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Ferric-sulfide-based catalysts made using reverse micelles: Effect of preparation on performance in coal liquefaction

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Abstract

Reverse micelles have been used to create small particles of ferric sulfide catalyst for direct coal liquefaction (DCL). Tests were performed to determine the effect on DCL of the procedure and conditions for the production of the reverse-micellar catalyst. The parameters varied include the water-to-surfactant ratio, the coal and catalyst mixing time, and the nature of the surfactant. The performance of the catalyst was quantified by the conversion and oil yield of the DCL experiments. After the optimum procedure for reverse-micellar catalyst production was determined, the optimal conditions were found to be a water-to-surfactant ratio of 10, a coal and catalyst mixing time of 60 min, and AOT as a surfactant. In further studies, hexane was found to affect coal liquefaction negatively. Hexane was found to have a significant effect on the surface area and pore volume distribution of coal. This may be due to small pores being plugged or due to the agglomeration of coal particles.
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1. Introduction

In direct coal liquefaction (DCL), catalysts are used to increase the conversion of the coal feed and the selectivity to oil-range products. The separation of the catalyst from the unreacted coal could have a significant cost. Ferric-sulfide-based catalysts are active for DCL [1], but yet sufficiently cheap and environmentally benign enough that they do not need to be removed from the residue after reaction, hence these catalysts are advantageous for use in DCL. Mixing ferric chloride with sodium sulfide creates the precursor ferric sulfide, even at very low temperatures, with sodium chloride as side product. The ferric sulfide then disproportionates into pyrite (PY, FeS_2) and pyrrhotite (PH, FeS_x), where x is approximately equal to unity [1]. Methods for introducing this catalyst as small particles intermingled with coal particles include impregnation in situ [2] and the use of aerosols [3]. Small catalyst particles can also be created using reverse micelles [4,10].

A reverse-micellar system is a microemulsion of water in a continuum oil phase. The discrete phase, water droplets, are separated from the continuum phase by molecules of surfactant, ordered such that the hydrophobic end of the surfactant molecule, the “tail”, aligns toward the continuum oil phase while the hydrophilic end, the “head”, aligns toward the discrete water droplets. The aggregates, or reverse micelles, containing surfactant molecules on the outside surrounding the discrete aqueous phase, can be 50 nm or smaller in size [10]. A mechanism [5] for the forma-

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tion of small particles by reaction in reverse micelles proposes that two reverse micelles containing stoichiometric concentrations of reactant solutions collide; the solutions then react to form the product, with further increase of the size of the product particle being prevented by the presence of the surfactant. Catalysts using iron and other materials have been prepared using micelles and studied for DCL by Moll and Quarderer [4] and by Linehan et al. [10]. Preliminary work into the production of a ferric-sulfide-based catalyst using reverse micelles, and the performance of the catalyst in DCL, was carried out by Chadha et al. [6].

2. Experimental

2.1. Materials

The catalyst precursor ferric sulfide was created using ferric chloride and sodium sulfide in their hydrated forms, $FeCl_3{\cdot}6H_2O$ and $Na_2S{\cdot}9H_2O$, obtained from Fisher Scientific with a purity of 98%. Hexane, used as the oil phase, was also purchased from Fisher Scientific as a mixture of hexane isomers with about 50% *n*-hexane and an overall purity of greater than 90% hexane. Three surfactants were used in the reverse-micellar catalyst production: sodium bis(2-ethylhexyl) sulfosuccinate sodium salt (AOT), polyoxyethylene-4-laurylether (“Brij 30”), and sodium dodecyl sulfate (SDS). The surfactants used were chosen for their structural differences. While AOT and SDS are both ionic surfactants, AOT has two hydrophobic tails while SDS has only one. Brij 30 is a nonionic surfactant with only one hydrophobic tail. All three surfactants were purchased from Aldrich Chemical Company. The AOT had a purity of 98%. SDS was a combination of three isomers.

The coal used for catalyst testing by DCL was DECS-6, a high-volatile A bituminous coal from the Blind Canyon seam in Utah. The coal was used at a particle size of 60 mesh. The proximate and ultimate analyses of the coal are shown in Table 1. Tetrahydrofuran (THF), used as a solvent in the DCL experiments, was obtained from Fisher Scientific with a purity of 99%.

