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Liquefaction of Biomass

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INTRODUCTION

The precarious state of energy resources in the United States and indeed in the world is of much concern to most knowledgeable persons. There is a general realization that additional energy supplies of all kinds will be needed if there is to be a modest increase in or perhaps even maintenance of the standard of living of all peoples. The inordinate proportions of oil and gas that are being used today to satisfy these needs have led to many political and economic problems. Indeed all of the major energy sources are beset with difficulties of one kind or another that prevent any one from being a panacea for the world's energy needs. Consequently it is generally accepted that intensive efforts must be devoted to developing energy sources that are now considered minor or even exotic. This has been a keynote in the national energy plans that have been set forth by the U. S. Department of Energy.

The recent trends in energy consumption in the U. S. are illustrated in Table 1. It is evident that major reliance is being placed on sources that involve serious problems of various kinds. It is also apparent that the suggested minor sources such as the various solar-related types and the geothermal do not now have an appreciable role. There is a rapidly increasing proportion of popular opinion that advocates immediate major attention to development of such sources.

One of the most important of the solar technologies is fuels from biomass. This includes wood, agricultural and municipal wastes and animal wastes; wood and related wastes are and will continue to be the principal source of domestic biomass. In 1900, wood supplied 25 percent of the total energy in the U. S.; as late as 1940, 20 percent of the homes used wood for space heating. Today wood contributes less than 1.5 percent of all U. S. energy or about the equivalent of 1/2 million barrels of oil per day. It has been estimated that the potential supply of energy from commercial forest lands in the U. S. that are available for fuel would be about 3 million bbl/day of oil equivalent. It was also estimated that biomass could provide 6 percent of U. S. energy needs by the year 2000.

The use of biomass as an energy source offers many benefits. Among these are:

- o There are large supplies of biomass, including current wastes and potential energy crops.
- o It is a renewable, non-depleting resource.
- o It is an indigenous resource that can reduce our dependence on foreign fuel.

Table 1. U. S. Energy Consumption, 1977.

Sources	Millions of Barrels per Day of Oil Equivalent	Percentage of Total	Quantity in Units Commonly Used for Each Source
Petroleum	18.4 ^a	50	18.4 million barrels per day
Natural Gas	9.2 ^b	25	19.2 trillion cubic feet per year
Coal	6.7 ^c	18	625 million tons per year
Nuclear	1.3	4	251 billion kilowatt hours per year
Hydro	1.1	3	230 billion kilowatt hours per year

^aIncludes imports of 8.7 million barrels per day (mbd), or 47 percent of total oil consumption; excludes 0.2 mbd of exports.

^bIncludes imports of 0.5 mbd oil equivalent, or 5 percent of total natural gas.

^cExcludes exports of 0.6 mbd oil equivalent.

Source: Department of Energy, Energy Information Administration, Annual Report to Congress, Volume III, 1977 (Washington, D.C.: Government Printing Office, 1978), pp. 5, 23, 51, 145.

- o It is cleaner and safer than some other sources, particularly in being low in sulfur content. Consumption of wastes could lessen environmental problems, by reducing open burning or use of landfills.
- o It is a highly flexible energy option due to the great density of sources, conversion processes and end products.
- o Benefits such as these can be realized in the near-term.

Biomass wastes are abundant today but there is a need to investigate methods of renewing biomass resources for the future. Cultivation of trees (silviculture) specifically for high energy yield per acre and short growth cycle could be explored. Such energy farms would contain trees that grow quickly and are planted in closely spaced rows. The time from planting to harvest would be about 4 to 7 years.

Trees for energy production could be managed in much the same way as progressive companies handle renewal of forest resources for wood products. Despoiling of natural forestlands in locales and terrains peculiarly suited to trees would not be an acceptable energy alternative.

Most of the energy available from wood (and wastes) has come from combustion and this will continue to be a considerable part of its contribution. However, there is also a significant potential for conversion of wood and wastes into liquid and gaseous products that could satisfy other needs (Fig. 1). The principal advantages in addition to the contribution to total energy availability would be in substitution for established fluid fuels that are in short supply and the decrease in harmful environmental effects.

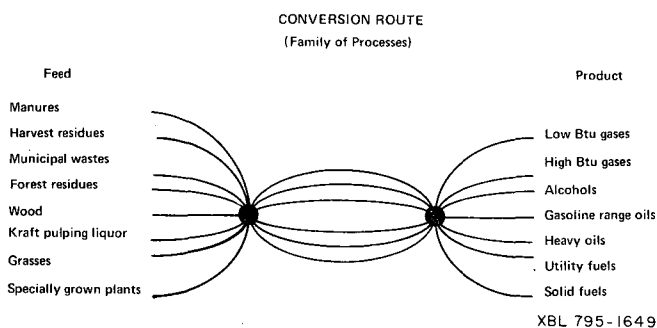


Fig. 1

HISTORY OF WOOD-TO-OIL CONVERSION

Serious efforts to investigate the conversion of biomass into oil or gas really began in the late 1960s at the Pittsburgh Energy Research Center (PERC) of the U. S. Bureau of Mines. Researchers there used a process originally developed for lignite conversion to liquefy organic municipal wastes. Under several thousand PSI of steam and carbon monoxide or synthesis gas (carbon monoxide plus hydrogen) these wastes liquefied at milder conditions than were required

for lignite. Sawdust, manure and other types of biomass were also suitable substrates. Liquefaction of wood was achieved by reaction at about 700°F for about 1 hour with a sodium carbonate solution as catalyst.

Encouraged by the results of bench-scale research and the economic feasibility analysis provided by the Blaw Knox Chemicals Division of the Dravo Corporation, the U. S. Bureau of Mines proceeded with the design and construction of a process development unit (PDU) capable of processing 3 tons of wood daily to yield roughly 5 bbl oil. The plant site chosen was adjacent to the Metallurgical Station of the Bureau of Mines at Albany, Oregon.

