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REFINING OF FOSSIL RESIN FLOTATION CONCENTRATE FROM WESTERN COAL
First Quarterly Final Report (February 23, 1993 - March 31, 1993)

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During the first quarter of the project, from February 22, 1993 to March 31, 1993, the major work is to conduct project work plan and to organize the research term in order to successfully conduct this fossil resin refining project. Besides the following work has been carried out during first quarter:

I. Sample Identification and Preparation

Under a previous DOE-funded program, University of Utah and APT conducted a serious pilot-plant tests of selective flotation of fossil resin from Wasatch plateau coal (both UPL coal and CO-OP coal mines) of south central Utah. About 200 lbs high grade fossil resin flotation concentrate (approximate 75% resin content) was generated from those pilot-plant flotation tests. The resin flotation concentrate were naturally dried, sampled and storied into one kilo plastic bag for future use. The ash and moisture content of the resin concentrate is found to be 1.23% and 1.03 by weight respectively.

II. Particle Size Distribution of Fossil Resin Flotation Concentrate

The particle size analysis for fossil resin concentrate is firstly carried out by dry screening on a mechanical screen shaker. About two hundred grams of sample was used to screen for 4 hours. Seven size fractions (+28, 28x65, 65x100, 100x200, 200x270, 270x400, -400 mesh) were obtained and weighed. The result of particle size analysis is presented in Table 1. As shown in Table 1, it is seen that most of fossil resin concentrate are very fine or less than 200 mesh (about 82% wt.) which might be suitable for solvent refining.

As concluded from our previous research project, it was found that macroscopic fossil resin is friable and can be easily liberated from other coal macerals. Consequently the fossil resin particles tend to concentrate into the fine sizes during size reduction and coal preparation. Because of this property, the fine resinous coal streams in a coal preparation plant contain more than 6% hexane-soluble fossil resin, even when the run-of-mine coal contains only 3% fossil resin. Thus, the fossil resin flotation concentrate contains more fine resin particles (-200 mesh). Under microscopic examination, it is also found that a notable amount of fine coal particle was still floated into the froth concentrate along with fossil resin particles.

Table 1. Particle size distribution of fossil resin flotation concentrate

Size (mesh)	Weight		Cumulative Weight	
	(gram)	(%)	Over (%)	Under (%)
+28	.5	0.25	0.25	100.00
28-65	4.1	2.08	2.33	99.75
65-100	4.9	2.49	4.82	97.67
100-200	26.6	13.51	18.33	95.18
200-270	45.1	22.91	41.24	81.67
270-400	73.4	37.28	78.52	58.76
-400	42.3	21.48	100.00	21.48
Total	196.9	100.00		

III. Microscopic Characterization of Fossil Resin Concentrate

Because of their fine particle size it is difficult to sort the resin grains from the flotation concentrate by hand according to their natural color difference. The microscopic characterization of fossil resin concentrate has been conducted to classify the fossil resin types in the flotation concentrate. Since fossil resin fluoresces under uv and blue light, the petrographic characteristics of fossil resin types and coal content in the resin concentrate can be examined by fluorescence microscopy. The polished briquettes (pellets) were used for such examination. About 1-2 g of the fossil resin sample (flotation concentrate) and a suitable amount of epoxy/resin mixture (in the ratio of about four parts coal to one part epoxy by volume) were thoroughly mixed and filled into a cylindrical rubber mold (1.5 cm in height and 2.4 cm in diameter). The briquette was then dried and hardened in the mold overnight. After removal from the mold, the briquette was ground and polished with a set of successive silicon carbide papers followed by polishing with a 1-micron and a 0.05-micron alumina suspension, according to ASTM Standard D-2013-79.

An Axioplan Universal Microscope manufactured by Carl Zeiss, West Germany, was

employed for the analysis of resin type and grain size for the polished samples. The microscope was equipped with a 150-watt xenon arc lamp (model 770), and a blue-light excitation set (450-490 SB) with an exciter filter (BP 450-490), a chromatic beam splitter (FT 510), and a barrier filter (LP 520).

Microscopic photographs of the samples were taken using a Carl Zeiss Microscopic attachment (MC 100) system with a 35-mm camera and using Kodak 100 film to produce colored photographs of the polished section. A 20X dry objective lens and a 10X photo eyepiece were used for all the photography, which gives 200X magnification of the image.

