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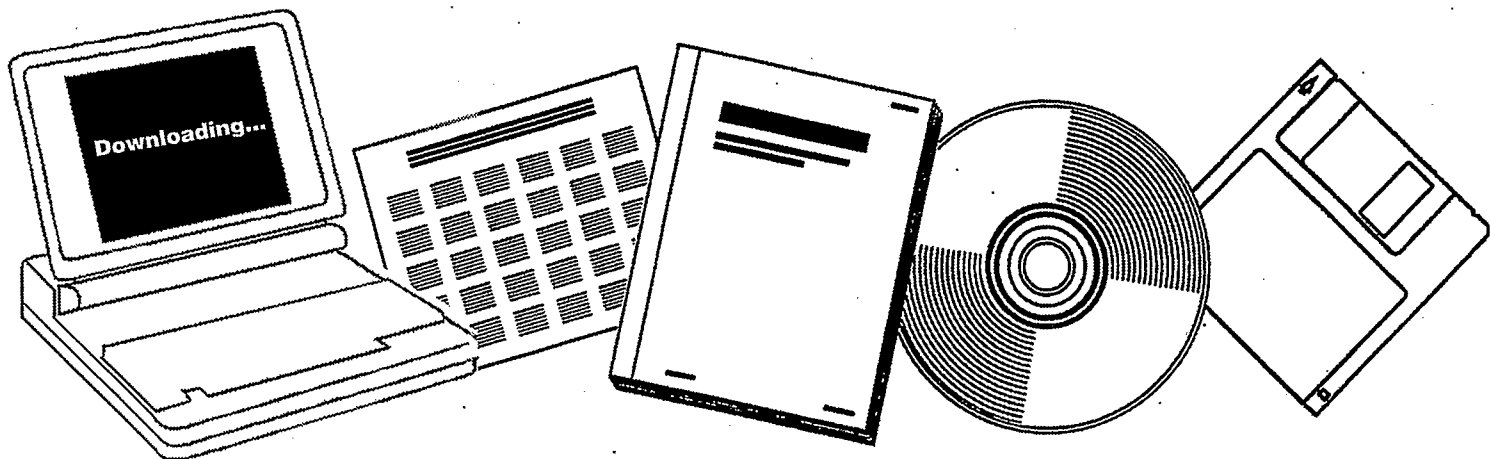
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**NEW CATALYSTS FOR THE INDIRECT  
LIQUEFACTION OF COAL. SECOND ANNUAL  
TECHNICAL REPORT, AUGUST 1, 1981-JULY 31,  
1982**

VIRGINIA COMMONWEALTH UNIV., RICHMOND.  
DEPT. OF CHEMISTRY

1982



U.S. Department of Commerce  
**National Technical Information Service**

DOE/PC/30228--T6

DOE/PC/30228--T6

DE83 001584

NEW CATALYSTS FOR THE INDIRECT LIQUEFACTION OF COAL

Second Annual Technical Report

Grant No: DE-FG22-80PC30228

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Period Covered: August 1, 1981 - July 31, 1982

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## New Catalysts for the Indirect Liquefaction of Coal

### Abstract

Series of zeolite-supported iron-containing catalysts with weight percent iron (% Fe) varying from ~1 to ~17% Fe have been prepared from  $\text{Fe}_3(\text{CO})_{12}$  and the synthetic zeolites ZSM-5, mordenite and 13X by an extraction technique. The zeolites ZSM-5 and mordenite were used in the acid form, 13X in the sodium form. The catalysts were characterized by a variety of techniques including infrared spectroscopy, X-ray powder diffractometry, X-ray photoelectron spectroscopy, ion-scattering spectrometry and Mössbauer spectroscopy. All catalysts contain highly dispersed, small particle-sized  $\gamma\text{-Fe}_2\text{O}_3$  with a small amount of the iron (0.6-1.5% Fe depending on the support) located in the pores of the support. Evaluation of the catalytic ability of some of these materials for synthesis gas conversion was conducted at 280°C and 300°C by using a fixed-bed continuous flow microreactor. All catalysts evaluated produce significant quantities of hydrocarbons. The distribution of hydrocarbons varies, depending upon the support used, for catalysts with similar weight percent iron. For the liquid hydrocarbons, Fe/ZSM-5 produces the highest percentage of aromatics, Fe/mordenite produces the highest percentage of olefins, and Fe/13X produces the highest percentage of saturates. The effect of support acidity and pore structure on hydrocarbon product distribution is discussed.

## Introduction

The use of bifunctional metal/zeolite catalysts for the conversion of synthesis gas (carbon monoxide and hydrogen) to gasoline range hydrocarbons has recently attracted much attention. For example, the combination of metal oxides with the medium pore (~6Å) zeolite ZSM-5 and the use of a metal nitrate impregnated ZSM-5 catalyst have been shown to produce gasoline range hydrocarbons containing a high percentage of aromatics from synthesis gas (1-3).

The efficiency and selectivity of a supported metal catalyst is closely related to the dispersion and particle size of the metal component and to the nature of the interaction between the metal and the support. For a particular metal, catalytic activity may be varied by changing the metal dispersion and the support; thus, the method of synthesis and any pre-treatment of the catalyst is important in the overall process of catalyst evaluation. Supported metal catalysts have traditionally been prepared by impregnation techniques that involve treatment of a support with an aqueous solution of a metal salt followed by calcination (4). In the Fe/ZSM-5 system, the decomposition of the iron nitrate during calcination produces  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> of relatively large crystallite size (>100 Å). This study was initiated in an attempt to produce highly-dispersed, thermally stable supported metal catalysts that are effective for synthesis gas conversion. The carbonyl Fe<sub>3</sub>(CO)<sub>12</sub> was used as the source of iron; the supports used were the acidic zeolites ZSM-5 and mordenite and the non-acidic, larger pore zeolite, 13X.

## Experimental

The zeolite supports were calcined in air prior to use. ZSM-5 (SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> = 30) and mordenite (SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> = 11.4) were used in the acid form (H-ZSM-5, H-Mord), and 13X (SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> = 1) used in the sodium form. Series of zeolite-supported iron catalysts of weight percent iron (% Fe) varying from ~1% to ~17% Fe were prepared from Fe<sub>3</sub>(CO)<sub>12</sub> and the calcined zeolites with cyclohexane as solvent by an extraction technique developed in our laboratory (5). All materials were characterized by infrared spectroscopy, X-ray powder diffractometry (XRPD), X-ray photoelectron spectroscopy (XPS), ion-scattering spectrometry (ISS), and Mössbauer spectroscopy. The weight percent of iron was determined by atomic absorption spectroscopy.

