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LIQUID-ENTRAINED CATALYST OPERATIONS AT LAPORTE PILOT PLANT FOR LIQUID-PHASE METHANOL PROCESS, 1984-1985: FINAL REPORT

AIR PRODUCTS AND CHEMICALS, INC.
ALLENTOWN, PA

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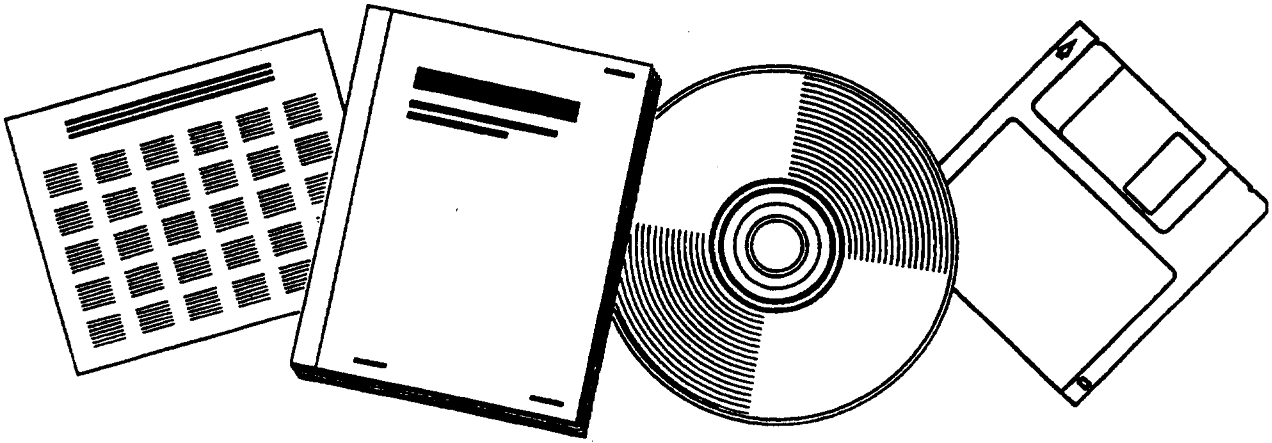

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Final Report
February 1987

Liquid-Entrained Catalyst Operations at LaPorte Pilot Plant for Liquid-Phase Methanol Process, 1984–1985

Prepared by
Air Products and Chemicals, Inc.
Allentown, Pennsylvania

REPORT SUMMARY

SUBJECTS	Coal-derived liquids / Gasification power plants	
TOPICS	Methanol Coproduction	Synthetic fuels GCC power plants
AUDIENCE	Fuels and generation planners	

EPRI-AP--5049
DE87 006950

Liquid-Entrained Catalyst Operations at LaPorte Pilot Plant for Liquid-Phase Methanol Process, 1984–1985

Sustaining catalyst life and increasing reactor productivity are key elements in improving methanol synthesis from coal gasification products. Tests at the LaPorte pilot plant using carbon monoxide–rich gas and an in situ reduction technique improved catalyst activation and demonstrated excellent catalyst deactivation rates.

BACKGROUND Synthesizing methanol in a gasification–combined-cycle (GCC) power nt offers utilities greater flexibility in handling variable load requirements. e liquid-phase methanol (LPMeOH)™ process, which is particularly suited for use with carbon monoxide (CO)–rich coal-derived gas, has the potential to produce methanol at lower costs than traditional gas-phase processes. Previous 5-t/d tests at the LPMeOH pilot plant in LaPorte, Texas, have improved the process and demonstrated methanol productivity and catalyst deactivation rates equivalent to those of bench-scale experiments.

OBJECTIVES To test high-catalyst slurry concentration using a commercial synthesis catalyst with CO-rich gas and to evaluate the short-term activity of a commercial catalyst with CO-rich gas at the 5-t/d scale.

