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*THE HYDROCRACKING OF RESIDUAL
OILS AND TARS
PART 5: SURFACE-COATED COBALT
MOLYBDATE CATALYSTS FOR
HYDRO-TREATING*

E. C. MCCOLGAN, P. S. SOUTAR, M. A. RETHIER, AND B. I. PARSONS

FUELS RESEARCH CENTRE

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THE HYDROCRACKING OF RESIDUAL OILS AND TARS
PART 5: SURFACE-COATED COBALT MOLYBDATE CATALYSTS FOR HYDRO-TREATING

by

E. C. McColgan*, P. S. Soutar*, M. A. Rethier**,
and B. I. Parsons***

ABSTRACT

A surface-layering technique is described for preparing low-cost, powdered cobalt molybdate catalysts for hydrocracking and desulphurizing residual oils in liquid phase systems. A small quantity of peptized alumina is milled on to the surface of 100 to 500-micron particles of alumina monohydrate (Boehmite). Concentrated solutions of salts of the active ingredients are dispersed in the peptized alumina, then the mixture is dried and calcined. Catalysts were prepared containing (over-all) concentrations of combined oxides of 13.0, 3.3, and 1.6 % by weight. The hydrocracking and desulphurizing capability of each catalyst was determined with bitumen separated from the Athabasca tar sands at a liquid hourly space velocity of 1.05, and 2000 psi hydrogen pressure. The activity of the 13.0 and 3.3 % catalysts were quite similar. The activity of the 1.6 % combined oxides system was measurably less. The practical lower-limit of oxides concentration for catalysts prepared by the described method is about 3 % by weight.

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Direction des mines
Rapport de recherche R 263

L'HYDROCRAQUAGE DES HUILES RÉSIDUELLES
ET DES GOUDRONS

5^e Partie: Les Catalyseurs couverts de cobalt et de
molybdène pour l'hydrotraitement.

par

E. C. McColgan*, P. S. Soutar*, M. A. Rethier**,
et B. I. Parsons***

RÉSUMÉ

Dans ce rapport, les auteurs décrivent une technique d'enduisage de surface pour la préparation peu coûteuse des catalyseurs de cobalt et de molybdène en poudre pour l'hydrocraquage et la désulfuration des huiles résiduelles dans des systèmes à phase liquide. Ils ont enduit une petite quantité d'alumine peptisée sur la surface des particules de 100 à 500 microns de monohydrate d'alumine (Boéhmite). Ils ont dispersé les solutions concentrées de sels d'ingrédients actifs dans l'alumine peptisée puis ils ont séché et calciné le mélange. Les auteurs ont préparé les catalyseurs contenant des concentrations (totales) d'oxydes combinés de 13.0, 3.3, et 1.6 % par pesanteur. Ils ont déterminé la capacité de l'hydrocraquage et de la désulfuration de chaque catalyseur avec le bitume séparé des Sables bitumineux d'Athabasca à la vitesse spatiale liquide par heure de 1.05 et à une pression d'hydrogène de 2000 psi. Ils ont trouvé que l'activité des catalyseurs de 13.0 et de 3.3 % était très semblable. Ils ont aussi trouvé que l'activité des systèmes d'oxydes combinés de 1.6 % était très inférieure. La limite inférieure et pratique de la concentration d'oxydes pour les catalyseurs préparés par la méthode décrite est à peu près 3 % par pesanteur.

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INTRODUCTION

The prospects for hydro-treating residual feed stocks appear best with a liquid-phase, fluidized bed system embodying small-particle (powder) catalyst (1, 2, 3). The main advantages of the fluidized bed arrangement are:

- a) improved temperature control.....a vital requirement in a system involving so many highly exothermic reactions, and
- b) the fact that catalyst can be withdrawn and added continuously, or semi-continuously, as required without process interruption.

In very general terms these advantages mean that the activity or conversion requirements can be held at almost any desired level, or changed rapidly, as circumstances dictate. The following report is concerned with an investigation of a technique for preparing low-cost cobalt molybdate catalysts for hydro-cracking residual oils in this type of bed.