For the catalytic activity evaluation, selected materials were pressed into 1/8 inch diameter pellets, reduced under H<sub>2</sub> at 300 psig (20.7 bar) and 450°C for 24 hours, and then treated with synthesis gas (H<sub>2</sub>/CO = 1/1) at 100 psig (6.9 bar) and 250°C for 24 hours. Catalytic data were obtained by using a fixed-bed continuous flow microreactor. Conditions employed were 300 psig (20.7 bar) pressure of 1/1 synthesis gas, GHSV ~1000 h<sup>-1</sup> and reactor temperatures of 280°C and 300°C. The products obtained over a 48 to 72 hour period from the microreactor were analyzed in two fractions. The gaseous effluent, including C<sub>1</sub>-C<sub>4</sub> hydrocarbons, was analyzed by use of a gas chromatograph that is an integral part of the microreactor. The liquid product was collected in an ice-cooled trap located after a heated (175°C) trap to collect high molecular weight (wax) products. The liquid product was separated into oil and aqueous fractions; analysis of the oil fraction was

accomplished by FIA chromatography on silica gel (6) and by simulated distillation (7).

## Results and Discussion

Characterization. Infrared spectra of the prepared materials do not show the presence of  $\nu(\text{CO})$  bands; thus, it is concluded that decomposition of  $\text{Fe}_3(\text{CO})_{12}$  accompanies adsorption onto the supports during the preparative procedure. For catalysts with low (<10%) iron loading, only the support is detected by XRPD suggesting that the particle size of the iron-containing species is <50 Å. However, above ~10% Fe loading a diffraction pattern associated with  $\gamma\text{-Fe}_2\text{O}_3$  and/or  $\text{Fe}_3\text{O}_4$  is observed in addition to that for the support. The XPS data confirm the presence of iron oxide in all the prepared catalysts; a detailed study of the XPS data for a series of Fe/ZSM-5 catalysts has recently been described (8). Ion scattering spectrometry, with  $^4\text{He}^+$  sputtering, has been employed to study the surface of the iron/zeolite catalysts. For all samples studied, large Fe/Si intensity ratios are obtained in comparison with those ratios from iron/zeolite catalysts prepared by wet-impregnation techniques using an aqueous solution of iron nitrate. It is concluded that the iron oxide particles in the catalysts prepared from  $\text{Fe}_3(\text{CO})_{12}$  are considerably smaller than those obtained by using iron nitrate. Depth profiling studies (XPS and ISS) have demonstrated that some iron (~1%) is incorporated into, and is strongly interacting with, the zeolite support; data for the Fe/ZSM-5 catalysts have been reported (8). Recent studies (9) of the infrared spectra of chemisorbed pyridine on Fe/ZSM-5 catalysts prepared by the extraction technique from  $\text{Fe}_3(\text{CO})_{12}$  has suggested that, in the prepared form, the iron is not interacting with the support at the Brønsted acid sites whereas for iron nitrate impregnated Fe/ZSM-5 catalysts some ion exchange does take place. Mössbauer spectroscopy has identified the surface iron-containing species as small particle-sized  $\gamma\text{-Fe}_2\text{O}_3$  in a superparamagnetic form (10, 11). For Fe/ZSM-5 catalysts, (15% to 16% Fe) magnetic and Mössbauer studies (12) have concluded that the carbonyl-impregnated samples contain  $\gamma\text{-Fe}_2\text{O}_3$  particles of diameter 60-66 Å whereas the nitrate-impregnated samples contain  $\alpha\text{-Fe}_2\text{O}_3$  of ~100 Å diameter.

Characterization of the used catalysts (~15% Fe) has shown the presence of  $\chi\text{-Fe}_5\text{C}_2$  (Hägg carbide) and  $\text{Fe}_3\text{O}_4$  for all materials investigated. For an Fe/ZSM-5 catalyst, it is estimated that ~80% of the iron is in the metallic form after the reduction step (12). Following the carbiding step,  $\chi\text{-}$  and  $\epsilon\text{-}$ carbides are detected in addition to  $\text{Fe}_3\text{O}_4$ .

It is concluded from the characterization studies, that the extraction technique which we have employed for the preparation of zeolite-supported iron catalysts results in the formation of highly dispersed, small particle-sized  $\gamma\text{-Fe}_2\text{O}_3$  on the support surface and, in addition, a small amount (~1%) of iron present as a spe-

cies, possibly in a non-oxide form, that interacts strongly with the zeolite support. It is suggested that the strongly interacting iron may be incorporated into the pores of the zeolite. The iron component in the prepared catalysts is readily reduced to the metallic form which in turn forms iron carbides which are active for hydrocarbon synthesis.

Catalytic Evaluation. In order to investigate support effects in these iron/zeolite catalysts prepared from  $\text{Fe}_3(\text{CO})_{12}$  by the extraction technique, three catalysts of similar weight percent iron loading were evaluated for their ability to catalyze synthesis gas conversion; these catalysts were 15.0% Fe/ZSM-5, 16.4% Fe/Mordenite and 15.0% Fe/13X. All catalysts were evaluated under similar conditions as described in the experimental section. Catalytic data is presented in the accompanying figures; in each figure the first three points for each catalyst are data obtained at 280°C, the second three points are data at 300°C.

The catalysts evaluated are active for synthesis gas conversion; the percent conversion of  $\text{H}_2$  and CO is shown for each catalyst in Figure 1 as a function of time under evaluation conditions and temperature. At 280°C the percent conversion of synthesis gas increases with time for the acidic zeolite-supported catalysts, Fe/ZSM-5 and Fe/Mordenite, but decreases for the larger pore, non-acidic zeolite-supported catalyst Fe/13X. The percent conversion increases for all catalysts at 300°C; for Fe/ZSM-5 and Fe/Mordenite the conversions remain constant at this temperature for several days, although for Fe/13X the conversion increases with time. The trends in % synthesis gas conversion, particularly % CO, are reflected in the weight % hydrocarbons, carbon dioxide and water obtained in the reactor effluent over the period of evaluation, see Figure 2. It is apparent that the catalysts are effective for the production of hydrocarbons from synthesis gas, but also catalyze the water gas shift reaction; the % hydrocarbons and %  $\text{CO}_2$  obtained are greater at the higher temperature (300°C) whereas the %  $\text{H}_2\text{O}$  is less at this temperature than at 280°C.

