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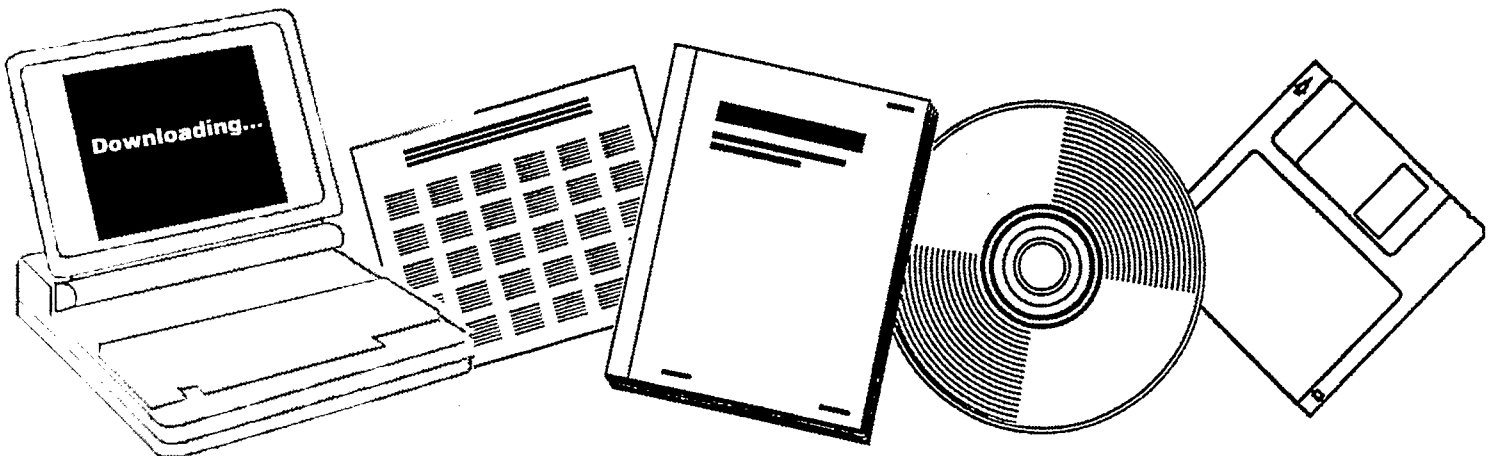
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**COAL LIQUEFACTION: INVESTIGATION OF  
REACTOR PERFORMANCE, ROLE OF CATALYSTS AND  
PCT PROPERTIES. QUARTERLY PROGRESS REPORT,  
APRIL 1-JUNE 30, 1984**

PITTSBURGH UNIV., PA

1984



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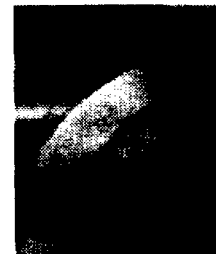
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Quarterly Progress Report

Coal Liquefaction - Investigation of  
Reactor Performance, Role of Catalysts  
and PCT Properties

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## 1. Objective and Scope of Work

The objective of this work is to investigate areas of science and technology that have been defined as being of prime interest to coal processing technology development. These areas include properties of coal liquids and slurries, reactor design, and performance in relation to reaction mechanisms. The work comprises investigations of the following high priority areas:

- . Reaction kinetics and reactor performance in direct coal liquefaction
- . Role of catalysts in indirect liquefaction and direct coal liquefaction
- . Physical, chemical and thermodynamic properties of coal liquefaction products.

## 2. Progress During Quarter

A detailed description of work in each of the three tasks is given below. Fifteen abstracts were prepared and are attached as an Appendix.

### Task 1 Reactor Kinetics and Reactor Performance in Direct Coal Liquefaction

The simulator developed by Schneider Consulting Engineers (SCE) models a preheater and a single reactor. This simulator has been modified to incorporate a number of reactors in series (at present, this number has been limited to 4, but it can be increased, if desired). Also, the SCE simulator assumed the same reaction network to occur in both the preheater and reactor. However, the preheater is considered to be a fast reaction zone where the fragmentation of coal into gases and intermediate products is complete and the reactor is considered to be a slow reaction zone where intermediates react to form final products. The program has thus been

appropriately modified so that the user can input different reaction networks for the preheaters and reactor.

Work is in progress to incorporate information regarding stability of the steady state if the operating conditions are in a region of multiple steady states. The simulator is also being modified to include kinetic data from existing pilot plant data.