APPROACH The investigators tested LPMeOH process performance and commercial catalyst activity at the LaPorte pilot plant over a wide range of operating conditions. In particular, they performed two test runs of a liquid-entrained catalyst operation, in which powdered catalyst particles are activated as they circulate through the reactor in slurry form. Performing these tests also enabled the project team to assess the plant's capability to operate for an extended period.

RESULTS The LaPorte liquid-entrained operations using a commercial catalyst over a wide range of slurry concentrations contributed significantly toward demonstrating LPMeOH technology at the 5-t/d scale. Major accomplishments were as follows:

- The LaPorte pilot plant demonstrated excellent mechanical performa achieving slurry concentrations of 25–49 wt%.

- An in situ reduction technique resulted in effective activation of the catalyst powder.
- The slow rate of catalyst deactivation achieved with CO-rich gas was comparable to the best results obtained in autoclave reactors.
- Selective changes in metallurgical processes reduced formation of metal carbonyl catalyst poisons.
- Performance of the LaPorte pilot plant at higher slurry loadings (above 30 wt%) was inferior to that of bench-scale reactors at comparable loadings.

EPRI PERSPECTIVE

Operations at the 5-t/d LaPorte pilot plant have significantly advanced development of the technology for producing methanol from coal gas. At this point, the results of this project suggest a need for tests to demonstrate adequate maintenance of catalyst activity for commercial applications. Such refinements of the LPMeOH process would enhance the economic incentive for methanol synthesis in GCC plants.

This project is a continuation of earlier work described in EPRI report AP-4430. For an economic feasibility study of the LPMeOH process, see EPRI report AP-3749. Results of another high-catalyst slurry concentration run are discussed in EPRI report AP-5050.

PROJECT

RP317-3
EPRI Project Manager: N. Stewart
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Contractor: Air Products and Chemicals, Inc.

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Research Project 317-3
DOE Contract DE-AC22-81PC30019

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Prepared by
Air Products and Chemicals, Inc.
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ABSTRACT

Liquid-entrained operations at the LaPorte LPMEOH* Process Development Unit (PDU) were performed under Task 10 of Contract No. DE-AC22-81PC30019 for the U.S. Department of Energy. During June 1984, a 6-day, high-slurry concentration run (45 wt%, Run E-2) demonstrated the ability of the PDU to operate smoothly with a high solids loading. The catalyst activity observed during Run E-2 did not compare well with the bench-scale predictions. This was in part due to an inadequate reduction. Mass transfer limitations may have also contributed to reduced catalyst performance, but this effect was masked by the inadequate reduction of the catalyst.

Data from a previous activity maintenance run (Run E-1) indicated that metal carbonyl catalyst poisons were limiting catalyst performance. In order to determine catalyst activity maintenance characteristics in a poison-free system, specific materials of construction were upgraded based upon their potential for metal carbonyl formation. This work culminated in a successful 40-day run (25 wt%, Run E-3) during May - June 1985. A successful in-situ reduction was accomplished, and catalyst deactivation rates were comparable to the best laboratory performance under CO-rich synthesis gas ($H_2/CO = 0.69$). The average decline in methanol productivity was 0.28%/day, and a nominal 4,540 kg/day (5.0 ton/day) crude methanol was produced.

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I. EXECUTIVE SUMMARY

Two liquid-entrained operations at the LaPorte LPMEOH* Process Development Unit (PDU) were performed under Task 10 of Contract No. DE-AC22-81PC30019 for the U.S. Department of Energy. The operations included a 6-day high slurry concentration run (Run E-2, June 1984) and a 40-day activity maintenance run (Run E-3, May - June 1985). The two runs, both starting with a commercial catalyst powder (F21/0E75), totaled 1100 hours of steady operation at the LaPorte LPMEOH PDU.

