PNL-SA--17707

DE91 001318

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OF WET, INDUSTRIAL WASTES

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April 1990

Presented at the
25th Intersociety Energy Conversion
Engineering Conference
Reno, Nevada
August 12-17, 1990

Work supported by
Department of Energy
under contract DE-AC06-76RLO 1830

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BENCH-SCALE REACTOR TESTS OF LOW-TEMPERATURE, CATALYTIC GASIFICATION OF WET INDUSTRIAL WASTES

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ABSTRACT

Bench-scale reactor tests are under way at Pacific Northwest Laboratory to develop a low-temperature, catalytic gasification system. The system, licensed under the trade name Thermochemical Environmental Energy System (TEES®), is designed for a wide variety of feedstocks ranging from dilute organics in water to waste sludges from food processing. The current research program is focused on the use of a continuous-feed, tubular reactor. The catalyst is nickel metal on an inert support. Typical results show that feedstocks such as solutions of 2% para-cresol or 5% and 10% lactose in water or cheese whey can be processed to >99% reduction of chemical oxygen demand (COD) at a rate of up to 2 L/hr. The estimated residence time is less than 5 min at 360 °C and 3000 psig, not including 1 to 2 min required in the preheating zone of the reactor. The liquid hourly space velocity has been varied from 1.8 to 2.9 L feedstock/L catalyst/hr depending on the feedstock. The product fuel gas contains 40% to 55% methane, 35% to 50% carbon dioxide, and 5% to 10% hydrogen with as much as 2% ethane, but less than 0.1% ethylene or carbon monoxide, and small amounts of higher hydrocarbons. The byproduct water stream carries residual organics amounting to less than 500 mg/L COD.

INTRODUCTION

A catalytic gasification system that operates in a pressurized water environment has been under development at Pacific Northwest Laboratory for over 10 years. Initial experiments, performed for the Gas Research Institute, were aimed at developing kinetics information for steam gasification of biomass in the presence of catalysts [1]. In that work, the combined use of alkali and metal catalysts was first reported for gasification of biomass and its components at low temperatures (350 °C to 450 °C)[2]. From this research evolved the concept of a pressurized, catalytic gasification system for wet biomass feedstocks to produce fuel gas [3]. Extensive batch reactor testing [4] and limited continuous reactor system (CRS) testing [5] were undertaken in the development of this system under sponsorship of the U. S. Department of Energy. A wide range of biomass feedstocks were tested and the importance of the nickel metal catalyst was identified. Specific use of this process for treating food processing wastes has also been reported [6]. More recently, the concept application was expanded to encompass cleanup of hazardous wastewater streams, and both batch reactor test results [7] and continuous reactor test results [8] have been reported. The process has now been licensed as the Thermochemical Environmental Energy System (TEES®) to Onsite*Ofsite, Inc., of Pasadena, California, a turnkey design engineering and construction management firm. TEES® was recognized with an R&D 100 Award in 1989 as one of the top 100 new technical developments to reach the

marketplace during 1988. The subject of this paper is the use of TEES® in bench-scale CRS experiments for the recovery of energy from waste streams representative of the food processing and organic chemical industries.

CONTINUOUS REACTOR SYSTEM

The relative simplicity of the TEES® process allows straightforward bench-scale testing. The bench-scale CRS, shown schematically in Figure 1, was composed of a pump, tubular reactor, cooler, pressure control device, product separator, gas measuring system, and condensate collector.

Description of Equipment

A reciprocating, packed-plunger, positive-displacement pump was used to feed the system. System piping included 0.5-in. O.D. (0.065-in. wall) 304 stainless steel tubing on the outlet of the pump. Pump inlet piping was 0.5-in. O.D. (0.035-in. wall) 304 stainless steel tubing. All valves and valve trim (except the pressure control valve) were also made of stainless steel.

The tubular reactor was a 2-in. O.D. x 1-in. I.D., 304 stainless steel pipe reactor. The screwed-on end-caps contained hold-down bolts for the 304 stainless steel end-pieces and O-ring seals. The vessel was heated by a three-piece ceramic furnace. Temperature was monitored on the outside wall and at the center of the catalyst bed at three points along the length of the reactor.

