

DEASHING OF COAL LIQUEFACTION PRODUCTS VIA PARTIAL
DEASPHALTING. II - HYDROGENATION AND HYDROEXTRACTION EFFLUENTS

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INTRODUCTION

A previous paper described the deashing of extraction effluents produced by hydrogen-donor extraction of bituminous and subbituminous coals.⁽¹⁾ The method described was the use of batch and continuous gravity settling with and without the use of deasphalting solvents to accelerate the settling process.

The present paper presents data on the extension of the same deashing technique to coal liquefaction products produced by direct hydrogenation of Illinois No. 6 bituminous coal. Deashing experiments were conducted both with products produced by liquid-phase hydrogenation in the presence of an ebullated bed of hydrofining type catalyst (H-Coal Process)^{2} and with products produced by hydroextraction, i.e., without added catalyst (PAMCO Process).^{3}

In contrast with the previous donor extraction case, the addition of a deasphalting solvent was required in both the H-Coal and hydroextraction cases to achieve an acceptable rate of deashing.

The hydroextraction products were produced in the CCDC bench-scale unit. Data are presented with respect to solvent balance, product yields and properties in hydroextraction as well as the deashing behavior of the hydroextraction effluents. Continuous unit data are presented in detail for the hydroextraction work while the batch work is briefly summarized.

Data are presented for deashing of H-Coal bottoms in both batch and continuous units.

EXPERIMENTAL

H-Coal Bottoms - Materials and Procedure

The basic material for the deashing studies was the vacuum bottoms from processing of Illinois No. 6 coal via the H-Coal Process. The material was produced by Hydrocarbon Research, Inc. for the Office of Coal Research in their 8" I.D. Process Development Unit. The detailed conditions for production of this material are given in Table 15 of Reference (2). Operating conditions were varied somewhat during this run. The average operating conditions were 2700 psig, 30 lbs coal fed/hr-ft³, 825°F and an average catalyst age of 230 hours.

The material "as received" consisted of the vacuum distillation residue from the H-Coal hydro bottoms. The material accordingly had most of the -975°F distillate oils removed. The analysis of the material is given in Table I.

The deashing in practice likely would be done prior to removal of the distillate oils. The material was accordingly reconstituted to simulate its original form by solution at 600°F in natural recycle solvent from Cresap, W. Va. A ratio of 1.25 parts solvent to 1 part of vacuum bottoms was used for both batch and continuous runs.

The origin and analyses of the Cresap recycle solvent were given previously.⁽¹⁾ n-Decane (Phillip's Technical Grade Minimum Purity 95%) was used exclusively as the deashing solvent in this work.

The batch settling experiments were carried out in the same manner as previously described for coal extraction products. The "reconstituted" slurry was heated with stirring to 720°F, n-decane was then added in prescribed amount and the mixture cooled to 600°F. The agitation was stopped and the slurry allowed to settle. Both settling rates and clarity of the upper phase after about three hours of settling were determined.

The same continuous unit⁽¹⁾ used for extraction and deashing of extraction effluent was used for deashing of the "reconstituted" H-coal bottoms. The extractor in this case served to preheat the slurry to 720°F. n-Decane was introduced into the middle stage of the extractor. The outlet slurry was cooled to 600°F before entering the settler which was also operated at 600°F. Only one continuous run was made due to limitations in supply of material.

The unit was started with solvent flow through all vessels until proper temperature conditions were established. The H-Coal slurry was then passed through the unit for a line-out period of nine hours to displace the start-up solvent. A material balance ensued for a period of four hours during which time samples of all appropriate streams were taken for analyses.

Hydroextraction - Materials and Procedure

The hydroextraction work was carried out with an Illinois No. 6 bituminous coal from the Burning Star Mine of Consolidation Coal Co. Analysis of the coal as fed to the hydroextraction unit is given in Table I.

A batch program was carried out prior to the continuous work in the same one-gallon autoclave previously used for donor extraction.⁽¹⁾ Anthracene oil was used as solvent and n-decane as deashing solvent. The experimental procedure was very similar to that previously described.⁽¹⁾ The autoclave was heated to 825-850°F with pure H₂ flowing slowly through the autoclave maintained at 1000 psig. The hydrogen flow was then stopped, n-decane added, and the reactor cooled to 600°F. The agitation was stopped for measurement of the settling rate.

Continuous hydroextraction runs were carried at two operating pressures of 950 psig and 2000 psig, respectively.

