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ADSORPTION OF POLYSACCHARIDES AND RELATED COMPOUNDS ONTO COAL AND THEIR EFFECT ON THE FLOTATION OF COAL AND PYRITE

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ABSTRACT

While polysaccharides are known to depress pyrite during coal flotation, many of them are also known to depress coal. This report gives a quantification of their adsorption onto Pittsburgh seam coal and an evaluation of the polysaccharide types and flotation conditions which may allow them to depress pyrite without, at the same time, also depressing the coal. Their mechanism of adsorption onto coal appears to be by hydrogen bonding and electrostatic attraction. Several effective pyrite depressants have been identified.

INTRODUCTION

Flotation is an important separation method for fine particles, and its use should increase substantially as the need for clean coal, low in sulfur, increases. By analogy with ore flotation used to process over 400 million metric tons of ores in the U.S. per year (1), coal flotation offers the potential of being an effective, large tonnage, low cost means of removing pyritic sulfur from coal. The need to crush raw coal to finer sizes in order to liberate additional fine pyrite from the coal should make this fine cleaning process even more attractive. However, with only about 30 million tons of coal processed by flotation

(2), the method is obviously far from achieving its potential. The many reasons it has not been more widely adopted have been summarized by Aplan (3), but chief among these is the less than ideal removal of pyritic sulfur from coal presently achievable.

Pyritic sulfur can be removed from fine coal by flotation in one of the following ways:

1. Improved flotation circuitry, machines, and processes.
2. Depression of pyrite and flotation of coal.
3. Depression of coal and flotation of pyrite.

Use of the last two methods has shown a wide variation in their effectiveness. The direct depression of pyrite works well on some raw coals and poorly on others while the reverse process suffers from the same problem and in addition requires reagents in quantities often very much greater than that required for ore flotation. A further complication is the finding in our laboratories that nearly every effective pyrite depressant is also a coal depressant at a similar or at a greater concentration. Thus, the proper selection of a pyrite depressant dictates that the experimentalist search for a 'window of opportunity' which either provides good pyrite depression without significant coal depression, or vice versa.

Our studies have shown that the polysaccharides and similar natural or synthetic polymers can be excellent depressants for both pyrite (4,5) and coal (6). The present report is a part of a larger study (4-6) on evaluating reagents which can depress pyrite during coal flotation or depress coal for pyrite flotation (the two-stage, reverse process proposed by Miller (7)). This study will focus specifically on the adsorption of these polymers onto coal, and the evaluation of those flotation conditions which allow for the depression of pyrite without at the same time seriously interfering with the flotation of coal.

EXPERIMENTAL MATERIALS AND METHODS

The coal used in these studies was from the Pittsburgh seam, Robena mine, Greene Co., PA. This cleaned, high volatile A bituminous coal had a proximate analysis (dry basis) of 7.11 % ash, 35.30% volatile matter, 57.60% fixed carbon, 1.14% total sulfur (of which 0.70% was pyritic sulfur) and 14,153 BTU/lb. The polymers used, together with their identifying characteristics, are given in Table 1. Their structures have been given previously (5,6), and greater detail is available in the chemical literature (8,9).

For the polymer adsorption studies, the as-received clean coal was picked to eliminate pieces containing obvious impurities. It was then crushed and screened to produce a 100x400 mesh fraction. This fraction had a surface area of 0.0725 m^2/g by

TABLE 1. Polymers Used for Adsorption and Flotation Studies

Ident. No.	Trade Name	Manufacturer	Charge	Identification
1	Hylon VII	Nat'l. Starch	(0)	Corn starch, high amylose ($\sim$60%)
2	Amioca	Nat'l. Starch	(0)	Corn starch, high amylopectin ($\sim$100%)
3	CPC 3005	CPC Int'l.	(0)	Corn starch, unmodified, amylose $\sim$ 25%
8	Sta-Lok 1303	Staley	(+)	Corn starch, cationic, amylose $\sim$ 25%
9	Cato-2	Nat'l. Starch	(+)	Corn starch, cationic, amylose $\sim$ 25%
10	Cato-14	Nat'l. Stacrh	(+)	Corn starch, cationic, high amylopectin
12	Jaguar MDD	Celanese	(0)	Guar gum, unmodified
13	Jaguar C-13	Celanese	(+)	Guar gum, quatemary, DS $\sim$ 0.13
14	Gendriv 162	Henkel	(+)	Guar gum, cationic
15	Gendriv 307	Henkel	(-)	Guar gum, carboxymethyl ether, Na form
17	Irish Moss	Meer Corp.	(0)	Mostly λ carrageenan
18	CPC 8071	CPC Int'l.	(0)	Corn starch dextrine, canary
19	CMC 4H1	Hercules	(-)	Carboxymethyl cellulose, DS=0.4, high vis.
20	CMC 7H	Hercules	(-)	CMC, DS=0.7, high viscosity
21	CMC 12M31	Hercules	(-)	CMC, DS=1.2, med. viscosity
22	Natrcsol 150 GXR	Hercules	(-)	Hyydroxethyl cellulose (HEC), MS=1.5
23	Natrosol 250 MR	Hercules	(-)	Hydroxyethyl cellulose (HEC), MS=2.5