Once the products left the reactor, they were cooled in a primary condenser and reduced in pressure before entering the separator. Pressure was controlled in the reactor by either a control valve or a dome-loaded back-pressure regulator. Piping downstream from the reactor was 0.25-in. O.D. with thick wall (0.049 in.) tubing before pressure letdown and thin wall (0.035 in.) tubing after. The valve itself contained a 3/32-in. orifice. The valve stem and seat were made of stellite. Excessive wear on the stellite trims led to replacement of the valve with the back-pressure regulator. Both systems functioned adequately while in good condition.

After pressure letdown, separation of phases was effected in a 24 in. x 0.5-in. diameter tube vertical separator. Most of the liquid products were removed via the bottom of the separator. In order to eliminate carryover of water into the gas analysis system, a ball float trap was placed after the secondary condenser as well as a T-type filter with a 0.02- μ m hollow fiber membrane filter. The float trap

*Pacific Northwest Laboratory is operated by Battelle Memorial Institute for the United States Department of Energy under Contract DE-AC06-76RLO 1830.

1. Reciprocating Pump
2. 1" ID x 72" Tubular Reactor
3. 6 KW, 3-zone Furnace
4. 60 Micron Filter
5. Primary Condenser
6. Pressure Control Valve
7. Condensation Pot
8. Liquid Collection Tank
9. Liquid Sample Loop
10. Separator Gas/Liquid
11. Filter
12. Gas Flow Meter
13. Float Trap for Condensed Liquid

FIGURE 1. Bench-Scale CRS Configuration

was self-draining, and the separator was drained manually. Condensate product from both drains was collected in a liquid receiving tank mounted on an electronic load cell.

A liquid sample loop, upstream of the separator, allowed recovery of small volumes of liquid product before it was dumped into the condensate collecting vessel. These samples were believed to be representative of the reactor contents.

The data acquisition and control (DAC) system employed in the CRS was a hybrid personal computer (PC)-based system employing discrete data acquisition devices and single loop process controllers communicating to a central PC via RS232 serial communications lines. The PC was used during experiments to monitor the process, calibrate instruments, and record data onto an ASCII disk file for later analysis. A custom-made program was used to coordinate these activities. Distributed, single loop control by stand-alone controllers was selected in order to provide additional safety in the event of a DAC system failure, and to reduce the amount of computing overhead assigned to the DAC software. Non-control sensors such as thermocouples and the gas mass flow meter were monitored via a data acquisition unit. The unit processed all raw signals to engineering units before sending the data to the PC for recording. Use of this system permitted a number of data channels to be relayed via a single RS232 serial port, and permitted additional computing overhead to be shifted from the main PC.

Product gas flow rate was measured by a thermal conductivity, mass flow sensor. The unit was calibrated to nitrogen gas (0-2 SLPM), but actual flow rates of mixed gases could be calculated based on known calibration factors and known gas composition. The product gas also flowed through a wet test meter to determine the total flow of gas.

Reactor System Operation

The tubular reactor was sealed once the specified amount and type of catalyst was loaded into the reactor vessel. After the reactor was sealed, the system was pressure-checked with nitrogen. The purge gas was vented from the system and the reactor refilled with hydrogen. The catalyst bed was heated up overnight in the hydrogen to assure a reduced nickel metal catalyst.

When the system had heated to operating temperature, the feedstock was pumped into the reactor system and the final pressurization of the system was achieved. Feed rates, temperatures, and pressures were controlled throughout the experiment while products were recovered and quantified.

To terminate an experiment, the feed pump was stopped and the reactor furnaces were turned off. Pressure was usually left in the reactor during cooldown overnight.

The residence time in the reactor can only be approximated. At the temperatures of the experiments (around 360 °C), the densities of the liquid and vapor phases of water change quickly as a function of temperature. In addition, the vapor pressure of water increases dramatically as the temperature increases and approaches the critical point. In our reactor the amount of water in the vapor phase was a function of the temperature of the reactor since it was a direct function of the pressure differential between the vapor pressure of water at that temperature and the total operating pressure of the reactor. Also, the amount of water vapor depended on the amount of product gases generated from the organic feed. The product gases acted as diluents to reduce the vapor pressure of the water in the reactor, which caused more water to pass into the vapor phase to maintain its vapor pressure. As more water was vaporized in proportion to more gas production or a smaller difference between the operating pressure and the vapor pressure of water at the operating temperature, the total residence time of all phases in the reactor was reduced. However, the reactor was not isothermal nor was the gasification instantaneous. Therefore, the effects of temperature and gas production varied along the length of the reactor.

Since this CRS is intended for process development purposes, we wish to derive usable design information from the experiments.

