

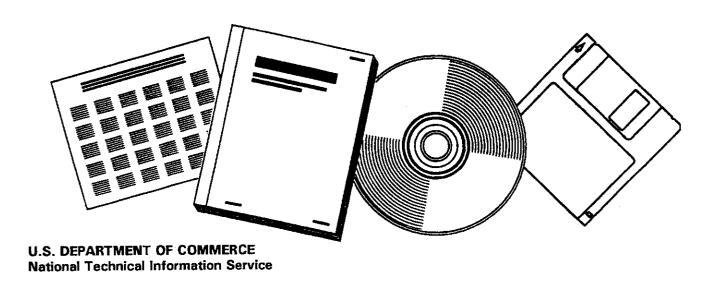
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# CONVERSION OF CELLULOSIC WASTES TO LIQUID HYDROCARBON FUELS: VOL. 1, PROJECT OVERVIEW: FINAL REPORT

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# CONVERSION OF CELLULOSIC WASTES TO LIQUID HYDROCARBON FUELS: VOL 1: PROJECT OVERVIEW

Submitted

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### ABSTRACT

A thermochemical conversion process to convert various biomass materials to diesel type fuels has been under development at Arizona State University (ASU) since 1975. An indirect liquefaction approach is used, i.e., gasification to synthesis gas followed by liquefaction of the synthesis gas. The primary virtue of an indirect liquefaction approach for cellulosic type feedstocks is that oxygen contained in the materials is easily separated. Thus the hydrocarbon liquid product is free of oxygenated compounds and can therefore be tailored to match transportation fuel products currently derived from petroleum. Approximately 100 biomass materials were studied as received from private industry, government laboratories and other university laboratories. The feedstock candidates included industrial wastes, agricultural and forest residues and crops that would be deliberately grown for energy conversion purposes. The product of the process is a liquid hydrocarbon transportation grade fuel similar to diesel. This can be upgraded to high octane gasoline via catalytic reforming if desired. The products should be compatible with existing engine designs and fuel distribution and marketing systems. The major virtue of the process is that a renewable, often low valued material is used as the feedstock to produce a quality product.

This final report consists of six volumes. This first volume contains an overview of the project from inception. Volumes 2-6 report on tasks completed and/or in progress in the reporting period from June 1, 1984 through May 31, 1985 that were at least partly funded by the Department of Energy. The volume titles are as follows:

Volume 1: Project Overview

- Volume 2: Kinetic Study of the Modified Fisher-Tropsch Synthesis over an Alumina-Supported Cobalt Oxide Catalyst
- Volume 3: Supervisory Control System Development for an Indirect Liquefaction Process
- Volume 4: Slurry Phase Synthesis of Liquid Hydrocarbon Fuels from Biomass Pyrolysis Gas Using Iron Catalysts
- Volume 5: Microwave Heating of Fluidized Bed Reactors--Pyrolysis and Calcination Applications
- Volume 6: The Modelling and Design of a Staged Indirect Liquefaction

  Reactor
- The individual report volumes are intended to be self standing.

### TABLE OF CONTENTS

	Pa ge
PRODUCT QUALITY	1
PRODUCT YIELDS	3
OPERATIONAL RELIABILITY	11
ENVIRONMENTAL COMPATIBILITY	15
THROUGHPUT	16
SIMPLICITY/AUTOMATION	16
SCALEUP	17
ECONOMICS	17
DIDI TOCO ADUV	18
BIBLIOGRAPHY	10
ACKNOWLEDGEMENTS	23

### PROJECT OVERVIEW

This volume will provide an overview of the project from the starting date of June 1, 1976 through the termination date of December 30, 1985. The general steps to be addressed for the project were as follows:

- 1) product quality
- 2) product yields
- 3) operational reliability
- 4) environmental compatibility ...
- 5) throughput
- 6) simplicity/automation
- 7) scaleup
- 8) economics

Product Quality. It was decided at project inception that the desired product would be a high quality, transportation grade liquid hydrocarbon fuel equivalent to that derived from petroleum. Thus, as opposed to the flurry of alcohol fuel development projects popular at the time, engine modification and alterations in the fuel distribution system would not be required.

A fundamental consideration in choosing a conversion path for converting cellulosic type materials to liquid hydrocarbon fuels is the elemental compositions of the feedstock and product. Typical values are as follows (wt%):

