## Title

DIRECT LIQUEFACTION OF BIOMASS - CORRELATIVE ASSESSMENT OF PROCESS DEVELOPMENT



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## Publication Date

1979-06-01

Lawrence Berkeley Laboratory
UNIVERSITY OF CALIFORNIA

ENERGY & ENVIRONMENT
DIVISION

Presented at the 3rd Annual Biomass Energy Systems Conference, Golden, CO, June 5-7, 1979

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June 1979



Prepared for the U. S. Department of Energy
under Contract W-7405-ENG-48

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# DIRECT LIQUEFACTION OF BIOMASS - CORRELATIVE ASSESSMENT OF PROCESS DEVELOPMENT

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## Abstract

Lawrence Berkeley Laboratory was assigned by DOE the responsibility of directing and technical monitoring of the projects dealing with direct liqeufaction of biomass. LBL found it desirable to fulfill its responsibility by undertaking a continual correlative assessment of process development activities and by initiating and undertaking studies to fill the missing gaps in order to bring the biomass liquefaction program to a successful conclusion speedily. This presentation deals with the correlative assessment efforts of LBL in the development of an oil-from-biomass technology.

## CORRELATIVE ASSESSMENT OF PROCESS DEVELOPMENT

The wood-to-oil process development unit (PDU) located at Albany, Oregon was designed on the basis of bench scale batchwise experiments conducted at the Bureau of Mines. The translation of the results of batchwise experiments into a continuous unit required a lot of guesswork. Major process units such as blender, preheater, pressure letdown vessel, and centrifuge were designed on the basis of meager or no data. Successful operation of these units and their modifications required engineering R & D before they could be operated successfully.

Of equal concern was the fact that the fate of the biomass liquefaction program depended upon the fate of a single process conceptualized by the Bureau of Mines. The Albany PDU was primarily designed to evaluate the technical feasibility of the Bureau of Mines process and to gather sufficient data in order to assess the economic feasibility of the process and to provide a data base for the design of a demonstration unit. If was tacitly assumed that the chemical feasibility of the process was a certainty. Preliminary experiments conducted at Albany cast some doubts on the chemical feasibility of the process. Accordingly process modifications and/or new process options had to be researched and a data base provided for testing at Albany.

Lawrence Berkeley Laboratory (LBL) was given the responsibility of directing and technical monitoring of the projects dealing with direct liquefaction of biomass. In discharging this responsibility LBL interfaced with the Biomass Energy Systems Branch of DOE, the operators of the Albany, Oregon PDU and with institutions providing supporting research and other services. Interfacing with the operators of the PDU required undertaking complementary studies, providing engineering support, providing input from supporting basic research, and undertaking any other activity to speed the development program. This presentation deals with the correlative assessment activity undertaken by LBL.

The results of Phase I operations (modify, commission, and conduct preliminary experiments) more or less identified the areas of concern. They pertained to:

- Mechanical problems
- Unit operations
- Chemistry of the process(es) and physical chemical aspects,
- Multiplicity of the variable parameters.

Mechanical problems proved to be formidable; they were largely handled by the operators of the facility. The unit operational problems encountered were both of mechanical and chemical engineering nature. In this respect LBL interfaced with Rust Engineering Company. For example, our mechanical engineer provided the design for the modification of the wood flour feeding system and worked with the Rust staff in the analysis of problems encountered with the preheater, pressure letdown vessel and thermal stresses, etc. Recognizing that the design of major process units such as preheater, pressure letdown system, and centrifuge was based on meager data or guess work, LBL developed procedures for the evaluation of the performances of the various process units. We may cite the centrifuge as an example. The centrifuge of the Albany PDU, a three phase unit, did not work and was removed from the system. This centrifuge was supposed to separate the solid residue as a sludge, and an aqueous phase containing the catalyst from the flash tank bottoms to provide a clear oil for recycling and as a product. A three phase centrifuge is as complicated a unit as a fractionation column. An LBL analysis indicated that the centrifuge chosen is not likely to separate an aqueous phase; it may separate the oil formed in the process from the startup oil if the properties of the two oils are very different and eventually become inoperative or useless as the startup oil becomes depleted.

