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Thermocatalytic Conversion of Food Processing Wastes

Topical Report FY 1988

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January 1989

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**Pacific Northwest Laboratory
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SUMMARY

Pacific Northwest Laboratory (PNL) is developing a thermocatalytic gasification system that converts high-moisture organic wastes to a medium-Btu (450 to 550 Btu/scf) fuel gas. Liquid wastes with a high chemical oxygen demand can also be treated but may require the use of supplemental fuel. A 1-L batch reactor has already been used to convert a number of food processing wastes, including fruit pomace, potato peeling sludge, cheese whey, and brewer's spent grain. The waste is converted in the presence of a nickel catalyst at 350°C to 400°C and 2000 to 4000 psig. Carbon conversion to gas is typically 90 to 95 wt% after 15 to 30 min. Methane yields of up to 6.3 scf/dry pound of spent grain have been obtained, with the methane content of the raw gas ranging from 45 to 55 vol%. Data obtained from the batch reactor studies indicate that the process is a net energy producer for most organic waste streams containing more than 2 wt% solids.

The PNL process is currently being tested in a continuous reactor system (CRS) designed to obtain reactor engineering and design data, which will be required for process scaleup. Initial data obtained in the CRS with spent grain and cheese whey indicate that the rate of conversion is high for the food processing wastes tested. Based on these initial results, it appears that high conversion can be achieved in less than 10 min in a plug-flow tubular reactor. The CRS tests also identified catalyst deactivation, which apparently was caused by some component of the food processing wastes, as an area where additional research is required. We are working with United Catalysts, a catalyst manufacturer, to determine the cause of deactivation and to select or develop catalysts resistant to deactivation.

Onsite*Ofsite, Inc., a design and construction management firm, has estimated the capital cost of a full-scale facility to convert food processing wastes to energy. The net cost of the gas produced by the process is highly dependent on the cost of waste disposal. As the cost of waste disposal increases, the PNL process will become very attractive economically. Onsite*Ofsite has also completed a preliminary design of a process development unit that can be used to demonstrate the concept on a near-commercial scale.

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INTRODUCTION

The efficient utilization of waste produced during food processing operations is a topic of growing importance to the industry. As landfill costs rise and the regulatory environment eliminates some of the alternatives to landfilling, processors are faced with increasing pressure to find economically attractive and environmentally sound disposal and/or utilization options. One option that has been used successfully for some wastes is incineration, which not only reduces waste volume but produces a valuable co-product (process steam) as well. For wastes such as bagasse, incineration has been standard practice in the industry for many years. Other wastes, including nut shells, citrus peels, and rice hulls, have also been incinerated (Inaba, Eakin, and Clark 1981).

While incineration is an attractive option for wastes with relatively low ash and moisture contents (i.e., under about 50 wt% moisture), it is not suitable for wastes with high moisture contents. Cheese whey, brewer's spent grain, and fruit pomace are examples of food processing wastes that are generally too wet to burn efficiently and cleanly.

Another potential option for organic wastes with high moisture content is anaerobic digestion. Anaerobic digestion has not been used for food processing wastes with high chemical oxygen demand (COD) because of the slow rate of conversion and the inability to convert the lignin fraction of the feedstock.

Pacific Northwest Laboratory (PNL)^(a) is developing a thermocatalytic conversion process that can convert high-moisture wastes (up to 98 wt% moisture) to a medium-Btu fuel gas consisting primarily of methane and carbon dioxide. At the same time, the COD of these waste streams is reduced by 90% to 99%. Organic wastes are converted by thermocatalytic treatment at 350°C to 400°C and 3000 to 4000 psig. The process offers a relatively simple

(a) Operated for the U.S. Department of Energy by Battelle Memorial Institute.

solution to waste treatment while providing net energy production from wastes containing as little as 2 wt% organic solids (this is equivalent to a COD of approximately 25,000 mg/L).