Operation at 950 psig

The 950 psig runs were conducted with essentially the same equipment as used previously in the donor extraction work.⁽¹⁾ Revisions were made, however, to accommodate the use of hydrogen as shown in the schematic flow diagram of Figure 1. Facilities were added for feeding a metered stream of pure hydrogen. The hydrogen was preheated and added to the feed slurry just before the latter entered the horizontal stirred preheater.

Another substantial change was the introduction of a primary receiver or "hot separator" between the extractor and the additive contactor. This served to disengage the gas and volatilized solvent from the product slurry. The primary receiver was normally operated at 650°F.

The operation of the hot separator resulted in a removal of a considerable fraction of the product slurry as vapor. The experimental results shown in Table II give the actual liquid feed rate to the settler as a weight fraction of slurry feed to the hydroextraction unit.

The liquid level in the separator was maintained by operation of a tungsten carbide-trimmed, let-down valve actuated by a liquid level controller. The final product was similarly handled in the overflow receiver where the product was let down to the pot of a vacuum still.

A series of runs was first conducted while bypassing the settler to survey the effect of the variables.

The start-up solvent was a coal-tar derived anthracene oil obtained from Allied Chemical Co. The oil was stabilized before use by running it through the unit with hydrogen at 950 psig, but without coal added, for six cycles. Polymer formed was removed by fractionation in the vacuum still. The still was operated in a semi-continuous fashion. Before the contents of the pot were removed, feed to the still was interrupted, the vacuum was reduced to 1 mm Hg while the temperature of the stirred liquid in the pot was maintained at 240°C until no more vacuum distillate was collected. The composition of the "stabilized" anthracene oil is given in Table II.

The second series of runs was conducted with the settler operating and n-decane added to increase settling rate.

A final series of runs was conducted in an attempt to generate natural solvent. To minimize physical solvent losses, the operation of the settler was bypassed. The total hydroextraction effluent was processed through the vacuum still to recover all distillates. The overhead distillate was then fractionated in a continuous atmospheric fractionating column packed with Goodloe packing, and having the equivalent of about 50 theoretical plates. The light cut boiling below 240°C at 1 atm was removed in this way before recycle of the solvent to the extraction unit.

Two types of operation were conducted in the above recycle solvent program. The initial method consisted of direct recycle of spent solvent to the hydroextraction unit. The second operation consisted of a "hybrid" type operation where either one-half or all of the recovered solvent was rehydrogenated over a fixed-bed, nickel molybdate catalyst before recycle to the hydroextraction unit. The final operation returned to direct recycle. Results of the final recycle operation only are reported here.

The hydroextraction was normally operated with a stirrer speed of 600 rpm. The system was inoperable due to coke formation when the stirrer speed was reduced to 300 rpm.

Operation at 2000 psig

The 2000 psig operations were conducted in a similar manner. A new five-stage, stirred extractor was built for the higher pressure with overall dimensions of (pre-heater and extractor) of 5.2" I.D. by 49.8" in height.

The initial operations at 2000 psig were conducted in two phases without operation of the settler. The first phase consisted of a short study of the system variables using the anthracene oil stabilized at 2000 psig. The second phase consisted of operation at constant operating conditions to generate natural solvent.

The natural solvent was then used in a final campaign in which both the continuous extraction and settler units were operated simultaneously. In this campaign the overflow and underflow streams were mixed and sent to the vacuum still for recovery of solvent. n-Decane was again used exclusively as the deashing solvent in this work.

The vacuum still during the 2000 psig hydroextraction campaign was operated to a final pot temperature of 290°C at 1 mm Hg, as contrasted to the final pot temperature of 240°C at 1 mm Hg used in the 950 psig campaign. The corresponding atmospheric boiling points are 474°C and 535°C, respectively. It should be recognized, of course, that some lower-boiling materials will be retained in the pot liquids by virtue of dilution with higher-boiling residue.

Analytical and Calculational Procedure

The analytical procedures were for the most part identical to those reported earlier.⁽¹⁾ Some slight differences are noted, however. Boiling tetrahydrofuran was used in the H-Coal work instead of cresol (in all other work) to characterize the quantity of insoluble residue. Results of the two solvents are, however, very similar. Slightly-higher insolubles are obtained with tetrahydrofuran.

The characterization of the solvents by boiling point in this work was determined by retention time in gas chromatography. A Perkin-Elmer 900 gas chromatograph was used. The column used was of an open tubular type, 100' x 0.0465" I.D., support coated with an Apiezon L substrate containing a finely divided siliceous support in the coating solution. The column retention times were calibrated with known compounds of the type present in the coal derived solvent such as tetralin, naphthalene, methyl-naphthalenes, phenanthrene, pyrene and chrysene.