	Feedstock	Product
С Н	47 6 47	85 15 0

Thus oxygen has to be eliminated and the H/C ratio has to be enhanced. An indirect liquefaction route was chosen to achieve this goal. The first step was to consist of decomposing the feedstock to the basic reactive compounds of hydrogen, carbon monoxide and ethylene. In addition, carbon dioxide and methane were to be expected. To achieve efficient heat transfer and thus promote the formation of olefins, a fluidized bed system was selected for the decomposition step (pyrolysis, gasification). To avoid the probable gas separation of combustion products, a circulating bed mode was chosen (separate fluidized bed combustor (regenerator) and pyrolyzer (gasifier)). The generated gases were passed through a cyclone (to remove any particulates) and scrubber (to cool the gas and remove any condensible liquids) before being compressed into a catalytic liquefaction reactor. Here, the carbon monoxide, hydrogen and ethylene were to be converted to a paraffinic hydrocarbon fuel by the following general stoichiometry,

$$c_2H_4 + (n-2) co + (2n-4) H_2 + c_nH_{2n+2} + (n-2) H_2O$$

$$\frac{1}{2}(n-1) c_2H_4 + co + 3H_2 + c_nH_{2n+2} + H_2O$$

In addition, a secondary reaction to produce normal propanol was expected,

$$c_2H_4 + co + H_2 \neq c_2H_5CHO$$
  
 $c_2H_5CHO + H_2 \neq c_3H_7OH$ 

A fluidized bed system was initially selected for the catalytic liquefaction step to control the temperature in the presence of the significant exothermic heat of reaction. A slurry phase mode was later tested with the primary objective of relieving the complexity of operating fluidized bed systems in series.

The net result was that an oxygen free product  $(C_nH_{2n+2})$  could be produced with product distribution dependent upon catalyst choice and system operating conditions. The 11/C ratio could be manipulated by addition of steam to the gasification stop thus promoting the water-gas shift reaction,

$$co + H_2 O + CO_2 + H_2$$

The early stages of the project also explored the concept of producing a high octane gasoline product. This was achieved by passing the paraffinic fuel product through a fixed bed catalytic reformer using a commercially available catalyst. Here, the desired reactions to regulate molecular weight and introduce structural complexity were introduced. Octane rating could be manipulated over a broad range with little difficulty.

A schematic of the integrated conversion system (without the reforming step) is shown in Figure 1. This basic processing configuration remained essentially intact throughout the project, although numerous improvements were implemented for the various steps. An indication of the product quality attainable for this system (without product refining) is given in Table 1, as compared with commercial materials.

Product Yields. Calculated elemental balances for the conversion system indicated a maximum product yield (e.g. No. 2 diesel) of about 100 gals. per ton of feedstock (dry, ash free basis). Numerous factor studies were performed over the course of the project to approach this limit. Much of this experimentation was performed in separate, smaller scale equipment (see Table 2). A general list of factors is contained in Table 3. Details of these studies have been reported (1-6,10-52). The status at the termination of the project is summarized in Tables 4 and 5. As indicated, a yield of approximately 50 gals. of diesel fuel type product appears possible. Single

# CONVERSION SYSTEM SCHEMATIC

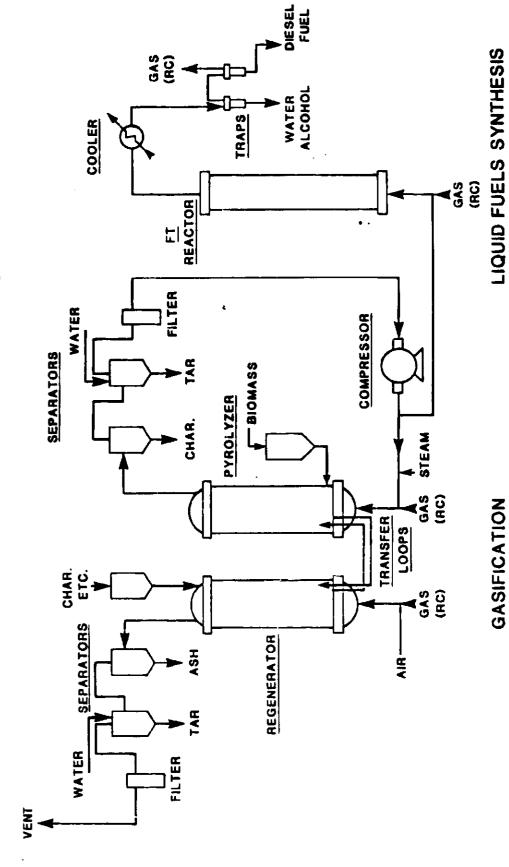


Figure 1. Indirect Liquefaction System Schematic (RC = recycle)