D.S. = degree of substitution
M.S. = moles of substituent, combined.

Blaine air permeametry. This area was used in preference to a BET surface area because the fine pore structure of coal measured by nitrogen gas adsorption would not always be accessible to high molecular weight polymers. The coal was mixed with 10 ml of a 150 ppm solution of the desired polymer (an amount found by prior test to be sufficient to produce an adsorption density not sensitive to concentration effects), the pH was adjusted by adding NaOH or HCl and the mixture was shaken for 10 minutes. The coal particles and solution were separated and the residual polymer concentration determined by U.V. spectroscopy at $\lambda = 486$ nm using the phenol-sulfuric acid method of Dubois et al. (10). In some cases xanthated polymers were used and in these cases the characteristic xanthate adsorbance at $\lambda = 301$ nm was used.

Desorption of the adsorbed polymer on coal or pyrite was done by contacting the coated solid with water and agitating for 10 min. The sample was filtered and the polymer concentration in the filtrate determined. The procedure was repeated several times to generate a desorption curve.

The zeta potential of the coal was determined by use of the Zeta Meter. Flotation tests were run in the older-style WEMCO-Fagergren flotation cell using 200 g (190 g. coal and 10 g. purified pyrite from the same seam), freshly crushed to a nominal -28 mesh. Reagents used were 0.24 kg/t MIBC, 0.04 kg/t No.2 fuel oil and 0.1 kg/t of the polymer under study. Greater details of all the procedures used may be found elsewhere (5,11).

EXPERIMENTAL RESULTS AND DISCUSSION

Polymer Adsorption onto Coal

The adsorption of a series of nonionic polysaccharides is given in Fig. 1. The average adsorption density (mg/m^2) onto coal is substantially less, on average, then is the adsorption of these same polymers onto pyrite from the same coal seam (5). Further, the adsorption of these non-ionic polymers onto coal is only modestly influenced by pH, whereas, polysaccharides in general invariably show their greatest adsorption onto pyrite at pH 7, influenced only modestly by the charge on the polysaccharide. This has been attributed by Perry and Aplan (5) to the formation of an iron-polysaccharide complex at the surface of the pyrite at this pH.

Fig. 1 shows that the high amylose content, straight chain Hylon VII and the corn starch dextrine, CPC 8071 are adsorbed to the highest degree. Prior work (6) has shown that dextrines are among the strongest of coal depressants, though neither amylose, amylopectin, nor ordinary corn starch (CPC 3005) were found to be particularly effective as coal depressants for a low volatile bituminous coal. This seeming riddle may be explained by recalling that depression during flotation is a two step process:

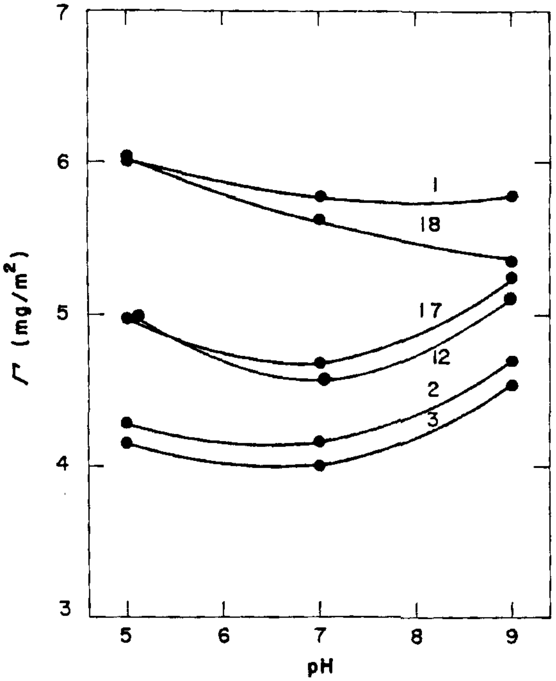

Fig. 1. Adsorption Density Profile of Nonionic Compounds onto Pittsburgh Seam Coal.
1 - Hylon VII (0) 12 - Jaguar MDD (0)
2 - Amioca (0) 17 - Irish Moss (0)
3 - CPC 3005 (0) 18 - CPC 8071 (0)

(1) the adsorption of the reagent onto the solid to be depressed, and (2) the establishment of a hydrophilic coating which will reject an air bubble. While amylose is strongly adsorbed into this coal, in its adsorbed form it may not provide a sufficient number of available hydroxyl groups to reject air bubbles. Alternatively, the adsorption of these components onto coal may differ with differing ranks of coal. The adsorption superiority of the straight chain high amylose starch (Hylon VII) over the branched amylopectin starch (Amioca) demonstrates the counter-productive steric effect of branching on adsorption. This factor has also been noted by Haung et al. (12).

Fig. 2 shows that all of the anionic polymers act similarly in that they are all much better adsorbed at pH 5 than at pH 9.

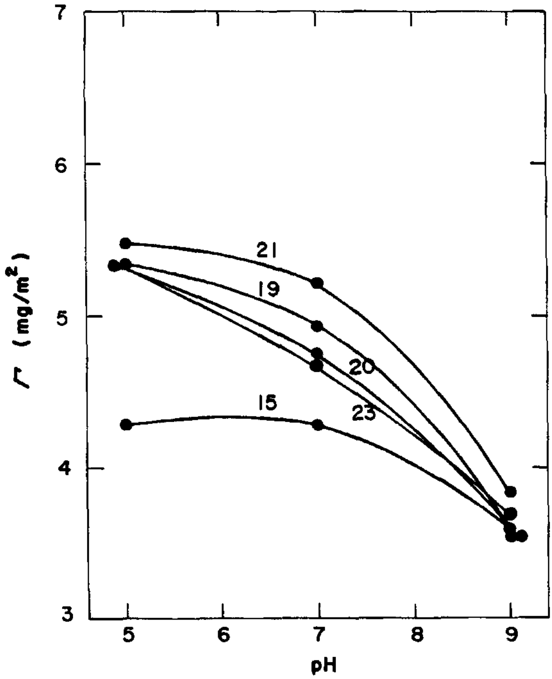

Fig. 2. Adsorption Density Profile of Anionic Compunds onto Pittsburgh Seam Coal.

15 - Gendriv 307 (-)	21 - CMC 12M31 (-)
19 - CMC 4H1 (-)	23 - Natrosol 250 MR (-)
20 - CMC 7H (-)	

By way of contrast, Fig. 3. illustrates that the adsorption of cationic polysaccharides follows just the opposite pattern with high adsorption densities noted at pH 9. The adsorption of two cationized corn starches (Sta-Lok 1303 and Cato-2) is seen to be substantially different. Though the production methods are proprietary, a review of the patent literature (13) suggests that Cato-2 (National Starch) was probably made by reaction with epichlorhydrin and ammonia (U.S. Pat. 3,666,751) while Sta-Lok 1303 (Staley) was probably made by quaternary ammonia etherification (U.S. Pat. 3,346,563). Interestingly enough, the Sta-Lok 1303 (though better adsorbed onto coal, see Fig. 3) has previously been found to be an inferior coal depressant to Cato-2 (6). Perhaps different hydroxyls in the structure have been cationized by the differing procedures.

