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SYNTHESIS OF OCTANE ENHANCERS DURING SLURRY-PHASE FISCHER-TROPSCH. QUARTERLY TECHNICAL PROGRESS REPORT NO. 8, JULY 1, 1992--SEPTEMBER 30, 1992

ALTAMIRA INSTRUMENTS, INC.
PITTSBURGH, PA

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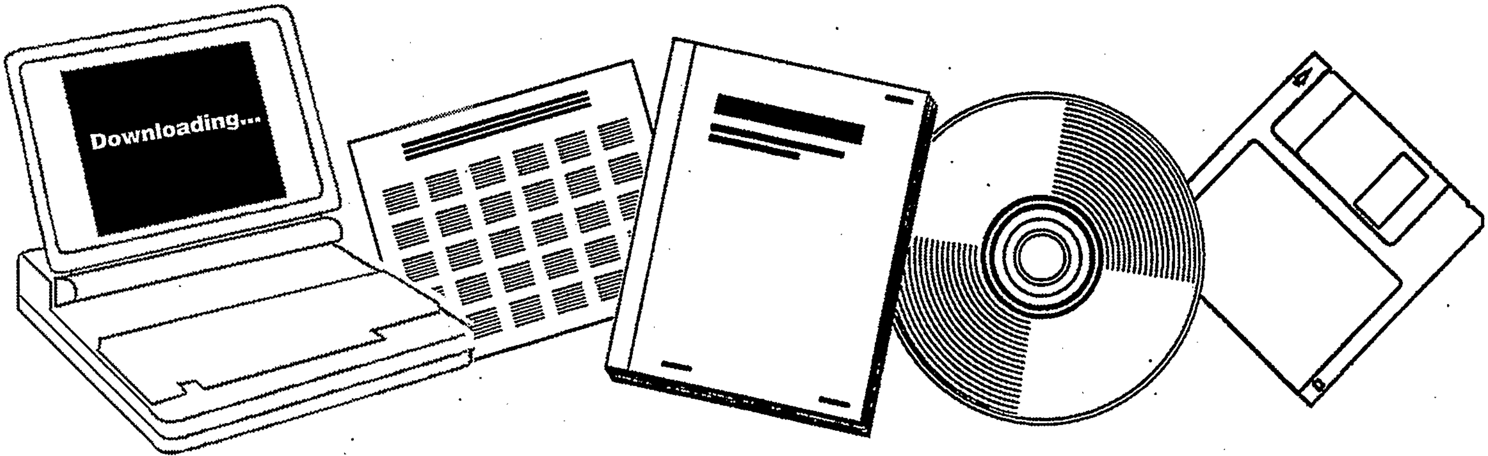

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QUARTERLY TECHNICAL PROGRESS REPORT No. 8

Covering the Period July 1, 1992 to September 30, 1992

SYNTHESIS OF OCTANE ENHANCERS DURING SLURRY-PHASE FISCHER-TROPSCH

Prepared for the U.S. Department of Energy Under
Contract No. DE-AC22-90PC90047

July 7, 1993

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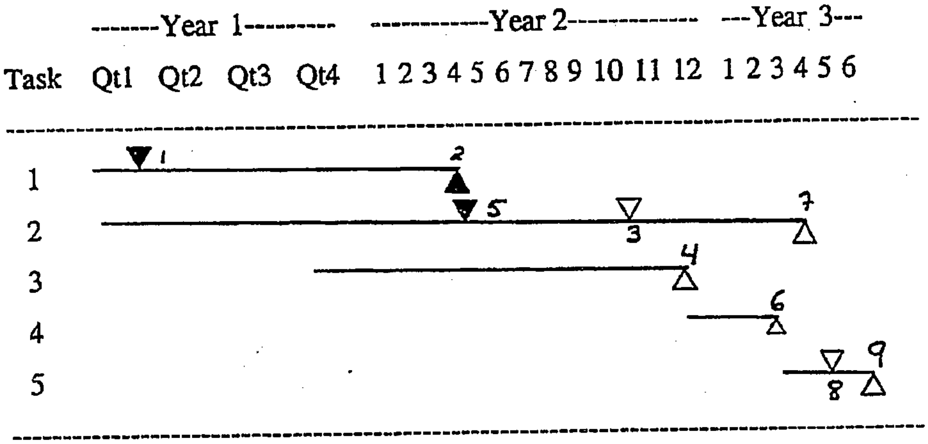
PLANNED TASK AND MILESTONE SCHEDULE



M = denotes milestone

List of Milestones

Number	Description
M1	Finalize design of slurry bubble column reactor (SBCR).
M2	Finalize construction and testing of SBCR. Topical report prepared.
M3	Completion of catalyst screenings for study of i-butylene addition during formation of alcohols. Decision made on catalysts to be studied in SBCR.
M4	Completion of slurry reaction of i-butylene addition during formation of alcohols.
M5	Completion of catalyst screenings for study of i-butylene addition to FT liquid products using acid catalysts. Decision made on catalysts to be studied in SBCR.
M6	Completion of slurry reaction of i-butylene addition to FT liquid products using acid catalysts.
M7	Completion of catalyst screenings.
M8	Completion of slurry reaction of alcohol addition during iso-olefin synthesis.
M9	Final report issued.

OBJECTIVES

The objective of this project is to investigate three possible routes to the formation of ethers, in particular methyl tert-butyl ether (MTBE), during slurry phase Fischer-Tropsch reaction. The three reaction schemes to be investigated are:

- Addition of i-butylene during the formation of methanol and/or higher alcohols directly from CO and H_2 during slurry-phase Fischer-Tropsch.
- Addition of i-butylene to FT liquid products including alcohols in a slurry-phase reactor containing an MTBE or other acid catalyst.
- Addition of methanol to slurry phase FT synthesis making iso-olefins.

WORK ACCOMPLISHED THIS QUARTER

During the eighth quarter we concentrated on examining catalysts for scheme 1, i.e., the addition of iso-butylene during CO hydrogenation. Using both a dual bed arrangement and bifunctional catalysts, we have demonstrated the synthesis of MTBE from syngas and isobutylene.

EIGHTH QUARTER'S TECHNICAL REPORT

1. ETHER SYNTHESIS DURING FT REACTION

1.1 INTRODUCTION

One of the proposed schemes for the direct synthesis of MTBE from syngas involves the incorporation of i-butylene during methanol synthesis from syngas. This incorporation can be envisioned to take place in one of two ways: by interception of a methanol reaction intermediate by i-butylene at a methanol synthesis site; or by secondary reaction of methanol and i-butylene on an acid site. The purpose of this portion of the work is to test each of these approaches to the synthesis of MTBE.

Because the formation of MTBE from methanol and i-butylene is thermodynamically unfavored at high temperatures, we have concentrated our work in low temperature alcohol synthesis catalysts, such as Rh or Pd-based catalysts. Some of these catalysts were promoted with either Mo (to increase the methanol yield) or alkali metals (to reduce the hydrogenation activity). The latter promotion was found necessary to reduce the undesirable hydrogenation of i-butylene to i-butane. Composite catalysts containing acid sites, suitable for secondary synthesis of MTBE, consisted of physical mixtures of the low-temperature methanol synthesis catalyst and a zeolite material.

This section of the report details some of the work in this area.

1.2. EXPERIMENTAL

1.2.1 Preparation of Catalysts

1.2.1.1 Catalysts Active for methanol synthesis

The procedure for the preparation of the Rh and Pd based catalysts active for MeOH synthesis has already been discussed in previous technical reports.

1.2.1.2 Bifunctional Catalyst

PdNaY was chosen as a bifunctional catalyst because of its known activity for MeOH synthesis[1] and for having acid sites which are required for MTBE synthesis. PdNaY was prepared by exchanging the Na^+ in a commercial NaY zeolite (Strem Chemicals) with an aqueous solution of $Pd(NH_3)_4(NO_3)_2$ (Aldrich). After the exchange, the solid was washed several times with deionized water and dried overnight at 60°C. The resulting catalyst was activated by heating in air to 300°C for 5 hours and reducing in flowing H_2 at 300°C for 30 minutes.

1.2.1.3 Composite catalysts

Composite catalysts were prepared by physically mixing the various Li-Pd/SiO_2 catalysts with either an HY zeolite (UOP's LZ210-12) or HZSM-5 (Mobil). The Pd catalyst and zeolite were mixed in a 10:1 ratio. Prior to reaction, the catalyst was treated in-situ by heating in flowing hydrogen at 1°C/minute to 400°C and holding the temperature for 5 hours.

1.2.2 Reaction Studies

The reaction system and experimental conditions used were described in detail in the Seventh Quarterly Technical Report.

1.2.3 Dual Bed Experiments

The reaction was also studied using a dual-bed arrangement of catalysts with the methanol synthesis function and the acid function separated. The dual bed consisted of a bed of 1.0 g of Li-Pd/SiO_2 followed by a bed of 0.10 g of LZ210-12 or ZSM-5 zeolite.