Considerable development work on the mechanical engineering associated with the operation of a liquid-phase fluidized bed has been undertaken at the Fuels Research Centre and the process appears viable (4). One serious problem remaining is the catalyst system. Unfortunately, catalyst consumption is excessive with asphaltic feed stocks containing high concentrations of nickel and vanadium*. Effective processes must be developed for lowering catalyst cost if a catalytic treatment for residual oils is to be competitive with conventional "coking" or thermal refining methods. Either the initial cost of the catalyst must be so low as to permit discarding it after single use or a means of regenerating it must be devised to permit repeated use. Both approaches to lowering catalyst cost are under investigation, but the emphasis is presently on the development of intrinsically low-cost formulations to minimize the requirement for regeneration.

The most widely used catalysts for hydrocracking and desulphurizing heavy oils involve cobalt and/or nickel and molybdenum oxides and sulphides

*With a feed stock such as the Athabasca bitumen, catalyst life is only 100 to 150 hours at moderate pressures (1500 to 2000 psi).

dispersed on alumina gel. The concentration of metal oxides is usually between 12 and 15 % by weight. In a series of preliminary experiments on the hydro-treating of Athabasca bitumen using commercially available, powdered, cobalt molybdate catalyst, a visual examination of the working catalyst suggested that the internal pore structure of the particle was not contributing significantly to the refining process. During the first few hours of operation, the internal pore structure appeared to become fouled with coke and tar residues leaving only a thin layer of working catalyst on the outer surface. This observation and the knowledge that diffusion rates in the liquid state are fundamentally much lower than those encountered in the vapour state suggested that the surface-coating of an inert particle with catalytic ingredients would lower catalyst cost without sacrificing effectiveness.

In the work described here small quantities of cobalt and molybdenum salts were milled on to the surface of a large quantity of powdered alumina monohydrate. The objective was to coat the outer surfaces of the support particles with a thin layer of active catalyst and thereby reduce component concentration and cost. Ideally, the best way to test the catalysts produced was in powder form in the pilot-scale fluidized bed reactor (1). Unfortunately, the time required to load, start-up, and run each catalyst was prohibitive and it was necessary to resort to other means of evaluation. Catalysts containing over-all concentrations of 13.0, 3.3, and 1.6 % by weight of the oxides of cobalt and molybdenum were prepared (as powders) then formed into 1/8 x 1/8-inch-diameter pellets and tested in a bench-scale flow system using Athabasca bitumen as feed stock. The operating conditions for the hydro-treating tests were selected with the view to producing low-sulphur distillate fuel oils.

EXPERIMENTAL

Catalyst Preparation

The catalyst system investigated was the traditional "cobalt molybdate" catalyst developed many years ago for hydrodesulphurization and hydrocracking (5). In principle, the procedure used to study the concept of building up a layer of active catalyst on an inert particle was quite simple. First, hard inert particles

of alpha alumina monohydrate (Boehmite) were coated lightly with peptized alumina in a mix-muller, then appropriate amounts of concentrated solutions of salts of the active ingredients were added. After a thorough mulling, the mixture was dried, calcined, and pelleted.

The hard inert particles, and the alumina used in the peptizing operation, were both high-purity Boehmites produced by the hydrolysis of aluminum alkoxides in the manufacture of alcohols. The hard (peptizing resistant) particle form was kiln-dried material in which 93 % of the particles occurred in the size range 100 to 500 microns in diameter. The easily peptized monohydrate was spray-dried material with the bulk of the particles in the size range 30 to 100 microns. Both were obtained from the Continental Oil Company of Teterboro, New Jersey.

In the monohydrate form the alumina possesses little or no pore structure or surface area in the catalytic sense. It is only on calcination that the high surface area and pore structure develop. For the grades of the material used here, the manufacturer reports calcined surface areas of approximately $200 \text{ m}^2/\text{g}$ and a total pore volume of 0.5 ml/g . To ensure that the catalytic ingredients were located on the outer surfaces of the particles, the powders were impregnated as the monohydrate, then calcined. As mentioned previously, the ultimate objective was to use the catalysts in the powder form in a liquid-phase fluidized bed system. The larger particle size of the Boehmite was selected to minimize the concentration of fine particles which tend to float out of such a system causing operational difficulties.

The details of the procedure used in the preparation of the catalysts were as follows:

1. A mixture of 1000 g of the spray-dried (peptizable) alumina monohydrate and 4000 g of the large-particle (inert) monohydrate was prepared in a twin-shell blender and placed in a Simpson mix-muller (Model LF).
2. A solution of 1200 ml of distilled water and 14 ml of nitric acid (70 %) was added slowly to the mix-muller to dampen the powder. (Where necessary, the amount of water added in this step was adjusted in accordance with the amount of water added in the solutions of the active ingredients so as to maintain the total approximately constant). The dampened mixture was thoroughly mullled for 3 minutes then allowed to stand for

10 minutes to ensure complete peptization of the spray-dried monohydrate. Preliminary experiments with the kiln-dried monohydrate under similar conditions indicated no peptization.