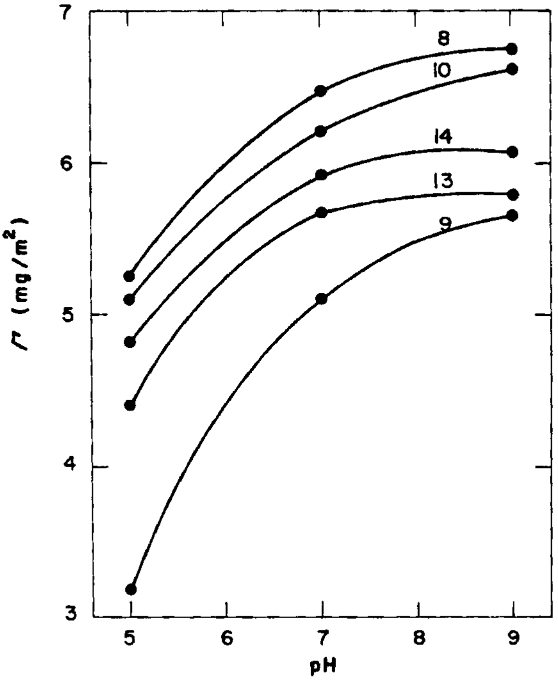

Fig. 3. Adsorption Density Profile of Cationic Compounds onto Pittsburgh Seam Coal.
8 - Sta-Lok 1303 (+)
9 - Cato-2 (+)
10 - Cato-14 (+)
13 - Jaguar C-13 (+)
14 - Gendriv 162 (+)

An overall comparison of the adsorption of polymers onto coal and pyrite is given in Fig. 4. It will be readily seen that, on average, the adsorption density of the polymers used here is much greater onto pyrite than onto coal. The increase in polysaccharide adsorption onto pyrite at pH 7, likely due to the formation of an iron-polysaccharide complex (5), is clearly seen. Xanthating the polymers at the 10% level (i.e., sufficient CS_2 reactant is added to react with 10% of the available OH groups), gave a strong adsorption onto pyrite at pH 5 (5). Though the data for the adsorption of xanthated polysaccharides onto pyrite have been omitted from Fig. 4 to avoid crowding, the range of adsorption density was 4.1 to 13.8 mg/m^2 with an average of 9.1 mg/m^2 at pH 5 (5). However, at pH 9 the average adsorption density was 4.4 mg/m^2 (range: 2.2-8.5) (5). This substantial

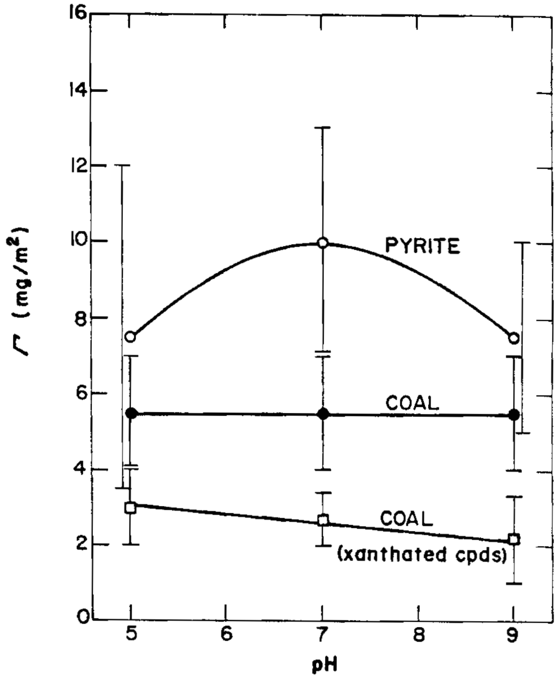

Fig. 4. The Average (Data Points) and Range (I) of Adsorption Density for Various Systems.
○ - Non-xanthated onto Pittsburgh Seam Pyrite, λ = 486 nm
● - Non-xanthated onto Pittsburgh Seam Coal, λ = 486 nm
☐ - Xanthated onto Pittsburgh Seam Coal, λ = 301 nm

decrease in the amount of the xanthated polymer on pyrite is not surprising since xanthates are adsorbed well onto pyrite only at neutral to slightly acidic pH values (14).

By xanthating the polymers the average adsorption density onto coal is decreased from that noted with the unxanthated compounds (Fig. 4). The xanthating process, which will usurp the most reactive hydroxyl groups, results in a polysaccharide less well adsorbed onto coal. This infers that the attachment of a polysaccharide to coal functions through these active OH groups; most likely by hydrogen bonding with OH groups on the coal surface. Previous work has also indicated that other steric effects in polysaccharides are important since the β/cis configuration (as in guars) is more strongly adsorbed onto pyrite than is the α/trans (starches) or β/trans (cellulose) forms (5). No clear configuration pattern is seen in Figs. 1-3 for the adsorption of similar polysaccharides onto coal.