Coal conversion in the hydroextraction runs was defined on the same basis as in the previous donor extraction case.⁽¹⁾

The hydrogen consumption was measured in two ways, by direct measurement of hydrogen gas in and out excluding flash gases and by forced elemental balance. The latter generally was higher than the former as might be expected due to small leaks, etc. Accordingly, only the latter values are reported.

The yield structure and material balance were arrived at by the following procedure:

- a. Force H₂S, NH₃ and H₂O yields by forced N, O and S balances.
- b. Force total material balance, i.e., solvent plus coal plus H₂ consumed = products ex H₂ gas.
- c. Iterate steps a) and b) until a simultaneous solution is obtained.

The material balance invariably was quite good such that very little forcing under step b) was required.

EXPERIMENTAL RESULTS

H-Coal Bottoms

A summary of the batch deashing runs is given in Table III. The initial settling rate for the 500 and 600°F runs are plotted in Figure 2 versus decane/slurry weight ratio. It is noted that the settling rates are nearly identical at both temperatures. When the decane/slurry ratio was increased to 0.35, the settling rate was too fast for accurate measurement. Only a minimum rate can be specified as shown in Table III.

The "equilibrium" clarity of the upper phase is plotted against the decane/slurry ratio in Figure 3. It is noted that a much lower ash "ultimate" product can be achieved at 500°F. Wall deposits were noted, however, in some of the 500°F runs which raised some doubt about operability of a continuous settler operated at this temperature.

For this reason, the continuous demonstration run was carried out at 600°F. Conditions and results of this run are given in Table IV.

The results of the continuous and batch runs are in good agreement. The settler upflow velocity of 0.37 in/min (Table IV) is equivalent to an initial settling velocity in the batch unit at a slightly lower decane/slurry ratio of 0.20 (Table III). The ash content of 0.13 wt % is equivalent to the equilibrium clarity of the batch run of 0.18 wt %.

The amount of precipitated residuum was calculated by the method outlined in a previous paper.⁽¹⁾ The value obtained, i.e., 7.9 wt % of the +474°C feed, is also in the same range indicated by the batch measurements.

Recovery of "soluble" residuum in the overflow was 76.5%. This value could be improved, of course, by back washing of the underflow.

Hydroextraction

The initial batch experiments on hydroextraction were made with anthracene oil solvent at 1000 psig, 825°F extraction temperature and 600°F settling temperatures. The settling rate was substantially zero without addition of deasphalting solvent. A relatively high settling rate of 1.9 in/min was obtained, however, by addition of decane in a weight ratio of 0.3 to extraction solvent.

The initial operations in the continuous unit utilized stabilized anthracene oil as the solvent at 950 psig total pressure. A survey of the temperature variable was made at constant slurry feed rate of 30 lb/hr and constant total pressure of 950 psig. The hydrogen partial pressure at the extractor exit was in the range of 800-830 psia. The optimum temperature at this pressure was 800°F, giving a conversion of 82%, as results in Table V indicate. A temperature of 825°F was chosen for more intensive study, however, since the residuum extract showed a more favorable lower sulfur content, i.e., 0.86% S versus 1.00% S at 800°F, as noted in Table V.

It was therefore decided to generate natural solvent from coal by continuous recovery and recycle of solvent at 825°F and to simultaneously study the deashing and extraction during the production of the natural solvent.

The deashing via continuous settling with decane addition was studied early in the solvent recycle program. A typical result is shown in Table VI where good deashing was obtained at a decane-to-extraction solvent ratio of 0.29 and a settler upflow velocity of 1 in/min. This result is roughly in agreement with batch results cited above where under comparable conditions the initial settling velocity was 1.7 in/min.

The solvent recycle program was finally abandoned after it was found that the low pressure operation could not be self-sustained without periodic addition of fresh anthracene oil.

The failure to close solvent balance is clear from the product yields cited in Table V. The data at 825°F and 950 psig represent average performance after about 25 cycles. It is noted that there is a net deficiency of 5.2% of recycle solvent in the boiling range used, i.e., 240 x 474°C. The use of a higher endpoint, as noted, in Table V would have substantially reduced the solvent deficit but would not have eliminated it. At any rate, handling losses were sufficiently high such that it would have been impossible to generate pure natural solvent even with the higher distillation endpoint. Accordingly, no further deashing studies were carried out at the low pressure level.