The distribution of the hydrocarbons is presented in Figures 3(a) through (d) and Figures 4(a) and (b) as a weight percent of the total hydrocarbon product. Figure 4(a) represents  $\text{C}_{5+}$  hydrocarbons obtained from the ice-cooled trap and Figure 4(b) the high molecular weight waxy products obtained from the heated trap. All catalysts produce a wide range of hydrocarbon products; the Fe/ZSM-5 catalyst produces the highest yield of methane and the lowest yield of oil of the three catalysts investigated. High methane and low oil yields may be related to the small particle size of the iron-containing species in this catalyst. Carbon monoxide chemisorption data on Fe/Mordenite catalysts have indicated the presence of larger particle sizes for the iron-containing species in these catalysts compared with those in the Fe/ZSM-5 catalysts (13). It has also been observed (14) that for the Fe/ZSM-5 catalyst, calcination in air (300°C and 500°C) leads to an increase in

particle size of the iron oxide which results in decreased methane and increased oil production for these catalysts compared with the as-prepared, uncalcined, catalyst. Simulated distillation data for the oil product ( $C_{5+}$  hydrocarbons) obtained by using Fe/ZSM-5 and Fe/Mordenite catalysts suggest that ~90% of the product boils in the gasoline range ( $<204^{\circ}C$ ) compared with ~70% for the oil obtained by using Fe/13X. The Fe/ZSM-5 and Fe/Mordenite catalysts produce only small amounts of wax whereas Fe/13X produces significant quantities, particularly at  $300^{\circ}C$ . It should be noted that for the Fe/13X catalyst, the increase in % CO conversion at  $300^{\circ}C$  with time is reflected in the increased production of high molecular weight hydrocarbons at this temperature. Thus, for synthesis gas conversion with the acidic zeolite-supported catalysts (Fe/ZSM-5 and Fe/Mordenite) a lower molecular weight distribution of hydrocarbons is obtained compared with that for the non-acidic, larger-pore zeolite-supported catalyst Fe/13X. It is apparent that the pores in the ZSM-5 and mordenite zeolites restrict the formation of high molecular weight hydrocarbon products and/or the presence of Brønsted acid sites in these zeolites results in the cracking of any high molecular weight products which may be formed. However, the 13X zeolite with larger-sized pores and the lack of Brønsted acid sites, does not have the ability to restrict the formation of high molecular weight products and thus a higher molecular weight distribution of hydrocarbons is obtained.

Fluorescence Indicator Absorption (FLA) chromatographic analysis of the  $C_{5+}$  hydrocarbon fraction was conducted on each sample obtained; the results are presented in Figures 5(a) through (c). Comparison of these results for the three catalysts studied shows that the use of the Fe/ZSM-5 catalyst results in the formation of the highest percentage of aromatics for all samples analyzed, for the Fe/Mord catalyst the highest percentage of olefins is obtained and for the Fe/13X catalyst, the highest percentage of saturates is obtained in the oil. The relatively high yield of aromatics for the Fe/ZSM-5 catalyst may be related to the low propene/propane ( $C_3^=/C_3$ ) ratio found by using this catalyst (see Figure 4(c)). This low ratio demonstrates the ability of the Brønsted acid sites in this catalyst to convert propene to aromatic products. It has previously been demonstrated that propene is readily converted into liquid hydrocarbons containing a high percentage of aromatics by the acidic form of ZSM-5 (15). For the Fe/Mordenite catalyst, it is concluded that the acid sites which are present catalyze the oligomerization of light olefins to heavier (liquid) olefins but are not as effective as those in the Fe/ZSM-5 catalyst for the production of aromatic hydrocarbons from propene. The absence of Brønsted acid sites in Fe/13X evidently allows the polymerization of hydrocarbon intermediates to occur resulting in the formation of higher molecular weight saturated hydrocarbons.

The effect of weight percent iron on the catalytic activity of Fe/ZSM-5 and Fe/13X catalysts was investigated by comparison of the catalytic rates obtained for 7.0% Fe/ZSM-5 with that for 15.0%

Fe/ZSM-5 and for 7.0% Fe/13X with that for 15.0% Fe/13X. These data indicate that an increase in the weight percent iron loading results in an increase in the carbon monoxide and hydrogen conversions and an increase in the total amount of hydrocarbons in the effluent. However, there is no appreciable difference in the distribution of the hydrocarbons that are produced.

### Conclusions

This study was initiated in an attempt to produce highly-dispersed, thermally-stable, zeolite-supported metal catalysts and to investigate the effect of acidity and pore size of the zeolite on the products obtained from synthesis gas conversion. As a result of this study, several conclusions can be made.

First, the use of  $\text{Fe}_2(\text{CO})_{12}$  and an extraction technique for the preparation of zeolite-supported iron catalysts results in the formation of highly-dispersed, small particle-sized  $\gamma\text{-Fe}_2\text{O}_3$  on the zeolite surface with a small amount, ~1 Wt% Fe, present in a non-oxide form that interacts strongly with, and may be incorporated into the pores of, the zeolite.

Second, the iron oxide readily forms the iron carbide  $\chi\text{-Fe}_5\text{C}_2$  after reduction in hydrogen and subsequent carburizing in synthesis gas. The thermally stable, long-lived catalysts that are obtained are active for synthesis gas conversion.

Third, the acidity and pore structure of the zeolite support are important factors that influence the distribution of products obtained during synthesis gas conversion. The catalysts Fe/ZSM-5 and Fe/Mordenite contain Brønsted acid sites and medium-sized pores and result in the conversion of synthesis gas to gaseous and liquid hydrocarbons containing a high percentage of unsaturates. Approximately 90% of the liquid (oil) products obtained by using these catalysts are in the gasoline boiling range. The catalyst Fe/13X, which consists of a non-acidic, larger-sized pore zeolite leads to the conversion of synthesis gas to a higher molecular weight distribution of largely saturated hydrocarbons. For this catalyst, approximately 70% of the liquid (oil) product obtained is in the gasoline boiling range.

