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ENERGY & ENVIRONMENT DIVISION

CATALYTIC BIOMASS LIQUEFACTION QUARTERLY REPORT

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CATALYTIC BIOMASS LIQUEFACTION
QUARTERLY REPORT

Thermochemical Conversion
Biomass Energy Systems Branch
Department of Energy

July-September, 1980

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1. THE LBL PROCESS FOR WOOD LIQUEFACTION

Essential features of the LBL liquefaction process remain as they have been since the first improvised run at the Albany PDU (TR-7). The chips are partially hydrolyzed with dilute sulfuric acid (about 0.075 wt %) at 180°C for 45 minutes. The acid, plus minor amounts of wood acids are neutralized and the crude slurry passed through a pulp and paper industry refiner. The result is a stable slurry containing 25% or more (perhaps as high as 35-37 wt %) dry solids. Although quite thick, the slurry is pumpable.

The slurry is preheated rapidly to about 360°C in the presence of synthesis gas (CO + H₂) and, of course, steam, and kept at 360°C for residence times of from several minutes to a half hour. A tubular reactor is recommended, although a stirred reactor is possible. Aqueous and oil product layers are separated for work-up.

Recommendations for a realistic test of this process at Albany have been made. Slurry will be made as described above, but concentrations above 25 wt % may not be feasible. A new tubular preheater (residence time < one minute) is ready to go as this is written. Since no tubular reactor exists, a take-off from the existing stirred reactor is being implemented to give about 50 gallons reaction volume. At the proposed slurry feed rate of 60 GPH, this gives a space time of 50 minutes and an actual average residence time — allowing for volume occupied by gas — somewhat lower.

Rust Engineering plans to install commercial phase separators to make the rather difficult oil/water separation. An LBL engineer will be present during both the slurry preparation and liquefaction phase of the planned run (TR-10, target date 11/18/80).

Since the shift reaction occurs during the wood liquefaction runs, recovered unreacted synthesis gas is richer in hydrogen and leaner in carbon monoxide than is the feed gas. Outlet gas contains much carbon dioxide from the shift reaction and also from decomposition and reduction of the wood. It is, however, possible that a recycled hydrogen-carbon dioxide mixture is, because of the reverse shift reaction, equivalent to carbon monoxide as feed gas. If so, this simplifies recycle of unreacted synthesis gas, since a reverse shift reaction can be avoided and the degree of CO₂ removal before recycle can be reduced. Laboratory and PDU experiments with H₂-CO₂ mixtures as feed gas have been recommended.

Earlier runs with the PERC process (TR-8 and -9) and also the TR-7 have used substantial amounts of sodium carbonate as "catalyst." There is considerable doubt about the need for such a catalyst. Indeed, there is evidence that simple neutralization of the slurry to about pH = 7 results in the best conversion to product oil. Certainly, the presence of sodium salts causes a serious water disposal problem. A further study of the effects of alkali addition and pH has therefore been undertaken. Stopping neutralization at pH 7 to 8, and substitution of sodium carbonate by sodium hydroxide greatly reduces the amount of sodium ion for disposal. Replacement with an alkaline material giving an insoluble sulfate (calcium or barium hydroxide) could further reduce disposal problems.

As is also true of the PERC process, the data necessary to optimize the LBL process with respect to time, temperature, pH and reaction gas composition are skimpy. A program to obtain better data is being initiated.

2. THE EFFECT OF PROCESS VARIABLES ON LIQUEFACTION UNDER THE CONDITIONS OF THE TUBULAR BOMB REACTOR

Abstract

During the past quarter we completed a planned set of experiments on the effects of process variables, on yields of acetone solubles and of solid residues in the liquefaction of wood. The variables studied were reaction temperature, reaction time, pH and initial gas composition. It had previously been shown that minimum heat-up time, other variables constant, gave maximum acetone solubles and minimum residue. Under tubular bomb conditions a reaction temperature of 360° with the minimum heat up time of about 2½ min and zero time at temperature gave us the highest acetone solubles and least solid residue. The response of yields to initial gas composition is complex. However, it seems clear that the presence of CO₂ in the initial gas is not harmful, and that a reducing gas is necessary.