The plant construction was completed in December 1976. However, by mid-1976, the facility was transferred to the Solar Energy Division of The Energy Research and Development Administration (ERDA), now the Department of Energy (DOE), due to a reorganization at Washington. ERDA contracted with Bechtel Corporation to commission the facility, perform shake-down tests and conduct test runs. The original contract period was one year, which later was extended to about 18 months. Plant commissioning and shakedown did not prove to be easy. Although small, the facility is extremely complex and contains process units operating under conditions heretofore unencountered. In addition, the plant was conceptualized by one company (Blaw Knox), designed by another (Rust Engineering), and was constructed by a third company (Maecon, Inc.). Since the Pittsburgh Energy Research Center turned its attention to lignite liquefaction upon transferral of responsibility, efforts involved in the various stages of plant design and construction could not be adequately coordinated. Bechtel did succeed in commissioning the plant and in conducting a limited number of test runs. However, a major process unit, the stirred tank reactor, could not be operated properly due to a design deficiency, and another, the centrifuge, had to be bypassed due to operational difficulties. Therefore, the results obtained by Bechtel did not shed much light on the chemical, technical, and economic feasibility of the process. In response to an ensuing Request for Proposal (RFP) a contract for operation was awarded to Rust Engineering Company, which earlier had provided the detailed engineering design.

Recognizing that further investigation of the process chemistry on a bench scale would be beneficial to the development program, Pacific Northwest Laboratories (PNL) proposed to ERDA that PNL would undertake bench-scale supporting activity. After conducting some experiments under conditions described by the Bureau of Mines researchers for purposes of comparison, PNL widened the scope of investigation to conditions not covered before, provided analytical support to Bechtel, recommended the necessity of an on-site analytical facility at Albany, and conducted bench-scale tests under conditions suggested by Rust. The University of Arizona noted that the injection of solids into high pressure vessels was a formidable unit operation

faced in coal liquefaction as well as biomass liquefaction and, accordingly, proposed to undertake research to develop extrusion techniques for biomass injection into high pressure vessels.

In October 1977, DOE contracted with Lawrence Berkeley Laboratory (LBL) of the University of California to monitor the development program at Albany and the supporting activity, to provide a third-party objectivity in the evaluation of results, to make recommendations regarding the future course of the program, and to provide assistance and appraisal to the new operators of the facility should a different company become a successful bidder to the RFP to be issued, i.e., to assure continuity.

The causes of the operational difficulties encountered at Albany remained controversial. DOE concluded that the difficulties were a formidable obstacle to assessment of the technical feasibility of the process and that the chemical feasibility of the process remained in question. Accordingly, in FY 79, LBL was given the responsibility of providing the operating directives for the test runs to be conducted at Albany, specifying the conditions of operation (e.g., temperature, pressure, wood/oil ratio, wood/catalyst ratio, wood/syngas ratio, slurry feed rates, residence time) to assess the chemical feasibility of the process. If one considers the fact that any operating condition that is deemed to be desirable from chemical or kinetic considerations must also be technically feasible (about which the engineers in charge of the operation of the facility will have something to say), one appreciates the checks and balances aspect of the DOE approach.

LBL was also given the responsibility of monitoring the supporting research activity on biomass liquefaction to utilize the results in the development program at Albany. Also, LBL proposed to DOE to conduct supporting engineering and development studies to provide operational approaches to biomass liquefaction.

CHEMISTRY OF WOOD-TO-OIL CONVERSION

Chemical Structure of Wood

Wood is composed of three major components, cellulose (30-50 weight percent), hemicellulose (10-35 weight percent) and lignin (15-35 weight percent). The chemistry of wood can be described for the most part by the chemistry of its constituents.

Cellulose is formed from D-glucose blocks joined by β -1,4-glucosidic bonds. Wood cellulose occurs as polymeric molecules with molecular weights generally in the range of 80,000 to 340,000. The structure of cellulose is shown in Fig. 2.

Hemicelluloses are complex molecular chains of xylose or arabinose backbones. Xylans combined with substantial amounts of uronic acids are the most important hemicelluloses in woods.

Cellulose Structure

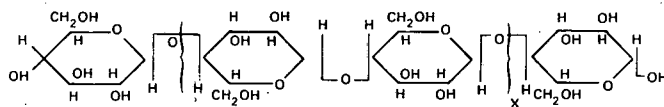


Fig. 2

XBL 795-1647

Lignin is a polymeric substance whose complexity results from the variety of ways in which constituent phenyl propane and other building blocks can be linked. Several models have been proposed for the structure of lignin. One such model, by Freudenberg [1], is shown in Figure 3. The molecular weight of lignin polymers in wood ranges from 8,000 to 11,000 (or higher) corresponding to a degree of polymerization of 35 to 55.

Lignin Structure

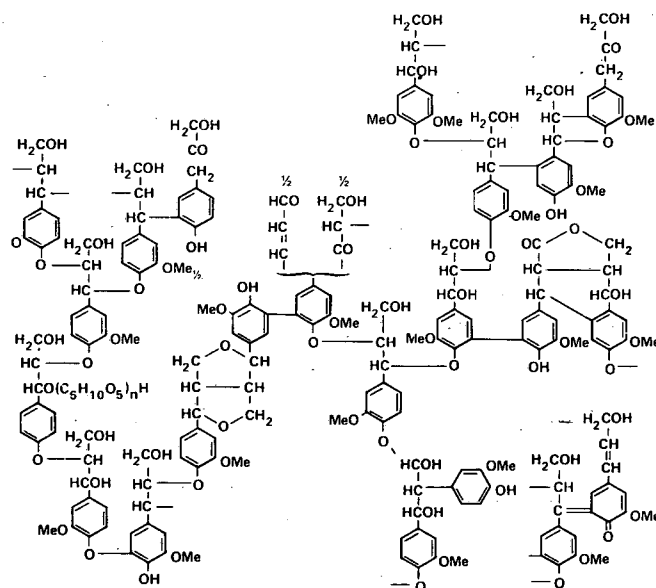


Fig. 3

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Wood Chemistry and Its Application to Liquefaction

Several areas of wood chemistry have been well explored. Pulping of wood for the manufacture of paper is probably the most studied and best understood area of wood chemistry [1,2]. The hydrogenation and hydrogenolysis of wood, lignin and cellulose have also been extensively studied. Substantial information also exists on the selective hydrogenation of carbohydrate materials such as cellulose, sugars and polyhydric alcohols.