Fourth, by careful choice of the zeolite support in the preparation of a bifunctional catalyst for synthesis gas conversion, the product distribution, particularly for hydrocarbons, may be controlled. This conclusion is of importance in the design of catalysts for the production of liquid transportation fuels from synthesis gas.



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### Figure Captions

- Figure 1. (a), % H<sub>2</sub> conversion; (b), % CO conversion. ■ = 15.0% Fe/ZSM-5; ● = 16.4% Fe/Mord; ▲ = 15.0% Fe/13X.
- Figure 2. (a), Weight % Hydrocarbons; (b), weight % Carbon Dioxide; (c), weight % water in the reactor effluent.
- Figure 3. (a), Weight % C<sub>1</sub>; (b), weight % total C<sub>2</sub>; (c), weight % total C<sub>3</sub> and (d), weight % total C<sub>4</sub> hydrocarbons of total hydrocarbons produced.

Figure 4. (a), Weight % oil ( $C_{5+}$ ); (b), weight % wax of total hydrocarbons produced; (c), propene/propane ratio for  $C_3$  hydrocarbons.

Figure 5. (a), Weight % aromatics; (b), weight % olefins; (c), weight % saturates in the oil ( $C_{5+}$  hydrocarbon) product.

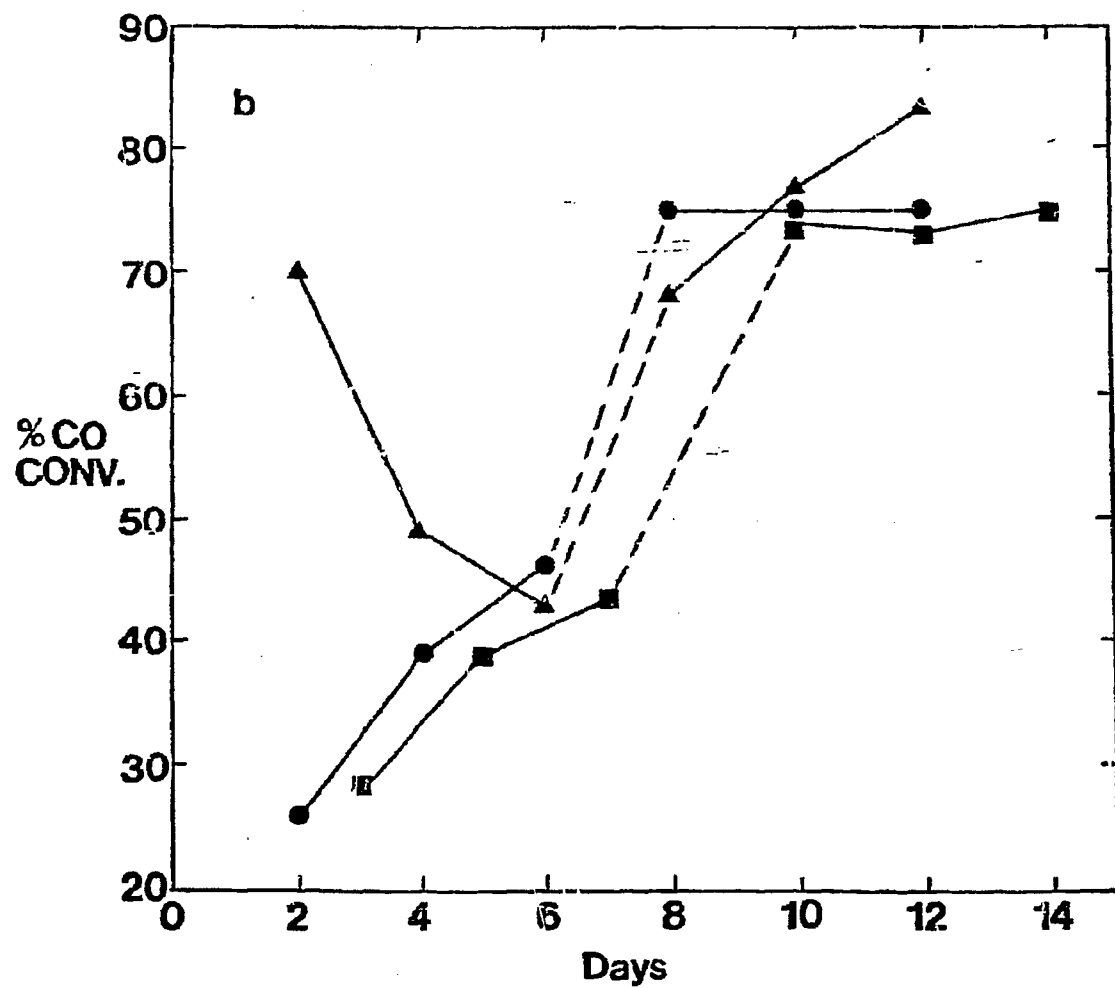
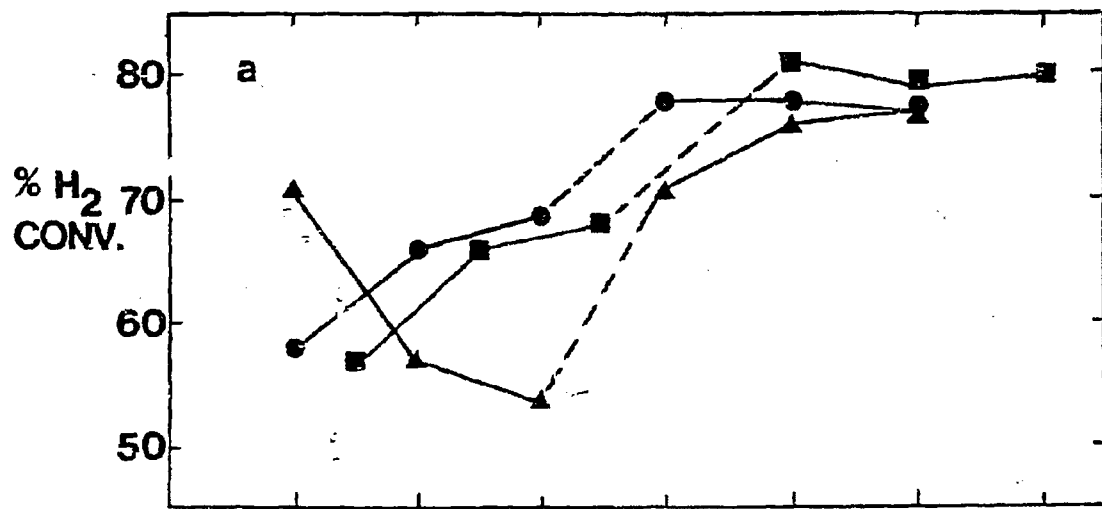