Although residence time in the catalyst bed is important in understanding the chemical mechanisms in order to optimize operation of the system, we can use liquid hourly space velocity (LHSV) in terms of L feedstock (at ambient conditions)/L catalyst/hr to scale up the operation of this tubular reactor. In this case, we used the portion of the catalyst bed at temperature as the catalyst volume. Since the feedstock entered the tubular reactor at ambient temperature, the bottom portion of the reactor was used to preheat the feedstock to operating temperature. In the preheat zone the metal catalyst acted primarily as a heat transfer surface. Although it has potential to facilitate the decomposition of the feedstock by catalysis of hydrogenation reactions, little hydrogen is available at the lower temperatures.

Product Analysis

Gas product analysis in conjunction with the liquids analysis was used to determine material balances and conversion rates around the reactor system. The gaseous stream was composed principally of CO_2 , CH_4 , H_2 , and C_2 + hydrocarbons, as well as water vapor. The online gas analysis equipment was able to measure concentrations of carbon oxides, hydrogen, methane, and C_2 hydrocarbons with reasonable accuracy and precision on a near-continuous (real-time) basis with a very short (<5 min) turnaround. In the CRS, a thermal-conductivity-based detector was used in a gas chromatographic analyzer. A secondary means used to determine product gas composition was a dedicated gas chromatograph. A thermal conductivity detector was used to analyze gases including hydrogen, carbon oxides, oxygen, nitrogen, methane, ethane, ethylene, and a backflush containing C_3 + hydrocarbons.

The aqueous feedstocks and products were analyzed for chemical oxygen demand (COD) and pH. The COD measurements were made with a closed reflux micromethod.

TEST RESULTS

Sufficient testing has been done to verify high conversion of three feedstocks in the bench-scale TEES[®] system. Para (p) -cresol has been tested as a 2% solution in water; lactose as both a 5% and a 10% solution in water; and cheese whey (6.5% dry solids), as received.

A number of different nickel catalysts have been tested for the TEES[®] process. These tests in the tubular CRS have demonstrated significant differences in the activity and stability of various commercial nickel catalysts. The catalysts themselves represent a range of steam-reforming, methanation, and hydrogenation catalysts. Properties of the catalysts are given in Table 1.

TABLE 1. Nickel Catalysts Tested in the TEES[®] Process

Property	Catalyst A	Catalyst B	Catalyst C	Catalyst D	Catalyst E
Type	Methanation	Hydrogenation	Hydrogenation	Steam Reforming	Steam Reforming
Surface Area	Moderate	High	High	Low	Low
Nickel Content	Moderate	High	High	Moderate	Low
Thermal Stability	Moderate	Low	Low	High	High

The nickel effectively catalyzes the reaction of the organics with the water to produce a mixed product gas of methane, carbon dioxide, and a lesser amount of hydrogen. The proportions of the three gases are determined by the concentration of the organic in water and the amounts of carbon, hydrogen, and oxygen in the organic material since the gas composition appears to be near equilibrium in the case of active catalysts [4]. In cases of less active catalysts, the hydrogen concentration is not driven to very low levels by catalysis of the methanation of carbon oxides with the hydrogen. With active catalysts the concentration of carbon monoxide in the product gas is reduced to an immeasurably low level (<0.05%). Two to four carbon alkanes (ethane, propane, and butane) are usually present at levels of <1%, but olefins are not found except in cases of low catalyst activity. This product gas would be valuable as a medium-Btu fuel or could be recovered as high-pressure pipeline quality gas after membrane separation of the carbon dioxide.

p-Cresol Results

Tests with p-cresol have verified earlier predictions of reactor requirements based on continuous stirred-tank reactor (Carberry) tests [5,6,8]. As shown in Table 2, better than 99% reduction in COD was achieved with an estimated residence time of less than 5 min (LHSV = 2.9 L feed/L catalyst/hr). The gas product was primarily methane with carbon dioxide and hydrogen also major components. The total gas yield was 1.6 L/g p-cresol of a medium-Btu fuel gas. These results were obtained throughout a period of 5 hr of operation with catalyst A.