Table 1
Proximate and ultimate analyses of DECS-6 coal (performed by Galbraith Laboratories)

Component	wt.%
Water	1.8
Ash[a]	6.3
Volatile matter[b]	49.0
Fixed carbon[b]	51.0
C[b]	81.9
H[b]	6.3
N[b]	1.5
S[b]	0.9
Cl[b]	0.12

[a] Dry basis.
[b] DAF basis.

2.2. Base-case procedure for production of reverse-micellar catalyst

The base-case procedure is modified from the method given by Chadha et al. [6]. The original procedure used *iso*-octane as the continuous phase, but hexane is used in the current work, due to the large amounts of the continuous phase required, the cost, and the high degree of flammability of *iso*-octane relative to the mixture of isomers of hexane. The catalyst is produced by reacting the ferric chloride and sodium sulfide precursors to create ferric sulfide which then disproportionates into PY and PH. The ferric chloride and the sodium sulfide are separately present in the discrete (aqueous) phase of a reverse micelle.

In the current base-case procedure, reactant solutions of 0.15 M Na_2S and 0.1 M $FeCl_3$ in water are made up first. Then a solution of 0.1 M surfactant in 1.5 l of hexane is made. These solutions are placed in a glove box that is purged with nitrogen gas. The surfactant/hexane solution is then divided into two equal portions. A calculated volume of one reactant solution is added to one portion of the surfactant/hexane solution, while an equal amount of the other reactant solution is added to the other portion of the surfactant/hexane solution, to form two reverse-micellar solutions. (The amount of reactant solution added is controlled by the water-to-surfactant ratio, W/S. For $W/S = 10$, a volume of 13.5 ml of each reactant solution is required.) These two reverse-micellar reactant solutions are then mixed together to create a reverse-micellar solution containing the ferric sulfide catalyst precursor.

The ferric sulfide disproportionates in the reverse micelle to form PY and PH. The mixture is agitated for 5 min.

After this agitation, a calculated weight of coal is added to correspond to a catalyst loading of 1.67 wt.% of dry, ash-free (DAF) coal. (For the amounts of reactant solutions added above, 8.3 g coal is used — this calculation is based on the assumption that all of the reactant solutions react to ferric sulfide.) The suspension of coal and reverse-micelle catalyst in hexane is agitated for 60 min. The slurry is then filtered and dried overnight at room temperature under a vacuum. The residue from the filtration is then used as the coal/catalyst feed for the DCL experiments.

2.3. Experimental procedures used for catalyst production

Experiments were performed to examine the effect of changes in the base-case procedure on catalyst performance in DCL. The first procedure used was the base-case procedure as described above. The second procedure was the use of a catalyst loading of 2.5%, which is greater than the nominal value of 1.67% used above. The third procedure was to use the base-case procedure, but to agitate the reactant solutions for 30 min before adding the coal, instead of 5 min. The fourth procedure was to use the base-case procedure, but to add acetone after the coal was added. Acetone can be used to break reverse micelles by increasing their size and making them thermodynamically unstable [7]. The fifth procedure was to wash the coal and catalyst feed produced from the fourth procedure with water, to remove NaCl formed in the reaction between sodium sulfide and ferric chloride. The sixth procedure was to use the base-case procedure, but to add equal portions of the coal to each of the reactant reverse-micellar solutions before they are mixed together and agitated. The seventh procedure was to use the base-case procedure, but to add all of the coal to the reverse-micellar solution containing ferric chloride reactant before mixing it with the other reactant reverse-micellar solution. The eighth procedure was to use the base-case procedure, but to add all of the coal to the sodium sulfide reverse-micellar solution before it was mixed with the other reactant reverse-micellar solution.

2.4. Experimental conditions used for catalyst production

The experimental conditions of the catalyst production procedure were systematically altered. Using the optimal experimental procedure from the eight used above, the water-to-surfactant ratio was varied (5, 10, 15); the surfactant was changed (AOT, Brij 30, and SDS); and the agitation time for the coal and catalyst slurry was changed (15, 60 and 120 min). All coal and catalyst feeds produced were tested using the same conditions for DCL, as described below.