Figure 1

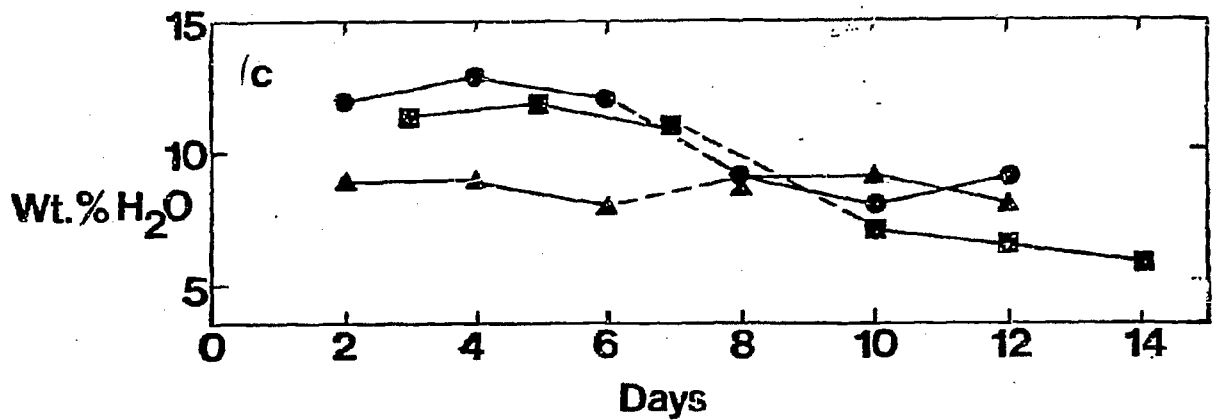
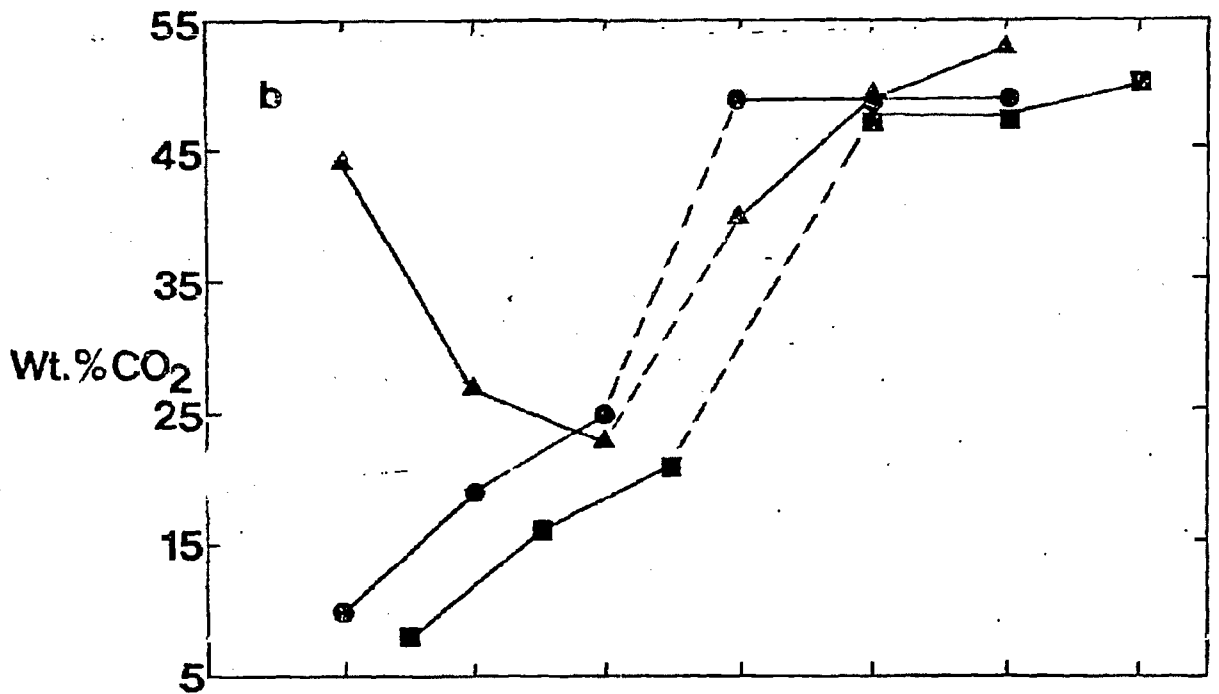
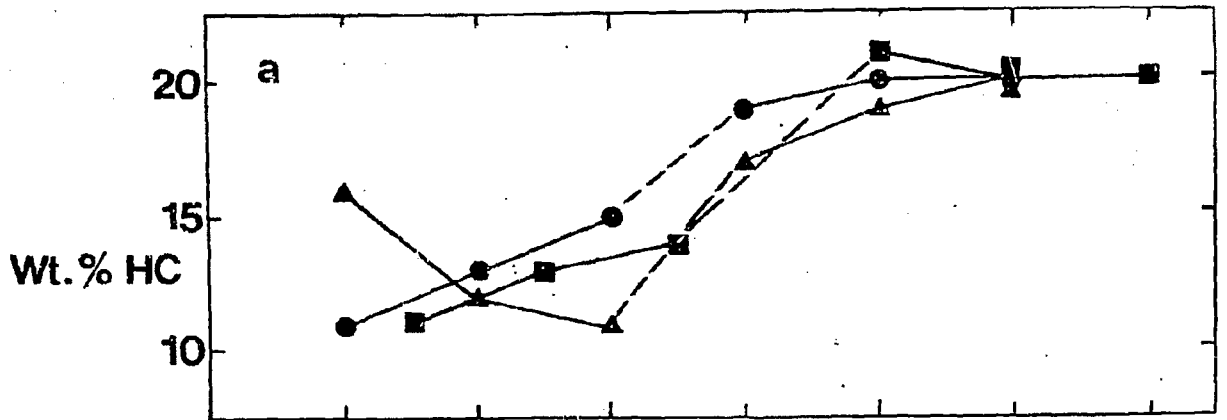