It is apparent, however, from the data given in Table II, that significant even though partial displacement of the initial solvent was obtained in the 950 psig operation. This is illustrated by the lower boiling range and higher hydrogen content after 25 cycles as compared with the stabilized anthracene oil.

The hydroextraction operation was then carried out at a higher total pressure, i.e., 2000 psig, with a hydrogen partial pressure approximately 1850 psia. A solvent surplus was readily generated at 2000 psig, partly because of the higher pressure and partly because of the higher distillation endpoint used in recovery of recycle solvent.

Steady operation was sustained for a total of 500 hours until the initial solvent was largely displaced. The product and yield distribution after generation of natural solvent is given in Table V. The principal differences as compared with the 950 psig operation, i.e., higher coal conversion, positive net solvent generation, higher overall distillate yield and lower extract sulfur content, are clear from the data presented in Table V. The apparent reduction in extract sulfur is not very great, i.e.,

0.86 to 0.7. The actual reduction likely is considerably greater. The sulfur content of the 950 psig residuum is artificially low since it contains a significant quantity of 474 x 535°C solvent polymer and heavy oil not present in the 2000 psig product.

The continuous deashing of the 2000 psig product with n-decane additive was then studied with the results shown in Table VI. It is again shown that very little ash removal is obtained without addition of decane. Almost complete deashing is achieved by use of a ratio of decane-to-extraction solvent of 0.50. At this condition, it was determined by the method outlined previously,⁽¹⁾ that the deashing was accompanied by precipitation of 16 wt. % of the cresol-soluble residuum fed to the settler.

DISCUSSION OF RESULTS

The particle size distribution of the cresol insolubles in the H-Coal bottoms hydroextraction at 2000 and 950 psig are compared in Figure 4. It is noted, i.e., the size of the residue decreases with increasing severity of hydrogenation in going from 950 to 2000 psig hydroextraction to 2700 psig catalytic hydrogenation (H-Coal). All of the products show a finer size residue than the donor extraction products discussed in the earlier paper.⁽¹⁾

The relative ease of deashing might be expected to follow the same sequence as residue size. The results in Table VI are in agreement with this in that a higher decane-to-extraction solvent ratio is required to deash the 2000 psig as compared with the 950 psig hydroextraction product. Unfortunately, no valid comparison can be made between the H-Coal and the hydroextraction products because of the fact that the latter was artificially "reconstituted" by use of Cresap recycle solvent.

The mechanism of deashing as discussed previously, undoubtedly involves agglomeration of fine residue particles by precipitation of heavy asphalt.

The data in Table VII clearly show that the benzene-insoluble content follows the same trend as residue size with increasing severity of hydrogenation treatment. Thus, from this point of view also, a larger quantity of deasphalting solvent would be required to achieve an equivalent amount of benzene-insoluble rejection. Unfortunately, insufficient data are available to verify this supposition.

The failure to close the solvent balance in hydroextraction at 950 psig is consistent with results published previously.⁽⁸⁾ The successful generation of natural solvent at 2000 psig is consistent with recent results on the PAMCO process published by Anderson.⁽⁹⁾

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TABLE I

Analysis of Feed Materials

	<u>Illinois No. 6 Coal</u> <u>Burning Star Mine</u>	<u>H-Coal Vacuum Bottoms</u>	
<u>Proximate Analysis</u>			
<u>Moisture and Distillate</u> <u>(-474°C) Free, Wt %</u>			
Volatile Matter	41.4	--	
Fixed Carbon	48.4	--	
Ash Oxidized	10.2	24.3	
<u>Ultimate Analysis</u>			
<u>Moisture and Distillate</u> <u>(-474°C) Free, Wt %</u>			
Hydrogen	4.71	5.01	
Carbon	70.79	66.63	
Nitrogen	1.27	1.20	
Oxygen (diff.)	10.14	0.92	
Total Sulfur	2.93	1.98	
Organic Sulfur	2.15	1.45	
Pyritic Sulfur	0.54	--	
Sulfate Sulfur	0.24	--	
<u>Distillation, Wt %</u>			
Distillates (-474°C)	0	6.3	
Residue (+474°C)	100	93.7	
<u>Solvent Fractionation, wt %</u>			
THF Insoluble	--	38.0	
Benzene Insoluble-THF Soluble	--	7.0	
Asphaltenes	--	29.0	
Oils	--	26.0	
<u>Screen Analysis, Tyler</u>		<u>Particle Size Analysis,</u>	
	<u>Mesh, Wt %</u>	<u>THF Insolubles, Wt %</u>	
+28	0.1	+26 microns	1.8
28 x 48	1.6	20.7 x 26	3.3
48 x 100	18.0	13 x 20.7	13.2
100 x 200	22.4	6.5 x 13.0	34.0
200 x 325	19.5	4.1 x 6.5	35.7
-325	38.4	-4.1	12.0