1.3 RESULTS AND DISCUSSION

1.3.1 Elemental Analysis

All the Pd based catalysts used in this study were analyzed for the various metals in their composition using inductively coupled plasma (ICP) spectroscopy. Table 1 shows the weight percent of the various components along with the intended loading and the nomenclature used for referring to these catalysts. The last two columns show the intended and analytical Li/Pd atomic ratio, respectively. The metal loading determined by ICP analysis for most of the catalysts was close to the intended loading except for the Li1/Pd/S1.

Table: 1

Metals loadings from elemenatal analysis

Catalyst Name	Description	Pd (wt%) (Loading)	Pd (wt%) (Analysis)	Li (wt%) (Loading)	Li (wt%) (Analysis)	Li/Pd@ (Atomic)	Li/Pd# (Atomic)
Pd/S1	Pd/SiO_2	5.0	4.36	-	-	-	-
Li1/Pd/S1	Li-Pd/SiO_2	"	"	0.33	1.66	1	5.85
Li4/Pd/S1	Li-Pd/SiO_2	"	"	1.3	1.17	4	4.1
Pd/S2	Pd/SiO_2	"	5.01	-	-	-	-
Li1/Pd/S2	Li-Pd/SiO_2	"	"	.33	.26	1	0.8
Li4/Pd/S2	Li-Pd/SiO_2	"	"	1.3	1.14	4	3.6
PdNaY		"	6.21	-	-	-	-

@ Based on intended loading
\# Based on ICP analysis

1.3.2 CO hydrogenation

1.3.2.1 Synthesis of MeOH During CO Hydrogenation

CO hydrogenation has been studied over all the Pd/SiO_2 and Li-promoted Pd/SiO_2 catalysts described in Table 1. Preliminary results for some of these catalysts were described in the Seventh Quarterly Technical Report. Reaction data for the Pd/S1 and Li1/Pd/S1 and Li4/Pd/S1 are shown in Table 2. In all three cases the main product observed was methanol. Methane and trace amounts of other hydrocarbons were also observed. For all of the catalysts the selectivity for methanol was above 90%. Figure 1 shows the effect of reaction time on the rate of methanol formation over Li-promoted Pd/SiO_2 catalysts, Li1/Pd/S1, and Li4/Pd/S1 at 175°C. For both catalysts their activity increased with time-on-stream and reached steady state after ten to twelve hours. This type of behavior is in agreement with previous reports in the literature[1,2].

1.3.2.2 Synthesis of Branched Hydrocarbons and Methanol During CO Hydrogenation Over a Bi-Functional Catalyst

CO hydrogenation was also carried out over the PdNaY catalyst. Figures 2 and 3 show the rate of MeOH and methane formation, respectively, as a function of time. As in the case of the SiO_2-supported Pd catalysts, this catalyst also required 10-12 hours to reach its highest activity. This catalyst, however, was not as stable as the SiO_2-supported catalysts and its rate for MeOH formation was found to decrease rapidly after it reached its highest level of activity. The initial rate of methane formation was approximately one order of magnitude higher than for the SiO_2-supported catalysts but quickly dropped to a steady-state value. A significant amount of branched hydrocarbons were also produced over this catalyst. An important observation was that trace amounts of MTBE were observed. We believe that this is the first time that MTBE has been observed to form directly from syngas during CO hydrogenation without the addition of i-butylene from an external source over this catalyst. The effluent analysis for this experiment is summarized in Table 3.

Table: 2

Product Distribution for CO Hydrogenation[1]

over Li-Pd/SiO_2

Catalyst	Pd/S1	L1/Pd/S1	Li1/Pd/S4
Reaction Time (hrs)	*	30	24
Temperature °C	170	175	175
Rate (μmol/g.hr)	32.3	54.0	69.3
Conversion (CO %)	0.03	0.2	0.2
<u>Selectivities (mol %)</u>			
MeOH	96.5	90.2	98
C_1	1.7	7.3	1.7
C_2	1.8	2.3	0.3
C_3	-	0.2	-

1. P = 7 atm., H_2/CO = 1
* Initial reaction data

FIGURE 1.