## Task 2 Indirect Coal Liquefaction and the Role of Added Catalyst Direct Coal Liquefaction

The first draft on an analysis of Fischer-Tropsch (F-T) synthesis prepared in the previous quarter was revised during this quarter. The revision has been completed. This part of the report, besides F-T synthesis, includes the Kolbel-Englehardt reaction and Isosynthesis. The first drafts on the Mobil methanol-to-gasoline (MTG) technology and on the synthesis of methanol and the use of methanol as a fuel were prepared during this quarter. The history of methanol production, chemistry of and catalysts in methanol synthesis, kinetics of methanol synthesis in an industrial reactor, current production technologies, methanol from coal, three-phase methanol synthesis and methanol as a fuel are discussed in the section on methanol.

## Task 3: The PCT Properties of Coal Derived Liquids

The final report on heat of reaction is almost complete. Material and energy balances around the dissolver were done with existing data, and correlations available on coal liquids were compared with the actual experimental data reported by Stephenson (1981). A comparison of the predictions of existing correlations of the vapor pressure of coal liquids and the experimental data reported by Gray and Holder (1982) is being done. Work

on surface tension of coal liquids and heat transfer correlation of coal liquids is underway.

#### References

1. Stephenson, J.L., "SRC-II Process - Exothermic Heat of Reaction Evaluation", Letter to Dr. F.S. Antezena, Gulf Science and Technology, Ref # 560IFT12, Feb. 12 (1981).
2. Gray, J.A. and Holder, G.D., "Solvent Refined Coal (SRC) Processes - Selected Physical, Chemical and Thermodynamic Properties of Narrow Boiling Range Coal Liquids from the SRC-II Process, Supplemental Property Data," DOE report #DOE/ET/10104-44, work performed under contract #AC05-76ET10104 (1982).

**ABSTRACTS**

Title: Solvent Refined Coal (SRC-Process) - Selected Physical, Chemical and Thermodynamic Properties of Narrow Boiling Range Coal Liquids from the SRC-II Process, Supplemental Property Data, Section IV C4

Author: Gray, J.A. and Helder, G.D.

Source: DOE Report # DOE/ET/10104-44, work performed under Contract # AC05-76ET10104.

Vapor pressure measurements were performed on six narrow boiling heart cuts (4HC-A, 6HC, 7HC-B, 10HC-B, 15HC-B and 18HC-B) and critical temperature and pressure were measured on three different heart cuts (4HC-A, 6HC, and 7HC-B). A comparison of the vapor pressure equations of Starling et al. (1980) and Wilson et al. (1981) indicated that Starling's equation of state can more accurately reproduce the experimental data when only boiling point and specific gravity are available. This is true only if the boiling point is used in conjunction with the equation of state to determine the orientation parameters rather than using a separate correlation. The equation of state method produces roughly half the average deviation of the Wilson method for the various many heart cuts.

#### References

1. Starling, K.E., M.R. Brule, C.T. Lin and S. Watansani; Coal Gase Project Report to DOE, Coal Conversion Systems Technical Data Book Project, Institute of Gas Technology, Report OU/IGT/S-14366-1, Published by the School of Chemical Engineering and Materials Science, University of Oklahoma, Norman, OK (1980).
2. Wilson, G.M., Jhonston, G.H., Hwang, S.C. and Tsonopoulos, C., Ind. Eng. Chem. Proc. Des. Dev. 20 (1), 94 (1981).

Number of pages: 5  
Abstract prepared by: Sekhar Bhattacharjee



Title: Correlations Evaluated for Coal Tar Liquids

Author: Newman, S.A.

Source: Hydrocarbon Processing 60 (12), 133 (1981).

A comparative study was reported in this paper in which several available correlations for molecular weight, critical constants and vapor pressure were compared to available experimental data on coal tar components. Nine existing correlations were evaluated for vapor pressure and the authors concluded that in general no single method is clearly superior to the others. For the entire group of compounds, the Mobil vapor pressure equation is best if the Watson critical properties are used; the extended Exxon Maxwell-Bonnel correlation is best if Mobil critical and acentric factors are used; and if solids are eliminated from the data set the Exxon Maxwell-Bonnel correlation gives the best result. For predicting the vapor pressure of condensed ring hydrocarbons, aromatics, the Pitzer-Curl and either the Exxon or Hariu Maxwell-Bonnel correlations appear equally applicable.

