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Low-Temperature Catalytic Gasification of Food Processing Wastes

1995 Topical Report

D. C. Elliott
T. R. Hart

August 1996

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest National Laboratory
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Pacific Northwest National Laboratory
Richland, Washington 99352

Summary

Process development research is continuing at Pacific Northwest National Laboratory on a low-temperature, catalytic gasification system that has been demonstrated to convert organics in water, such as food processing wastes, to useful and environmentally safe gases. The system, licensed in the U.S. and Canada under the trade name Thermochemical Environmental Energy System (TEES®),[a] treats a wide variety of feedstocks ranging from hazardous organics in water to waste sludges from food processing. The current research program is focused on the use of continuous-feed, tubular reactors systems for testing catalysts and feedstocks in the process.

Results of the testing reported here show that food processing waste feedstocks, ranging from sugar syrups to potato waste, can be processed to >95% reduction of chemical oxygen demand (COD). The estimated residence time is about 10 min at 350°C and 21 MPa, not including about 3 min required in the preheating zone of the reactor. The liquid hourly space velocity varies around 2 L feedstock/L catalyst/hr, depending on the feedstock. The product fuel gas contains from 40% to 50% methane, depending on the feedstock. The balance of the gas is mostly carbon dioxide with <10% hydrogen and usually <1% ethane and higher hydrocarbons. The byproduct water stream carries residual organics from 100 to 1000 mg/L COD, again depending on the feedstock.

The TEES process has now been demonstrated in continuous-feed, fixed-bed catalytic reactor systems on four scales of operation ranging from 0.03 L/hr to 33 L/hr. The systems have been operated with consistency at conditions of 350°C and 21 MPa. The demonstrated effective heat recovery in a tube-in-tube heat exchanger should be beneficial for economical operation of TEES. Aqueous effluents with low residual COD (<1000 ppm) and a product gas of medium-Btu quality have been produced.

Development has progressed to the initial phases of industrial process demonstration. Testing of industrial waste streams is underway at bench and engineering scales. Plans for FY 1996 include operating an Industrial Onsite Demonstration Unit at industrial sites of cooperating companies. However, these tests will not include food processing wastes within the U.S. Department of Energy-sponsored program of research and development. Budget limitations and a revised program focus have eliminated food processing wastes from further testing for the foreseeable future. Alternative sources of funding will be required to continue with food processing waste testing demonstrations.

(a) TEES is a registered servicemark of Onsite*Ofsite, Inc., Duarte, California, the process licensee.

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We have had significant industrial input and support for this project, in particular Dr. J. Strasser, Process and Equipment Technology; Dr. I. Feins, Engelhard Corporation; and Dr. P. Polanek and Mr. J. Markert, BASF Corporation. The industrial input included catalysts for testing and insight on both the results of the tests and the utility of the results. We acknowledge the support and participation through supplying waste samples by Anheuser-Busch Companies, St. Louis, Missouri; Del Monte Foods, Toppenish, Washington; Lamb-Weston, Inc., Richland, Washington; and TreeTop Corporation, Selah, Washington.

We also acknowledge the continuing support of our process licensees, Onsite*Ofsite, Inc., Norman Banns, president; and Alligator Corporation, Richard Locke, president.

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Introduction

The catalytic gasification system described in this report has undergone continuing development and refining work at Pacific Northwest National Laboratory (PNNL)[a] for over 16 years. The original 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).

From the fundamental research evolved the concept of a pressurized, catalytic gasification system for converting wet biomass feedstocks to fuel gas. Extensive batch reactor testing [2] and limited continuous reactor system (CRS) testing [3] were undertaken in the development of this system under sponsorship of the U.S. Department of Energy (DOE). A wide range of biomass feedstocks were tested, and the importance of the nickel metal catalyst was elucidated.

Specific use of this process for treating industrial processing wastes was first studied by Baker et al.[4] using food processing wastes. That early work focused on brewer's spent grain and cheese whey. Batch and continuous stirred-tank reactor tests provided useful design information for evaluating the preliminary economics of the process. Long-term catalyst stability was identified as an area needing further attention. Continuing work at PNNL has addressed improvement of the catalyst [5] and scaling the system to long-term industrial needs. Scaling has included both bench-scale and engineering development systems [6,7].