MeOH formation during CO hydrogenation over

Li promoted Pd/SiO_2 catalysts

T = 175 °C, P = 7 atm., $H_2/CO = 1$

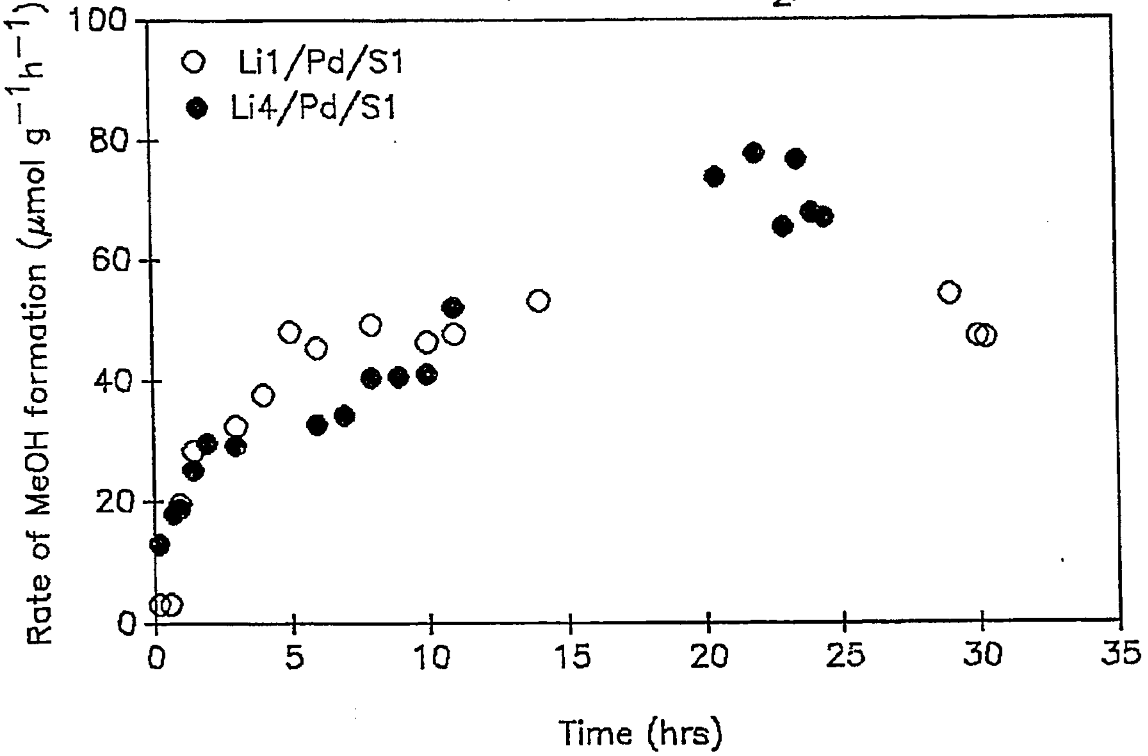


FIGURE 2.
Methane formation during CO Hydrogenation over PdNaY

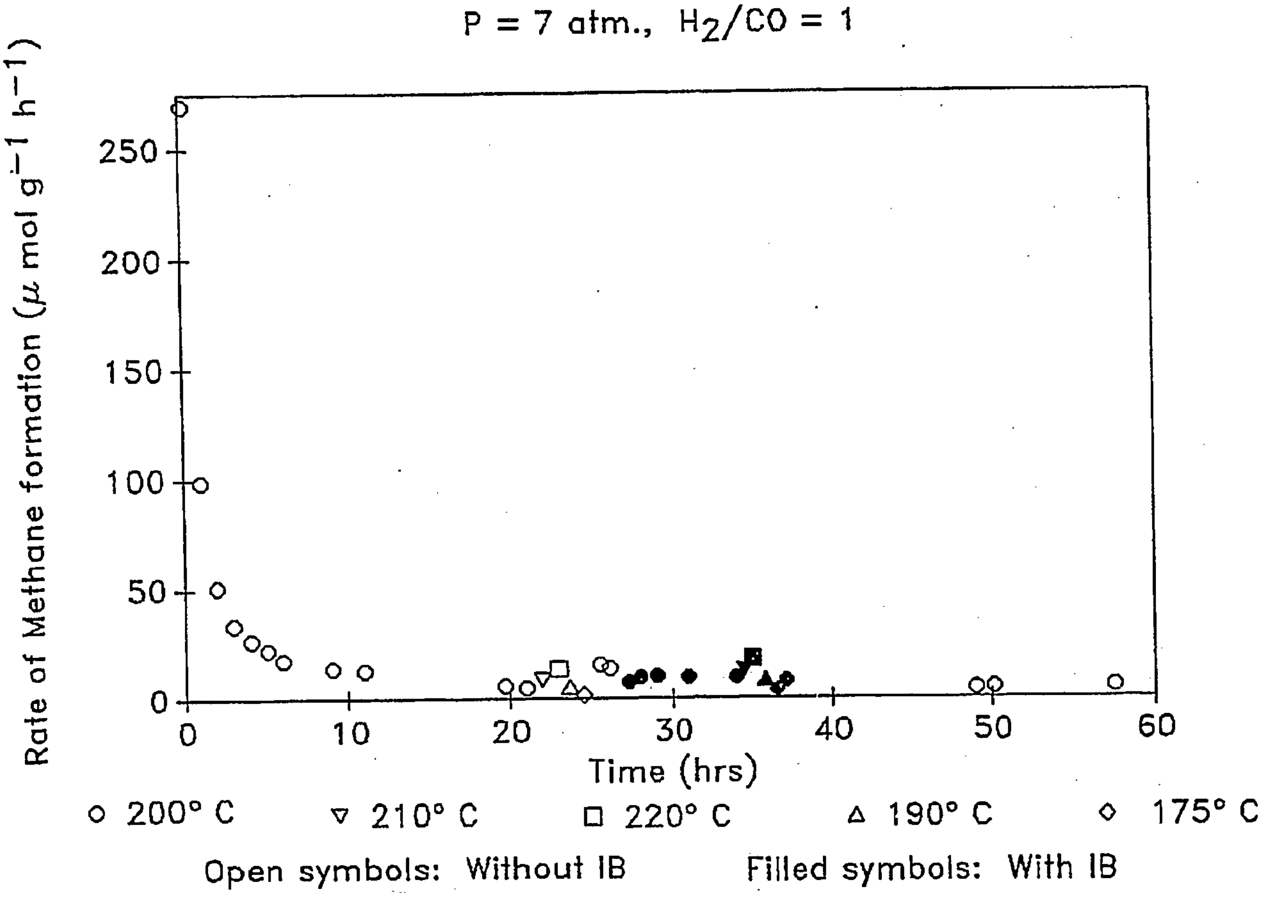
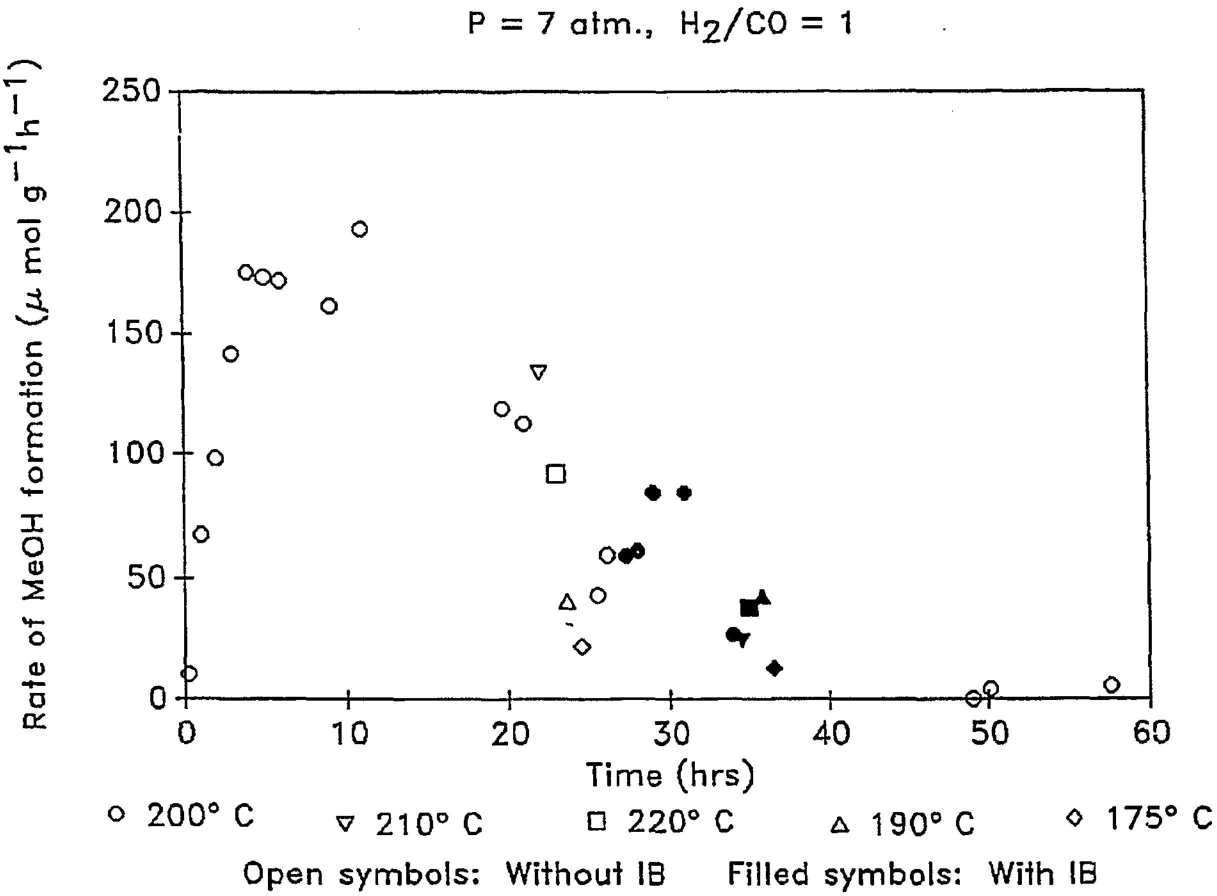


FIGURE 3.
MeOH formation during CO hydrogenation over PdNaY



1.3.3 Addition of i-Butylene During CO Hydrogenation

The addition of i-butylene during CO hydrogenation was studied over the following four different catalysts systems:

i. FTS catalysts active for MeOH synthesis;

ii. Bi-functional catalysts active for MeOH synthesis and having acid sites;

iii. Composite catalysts, i.e., physical mixtures of (i) and an acid zeolite; and

iv. Dual bed arrangements of (i) with an acidic zeolite.

1.3.3.1 Addition of i-Butylene During CO Hydrogenation Over FTS Catalysts

Table 4 summarizes the reaction results for CO hydrogenation in the presence of i-butylene over the Pd/S1 and Li1/Pd/S1 catalysts. The only significant product observed during the addition of i-butylene to the syngas feed over these Pd catalysts was the hydrogenated product isobutane. No MTBE was observed during the reaction. Figure 4 shows the effect of addition of i-butylene on the rate of MeOH formation over Li1/Pd/S2 at steady-state conditions. Upon addition of i-butylene, a sudden decrease in the activity for MeOH formation was noted leading to a new steady-state. The Arrhenius plot for this experiment is shown in Figure 5.

It is likely that the decrease in the rate of MeOH formation with the addition of i-butylene is due to the i-butylene blocking some of the active sites for methanol synthesis. Alternatively, it may be due to the fact that the hydrogenation reaction consumed some of the surface H_2, resulting in lower H_2/CO ratio at the catalyst surface. Figure 6 summarizes the results for the initial rate (10 min) for the hydrogenation of i-butylene to i-butane during CO hydrogenation over catalysts Pd/S1 and Li1/Pd/S1. It can be seen that Li promotion significantly decreased the hydrogenation activity of the catalyst resulting in a lower rate of i-butylene hydrogenation as compared to the unpromoted Pd/SiO_2 catalysts. In spite of this lowered hydrogenation activity, however, no interception of the methanol intermediate by the i-butylene, resulting in MTBE, was observed. This further suggests that the metal sites are not capabie of carrying out the etherification reaction.

Table No: 3

Effluent Analysis during CO Hydrogenation[1] over PdNaY

	Before IB addition	After IB addition
Reaction Time (hrs)	26.1	34
Conversion (CO %)	0.49	-
Rate of CO conversion (μmol/g.hr)[2]	175.7	-
Rate of MTBE formation (μmol/g.hr)[2]	-	1.7
Relative molar conc. (mol %)		
C_1	14.0	1.5
C_2	6.2	0.7
C_3	4.9	2.7
C_4	1.4	4.2
I-Butane	5.4	62.0
I-Butene	2.6	9.2
C_5	4.2	10.2
C_6	1.6	3.6
C_7	0.9	1.6
C_8	-	-
MeOH	58.8	4.0
DME	0.0	0.0
MTBE	Traces	0.3

1. T = 200 °C, P = 7 atm., $H_2/CO = 1$
2. Based on per gram of Li-Pd/SiO_2

Table No: 4

Effluent analysis during CO hydrogenation[1] over Pd/S1 and Li1/Pd/S1 before and after iso-butylene addition

Catalyst	Pd/S1		Li1/Pd/S1	
	Before	After	Before	After
Rate of CO conversion (μmol/g.hr)	32.3	-	45	-
CO conversion (%)	0.03	-	0.05	-
<u>Relative Molar Conc. (mol%)</u>				
MeOH	96.5	7.8	96.9	13.7
C_1	1.7	0.2	2.7	0.3
C_2	1.8	0.3	0.4	0.2
Iso-butane	-	47.7	-	6.3
Iso-butene	-	44	-	79.5

[1] T = 17 °C, P = 7 atm, $H_2/CO = 1$

FIGURE 4.

