
Selective flotation of fossil resin from Wasatch Plateau high-volatile bituminous coal

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Abstract — *Certain bituminous coals are known to contain appreciable quantities of natural fossil or subfossil resin. Such resinous coals are found in the western US, particularly the Wasatch Plateau coalfield of UT. Some of the seams in this*

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field contain an average of 5% resin. This fossil resin has been recovered by gravity and/or flotation processes since 1929. Resin concentrates thus produced are of low quality and are usually refined by solvent extraction. The purified resins are of commercial importance in the adhesive, coating, rubber, and ink industries, etc. An improved flotation technique has been developed which involves ozone conditioning to selectively float resin from high-volatile bituminous coal. With this flotation process, a concentrate product which contains 95%

resin at a recovery of 80% can be obtained in single-stage flotation. By comparison, conventional flotation at 80% recovery produces a concentrate having a resin content of 30 to 40%.

Introduction

Many bituminous coals are known to contain small quantities of natural (fossil or subfossil) resin. In the US, highly resinous coals are found in western coalfields, particularly the Wasatch Plateau coalfield in UT (Spieker and Baker, 1928; Tomlinson, 1932; Thiessen and Sprunk, 1937; Crawford and Buranek, 1952). The geographic location of the Wasatch Plateau coalfield together with mine locations is presented in Fig. 1. Some of the seams in this field average even more than 5% resin. Such resinous coals are also found in Colorado, New Mexico, and Wyoming.

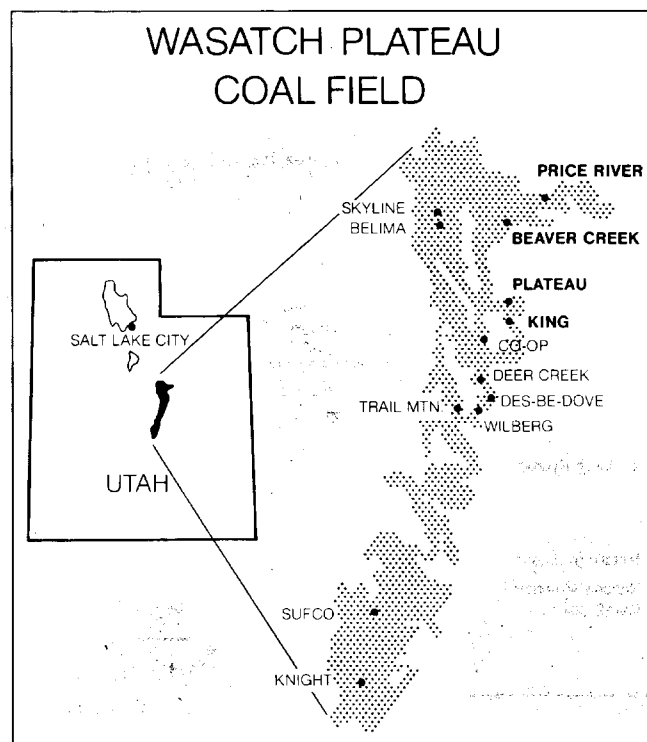


Fig. 1 — Mine locations in the Wasatch Plateau coalfield. Preparation plants located at Price River, Beaver Creek, King, and Plateau.

Fossil resin has been recovered from the Wasatch Plateau coalfield intermittently since 1929 by gravity and/or flotation processes. Resin production nevertheless has been on a very small scale. Of the four coal preparation plants in the Wasatch Plateau coalfield (US Fuel, Plateau, Beaver Creek, and Price River), only one plant (US Fuel) recovers this valuable resource at a rather low level of production.

Resin concentrates thus produced are usually refined by solvent extraction and precipitation. Such purified resins typically have a molecular weight of about 730, a melting point of about 170°C, and an iodine number of about 145. The product, at the present time, has a market value of approximately \$1.00/kg and is used in the adhesive, rubber, varnish, enamel, paint and coatings, thermoplastics, and ink industries. Most recently, it has been suggested that these resins could have a special value as feedstock for high density jet fuels.