Review of the various methods of degradation and hydrogenolysis of wood and its components suggested that four major chemical routes to liquefaction offered promise. These may be des-

cribed as: 1. Treatment with inorganic salt solutions as in paper manufacture; 2. Treatment with hydrocarbon hydrogen-donor solvents; 3. Solvolytic degradation with water or alcohols in an acidic medium; 4. Hydrogenation (or reduction), perhaps aided by homogeneous organometallic complexes as catalysts in dilute aqueous acids.

The PERC and LBL processes require the presence of a pressure of carbon monoxide, and are thus not strictly hydrogenolyses. In the former, recycle oil and water constitute the carrier and sodium carbonate or other alkali is added to the water. In the LBL process the wood is hydrolyzed in an aqueous acidic medium and the conversion to oil is again catalyzed by a water solution. The essential differences include the hydrolysis step of the latter, which eliminates the drying and grinding of chips to flour in the PERC process and the recycle requirement.

Research is continuing on other combinations of the basic chemistries to seek further improvement in liquefaction.

NATURE AND USES OF WOOD LIQUEFACTION PRODUCTS

The nature and distribution of products from liquefaction of wood may be estimated from an analysis of the depolymerization products of cellulose and lignin. A partial list of cellulose and lignin decomposition products is shown in Figures 8 and 9, together with their estimated boiling points and solubilities in water.

	Example	Boiling point	Solubility in water
Cellulose			
↓			
Oligomers			Insoluble
↓			
Sugars	Glucose	—	Soluble
↓			
Polyalcohols	Sorbitol	295(@ 3.5mm Hg)	Soluble
↓			
	Glycerol	290	v. Soluble
↓			
Alcohols	Propanol	98	v. Soluble
↓			
	Ethanol	79	v. Soluble
↓			
Alkanes			

Fig. 4

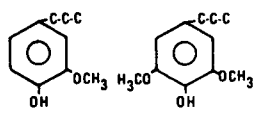
	Example	Boiling point (°C)	Solubility in water
Lignin			Insoluble
↓			
Oligomers			
↓			
Phenylpropanes		265-400	
↓			
	4-methoxy-2 methyl phenol	252	Insoluble
↓			
Substituted phenols and cresols	Cresolol	249	
	Ethyl phenol	207	
↓			
Phenols	Phenol	182	Soluble
↓			
Cyclohexanols	Methyl cyclohexanol	155	Insoluble
↓			
Cyclohexanes	Cyclohexane	81	Insoluble

Fig. 5

The depolymerization of wood in reducing atmospheres results in the formation of products of progressively lower oxygen content and polarity. The lignin largely decomposes to form water-insoluble, high boiling products, whereas cellulose decomposition results in the formation of water-soluble products having lower boiling points. This is a rather simplified view of wood liquefaction since it ignores the possibility of the formation of polymeric materials from condensation and degradation reactions. It does, however, permit estimation of the physical properties of process streams and selection of possible separation processes.

A two-phase liquid effluent can be expected from the liquefaction reactor. The light aqueous phase would contain added process water and water from wood dehydration together with dissolved low molecular-weight oxygenated products. Higher molecular weight wood-derived oils and any non-polar organic slurring solvents could be expected in the heavy organic phase. The organic phase would also contain any unreacted wood or high-molecular-weight condensation and degradation products or char. Distribution of products between oil and water phases will depend upon the relative amounts of the two phases present.

Where the aqueous phase is a large fraction of the product stream, decantation or centrifugal separation is needed to avoid high evaporation costs. If water soluble catalysts (other than simple alkali) are added, these could be recirculated in the separated water phase.

Of the organic phase only materials lower boiling than dilignols (from lignin) and sugars (from cellulose) can be distilled. The non-distillable fraction of the organic phase could be used for the production of reducing gases and/or process heat generation.

The character and composition of the oils produced by liquefaction of biomass will vary depending upon the type of biomass feedstock and the processing conditions. An approximate estimation of the properties of the oils is as follows:

Composition of oil phase (weight percent)

Solids	= 0.6
Water	= 7.1
Oil	= 92.3 (acetone soluble)

Elemental Composition of oil (weight percent)

Carbon	= 81.2
Hydrogen	= 7.9
Nitrogen	= 0.1
Oxygen	= 10.8 (by difference)

Additional properties

Heating value	= 15,800 btu/lb
Specific gravity	= 1.09
Viscosity	= 40 cp at 210°F
Distillate yield (275°C at 3 mm Hg)	= 70 percent

Distillate equivalency	= between No. 2 and 4
No. 2 equivalent yield	= 40 percent
No. 4 equivalent yield	= 30 percent
Raw oil equivalency	= No. 6 fuel oil

In general the oils may be suitable substitutes for petroleum fractions in several applications. Mainly they should be useful as boiler fuels and as petrochemical feedstocks. Because of their rather strong phenolic character, they may be especially adaptable to plastics manufacture.

TECHNOLOGY

Process Definition

Process development involves establishing of chemical, technical and economic feasibility of a process scheme, usually in that order. When an array of partially understood chemical conversion schemes exist, even a limited prior understanding of process parameters and economics can be of invaluable assistance in formulating a reasonable experimental program. The changes in physical structure and elemental composition of wood as it is converted to liquid products have been analyzed for a first overview of process concepts.