MeOH formation over Li1/Pd/S2

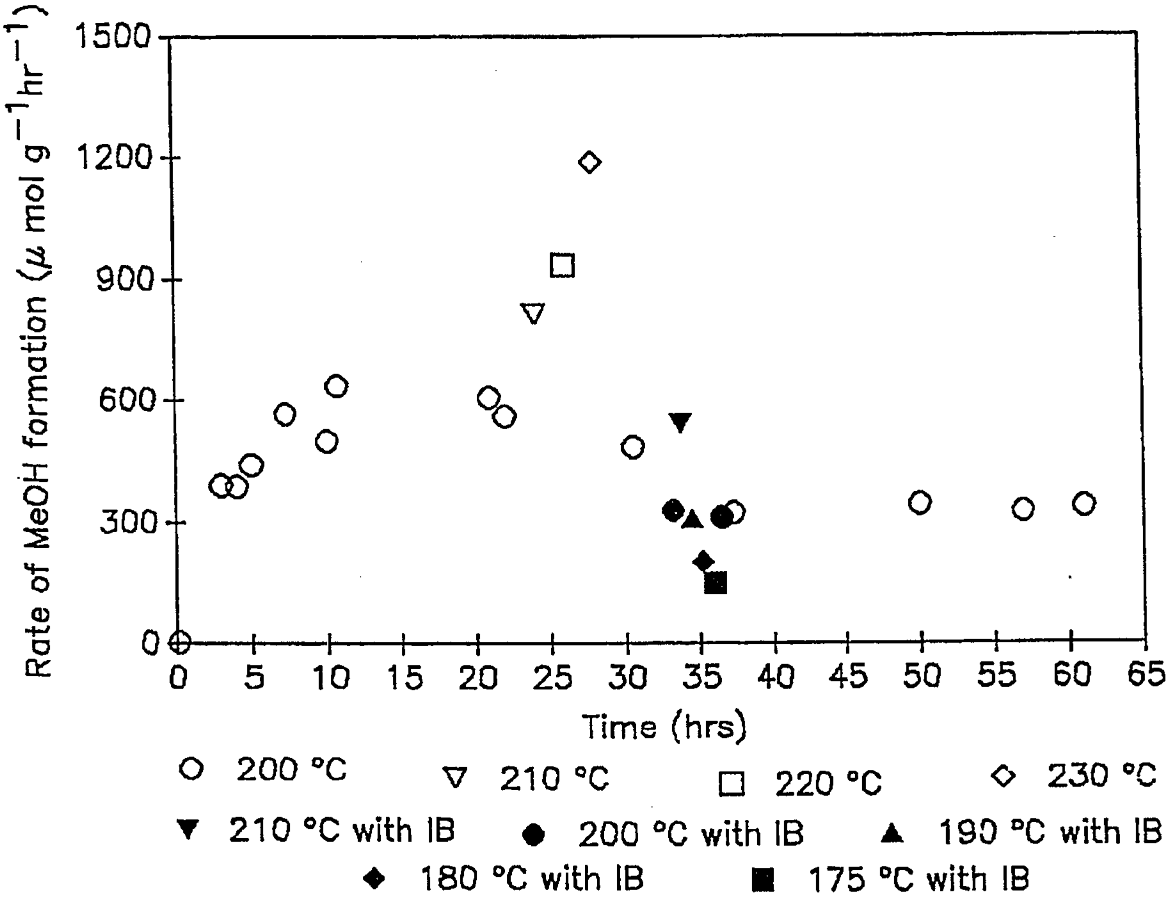


FIGURE 5.

Arrhenius Plot for CO hydrogenation over Li1/Pd/S2

$H_2/CO = 1$,   P = 7 atm.

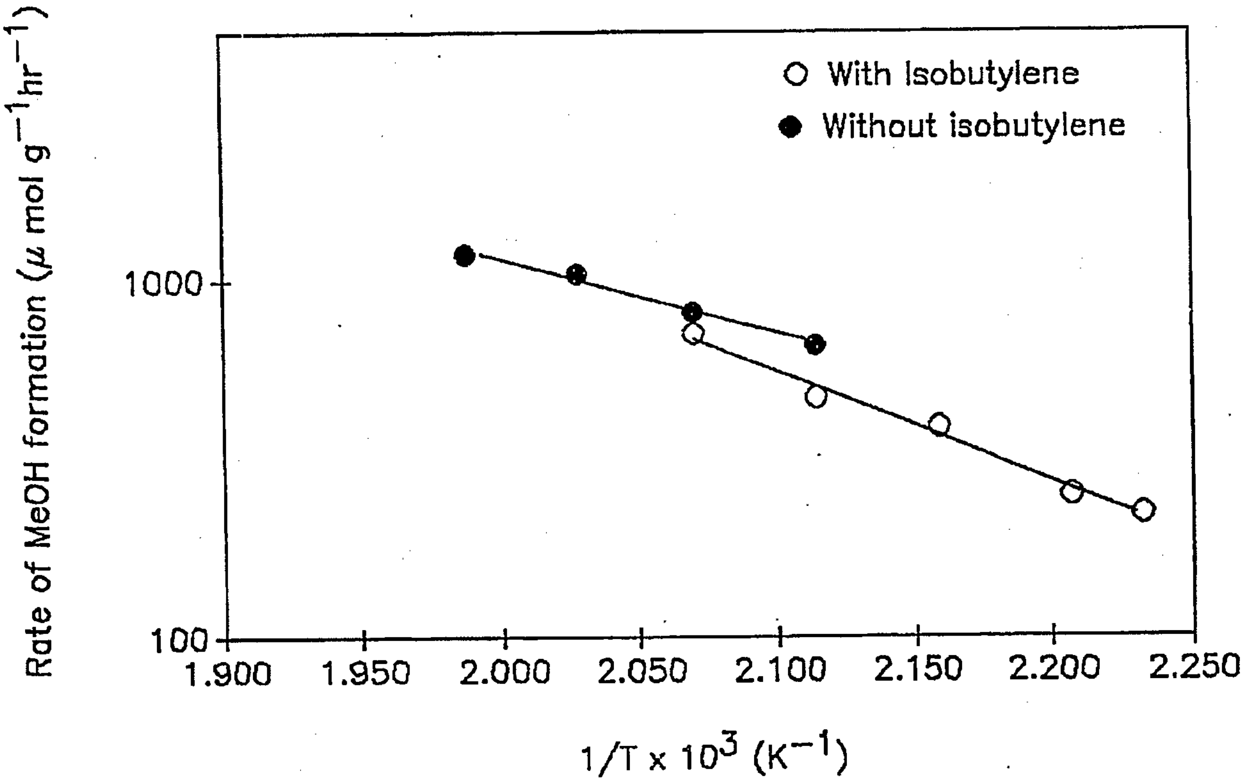
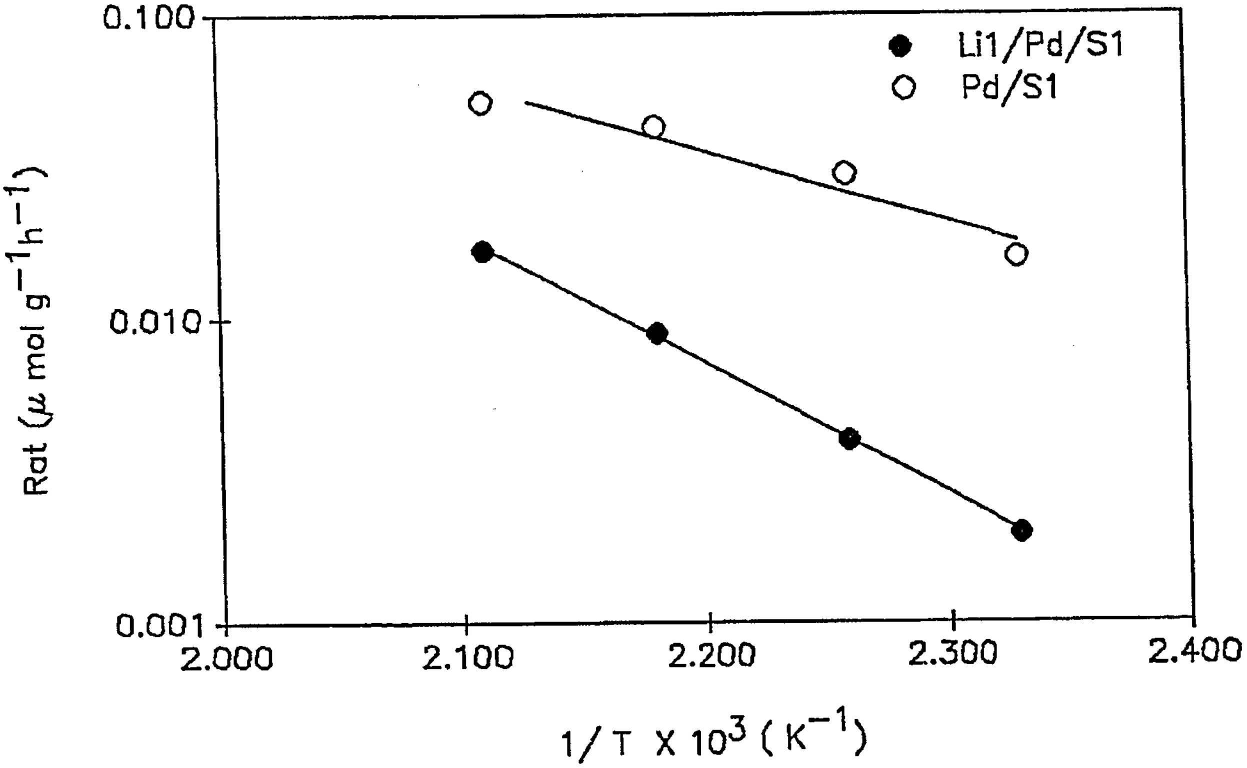


