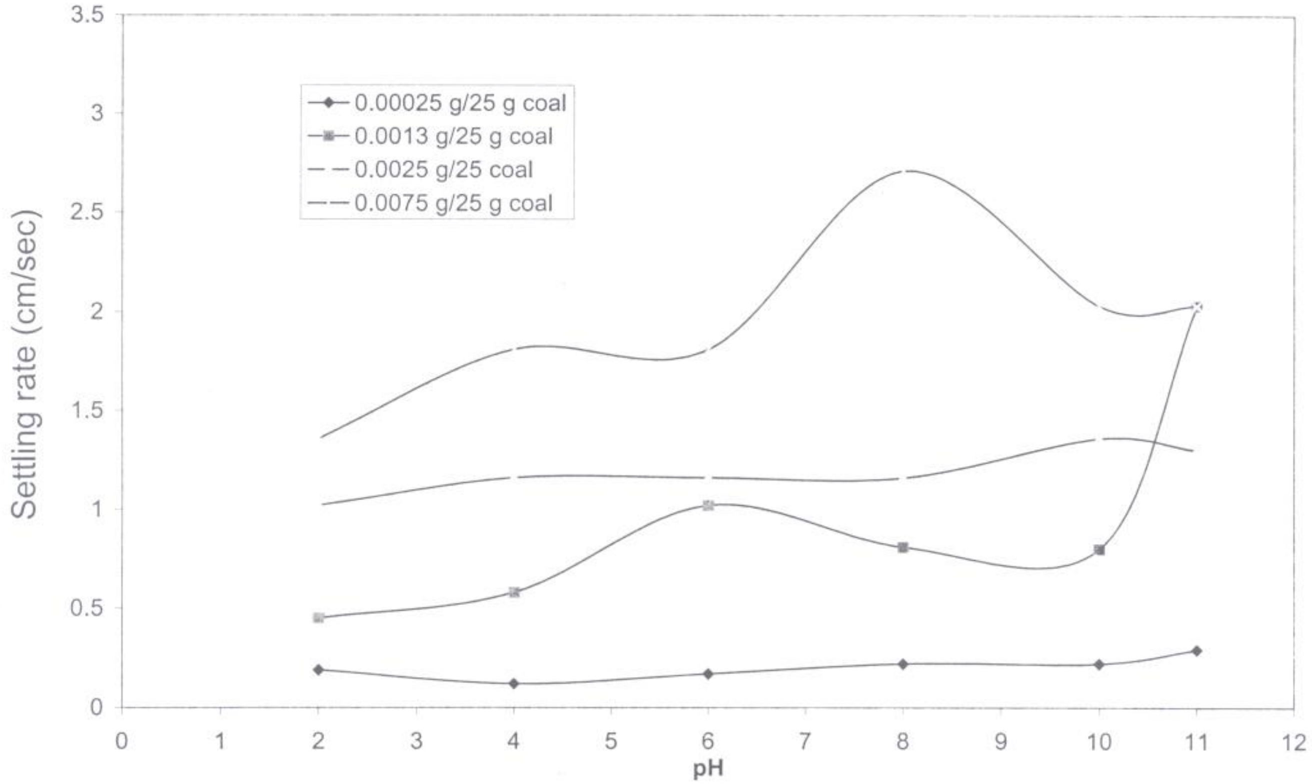


Figure 8. Settling rate (cm/sec) of Shirebrook coal treated with P.E.O. at different dosage and pH values.