TABLE II

Analysis of Feed and Product
Recycle Solvents

	Solvent		
	Stabilized Anthracene Oil	After 25 Cycles at 950 psig	Natural Solvent After 2000 psig Operation
<u>Boiling Range by G.C., Wt. %</u>			
-218°C	0.57	1.12	5.03
218 x 240°C	2.29	5.24	15.33
241 x 260°C	6.91	15.32	18.92
261 x 294°C	28.13	27.98	21.81
295 x 340°C	36.92	33.42	24.09
+340°C	25.18	16.92	14.82
<u>Ultimate Analysis, Wt. %</u>			
Hydrogen	5.92	7.48	7.84
Carbon	91.42	90.49	88.43
Nitrogen	0.94	0.36	0.67
Oxygen	1.28	1.52	2.71
Sulfur	0.44	0.15	0.35

TABLE III

Summary of Batch Settling
Runs with H-Coal Bottoms

Run Number	1	6	3	5	7	4
Temperature, °F	500	→	←	600	→	→
Additive	None	n-Decane	→	→	→	→
Additive/Reconstituted Slurry, Wt Ratio	0.0	0.10	0.35	0.10	0.20	0.36
<u>Cresap Recycle Solvent, Wt Ratio</u>						
H-Coal Vacuum Bottoms	2.04	1.25	→	→	→	→
Initial Settling Rate, in/min	0.04	0.12	>> 1.1	0.12	0.38	> 1.71
Wt % Ash in Ultimate Clarified Layer	0.48	0.12	0.02	0.23	0.18	0.11
Precipitated Residuum Solubles, Wt % of Residuum Fed	(1)	(1)	(1)	(1)	(1)	7.9 ⁽²⁾

(1) Consistent values by solvent balance could not be obtained.

(2) Determined by filtration.

TABLE IV

Conditions and Results of Continuous
Deashing Run with H-Coal Bottoms

Feed Slurry	<u>1.25 lb Cresap Recycle Solvent</u> 1b H-Coal Vacuum Bottoms
<u>A. Temperatures, °F</u>	
Pumping	600
Preheat	720
Settler	600
<u>B. Feed and Product Rates</u>	
Raw Slurry, lb/hr	31.3
Decane Feed, Wt % of Slurry	25
Calculated Settler Upflow Velocity, in/min	0.37
<u>Product Split, Wt %</u>	
Overflow	66.1
Underflow	28.7
Vents - Condensed Vapor	5.7
<u>C. Product Quality</u>	
<u>Ash in Overflow, Wt %</u>	
in Total	0.034
in +474 C Residuum	0.13
<u>THF Insolubles in Underflow, Wt %</u>	
Total Underflow	46.8
in +474°C Residuum	71.9
<u>D. Calculated Wt % Residuum in Feed Precipitated</u>	
	7.9
<u>E. Wt % Recovery, THF Soluble Residuum in Overflow</u>	
	76.5

TABLE V

Yields and Solvent Balance in
Hydroextraction

A. Conditions

Total Pressure, psig	950	950	2000
Extraction Temperature, °F	800	825	822
Solvent/MF Coal, Wt Ratio	2.5	2.5	2.0
Slurry Feed Rate, lb/hr	30	30	28.1
Coal Throughput, lb MF/hr-ft ³	38	38	41
H ₂ Rate, SCF/lb MAF Coal	27.8	27.8	26.3
H ₂ Partial Pressure at Exit, psia	~ 830	~ 800	~ 1850
Solvent Age, Cycles	15	avg. 17	Natural

B. Results

Coal Conversion, Wt % of MAF Coal Fed	82.4	77.8	94.3
<u>Yields, Wt % of MAF Coal</u>			
MAF Cresol Insolubles	17.6	22.2	5.7
Extract (+474°C Residuum Solubles)	66.6	62.4	--
Extract (+535°C " ")	--	57.8	56.2
Water	6.6	6.4	9.1
H ₂ S + NH ₃	2.5	2.2	2.3
CO ₂ + CO	1.3	1.6	1.6
C ₁ -C ₅ Hydrocarbons	4.7	5.0	7.4
C ₆ x 240°C Light Distillate	4.1	6.8	6.6
240 x 474°C Recycle Solvent	-1.9	-5.2	--
240 x 535°C " "	--	-0.6	14.0
Total	101.5	101.4	102.9
H ₂ Consumed, Wt % MAF Coal	1.5	1.4	2.9
<u>Extract Sulfur, Wt %</u>			
+474°C Residuum	1.00	0.86	--
+535°C " "	--	--	0.7