3. With the mix-muller in operation, appropriate amounts of concentrated solutions of reagent-grade cobalt nitrate $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and ammonium paramolybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ were added slowly. The usual procedure was to add the cobalt solution first, though this procedure was reversed for one batch of catalyst. The mixture was muller for 4 minutes after the addition of each ingredient.
4. The impregnated mixture was spread as a half-inch layer in a stainless steel tray and dried in air at 110°C for 3 hours, crushed lightly with a wooden rolling pin to break up lumps, then calcined at 500°C for 3 hours. The particle size distribution in the mixture at this point was essentially the same as that of the original kiln-dried monohydrate, i.e. 100 to 500 microns.
5. The calcined alumina was formed into $1/8 \times 1/8$ -inch pellets at 40,000 psi in a continuous pelleting press using 2 % stearic acid as a binder-lubricant. The pellets were recalcined at 500 to 550°C for 4 hours to remove the stearic acid.

Catalysts were prepared in which the over-all concentrations of cobalt and molybdenum oxides were 13.0, 3.3, and 1.6 % by weight. For comparison purposes, a batch of the pure alumina support material was put through the same procedure (adding distilled water in place of the salt solutions) for "zero" metals baseline experiments. The compositions were:

- No. 1 : 4.4 % CoO and 8.6 % MoO_3 (cobalt added first),
- No. 2a: 1.1 % CoO and 2.2 % MoO_3 (cobalt added first),
- No. 2b: 1.1 % CoO and 2.2 % MoO_3 (molybdenum added first),
- No. 3 : 0.5 % CoO and 1.1 % MoO_3 (cobalt added first), and
- No. 4 : pure Al_2O_3 support.

The catalyst numbers correspond to the graph designations indicated on the figures in the section describing the experimental results.

Apparatus and Procedure for Testing Catalysts

The apparatus used was the bench-scale flow system described in the first report in this series (6). In all of the experiments, the oil feed and hydrogen were pumped in at the bottom of the reactor. The unit was operated, in effect,

as a continuous-flow, liquid-phase system with the oil and hydrogen percolating up through the reactor. All of the experiments were undertaken at 2000 psi, LHSV = 1.05 and at an exit gas rate of 5000 standard cubic feet per barrel (scf/bbl). A series of experiments with each feed stock consisted of four consecutive 3-hour runs at increasing temperatures, 430, 440, 450, and 460°C followed by a 3-hour repeat run at 440°C. The product of the second run at 440°C was compared with that from the first to obtain a measure of the extent of catalyst deactivation. The background to this particular procedure is explained fully in the second report in this series (7). The analytical procedures used on the feed stocks and products are also described in the second report. Hydrocarbon material boiling up to the equivalent of 975°F at one atmosphere pressure is classed as distillable oil, and material boiling above 975°F equivalent temperature is residuum.

Feed Stock

The feed stock for this work was the same as that used and described in the first and second reports in this series (6, 7), i.e., bitumen from Great Canadian Oil Sands Ltd. operating in the Athabasca tar sands reserve at McMurray, Alberta. The Athabasca material is separated from the sand using the Clark Hot-Water Process and froth flotation followed by dilution with naphtha and centrifuging. The residual concentration of clay and silt in the bitumen after recovering the solvent naphtha was about 0.9 % by weight, and the concentrations of +975°F residuum and sulphur were 51 wt % and 4.72 wt % respectively.

EXPERIMENTAL RESULTS AND DISCUSSION

The surface-layering technique accomplished many of the objectives of the investigation. The effectiveness of the 13.0 and 3.3 %-oxide catalysts were very similar. Both of these catalysts could hydrocrack and desulphurize 80 to 85 % of the bitumen to meet the sulphur requirements for distillate fuel oils. The activity of the catalyst that contained only 1.6 % combined oxides was measurably less. The practical lower-limit of oxides concentration appears to be about 3 % by weight. None of the catalysts, of course, could desulphurize

the dregs of the hydrocracked bitumen (the +975°F pitch fraction) to meet the specifications for residual fuel oil. However, this was not expected because, as yet, no catalyst formulation of any sort has been devised which can do this on a commercial basis.