TABLE 2. Experimental Results with Different Feedstocks

	p-Cresol, 2%	Cheese Whey	Lactose, 10%	Lactose, 5%	5% Lactose w/ Na_2CO_3
Catalyst	A	C	C	C	C
Result					
Gasification of Carbon, %	94.3	94.3	87.5	94.8	93.8
Reduction COD, %	99.1	99.3	99.3	99.7	99.3
Feed, L/hr	1.7	1.2	1.0	1.2	1.2
Gas, L/hr	53.8	50.2	66.4	50.2	45.3
Gas, L/g	1.58	0.68	0.67	0.78	0.78
Effluent, ppm COD	470	360	530	140	440
Effluent pH	6-8	7	3	3-5	3-5
Gas Composition:					
Methane, %	53	48	47	40	44
C Dioxide, %	36	45	49	47	48
Hydrogen, %	9	6	3	10	7
Ethane, %	0.3	1.0	0.4	2.2	1.0
Btu/SCF	606	550	509	503	498

@ 360 °C and 3000 psig catalyst bed conditions

Cheese Whey Results

Similar results, also shown in Table 2, have been obtained using cheese whey as the feedstock. Over 99% reduction in COD was achieved with an LHSV of 2.3 L/l/hr. The gas product contained less methane, reflecting the more highly oxidized state of the

feedstock. Again, carbon dioxide and hydrogen composed most of the rest of the gas product. The total gas yield was 0.7 L/g dry whey solids of a medium-Btu fuel gas.

Lactose Results

Lactose has been tested in a number of experiments as a model compound for cheese whey. Approximately 3/4 of the dry solids in cheese whey are lactose. Inorganic components and sulfur-containing proteins in cheese whey may deactivate the nickel metal catalyst. The lactose experiments allow us to test the catalytic gasification of the organic structure without possible catalyst deactivation by the other trace elements.

Catalyst C exhibits the highest activity seen thus far for lactose gasification (Table 2). The activity is nearly unchanged over a 24-hr operating period. Over 99% reduction in COD was achieved with an LHSV of 1.8 L/L/hr. Good quality medium-Btu gas was produced containing primarily methane and carbon dioxide with a low level of hydrogen. A similar test (results shown in Table 2) was done with sodium carbonate added with the lactose, both as a simulant of the alkali found in cheese whey and also as a possible buffer for the acidic nature of the reaction environment (pH 3 to 5 in the effluent). Steady, high conversion was also achieved over a 5-hr period in this test (LHSV = 2.0 L/L/hr). Although the feedstock entered the reactor at pH 10.5, we do not know if significant buffering of the reaction environment was achieved. Effluent samples from the reactor were measured at pH 3 to 5 as in the unbuffered experiments.

Results with Other Catalyst Types

Tests with steam reforming type catalysts D and E were not as successful. The D catalyst was tested in a prereduced form and exhibited only moderate activity (see Table 3) with either lactose or p-cresol as the feedstock. The E catalyst was obtained in the commercial oxidized form and was reduced in dry hydrogen at 400 °C in the reactor. Only partial reduction was achieved at these conditions, and further reduction while on-line in the gasification mode (as is commercially done in steam reformers) was not apparent. Moderate catalyst activity was achieved, as shown in Table 3. Another type-E catalyst was also tested after treatment in dry hydrogen at only 350 °C. No reaction of the hydrogen was noted, so the catalyst used in the experiment is considered to be nickel oxide, which has been found to have no catalytic activity for this reaction [7]. Only pyrolysis and limited gasification was achieved in this test, as shown in Table 3.

Catalyst Deactivation Mechanisms

Maintaining the catalyst activity is of paramount importance in any catalytic process. The successful use of the nickel metal catalyst in the TEES[®] reaction environment is a priority in this research. Batch tests [1-4,7] demonstrated the short-term activity of the catalyst; use of a continuous-feed reactor system is required to demonstrate long-term stability of the catalyst. Initial CRS experiments [5,6,8] identified catalyst stability as a problem area requiring further research and development. Early hypotheses about catalyst deactivation focused on the organic overload of the catalyst sites and resulting carbon fouling of the catalyst or slow oxidation of the nickel metal catalyst under these steaming conditions.

Concerns about oxidation of the catalyst or carbon laydown on the catalyst have been alleviated by analysis of used catalysts, which show neither of these problems exist. We have now determined that carbon fouling is a symptom of loss of catalyst activity, not the cause. Carbon deposition on the catalyst has barely been measurable at less than 1%. Oxidation of the catalyst has not been encountered in any of the used catalysts. The crystalline forms in the catalyst were determined by x-ray diffraction (XRD). Nickel metal was the

primary crystalline form of nickel in all cases. However, XRD also indicated that significant crystallite growth occurred in the TEES[®] reaction environment.