Figure 2

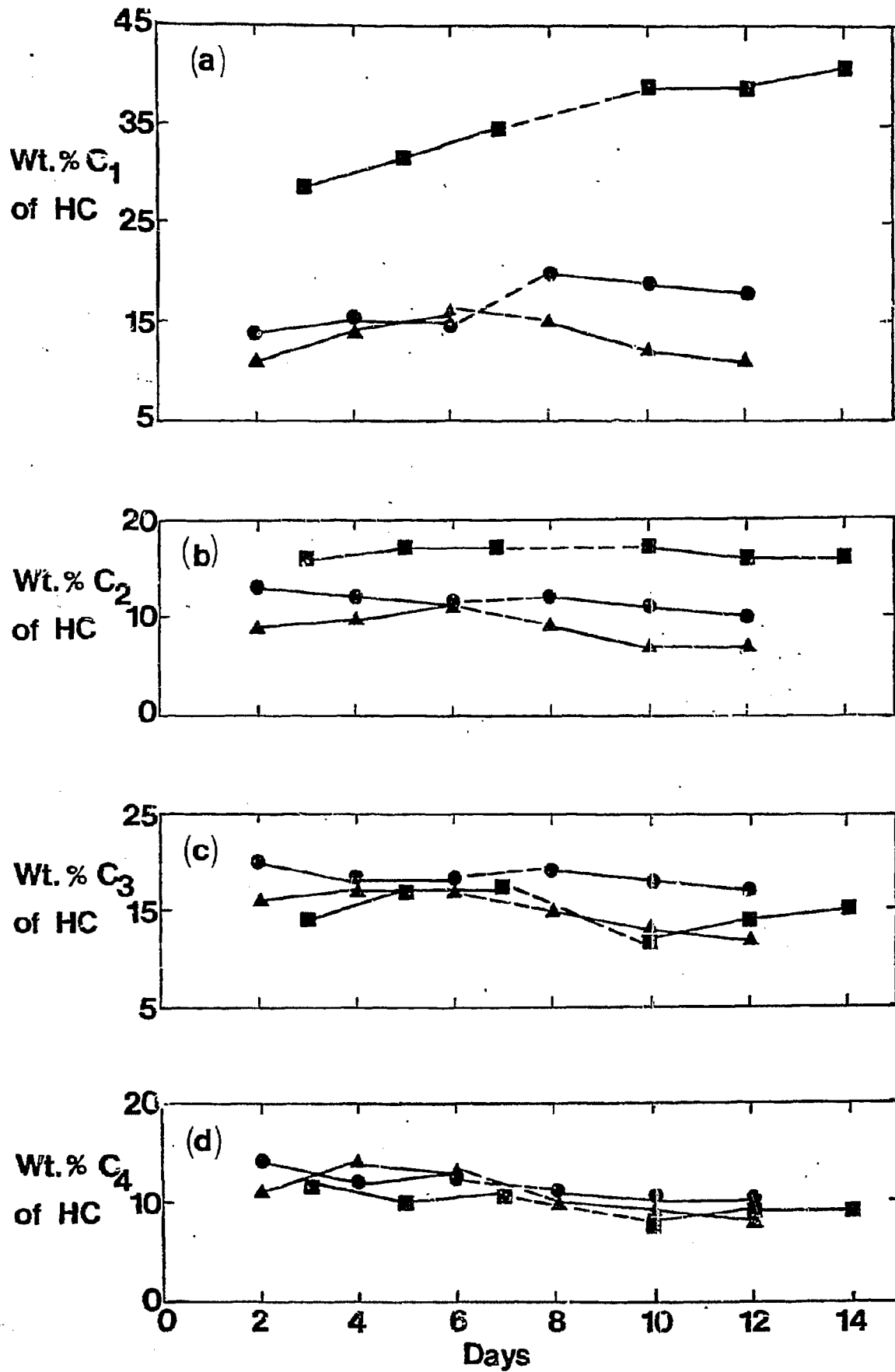


Figure 3

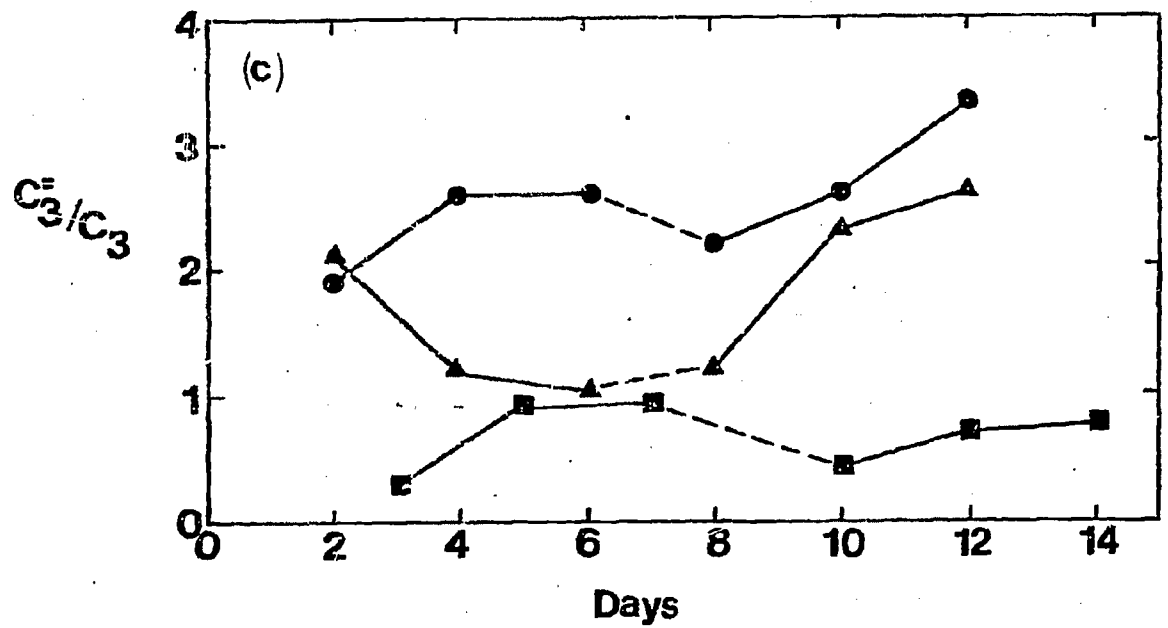
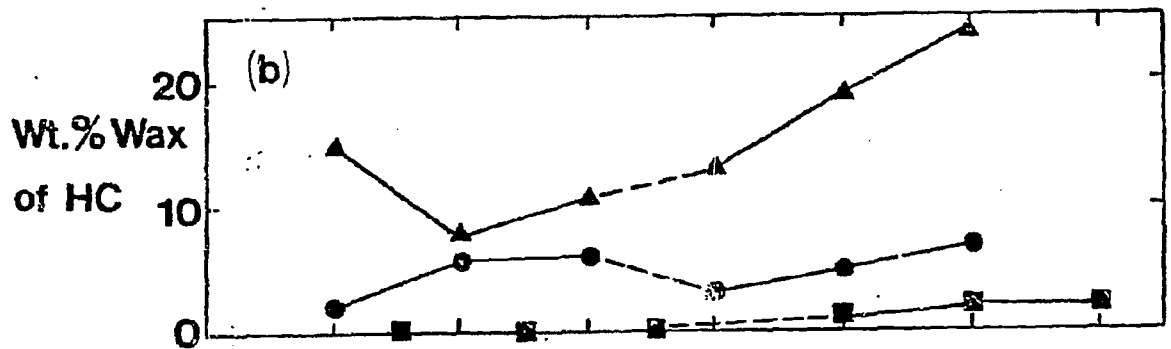