As Dr. Larry Schaleger informed you in his presentation, LBL developed a modified process option that requires pretreatment of biomass. Although two large stirred autoclaves were available at Albany

for wood pretreatment, they were not designed for the operational procedures developed at LBL. Accordingly the LBL and Rust engineers worked together to modify the autoclaves available at Albany and to develop operational procedures that would meet the reaction conditions imposed by the chemistry of the pretreatment.

Dr. Manu Seth informed you of the results of LBL's investigations on the chemistry of the liquefaction of biomass. However promising, any new process or modified process option requires scrutiny regarding the adequacy of the data base provided for evaluation at Albany. The bench seale results have to be translated into the operation of a continuous unit such as exists at Albany, Oregon with minimum modifications, as major modifications are time consuming. The translation involves the specification of the following:

- o Preliminary modifications of the PDU, e.g., piping and instrumentation.
- o Startup procedures.
- o Operational procedures
- o Likely deviations from the anticipated procedures.
- o Operating conditions.
- o Data to be recorded and its frequency.
- o Equations for the analysis of the data recorded.

It is needless to elaborate that in order to develop valid equations for the analysis of the data recorded, one must consider heat and mass balances and kinetics of the reactions. The last factor is largely unknown to begin with, and in a strict sense, probably will remain unknown whether or not a viable process evolves from the effort. However, effective kinetic parameters must be formulated, realizing what can or cannot be measured or analized on the basis of their sensitivity to the operating conditions imposed. Process optimization boils down to finding the operating conditions that render the process most attractive economically. In the operating directives issued for testing the feasibilities of the Bureau of Mines process and a modified version of the Bureau of Mines process, the points raised above have been considered in detail.

The last concern that we cited in this presentation, i.e., the number of variable parameters, are shared by LBL and Rust Engineering Company. To be specific, they are listed below:

VEHICLE OIL/BIOMASS RATIO
CATALYST/BIOMASS RATIO
SYNTHESIS GAS/BIOMASS RATIO
SYNTHESIS GAS COMPOSITION
SLURRY FEED RATE ( RESIDENCE TIME IN PREHEATER)
PREHEATER EXIT TEMPERATURE
PRESSURE
REACTOR TEMPERATURE
REACTOR INVENTORY (RESIDENCE TIME IN REACTOR)
TYPE OF BIOMASS
TYPE OF STARTUP OIL
TYPE OF CATALYST

The experience at Albany has shown that the number of test runs that can be conducted is limited, i.e. about one run a month. About twelve runs that can be conducted between now and August of 1980 will not be sufficient to evaluate the influences of twelve variables. Considering the chemistry of the reactions taking place, the unit operations involved in the process, and the confidence desired, we believe about 60 test runs have to be conducted. For this reason LBL has designed and constructed a mini PDU that in many respects simulates the Albany PDU. In the second part of his presentation, Dr. Seth has described two additional pieces of equipment designed to simulate continuous operations by batchwise experiments. Our plans call for screening the influences of as many parameters as possible in order to zero in on the conditions of the critical test runs that can be conducted at Albany. Of course we plan to establish a one-to-one correspondence between the results that can be obtained at Berkeley and Albany. For wood hydrolysis, for example, we started the tests in a 400 ml autoclave and graduated to one liter and later to a 10 gallon autoclave. Having established an identity in the results obtained, we requested that a test run be conducted at Albany using the 400 gallon autoclaves of the PDU. We are happy to report that the results obtained at Albany were identical to those obtained at Berkeley.

In summary, LBL found it highly desirable to fulfill its responsibility in the DOE's direct liquefaction of biomass program by undertaking a continual correlative assessment of process development activities and by initiating and undertaking studies to fill the missing gaps to speed the course of the program.

This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

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