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QUARTERLY TECHNICAL PROGRESS REPORT

Submitted to U.S Department of Energy

GRANT TITLE:	Investigation of Syngas Interaction in alcohol Synthesis Catalysts
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*U.S. / DOE Patent Clearance is not required prior to the publication of this document

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Feb 6, 1996
Date

DISCLAIMER

Quarterly Technical Progress Report
(Period October 1 St. to December 31 St.)

This report presents the work done on " Investigation of Syngas Interaction in Alcohol Synthesis Catalysts" during the last three months. In this report the results of the work done on the effect of CO adsorption on the magnetic character of cobalt in the Cu/Fe/Zn catalysts is discussed.

Introduction: Transition metals copper, cobalt, and iron play a significant role in the conversion of syngas(CO & H_2) to liquid fuels. Several catalytic studies indicate that product selectivity is governed by the inter-metallic ratios and the method of preparation. The catalytic as well as the magnetic character of the catalyst are known to be influenced by the modifications in the metal crystallites. With a view to examine the relations between the magnetic and catalytic properties, a series of Cu/Fe/Zn catalysts were prepared using the co-precipitation method. During this quarter, the magnetic characteristics of the samples before and after adsorption of carbon monoxide and after desorption of CO were investigated.

Experimental Studies: A total of 7 samples were prepared by co-precipitation method. One Molar solutions of copper, Iron and Zinc nitrates are mixed in the proportions needed to obtain the desired cation ratio in the catalyst. By adding one molar solution of sodium hydroxide, the catalyst was precipitated while maintaining neutral pH conditions at a temperature of 80°C. The precipitate was thoroughly washed in warm water, dried at 80°C and calcined in air for 18 hours at 350°C. The calcined sample was reduced in a flowing stream of hydrogen for 18 hours at 350°C. Hydrogen was outgassed while reducing the temperature to 250°C. At this temperature a steady flow of carbon monoxide was introduced for 30 minutes. Continuing the flow of carbon monoxide, the temperature was reduced to 50°C. The sample tube was sealed under partial vacuum and magnetization studies of the catalyst were investigated at Grambling State University.

Results and Discussion:

The saturation magnetization data is shown in Table 1. The following inferences can be drawn from an examination of the magnetic moments of the three ferro magnetic materials and their 3d electronic structures: Fe ($3d^6$ - 218 emu/g), Co ($3d^7$ - 161 emu/g), and Ni ($3d^8$ - 54 emu/g). It seems the magnetic moment decreases in the ferro magnetic materials as the 3d electron density increases.

In all the Iron samples analyzed, the magnetic moment of the Co adsorbed samples is significantly less than the samples with out CO. This is indicative of charge transfer from

carbon monoxide to Iron. Unlike the Cu/Co/Cr (Quarterly Report , October 1995) samples, this feature is independent of Cu/Fe ratio.
The last column in Table 1 represents the magnetization data of the CO desorbed samples. Desorption of CO was done by simply outgassing the samples over night in high vacuum at room temperature. All the samples consistently show a significant further drop of magnetic moment. This feature seems to indicate that a sharing of charge between CO and Iron metal prevails as long as the solid and the gas are in equilibrium. Further decrease in magnetic moment when Co was driven out could result if there occurs an enhancement in the metallic charge distribution.

These features, decrease in the magnetic moment of the adsorbed samples independent of the metal ratio , and a further drop in the moment as carbon monoxide is desorbed is interesting and intriguing. Further study of this phenomena is necessary to understand this unusual behavior.

The FTIR spectrometer has been setup for DRIFT studies. Initial test runs are conducted using transimittance spectroscopy of known substances. A few of the spectra taken on the system are attached.

Future Plans: The present studies indicate a possible charge transfer between the Ferromagnetic metal and carbon monoxide due to the adsorption process. We plan to repeat these experiments to confirm the results both on Cu/Co/Cr and Cu/Fe/Zn samples. FTIR studies of CO adsorbed on cobalt , copper and chromium will be investigated during the next quarter.

Student Training:

One of the objectives of this project is to provide research training for minority undergraduate students at a school with predominant African American enrollment. Out of four new students involved in the project, three are engineering majors and one is a physics major. All these students have learned the catalyst preparation techniques by the three different methods. One of the students worked with Dr. A.N. Murty , the Co-PI , and a Grambling State University physics student involved in the project, received training in the operation of ZFNMR spectrometer and Vibration sample magnetometer during the summer. She acquired the needed skills to collect and analyze the data on the samples investigated. Currently all the four students are getting acquainted with the operation of the

FTIR Spectrometer and learning use of the WIN First software package to run the instrument.