FIGURE 6.
Arrhenius Plot for hydrogenation of isobutylene in the presence of CO and H_2



1.3.3.2 Addition of i-Butylene During CO Hydrogenation Over a Bi-functional Catalyst

Iso-butylene was added during CO hydrogenation over the PdNaY catalyst. Figure 7 shows the rate of MTBE formation with time on stream after the addition of i-butylene. The rate of MTBE formation gradually increased after the addition of i-butylene. After the temperature reached 210°C, the catalyst was noted to deactivate with a corresponding decrease in the rate of MTBE formation. This trend suggests that at above 200°C this catalyst is not as stable as Pd/SiO_2.

1.3.3.3 Addition of i-Butylene During CO Hydrogenation Over Composite Catalysts (Physical Mixtures)

Table 5 summarizes the effluent analysis for a composite catalyst consisting of $Li/Pd/SiO_2$ (Li1/Pd/S1) and LZ210-12 (HY) zeolite before and after i-butylene addition. Figure 8 shows the rate of MTBE formation with time on stream after i-butylene addition for the same catalyst. The MTBE formation rate decreased rapidly after i-butylene addition and fell to nearly zero after 10 hours.

A similar study was done using ZSM-5 zeolite. Table 6 summarizes the effluent analysis during CO hydrogenation over this composite catalyst. In this case, the rate of MTBE formation was much higher than the one observed during experiments using Li1/Pd/S1 and LZ210-12. Figures 9 and 10 show the rate of MTBE and MeOH formation over this catalyst. This catalyst was found to be much more active, and unlike Li1/Pd/S1 + LZ210-12, it did not deactivate at higher temperatures.

1.3.3.4 Addition of i-Butylene During CO Hydrogenation Over a Dual-Bed Catalyst Arrangement

The results for the experiments utilizing the dual bed of LZ210-12 and Li1/Pd/S1 are summarized in Table 7. Figure 11 shows the effect of time-on-stream on the rate of MeOH formation. The results of this experiment are hard to understand as the rate of MeOH formation actually increased with the addition of i-butylene. This is the opposite effect to what was previously observed with the Li-Pd/SiO_2. Interestingly, in this

FIGURE 7.

MTBE formation during CO hydrogenation over PdNaY

P = 7 atm., $H_2/CO = 1$

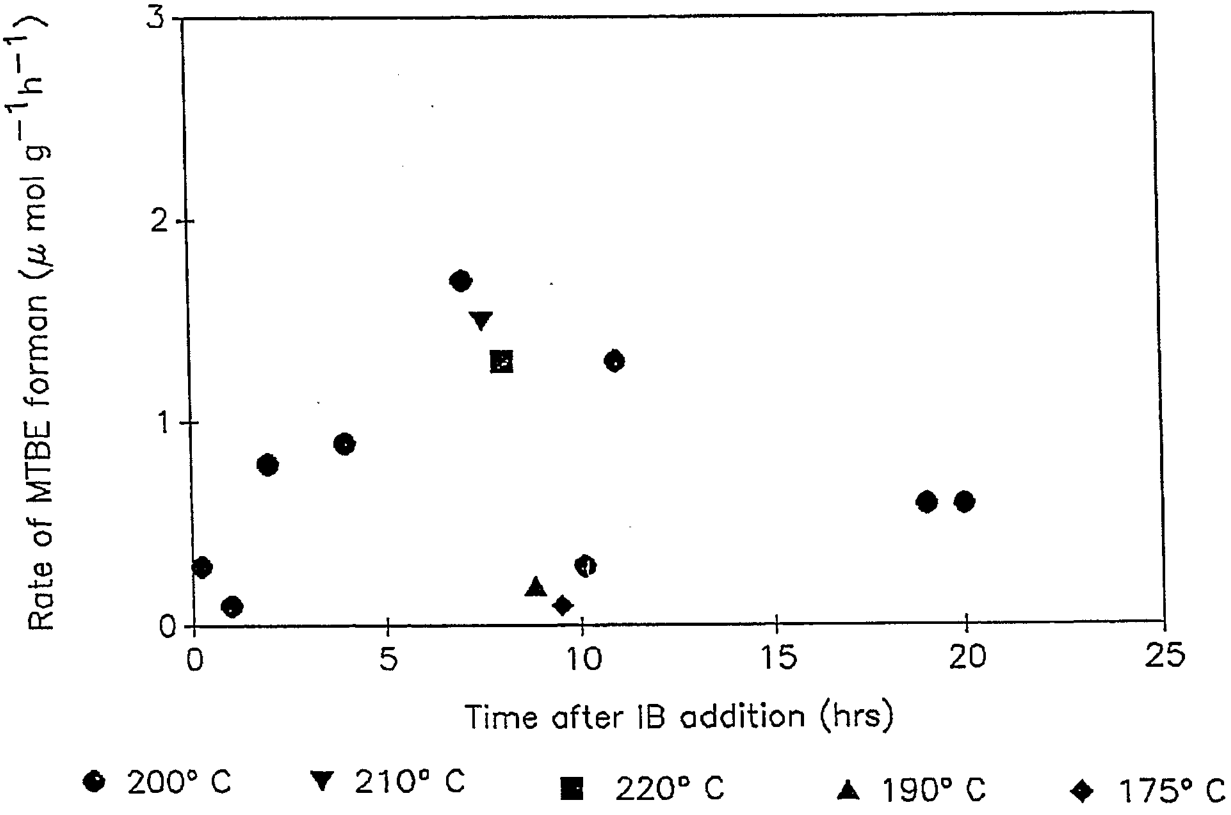


Table No: 5

Effluent Analysis during CO Hydrogenation[1] over

Li1/Pd/S1 + LZ210-12 (10:1) Physical Mixture

	Before IB addition	After IB addition
Reaction Time (hrs)	22	24.8
Conversion (CO %)	0.02	-
Rate of CO conversion (μmol/g.hr)[2]	9.15	-
Rate of MTBE formation (μmol/g.hr)[3]	-	2.2
<u>Relative molar conc. (mol %)</u>		
C_1	22.1	0.2
C_2	4.0	0.1
C_3	-	0.1
C_4	-	0.2
I-Butane	-	6.3
I-Butene	-	88.3
C_5	-	0.2
C_6	-	0.1
C_7	-	-
C_8	-	3.8
MeOH	55.7	0.8
DME	18.2	-
MTBE	-	0.02

1. T = 175 °C, P = 7 atm., $H_2/CO = 1$
2. Based on per gram of Li-Pd/SiO_2
3. Based on per gram of Zeolite

FIGURE 8.