TABLE VI

Results of Deashing Hydroextraction Effluent

<u>Extraction</u>					
Solvent/MF Coal, Wt Ratio	2.5	2.0	2.0	2.0	2.0
Solvent Age, Cycles	~ 3	Natural →			
Temperature, °F	825	822 →			
Pressure, psig	950	2000 →			
<u>Conditioning</u>					
Temperature, °F	599	600 →			
n-Decane/Extraction Solvent, Wt Ratio	0.29	0.00	0.31	0.49	0.50
<u>Settling</u>					
Temperature, °F	611	600 →			
Free Cross Section, ft ²	.079	→			
Settler Feed as Wt Fraction of Slurry Fed to Extractor	--	0.75	0.77	0.63	0.70
Overflow, lb/hr-ft ²	282	285	368	343	414
Overflow Velocity, in/min	1.0	0.9	1.6	1.7	1.8
Underflow, Wt % of Settler Feed	25.3	22.8	17.0	18.9	7.5
<u>Results</u>					
<u>Quality of Residuum in Overflow, Wt %</u>					
Cresol Insolubles	8.6	17.1	14.7	0.25	0.14
Ash	0.40	11.3	9.9	0.17	0.10
Sulfur	0.80	1.3	1.2	0.65	0.76
Extract Recovery in Overflow, Wt % of Extract Fed	70.1	81.1	75.0	69.0	80.4
Wt. % of Ash Fed in Settler Overflow	1.6	54.7	43.8	0.6	0.4

TABLE VII
 Solvent Fractionation Analyses
 - Cresol-Soluble Residue

Process	<u>Hydroextraction</u>		<u>H-Coal Bottoms</u>
Operating Pressure, psig	950	2000	2700
Operating Temperature, °F	825	825	825
Benzene Insolubles, Wt %	31.8	17.8	11.3
Asphaltenes, Wt %	40.2	60.2	46.8
Oil (+474°C), Wt %	28.0	--	41.9
Oil (+535°C), Wt %	--	22.0	--