The process is licensed in the U.S. and Canada as the Thermochemical Environmental Energy System (TEES®) to Onsite*Ofsite, Inc., of Duarte, California, a turnkey design engineering and construction management firm. In 1989 TEES was recognized with an **R&D 100 Award** from *Research and Development Magazine* as one of the top 100 new technical developments to reach the marketplace. In December 1995, Alligator Corporation of Richland, Washington, acquired a license for the technology in several countries in the Far East and is currently actively marketing the technology.

This report is a follow-on to previous Interim Reports [8-10], which reviewed the results of the studies conducted with batch and continuous-feed reactor systems from 1989 to 1994, including much work with food processing wastes. The discussion here provides details of experiments on food processing waste feedstock materials, exclusively, that were conducted in batch and continuous flow reactors. However, DOE-sponsored research in this application of the technology was suspended by direction of DOE-Headquarters in mid-1995.

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Technology transfer issues are also addressed here, including both domestic and foreign applications for the technology. Of particular importance is the focus on operating the Industrial Onsite Demonstration Unit in cooperation with industrial participants.

Conclusions and Recommendations

As a result of the work presented here, we can make the following conclusions and recommendations.

Conclusions

- Organic materials in food processing wastes can be converted to near extinction in TEES. High methane yields are produced from reaction of the organics with water, and the product effluent is substantially clean.

- Certain food processing waste components, for example, calcium and sulfur, may require pretreatment to remove them before the waste is processed.

- The tubular reactor design with the fixed-bed of pelletized catalyst has been scaled up, including heat recovery by exchange between the feed and the reactor effluent, and has been demonstrated successfully.

Recommendations

- Continue bench-scale testing of TEES to include other feedstocks of industrial interest and verify catalyst compatibility.

- Evaluate ion exchange further to determine its applicability as a pretreatment method for calcium and sulfate removal from food processing wastes.

- Perform long-term tests to evaluate catalyst stability with industrial waste streams.

- Operate the Industrial Onsite Demonstration Unit to provide in-plant experience with TEES for potential users.

Batch Reactor Studies

Batch reactor tests were an important beginning step in developing industrial systems based on this technology. These studies provided a quick and cost-effective method to determine the applicability of TEES to the specific feedstock. While the tests gave only a preliminary view of the kinetics and the catalyst compatibilities, they also provided products for analysis.

Summary

The batch reactor was a standard Autoclave Engineers bolted-closure vessel modified in our laboratory to allow samples to be withdrawn at reaction conditions. With this capability, a single batch cycle could provide several data points over time to allow a preliminary assessment of kinetics. The reactor and procedure are described in detail in an earlier report [6]. For the experiments, a nominal 10 wt% organic in water feed was used with a standard nickel catalyst (Engelhard Ni-0750, powdered). Standard test temperature was 350°C for 1 to 2 hours. Multiple gas samples were recovered during each test to increase the amount of information gained from each test.

Of the 12 total tests, the experiments included nine different industrial waste streams. Industrial input to this research program was an important aspect. For these batch tests, waste samples were received from four different producers.

Preliminary Feedstock Tests

A number of different food processing waste feedstocks were evaluated in the batch reactor system. The list of feedstocks and results of the batch reactor tests are shown in Table 1. A primary measure of the process is the reduction in the chemical oxygen demand (COD). The time at temperature given in the table is the length of time for the reaction to come to completion as indicated by an increase in reactor pressure and change in gas composition. The residual ammonia content of the process effluent was not measured in all cases, but the range of values given is believed to be representative of expected levels for all the feedstocks shown.

The corn DAF (dissolved air flotation) float came from a new process unit installed by Del Monte Foods at Toppenish, Washington, to clarify the wastewater. In the DAF system, sulfuric acid is added to facilitate the clarification; therefore, the DAF float, a starch product, also contains sulfate. The resulting catalytic inhibition is seen, as expected, in the reduced methane concentration and the high residual COD.