In the recovery of resin from coal, most of the resin phase is liberated at a relatively coarse size, approximately 20 mesh, and has a variable shade of resinous yellow/brown color. In addition to its color, the resin is distinguished by a specific

gravity of about 1.00 to 1.08 g/cm³. From a chemical standpoint, the resin is characterized as being considerably less aromatic than other coal macerals with an aromaticity of 0.16 to 0.23, compared to more than 0.60 for other coal macerals as determined by nuclear magnetic resonance (Wender, et al., 1981). Recent thermogravimetric and spectrometric (MS, NMR) analysis suggests that the fossil resins from the Wasatch Plateau region consist of macromolecular aggregates composed of polymerized sesquiterpenoids (Crelling, et al., 1987); see Fig. 2. Further, the structural difference between resin and other coal macerals manifests itself by a slight difference in the respective hydrophobicities of the two components as revealed in Table 1.

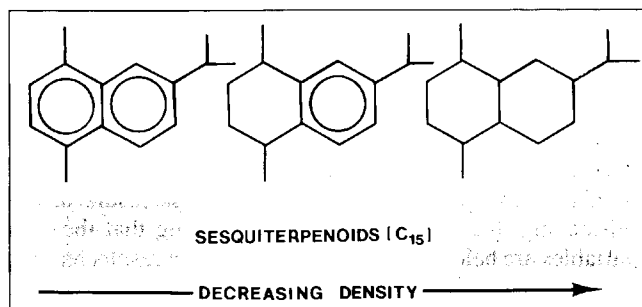


Fig. 2 — Sesquiterpenoid structures similar to the fossil resin structure found in Utah coals.

Table 1 — Bubble Attachment-Time Measurements for Resin and Coal Particles from the Hiawatha Seam, Utah (Particle Size 212x300µm, pH 6.5) and Contact-Angle Measurements for Polished Samples (Miller and Ye, 1988).

Component	Bubble Attachment Time, ms	Contact Angle, degrees
Resin	5.0	58-59
Coal	15.0	48-51

From this small difference in the respective hydrophobicities, it might be expected that a flotation separation would be possible as has been reported (Green, 1930; Klepetko, et al., 1947, 1950, 1952). However, because the difference in the respective hydrophobicities of coal and resin is not great, the flotation separation of resin from coal is poor, and a multistage flotation circuit is needed even to produce a resin concentrate of modest quality. In this regard, efforts have been made to improve resin flotation from coal by controlled reagent addition (Laros and Pick, 1983) and by using coal depressants/oxidants (Arabidze, et al., 1980). Only modest success has been realized.

Also, it has been found that the high-capacity air-sparged hydrocyclone is effective for the flotation of fine resin from coal using a traditional reagent schedule. Resin concentrates containing about 50% resin can be produced in a single stage of flotation at a dry solids specific capacity of about 91 t/d/m³ (100 tpd per cubic ft) of cell volume (Miller, et al., 1986).

Research efforts at the University of UT have been devoted to finding a more effective coal depressant in order to increase the difference in hydrophobicity between resin and coal and hence make a more selective flotation separation. The results from this research program are reported herein, and, most significantly, it has been found that ozone conditioning for coal depression is particularly effective (Miller and Ye, 1988).

Evaluation of flotation reagents

The first effort made in this study was to evaluate flotation strategies to improve the separation efficiency for resin concentration. The resin/coal slurry samples were taken from a

thickener underflow product from the US Fuels preparation plant at Hiawatha, UT, where coarse Hiawatha-seam coal is cleaned and the fines are discarded. The resin content of these samples varies from 7.2 to 14.0%. Table 2 lists the flotation reagents considered. As can be seen from the table, a variety of combinations of frother, collector, and depressant were examined. Table 3 gives the best flotation results obtained for each case. The flotation was carried out in a 3-L Galigher flotation machine with an air flow rate of 4 l/min and a stirring speed of 900 rpm.

It is necessary to point out that the results given in Table 3 present only the best flotation results obtained for these different reagent schedules. In many instances, less-efficient separations were obtained at different conditions. Flotation conditions and procedures varied from case to case and have been omitted here to avoid a lengthy description of this phase of the experimentation.