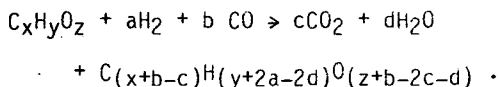
Physical Transformations

Wood may be fed to a process as chips (3/8 in. to 1 in. size pieces) or as flour (typically -60 mesh powder). Use of wood chips avoids the cost of drying, crushing and grinding associated with the production of flour. Chemical (or mechanical) degradation of chips to fine particles is, however, necessary if the wood is to be fed as a slurry through high pressure pumps to pressurized reaction tanks. In the first step wood chips must be transformed into a pumpable slurry and/or material soluble in the slurring solvent. Early depolymerization of wood to soluble material is advantageous because of greater accessibility of the substrate--here catalyst and carbon monoxide--to the reagents that promote liquefaction.

The next major physical transformation is the conversion of the pumpable slurry to low molecular weight product. This step occurs at high pressures in the processes using carbon monoxide reduction.

Elemental Transformations

The overall mass balance for the biomass conversion scheme can be represented by:



This equation is a plant-battery-limit mass balance that ignores ash, nitrogen and sulfur but includes the consumption or production of any reducing gases or wood derived reagents needed for the overall conversion (e.g., CO, H₂ or alcohols).

An essential element for wood liquefaction is the removal of most of the chemically bound oxygen which, together with depolymerization, can lead to formation of liquid products with a high heating value. Oxygen removed from wood would exit any processing scheme as water, carbon dioxide or carbon monoxide. Removal or addition of water to or from an organic molecule results in little change in its molar heating value, as indicated by the Dulong Formula for estimation of heating values. Spontaneous removal of carbon dioxide in the major reaction step would lower the consumption of reducing gases and hence the load on any auxiliary gasification system.

Bureau of Mines Process

The process development unit at Albany, Oregon is presently being operated essentially as designed in the PERC process. A schematic representation of the process is shown in Fig. 4. The PDU is shown in the photograph of Fig. 5.

PERC PROCESS SCHEMATIC

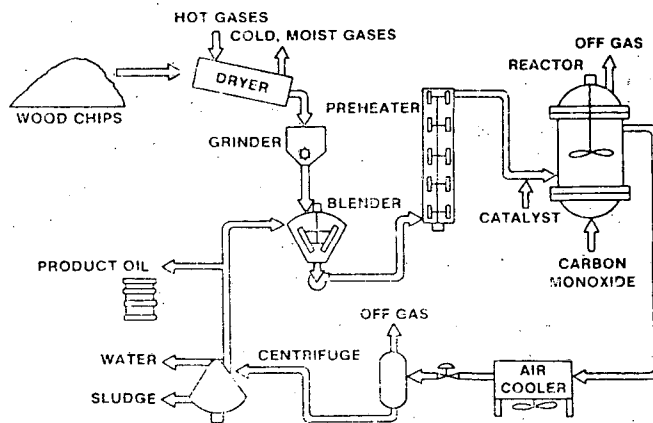


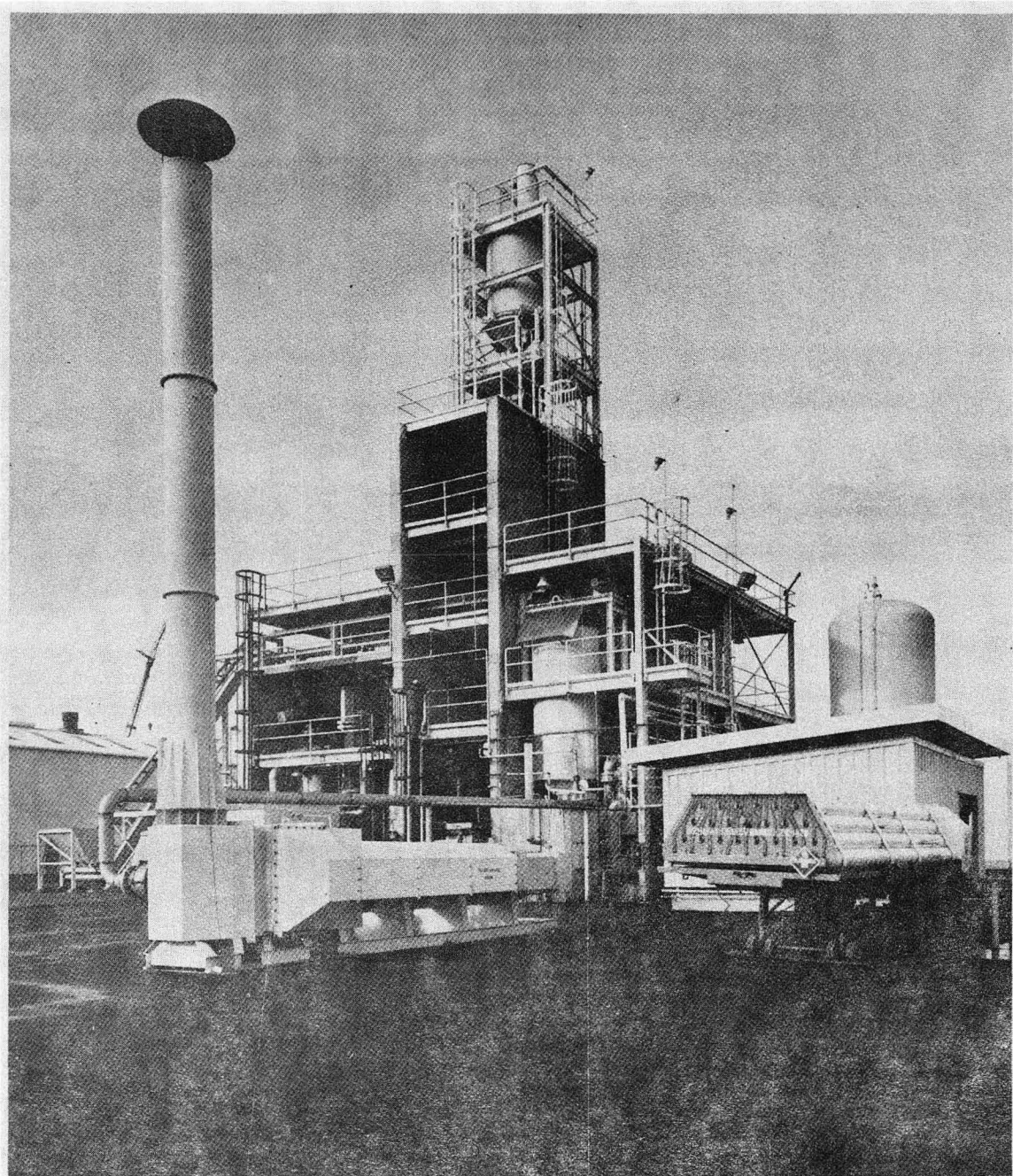
Fig. 6

The PDU was designed to demonstrate the PERC process for conversion of wood to oil. Biomass feedstock in present use is Douglas Fir wood chips, purchased from a nearby paper mill. These chips are stored in an open shed. They are moved by a front end loader to the feed conveyor for transfer to the dryer feed storage bin.