The high slurry loading test was conducted to evaluate the performance of the LaPorte PDU starting with a catalyst powder. The catalyst was slurried to a high concentration in order to provide a very demanding test of the slurry mixing and handling system, in-situ reduction technique, and methanol synthesis performance. A 43 wt% oxide, slurry composed of Freezene-100 oil and a catalyst powder (F21/0E75-29), was prepared in the slurry prep tank and successfully tranferred to the reactor/slurry loop. The concept of in-situ reduction was demonstrated for the first time at the LaPorte PDU scale. After reduction, balanced feed gas was introduced into the reactor at 250°C (482°F) and 6,310 kPa (915 psia). During the early part of the run, the slurry concentration was further increased to 49 wt% oxide by limiting the pump seal flush flow. At this solids loading, the productivity data indicated that the reactor performance was achieving only 55 to 60% of autoclave performance. Furthermore, nuclear density gauge surveys revealed an apparent accumulation of solids and/or a decrease in gas holdup in the bottom of the reactor. Subsequently, a series of process variable changes was made to identify the problems and to improve the PDU performance.

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Reactor performance relative to the laboratory prediction did not improve significantly when temperature, superficial gas and liquid velocities, or slurry concentration were varied. However, the dilution steps did appear to eliminate the presence of the axial density profile in the reactor after the solid concentration was reduced below 45 wt% (as oxide).

Subsequent laboratory studies indicted that inadequate catalyst reduction was a prime contributor to the off-performance of this run. Changes in the reduction procedure are expected to remedy this problem. Mass transfer limitations may have also contributed to reduced catalyst performance at high slurry concentration, but this effect was masked by the inadequate catalyst reduction. Another high slurry concentration run in the LaPorte LPMEOH PDU will be necessary to isolate this effect once the in-situ reduction procedure is modified. Mechanical performance throughout this run was excellent, having achieved a 99% on-stream factor. The post-run inspection revealed no solids blockages in the slurry circulation loop. This operation provided valuable experience for the operations and engineering staff on handling catalyst slurries above 40 wt%, which until this time had not been attempted at the LaPorte PDU scale.

Following Run E-2, an extensive research and engineering effort was devoted to the development of new reduction procedures and to upgrading the materials of construction at the LaPorte PDU. Changes to the materials were necessary to prevent the formation of metal carbonyls (a known catalyst poison) from the low alloy and carbon steel surfaces in the unit. Extensive carbonyl surveys were performed to determine the areas to be modified. Follow-up testing to confirm the effectiveness of the resulting modifications showed a significant drop in carbonyl generation rates.

The activity maintenance operation (Run E-3) using a 25 wt% oxide slurry (catalyst F21/0E75-35) and a CO-rich ($H_2/CO = 0.69$) reactor feed gas was a milestone in the development of the LPMEOH technology. Catalyst activation using the in-situ reduction procedure was successfully demonstrated with hydrogen consumption approaching the stoichiometric value for a properly reduced catalyst. The nominal operating conditions for the run were 250°C, 5,300 kPa, and 10,000 1/hr-kg space velocity. The PDU performance compared well with the laboratory predictions throughout the duration of the run, with productivity ranging from 28.3 g-mol/hr-kg early in the run to a final value of 24.7 g-mol/hr-kg. The decline in relative methanol productivity (0.28% per day) was a significant improvement over the results of the last activity maintenance run (Run E-1), and approached the performance of smaller bench-scale reactors. The low rate of catalyst deactivation achieved in this run is believed to be the direct result of chemical cleaning and the metallurgical upgrade performed before the run. Over the 40 days of operation, the unit achieved a 97% on-stream factor and produced 186 metric tons of 96 wt% methanol product. An electrical fault in the 01.10/01.20 compressor motor accounted for 34 hours of the downtime. The ability to maintain catalyst activity during an extended shutdown was proven when the productivity remained unchanged from levels present before the compressor outage.