2.5. DCL experiment

The DCL runs were conducted in 17 ml tubular bomb reactors. Each experiment was performed in duplicate. Each reactor was filled with 3.1 g of coal and catalyst feed. Reaction conditions included a temperature of 400°C, a time of 30 min, and a hot hydrogen pressure of 1000 psi along with a total hot pressure of 2000 psi. These reaction conditions coincide with conditions used in previous coal liquefaction experiments performed in our laboratory and reported in the literature.

After reaction, the gas products are analyzed using a gas chromatograph (GC) as described by Liu et al. [2]. The solid and liquid products are removed from the reactor using THF as a solvent. The THF solution is refluxed overnight. The THF-insoluble (TI) material is then removed through filtration. The THF is then removed from the THF-soluble material. The remaining material is refluxed in hexane for 2 h. The hexane-insoluble (HI) material is then removed through filtration. Consistent with our earlier work, the total conversion, X, is defined as the percentage by weight of material after reaction that is soluble in THF or in the gas phase, and X is calculated using

$$X\,[\text{wt.\%}] = \frac{\text{mass of dry coal} - \text{TI}}{\text{mass of DAF coal}} \times 100 \tag{1}$$

The asphaltene/pre-asphaltene yield, Y_A, is defined as the percentage by weight of material after reaction that is soluble in THF but insoluble in hexane, and Y_A is calculated using

$$Y_A\,[\text{wt.\%}] = \frac{\text{HI}}{\text{mass of DAF coal}} \times 100 \tag{2}$$

Oil is defined as the product that is soluble in hexane. For the greatest accuracy, the oil yield, Y_O, is obtained by the difference

$$Y_O\,[\text{wt.\%}] = X - Y_A - Y_G \tag{3}$$

where Y_G is the gas yield obtained from the GC analysis.

The data shown below are averages of (at least) two experimental runs. The experimental error is ±2 percentage points in X and in Y_A. In general, Y_G was found to have values from 3 to 5%, and there was little or no change in the gas yield with any experiments. Hence, in the following data, values for gas yield are not explicitly reported, although measured and used to calculate Y_O. The oil yield has an error of approximately ±4%, since errors in X and Y_A are much greater than errors in Y_G.

3. Results and discussion

3.1. Results of experiments without ferric-sulfide-based catalyst

A set of experiments was performed to determine whether the ferric-sulfide-based material or the other components present in the reverse-micelle system were causing the changes in conversion and yields relative to the liquefaction of coal alone. In one set of runs, coal was mixed in 1.5 l of hexane for 1 h, then filtered and dried, and the reaction was carried out on this solid. In another set of runs, coal was mixed in 1.5 l of a 0.1 M solution of AOT in hexane. The coal was then filtered and dried before the DCL reaction was carried out. The results from these experiments are compared in Fig. 1 with the results of DCL using coal alone, washed and dried, and DCL using the full coal/catalyst system as described above. As shown in Fig. 1, the conversion and oil yield for DCL experiments using coal decrease substantially when the coal is pretreated with AOT and hexane without catalyst. This indicates that any increase in conversion or oil yield for DCL with the full coal/catalyst system relative to DCL with coal alone is due to the presence of the ferric-sulfide-based catalyst, and not due to the other components present in the reverse-micelle system.

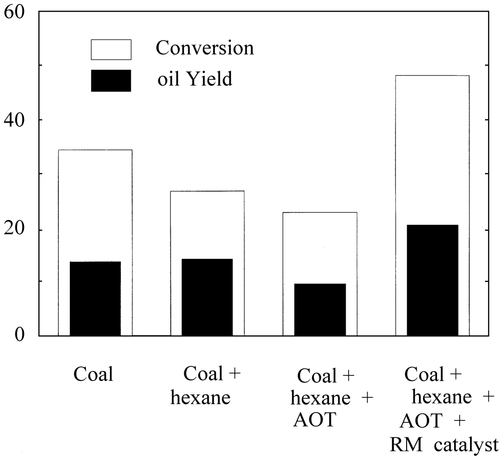

Fig. 1. Effect of components on DCL. DCL was carried out at 400°C and 1000 psig hydrogen pressure for 30 min.