The wood chips are withdrawn from the storage bin by a controlled rate table feeder to a gas fired rotary dryer, where the moisture content is reduced from about 50 percent to about 4 percent maximum, using hot combustion products for heating. The dried chips are then fed to a hammer mill grinder and screened through 35 mesh.

The grinding and subsequent wood processing operations are kept in a nitrogen atmosphere to minimize moisture pick up and explosion hazard.

The wood powder is conveyed pneumatically to an elevated feed bin in a closed system using nitrogen as the conveying medium.



**THE BIOMASS LIQUEFACTION
PROCESS DEVELOPMENT UNIT
ALBANY, OREGON, U.S.A.**

Fig. 7

The "catalyst," sodium carbonate, is prepared as a 10-15 percent aqueous solution for injection into the reaction system by means of a conventional high pressure metering pump. The catalyst solution is injected either ahead of the preheater or directly into the reactor. Since sodium carbonate is primarily for pH control and reacts with organic acids found in the process, it is not truly a catalyst.

Wood powder is continuously fed from the wood surge bin through a weigh feeder to the wood-oil blender. Anthracene oil is initially used as a carrier vehicle; it is pumped from the oil feed tank to the wood-oil blender where it is mixed with wood powder. The cone-shaped blender has helical screws on rotating arms that sweep the side walls.

The wood-oil slurry from the blender is pumped into the reactor through a feed slurry circulating pump and a high pressure feed slurry pump. The slurry circulating pump is a Moyno resilient stator type, while the high pressure slurry feed pump is a Bran and Lubbe variable stroke, positive-displacement type with ball check valves. The wood-oil slurry passes through an electrically heated reactor, where temperature and pressure are maintained and reaction occurs.

The reactor effluent is cooled in an air-cooled heat exchanger prior to pressure letdown; upon letdown, water and light oils flash and are separated in a low pressure flash system. The vented gas is cooled to recover light oils and water. Vented gas and non-condensibles are incinerated.

Pretreatment of Biomass Prior to Liquefaction

Pretreatment of biomass prior to liquefaction was proposed by the Bureau of Mines as an optional procedure for injecting biomass into high pressure vessels. In the Bureau of Mines process the purpose of pretreatment was to change the rheological properties of biomass so that biomass flour/oil slurries containing up to 50 percent treated biomass could be injected into high pressure vessels. Lawrence Berkeley Laboratory researchers investigated the rheological and chemical changes occurring upon pretreatment of wood and conceptualized a process scheme in which wood chips are hydrolyzed under mild conditions to obtain a concentrated aqueous slurry that can be directly injected into the liquefaction system without a carrier oil.

In the BOM pretreatment technique wood chips and water (30/70)* are heated to 500°F and 700 psig for 60 minutes. After cooling, the solids are separated by vacuum filtration, dried and pulverized to -50 mesh, mixed with recycle oil and injected into the liquefaction loop. The results of four pretreater test runs conducted at Albany were generally unsatisfactory.

Several pretreatment runs were conducted by LBL on a small scale, duplicating the conditions used at Albany. Significantly, it was found

that the carbohydrate fraction of the wood was almost entirely destroyed under these conditions; the product analyzed as a dehydrated char. This was a serious shortcoming since carbohydrates have been shown to liquefy readily under BOM/Albany conditions, whereas chars typically are intractable.

On the other hand it was noted that extensive size degradation occurred in the course of pretreatment, indicating that it might be possible to eliminate filtration, drying and grinding altogether. Thus a new goal for the development of a pretreatment process was established: to produce, with no sacrifice of the original heating value, a concentrated slurry of biomass capable of being pumped directly into the liquefaction system. A five-point process development plan was formulated for the purpose of guiding the overall research effort:

1. research chemical pretreatment methods
2. research
 - a. size degradation efficiency
 - b. rheological properties of solids
3. research liquefaction of treated biomass
4. obtain data base for large scale test run at Albany
5. conceptualize a process.

Chemistry of Pretreatment

Initial guidance as to research direction was provided by an examination of the chemical composition of Douglas Fir (*Pseudotsuga menziesii*), the predominant species in the Albany area (Table 2).

Table 2. Composition of Douglas Fir (moisture free).

	Percent
Cellulose	41
Hemicellulose	26
Lignin	28
Uronic anhydride	3
Ether-soluble extractives	1.0
Acetyl	0.6
Ash	0.3
	99.9

Cellulose and hemicellulose, comprising the carbohydrate fractions, should be preserved, either as such, or in the form of their constituent sugars which are liberated upon hydrolysis. Lignin, a three-dimensional matrix of oxygenated phenylpropane units, accounts for most of the remainder.

*Plant practice established 23 percent solids as a more manageable concentration.

The conditions specified by the Bureau of Mines are apparently too severe since they result in charring and destruction of carbohydrate. Milder conditions require the use of a catalyst. Obvious catalysts are acids or bases.

The most promising results were obtained in the case of mild acid hydrolysis accompanied by vigorous agitation. Conditions have been found which result in complete hydrolysis of the hemicellulose with little loss of carbohydrate values (Table 3). Under these conditions the remainder of the wood undergoes extensive size reduction, forming spherical rather than fibrous particles. The resulting slurries have been shown to possess superior rheological characteristics.