Petrographic examination of the fossil resin types was conducted by microscopic counting. Total 354 counts were conducted. Under blue light fossil resin grains can be classified into four groups: green, green-yellow, yellow-orange and orange-brown which are similar to the hand-sorted yellow, amber, light-brown, and dark-brown resin. The resin type distribution of the flotation concentrate is listed as follows:

Resin Type	Green	Green-Yellow	Yellow-Orange	Orange-Brown	Coal
% Counting	12.62	28.65	34.97	11.04	12.72

Major portion of fossil resin flotation concentrate are green-yellow or yellow-orange type resins (about 63%). Few green resin (12.6%) and orange-brown resin (11%) are found in the resin concentrate. A notable amount of fine coal particles (12.7%), mostly vitrinite and less than 20 micron, is observed in the concentrate. Some fine clay minerals are also occasionally found in the concentrate. All resin types fluoresce under blue light with a greenish yellow color, which gradually diminishes in intensity from green to orange-brown resin. While the microphotograph of green resin indicates that it is homogeneous in appearance, the other resin types show inclusions of what appear to be fine coal particles. These colloidal coal particles were found to be 1 to 20 microns in diameter and gradually increase in number from green-yellow to orange-brown resin. These observations lead to the conclusion that the differences among the four resin types, particularly their color in the natural state may be partly due to the differences in the quantity of finely suspended coal particles.

IV. Preliminary Solvation Examination

Both solvent loading limits and maximum extraction yield has been tested for the fossil resin concentrate in order to obtain some basic parameters of solvent refining property. The tests of solvent loading limits for four different solvents (ethyl acetate, hexane, heptane, and toluene) has been carried out at room temperature. For each solvent loading test about 90 grams of fossil resin concentrate was placed into a 500-ml long-neck flask with 300-ml technical grade solvent. The flask was properly sealed and was then agitated on a Junior Debit shaker at 250 RPM. The extraction lasted for 24 hours. After extraction the flask was stationed for another 72 hours to let the undissolved particles settle down. After filtration of resin-loaded top solutions 20 ml of the filtrated solution was taken into a beaker and dried in a vacuum oven for 48 hours. After all solvent evaporated, the resin loading limits for these four solvents were determined and given below.

Solvent Type	Ethyl Acetate	Hexane	Heptane	Toluene
Loading Limit (gm resin / 100 ml solvent)	8.72	22.56	23.03	30.17

It is seen that the solubility of the four selected solvent for fossil resin concentrate follows the order (from weak to strong): ethyl acetate > hexane > heptane > toluene. It can also been seen from the data that toluene is a strongest solvent for fossil resin extraction (30.17 gm resin / 100 ml toluene) in the four solvents, next is heptane (23.03 gm resin / 100 ml heptane) then hexane (22.56 gm resin / 100 gm hexane), and that ethyl acetate is a weak solvent for fossil resin extraction (8.72 gm resin / 100 ml solvent). Therefore, toluene will gives a highest extraction percentage for fossil resin concentrate, ethyl acetate will give a lowest extraction percentage and hexane and heptane in between. Such a finding might be expected due to the fact that ethyl acetate is a very short-chain solvent that is difficult for to interact with or penetrate into fossil resin matrix. Hexane and heptane are a non-polar solvent with medium chain length which give almost the same loading ability for fossil resin concentrate. Hexane and heptane can easily dissolve low molecular weight resin components from resin matrix, while toluene is weakly polar with a permanent dipole and π -electron system that provides for stronger interaction with or penetration into fossil resin. It should be noted that the color of the extracted solution changes

gradually from light amber for ethyl acetate solution to dark for toluene solution.

The maximum resin extraction yield of fossil resin concentrate for the four different solvents was conducted with Soxlet extraction unit (HT-6 model). In each solvent extraction test, about one and half gram of fossil resin concentrate was placed into a single-thickness cellulose extraction thimble and about 60 ml solvent was used for extraction. First, the resin concentrate was set for boiling for about 4 hours then the thimble was lift for rinsing for another 10 hours. After extraction the solvent was evaporated and both soluble resin and insoluble residue was dried and analyzed. The results of maximum resin extraction test are given as below:

Extraction Solvent	Ethyl Ace.	Hexane	Heptane	Toluene
Resin (wt. %)	65.35	70.27	78.34	85.14

The extraction ability of the four selected solvents for fossil resin concentrate follows the order (from weak to strong): ethyl acetate > hexane > heptane > toluene. It is seen that toluene has strongest extraction ability for fossil resin concentrate which can extract 85.14% resin from flotation concentrate. Heptane can extract 78.34% resin and hexane can extract 70.27% resin from the fossil resin flotation concentrate. It is found that ethyl acetate is a weaker solvent for fossil resin which can only extract about 65.35% resin from resin concentrate about 20% less than that extracted by toluene under the same conditions.

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