The process chemistry described in this report was first identified under a basic science research study for the Gas Research Institute (GRI) (Sealock, Elliott, and Hallen 1982). Further development of the concept was funded by the U.S. Department of Energy (DOE), Office of Conservation and Renewable Energy through the Biofuels and Municipal Waste Technology Division. Over 180 experiments were conducted in a batch autoclave (reactor) with various biomass materials (Sealock et al. 1988). Additional tests with a variety of different feedstocks have been funded by private industry to broaden the application of the process. Further research directed at using the same technology to destroy hazardous organics in water is being funded by DOE through the Northwest Hazardous Waste Research, Development, and Demonstration Center at PNL (Baker and Sealock 1988). Based on all of this previous research, two patent applications have been filed.

This report describes continuous reactor system (CRS) experiments that have been conducted with food processing wastes. The purpose of the CRS experiments was to provide kinetic and catalyst lifetime data, which could not be obtained with the batch reactor tests. These data are needed for commercial scaleup of the process. The CRS experiments were sponsored by DOE through the Waste Energy Reduction Division of the Office Industrial Programs, Conservation and Renewable Energy. The results of the batch reactor tests are also included here for comparison with the CRS results.

EXPERIMENTAL PROCEDURES

Two experimental systems were employed to study the conversion of food processing wastes in the thermocatalytic system. A 1-L batch reactor designed to operate at pressures of up to 5000 psig and temperatures up to 450°C was used for preliminary screening of the wastes to determine their relative reactivity and ultimate methane yields. Wastes already tested in the batch system include cheese whey, grape and cranberry pomace, brewer's spent grain, poultry and fish processing wastewaters, sludge from steam peeling of potatoes, and cow manure. The experimental system and procedures are described in detail elsewhere (Sealock et al. 1988).

The CRS contains a 1-L reactor, which was modified to operate as a continuous-flow reactor. A high-pressure metering pump is used to pump the feedstock through the reactor at rates of 0.25 to 2.0 L/h. With the CRS, the effects of reactor space velocity and reaction pressure and temperature on product yield and quality can be determined, and catalyst stability can be evaluated over extended periods of time. The reactor in the CRS is a Carberry spinning basket reactor in which the catalyst is loaded into a wire basket that rotates around a central axis. This reactor is gradientless (isothermal and uniform composition throughout) and is equivalent to a continuous stirred-tank reactor (CSTR). It operates at constant temperature with a constant composition. The high-speed rotation of the catalyst basket ensures that mass transfer does not limit the rate of reaction. These factors make the reactor ideal for obtaining true reaction rate data. Kinetic information obtained with this reactor can then be used in design equations for other types of reactors, such as a fixed-bed tubular reactor, which is more appropriate for larger-scale commercial operations. Food processing wastes that have been tested in the CRS are cheese whey and spent grain.

The wastes used for these tests were obtained from a number of food processing plants, and were shipped and stored frozen. The required amount of waste was thawed just prior to use. The fruit pomace samples were blended with deionized water to make a slurry containing approximately 10 wt% solids. The spent grain and cheese whey were charged to the batch reactor as received. The brewer's spent grain was prepared for the CRS experiments by

processing it in a batch attrition mill employing a stainless steel grinding medium. This physical pretreatment is required to facilitate handling of the waste materials and does not appear to affect the conversion process itself. Cheese whey was fed to the CRS as received. Table 1 shows the properties of the food processing wastes used in both the batch and CRS experiments.

Two different catalysts were used in this research. Harshaw Ni 1404 (68% nickel on a proprietary support) was used for the batch reactor experiments. It was crushed to a powder before being placed in the reactor. United Catalyst G-65 RS and Ni 1404 were both used for the CRS experiments. The Ni 1404 was used as 1/8-in. x 1/4-in. extrudates, and G-65 RS was used as 1/4-in. x 1/4-in. tablets.