The advantages and limitations of the tubular bomb reactor are discussed. The major advantages are the short heat-up and cooling times, ability to do many experiments and freedom from leaks. The disadvantages lie in the lack of agitation, the limited contact between reducing gases and the substrate, and the degree of error introduced by using a very small sample.

While we continue to use conventional stirred autoclaves to study biomass liquefaction, there are inherent disadvantages. Most importantly, heat up and cooling times are long for such a system, making kinetic interpretation difficult, time per experiment is long and problems with leaks are frequently encountered.

The tubular bomb reactor consists of a short horizontal length of 0.5" SS-316 tubing (5" x 3/8" ID), supported by a vertical length of 1/4" SS-316 tubing (14" x 1/8" ID) connected by a right angle elbow at one end of the 0.5" piece, with the other end connected to a vertical thermocouple well.

Total volume of the system is about 11 cm³ including about 2-3/4 cm³ in the vertical section. Since over 4 cm³ of slurry is packed into the 1/2" OD section at the start of the reaction, much of the charge gas (CO, H₂, N₂, CO₂ or mixtures) is in the vertical arm. After loading the slurry sample, the reactor is pressurized, usually to 500 psig, closed off, and plunged into a molten salt bath initially slightly above the desired reaction temperature. The reaction temperature registers 360°C in about 2.5 minutes. After the desired time at temperature, the bomb is cooled rapidly by a plunge into a water bucket.

Two deficiencies are obvious: there is no agitation other than that provided by thermal effects; and contact of the pressurizing gases (CO etc.) with the slurry is somewhat limited. Half the volume of the horizontal reactor section is filled with slurry. As the temperature is raised and steam is formed, this will tend to push the pressurizing gases out into the vertical section. With the short heat-up and reaction times used, there will be little that diffusion can do to overcome this effect.

The conclusions drawn from these experiments must be understood in the light of the above.

Feedstock and Experiment Procedure

The slurry samples used were taken from a large batch made at the Albany, Oregon PDU by prehydrolysis and refining of Douglas Fir wood chips. For each run, slurry containing 1 to 1.5 g wood (dry basis was weighed into the reactor section). The slurry had been previously adjusted to the desired pH with sodium carbonate or sodium hydroxide. The reaction was pressurized to 500 psig with CO, H₂, CO₂ or various mixtures. Runs with no reducing gas present were pressurized with 500 psig N₂.

Results and Discussion — Effect of Reaction Temperature

The effect of operating at reaction temperatures up to 400°C was studied and the results are shown in Table I. At 250°C the low reaction temperature and short heat-up time result in a very high insoluble residue (50%) and 26% acetone solubles for a total mass recovery of 76%. This probably means that very little gasification and incomplete reaction have occurred. At the highest temperature studied, 400°C, acetone solubles are lower (19.7%), char is again increasing and the total mass recovery is reduced, indicating strong formation of CO₂, H₂O and other volatiles. Clearly 400°C at the minimum heat-up time and the reaction time is too high a temperature.

Although reaction temperatures of 300°C produced more acetone solubles than reaction at 360°C, the insoluble residue and mass recovery are also higher at the lower temperature. Overall reaction must therefore be less (i.e., the overall recovered product must be high in oxygen). Further exploration of the temperature region from 300 to 360° is indicated.

The effect of heat-up time has been previously reported. In terms of maximizing acetone solubles and of limiting char formation, rapid heat-up was shown to be desirable. The effect of reaction time on yield of acetone solubles and insoluble residue was investigated in several runs at 360°C. The results are given in Table II. In every case acetone solubles increased with decrease in reaction time regardless of the pH of the slurry. In general residue increased with increasing time.

Evidently, under the conditions existing in the tubular bomb reactor at 360°C, the highest yields of acetone solubles and lowest yields of insolubles were obtained with minimum heat-up time and zero holding time at temperature. Again, this suggests that further exploration of the temperature range from 300°C to 360°C (probably, especially 330°C-360°C) is desirable.

Effect of Initial pH of Slurry

The pH range studied was 4 to 9.7. Under tubular bomb conditions, pH's around 7 seem to be better than pH=4 and considerably better than pH=9.7 (see Table III).