The experimental results obtained with the three catalysts, 13.0 %, 3.3 %, and 1.6 % combined oxides and the pure Al_2O_3 support material are summarized in Figures 1 and 2 and Table 1. The effect of reaction temperature on the conversion of the residuum hydrocarbon to distillable oil and on sulphur removal is shown in Figure 1 and the yields of liquid product and the specific gravities are given in Figure 2. The yield and sulphur content of the product fractions formed at 450°C are shown in Table 1. The comparison was made at 450°C as the level of conversion of the +975°F residuum to distillate (65 to 70 %) correspond very closely to that which would be required to sustain a typical tar sands operation (8).....assuming that the bulk of the energy required for the mining and separation processes can be derived from the +975°F pitch fraction either by direct combustion followed by flue gas desulphurization, or by gasification followed by combustion.

It is immediately apparent from Figure 1 that whereas the cobalt molybdate system is a strong desulphurizing catalyst for distillates, it is a comparatively weak catalyst for hydrocracking residuum. For example, at a reaction temperature of 460°C, the sulphur content of the distillate hydrocarbon produced catalytically is of the order of 0.5 % compared to 2 % over the alumina support (a four-fold reduction in sulphur concentration). On the other hand, the conversion of the +975°F residuum to distillate hydrocarbon was only 82 % with the strongest catalyst compared to 71 % over the pure alumina support.....a significant but relatively small improvement. This aspect of the investigation suggests that a more powerful catalyst system for the liquid-phase hydro-treating of residual feed stocks might be a blend of two catalyst powders; one specifically for hydrocracking and one for desulphurizing. A dual-function catalyst is not a new concept, but a powdered catalyst system for liquid-phase hydrogen treating offers a unique opportunity to mix two (or more) separate and distinct catalyst systems into one reaction vessel.