TABLE 1. Properties of Food Processing Wastes Used in the CRS

	<u>Spent Grain</u>	<u>Cheese Whey</u>	<u>Cranberry Pomace</u>	<u>Grape Pomace</u>
<u>COD, mg/L</u>				
As Received	114-163	56-70	--	--
As Processed	100-110	69-70	--	--
<u>Solids Content, wt%</u>				
As Received	9.2-12.3	5.1-6.5	31.0	47.5
As Processed	7.5-9.2	5.1-6.5	10.4	
<u>Composition of Dry Solids</u>				
<u>Elemental Analysis, wt%</u>				
C	46.6	37.4	47.3	52.5
H	6.7	5.3	6.3	6.0
O	38.4	46.3	38.4	33.7
N	4.3	1.0	1.2	3.1
S (ppm)	0.034 (340)	0.12 (1230)	--	0.23 (227)
Ash	4.0	10.0	6.8	5.4
<u>Ash Analysis, wt%</u>				
CaO	7.9	14.6	--	15.8
K ₂ O	1.2	29.6	--	32.5
Na ₂ O	1.1	9.2	--	--
MgO	10.6	20	--	--
P ₂ O ₅	29.7	19.7	--	12.7
SiO ₂	32.0	--	--	11.9

RESULTS

The results of batch reactor tests with several food processing wastes are given here for comparison with bench-scale CRS test results.

BATCH REACTOR TESTS

In the batch tests, the feedstock and catalyst were charged to the reactor, which was then heated to the desired reaction temperature (usually 350°C to 400°C) at an average heating rate of 5°C/min. The reactor and its contents were held at the desired temperature for 60 to 90 min. The pressure in the reactor varied between about 4500 psig at 400°C and 3000 psig at 350°C. Small volumes of gas were withdrawn periodically so the progress of the reaction could be monitored. At the end of the experiment, the reactor was cooled rapidly and the product gas volume and composition were measured. The reactor contents, which included water, catalyst, and partially reacted feedstock, were removed, weighed, and analyzed for carbon content and COD. Table 2 summarizes the results of batch reactor experiments with four different feedstocks.

TABLE 2. Results of Batch Experiments

Feedstock	Temp., °C	Solids Content, wt%	COD, mg/L		Product Gas Yield, scf/lb	Product Gas HHV, (a) Btu/scf
			Feed	Effluent		
Spent Grain	395	9.2	114,000	640	12.9	572
Spent Grain	350	9.2	114,000	1,000	13.2	549
Cheese Whey	400	6.4	69,000	1,100	13.5	589
Cheese Whey	350	5.1	56,000	1,280	11.5	577
Cranberry Pomace	400	10.4(b)	--	300	14.4	597
Grape Pomace	400	12.1(b)	--	1,200(c)	14.5	577

(a) HHV = higher heating value.

(b) Cranberry pomace was 30.7 wt% solids as received; grape pomace was 54.3 wt% as received.

(c) Estimated COD; estimate was based on carbon content of effluent.

Product gas composition varied somewhat but was typically in the range of 42 to 57 vol% methane, 39 to 43 vol% carbon dioxide, and 2 to 11 vol% hydrogen. Smaller concentrations (under 1 vol%) of ethane, ethylene, and carbon monoxide were also measured. Nitrogen present in the feedstocks as protein appeared as dissolved ammonia in the reactor effluent.

The batch reactor experiments demonstrated the significant (>90%) reductions in COD attainable using the PNL thermocatalytic gasification concept. Plotting the batch reactor conversion data versus time at temperature (see Figure 1) indicated that, for some feedstocks, residence times of less than 30 min should be sufficient to obtain this level of conversion. Because of the possibility of poor catalyst contacting and mass transfer limitations, the time required for conversion in the batch reactor should be used only to compare the relative rates of different feedstocks and not as an indicator of the true rate of reaction.

FIGURE 1. Carbon Conversion Versus Reaction Time (Batch Experiments)

BENCH-SCALE CRS EXPERIMENTS

A blend of sweet and acid cheese whey (at approximately 6.4 wt% solids) and a ball-milled slurry (7.5% solids) prepared from brewer's spent grain were also tested in the CRS. Two experiments were carried out for each feedstock--one at 350°C and one at 400°C.