Figure 1
Hydroextraction Unit With Settler

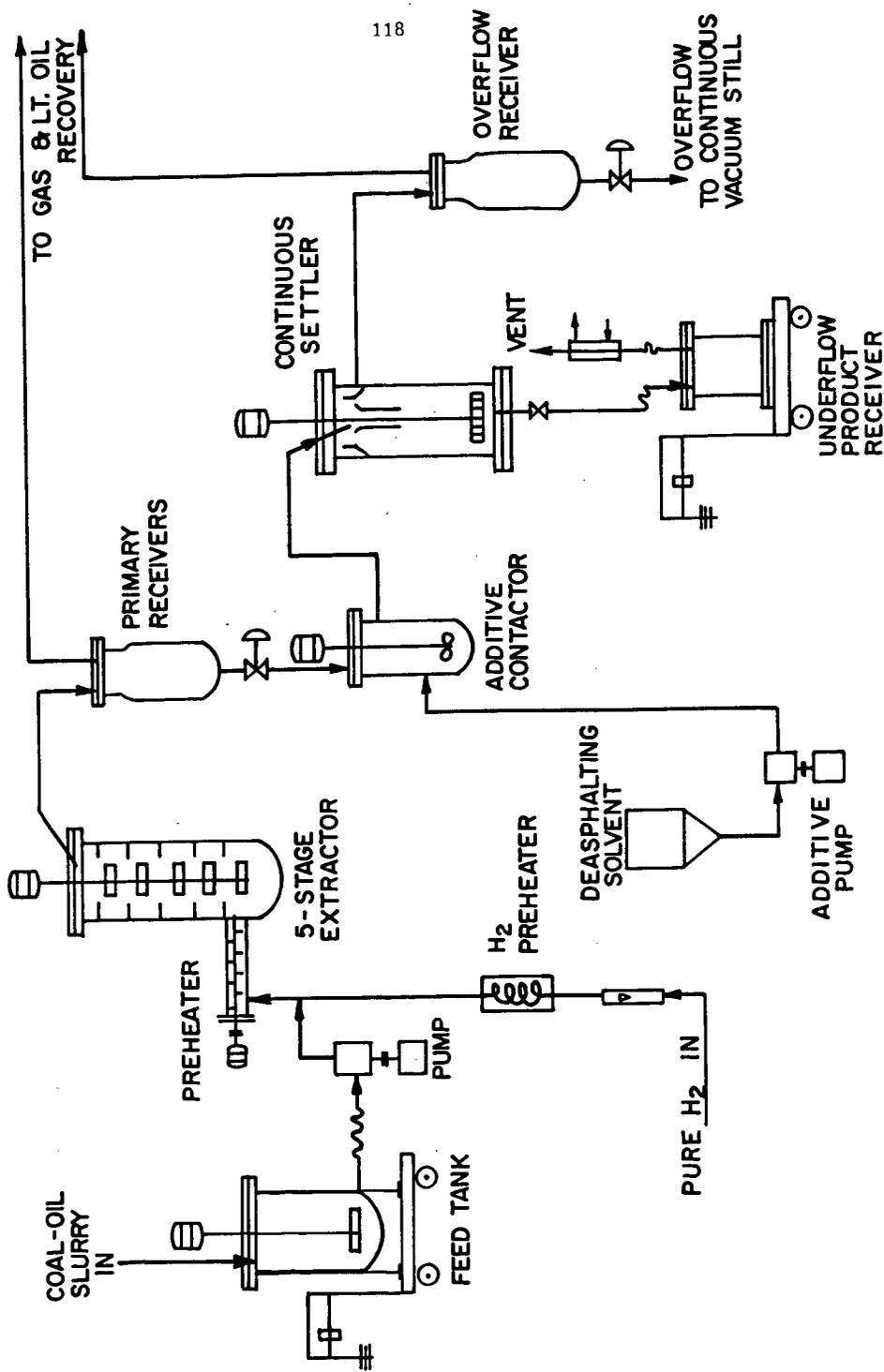


Figure 2
Settling Rate of H-Coal Bottoms Slurry
(Effect of Decane Addition)