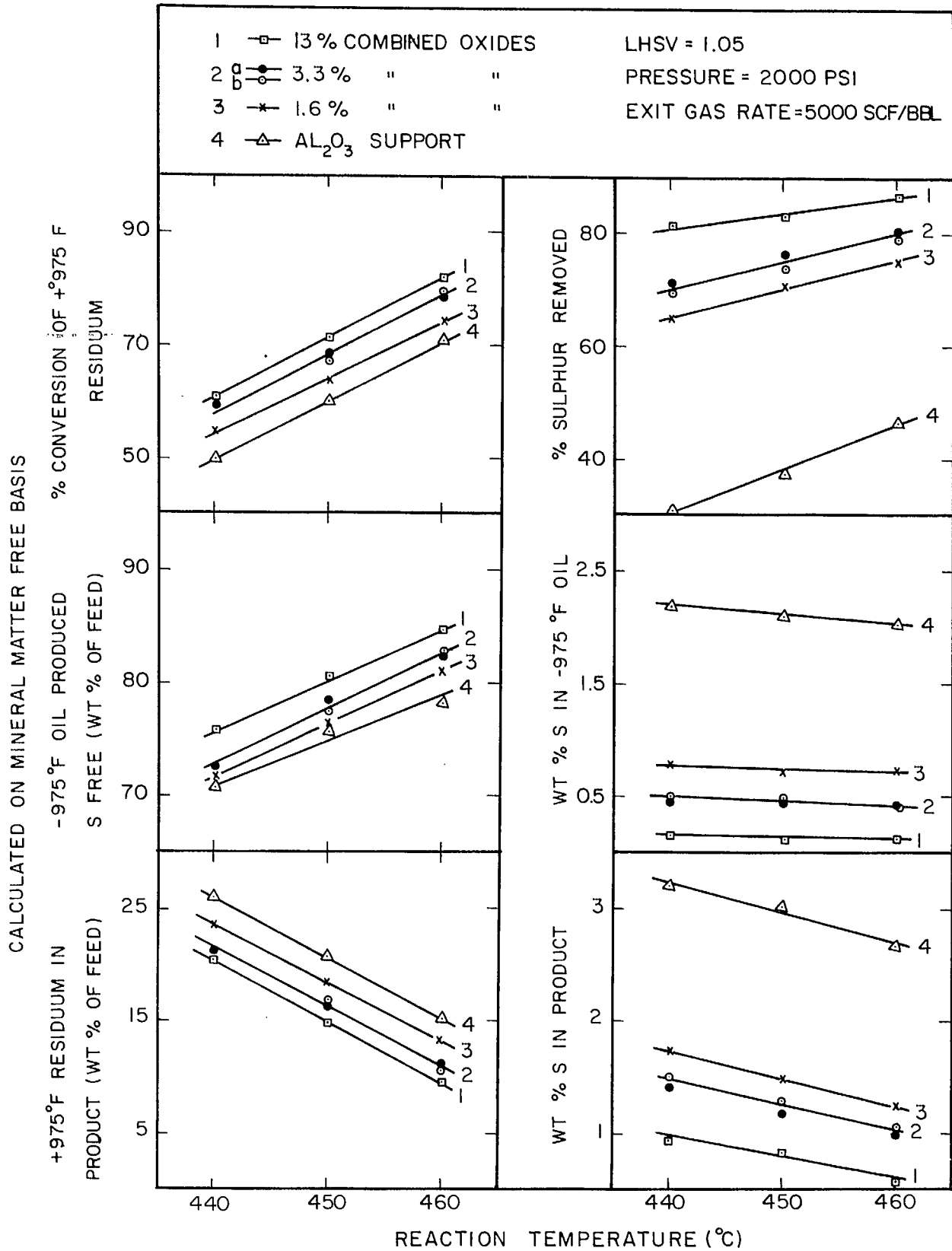


Figure 1 - The effect of reaction temperature on the conversion of the +975°F residuum to distillable oil and on sulphur removal.
 Note: the residuum remaining in the product and the distillable oil produced are expressed as weight % of feed.