Table 3 summarizes the preliminary results of these CRS experiments. The residence times in the CRS are based on the volumetric flow through the reactor taken at reaction conditions, with the feedstock density assumed to be the same as water at the same conditions. The residence times in Table 3 are mean residence times for all molecules. Because of the high degree of back-mixing in the reactor there is a wide range of residence times for individual molecules. The flow rates at ambient conditions were about 850 mL/h for spent grain and 450 mL/h for cheese whey. The difference in the residence times at 350°C and 400°C is due to the difference in the density of water at the different reactor conditions.

TABLE 3. Preliminary Results from Continuous Reactor System^(a)

<u>Feedstock</u>	<u>Residence Time, min</u>	<u>Solids Content, wt%</u>	<u>COD, mg/L</u>		<u>Product Gas Yield, scf/lb</u>	<u>Product Gas HHV, Btu/scf</u>
			<u>Feed</u>	<u>Effluent</u>		
Spent Grain @ 400°C	34	7.5(b)	100,000	15,000	9.3	442
Spent Grain @ 350°C	70	7.5(b)	110,000	30,000	3.5	523
Cheese Whey @ 400°C	17	6.4	69,000	3,000	11.3	465
Cheese Whey @ 350°C	38	6.4	70,000	24,700	4.5	369

(a) Experiments at 400°C were conducted at 4100 psig; experiments at 350°C were conducted at 3100 psig.

(b) 12.3 wt% solids as received; diluted to 7.5 wt% during milling.

Rate of Reaction

Gas yields are lower and the effluent COD is higher in the CRS than in the batch reactor. This difference is primarily a function of the type of reactor. The Carberry spinning basket reactor used in the CRS is designed to obtain reaction rate data in a gradientless bed (isothermal and uniform concentration throughout the bed). By plotting the log of the rate of the conversion against the log of the reactant concentration in the reactor, the order of the reaction and the rate constant can be determined. This kinetic information can then be used to design other types of reactors, such as a fixed-bed tubular reactor, which is of more practical interest for commercial systems where high conversion is desired.

The limited experimental data obtained to date with food processing wastes are not sufficient to determine the order of the reaction or the rate constant. However, numerous experiments have been completed in the CRS with model organic compounds (research funded by the Northwest Hazardous Waste Research, Development, and Demonstration Center at PNL). The model compounds were methyl isobutyl ketone, sucrose, and p-cresol. Data for p-cresol are shown in Figure 2.

Figure 2 shows that conversion of p-cresol (based on reduction of COD) is approximately first order with a rate constant of 0.015 s^{-1} at 350°C and 0.06 s^{-1} at 400°C . Also shown in Figure 2 are the initial data points for cheese whey and spent grain. These data appear to fall in line with the data obtained with p-cresol. Making the assumption that the kinetic data obtained with p-cresol provide a good model for food processing wastes, the required residence time in a plug-flow tubular reactor can be calculated from the following equation for first-order reactions:

$$T = -\ln(1-X_a)/k \quad (1)$$

where T is the required residence time, X_a is the fractional conversion, and k is the first-order rate constant. Using this expression, the required residence time for 95% conversion in a plug-flow tubular reactor is 3.4 min. There is some indication the conversion might not be truly first order at

FIGURE 2. Comparison of Food Processing Waste Conversion with p-Cresol Conversion

higher concentrations and might tend toward zero order. Zero order indicates that the surface of the catalyst is saturated with organic and the rate is purely a function of the turnover frequency of the catalyst. The estimated residence time for 95% conversion based on zero-order kinetics is about 15 min, which represents the required maximum residence time.

The design of tubular reactors for the process development unit and commercial-scale units must consider several factors to ensure that the flow in the reactor approaches plug flow. This requires that very little axial dispersion (back-mixing) should occur in the reactor. The design of the tubular reactor must also ensure that mass transfer does not limit the rate of conversion. The appropriate flow conditions in the reactor are achieved by proper selection of the reactor length-to-diameter ratio and the catalyst size.