Number of pages: 10

Abstract prepared by: Sekhar Bhattacharjee

Title: Vapor Pressure of Coal-Liquid Fractions - Data and Correlations

Author: Zudkevitch, D., Krautheim, P.D. and Gaydos, D.

Source: Fluid Phase Equilibria 14, 117 (1983).

A new empirical correlation of the Cox-Maxwell-Bonnell type was developed for predicting the vapor pressures of aromatic compounds and liquids derived from coal. The Cox-type correlation of Maxwell and Bonnell is used widely for prediction of vapor pressure in the petroleum industry. In the present work, new data on coal tar oils and data from literature on pure compounds and coal derived liquids were used for testing and prediction of MB correlation. An analysis of the actual experimental data on coal liquids indicated that the correlation is reliable within  $\pm 3\%$  when applied to pure aromatic compounds and liquids. Larger deviations up to 25% were observed for oxygen containing compounds as for example, cumarone or compounds such as dibenzofuran where the oxygen atom is in the middle ring of the aromatic structure.

Number of pages: 30

Abstract prepared by: Sekhar Bhattacharjee

Title: Solvent Refined Coal (SRC) Process Selected Physical, Chemical and Thermodynamic Properties of Narrow Boiling Range coal Liquids from the SRC-II Process, Supplemental Property Data, Section IV B4 and 5

Authors: Gray, J.A. and Holder, G.D.

Source: DOE Report # DOE/ET/10104-44, work performed under Contract # AC05-76ET10104.

Surface tension measurements were performed on cuts ~~4HC-B~~, 6HC, 7HC-A, 10HC-A, 15HC-A and 18HC-A at temperatures to 672K and pressures just above the saturation pressure. In general, surface tension of the various heart cuts decreased with increasing temperature. Surface tension increased with increasing boiling point. The surface tension data were correlated with reduced properties which are available from the vapor pressure measurements or were estimated from the existing correlations when a maximum deviation of 30.6% was observed. Experimental data were also reported by the authors on samples of vacuum tower overhead, heavy distillate products and recycled slurry obtained from PDU P-99 at temperatures to 672K and pressures up to 13.8 MPa. At temperatures below 525K, the surface tension of the recycled slurry was significantly greater than that for the heavy distillate and heavy distillate surface tension was slightly higher than that for the vacuum tower overhead. The effect of hydrogen pressure on surface tension of process streams was determined at 13.8 MPa at temperatures of 616.5K and 672.0K. The 13.8 MPa results averages about 15% lower than the values measured at lower pressures and the individual percentage differences varied from 0-28%.

Number of pages: 8

Abstract prepared by: Sekhar Bhattacharjee

Title: Vapor Pressure of Some Nitrogen Containing Coal Derived Liquids

Author: Rostyne, C.V.D. and Frausnitz, J.M.

Source: J. Chem. Eng. Data 25, 1 (1980).

In this work vapor pressure data for four nitrogen-containing heterocyclic compounds formed in coal derived liquids are reported. At elevated temperatures, there is a possibility of thermal degradation of coal liquids. These authors used a gas saturation apparatus to measure the vapor pressure at or near ambient temperatures. Vapor pressure data for quinaldi 5-ethyl-2 methylpyridine, quinoline and N-ethylcarbazol were experimentally determined and fitted by a standard Antoine type of equation. An average percent deviation of 1.62-2.80% was observed. The range of pressure studied in this paper was  $10^{-3}$  -  $10^{-1}$  mm Hg.

Number of pages: 3

Abstract prepared by: Sekhar Bhattacharjee

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Title: Effect of Nitrogen Content of Solvent on Coal Liquefaction

Authors: S.V. Fanvelkar, W. Ge, Y.T. Shah and D.C. Cronauer

Source: Ind. Eng. Chem. Fundam., 23, 202 (1984).

The effect of nitrogen content on the mechanism of coal liquefaction was studied using Powhatan No. 5, a high volatile bituminous coal. Three liquefaction solvents were used in the study: hydrogenated phenanthrene compounds, anthracene oil and SRC-II solvent. The premise that addition of heterocyclic compounds like tetrahydroquinoline (THQ) would maintain the hydrogen-donor capability of recycle solvents at higher levels during all phases of coal conversion was shown to be false for hydrogenated phenanthrene and SRC-II solvent as there was no change on the product distribution from coal liquefaction. THQ addition however had a major effect on the product distribution when added to the poor hydrogen-donor solvent, anthracene oil. But, in place of an increase in the yield of oils (pentane solubles), a net loss of solvent and a sizable increase in asphaltenes was observed. This was attributed due to adduction reactions of solvent and coal species. The level of adduction decreased with reaction time indicating some reversibility.