Frother/Collector	Organic Colloids	Electrolyte	Oxidants
Dowfroth 250	Tannic Acid	NaCl	NaOCl/MIBC
MIBC	Tannic Acid/MIBC	NaCl/MIBC	H ₂ O ₂ /MIBC
MIBC/Kerosene	CMC		KMnO ₄ /MIBC
Dowfroth/Kerosene	CMC/MIBC		

Reagent Schedule	Feed Grade, % Resin	Concentrate Grade, % Resin	Resin Recovery, %
Frother only	14.0	62.0	74.0
	7.2	24.9	75.0
Frother/Kerosene	14.0	45.8	82.0
Organic Colloids	14.0	46.6	84.7
Electrolyte	14.0	29.4	90.1
Oxidants	7.2	42.3	74.0

It can be seen from Table 3 that traditional frothers (MIBC and Dowfroth 250) and collector (kerosene) in various combinations did not provide any significant improvement in separation efficiency. Further, in the absence of depressants, the addition of kerosene brought about a decrease in concentrate grade. This limited discrimination between resin and coal is due to the natural hydrophobicities of resin and coal and the bubble attachment times of similar magnitude (although there is some difference as shown in Table 1). Not unexpectedly, with kerosene addition the discrimination is even less due to kerosene wetting of both resin and coal particles, such that both components have even closer hydrophobic character, and a flotation separation becomes even more difficult.

Organic colloid depressants such as dextrin are known to depress coal at low levels of reagent addition (Miller et al., 1983). However, for the organic colloids used in this present study, no selectivity for resin flotation could be achieved.

Salt flotation of coal has been known for some time (Laskowski and Iskra, 1970), and so it was not particularly surprising to find that the resin separation was not improved by the addition of high concentrations of inorganic electrolytes such as NaCl.

With oxidant addition, coal was depressed to a certain extent, and the separation efficiency was improved. The concentrate grade of resin increased to some extent from 24.9 to 42.3% at the same recovery and a feed grade of 7.2%. However, it needs to be emphasized that the quantity of the oxidants needed to gain this improvement was so high that such a reagent schedule could not be justified in terms of reagent cost. Clearly, these oxidants were not particularly cost-effective in improving the flotation of resin from coal.

From the results presented in Table 3, it is evident that the

separation efficiency for resin recovery was not improved appreciably with these reagents.

However, a significant improvement in resin flotation from coal was found when ozone was used for oxidation, and excellent separations were achieved with concentrates containing 95% resin at 80% recovery, as described in the following section.

Ozone depression

Flotation experiments

For ozone conditioning, the air inlet on the flotation machine was first connected to the outlet of an ozone generator (Model 03B-O, product of Ozone Research and Equipment Co.). The ozone with oxygen as the parent gas was introduced into the resin/coal slurry through the air inlet and sparged naturally into the stirred suspension. The ozone was added at a rate of 0.18 g/min for various times (the conditioning time). After conditioning, MIBC was used as a frother at a dosage of 0.03 kg/t. The flotation was then accomplished with an air flow rate of 4 l/min for 5 min at 900 rpm and 10% solids. The resin content of the feed was at 7.2%. All results reported are for single stage flotation.

The first interesting result showed that the effectiveness of coal depression by oxidation with ozone appears to be significantly affected by the initial surface state of the coal particles, as shown in Fig. 3. Without grinding the feed (zero grinding time), 35.8% of the coal was recovered in the concentrate. But with 1 min of grinding and the same ozone treatment, flotation recovery of the coal was only 2.8%. When the grinding time was increased to 5 min, the recovery of the coal was reduced to less than 1%, producing a concentrate containing 90% resin. The results presented in Fig. 3 further suggest that oxidation with ozone at a fresh coal surface is significantly different than at the resin surface. After oxidation by ozone, the coal particles become strongly hydrophilic and no longer

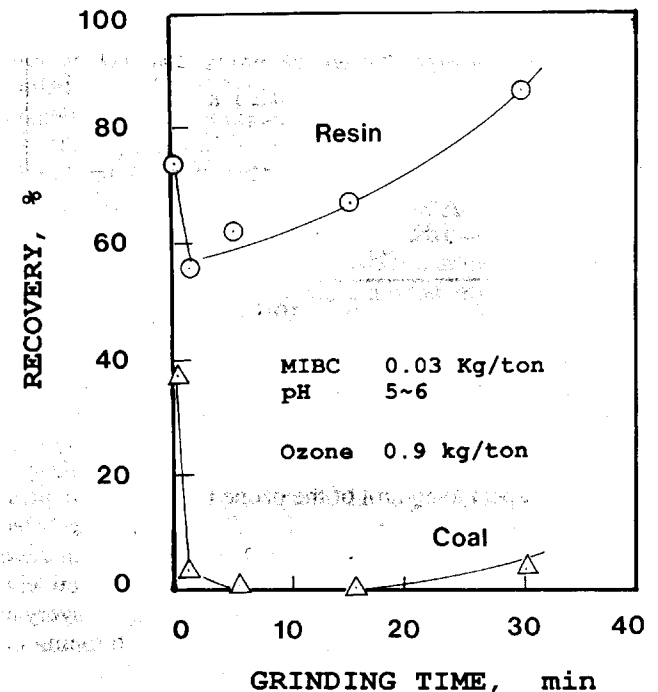


Fig. 3 — Single-stage flotation recovery of resin from coal with the ozone depression technique as a function of grinding time (grinding done at 50% solids).