3.2. Effect of experimental procedure used for catalyst production

Experiments were carried out to determine the effect of varying the base-case procedure for making the reverse-micellar catalyst. The different procedures used are described above. The results of DCL experiments on coal/catalyst systems produced with these different procedures are shown in Fig. 2. When the expected error bars in conversion and yield are taken into account, none of the procedures attempted is significantly better than the base-case procedure. Since the base-case procedure is the simplest and uses the least amount of raw materials, this procedure was chosen as the optimal procedure and was used for the subsequent runs.

3.3. Effect of experimental conditions used for catalyst production

Using the base-case procedure to add the catalyst to the coal, experiments were conducted while varying the conditions under which the reverse micelles are formed. The various experimental conditions used were described above. For this set of experiments, surfactants AOT and Brij 30 were used. For each of these surfactants, experiments were conducted using water-to-surfactant ratios (W/S) of 5, 10, and 15 and coal/catalyst mixing times of 15, 60 and 120 min. The

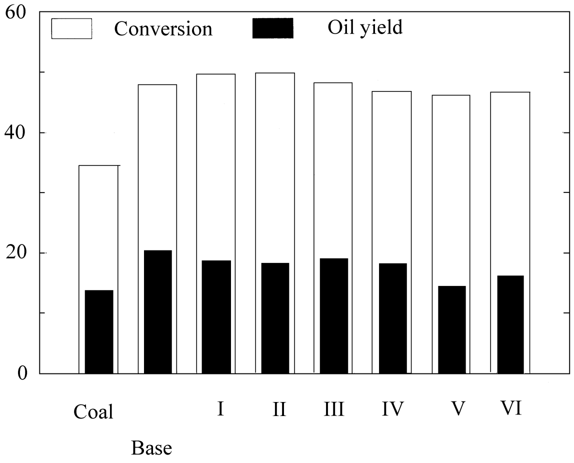

Fig. 2. Effect on DCL of changing the production procedure for the reverse-micelle (RM) ferric-sulfide-based catalyst. “Coal” represents the results of the uncatalyzed DCL. The “base case” is as described in the text. Cases “I”–“VI” represent changes from the base case: “I”, RMs mixed for 30 min; “II”, acetone used (to breakup RMs before DCL); “III”, procedure of “II” plus water added (to remove NaCl reaction byproduct); “IV”, coal added to both RMs individually, before mixing; “V”, all coal added to RM solution containing $FeCl_3$; “VI”, all coal added to RM solution containing Na_2S. Conditions for DCL are as in Fig. 1.

changes in W/S were accomplished by varying the amount of precursor solutions used, but this results in a change in the amount of catalyst produced. The amount of coal used is therefore adjusted accordingly, to keep the catalyst loading at 1.67%. For example, with $W/S = 5$, only 7 ml of each precursor solution is used, compared to 13.5 ml when $W/S = 10$. Accordingly, only 4.1 g of coal is added to the reverse-micellar catalyst solution (when $W/S = 5$), compared to 8.3 g of coal added to the reverse-micellar catalyst solution when $W/S = 10$.

Figs. 3 and 4 show the conversion and oil yield data for these runs using AOT and Brij 30 surfactants, respectively. The results for AOT are greater than the results using Brij 30. For both AOT and Brij 30, maximum conversions and oil yields are obtained at $W/S = 10$ and coal/catalyst mixing times of 60 min. Some explanations of why maxima are expected in X and Y_O at these optimum conditions are given in Section 3.4.

An additional run was performed at the optimum conditions, using SDS as the surfactant. This run resulted in a conversion of 18% and an oil yield of 7%.