Table 3. Data Base for Hydrolysis

Wood/Water Ratio	23/77
Heat-up Time	12 min
Temperature	180°C (356°F)
Pressure	130 psig
Acid (% H ₂ SO ₄)	0.05
pH	2.0
Retention Time	45 min

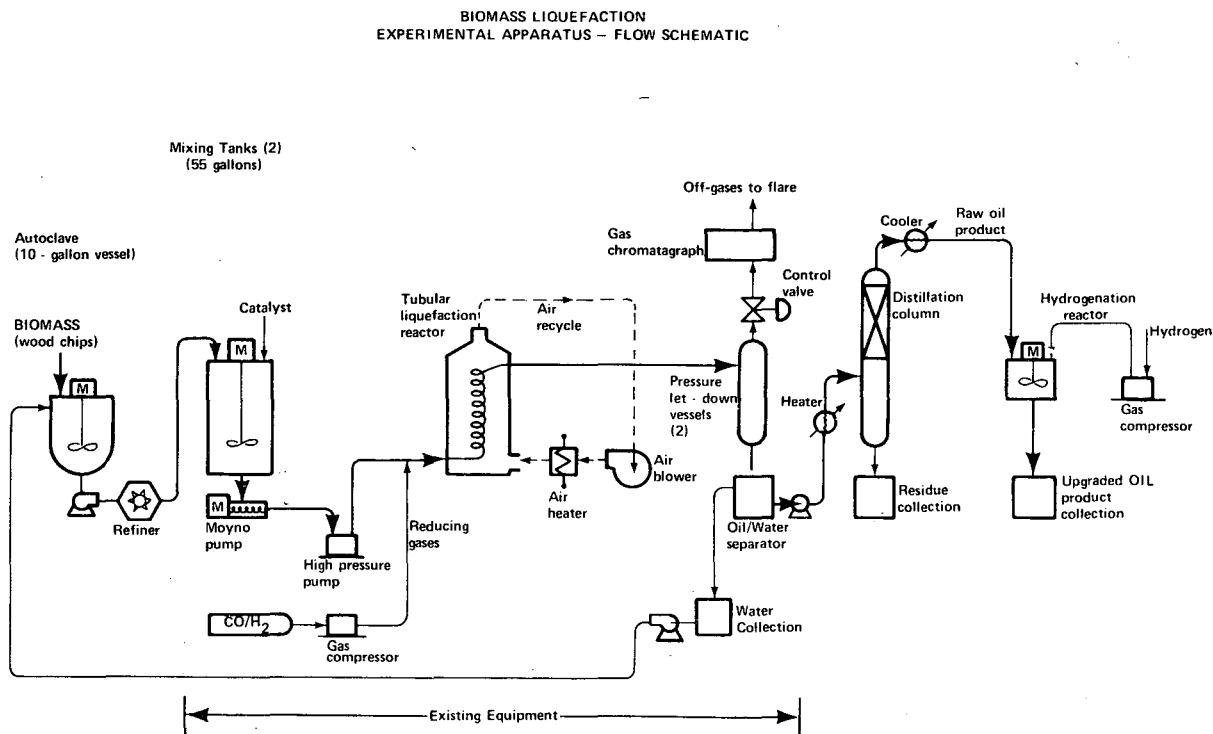
The success of hydrolytic pretreatment in reducing wood chips to fine particles is probably due to a combination of chemical and physical action. Typically about half of a charge of dry

wood chips, nominally 1-1/4 in. in length, is reduced to -20 mesh or finer. Those particles larger than -20 mesh can for the most part be crumbled between the fingers.

In order to adapt the acid pretreatment technique to the Albany process, neutralization with alkali would be required. This is an obvious disadvantage which could be avoided if (a) a basic pretreatment or (b) an acidic liquefaction method could be developed. Active research in this area is under way. Several promising acid catalysts for liquefaction have been discovered but have not yet been fully evaluated.

LBL Process

In order to incorporate the major changes from the Bureau of Mines process pretreatment of the wood chips prior to liquefaction, an LBL process was conceptualized. The conversion chemistries involved in the two processes are similar but there are significant process differences. Besides the pretreatment changes, the LBL process differs in the handling of the exit streams. The spent synthesis gas from the pressure let-down vessels may be sent to heat the reactor and the bottoms to a continuous separator for recovery of oil which may then be distilled into desired fractions. None of it is needed for recycle. A schematic of the complete equipment is shown in Figure 8; the presently operating liquefaction section is indicated and a photograph appears in Figure 9.