floatable, but the resin particles still retain their hydrophobicity to a certain extent, so that a selective separation is possible. Furthermore, Fig. 3 indicates that the flotability of oxidized resin is directly related to its particle size. The flotation recovery of resin increases as the particle size becomes finer (longer grinding time). The size analyses of the ground particles for these experiments are given in Table 4.

Table 4 — Size Analyses of Resinous Hiawatha Coal at Different Grinding Times

Size mesh	% Weight Grinding Time, min			
	1	5	15	30
>50	6.6	3.2	0.6	0.1
50/70	13.1	4.8	2.3	0.9
70/100	10.9	5.0	3.4	1.1
100/140	16.4	14.7	6.7	4.3
140/200	8.2	9.9	8.0	5.1
200/325	5.3	13.1	14.9	11.6
325/400	9.2	13.6	19.7	14.6
<400	30.3	35.7	44.4	62.3

Although the flotability of oxidized resin can be improved as the particle size becomes finer, extensive oxidation with ozone will eventually depress the resin as found in another set of experiments, the results from which are shown in Fig. 4. In these experiments, the flotation recovery of resin from material ground for 30 min was tested at different ozone-conditioning times. From the data presented in Fig. 4, the resin is significantly depressed when the ozone-conditioning time is greater than 100 sec (1 kg/t solids). It is evident that proper control of the ozone addition is a critical feature for selective flotation of fossil resin from coal.

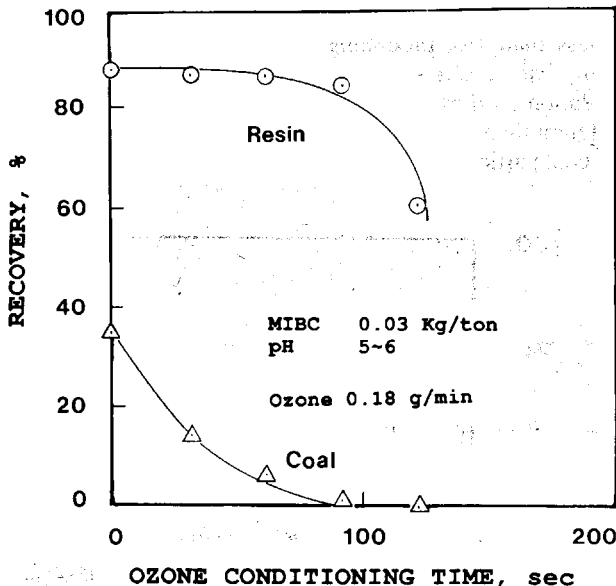


Fig. 4 — Single-stage flotation recovery of resin from coal as a function of ozone-conditioning time (grinding done at 50% solids for 30 min).

With respect to control of the ozone level in coal depression, a set of experiments for the four different samples listed in Table 4 was carried out. Ozone-conditioning time in these experiments was controlled at 30, 60, and 90 sec, respectively. In Fig. 5, the results are plotted as resin grade vs. recovery in the concentrate so that the effect of ozone conditioning on both grade and recovery can be represented. The highest recovery observed corresponds to 30 sec of ozone conditioning while the lowest recovery is for 90 sec of conditioning. The results obtained without ozone conditioning (closed

symbols) are also included in Fig. 5 for comparison. It is evident from Fig. 5 that ozone conditioning is an effective way to significantly improve the flotation separation efficiency for resin recovery from coal. For example, in the case of 30 min of grinding, a concentrate containing 95% resin with 80% recovery for single-stage flotation after ozone conditioning can be obtained, while the grade of the concentrate without ozone conditioning at the same recovery is only 20 to 30% resin.