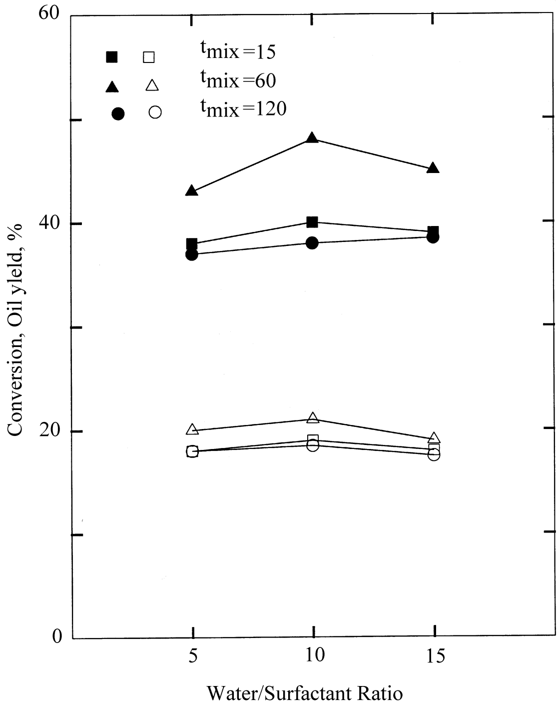

Fig. 3. Effect of conditions of preparing the RM catalyst (the water-to-surfactant ratio and the coal/catalyst mixing time) on DCL conversion (filled symbols) and oil yield (open symbols). DCL conditions are as in Fig. 1. AOT is the surfactant used.

These numbers are smaller than those for either AOT or Brij 30. A possible explanation of this phenomenon is that the structural differences of the three surfactants lead to the differences in their ability to produce reverse-micellar catalyst. However, the solubility of the surfactants in hexane are different. Equal amounts of hexane and surfactant were used in the runs, so as to compare surfactants on an equal footing. However, it was observed that some of the SDS was not dissolved. This could affect the results of DCL. A proper comparison of surfactants of different types should be carried out under the most favorable production procedures and conditions for each surfactant. Since this was not done in the present case, the results (with SDS at least) should be considered preliminary.

3.4. Analysis of conditions past optimum

In analyzing the effect of production conditions, the conversions can be expected to increase with increas-

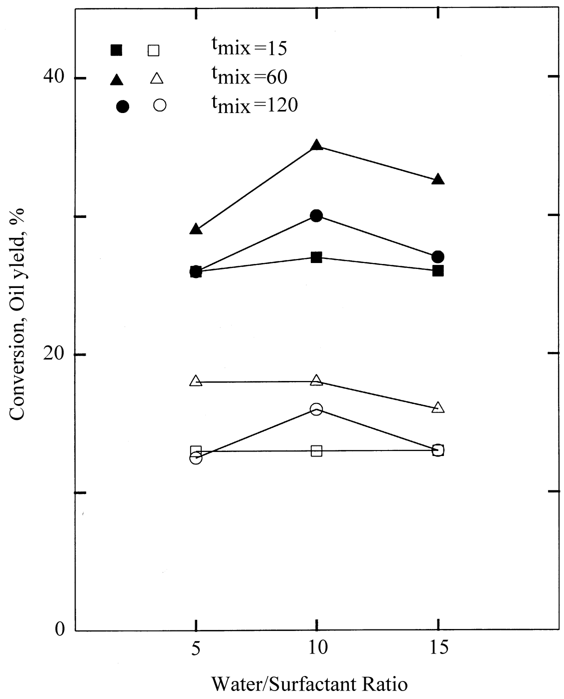

Fig. 4. Effect of conditions of preparing the RM catalyst (the water-to-surfactant ratio and the coal/catalyst mixing time) on DCL conversion (filled symbols) and oil yield (open symbols). DCL conditions are as in Fig. 1. Brij 30 is the surfactant used.

ing mixing time, as this would improve the distribution of the catalyst. Similarly, the catalytic performance could be expected to decrease with increasing values of W/S, since decreasing amounts of surfactant should lead to larger, unstable reverse micelles. The decreasing performance of the catalyst with increasing coal/catalyst agitation times at greater than the optimum value bears examination, as does the improvement in performance at increasing values of W/S at less than the optimum value of W/S. The drop in performance with both increasing agitation time of the slurry at large times, and with decreasing W/S at low ratios can be explained by postulating that excessive agitation breaks up the reverse-micellar structure. Further, note that W/S decreases at constant amounts of water with increasing amounts of surfactant, and thereby with increasing amounts of hexane. Hence, it could be postulated alternatively that the decrease in performance with the two phenomena noted above could be explained by excessive interactions of hexane with the coal or the catalyst, or both. To determine which (if either, or both) of these postulates are responsible for the decrease in performance, three sets of experiments were carried out. The experiments all used reverse-micellar catalysts created with W/S = 10 and AOT as the surfactant.