Number of pages in document: 6

Abstract prepared by: S. Joseph

Title: Coal Liquefaction in Nitrogen Compounds

Authors: F.J. Derbyshire, G.A. Odoerfex and D.D. Whitehurst

Source: Fuel, 63, 56 (1984).

The liquefaction of subbituminous coal (Wyodak-Anderson, Bell Ayr) was carried out with tetrahydroquinoline (THQ), a mixture of THQ and pyrene and tetralin as solvents. The conversion of coal to THF soluble products approached 100% under relatively mild reaction conditions (420°C) with THQ as solvent, after 1 hr of reaction time. The conversions obtained in a mixture of 30 wt % THQ/70 wt % pyrene although lower than in THQ, were significantly higher than in tetralin. A maximum in conversion with both THQ and mixture with pyrene was observed at 425°C. The extent of coal dissolution in THQ appeared to be influenced by its concentration relative to coal. The unusual ability of THQ to liquefy coal was attributed to these properties: it is an active hydrogen donor, it is regenerable during liquefaction by the reaction of hydrogen and quinoline and its polarity allows penetration of the coal structure and aids in dispersion of the dissolved coal. It was observed that during reaction with coal, THQ and other nitrogen compounds underwent extensive condensation reactions which resulted in an increase in the nitrogen content of the high boiling and non-distillable liquefaction products.

Number of pages in document: 5

Abstract prepared by: S. Joseph

Title: Short Contact Time Dissolution of Lidell Coal

Authors: N.R. Foster, R.G. Weiss, M.M. Young and K.N. Clark

Source: Fuel, 63, 66 (1984).

The thermal dissolution of Lidell coal in tetralin was studied at 400°C for reaction times ranging from 0 to 60 min. The initial dissolution of coal at the relatively mild liquefaction conditions (400 °C, 13.8 MPa) was rapid. No denitrogenation and negligible oxygen/sulphur elimination reactions occurred. At longer reaction times oxygen was eliminated via hydrogenolysis reactions. The product distributions were simulated using a system of second order pseudo kinetic equations. Good agreement between experimental data and calculated conversions was obtained (as revealed by the magnitude of the sums of squares of residuals (experimental conversion - calculated conversion) for all product species ( $6.5 \times 10^{-4}$ )).

Number of pages in document: 5

Abstract prepared by: S. Joseph

Title: Hydroliquefaction of Victorian Brown Coal in a Continuous Reactor  
1. Effects of Residence Time, Temperature and Catalysts on  
Conversion

Authors: J.B. Agnew, W.R. Jackson, F.P. Larkins, D. Rash, D.E. Rogers, P.  
Thewlis and R. White

Source: Fuel, 63, 147 (1984).

Slurries of two Victorian brown coals in tetralin were reacted with hydrogen in a vertical tubular bubble column reactor, 19 mm diameter and 2.22 m long. The maximum design operating conditions were 450°C and 27 MPa. The reaction was studied with and without the addition of iron and iron-tin based catalysts. The addition of tin to iron based catalysts caused a synergistic catalytic effect. The conversion to liquid products was greater when the catalysts were ion-exchanged onto the coal rather than being added as powders. Increases in reaction time and temperature and the addition of iron/iron-tin based catalyst system resulted in an increase in conversion. The increase in conversion was mainly reflected in an increase in the oil yield rather than in increased asphaltene or asphaltol formation.

Number of pages in document: 6

Abstract prepared by: S. Joseph



Title: Interrelationship of Chemical Characterization and Rheological Behavior of Coal Liquefaction Bottoms

Authors: F.F. Tao and R. Billimoria

Source: Fuel, 63, 158 (1984).

In the development of the Exxon Donor Solvent process, bituminous and subbituminous coals were processed in a 1 tpd coal liquefaction pilot plant operated at 700-723 K, 10.3-13.8 MPa hydrogen pressure using coal slurry feed with and without bottoms recycle. The liquefaction bottoms from both once-through and bottoms recycle operation exhibited non-Newtonian behavior. The recycled bottoms had a lower viscosity, were more thermally stable and showed more shear thinning behavior when compared to once-through bottoms. Chemical characterization and thermal analyses revealed that cross linkages between aromatic clusters were the major contributing factors for the high bottoms viscosity. The viscosity increase of molten bottoms in an inert atmosphere was postulated as being due to the formation of cross-linkages by condensation reactions of alkyl and phenolic functional groups.