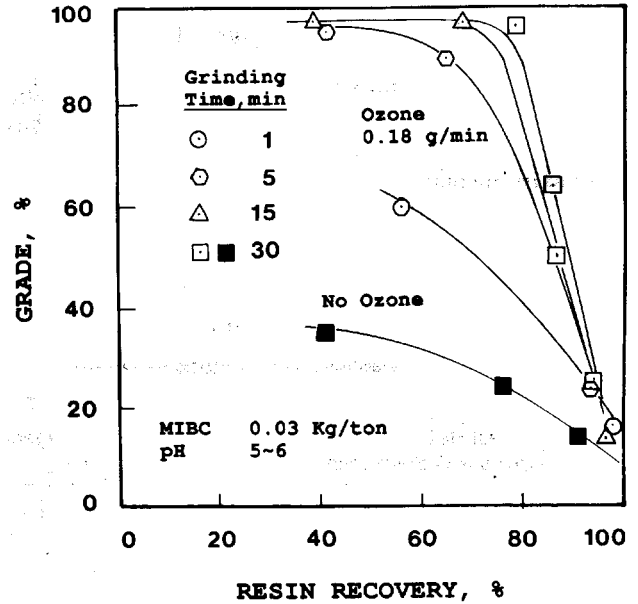


Fig. 5 — Concentrate grade vs. recovery for single-stage flotation with (open symbols) and without (closed symbols) ozone pretreatment for different grinding times.

Bubble attachment-time measurements

Flotation separation phenomena can be well explained from bubble attachment-time measurements for different particle components of a mixture. The bubble attachment time (or induction time) is a function of surface chemistry, particle size, pH, temperature, surfactant concentration, etc. (Eigeles and Volova, 1960; Yordan and Yoon, 1985; Ye and Miller, 1988; Ye, et al., 1989). Nevertheless, with all these parameters fixed, bubble attachment-time measurement (kinetic criterion) can provide a rather precise description of hydrophobic character and flotation response.

Bubble attachment-time measurements in the present study were made with an electronic induction timer (product of Virginia Coal and Mineral Service, Inc.) at a particle size of 212x300 μm and pH 6.5. Because the actual contact time between the bubble and the particle bed in the apparatus can be biased due to the motion characteristics of the bubble, the measured attachment time was carefully monitored and calibrated with an SP 2000 high-speed video system. In the bubble attachment-time experiments, fresh-ground resin or coal particles were placed into a beaker and gently stirred. The ozone gas with oxygen as the parent gas was introduced at the bottom of the beaker and naturally sparged through the system at an ozone flow rate of 0.18 g/min. After a given conditioning time, the oxidized particles were filtered and transferred to the induction timer apparatus for attachment time measurements.

The effect of ozone oxidation on bubble attachment time is shown in Fig. 6. First, the results show that a small difference in natural hydrophobicity of resin and coal, represented by a

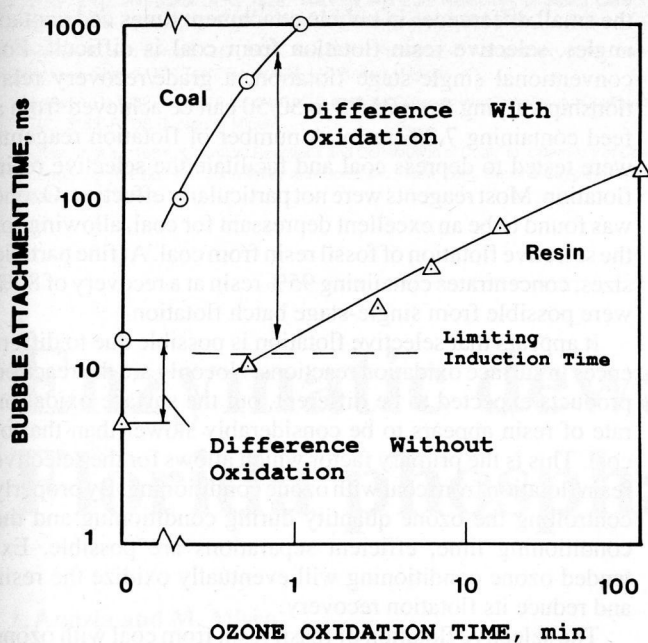


Fig. 6 — Measured bubble attachment times of fossil resin and coal for a particle size of 212x300 μm at pH 6.5 as a function of ozone pretreatment time.