Desorption

As illustrated in Fig. 5, once a polysaccharide is absorbed onto either coal or pyrite, it clings tightly to the surface with most of it remaining adsorbed even after repeated washing. Though not shown here, the desorption of five other polysaccharides was also evaluated. Except in one case, the amount desorbed from either coal or pyrite did not exceed 21% (11). In the case of the Amioca and CPC 3005 xanthated compounds, the amount desorbed from pyrite was zero in the range pH 5-9. For coal, about half of the 10% xanthate of ordinary corn starch, CPC 3005, can be washed from the surface at pH 7 or 9 but none of the Amioca xanthate at pH 5 to 9.

Adsorption Mechanisms

Based on the literature data for the adsorption of polysaccharides and other polymers onto a variety of minerals (mostly oxides), Im and Aplan (6) concluded that the following mechanisms are most likely for the adsorption of similar organic polymers onto coal:

1. Electrostatic attraction (15,16)
2. Hydrogen bonding (15,16)
3. Ester formation at the surface (15)
4. A 'squeezing out' effect due to the lower potential energy of the polymer molecule at the surface than in solution (17)
5. Hydrophilic bonding with the polymeric depressant displacing water molecules at the surface (18).
6. Specific adsorption through selected active groups (19).

Because Figs. 2 and 3 showed the likelihood of electrostatic attraction, the zeta potential of this coal was determined as a function of pH (Fig. 6). The isoelectric point (IEP) was found at pH 4.5 shifting downward to pH ∿ 4.0 upon aging for 24 hours. This likely reflects some surface oxidation since Wen and Sun (20) have shown that a decrease in the IEP of coal results from oxidation. Note that the zeta potential of the coal is slightly negative at pH 5 and strongly negative at pH 9. These data are compatible with the data for Fig. 2 showing a modest preference of an anionic polymer for the coal surface when it is slightly charged negatively and poor adsorption for the strongly negative

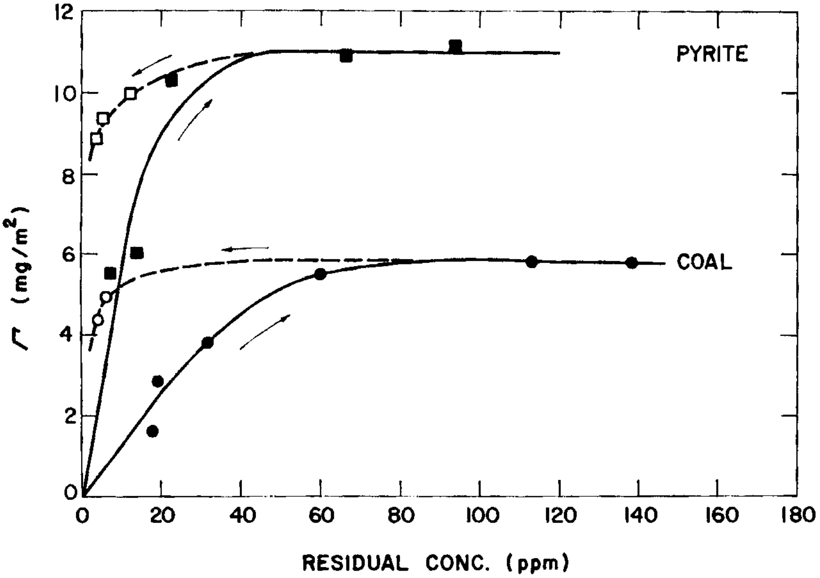

Fig. 5. Adsorption and Desorption Curves for Hylon VII onto Pittsburgh Seam Pyrite and Coal. pH 7.

■- Pittsburgh Seam Pyrite Adsorption Isotherm
□- Pittsburgh Seam Pyrite Desorption Isotherm
●- Pittsburgh Seam Coal Adsorption Isotherm
○- Pittsburgh Seam Coal Desorption Isotherm