MTBE formation during CO hydrogenation over
Li1/Pd/S1 + LZ210–12 (Physical mixture)
P = 7 atm., $H_2/CO = 1$

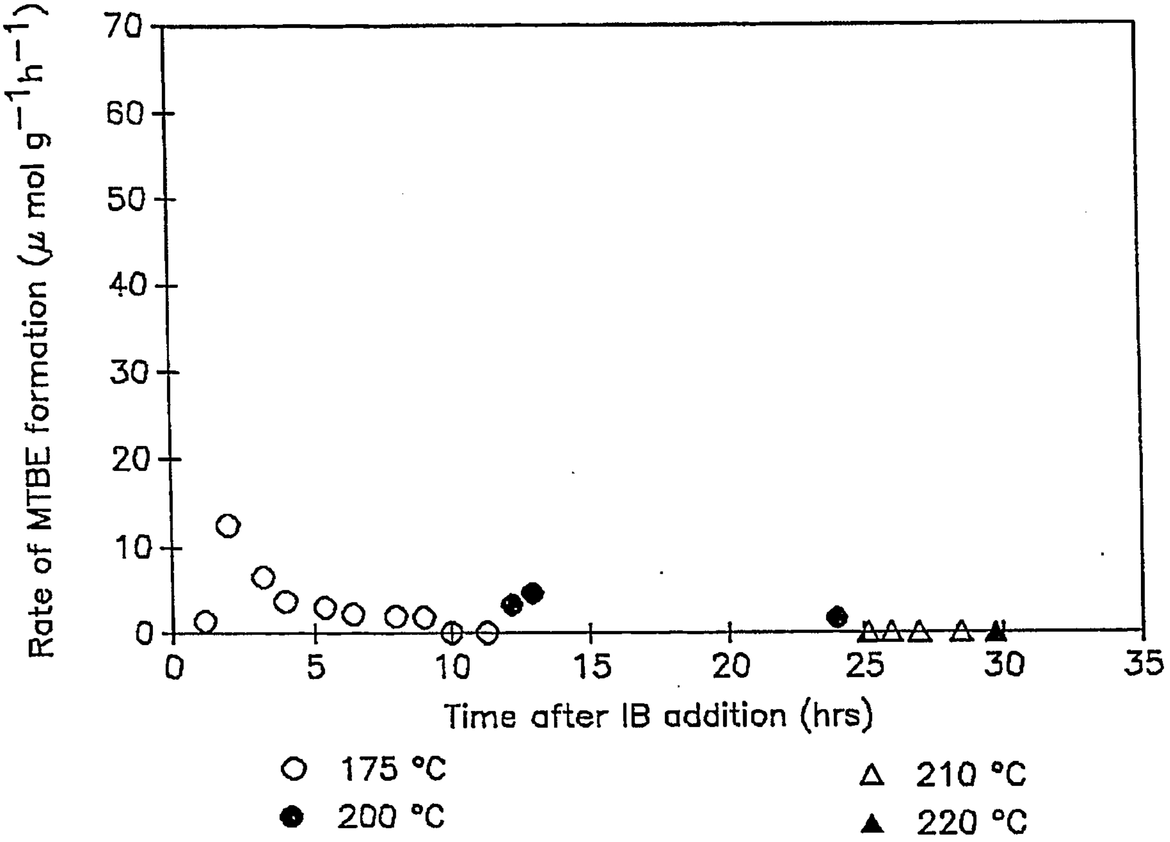


Table No: 6

Effluent Analysis during CO Hydrogenation[1] over Li1/Pd/S1 + ZSM-5 (10:1) Physical Mixture

	Before IB addition	After IB addition
Reaction Time (hrs)	21.6	28
Conversion (CO %)	0.03	-
Rate of CO conversion (μmol/g.hr)[2]	12.65	-
Rate of MTBE formation (μmol/g.hr)[3]	-	17.9
Relative molar conc. (mol %)		
C_1	12	0.2
C_2	4.1	0.1
C_3	1.4	0.4
C_4	-	1.1
I-Butane	-	58.4
I-Butene	-	4.7
C_5	-	3.3
C_6	-	0.9
C_7	-	1.3
C_8	-	27.2
MeOH	82.5	2.1
DME	-	-
MTBE	-	0.2

1. T = 175 °C, P = 7 atm., $H_2/CO = 1$
2. Based on per gram of Li-Pd/SiO_2
3. Based on per gram of Zeolite

FIGURE 9.

MTBE formation during CO hydrogenation over

Li1/Pd/S1 + ZSM-5 (Physical Mixture)

P = 7 atm., $H_2/CO = 1$

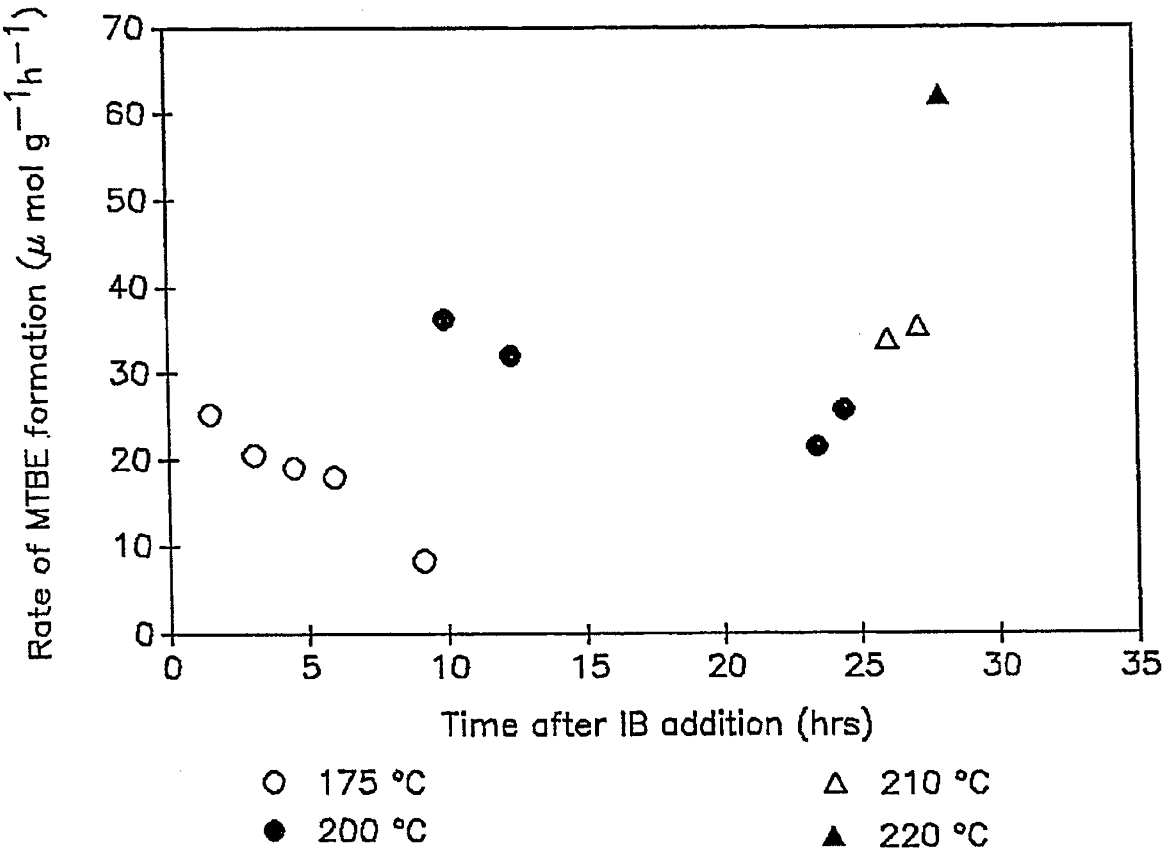
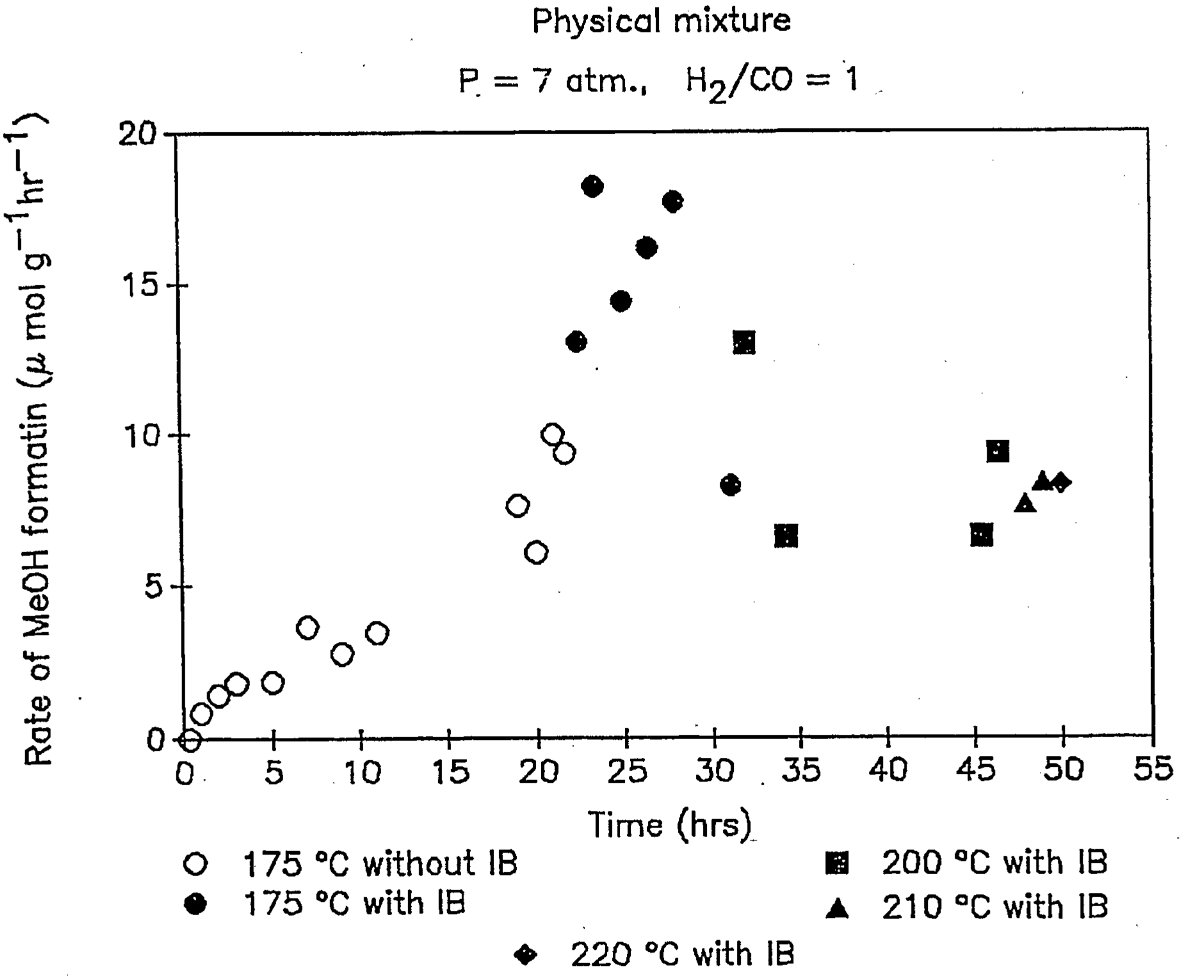