TABLE 3. Experimental Results with Different Catalyst Types

Feedstock	Catalyst D	Catalyst D	Catalyst E(reduced)	Catalyst E(oxidized)
	10% Lactose	2% p-Cresol	10% Lactose	10% Lactose
Results				
Gasification of Carbon, %	43.3	79.8	48.1	15.2
Feed, L/hr	0.9	1.2	0.8	0.6
Gas, L/hr	36.5	38.6	40.2	7.2
Gas, L/g	0.42	1.54	0.50	0.13
Effluent, ppm COD	30500	3350	13700	40000
Gas Composition:				
Methane, %	25	36	19	3
C Dioxide, %	49	34	50	83
Hydrogen, %	24	28	27	12
Ethane, %	0.7	0.3	0.6	0.5
Btu/SCF	392	499	326	145

@ 360 °C and 3000 psig catalyst bed conditions

Nickel crystallite growth and resulting loss of active catalyst sites is usually viewed as a high-temperature problem. Our operation at relatively low temperature does not avoid the problem in the presence of a high-pressure steam/water environment. The crystallite growth may result from organic overload of the catalyst through increased organic acid concentration. Organic acid intermediates in the pyrolytic breakdown of the feedstock are believed to be present in a transitory role. Buildup of the concentration of these intermediates would be avoided by stable operation of the system at appropriate temperature and pressure and operation of the catalyst only under proper conditions of flow and concentration that allow high conversion of the organics.

Effects of Catalyst Deactivation

Measurements of the crystallite sizes of the nickel catalyst have been made before and after use in TEES[®]. Typical active catalysts of this type have nickel crystallites of around 100 Å or less. Measurements of the crystallites after use showed growth to the range of 300 to 500 Å in some catalysts after only a few hours on stream. Such a crystallite size change would cause a significant loss of surface area and, therefore, result in loss of catalyst activity [9]. Results with the catalysts after crystallite growth are shown in Table 4. As seen by the data, loss of activity for both gasification and methanation is evident.

Research Plans

Tests have shown that at least 24 hr of operation can be achieved with a type-C catalyst with lactose as the feedstock. Longer catalyst lifetimes must be proven in order to optimize process economics. Testing will continue to verify the catalyst operability with other feedstocks of interest, including cheese whey, brewer's spent grain, and grape pomace. Also, alternative shutdown and restart procedures will be investigated to find the correct set of

conditions to minimize crystallite growth in the catalyst. Commercial operation of TEES[®] for waste cleanup is the goal of this continuing research.

TABLE 4. Experimental Results with Deactivated Catalysts

Catalyst	p-Cresol, 2%	Cheese Whey	Cheese Whey	Lactose, 10%
	A	B	C	C
Result				
Gasification of Carbon, %	50.0	50.2	59.8	40.2
Feed, L/hr	2.0	0.9	1.2	1.0
Gas, L/hr	33.1	24.8	36.5	66.4
Gas, L/g	0.75	0.34	0.50	0.67
Effluent, ppm COD	11500	17000	11600	22000
Gas Composition:				
Methane, %	48	21	28	21
C Dioxide, %	38	59	48	70
Hydrogen, %	9	15	20	5
Ethane, %	0.5	1.6	1.2	1.5
C Monoxide, %	0	0	0	0.2
Backflush, %	2.3	2.3	1.8	2.8
Btu/SCF	597	361	430	341

@ 360 °C and 3000 psig catalyst bed conditions

CONCLUSIONS

The TEES[®] process has been demonstrated in a bench-scale, continuous-feed tubular reactor system. Feedstocks tested included p-cresol and lactose water solutions and cheese whey. High-surface area, high-concentration nickel metal catalysts have been shown to be most effective.

High levels of conversion of organics in water have been achieved as suggested by >99% reduction of COD in the organic-containing water. The gas product is composed primarily of methane, carbon dioxide, and a lesser amount of hydrogen. The product gas would be useful as a medium-Btu fuel (higher heating value of ≥ 500 Btu/SCF) or as a source of high-pressure pipeline quality gas and carbon dioxide following membrane separation.

Catalyst activity is degraded by flow upsets and process idling. A potential cause of the catalyst deactivation is increased nickel crystallite size.

ACKNOWLEDGMENTS

The authors acknowledge the financial support of the U. S. Department of Energy, primarily through its Office of Industrial Programs (OIP) and also through the Research, Development, Demonstration, Testing & Evaluation program. We thank Mr. Stuart Natof, program manager in OIP, for his support of the research.

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