The apple processing wastes were provided by TreeTop from several plant locations. The apple materials (pomace and seeds/peel) process fairly well in our system with moderately good gas compositions. The apple biosludge shows signs of catalyst inhibition, which is even more pronounced in the

Table 1. Preliminary Feedstock Test Results

Feedstock	Time at Temperature	Product Gas Composition, vol%					Destruction of COD, %	Product COD, ppm	NH_4^+, ppm
		CH_4	CO_2	H_2	C_2	BF			
Corn DAF	110 min	35.7	50.4	11.0	0.7	1.7	84.6	18,750	NA
Apple pomace	60 min	40.3	51.4	6.4	1.0	0.8	97.1	5,800	NA
Apple seed/peel	100 min	35.9	57.0	5.4	0.9	0.8	98.4	4,100	NA
Apple biosludge	45 min	33.4	42.0	23.8	0.4	0.4	99.2	550	NA
Apple wastewater	48 min	9.4	38.5	51.8	0.0	0.0	99.6	32	NA
Potato crumbs	105 min	54.2	38.7	6.2	0.4	0.4	99.8	290	NA
Potato piece	100 min	45.9	45.4	7.0	0.7	0.7	99.7	475	NA
Potato peels	60 min	43.1	45.1	10.0	0.8	1.0	99.2	680	6,758
Potato biosludge	80 min	33.9	38.2	24.8	1.4	1.6	83.4	18,750	~1,500
Onion, yellow	60 min	34.2	48.9	15.0	1.2	0.6	99.7	650	1,490
Dairy wastewash	100 min	3.6	55.0	41.4	0.0	0.0	97.2	60	118

BF = backflush from chromatography columns, assumed to be higher hydrocarbons.
NA = not analyzed.

apple wastewater. The methane content is low and the hydrogen is high, as methane formation ceased rather early on in the tests. A high percentage of COD reduction is seen in both cases, however.

The potato tests were performed with feedstocks from the Lamb-Weston potato processing plant in Richland, Washington. The first test was performed with the crumbs of spiced frozen potato product, and the second test was made with larger pieces of the same product. The reactivity of the two tests is comparable, but the methane yield from crumbs appears inordinately high, suggesting a higher level of residual frying oil. These tests can be compared with earlier tests using the crumbs from the potato fryer [10]. All the tests suggest, however, that potato crumbs, as produced, would be a good TEES feedstock. The potato peels seem to exhibit only slightly less activity. There is a suggestion of catalyst inhibition in the higher hydrogen concentration and in the early time of cessation of reaction. The potato biosludge test shows a distinctly reduced level of activity. The effluent COD is high; hydrogen product gas is high; and methane is low. The effect may result from catalyst fouling by inorganic components or by sulfur, which is expected to be present in this stream.

Both the onion and dairy waste show high levels of COD reduction. However, the gas product composition suggests a significant inhibition of the nickel catalyst relative to the methane formation reactions. Sulfur compounds are expected to be present in significant quantities in both of these feedstocks and have likely poisoned the catalyst. Longer-term testing is required to verify this speculation.

Continuous-Flow Reactor Waste Treatment Studies

The continuous-flow reactor studies were designed to provide information for further scaling the TEES process to industrial use.

Summary

Gasification tests were carried out in a fixed-bed catalytic tubular reactor. The bench-scale unit, described in detail in an earlier report [8], consisted of a 6 ft long x 1 in. I.D. tube that was fed from a cylindrical feed tank by a reciprocating plunger pump. The reactor was heated by an electrical resistance furnace and served as both the preheater and the reactor. Pressure was controlled in the reactor by a dome-loaded, back-pressure regulator. The pressure regulator was followed by a condenser/separator system in which liquid samples were recovered. Uncondensed product gas was passed through flow meters and vented. The bench-scale reactor was operated in three experiments lasting from 4 to 5 hours for a total operating time on stream of 14 hours.