Between the two runs, the hydrodynamics of the bubble column reactor at the LaPorte LPMEOH PDU were studied. Two-phase gas holdup measurements were taken to determine the effects of gas and liquid superficial velocities, temperature, and pressure on the gas holdup. The results were consistent with past studies (Reference 1), except for the detection of axial profiles at conditions favoring higher gas holdup in the reactor. The data also showed that gas holdup increased with increasing pressure and was relatively insensitive to temperature at 6,310 kPa (915 psia). Further, gas holdup was found to be directly proportional to superficial gas velocity, while changes in the superficial liquid velocity had no significant effect on gas holdup. These studies yielded valuable information for use in analysis of run results and in the design/scale-up of the LPMEOH reactor.

II. INTRODUCTION

The U.S. Department of Energy (DOE) and Air Products and Chemicals, Inc. are now in the final phase of an R&D program that began 28 September 1981 and aimed at further developing the Liquid Phase Methanol (LPMEOH) process invented by Chem Systems Inc. Air Products and Chemicals, Inc. is the prime contractor, providing overall program management, and is responsible for engineering design, procurement, construction, and operation of the Process Development Unit (PDU). Chem Systems performed as the key subcontractor in the program. Cost sharing was provided by Air Products, the Electric Power Research Institute, and Fluor Engineers, Inc.

LPMEOH technology has the potential to be a lower cost conversion route to methanol from coal than current gas phase processes. Laboratory work to date shows LPMEOH technology particularly suited to coal-derived synthesis gas rich in carbon monoxide. The LPMEOH process is capable of processing feed gas containing CO and H_2 at high concentrations and in variable proportions. The LPMEOH process allows high CO conversion per pass because it readily accommodates the heat liberated by the exothermic methanol synthesis reaction.

The LaPorte PDU operation is supported by an extensive laboratory program principally conducted at Air Products' facilities in Allentown. The laboratory program includes catalyst screening, testing, development, preparation, and analysis of catalyst samples from the PDU during operation at LaPorte.

A DOE-owned, skid-mounted pilot plant at the IGT HYGAS facility in Chicago was disassembled, transferred to Air Products' synthesis gas facility in LaPorte, Texas, refurbished, and expanded for service as the LPMEOH PDU. The construction of the LaPorte LPMEOH PDU was completed in October 1983. Commissioning began in November 1983 and was completed in early 1984. Subsequently, four runs with methanol synthesis operation totaling 2,305 hours of reaction time have been accomplished:

- Shakedown run with R71/OF12-26 catalyst (Run F-1)
- Activity maintenance run with R71/OF12-26 catalyst (Run E-1)
- High-slurry concentration run with F21/OE75-29 catalyst (Run E-2)
- Activity maintenance run with F21/OE75-35 catalyst (Run E-3)

The first two runs began in the liquid-fluidized mode, but the catalyst extrudates attrited rapidly to powder form. The PDU performed well, both during the transition from extrudates to slurry and at the complete slurry state. Process performance and catalyst activity over a wide range of operational conditions were investigated, and the capability of the PDU to operate for an extended period was demonstrated. The results of Runs F-1 and E-1 are presented in the Topical Reports for Task 5 and Task 6 of this DOE contract (References 1 and 2), respectively.

The latter two runs (E-2, E-3) were performed under Task 10, Liquid-Entrained Operation, using a commercial catalyst powder. Both of these runs used an in-situ reduction procedure in which the powdered catalyst is activated as it is circulating in slurry form (liquid-entrained) through the reactor. Runs E-2 and E-3 also marked the first demonstration of the in-situ reduction technique at the PDU scale. Results of Runs E-1 and E-2 revealed the need to upgrade the materials of construction to minimize metal carbonyl formation at the LaPorte LPMEOH PDU. The planned material change-out was made after Run E-2. Subsequently, excellent reactor performance was achieved in Run E-3, with catalyst deactivation comparable to laboratory predictions at similar conditions. The pertinent data for Runs E-2 and E-3 and the discussion on the progress of the Liquid Phase Methanol Program are presented in this topical report.