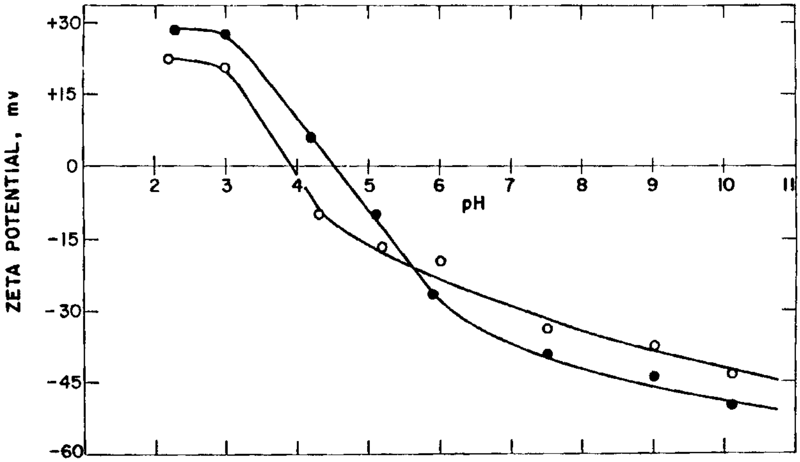

Fig. 6. Zeta Potential of Pittsburgh Seam Coal (Ground Dry), Fresh and Aged.

●- Fresh ○- Aged (24 Hours)

surface at pH 9. Cationic reagents, on the other hand, are strongly adsorbed to a strongly negative surface (Fig.3). These data strongly support the electrostatic attraction premise.

Other mechanisms are obviously also operating since all of the compounds used here, irrespective of their charge or the charge on the coal, are reasonably well adsorbed onto coal. The authors believe that the most likely of the mechanisms is hydrogen bonding. The data of Fig. 4 supports this premise since xanthating the polysaccharide, which would be expected to occur at the most active hydroxyl sites, results in a substantial decrease in the adsorption density of the polysaccharide onto coal.

Specific adsorption of the polysaccharide onto coal could also occur by formation of an iron-polysaccharide complex at the surface, as iron is universally present in coal in the form of, e.g., pyrite and iron oxides. Ester formation is probably not very important here because the high rank coal used (HVA bituminous) contains a negligible carboxylic content, though ester formation may well be an important mechanism for adsorption of an organic polymer onto sub-bituminous and lignite coals which contain a plethora of carboxylic groups. No definite evidence is available here on the other two possible mechanisms.

Flotation Testing

The overall flotation data for one minute of flotation is shown in Table 2. This was a fast-floating coal and the results for a two minute flotation time were similar except the yield was increased ∿ 5% and the sulfur content of the floated coal also increased. Note that some of the tests were excellent, producing low sulfur contents at relatively high yield (∿ 80%). Unfortunately, some of the compounds seriously depress coal. These were found to be largely dextrine, Irish moss and guar gums, carboxymethyl cellulose and hydroxyethyl cellulose. Flotation data on these systems have been reported in greater detail previously (5) or may be found in our primary report (11). A summary of the flotation results obtained by using these polymers as pyrite depressants, and their influence on pyrite rejection and coal flotation, is given in Table 2. A low % S in the clean coal resulting from flotation indicates pyrite depression and a high yield indicates the coal was not significantly depressed. The following features for the flotation of this Pittsburgh seam coal can be seen from the tabulated data:

1. The coal yield at pH 5 is generally better than at pH 7 or 9.
2. The lowest sulfur content of the clean coal frequently occurs at pH 7.

Table 2. Flotation Results Using Polymeric Depressants to Depress Pyrite
(Heads: 3.4%S; flotation time: one minute.)

Compound	pH	%S*	% Yield	Compound	pH	%S*	% Yield	Compound**	pH	%S*	% Yield
Hylon VII	5	1.18	78	Jaguar MDD	5	1.17	59	Hylon VII-X	5	1.12	83
(amylose)	7	1.13	68	(guar gum)	7	1.10	58		7	1.13	60
	9	1.12	55		9	1.16	44		9	1.14	69
Amioca	5	1.29	77	Gendriv 162	5	1.27	57	Amioca-X	5	1.25	76
(amylopectin)	7	1.23	66	(cationic	7	1.23	56		7	1.23	67
	9	1.27	59	guar)	9	1.24	42		9	1.26	68
CPC 3005	5	1.27	78	Gendriv 307	5	1.21	60	CPC 3005-X	5	1.16	87
(corn starch)	7	1.19	80	(anionic	7	1.18	56		7	1.21	80
	9	1.19	66	guar)	9	1.25	43		9	1.22	69
Cato-2	5	1.18	82	Irish Moss	5	1.21	33	Cato-2-X	5	1.17	74
(cationic corn	7	1.15	76	(λ Carrageenan)	7	1.15	38		7	1.16	72
starch)	9	1.19	57		9	1.16	48		9	1.13	73
Cato-14	5	1.34	79	CMC 7H	5	1.29	48	CPC 8071-X	5	1.22	51
(cationic	7	1.25	76	(carboxymethyl	7	1.24	31		7	1.12	46
amylopectin)	9	1.27	65	cellulose)	9	1.29	68		9	1.17	51
CPC 8071	5	1.22	26	Natrosol 150GxR	5	1.20	56	Gendriv 162-X	5	1.19	69
(corn starch	7	1.17	31	(hydroxyethyl	7	1.14	47		7	1.18	50
dextrine)	9	1.19	23	cellulose,MS 1.5)	9	1.15	33		9	1.12	49
								Irish Moss-X	5	1.21	69
									7	1.12	52
									9	1.21	53