Effect of Composition of Pressurizing Gas

Experiments were run at 360°C and these pH levels with various combinations of CO, H₂ and CO₂ and with inert gas (N₂). Maximum heat-up rate (2½ minutes to 360°) and 7½ minutes holding time were used. Reaction conditions and yields are shown in Table III. For the liquefaction run at pH 4, the yields of acetone solubles range from 24 to 47% by weight of dry wood feed. The highest yield was obtained in run RT 51 in which a mixture of CO + CO₂ + H₂ was used. The lowest yields of acetone soluble and highest residues were obtained with N₂ as pressurizing gas (no reducing gas). At pH=7 the runs with CO or H₂ alone showed the highest acetone solubles and lowest residues, the runs with 1/1 CO + H₂ and CO + CO₂ + H₂ gave roughly equivalent results at somewhat lower acetone solubles. However, at pH 9.7, the highest yield of solubles was obtained with the mixed synthesis gas.

It is concluded that, despite the poor contacting and lack of agitation in the tubular bomb reactor, the presence of reducing gas is important in increasing acetone solubles and decreasing insolubles. Hydrogen, carbon monoxide and mixtures of these are probably equivalent within the uncertainties of these results. Carbon dioxide is not harmful, provided sufficient reducing gas is also present.

Summary of Conclusions

- There are obvious disadvantages as well as advantages to the use of the small tubular bomb reactor. Advantages are quick heat-up and cooling and relatively rapid experimentation. Disadvantages include lack of agitation, poor gas-substrate contact and small sample size. These limit the degree to which conclusions can be generalized. However, the reactor appears to be useful for rapid screening of variables.
- Under the tubular bomb conditions and in the ranges explored, highest yields of acetone solubles were achieved at initial slurry pH's around 7, with minimum possible heat-up and cooling times and with zero holding time at 360°C in the presence of reducing gas (CO or H₂ or both).
- Within rather wide limits, no significant differences among H₂, CO, or H₂-CO mixtures were found, the unmixed gases appearing to be better under one set of conditions (at pH = 9.7) and mixtures better at others.
- Mixtures containing CO₂ as well as reducing gas are at least as good as those without it.
- With only inert N₂ as pressurizing gas, results are distinctly poorer than when a reducing gas is added: solubles are down and insolubles are up.
- The temperature range between 300°C and 360°C appears to be worth exploring.
- In view of the usefulness of rapid heat-up experiments and quick experimental turn-over, an effort to design or purchase a modified tubular bomb with better agitation and gas/slurry contacting should be started.

TABLE I

Reaction No.	Temperature °C	Heat up time (min)	Final Pressure*	Acetone Soluble %	Acetone Insoluble %	Mass Recovery
RT46	250	1½	500	26.1	50.3	76.4
RT47	300	1½	620	43.9	26.0	69.9
RT42	360	2	700	31.5	15.3	46.8
RT53	400	2	800	19.7	23.0	42.7

Table I. Effect of reaction temperature

Residence time = 15 min

Initial Pressure = 500 Psi (CO/1½ = 1/1)

Slurry at pH = 4

* Pressure in reactor after recooling to ambient temperature. Increases reflect amount of carbon dioxide formed by decomposition of wood.

TABLE II

Reaction No.	pH of Slurry	Heat up time (min)	Residence time (min)	Acetone Soluble %	Residue %	Mass Recovery
RT42	} 4	2½	15	31.5	15.3	46.8
RT44		2½	7½	36.7	13.3	50.0
RT43		2½	0	51	8.1	59.1
RT41	} 7	2½	15	32	19.1	51.1
RT38		2½	7½	37.6	15.5	53.1
RT28		2½	0	57.3	5.4	62.7
RT5	} 9.7	2½	15	30.5	27.9	58.4
RT55		2½	7½	32.3	22.3	54.6
RT53		2½	0	36.2	21.4	57.6