small difference in induction times (5 ms for resin and 15 ms for coal), limits selective separation of fossil resins from coal. Second, at a particle size of 212x300 μm and with MIBC addition, a limiting bubble attachment time for flotation is about 10 to 13 ms as determined experimentally. Because the intrinsic bubble attachment time for coal particles is close to the limiting bubble attachment time, a portion of the coal particles are unavoidable floated into the froth product, lowering the concentrate product quality. Third, the attachment times for both resin and coal particles increase with ozone oxidation, but the increase in the attachment time for resin particles is at a much slower rate than the rate of increase in the attachment time for coal particles. Thus, by controlling the oxidation time at about 1 minute, the ratio of the attachment time for coal particles to the attachment time for resin particles becomes about 67 (compared to a ratio of 3 in the absence of oxidation). Under these circumstances, excellent separation and recovery of resin from coal can be achieved. A further increase in oxidation time should be avoided because the attachment time of the resin particle will far exceed the limiting attachment time, causing a drop in flotation recovery. These conclusions have been confirmed by the results from flotation experiments.

Low-Grade Feed

Other low-grade feed material from the Castlegate seam was supplied by Amax Coal, Price River operation. This material analyzed 0.62% resin and was ground to -100 mesh. Excellent fossil resin recovery was realized in single-stage batch flotation as revealed from the data presented in Table 5. In this case, multistage flotation appears to be necessary to achieve high quality resin concentrates.

Discussion

As illustrated by the experimental results, a small difference in natural hydrophobicity between resin and coal, represented by a small ratio of bubble attachment times ($\tau_{\text{coal}} / \tau_{\text{resin}}$), limits the selective flotation of resin from coal unless the

Table 5 — Selective Flotation of Fossil Resin from Low-Grade Castlegate Feed by Ozone Pretreatment (0.2 kg/ton)

Product	wt %	Resin, %	Resin Recovery, %
Concentrate	1.7	35.70	97.9
Tailings	98.3	0.01	2.1
Feed	100.0	0.62	100.0

coal particles are strongly depressed. Of the flotation reagents investigated, ozone treatment was found to be the most effective, requiring only a relatively small addition due to its high oxidation potential and specific reactivity at the more aromatic coal surface.

Fundamental considerations

There is little known about the oxidation of a coal surface with ozone. Unlike other oxidants such as HNO_3 , $\text{K}_2\text{Cr}_2\text{O}_7/\text{HNO}_3$, $\text{KMNO}_4/\text{OH}^-$, BUOOH/AIBN , NaOCl/OH^- , or peracetic acid, which selectively oxidize only the aliphatic portions of complex coal molecules, ozone oxidation seems to be different. In the case of oxidation with these oxidants, a polymeric amorphous and polydispersed product can be formed, which is often identified as a humic acid. The major functional group of the humic acids has been known for a long time to be the carboxyl group (Howard, 1945). Other constituents such as phenolic, alcoholic, carbonyl, and methoxyl groups are also present in humic acids in significant amounts (Kinney and Keschner, 1952). Furthermore, these humic acid compounds have been shown to influence the flotation response of coal in a complex manner (Laskowski, et al., 1986). Some authors have suggested that the humic acids thus formed are similar to low-rank coal (Yarab, et al., 1979). As the oxidation process extends further, it results in water-soluble acids with the color of the solution changing from rusty brown toward a yellow-green color and finally disappearing. On the other hand, it is reported that oxidation with ozone directly transforms the coal into dark water soluble acids without the formation of humic acids (Kinney and Friedman, 1952). Ahmed and Kinney (1950) tested the ozone oxidation of humic acids obtained from a bituminous coal and found that the reaction of the humic acids with ozone is relatively slow and that part of the humic acids (approximately 35%) is ozone-resistant.

If these humic acids may be considered to be similar to low-rank coal, it is relatively easy to explain the beneficial effect of grinding to create fresh surfaces for the ozone oxidation process in consideration of Ahmed and Kinney's (1950) work. Uncontrolled, oxidation and hydration at the coal surface in the as-received condition cause a relatively slow oxidation rate, and the surface is relatively ozone-resistant, passivated by humic acids. If this oxidized humic acid layer is removed and a fresh coal surface formed during grinding that is more susceptible to ozone oxidation, then different oxidation products form and the surface becomes strongly hydrophilic. Further, fresh grinding exposes unsaturated bonds at the coal surface, which also facilitates the ozone attack.