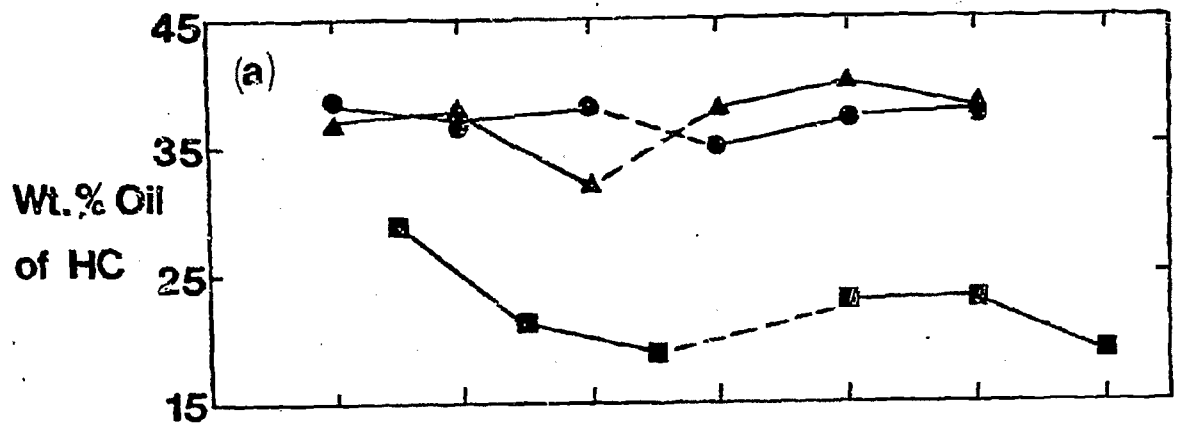


Figure 4

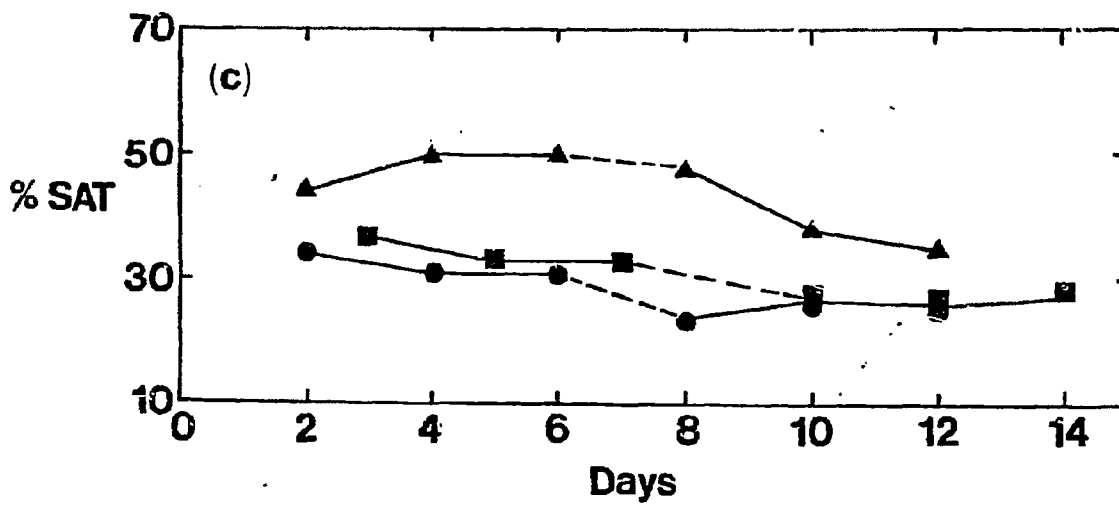
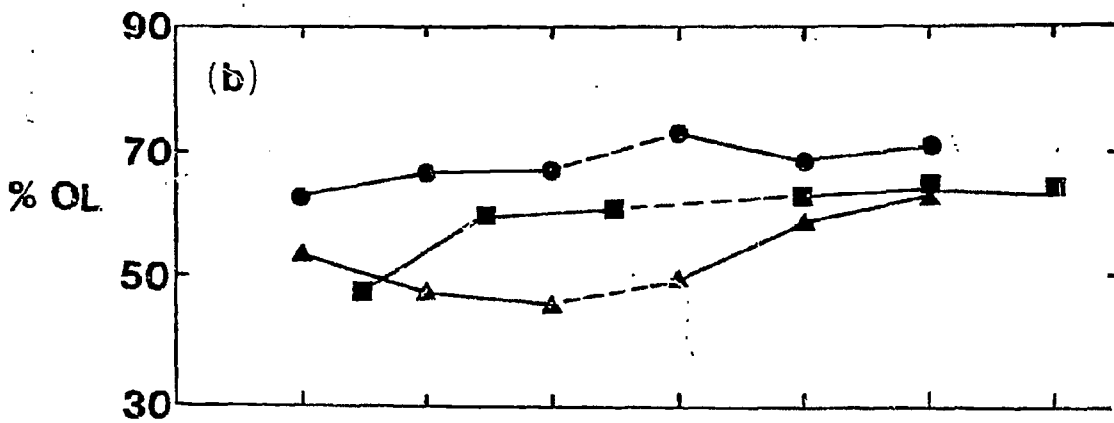
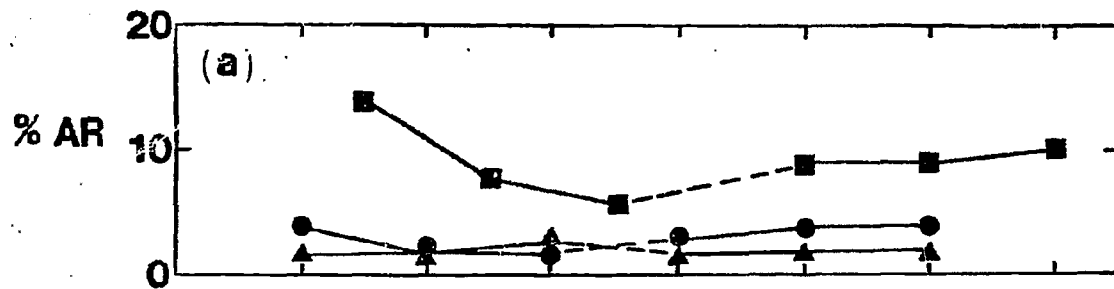