A mobile Industrial Onsite Demonstration Unit was designed and built based on the tubular reactors being used at PNNL. This mobile unit is a fifth-wheel mounted trailer containing the processing equipment designed at a scale of 60 gallons per day of wet feed. The test system is equipped with four 3½ ft x 1½ in. I.D. fixed-bed tubular reactors and supporting equipment to convert waste to clean water and fuel gas. Design working conditions for the reactors were 350°C at 22.1 MPa. The Industrial Onsite Demonstration Unit was operated in one test with a simulated food processing waste lasting for a total on-stream operating time of 60 hours.

Bench-Scale Reactor Tests

Tests in the bench-scale reactor were performed primarily to verify operability in continuous TEES systems. These tests also provided a preliminary verification of catalyst stability in processing the specific feedstock. Results of the bench-scale tests are given in Table 2.

Table 2. Bench-Scale Food Processing Waste Test Results

Feedstock	Catalyst	Product Gas Composition, vol%					Destruction of COD, %	LHSV,[a] L/L/hr	Gasification[b] of Carbon, %
		CH_4	CO_2	H_2	C_2	BF			
Potato peel/slice	BASF[c]	50	43	2.9	2.0	1.9	95.4	2.67	103
Potato peel	BASF[c]	48	50	2.0	0.1	0.3	99.3	6.52	15

(a) Liquid hourly space velocity.
(b) Gas yields include a correction for carbon dioxide dissolved in the aqueous effluent as alkali and ammonium bicarbonates.
(c) Modified BASF Corporation catalyst.

Pumping difficulties developed during the testing. In the first potato test listed in Table 2, plugging occurred after switching to the apple peel/seeds feedstock, and the system was operating for about ½ hour. In a second test, not reported in Table 2, the apple peel/seed feed began to plug at the reactor entrance after only a few minutes on line. As a result, no apple processing data in the continuous reactor could be generated. After the pressure buildup, several hours of attempts to purge the system with water and organic solvent solution proved unsuccessful. In the third test, intermittent pressure spikes occurred while feeding potato over a 1½-hour period. After a 2-hour attempt to clean the reactor bed with a dilute acid feed, pressure fluctuations were even greater for a 1-hour attempted operation. No useful information could be obtained, and the test was terminated. These plugging problems contrast with earlier results in processing both fried potato waste and brewer's spent grain in which no plugging problems occurred.

Inspection of the catalyst bed after the tests showed obvious evidence of the plugging. A carbonaceous material sealed the bed in a plug at an early point of the reaction zone. The hard tar or coke was removed from the catalyst in breaking apart the plug. Carbon deposition in the catalyst pellets was minimal, about 3 wt%, as in the fresh catalyst which contained graphite. Agglomeration of the nickel crystallites was also minimized by the use of the modified BASF catalyst. Samples from the first two tests ranged from 14- to 18-nm average nickel crystallite size by X-ray diffraction (XRD) line broadening approximation, while the catalyst in the actual plugged area in the third test had only 10-nm nickel crystallites.

Industrial Onsite Demonstration Reactor Tests

Tests in the Industrial Onsite Demonstration Unit were performed primarily to verify operability of the reactor system. The first test was performed with a dilute solution of orange drink syrup. Processing rates and temperatures were higher than usual during this test, as seen in Table 3. The operating pressure was somewhat unsteady. However, the test gave useful operating experience and was valuable in verifying the operability of the unit. The operation was relatively smooth for the first actual process test in the Unit. The results in Table 3 are for portions of the run with steady operations and represent 6- to 11-hour windows during the first 39 hours of a 60-hour test. The high initial activity of the catalyst was soon damped during the processing. By the end of the run, the activity of the catalyst had fallen much lower than shown here to a <62% conversion of COD at a 3.3 liquid hourly space velocity (LHSV).