The oxidation process at the resin surface is obscure. No research on this topic has been reported in the literature. Moreover, a study regarding the difference in the chemical composition of the resin and coal used in this work has not yet been initiated. However, unlike coal macerals, natural resins from plants are mainly volatile and nonvolatile terpenoids. The fossil or subfossil resins from coal (resinite) would be derived from the nonvolatile terpenoids through oxidation and polymerization. The chemical composition of these fossil

or subfossil resins varies with plant source, extent of polymerization, and conditions of coalification, etc. The western coal in the US is characterized as being formed in the Cretaceous period. It is expected that the oxidation and polymerization of the resin from the Wasatch Plateau coalfield would be less than that of amber, which is quite different, being known to contain mostly succinic, formic, butyric, and cinnamic acids and being known to be relatively difficult to extract with solvents. The relative resistance of the fossil resin to ozone oxidation that has been observed in this present work appears to be caused by its unique chemical composition and structure.

Engineering considerations

Selective flotation of resin from coal with ozone pretreatment should be commercially feasible. Other oxidants have been shown to be less effective for coal depression and require higher addition levels in conditioning and hence are not commercially viable. In the present work, flotation concentrates containing 95% resin with 75% recovery can be obtained for an ozone consumption of about 1 kg/t of coal feed. With today's technology, it is common to produce ozone at an energy demand of as low as 20 kWh/kg O₃ with air being used as the parent gas. Thus, the cost of the ozone pretreatment based on our laboratory work would be 0.05 kWh/kg resin, which is about 2¢/kg of resin concentrate.

For some markets, this high-purity resin concentration (90% resin) can be used without further processing by solvent refining. The typical operating cost for solvent purification adds significantly to the total cost for production. These costs should be reduced considerably if a high-purity concentrate is used as feed to the solvent purification process. Solvent-refined resin typically is sold for \$1.00/kg.

Other costs besides that of ozone generation are associated with the installation of an ozone pretreatment process and include a mill for grinding the feed as well as facilities for decomposition of released ozone in the vent gas after pretreatment. These operating costs together with the ozone generation cost can be reduced to a minimum by proper process design. Based on our experience in the laboratory, most of the ozone introduced into the resin/coal slurry was lost (sparged out) before it dissolved and/or reacted with the coal particles in the slurry. The actual quantity of ozone required is expected to be substantially less than that reported in this study. Thus, more effective ozone utilization can be realized by appropriate reactor design with gas recycle and conditioning at a high solids concentration. Also, the solubility of ozone in water is considerable (Emmerp and Pigford, 1963). The concentrated ozone-bearing water could be used and recycled to facilitate coal depression due to its residual ozone content. Alternatively, one may choose a low concentration gas and condition the slurry for a relatively longer time. Optimization with respect to time and concentration will no doubt reduce the operating costs and ozone generation cost so that it is expected that the operating cost for the process will be less than 2¢/kg of resin. Furthermore, it is recommended that the ozone pretreatment and resin flotation be practiced after fine coal cleaning in the preparation plant. The tailings from the resin flotation would be a low-ash coal/water mixture which can still be utilized for power generation.

Summary and conclusions

Because of their similar hydrophobicities, represented by

the small differences in bubble attachment times and contact angles, selective resin flotation from coal is difficult. For conventional single-stage flotation, a grade/recovery relationship varying from 25/75 to 50/50 can be achieved from a feed containing 7.2% resin. A number of flotation reagents were tested to depress coal and facilitate the selective resin flotation. Most reagents were not particularly effective. Ozone was found to be an excellent depressant for coal, allowing for the selective flotation of fossil resin from coal. At fine particle sizes, concentrates containing 95% resin at a recovery of 80% were possible from single-stage batch flotation.

It appears that selective flotation is possible due to differences in surface oxidation reactions. Not only are the reaction products expected to be different, but the surface oxidation rate of resin appears to be considerably slower than that of coal. This is the primary factor which allows for the selective resin flotation from coal with ozone conditioning. By properly controlling the ozone quantity during conditioning and the conditioning time, efficient separations are possible. Extended ozone conditioning will eventually oxidize the resin and reduce its flotation recovery.

The selective flotation of fossil resin from coal with ozone conditioning should be commercially profitable. The cost of ozone pretreatment is estimated to be less than 2¢/kg of resin produced.

Acknowledgments

The authors gratefully thank R. Jin for experimental help in the evaluation of some flotation reagents and Y. Yang for resin analysis.

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