In the first experiment, coal was pre-mixed with hexane and the slurry was agitated for 60 min, then filtered and impregnated with the reverse-micellar catalyst using the optimal production procedure. In this case, the coal and hexane are agitated for a total of 120 min, while the coal/catalyst slurry is agitated for only 60 min. After DCL using this material, the conversion is 39%. This is similar to that for DCL after the coal/catalyst slurry is agitated for 120 min (with no pre-mixing of coal and hexane), 38% as shown in Fig. 3. However, when the coal/catalyst slurry is agitated for only 60 min (again with no pre-mixing of coal and hexane), the value of conversion for DCL is much greater, 48%. The conversions are the same with a significant change in coal/catalyst agitation time, 60–120 min, but decrease when the interaction between hexane and coal is increased by pre-mixing. Hence, it would appear that the reverse-micellar structure does not break up, or in any case, does not cause the catalyst performance to diminish, for agitation times greater than 60 min; on the other hand, the interaction with hexane may be significant.

In the second experiment, coal was pre-mixed with hexane and agitated for 45 min before the reverse-micellar solutions of ferric chloride and sodium sulfide were added, the coal/catalyst slurry then being agitated for 15 min. The conversion obtained for DCL with this material can be compared with the runs in Fig. 3 with coal/catalyst agitation times of 15 and 60 min. The conversion obtained in the run where coal is pre-mixed with hexane is 41%, a conversion of 40% is obtained for the 15 min run from Fig. 3, while the conversion with the 60 min run from Fig. 3 is 48%. The difference between the 15 min run and the 60 min run of Fig. 3 can be explained by the improvement in distribution of the catalyst, as noted earlier. Agitating coal and hexane for less than 60 min appears to have little effect on the conversion.

Following these two experiments, it is reasonable to assume that catalyst performance for agitations of less than 60 min is governed by the time of coal/catalyst mixing, with increasing agitation times increasing

Table 2
Surface area and average pore volume of DCL feed samples (by Quantachrome) related to conversion and oil yield after DCL

Sample	Description	Surface area (m^2/g)	Average pore diameter (Å)	Conversion (%)	Oil yield (%)
S1	Coal	1.052	229.1	34	13
S2	Coal + hexane, 60 min	0.8795	281.5	27	13
S3	Optimum coal/catalyst feed	0.5725	393.1	48	21
S4	Optimum coal/catalyst feed, acetone wash	0.8288	110.5	50	19

performance. For agitations of greater than 60 min, catalyst performance is governed by coal/hexane interactions, with increasing times decreasing performance. Hence, the decrease in performance with decreasing W/S at small W/S values can also be ascribed to coal/hexane interactions rather than breakup of the reverse micelles.

In the third set of experiments, DCL runs were performed on coal samples with increasing amounts of hexane soaking, 60 and 120 min. No catalysts were used in these runs. When compared to thermal liquefaction of the coal, the conversion decreases with an increase in the coal and hexane mixing time. The conversion went from 34% with no hexane pre-mixing, to 26% with 60 min of hexane pre-mixing, to 22% with 120 min of pre-mixing. It is clear from these results that it is the effect of hexane on coal that causes the DCL conversion to decrease with an increase in coal/catalyst agitation.

3.5. Effect of hexane on coal and DCL

The effect of hexane on coal could be that the hexane simply causes the coal to agglomerate. This would result in larger effective coal particle sizes for DCL. Past work has shown the conversion of DCL to decrease by 10% with an increase of a particle size from 1 to 1000 μm [8]. Other previous work has shown a negative effect of hexane on coal. One study [9] found that THF and tetralin would extract 8 and 6% by weight, respectively, from coal when each solvent was used alone. Further, hexane would similarly extract about 1% by weight from the coal. However, if the coal was first extracted with hexane and then with either THF or tetralin, the amount of material extracted drops to 4 or 1%, respectively. Clearly hexane, while not extracting significant amounts of coal constituents, interacts with the coal in such a way as to prevent the more-powerful solvents from extracting material from the coal.