III. LAPORTE LPMEOH PDU DESCRIPTION

A. Background

The primary function of the LaPorte LPMEOH PDU is to acquire necessary plant data at a small but representative engineering scale for testing the feasibility of the LPMEOH process. The feasibility determination includes analyzing the sensitivity of major process variables and evaluating the process design. The PDU was designed with the capability of generating and collecting plant data over a wide range of potential operating conditions for the LPMEOH process.

Two types of reactor configurations have been developed for the LPMEOH process. The first, termed the liquid-fluidized catalyst reactor, employs an ebullated bed of 3-6-mm diameter particles in which the upward flow of inert hydrocarbon liquid and synthesis gas results in the fluidization of the catalyst to a desired bed expansion. Phase separation between solid and liquid/gas occurs at the top of the reactor. Thus, the catalyst bed remains within the fixed boundaries of the reactor. The second configuration is termed the liquid-entrained catalyst reactor. In this system, small catalyst particles (micron size) are suspended or slurried in the inert liquid and circulated through the reactor. Contact with the synthesis gas is made by feeding the gas cocurrently with the upward flow of liquid.

The LaPorte LPMEOH PDU was originally intended to be a virtual reuse of the Liquid Phase Methanation (LPM) Pilot Plant operated from 1977 to 1978 at the IGT HYGAS facility in Chicago, Illinois. Relocation of this equipment to LaPorte, Texas, and some renovation work was anticipated to allow operation in the liquid-fluidized mode. At a later date, a skidded train of equipment would be designed and tied into the first unit to allow for liquid-entrained operation.

However, past experience with PDU design and operation showed that full consideration of all modes of operation during the initial design stage would decrease the chance of extensive modifications later in the program that would not be identified otherwise. In recognition of this, a single "unified design" approach was adopted for this PDU. Equipment, instrumentation, and valving specifications included the consideration of both modes of operation from the start of the design effort. Under this premise, a process flowsheet of the unified design was developed jointly by Air Products and Chem Systems. The flowsheet incorporated a high degree of operational flexibility, and the resultant PDU design permitted switching operations from the liquid-fluidized to the liquid-entrained mode without equipment or piping alterations. A common reactor was designed to accommodate both modes.

B. Process Design Basis

High operating pressure favors the equilibrium of the methanol synthesis reaction, and it was desirable to design the PDU to achieve a reasonably high pressure. Ideally, this could have been as high as 10,450 kPa (1,515 psia). However, the reuse of existing equipment limited the maximum operating pressure to 6,310 kPa (915 psia). This was considered acceptable, since 6,310 kPa is close to the optimum pressure in many commercial methanol plants, and a wide operating pressure range remained available (3,550-6,310 kPa), 515-915 psia). The ranges of other operating variables were chosen to encompass operating conditions of interest (Table III-1). As will be apparent later, some of the design ranges were exceeded in actual operation.

Two principal reactor feed compositions were identified for the design basis (Table III-2):

TABLE III-1

RANGE OF OPERATING VARIABLES FOR LAPORTE LPMEOH PDU

	Minimum	"Normal"	Maximum
Reactor Pressure, kPa (psia)	3,550 (515)	5,270 (765)	6,310 (915)
Reactor Temperature, °C (°F)	220 (428)	250 (482)	270 (518)
Liquid-Fluidized Space Velocity, l/hr-kg cat	1,000	2,500	4,000
Liquid-Entrained Space Velocity, l/hr-kg cat	2,000	6,000	10,000
Superficial Gas Velocity, cm/s	2.0	9.0	12.0
Slurry Concentration, wt % oxide	10.0	25.0	33.0

TABLE III-2

LAPORTE LPMEOH PDU FEED GAS COMPOSITIONS

(Based on Texaco Gasifier)

	Balanced	CO-Rich
H_2	55%	35%
CO	19	51
CO_2	5	13
N_2	21	1
	100%	100%
H_2/CO Ratio	2.89	0.69
$H_2/(CO + 1.5\ CO_2)$	2.08	0.50