TABLE 1

MAGNETIZATION DATA OF CO ADSORBED AND DESORBED SAMPLES

Magnetic Moment (emu/g)

Sample	Cu/Fe	Cu/Fe/Zn	Cu/Fe/Zn CO-adsorbed	Cu/Fe/Zn CO-desorbed
8-71-21	0.11	223.4	83.5	42.18
17-78-5	0.22	164.6	82.9	79.09
37-37-26	1.00	154.6	78.9	40.48
43-26-31	1.70	204.6	58.8	22.28
22-12-66	1.80	138.3	54.7	9.01
58-29-13	2.00	135.5	66.2	26.19
54-22-24	2.50	142.7	64.5	19.87

FIG 1 FTIR SPECTRA OF ACETONE

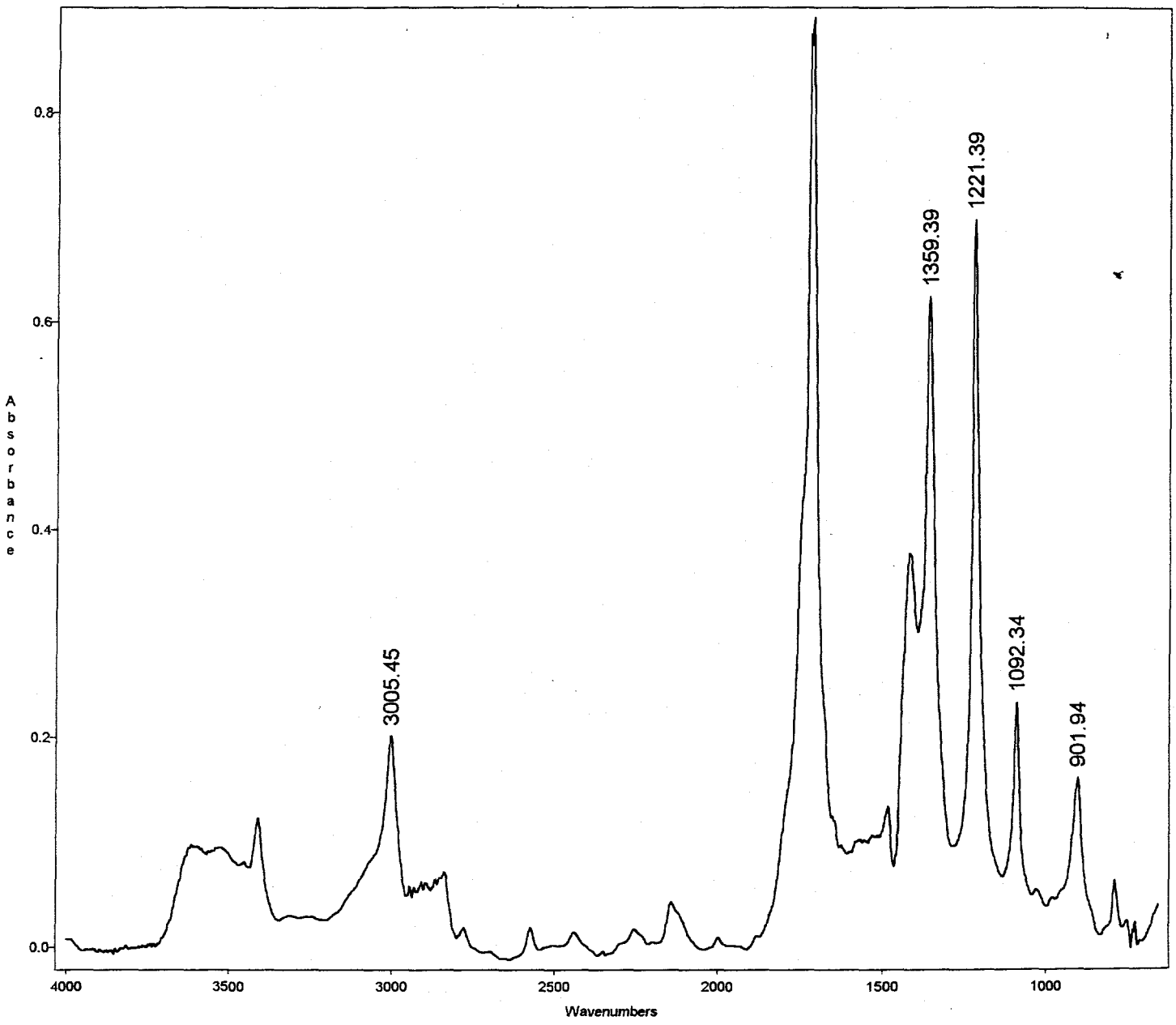

FIG 2

FTIR SPECTRA OF POLYSTYRENE