FIGURE 10.

MeOH formation over L1/Pd/S1 + ZSM−5 (Physical Mixture)



experiment measurable amounts of MTBE were formed only after 2.5 hours of i-butylene addition to the syngas.

Another experiment using ZSM-5 instead of LZ210-12 was conducted using a dual bed arrangement. Table 8 gives a summary of the results before and after i-butylene addition. Figures 12 and 13 show the effect of time on stream on the rate of MTBE and MeOH formation, respectively. These results are similar to those obtained with the composite catalyst. The rate of MTBE formation decreased sharply after addition of i-butylene and reached a steady-state after 5 hours. When the temperature was increased to 200°C the rate of MTBE formation increased again. At this temperature, rates were comparable those observed with similar catalysts in a composite arrangement.

1.4 SUMMARY

The initial work on the synthesis of MTBE during CO hydrogenation shows that MTBE cannot be formed directly on metal sites and likely requires the presence of an acid site. However, MTBE can be made successfully when an acid site, provided by the zeolites, is present in the vicinity of the methanol-synthesis metal sites. When i-butylene was added during CO hydrogenation over a composite catalyst consisting of Li-Pd/SiO_2 and a hydrogen-zeolite, MTBE was formed in measurable amounts. The major by-product of this reaction scheme was isobutane and the dimer of i-butylene. In general, ZSM-5 was found to be superior to LZ210-12 HY zeolite.

CO hydrogenation over a bifunctional PdNaY catalyst shows that branched hydrocarbons as well as MeOH can be made successfully at the same time. Addition of i-butylene over this catalyst only (i.e. without other zeolite) results in the formation of trace amounts of MTBE.

1.5 REFERENCES

1. Cavalcanti, F.A.P., Dossi, C., Shen, L.L., and Sachtler, W.M.H., Catal. Lett., 6, 289 (1990).
2. Shen, Y., Wang, S., and Huang, K., Appl. Catal. 57, 55 (1990).

Table No: 7

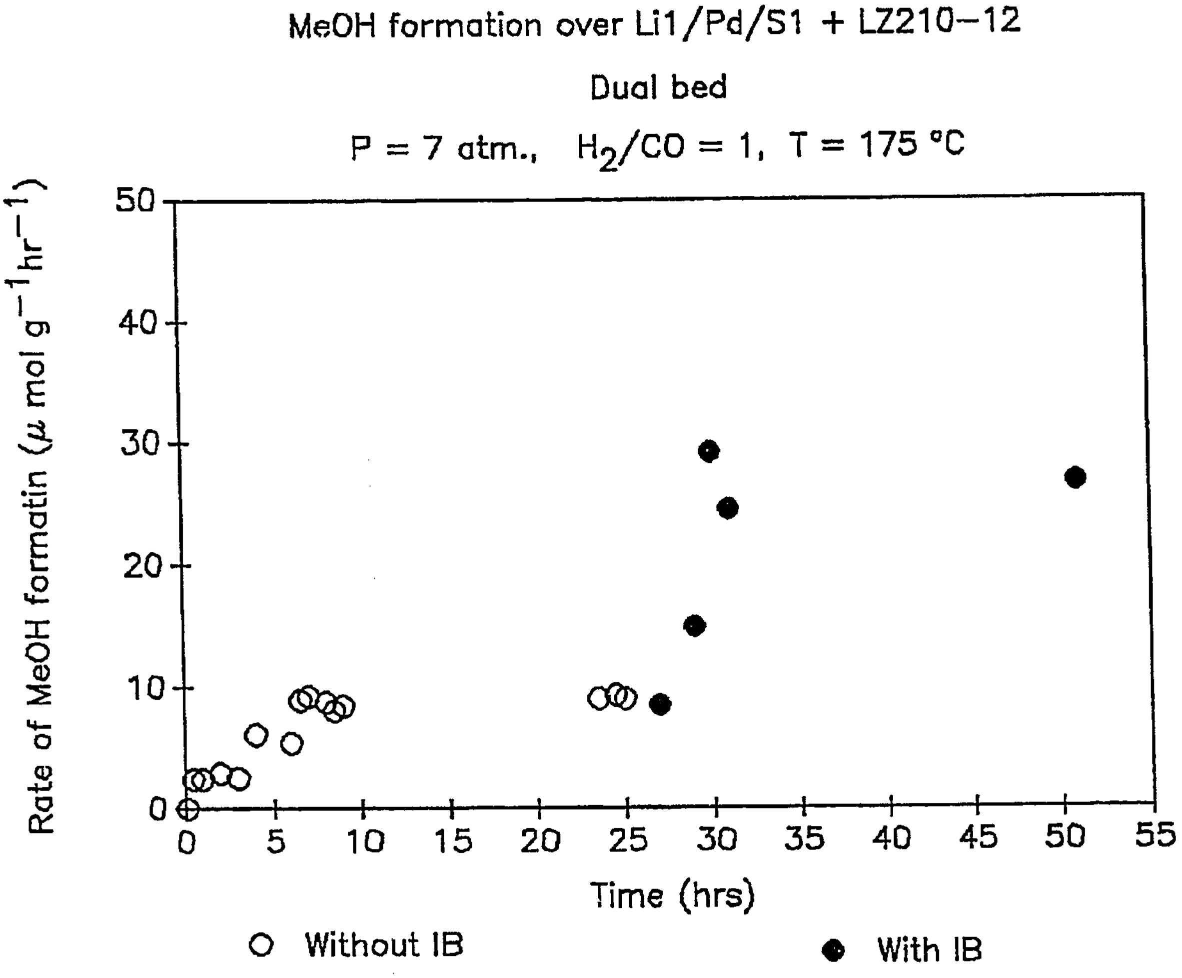
Effluent Analysis during CO Hydrogenation[1] over

Li1/Pd/S1 + LZ210-12 (10:1) Dual Bed

	Before IB addition	After IB addition
Reaction Time (hrs)	25	29
Conversion (CO %)	0.06	-
Rate of CO conversion (μmol/g.hr)[2]	28.4	-
Rate of MTBE formation (μmol/g.hr)[3]	-	5.3
Relative molar conc. (mol %)		
C_1	32	0.6
C_2	5.0	0.2
C_3	2.1	0.2
C_4	-	0.2
I-Butane	-	35.9
I-Butene	-	56.7
C_5	-	0.4
C_6	-	0.3
C_7	-	0.3
C_8	-	3.7
MeOH	41.1	1.15
DME	19.9	0.2
MTBE	-	0.01

1. T = 175 °C, P = 7 atm., $H_2/CO = 1$
2. Based on per gram of Li-Pd/SiO_2
3. Based on per gram of Zeolite

FIGURE 11.