Number of pages in document: 5

Abstract prepared by: S. Joseph

Title: Transportation Fuels from Synthesis Gas via ZSM-5

Authors: W. Lee, R.M. Gould, J.C.W. Kuo and S. Yurchak

Source: Proceedings of 10th Annual International COCLAC Conference,  
University of Pittsburgh, August, 1983.

The subjects of this paper are the Mobil's Methanol-to-Gasoline (MTG) fixed-bed and fluid-bed processes and two new concepts, which are extensions of the MTG process, for the conversion of syngas to transportation fuels. A plant based on the fixed-bed process is scheduled for completion in 1985 in New Zealand, which will produce about 570,000 tons annually of high-octane gasoline from natural gas. The fluid-bed process has been tested in a 100 BPD pilot plant in Germany with gratifying results. Further tests are in progress.

In the MTG process, the initial products are light olefins. By altering the process parameters and/or catalysts, the authors have obtained 80% C<sub>2</sub>-C<sub>5</sub> olefins, which can be converted to gasoline and distillates. The MTG technology should be used to convert coal to transportation fuels. The authors have tested a two-stage slurry FT/ZSM-5 process (MFT). Here, the H<sub>2</sub>-poor synthesis gas from second-generation gasifiers will be the source of syngas. In the MFT process, syngas is converted to hydrocarbons and oxygenates with an Fe/Cu/K<sub>2</sub>CO<sub>3</sub> catalyst in a slurry reactor and the effluents are upgraded to gasoline over ZSM-5 in a separate fixed-bed reactor. Single pass conversion in the slurry reactor is high and side reactions are minimized due to efficient temperature control. Future work planned with MFT concept are: coproduction of gasoline and distillates, chemicals and lube oil.

Number of pages in document: 17

Abstract prepared by: K. S. Seshadri

Title: Hydrocarbons from Methanol

Author: Clarence D. Chang

Publisher: Marcel Dekker, Inc., New York, 1983. ISBN 0-8247-7007-2

The book is divided into eight chapters with 178 references. The stoichiometry of conversion of methanol to paraffins and aromatics and the thermochemistry of these reactions are examined in the first two chapters. The third chapter deals with early catalytic reactions in which methanol is converted to a variety of hydrocarbons. Discussions are given of the structure of ZSM-5 and related zeolites, product distribution over various zeolites, the effect of pressure and temperature on product spectrum, high selectivity of phosphorous modified ZSM-5 for C<sub>2</sub>-C<sub>4</sub> olefins, and non-zeolite catalysts in methanol chemistry. The next chapter deals with the kinetics of methanol conversion to hydrocarbons. Published reaction schemes, the rate laws, rate constants, the fit of experimental data with theoretical rate equations are examined. Proposed reaction mechanisms are then reviewed. Several mechanisms are proposed in which divalent carbenoid species, methyl cations and methyl free radicals are reaction intermediates.

Another chapter is devoted to pilot plant work at Mobil to demonstrate the commercial possibilities of methanol-to-gasoline technology. The design of reactors, run conditions, material balance, gasoline properties and catalyst aging studies are given for fixed-bed as well as fluid-bed reactors.

Dual-function catalysts containing metal oxides for the conversion of synthesis gas to methanol and zeolites for the production of gasoline from methanol have been investigated at Mobil. The polystep process is accompanied by undesirable side reactions, particularly hydrogenation of olefin intermediates. The catalyst studied in detail is Zn-Cr-ZSM-5. With a

4 Zn:1Cr ratio C<sub>2</sub>-C<sub>4</sub> olefins are the main products. The work of others on dual-function catalysts, with similar results, is also discussed. The author points out that methanol-to-hydrocarbons has practical applications, but the mechanism and the kinetics of the reaction are not well-understood.

Number of pages in the book: 129

Abstract prepared by: K.S. Seshadri

Title: Methanol, Past, Present and Speculation on the Future

Author: Alvin B. Stiles

Source: AIChE Journal 23, 362, May, 1977.