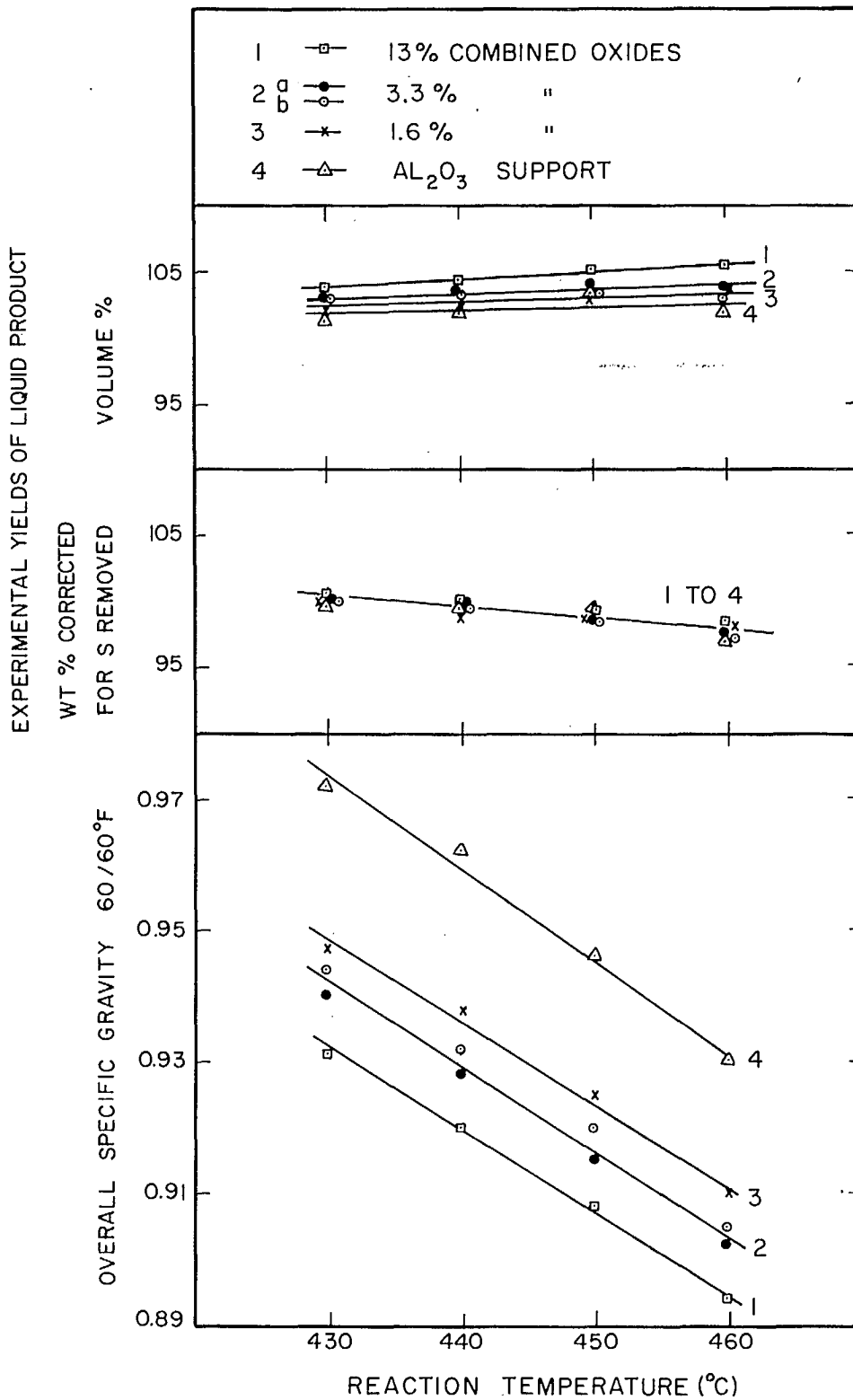


Figure 2 - The specific gravity and experimentally observed yields of liquid product formed with the various catalysts.

Table 1

The Yield and Sulphur Content of the Product Fractions Formed at 450°C over the Various Catalysts

(Note: the fractions are expressed as weight % of mineral-matter-free feed)

Boiling Range of Fraction Equiv. Temp. at one atm. (°C) (°F)		Feed Stock		Al ₂ O ₃ Support		13.0 % Oxides		3.3 % Oxides		1.6 % Oxides	
		Wt % of Fraction	Wt % Sulphur	Wt % of Fraction	Wt % Sulphur	Wt % of Fraction	Wt % Sulphur	Wt % of Fraction	Wt % Sulphur	Wt % of Fraction	Wt % Sulphur
Gas Produced		-	-	0.6	-	0.8	-	1.5	-	1.4	-
RT - 100	RT - 212	-	-	1.5	-	2.5	-	2.6	-	1.8	-
100 - 200	212 - 392	2.1	-	8.2	1.03	7.5	0.10	7.8	0.18	7.2	0.32
200 - 250	392 - 482	1.5	1.15	6.9	1.33	9.2	0.08	7.8	0.15	7.5	0.38
250 - 275	482 - 527	5.2	1.65	6.1	1.69	6.1	0.09	6.6	0.20	6.0	0.39
275 - 332	527 - 630	5.5	2.49	14.3	2.09	19.1	0.16	14.3	0.27	13.3	0.55
332 - 361	630 - 682	6.9	2.78	9.8	2.25	7.0	0.23	10.2	0.41	10.2	0.71
361 - 391	682 - 736	5.1	3.10	5.1	2.55	5.6	0.28	5.6	0.54	7.5	0.85
391 - 421	736 - 790	6.7	3.38	6.7	2.88	6.3	0.36	6.0	0.66	6.8	0.96
421 - 524	790 - 975	16	3.86	19	3.34	17	0.51	17	0.89	17	1.28
+524	+975	51	6.2	20	5.7	15	2.9	17	3.7	18	4.0
Sulphur Removed		-	-	1.8	-	3.9	-	3.6	-	3.3	-
Totals*		100	4.75	100	4.70	100	4.53	100	4.57	100	4.60

Pressure = 2000 psi

LHSV = 1.05

Exit Gas Rate = 5000 scf/bbl

Reaction Temperature = 450°C

*sulphur totals recapitulated from analytical results

On the basis of the desulphurizing capability, the surface-layering technique for catalyst preparation appears to be quite successful over the whole range of concentrations, 13.0 to 1.6 weight per cent. The sulphur content of the over-all distillate formed increased slightly from 0.3 to 0.7 % as the oxide concentration decreased, but it was still within the present commercially acceptable range* and considerably below that produced over the alumina support alone. The changes in hydrocracking activity with oxide concentration, however, were more significant. At 460°C, the conversion decreased from 82 % for the 13.0 %-oxide system, to 80 % for the 3.3 % combined oxides, to 74 % for the 1.6 %-oxide catalyst. The practical, lower limit of oxides for hydrocracking purposes appears to be in the region of 3 % combined oxides. A five degree (centigrade) increase in reaction temperature would be sufficient to restore the activity of the 3.3 %-oxide system to that observed with 13 % combined catalyst. Such a change would not grossly affect catalyst life or increase gas formation unduly.