Table 3. Results from the Industrial Onsite Demonstration Unit Tests

Temperature, °C	Pressure, MPa	Product Gas Composition, vol%					Destruction of COD, %	LHSV, L/L/hr
		CH_4	CO_2	H_2	C_2	BF		
360	20.0	41	46	13	0.1	0.0	97.9	5.16
360	20.4	40	43	16	0.1	0.0	96.9	2.59
366	19.3	23	38	38	0.5	0.8	95.6	3.11

Analysis of the BASF catalyst following the test showed significant evidence of deterioration. Calcium carbonate deposits were evident, derived either from the orange drink concentrate itself (0.067 wt% ash) or the city water used in the feedstock makeup (81 mg/L Ca). Nickel crystallite growth was also evident, having increased from 5.9 nm in the fresh catalyst to 15 to 25 nm in the several samples from the different reactor beds, based on XRD line broadening. Compared with earlier test results [7], the nickel had sintered less than the expected 35-nm average crystallite diameter. Since this result would indicate less loss of catalyst activity, the calcium deposits were apparently a significant factor in the degree of activity loss.

Technology Transfer

Transferring the TEES technology out of the laboratory and into commercial use is the primary goal of this research project. In order to achieve this goal, we have made several efforts to broaden the exposure of TEES to potential users. Also, we have made the primary goal of our research program the demonstration of TEES technology with industrial partners.

An important part of the Technology Transfer effort is responding to requests for information. From PNNL, publications and presentations have been generated to describe the technology and development activities. The process licensees, Onsite*Ofsite, Inc., and Alligator Corporation, use a more direct marketing approach through paid advertisements and direct mailings and contacts with industry. Furthermore, in addition to earlier citations, writeups have appeared in *Popular Science* (December 1994) and *Bottom Line Business* (August 1995).

Altogether, PNNL received and responded to 33 requests during 1995 from parties with biomass-type wet waste disposal interest. Table 4 gives an idea of the geographical distribution and the applications being considered.

Green Brewery Concept

A major effort was expended by PNNL in organizing the American brewing industry to initiate a pollution prevention effort. Executives of major brewers (Anheuser-Busch Companies, Miller Brewing Company, Coors Brewing Company, Stroh Brewery Company) and a representative of microbreweries, Brewers' Association of America, agreed to gather in Chicago to discuss development of a co-operative industrial review for process improvement regarding energy efficiency and waste reduction.

During the planning activities for the meeting, Anheuser-Busch, after years of cooperative efforts with PNNL, including funded research, proposed that TEES could be integrated in brewing operations to reduce wastes, minimize effluent streams from the brewery, improve energy independence, reduce greenhouse gas production, and improve overall economics and efficiency. With the cooperation of the other brewing operators, an industry-wide Cooperative Research and Development Agreement (CRADA) was anticipated to involve TEES in addressing the industry's needs in the area of pollution prevention. Unfortunately, on the eve of the meeting, the cooperation was squelched by budget limitations and DOE's current focus on industries other than food processing.

Table 4. Information Requests for TEES

Region	Type of Company	Waste Feedstock
	Domestic	
Kansas	engineering consultant	potato waste
North Dakota	engineering consultant	wet biomass
Virginia	waste treatment	biomass conversion
North Carolina	entrepreneur	livestock waste
Arkansas	industrial consultant	wastes
Indiana	entrepreneur	wastes
Florida	metro government	wastes
Colorado	entrepreneur	wastes
New Jersey	scrap reprocessor	wastes
Nebraska	manufacturer	wastes
California	industrial consultant	wastes
Minnesota	industrial consultant	wastes
Washington	industrial consultant	wastes
Kentucky	industrial consultant	clothes manufacturing wastewater
New York	state energy office	cheese whey
Illinois	farmer	wood slurry
Virginia	energy consultant	wet biomass
Michigan	state energy office	soybean wastewater
Ohio	chip manufacturer	potato wastes
California	county government	fish and cheese wastes
California	industrial consultant	algae
North Carolina	attorney	wastes
New Jersey	EPA data base	wet organic wastes
	Foreign	
Netherlands	environmental engineer	algae, manure
Switzerland	environmental engineer	wastes
Canada	environmental engineer	wastes
Canada	process designers	food wastes
India	biomass energy developers	wastes
Australia	process designer	palm oil, tannery waste
Indonesia	industry institute	palm oil
Greece	environmental engineer	winery wastewater
Japan	environmental engineer	food wastes
India	engineering consultant	textile wastes, sugar factory wastes

Industrial Onsite Demonstrations

A major new thrust of our Technology Transfer effort has been the operation of the Industrial Onsite Demonstration Unit. PNNL entered into a CRADA with Onsite*Ofsite so that TEES can be seen and operated by the potential users in industrial settings. The CRADA provides the framework for Onsite*Ofsite to use the Unit in cooperation with PNNL to demonstrate and market the technology to industry in concert with DOE.