This paper covers the history of methanol, early methods of production of methanol, such as destructive distillation of wood along with present commercial technology. In the section on methanol synthesis, competitive reactions that can occur during the hydrogenation of CO, thermodynamics of these reactions and active catalysts for each reaction are mentioned. Catalysts for methanol synthesis have to be carefully chosen to obtain high selectivity to methanol. Processes are discussed that were in operation for the coproduction of methanol and ammonia from syngas derived from coal or coke. Syngas preparation from natural gas and other feedstocks, gas purification, gas compression, converters and their design are described in great detail.

It is interesting that the author has singled out among other methods of methanol synthesis, the low-pressure, hydrogenation of CO via methyl formate. Types of catalysts used in commercial processes, specifications to commercial methanol, physical properties of methanol and its uses are given. The author agrees that coal should be the future feedstock, but advocates a technology differing from the current sledgehammer method in which all the bonds in coal are broken to produce syngas, which should include recovery of useful aromatic compounds from coal prior to its conversion to syngas. Other areas of research and developmental work suggested, assuming that methanol will be a major liquid fuel are: effective utilization of biomass; methods to

produce oxygen less expensively; novel catalysts to expand the product spectrum to include higher alcohols and other oxygenates.

No. of pages in the document: 14

Abstract prepared by K.S. Seshadri

Title: Methanol Synthesis

Author: H.H. Kung

Source: Catal. Rev.-Sci. Eng. 22 (2), 235, (1980)

In this paper, the basic chemistry of catalytic hydrogenation of CO to methanol is reviewed. The presence of both CO and H<sub>2</sub> enhances the adsorption of one or both components. This is considered as evidence for surface complexes. CO/H<sub>2</sub> adsorption on ZnO has also been studied by infrared spectroscopy and deuterium isotope labelling. The latter study suggests that methanol is formed via a surface species, possibly a surface methoxide.

Reports on homogeneous catalysts in methanol synthesis are reviewed in one section to find support to the discussed mechanistic schemes. The important part of the paper is the discussion on the composition and surface properties of catalysts, and criteria that an active catalyst should satisfy. In copper-based catalysts, Cu(I) in a solid solution of ZnO is the active species, whereas in ZnO-based catalysts, the activity is decided by the surface chemical composition as well as the atomic arrangement on the surface. The catalyst should be stable in a reductive atmosphere and non-dissociatively activate CO. Most oxides adsorb CO associatively, but CO may be weakly adsorbed. This difficulty is overcome by using mixed oxides with appropriate cations. Besides, a good catalyst should also have the ability to activate hydrogen. The most likely surface intermediate is methoxide. Catalysts which form stable metal methoxides are not suitable for methanol synthesis.

No. of pages in document: 25

Abstract prepared by K.S. Seshadri

Title: The Kolbel-Englehardt Synthesis

Authors: H. Kolbel and M. Ralek

Source: Chapter 7 in Fischer-Tropsch Synthesis by R.B. Anderson, Academic Press, NY, 1984.

The Fischer-Tropsch reaction for the fuels and chemicals from syngas is in commercial use. Another reaction is one in which hydrogen is completely absent and CO is directly reacted with steam in accordance with the equation  $3CO + H_2O \rightarrow -CH_2- + 2CO_2$ . Kolbel and Englehardt, while investigating the role of water-gas-shift (WGS) reaction in the F-T synthesis discovered this reaction and it is named the Kolbel-Englehardt (KE) synthesis.

The fundamentals of the KE synthesis, catalysts and reactors suitable for the reaction, extension of the reaction for the synthesis of aliphatic primary amines and high molecular weight paraffinic hydrocarbons, and the future of the reaction for commercial production of chemicals are the subjects of this chapter.

The KE reaction is formally regarded as the sum of F-T and WGS reactions. Iron, cobalt and nickel, the F-T catalysts, are also active in the KE reaction. But the iron catalysts are superior to others, because they are good catalysts for the WGS reaction. The best temperature for the reaction is 240°C. Above this temperature, CO conversion is low and excess methane is formed. Due to this temperature restriction, fixed-bed or slurry reactors are preferred and not fluidized-bed reactors. Since H<sub>2</sub> consuming reactions are suppressed, the products are rich in olefins and oxygen-containing molecules. Work on the slurry reactor used by Kolbel in the KE synthesis is being applied to F-T reactions carried out in the slurry phase.

No. of pages in document: 27

Abstract prepared by K.S. Seshadri



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