The order in which the metal salt solutions are added to the alumina support does not appear to be an important factor (compare graphs marked 2a and 2b in Figures 1 and 2). Within experimental error, the conversions attained at all temperatures, and the properties of the products formed, are essentially the same whether the cobalt solution is added first or the molybdenum solution is added first.

Gas formation was not a serious problem with any of the catalyst systems. The largest amount of gas produced was only 3.3 wt % (based on feed) with the 1.6 %-oxide catalyst at 460°C. This is to be compared with the 8 to 9 weight % gas formation encountered in the purely thermal (non-catalytic) hydrocracking process reported in Part 3 of the current series of papers (9). Even the weakest of the catalyst systems investigated produced 5 to 6 % by weight more liquid distillate fuel than the corresponding non-catalytic process. If a value

*the activity of the 1.6 % catalyst was just sufficient to produce 1%-sulphur distillate fuel oil (650 - 975°F) at 450 - 460°C.

of \$4.00/bbl is placed on the additional distillate product formed, a 5 % improvement in liquid yield represents approximately 6 million dollars of additional revenue per year for a 100,000-bbl/day plant*. This advantage is based on only the additional liquid product formed, and does not take into account the enormous improvement in product quality that inherently accompanies a catalytic treatment relative to a thermal process.

An interesting side issue on the matter of gas formation and liquid yield arises in connection with the experiments involving the pure alumina support. The extent of gas-make with the alumina support was as low as that encountered with any of the impregnated catalysts. When the investigation was begun, it was expected that the hydrocracking characteristics of the bitumen would be essentially the same over the pelleted support material as those observed in the non-catalytic or thermal system. In most respects (product quality, etc.) this was true, but the outstanding exception was in the amount of gas produced. At comparable conversion levels, substantially less gas was formed with the vessel packed with "inert" alumina than in the purely thermal experiments (9) where the reaction vessel was used empty (no packing). The important mechanical difference, of course, is the fact that the residence time of the oil in the packed vessel is only about one-third that with the empty vessel. It would seem that a potentially fruitful area for further research on the "thermal" hydrocracking process would be on the effect of the extent and nature of packing in the reaction vessel. The time-temperature dependence of the thermal process obviously has an important bearing on liquid-distillate yield.

The extent of catalyst deactivation observed with the catalyst systems decreased slightly with decreasing concentration of oxides. A comparison of the conversions attained (desulphurization and hydrocracking) in the first and second runs at 440°C is shown in Table 2. As one would expect, catalyst deactivation was greatest with the strongest catalyst and least with the weakest. The relative differences between 13.0 % and 3.3 %-oxide systems were quite small. Unexpectedly, there was also a measurable change observed in the hydrocracking

*this assumes that the +975°F pitch and the crack gas are in surplus (and of little value) at the plant site.

Table 2

A Comparison of the Conversions Attained in the First and Second Runs at 440°C

Concentration of Combined Oxides (%)	% Sulphur Removed			% Conversion +975°F Residuum		
	First Run	Second Run	Change	First Run	Second Run	Change
Al ₂ O ₃ Support only	30	29	1	50	48	2
13.0	81	72	9	61	57	4
3.3	71	64	7	59	55	4
1.6	65	59	6	54	51	3
Operating Pressure = 2000 psi				LHSV = 1.05		
Exit Gas Rate = 5000 scf/bbl						

activity of the alumina support material.....from 50 % conversion during the first run at 440°C to 48 % in the second. This suggests that alumina does possess some activity for hydrocracking which is being spoiled by either carbon fouling or metals deposition in much the same way as a conventional cobalt molybdate catalyst.

To conclude, the results of the study were very encouraging because they confirmed the validity of the basic principles of the preparative procedure. The activity of the catalysts prepared here compare favourably with the activity of commercially available cobalt molybdate catalysts (described in Part 2, Reference 7). There was no indication of loss of activity due to the occlusion of the metal oxides in the surface of the alumina during the phase changes in the calcination process. It must be emphasized that the experiments described are our first attempts at surface-layering. Additional experimentation will undoubtedly result in further improvements.

POSTSCRIPT

The mention of specific brand names for equipment and materials is made for information purposes only and does not imply endorsement by the Mines Branch.

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