Catalyst Activity

The initial data obtained in tests with spent grain and cheese whey in the CRS showed that the composition of the gas in the CRS was similar to that obtained in the batch reactor, and the rate of reaction was quite high. However, during the course of each test, a steady decline in conversion efficiency and gas composition (occurring over a period of several hours) was observed. Severe softening and swelling of the catalyst pellets was observed with Ni 1404, the catalyst used for most of the batch experiments and the initial CRS tests. Another catalyst, G-65 RS, was used for the remainder of the experiments. No softening or swelling was noted with this catalyst, but the initial rate of conversion was lower and there was still a loss of activity with time.

Catalyst deactivation was not noted in tests with the organic model compounds, methyl isobutyl ketone, sucrose, and p-cresol. This suggests that components of the spent grain and cheese whey, such as alkali, sulfur, and nitrogen-containing materials, could be responsible for the deactivation. The concentration of organic in the tests with food processing wastes was higher than it was in the tests with model compounds. Saturation of the catalyst surface with organic materials could lead to accumulation of deposits and eventual fouling. A more active catalyst may be needed to keep

the surface clean. We are working with United Catalysts to determine the cause of the loss of activity and to identify or develop a catalyst suitable for this application.

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cause of the loss of activity and to identify or develop a catalyst suitable
for this application.

PROCESS DESIGN AND ECONOMICS

Except for the period (noted above) during the CRS experiments when the catalyst had deteriorated, the quality of the gas produced by the process is quite good. The product gas is primarily methane and carbon dioxide, and should be compatible with existing natural gas burners without modification of the gas nozzles. The gas can be delivered at greater than 1500 psig, and in a commercial-scale process could be expanded through a power recovery turbine to further enhance energy recovery. The product gas is essentially free of nitrogen and can be processed using a number of commercially available technologies to obtain a near-pipeline quality gas. The gas could also be burned in a diesel or spark-ignition engine to produce shaft power or electricity.

When high conversion is achieved, the effluent from the process can be discharged directly to a publicly owned treatment works or an existing onsite treatment plant. Depending on local standards and the feedstock used, it may be possible to discharge the effluent directly to a surface water stream. The significant reduction in COD in the effluent stream compared with the feed material should be of great economic benefit to processing operations that are charged for water treatment on the basis of discharge quality, or for those operations that are operating at or above the capacity of their treatment facilities.

Based on the encouraging experimental results from both the batch reactor studies (Sealock et al. 1988) and the CRS studies, PNL is working with Onsite*Offsite, Inc., a design and construction management firm, to develop and build a 1 wet ton/day, skid-mounted process development unit to demonstrate the process on a commercial scale. The facility would be skid-mounted so it could be transferred to a number of food processing facilities for testing with several different wastes. Onsite*Offsite has prepared a detailed process flow diagram, shown in simplified form in Figure 3. The process uses a recycle water stream for feedstocks where water must be added to facilitate slurry preparation and pumping. The product gas can be treated and used in a number of different ways, depending on the plant requirements. The flow diagram in Figure 3 includes a membrane separation of

FIGURE 3. Simplified Process Schematic

the gas, which would be employed where the user required a high-Btu content gas (>900 Btu/scf) or desired a relatively pure carbon dioxide byproduct stream (i.e., for controlled-atmosphere storage or carbonation). Other end configurations can be developed for different needs.

Onsite*Ofsite is commercializing the technology as the Thermochemical Environmental Energy System (TEES). They have estimated (for a private client) the cost of a 150 gpm plant (approximately 850 tons per day of waste containing 9.2% solids) to handle food processing wastes. The estimated capital cost, based on a 10-min reactor residence time, is \$10.2 million. With a 12% discounted cash flow rate of return on investment, the cost of the gas produced would be \$6.20/million Btu assuming no credit for waste disposal savings. However, if it costs \$3/ton to dispose of the waste, the net cost of the gas produced falls to about \$3/million Btu; if it costs \$5/ton to dispose of the waste, the net cost of the gas produced falls to \$1/million Btu. This relationship is shown graphically in Figure 4. The graph does not credit the TEES process for integrated applications which would provide for potential electrical generation and use of the hot process water produced.