XBL 802-239

Fig. 8

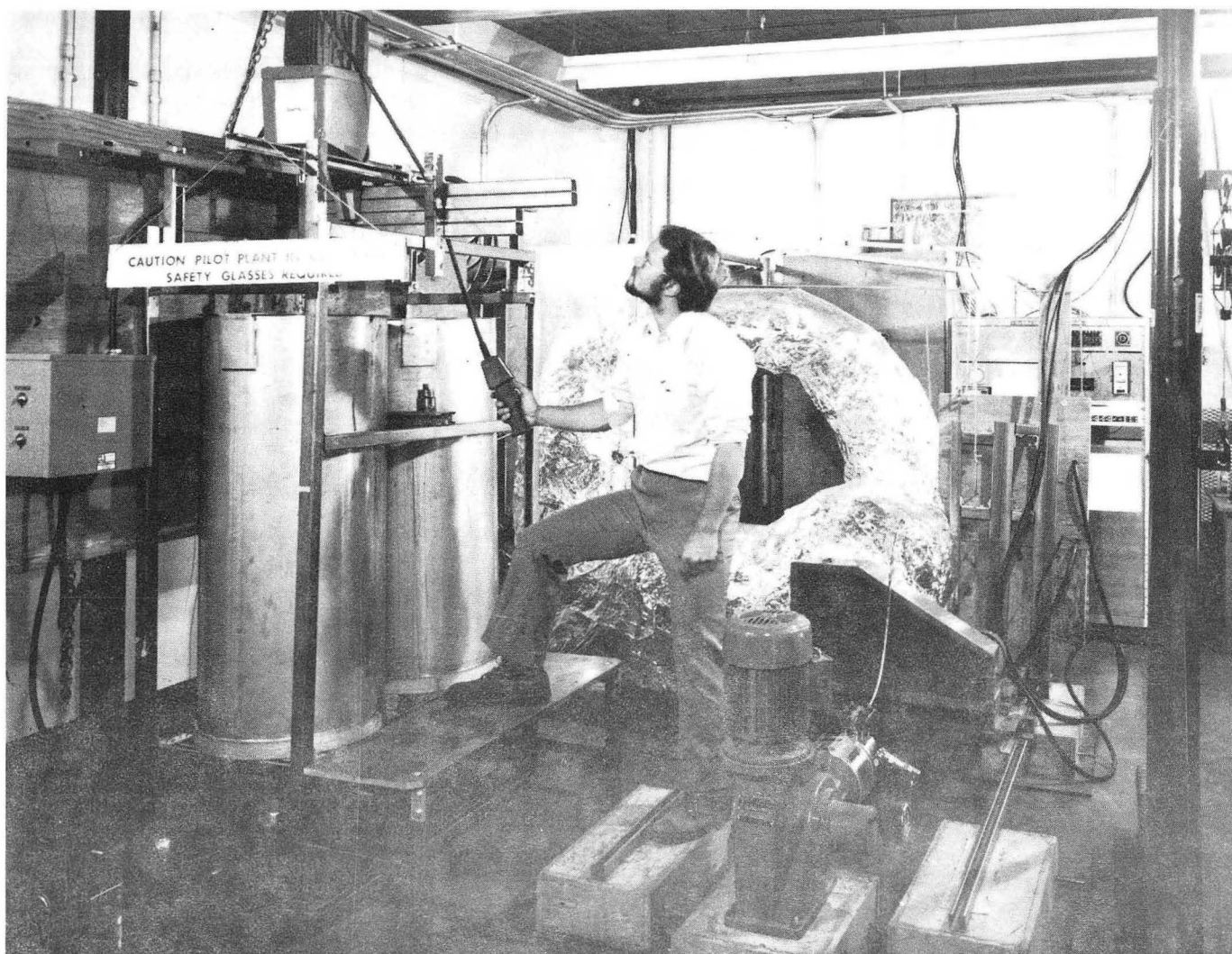


Fig. 9

Current Status

A small process evaluation unit at LBL is being devoted to studies of the effects of variable process parameters such as type of biomass, type of catalyst, catalyst/biomass ratio, slurry feed rate, synthesis gas composition, synthesis gas/biomass ratio, pressure, reactor temperature, and residence time in reactor. These and other studies provide a data base for contemplated changes and improvements in the Albany PDU and to direct the scalings of such parameter effect determination to the Albany scale. For example, the wood hydrolysis tests were started in a 400 ml autoclave, graduated to 1 liter and 10 gallon autoclaves, and finally to the 400 gallon Albany autoclaves. Results obtained at all scales were reasonably similar.

The scale of the LBL unit makes it amenable to possible innovative ideas for process changes that may be developed at LBL or at supporting or related facilities.

Further tests of the PERC process, utilizing a light-ends stripper and a vacuum distillation unit, are being conducted at the Albany PDU. Following these, installation of a new reactor to permit continued tests of the LBL process is planned.

ACCOMPLISHMENTS

Efforts to test and establish the chemical and technical feasibility of the Bureau of Mines wood liquefaction process in the Albany, Oregon process development unit were essentially unsuccessful. The unit could not be operated for reasonable periods of time without operational difficulties. A major problem has existed in the wood pretreatment steps such that a slurry containing more than 10 percent wood has not been routinely handled. Consequently a primary objective of the LBL research program has been to develop a pretreatment process that would result in a mobile slurry containing 30 percent wood. Considerable progress has been made toward

that objective as well as other improvements in the overall process. As a result a breakthrough has been attained in the process development by the production of seven barrels of oil through an improvised version of the LBL process, demonstrating the chemical feasibility of the process. Development of the new pretreatment process has established the technical feasibility of circulating a slurry containing at least 23 percent wood. In subsequent work the wood content has been increased to over 30 percent by optimization of prehydrolysis conditions.

In order to continue development of a commercially viable biomass liquefaction unit, LBL has designed and constructed a continuous process evaluation unit to screen the influences of process variables, to provide engineering data regarding heat and momentum transfer and reaction rates, and to screen promising new process concepts that come from bench-scale laboratory experiments. This unit simulates the Albany facility but incorporates a tubular reactor that has been lacking there. Experiments in this unit will provide data bases for scale-up to the larger development unit at Albany.

FUTURE PROGRAM

The future program of LBL for biomass liquefaction development includes the following objectives:

1. Conduct tests to establish a data base for designing a continuous pre-hydrolyzer to process 3-5 TPD of wood. Continue efforts to increase wood content of the slurry.
2. Select promising catalysts for biomass liquefaction and determine optimum operating conditions.
3. Select catalysts and optimum conditions of hydrogenation to upgrade syncrudes obtained from wood.
4. Conduct research on solvolysis of wood in phenols and alcohols and subsequent liquefaction.
5. Develop standardized procedures for characterizing oils from wood.
6. Operate the continuous process evaluation unit to establish optimum technical parameters.
7. Design a demonstration plant to test and optimize the continuous process.
8. Monitor other DOE research on biomass liquefaction, evaluate findings, and recommend improvements.

Economic Feasibility

Rust Engineering Company and SRI International made, independently, an economic feasibility analysis of the LBL process in

comparison with the BOM process. The LBL process compared favorably. From a small plant processing 1,000 TPD wood (dry basis) the cost of the oil produced (equity financing) was placed at \$48/bbl by SRI.