In an attempt to analyze further the effect of hexane on coal, surface area and pore volume distribution tests were performed by Quantachrome Corporation. Four samples were used: coal, coal soaked in hexane for 60 min, the coal/catalyst feed produced using the optimum procedure and conditions for catalyst production (AOT, water-to-surfactant ratio of 10, and 60 min of coal/catalyst agitation), and the optimum coal/catalyst feed with an additional acetone wash. This acetone breaks the reverse micelles and thereby removes some of the AOT from the coal/catalyst feed. The surface area and average pore size for each sample are given in Table 2. Pore volume distributions are shown in Figs. 5–8. The samples were also subjected

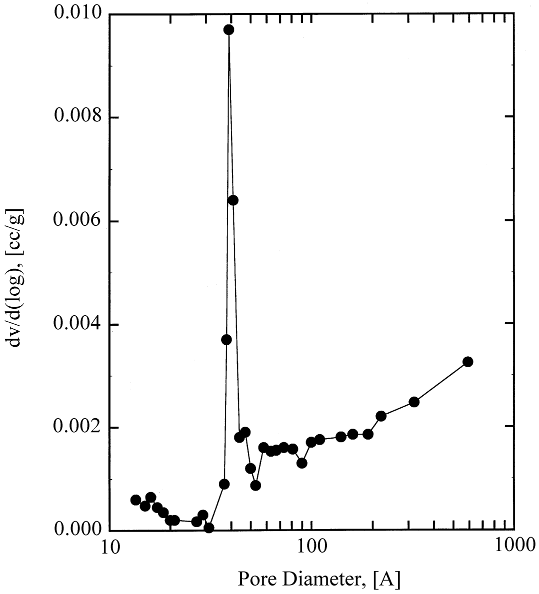

Fig. 5. Pore volume distribution for coal (sample S1).

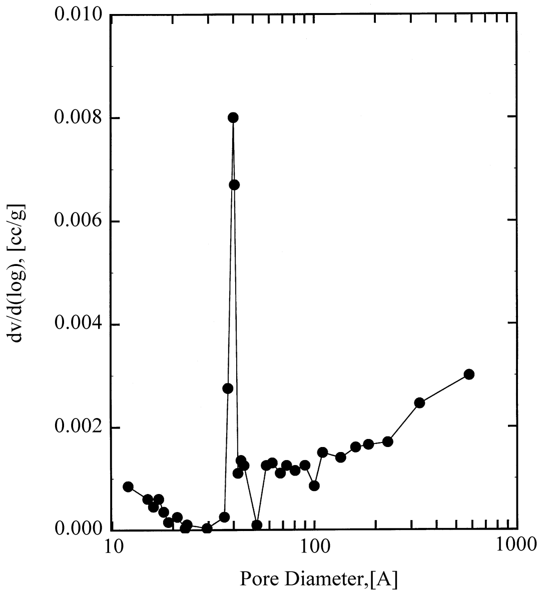

Fig. 6. Pore volume distribution for coal soaked in hexane for 60 min (S2).

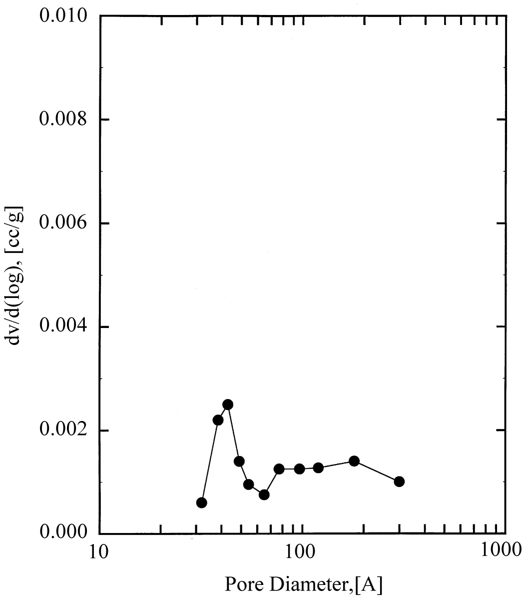

Fig. 8. Pore volume distribution for coal plus RM catalyst (optimum) after an additional wash in acetone to remove micelles.

to DCL; conversions and oil yields are also given in Table 2.