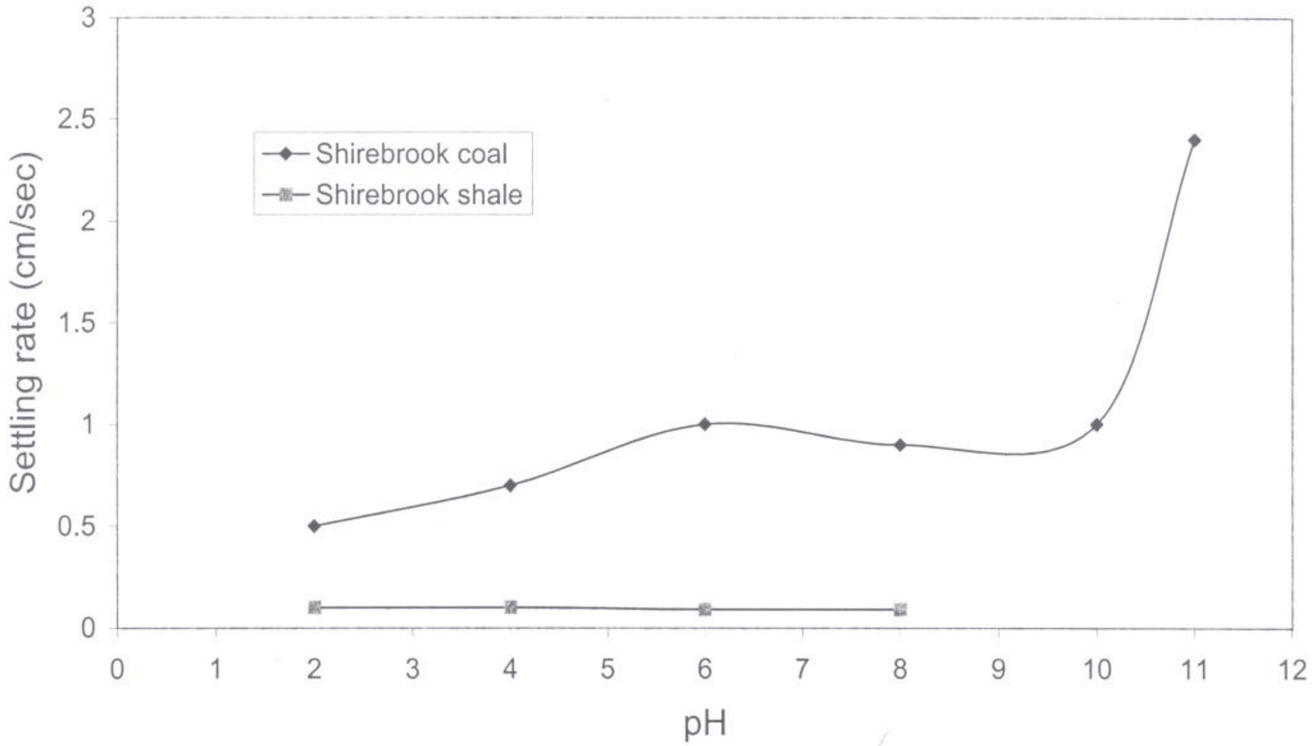


Figure 9. Settling rates (cm/sec) of Shirebrook coal and shale treated with P.E.O. at a dosage of 0.00125 g/25 g feed.

TABLE 4. Variations of Ash Content (%wt) with pH for Artificial Coal/Shale 50/50 w/w Mixture (Ash = 46.0 %wt) based o n Maximum Differences i n Settling Rates.

P.E.O. Dosage (g / 25 g Feed)	pH					
	2.0	4.0	6.0	8.0	10.0	11.0
0.00125	38.0	39.5	39.8	38.3	37.4	38.0

charge, and the shale particles carry significant electrostatic charge, then we would expect principally flocs of coal to be produced and the shale particles remain within the suspension. In this manner selective coal beneficiation by the addition of electrolytes could occur.

Initially, Bagworth R.O.M. (49% wt. ash) was subjected to selective flocculation experiments, but sediments of low ash content were not obtained. The reason for this failure was believed to be as a result of surface contamination of coal by the shale particles or vice versa.

In the next stage settling rates of separate coal and shale samples were performed with the three pre-screened polymers. Of the polymers tested P.E.O. produced remarkable results. Using P.E.O. dosages between 0.00025 to 0.0075 g/25 g of either coal or shale the coal particles settled nearly 20 times faster than the shales (Tables 2 and 3).

The results and the graphs indicate that P.E.O. probably because of its high molecular weight, 5,000,000 and possibly through its active groups in a suspension containing both the coal and shale particles would preferentially flocculate the coal particles and leave the shale particles within the suspension.

The ash analyses of the sediments showed that in a single operation the reduction in feed ash content, at pH = 10, of about 12 %wt achieved.

From Table 4 it is also obvious that the best results at pH=10 were obtained. This could be due to the fact that at high pH values the coal particles show high affinity towards the polymer while the shale particles remain indifferent.

The reason for this partial improvements could be explained from the fact that when coal particles flocculate and become larger in size (see micrographs in Figures 2a and 2b) on their settling, would carry much smaller shale particles with them to the bottom of the flask. The second reason could be that since shale particles having higher densities than the coal particles would settle faster. The third reason for the inclusion of non-carbonaceous in the flocs context is the fact that even when grinding samples to -45 μm still some mineral matters are not fully liberated (see micrographs in Figures 3a, 3b, 4a, 4b and 4c, and the description in section 2-3 , i.e. floc conditioning).

6. CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

Differences in the settling rates of shale and coal minerals (notably with P.E.O.) were observed. Reductions in ash content by selective flocculation, either of artificial mixtures of shale and coal or of high ash R.O.M. coals, were achieved.

The optimal use of P.E.O., the effect of pulp temperature and the operation parameters are worth investigating further.

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