Table II. Effects of pH and Residence Time

TABLE III

Reaction No.	pH of Slurry	Pressurizing Gas			Acetone Solubles wt %	Acetone Insolubles wt %	Mass Recovery
		CO %	H ₂ %	CO ₂ %			
RT48	pH=4	100	0	0	35	23	58.
RT49		0	100	0	45.2	15.7	60.9
RT44		50	50	0	36.7	13.3	50.0
RT51		24	44	32	47.6	19.2	66.8
RT54*		0	0	0	24.9	27.0	51.9
RT40	pH=7	100	0	0	46.6	11.1	57.7
RT39		0	100	0	46.6	11.7	48.3
RT38		50	50	0	37.6	15.5	53.1
RT27		24	44	32	38.8	19.9	58.7
RT15*		0	0	0	21.3	30.7	52.0
RT58	pH=9.7	100	0	0	24.9	27.5	52.4
RT56		0	100	0	26.4	25.0	51.4
RT55		50	50	0	32.3	22.3	54.6
RT60		24	44	32	24.5	30.7	55.2
RT59*		0	0	0	22.5	31.3	53.8

Table III. Effect of gas mixture

Reaction temperature = 360°C

Heat up time = 2½ min

Residence time = 7½ min

Initial Pressure 500 Psi

Final Pressure = 700 Psi

* Runs with no reducing gas were done with 500 Psi N₂.

3. PROCESS EVALUATION UNIT PROGRESS REPORT

- Installation of slurry feed tank scale and associated tubing.
- Complete revision and replacement of tubing, valves and fittings in the prototype. For example, new air-operated ball valves have been installed to avoid plugging problems experienced in the past. In addition, a new gas feed manifold system has been installed.
- High temperature "butterfly" valves (6") have been received for quick cool-down or venting of the reactor heating media.
- A central data acquisition system has been procured and is presently being programmed for use.

4. CHEMICAL CHARACTERIZATION OF WOOD-DERIVED OILS

The goals of chemical characterization are to develop techniques for assessing the quality of biomass oils, to gain an understanding through product identification of the probable chemical mechanisms operating in liquefaction, to provide analytical bases for eventual kinetics studies, and to identify potentially toxic or environmentally deleterious materials. In our last report we disclosed the results of a GC-MS (capillary gas chromatograph — mass spectrometry) study of the ether-soluble components of the aqueous effluent from Albany test run TR-7 (LBL process mode). We now report a similar study of material isolated from TR-7 oil by steam distillation. We have also analyzed or are in the process of analyzing the following materials: (a) oil derived from hydrolytically degraded glucose; (b) oil from pure cellulose; (c) oil from Klason lignin derived from Douglas Fir; (d) TR-8C oil (PERC process mode). Some preliminary results are reported and technical difficulties discussed.

Technicians at LBL's GC-MS facility report that biomass oils possess a remarkable ability to ruin capillary GC columns, apparently because of the deposition of thermally unstable tars near the column inlet. These materials, or their degradation products, elute continuously, creating intolerably high background noise levels. It appears that the latest column technology, to be commercialized in a matter of months, will be able to solve this problem. In the meantime, we chose to circumvent the difficulty by isolating for GC-MS examination only the most volatile fraction of TR-7 oil, that obtainable by exhaustive steam distillation.

A concentrated ether extract of steam distillate was analyzed directly, using an OV-225 SCOT column. The OV-225 stationary phase, a cyanopropyl phenyl silicone, cleanly separated the product into nonphenolic and phenolic groups. The 22 most abundant components, as arbitrarily judged by peak heights, are listed in Table IV. The same major classes of compounds previously found in TR-7 aqueous effluent are represented. Significantly, no hydrocarbons appear among the major components. Nor are there acyclic compounds of any type.

TABLE IV

Major Components of Steam Distillate of TR-7 Oil ^{a,b}

No. ^c	Time (sec)	Mass	Probable Identity	Type ^d
1	429	84	Cyclopentanone	CK-S
2*	446	98	Cyclohexanone	CK-S
3	486	98	3-Methylcyclopentanone	CK-S
4	694	112	Cycloheptanone (?)	CK-S
5	853	110	Dimethyl-2-cyclopentenone isom.	CK-S
6*	941	96	2-Methyl-2-cyclopentenone	CK-U
7	1114	124	Dimethyl-2-cyclopentenone	CK-U
8*	1173	110	Dimethyl-2-cyclopentenone isom.	CK-U
9	1246	110	Dimethyl-2-cyclopentenone isom.	CK-U
10	1302	124	Trimethyl-2-cyclopentenone isom.	CK-U
11*	1412	-	(Computer omission)	-
12	1467	-	(Computer omission)	-
13	1498	138	Alkyl cyclohexenone (?)	CK-U
14	1527	138	Alkyl cyclohexenone (?)	CK-U

