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REFINING OF FOSSIL RESIN FLOTATION CONCENTRATE FROM WESTERN COAL
Ninth Quarterly Final Report(Jan. 1 - March 31, 1995)

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Research work conducted in the ninth quarter, from January 1 to March 31, 1995, covered continuous operation of the proof-of-concept refining test circuit. The work was mainly performed by Advanced Processing Technologies(APT), Inc., a subcontractor of the University of Utah.

1. Materials

Technical grade of mixed hexanes (C_6H_{14}), normal heptane (C_7H_{16}) and toluene ($C_6H_5CH_3$) with the following physical properties were used as solvents for these experiments.

	<u>Molecule Weight</u>	<u>Density, g/cm³</u>	<u>Boiling Point, °C</u>
Hexane	86.18	0.659	68
Heptane	100.12	0.684	98
Toluene	92.15	0.867	111

Feed material for the testing was fossil resin concentrate obtained from the Wasatch Plateau coal (the same as was used in the kinetic studies and batch extraction tests presented in the Third Quarterly Report, 1994). The relevant properties of the concentrate are:

Density (gram/cm ³)	1.143
Ash Content (%)	1.23
Moisture (%)	1.03
Hexane-Soluble Resin (%)	73.34

The particle size distribution of the resin concentrate was determined by wet screening according to ASTM standard procedure (D 4749) and the particle size distribution was found to be as follows:

<u>Particle Size (mesh)</u>	<u>Weight %</u>	<u>Cumulative Weight %</u>
28 × 60	1.79	100
60 × 100	4.24	98.21
100 × 200	13.74	93.97
200 × 270	7.71	80.23
270 × 400	8.71	72.52
- 400	63.81	63.81
Total	100.00	

2. Experimental Apparatus and Procedure

A schematic diagram of the continuous refining test circuit is shown in Figure

1. All studies were carried out with this system. A thermocouple was used to monitor the temperature inside the solvent extractor. A heating coil using hot water

as a heating medium was placed inside of the extractor to supply heat in cases where a higher operating temperature was required. Temperature was controlled by the flow of hot water through the heating coil. Agitation of the resin concentrate/solvent suspension was accomplished with an air operated centrifugal pump.

Two to three liters of solvent were taken into the solvent extractor to start the test. When the temperature inside the extractor approached the desired set point, fossil resin concentrate was charged into the system using an Accurate dry material feeder operated at a certain pre-calibrated rate. After a period of time (determined based on feeding rate and solid/liquid ratio) fresh solvent at the same temperature as that inside the extractor was introduced into solvent extractor at a rate to sustain the desired solid/liquid ratio. After the desired residence time was established, the mixture was discharged to filtration through the outlet valve. A temperature accuracy of $\pm 2^{\circ}\text{C}$ was controlled by adjusting the hot water flow in the heating coil.

Figure 1. Schematic diagram of the continuous fossil resin refining circuit

3. Experimental Results and Discussion

During this period of work, technical grade heptane was mainly used as the refining solvent. When technical grade toluene was used as the extraction solvent great difficulty was encountered in the filtration step. The mixture of the extraction residue and pregnant solution was so sticky that it was impossible to separate the residue from the solution in a reasonable time period. Therefore, no experimental data for continuous operation with toluene were obtained in this period. All data presented in this section of the report were generated by using heptane as the solvent.

The effects of extraction temperature, average residence time, and resin concentrate/solvent ratio were investigated under similar conditions to those in which hexane was used as the solvent. Test results using heptane as the solvent are presented in Figures 2 to 4. It is obvious that the results are similar to the results obtained with hexane in all aspects and the conclusions are similar to those presented in the Eighth Quarterly Report.

Figure 2. Effect of heptane extraction temperature on resin yield at a feed rate of 5 gram/min. and a residence time of 10 min. (concentrate/solvent ratio = 0.07)

Figure 3. Effect of average residence time on resin yield with heptane at a feed rate of 5 gram/min. and 49 °C (concentrate/solvent ratio =0.07)

Figure 4. Effect of the resin concentrate to solvent ratio (by weight) on resin yield with heptane at a feed rate of 5 gram/min. 49 °C

4. Comparison of the Test Results Using Different Solvents

In order to evaluate the solvents used, a simple comparison was carried out from both a technical and economical viewpoint.

4.1 Technical Evaluation

Heptane showed a consistently higher extraction than hexane even though heptane contains only one more methylene group in its molecular structure. Figures 5 to 7 compare the extraction yields for these two solvents under the same extraction conditions. It is clear that heptane extracts a little more resin than hexane and is a more powerful solvent for fossil resin than hexane. With heptane as the solvent, both the extraction rate and the resin yield are enhanced. The reason for improved extraction with heptane appears to be due to kinetic considerations (laboratory Soxhlet extraction for 6 hours results in the same extraction for both the mixed hexanes and the normal heptane). Thus it seems that molecular structure has a significant effect on the rate and extent of extraction. Additional laboratory experiments are planned to examine this hypothesis further. In any event, based on the results from the continuous testing normal heptane would be the preferred solvent.

Figure 5. Comparison of resin yields with two different solvents under various temperatures at a feed rate of 5 gram/min. and a residence time of 10 min. (concentrate/solvent ratio = 0.07)

Figure 6. Comparison of resin yields with two different solvents under various residence times at a feed rate of 5 gram/min. and 49 °C (concentrate/solvent ratio = 0.07)

Figure 7. Comparison of resin yields with two different solvents under various concentrate to solvent ratios at 49 °C and a residence time of 10 min.

Toluene can not be used as a refining solvent for the following reasons:

As shown by the differential refining tests presented in the Sixth Quarterly Report, the melting point of the heptane-extracted resin is less than 155 °C while the melting point of the toluene-extracted residue (taken from the residue of heptane extraction) is higher than 230 °C. The difference is obviously due to the fact that the toluene-extracted material has a much higher molecular weight or a much larger molecular size. These large molecules remain insoluble during hexane or heptane extraction and bridge other insoluble residue particles (eg. coal) which are very fine and thus a porous structure is created for the insoluble residue particles. With heptane as the solvent, the integrity of the porous structure is preserved and with the relatively large porous particles filtration after extraction is facilitated. However, with toluene as the solvent, the porous structure is destroyed and the very fine insoluble residue particles (eg. coal with an estimated size of 0.5 to 20 μm) are released. These fine residue particles inhibit filtration. Finally, as expected, the color of the toluene extracted products is much darker than the color of the heptane or hexane extracted products and thus the toluene extracted products would be of significantly lower economic value.

4.2 Economic Evaluation

As mentioned previously, from a technical perspective, heptane is the better solvent to be used in the resin refining process. However economic factors must also be considered in the overall evaluation of the process. In this regard a simple economic evaluation was carried out taking into consideration the operating costs for the resin concentrate refining process.

First of all, the price of industrial grade heptane is about the same as hexane. Because the process operates in a recycle mode, the initial cost would be about the same for both solvents. But in order to obtain the final resin product, the extracted resin has to be recovered from solution using evaporation techniques, which consume energy. Due to the significant difference in boiling points between the two solvents, approximately 25 - 35% more energy will be required for resin recovery by evaporation if heptane is used as the solvent for extraction. This represents a very significant increase of the operating cost.

Secondly, based on bench scale tests the same yield can be achieved with hexane if the average residence time is increased. Such an increase in retention time only increases the capital cost by a small amount.

It appears then from an economic perspective that hexane is the most appropriate solvent.