The results for samples S1 and S2 from Table 2 show that hexane decreases the surface area of coal and increases the average pore size. From Figs. 5 and 6, the increase in pore size appears to be due to a loss in the number of pores of the most prevalent size, around 35–45 Å. The loss in DCL conversion when coal is pre-soaked in hexane is consistent with the interaction effects for hexane, noted earlier.

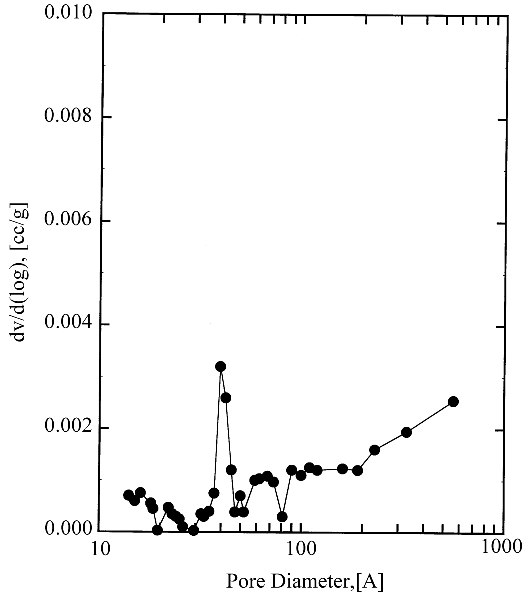

Fig. 7. Pore volume distribution for coal plus RM catalyst prepared in the optimal manner (S3).

Table 2 indicates that the surface area of S3, the optimum coal/catalyst sample, is even lower than that of S2, and the average pore size is considerably increased as well. Again, the corresponding pore-volume distribution curve (Fig. 7) shows a drastic reduction in the number of pores in the range 35–45 Å. The small pores in the coal/catalyst sample are either eliminated or filled with catalyst particles. As expected, the presence of the catalyst results in increasing conversion and yield values after DCL.

Finally, the surface area of S4 is higher than that of S3, and is about equal to that of S2. However, the pore-size distribution curve, Fig. 8, resembles Fig. 7 (S3) more than it does Fig. 6 (S2). There is a slight decrease in the number of pores around 40 Å, and the maximum in the pore-size distribution shifts slightly to around 45 Å, but the increase in surface area and the decrease in the average pore size is probably due to the absence of pores of size larger than 300 Å in Fig. 8 — note that Fig. 7 shows significant pore volumes associated with pores of size 200–550 Å. Therefore, the breakage of the reverse micelles and/or the presence of acetone decreases the number of the largest pores. However, note that the removal of the reverse-micellar structure and the presence of acetone does not affect the conversion or oil yield of the DCL experiments, relative to DCL of S3.

4. Summary and conclusions

1. In the optimal procedure for the production of a ferric-sulfide-based reverse-micellar catalyst for DCL, first the ferric-chloride reverse-micellar solution and the sodium sulfide reverse-micellar solution are mixed together for 5 min. Then coal is added and the suspension is agitated. This suspension is then filtered and dried, and used as the feed for DCL.
2. A coal/catalyst mixing time of 60 min and a water-to-surfactant ratio (W/S) of 10 are optimal conditions for the production of the reverse-micellar catalyst using AOT. When hexane and coal are contacted for more than 60 min, the hexane/coal interaction has a significant negative effect on DCL. Decreasing values of W/S result in an increased amount of hexane present, especially at low values of W/S, and thereby an increased hexane/coal interaction. Increased coal/catalyst agitation, which could lead to destruction of the reverse micelles, does not appear to decrease the performance of the catalyst during DCL.
3. The presence of hexane affects the surface area and the pore-volume distribution of coal. This could be the cause of the decreased performance with increasing hexane contact time. The effect of hexane could also be due to particle agglomeration. Agglomeration could cause the decrease in conversion and oil yield observed when coal was mixed in hexane. The presence of acetone also affects the surface area and pore-size distribution, but to a lesser extent. The acetone is added to breakup the reverse micelles. Neither the change in surface area nor the destruction of the reverse micelles in the presence of acetone causes any significant change in DCL conversion or oil yield.
4. AOT is a superior surfactant for reverse-micellar catalyst production relative to Brij 30, at least for the preparation conditions used herein. Lower conversions for SDS surfactant may be caused by the lower solubility of the surfactant in hexane relative to AOT.

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