15*	2184	124	Guaiacol	MP
16	2389	138	4-Methylguaiacol (Creosol)	MP
17*	2477	136	C ₉ H ₁₂ O (Alkyl phenol)	P
18	2548	152	4-Ethylguaiacol	MP
19	2630	122	C2 phenol isom.	P
20	2639	150	Alkyl Phenol	P
21	2709	166	4-n-Propylguaiacol	MP
22	2817	122	Ethylphenol isom.	P

^a Obtained from Albany Drum #5 (Sample TR-7 - 190)

^b GC conditions: OV-225 SCOT column; 50°C for 3-min, then 30/min to 250°C and hold.

^c The six most abundant components are starred.

^d CK-S: cyclic Ketone, saturated; CK-U: cyclic Ketone, unsaturated;
MP: methoxyphenol; P: phenol.

		<u>% O</u>	<u>% Phenols</u>	<u>H/C Ratio</u>
TR-7	Oil	14.4	32.2	1.2
TR-8C	Oil	6.5	13.5	1.2

The combined data suggest either that a mechanism for deoxygenating phenols operated to a much greater extent in test run TR-8C or that this oil still contains substantial quantities of the startup vehicle, anthracene oil.

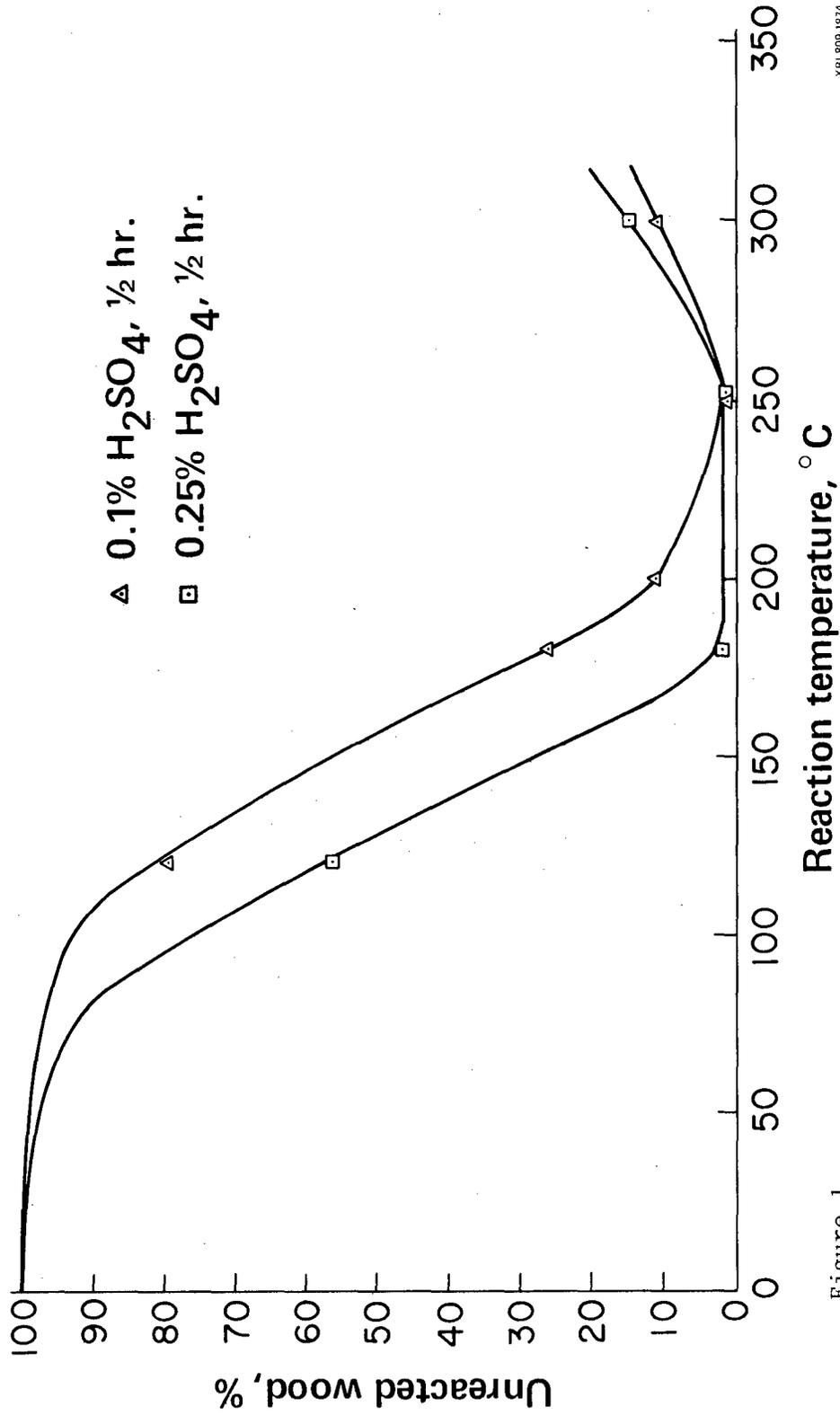
5. SOLVOLYTIC LIQUEFACTION

The feasibility of solubilizing up to 95 plus percent wood has been established using acidified alcohol or phenol as solvent. No hydrogen nor synthesis gas is required. The product is a soft black bitumen-like solid at room temperature with a heating value estimated to be 11,000 BTU/lb.

The effect of various process variables on the extent of wood solubilization was studied. Figure 1 depicts conversion versus temperature using phenol as solvent. It shows a u-shaped behavior indicating the existence of an optimum temperature above and below which less wood solubilization occurs. The increase in insoluble material at 300°C is indicative of repolymerization of depolymerized species. Acid concentration and reaction time versus conversion were also investigated. They all displayed the familiar u-shaped curve indicating existence of optimum process conditions. For example, for solvents like phenol, ethylene glycol and cyclohexanol, the optimum process conditions are 0.1 wt. % acid at 250°C for ½ hr.