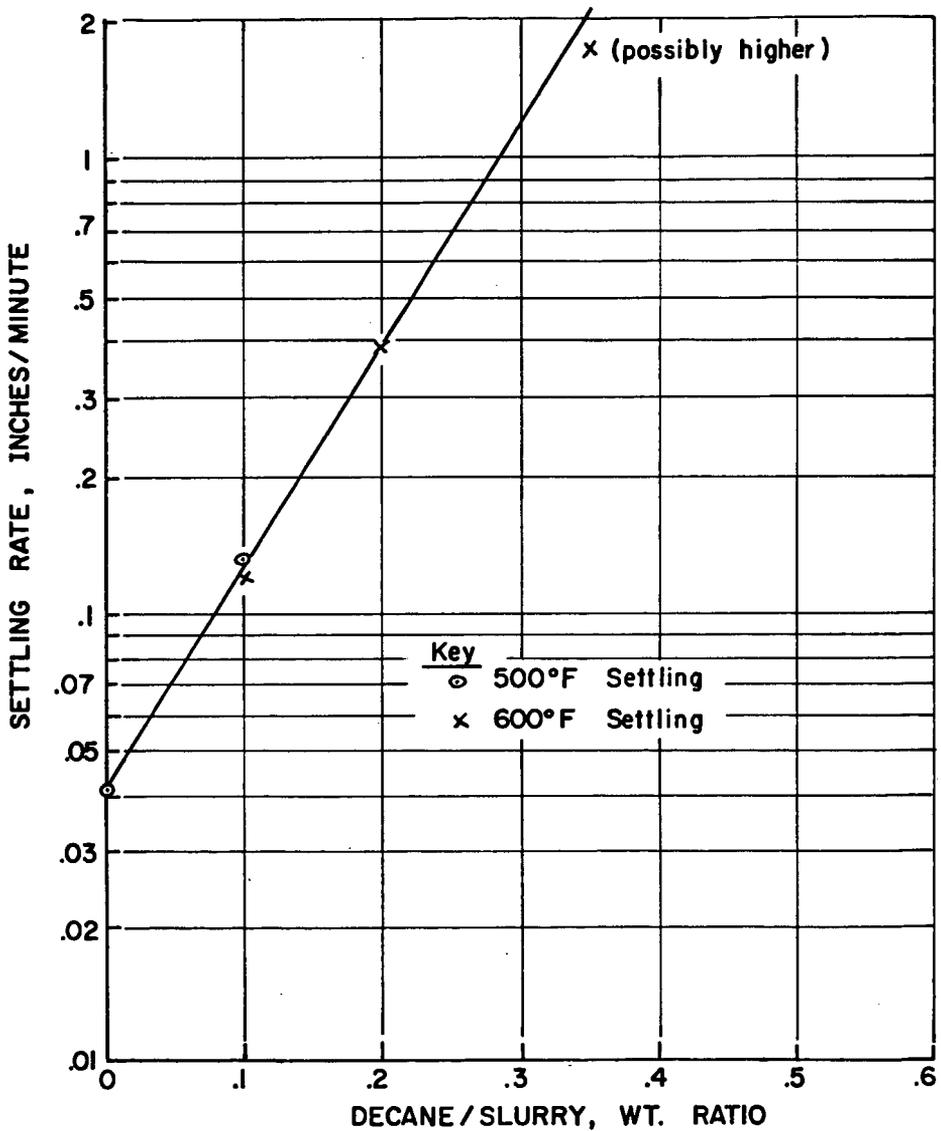


Figure 3
"Equilibrium" Clarity of Products
as a Function of Decane Addition
and Temperature

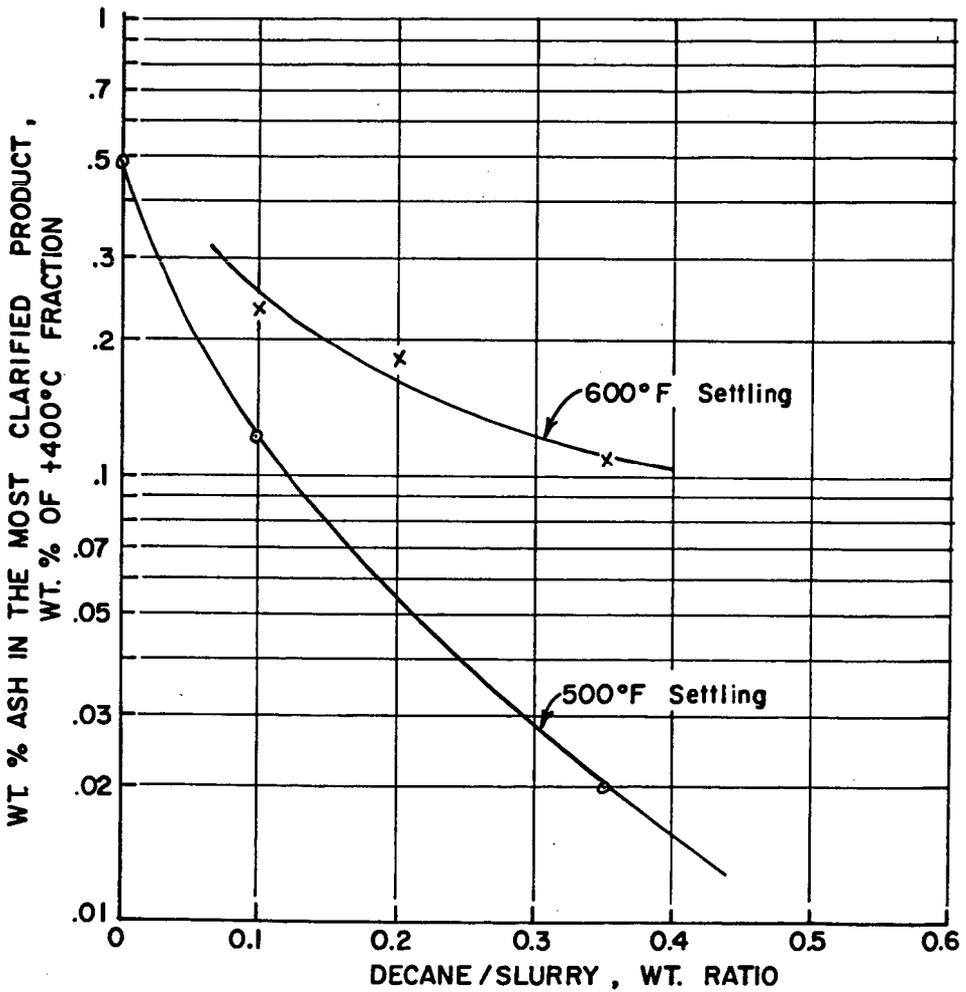


Figure 4
Particle Size of Cresol Insolubles