The benefit to DOE obtained through this CRADA is the transfer of a DOE-enhanced technology to the private sector. Utilization of this technology will provide an energy savings for industry through conversion of waste organics into fuel gas. In addition, the technology will result in a cleaner environment through the utilization of waste for energy production.

The benefit to Onsite*Ofsite is the demonstration of the technology in an industrial setting. This demonstration is expected to facilitate the marketing of the technology, which, in turn, leads to sublicensing the technology or sales of equipment or services.

Using the mobile unit should lead to a broader application of TEES. For DOE, it may be used for demonstrations within the Alcohol Fuels Program, for example.

Operations began in the Industrial Onsite Demonstration Unit just before the end of FY 1994. The initial testing was performed at Onsite*Ofsite's facilities in Duarte, California, by Onsite*Ofsite personnel to first develop operating capabilities, as well as an operating history for the Unit.

NICE^3 Proposal with Idaho Potato Research Committee

A presentation was made to the Potato Research Committee of the Idaho Association of Commerce & Industry in Boise on December 15, 1994. Technical results of PNNL research on potato waste stream processing by TEES were presented. The TEES option of potato peel waste treatment was discussed and a proposal suggested for development through DOE's NICE^3 program (National Industrial Competitiveness through Energy, Environment, and Economics). Proposal preparation continued through January with input from Onsite*Ofsite, Inc.; Idaho Supreme Potatoes, Inc.; the Idaho State Department of Water Resources; and PNNL. Unfortunately, the proposal, which must originate at the state level, was not completed on time in Idaho for submission to meet the NICE^3 deadline in January 1995.

Continuing contacts with the Idaho potato growers brought to light the research on membrane separation and recycling of water from potato starch wastewater. The concentrated organic stream would be a prime candidate for TEES processing in lieu of the entire wastewater stream. Arrangements were being made for a shipment of the concentrate to PNNL for testing in May 1995, but the work was halted by the DOE suspension of research on food processing wastes.

Future Research

Process development research will continue on TEES in FY 1996. DOE-sponsored testing of industrial waste streams will focus on chemical manufacturing wastewaters, excluding food processing wastes. This distinction was made in light of research budget limitations and the internal DOE Energy Efficiency and Renewable Energy/Office of Industrial Technologies focus on Industries of the Future, which do not include the food processing industry.

Further development work for food processing wastes will require an alternate funding source. Alligator Corporation, the new process licensee for the Far East, has identified a large market for the technology. They will be seeking support for continued process development based on feedstock materials such as palm oil and sugar cane processing wastewaters and brewery wastes.

Related Presentations and Publications

Within this project, during 1995, one presentation manuscript was prepared and will be published:

- "Chemical Processing in High-Pressure Aqueous Environments: Low-Temperature Catalytic Gasification" by D. C. Elliott and L. J. Sealock, Jr. Presented at the First International Conference on Science, Engineering and Technology of Intensive Processing, September 18-20, 1995, Nottingham, U.K.; to be published in *Chemical Engineering Research and Design*.

Three other formal presentations were made:

- "Innovative Wet Waste Treatment with TEES." Presented by Douglas C. Elliott at the Process Water and Energy Efficiency for the Northwest Food Processing Industry Conference, March 7, 1995, Kennewick, Washington.

- "Thermochemical Environmental Energy System (TEES)." Presented by Douglas C. Elliott at the Washington State Energy Office Northwest Industrial Energy Forum, April 20, 1995, Moses Lake, Washington.

- "Low-Temperature Catalytic Gasification of Wet Industrial Wastes." Presented by Douglas C. Elliott at the OIT Industrial Pollution Prevention Program Review, May 9-11, 1995, Chicago, Illinois.

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