Experimental data shows that the reaction is strongly catalyzed by acid. Low acid concentrations require a higher reaction temperature for good conversion and vice versa. Work is underway to better define this behavior.

After recovering the process solvent, the solvolytic product is a black, soft bitumen-like solid at room temperature which softens at 140°C. Scanning electron microscopy reveals that the fibrous cellulose elements of wood are completely destroyed. Instead the product is more or less crystalline. Elemental analysis of the product shows a 25-50% reduction in oxygen. Table V depicts a comparison of the solvolytic product with wood and coals of different rank. GPC study reveals that the mean molecular weight of the product is about 300 which accounts for its low melting point. HPLC and chromatographic work will be initiated shortly to characterize the chemical nature of solvolytic wood oil.



XBL605-1874

Figure 1

TABLE V.

	Wood	Solvolytic "Oil"	Lignite	Wyodak Subbituminous Coal
N	0.13	0.15	0.93	0.92
S	0.05	0.06	0.55	1.20
Ash	0.50	0.58	16.90	14.09
H/C	1.27	0.78	0.89	0.96
O/C	0.57	0.28	0.24	0.19

Table V. Ultimate Analyses of Wood, Solvolytic Oil and Selected Low-Rank Coals.

6. PROPOSED ACTIVITIES — FY 81

In FY 81 we expect finally to realize the promise of a unified, well-staffed and well-equipped group. Although two chemical engineers have left the group and will be missed, it is hoped that funding for their replacements will be available, along with money for the hiring of one or two technicians. Our graduate student is in the process of writing his doctoral dissertation now, but a candidate for an M.S. in chemical engineering is expected to join the group shortly.

The long-awaited fume hoods and laboratory benches for the analytical and heavy equipment facilities in the Dymo Building should be in place and operational by the end of the first quarter. This will permit high pressure batch autoclave equipment to be moved to the Dymo Building from the Richmond Field Station, resulting in the centralization of both personnel and equipment and many advantages in efficiency and safety control.