The economics of the TEES process are very site specific and are dependent primarily on the cost for waste disposal and the incremental cost of energy at individual facilities. The waste disposal cost and the cost of

FIGURE 4. Cost of Producing Gas with the TEES Process as a Function of Waste Disposal Costs

energy for several potential applications of the TEES process are shown in Table 4. The cost of fuel used at these facilities ranges from \$2.40 to \$5.00 per million Btu. Waste disposal costs have an even wider range from \$26.60 per ton for cranberry presscake to -\$13.80 per ton for spent grain currently sold as cattle feed.

TABLE 4. Current Fuel and Waste Disposal Costs for Selected Industries

<u>Waste Type</u>	<u>Location</u>	<u>Fuel Type</u>	<u>Fuel Cost, \$/MM Btu</u>	<u>Waste Disposal Costs, \$/wet ton^(a)</u>
Brewer's Spent Grain	Midwest	Coal	--	-13.3(b)
Brewer's Spent Grain	California	Natural Gas	5.00	-13.8(b)
		No. 6 Fuel	3.22	
		Oil		
Cranberry Presscake	Northwest	Bunker Oil	4.90	26.6
Fruit Pomace	Northwest	Natural Gas	4.80	3.3
Spent Coffee Grounds	California	No. 6 fuel	2.45	7.1
		Oil		
Cattle Feedlot Manure	Southwest	Natural Gas	3.75	1.5
Grape Pomace	California	Natural Gas	2.40	-38.0
Pineapple Silage Water	Hawaii	No. 6 Fuel	2.45	0(c)
		Oil		
Cheese Whey	California	Natural Gas	2.80	0.6
Cheese Whey	Northwest	Natural Gas	2.75	1.8
Water Treatment Sludge	Midwest	Natural Gas	2.80-4.77	
		No. 2 Fuel	3.36	2.5
		Oil		
		Coal	1.70	

(a) Negative number indicates that waste is currently being sold.

(b) Does not include cost of dewatering and drying the feed, and the associated wastewater treatment costs.

(c) 4 1/2 million gallons per day of silage water are currently being disposed of by deep well injection. Well injection is expected to be stopped in the near future.

For several companies, the TEES process would be economical today, e.g., for disposing of cranberry presscake and fruit pomace in the Northwest where both energy costs and waste disposal costs are high. Still other companies are quite interested in the process for future use because they anticipate a significant increase in their waste disposal and energy costs. Several examples of potential changes are listed below:

- Pineapple silage water is being disposed of by deep well injection, which is expected to be halted in the near future.
- Spent coffee grounds can no longer be "land treated" and are accumulating at a plant site until another disposal method is identified.
- Effluent water from spent grain dewatering is being disposed of in a publicly owned treatment works, and the cost is escalating rapidly.
- Cheese whey is being "land treated," which is likely to become more expensive or stopped completely.
- Grape pomace cannot always be sold and is accumulating at some plant sites.

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

The thermocatalytic system being developed by PNL for conversion of high-moisture food processing wastes and other biomass streams permits energy recovery from wastes that are too wet for incineration. The product gas can be used as a replacement for natural gas in most fired equipment, as a source of pure methane and carbon dioxide, or burned in an engine to produce shaft power or electricity. When high conversion is achieved, the effluent water will require little or no additional treatment. The process becomes very attractive economically as the cost of waste disposal increases.

Work is continuing at PNL to develop more complete engineering data for the process, including reactor design data, catalyst lifetime, and optimization of process parameters. These data will be used to design a process development unit that can be used to demonstrate the process on a near-commercial scale.

The primary objectives for continued development of the process in FY 1989 are listed below:

- Obtain additional rate data in the CRS with spent grain, cheese whey, and other food processing wastes.
- Determine the cause of catalyst deactivation and identify or develop catalysts resistant to deactivation (with United Catalysts).
- Work with Onsite*Offsite to obtain funding for construction and operation of a 1 wet ton/day process development unit (PDU).
- Begin construction of the PDU.

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