* % Sulfur in the floated coal (1 minute)

** Xanthated at the 10% level

3. The best pyrite depressants in terms of producing a low sulfur clean coal product (< ∿1.2% S) and at high coal yield (∿ 80%) are Hylon VII, Cato-2 and CPC 3005, and their xanthates. The preferred pH is generally at pH 5 or 7, especially if a higher coal yield is to be obtained.
4. Dextrine CPC 8071, guar gum (Jaguar MDD), anionic guar gum (Gendriv 307), Irish moss and HEC (Natrosol 150GXR) also produced a low sulfur clean coal in various cases. Unfortunately, they also were found to seriously depress coal under these same conditions.
5. Hylon VII, with a high content of straight chain amylose was found to produce a lower sulfur product than did the branch chain, high amylopectin, Amioca at a comparable coal yield.
6. Xanthating Hylon VII, CPC 3005, Gendriv 162 and Irish Moss at the 10% level was found to result in a decreased depression of coal (i.e. gave a higher yield) in several instances. Xanthating the dextrine, CPC 8071, resulted in a substantial increase in coal yield at all pH levels. This infers that xanthating decreases the number of active hydroxyl groups that would otherwise facilitate attachment of these polymers to the coal and result in subsequent coal depression.

CONCLUSIONS

The following general conclusions may be drawn from the data for Pittsburgh seam coal and pyrite:

1. In general, polysaccharides are much more strongly adsorbed onto pyrite than onto coal. This provides a 'window of opportunity' to select a concentration which will depress pyrite without, at the same time, seriously depressing the coal.
2. The experimental evidence indicates that adsorption of these polymers onto coal probably occurs through hydrogen bonding and electrostatic attraction.
3. The IEP of this Pittsburgh seam coal sample was found at pH 4.5. This decreased to pH 4 upon gentle oxidation (aging).
4. Polyaccharides are adsorbed onto coal, at least in part, by electrostate attraction. Cationic polysaccharides are strongly adsorbed onto coal at pH 9 where the coal shows a strong negative zeta potential, whereas anionic compounds are less well adsorbed at this pH. The adsorption of non-ionic polysaccharides is not greatly influenced by pH.
5. Xanthated polysaccharides are less well adsorbed onto coal than are their non-xanthated counterparts. This infers that the xanthating process has unsurped some of the active OH groups that would otherwise facilitate

polysaccharide attachment to the coal through hydrogen bonding.

6. Some evidence was found that steric accessibility of hydroxyl groups influences polymer adsorption onto coal.
7. There is a high degree of variability in the effectiveness of polysaccharides as pyrite or coal depressants during flotation.
8. A high amylose content corn starch (Hylon VII), ordinary corn starch (CPC 3005) and a cationic corn starch (Cato-2) were found to be the best pyrite depressants in terms of a low sulfur content of the clean coal and a high coal yield. The adsorption density of polysaccharides onto coal gives an imperfect correlation with coal depression during flotation.
9. Dextrine, guar and Irish moss gums and carboxyethyl and hydoxyethyl cellulose are relativly good pyrite depressants but they seriously depress coal.
10. The straight chain, high amylose content starch, Hylon VII is a superior pyrite depressant at a comparable coal yield than is the branch chain, amylopectin starch, Amioca. The Hylon VII is more strongly adsorbed onto both pyrite (5) and onto coal than is the Amioca.
11. In the presence of these polymeric depressants, coal generally floats better at pH 5 than at pH 7 or 9.
12. Once adsorbed onto either pyrite or coal, polysaccharides are not easily desorbed by simple water washing.

Work is in progress to determine if these conclusions can be extended to other coals.

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