New equipment on the way includes two 300-ml high pressure autoclaves, a capillary gas chromatograph and a 10-gal, electrically-heated, stirred, 600 psi autoclave for wood hydrolysis experiments. Also, a colloid mill for posthydrolysis refining of wood slurries has been rented; its suitability will be evaluated during the first quarter of FY 81.

The 10-gal autoclave and colloid mill (or other appropriate refiner) will be used to provide the process evaluation unit (PEU), now scheduled for start-up in January 1981, with wood slurry feed. As a result of preliminary tests this past quarter, the PEU is undergoing a complete rebuilding. Also, during October-November an outside shed for gas cylinder storage will be constructed, an alarm system for monitoring toxic and flammable gas levels will be installed and an emergency reactor cool-down will be completed. These latter changes have been ruled to be essential safety features. A central data logger has been procured, installed and programmed. It will also be used to monitor operation of the 10-gal autoclave. If shakedown tests of gas feed, slurry feed and pressure letdown systems prove successful during the month of December, then the first continuous run could be conducted as early as January 1981. Initial runs will employ more or less standard LBL process conditions in order to establish a link between the Berkeley PEU and the Albany reactor. Subsequent operation will be directed toward both engineering and basic research ends, e.g., process optimization and engineering data collection.

The use of high pressure batch autoclave runs to pin down process variables in these highly complex systems will not be neglected either. With the use of the 300-ml high pressure autoclave or an improved tubular bomb (provided that we satisfy ourselves that tubular bomb results are meaningful), three sets of factorially-designed experiments are planned. The first set entails a three level factorial design involving temperature, time and pH in the hope of obtaining information concerning rates of reaction, activation energies and the shape of the pH-rate profile. In the second set the effects of gas composition and total pressure as well as the role of the water gas shift reaction will be treated. The third set will be designed around the question of the efficacy of recycling the various process streams, organic, mineral and aqueous, involved in wood liquefaction. In addition to these experiments, batch autoclave work will be used to screen selected lignocellulosic residues — hardwood, bagasse, sugar beet pulp, cellulose fines — as to suitability for injection into the PEU.

Basic chemical — analytical studies directed towards product characterization and evaluation will be extended. We hope to characterize various solvent-extracted fractions of TR-8C oil as the first step of a systematic study of product upgrading by solvent extraction. The question of the nature and origin of water-soluble organics obtained in the LBL process mode and their fate upon recycle will be examined further. Product distributions resulting from the liquefaction of cellulose, Klason lignin, glucomannan and appropriate model compounds will be determined in order to shed light on the mechanism of liquefaction in general and the role of carbon monoxide as a reducing species in particular.

Regarding Albany operations, LBL will continue a limited monitoring role in connection with LBL process runs. For test run TR-10, scheduled for late November 1980, LBL will provide data from batch autoclave runs as requested by Rust Engineering and will also station one observer at Albany for the duration of the run. TR-10 is designed to operate under conditions of slurry concentration, gas feed and residence time judged more favorable to the LBL process than those employed last year in TR-7. A better mass/energy balance should be obtained. Also, a study of the feasibility of recycling off-gas will be conducted.

Successful conclusion of the above program should result in the following accomplishments:

- o Much closer characterization of the LBL liquefaction process as to optimum conditions, effects of gas atmosphere, yields and product separation and usage of synthesis gas.
- o Improved characterization procedures for liquefaction products in general.

- o Improved insight into the mechanisms of liquefaction, at least into the differing behavior of the individual component types of wood.
- o Required information about the behavior of certain product streams upon recycle:

unreacted synthesis gas
aqueous phase, including the organic solubles
primary oil product.

7. EXPENDITURES

Operating (basic fund) 767K)

1) Total expenditures	\$735,782
2) Average monthly expenditures	61,315
3) September 1980	106,568
4) % expended - 96%	

Operating (Prototype) (Received 64K
in June)

1) Total expenditures	64,228
2) Average monthly	16,057

Total Overall Operating Expenditures	<u>\$800,010</u>
Capital Equipment	

1) Prior Year Carryover	35,000	
2) FY 80 Allocation (June)	32,500	
3) Expended and Encumbered		\$51,600*
4) Approx. Balance, 9-30-80	_____	<u>15,900</u>
	\$67,600	\$67,500

* May not be true cost as equipment is received.

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