LBL originally (June, 1979) provided SRI a conservative data base. For example, water/wood ratio was given as 3.35. Experiments conducted since June 1978 revealed that this ratio could be reduced to nearly 2.0 resulting in a 40 percent reduction in reactor heat requirements and a 31 percent reduction in the number of reactors for a fixed oil production capacity. These translate into a minimum of 25 percent reduction in the operating costs. A second conservative data base concerns synthesis gas utilization. Additional studies revealed that, by adjusting the H₂/CO ratio in the synthesis gas introduced, the net synthesis gas consumption can be reduced to less than one half of the original estimates.

It is anticipated that sufficient data will be generated at LBL during 1980, using the continuous process evaluation unit, to warrant an updating of the cost estimates of the process (by SRI).

Technical Feasibility of Continuous Prehydrolysis

Prehydrolysis was performed batchwise at Albany, Oregon using one of the two 400-gallon autoclaves. The resulting slurry was refined using the laboratory refiner at Oregon State University. If this unit operation can be done continuously, the economics of the process can be assured. Engineers familiar with pulp and paper technology point out that modern pulping machines operate under higher pressures (250 psig) than those required for prehydrolysis (130 psig) and at higher temperatures. They foresee no difficulty in designing and constructing a unit capable of processing 2,000 TPD (smaller than modern pulpers).

Commercialization Potential

The LBL team believes that sufficient data can be obtained at LBL and Albany to design a demonstration or commercial plant (2,000 TPD) and obtain a firm cost estimate of the process. The target dates set are given below:

- o Design of a reactor capable of producing 2-5 bbl oil/day. May 1, 1980 (by LBL)
- o Fabrication and installation of the reactor at Albany July-Sept. 1980
- o Acquisition of a continuous hydrolyzer (20 TPD) and installation at Albany May-August 1980
- o Shakedown tests of the reactor and hydrolyzer September-October 1980
- o Test runs at Albany November-January 1981

- o Basic engineering data generation and integrated plant concept development Sept. 1980-Aug. 1981 (LBL)
- o Design of a commercial size plant in 1981.

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Regarding the next to the last item, LBL is continually evaluating various integrated plant concepts. An example of an integrated process concept is shown in Figure 10. The novel feature of the concept is a reactor for both gasification and liquefaction. The conceptual design is shown in Figure 11.

Conceptual Commercial Biomass Liquefaction Flow Schematic
LBL Process

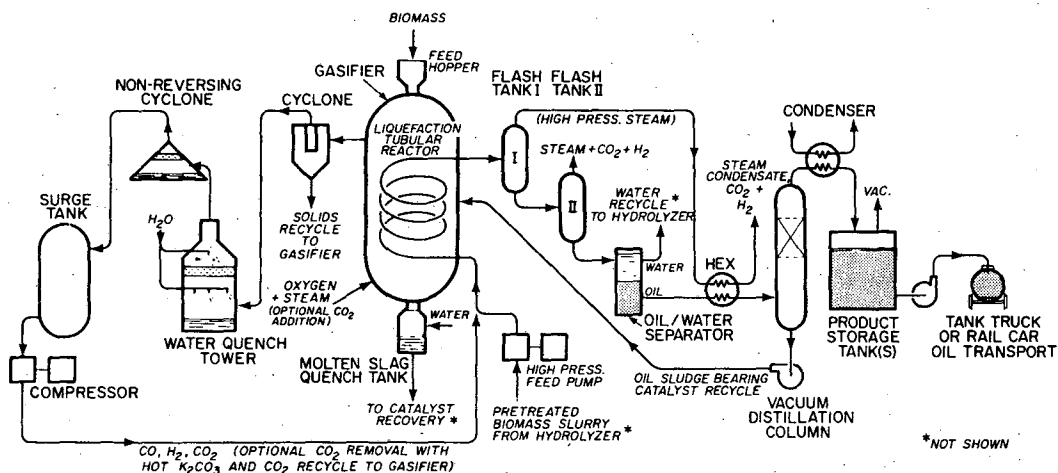


Fig. 10

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Modified Lurgi Gasifier with Liquefaction Reactor

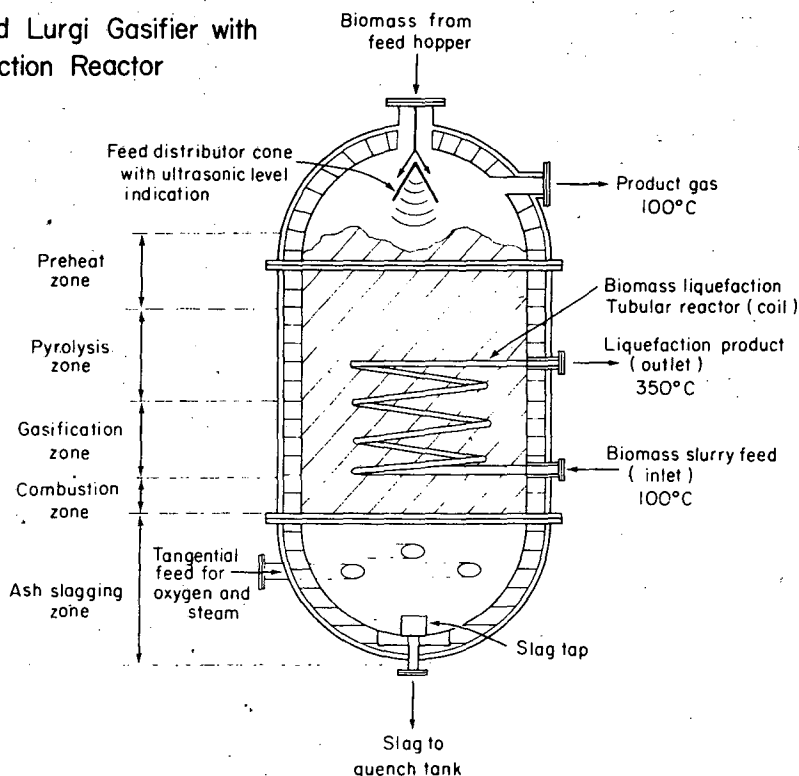


Fig. 11

XBL 7910-4543

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