Figure 5

Paper published

"Fe<sub>3</sub>(CO)<sub>12</sub> Impregnated ZSM-5: Characterization and Liquefaction Activity"

Stencel, J. M., Diehl, J. R., Douglas, L. J., Spitler, C. A., Crawford, J. E. and Melson, G. A. Colloids and Surfaces, (1982), 4, 331.

Papers presented

"Preparation, Characterization and Evaluation of Some New ZSM-5 and NU-1 Supported Iron and Cobalt Catalysts for the Production of Gasoline-Range Hydrocarbons from Synthesis Gas"

Crawford, J. E., Melson, G. A., Stencel, J. M. and Gormley, R. J. 7th North American Catalysis Society Meeting, Boston, MA, October 1981, (selected).

"New Catalysts for the Indirect Liquefaction of Coal"

Melson, G. A. U.S. Department of Energy Contractor's Meeting, Pittsburgh, PA, October 1981.

"Catalytic Activity of a Series of Carbonyl-Impregnated Iron/ZSM-5 Catalysts"

Crawford, J. E., Melson, G. A. and Rao, V. U. S. 33rd Southeastern Regional ACS Meeting, Lexington, KY, November 1981.

"Comparison of Ion Scattering Spectrometry Data for Some ZSM-5, Mordenite and 13X Supported Iron Catalysts"

Crites, J. W., Crawford, J. E., Mbadcam, K. J., Melson, G. A., Diehl, J. R. and Stencel, J. M. 33rd Southeastern Regional ACS Meeting, Lexington, KY, November 1981.

"Mössbauer Spectroscopic Investigations of Some Oxide-Supported Iron Catalysts"

Mbadcam, K. J., Melson, G. A., Vaishnava, P. P. and Montano, P. A. 33rd Southeastern Regional ACS Meeting, Lexington, KY, November 1981.

"Evaluation of Some New Zeolite-Supported Metal Catalysts for Synthesis Gas Conversion"

Melson, G. A., Crawford, J. E., Crites, J. W., Mbadcam, K. J., Stencel, J. M. and Rao, V. U. S. 183rd National ACS Meeting, Las Vegas, NV, March 1982 (invited symposium paper).

"Comparison of Nitrate and Carbonyl Impregnated Metals on Oxide Supports"

Stencel, J. M., Diehl, J. R., Spitler, C. A., Mbadcam, K. J. and Melson, G. A. 183rd National ACS Meeting, Las Vegas, NV, March 1982.



Papers presented (continued)

"Fe/ZSM-5 Catalysts: Effect of Particle Size on Activity for the Fischer-Tropsch Reaction"

Crawford, J. E., Melson, G. A. and Stencel, J. M. 21st Annual Spring Symposium, Pittsburgh Catalysis Society, Pittsburgh, PA, May 1982.

"Infrared Studies of Acidic Sites on ZSM-5 Impregnated with Transition Metals"

Rhee, K. H., Stencel, J. M., Rao, V. U. S., Gormley, R. J., Melson, G. A. and Crawford, J. E. 21st Annual Spring Symposium, Pittsburgh Catalysis Society, Pittsburgh, PA, May 1982.

"Modification of Acidity and Activity of ZSM-5 with Incorporation of Transition Metals"

Stencel, J. M., Rao, V. U. S., Rhee, K. H. and Melson, G. A. Tri-State Catalysis Meeting, Ashland, KY, May 1982.

Invited Seminars (G. A. Melson)

"Some New Supported Catalysts for Synthesis Gas Conversion: Characterization and Evaluation"

EXXON Research and Engineering Company, Linden, NJ, September 1981.

"New Fuels from Old: Synthetic Fuels from Coal"

East Carolina University, Greenville, NC, October 1981.

Longwood College, Farmville, VA, February 1982.

Randolph Macon College, Ashland, VA, February 1982.

University of West Florida, Pensacola, FL, March 1982.

University of South Florida, Tampa, FL, March 1982.

"New Supported Metal Catalysts for Synthesis Gas Conversion"

Air Products and Chemicals Inc., Allentown, PA, June 1982.

Degree Awarded (Virginia Commonwealth University)

Ph.D. - Janet E. Crawford. Thesis Title - "Some ZSM-5, NU-1 and Alumina-Supported Metal Catalysts for the Fischer-Tropsch Reaction." (May 1982)