MeOH formation over Li1/Pd/S1 + LZ210-12

Dual bed

P = 7 atm., $H_2/CO = 1$, T = 175 °C

Table: 8

Effluent Analysis during CO Hydrogenation[1] over Li1/Pd/S1 + ZSM-5 (10:1) Dual Bed

	Before IB addition	After IB addition
Reaction Time (hrs)	21	28.5
Conversion (CO %)	0.05	-
Rate of CO conversion (μmol/g.hr)[2]	18.2	-
Rate of MTBE formation (μmol/g.hr)[3]	-	4.3
Relative molar conc. (mol %)		
C_1	32.3	0.4
C_2	9.6	0.2
C_3	4.7	0.5
C_4	1.8	0.9
I-Butane	-	10.0
I-Butene	-	73.3
C_5	3.0	0.7
C_6	4.3	0.4
C_7	-	0.2
C_8	-	13.1
MeOH	44.3	0.2
DME	-	-
MTBE	-	0.1

1. T = 175 °C, P = 7 atm., $H_2/CO = 1$
2. Based on per gram of Li-Pd/SiO_2
3. Based on per gram of Zeolite

FIGURE 12.

MTBE formation during CO hydrogenation over Li1/Pd/S1 + ZSM
Dual Bed
P = 7 atm., $H_2/CO = 1$

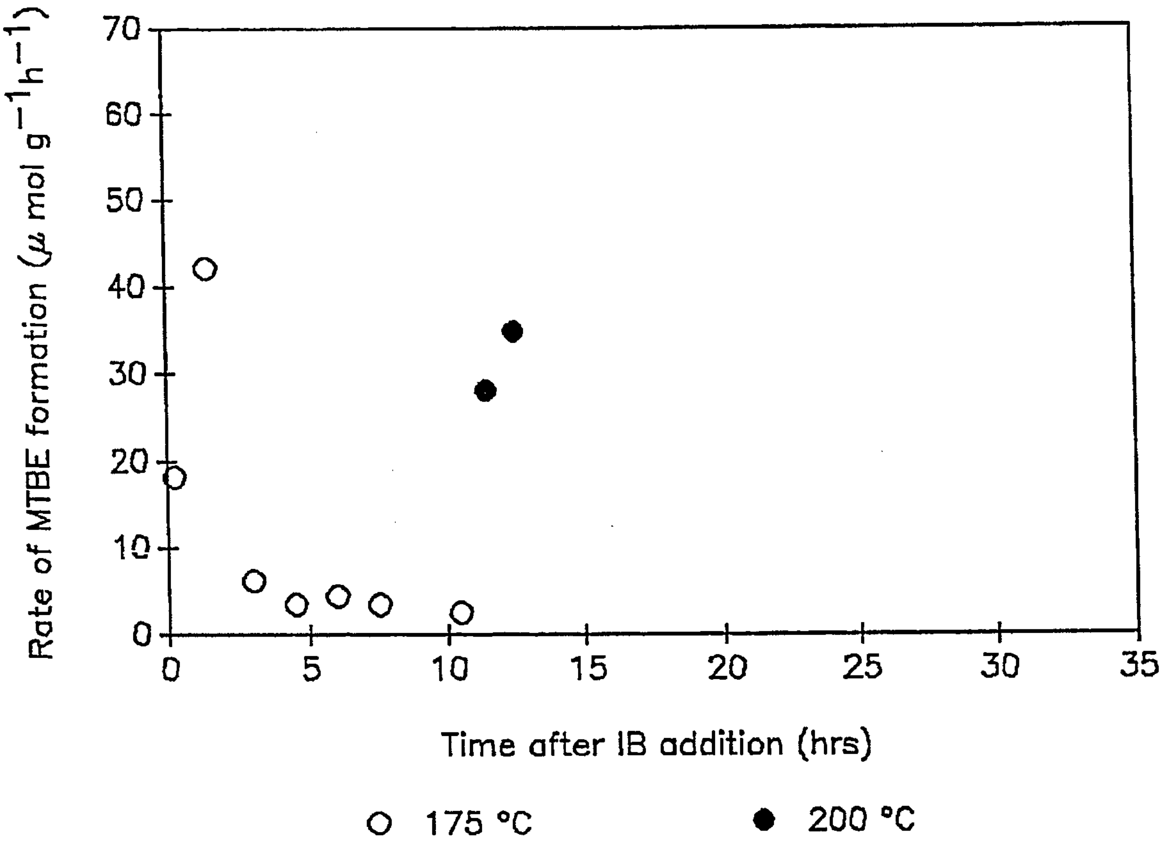
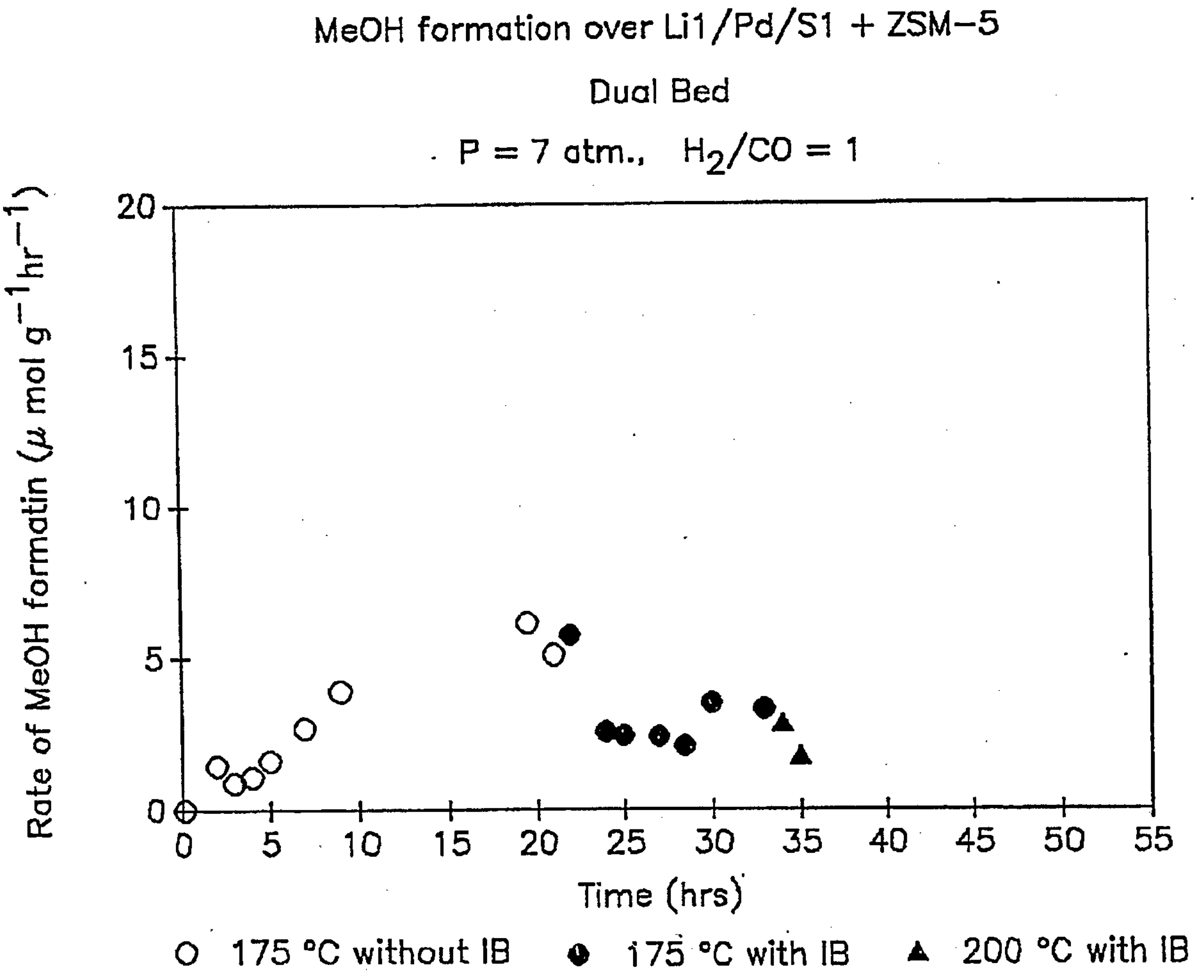


FIGURE 13.


MeOH formation over Li1/Pd/S1 + ZSM-5
Dual Bed
P = 7 atm., H_2/CO = 1
Rate of MeOH formatin (μ mol $g^{-1}hr^{-1}$)
0
5
10
15
20
0
5
10
15
20
25
30
35
40
45
50
55
Time (hrs)
○ 175 °C without IB
● 175 °C with IB
▲ 200 °C with IB

2. WORK PLANNED FOR NEXT QUARTER

Work for next quarter will concentrate in the SBCR runs for the synthesis of MTBE from i-butylene and methanol, and the completion of studies involving the addition of i-butylene during the synthesis of alcohols.

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