Table 1. Product Characteristics

	JP-4 (ASTH D1655)	JP-5 (ASTH D1655)	Kerosene (ASTK D3699)	Diesel #5. 2 (ASTM D975)	At Jet Fuel (ASTM D1655)	ASO Product	ASTH Test Method
Gravity, deg API # 60°F	57 max. 45 min.	51 max. 37 ada.			51 max. 39 más.	0.9	D-287
Specific gravity @ 60°F	.751 min.	.775 min.			.775 min.	.739	D-287
Distillation temperature, deg F							9 <del>-8</del>
10 percent evaporated 20 percent evaporated 50 percent evaporated 90 percent evaporated	290 max. 370 max. 470 max.	400 max.	401 max.	540 min. 640 max.	100 max.	230 256 336 503	
Final boiling foint, deg P	550 шаж.	572 mar.	572 mak.		572 MAK.	594	
Viscosity at 100 °F, ca			1.0 min. 1.9 min. (164 et)	1.9 sin. 4.1 sax.		96.0	p-445
Sulfur percent by weight	0.3 EAK.	0.3 max.	0.3 max. (No. 2-K)	0.5 max, 0.3	Esk.	90.0	P-129
Cloud point, deg F						21	76-4
Pour point, deg F						-43	D-97
Flash point, deg F			100 mta.	125 eds.		<75	p-92
Celane Number				to sin.		61.5	D-975
Spectrogiuphic Analysis Fe (Soap, AA Muthod), ppm Al Fe Cu Cu Ni						£2;555	

### Table 2. Experimental Systems

System	Description
Large Scale Integrated Unit:	10" pyrolyzer-regenerator circulating solid heating system with 6" liquefaction reactor. SIHI compressor. Gas recycle from liquefaction to gasification.
Small Scale Integrated Unit:	3" pyrolyzer staged with 6" liquefaction reactor. Electrically heated. Intermediate scrubber stage. Nash compressor for recycle.
Microreactor System:	microscale fixed and fluidized bed reactors with fluidized bath heater.
Slurry Phase Fischer-Tropsch Reaction System:	5" x 35" reactor with off gas and circulating circulating catalyst slurry system.
Liquefaction Catalyst Testing Unit:	2" fluidized bed reactors with common synthesis gas feed system. Six units in parallel. Additional reactor with recycle capability with compressor.
Process Control Equipment:	electronic controllers, recorders, data reduction equipment, etc. to monitor and control the various conversion systems.
Simulator Equipment:	plastic model units cross riser transfer loop, injector transfer loop, internal recycle transfer loop, etc.
Feedstock Preparation:	cutting, hammer and ball mills, pulverizer, chipper, screeners.
Feedstock Analysis:	heating values, extraction analyses, ash content, etc.
Catalyst Preparation:	hot plate mixers, calcining furnace, drying equipment, etc.
Analytical Support:	gas chromatographs, (5), gel permeation chromatograph.
Catalyst Characterization:	surface area, porosity, surface and bulk composition, etc.

### Continuous Extractor:

4" diameter x 7" screw extractor with continuous solids feeding and withdrawal and solvent delivery system.

## Microwave Heated Fluidized Bed:

915 MHZ, 0-30KW microwave source coupled to fluidized bed system with related control equipment.

### Table 3. Factor Studies

### Gasification:

- 1. Reactor system configuration
- 2. Feedstock characterization
- 3. Heat transfer media/catalyst
- 4. Fluidization gas composition
- 5. Residence Time
- 6. Temperature
- 7. Pressure
- 8. Recycle effects

### Liquefaction:

- 1. Catalyst composition
- 2. Catalyst preparation method
- 3. Catalyst calcination, reduction, pretreatment
- 4. Reactor system configuration
- 5. Conversion temperature
- 6. Conversion pressure
- 7. Conversion residence time
- 8. Feedgas composition
- 9. Recycle effects

### Table 4: GASIFICATION SYSTEM PRESENT STATUS

### Factors:

- a. sand, dolomite, catalyst heat transfer media
- steam + liquefaction reactor off gas fluidizing gas
- c. ~ 1 psig pressure
- d. ~ 1500°F temperature
- e. 1-5 secs. residence time
- f. no pyrolysis gas recycle

### Responses:

- a 85% feedstock conversion to gas
- b. gas composition, mole \$:
  - 15 olefins
  - 30 hydrogen
  - 30 carbon monoxide
  - 15 paraffins
  - 10 carbon dioxide

### Table 5. LIQUEFACTION SYSTEM PRESENT STATUS

### Factors: 1. Catalyst

- a. Co/Al203
- b. Impregnation (incipient wetness)
- c. No wash
- d. Calcination at 400°F, 4 hours
- e. Hydrogen reduction (1 atm, 750°F, 3 hours)
- f. No pretreatment

### 2. Conversion

- a. Fluidized bed, slurry reactors
- b. Temperature = 500°F
- c. Pressure = 140 psig
- d. Feed gas composition (mole \$) = 15 olefins, 30 H<sub>2</sub>, 30 CO, 15 paraffins, 10 CO<sub>2</sub>
- e. Residence time (single pass) = 15-30 secs.
- f. Recycle = 3/1

Responses: 1. Product quality = No. 2 diesel fuel

2. Product Yields = 40-50 gals. per ton of biomass feedstock (dry ash free).

pass high octane gasoline yields would be less (~20 vol. \$ yield loss through the reformer) but some of this could be recovered via off gas recycling.

A large list of feedstocks were investigated over the course of the project. (Table 6). These were supplied by private industry, government laboratories (various nations), municipalities, other universities, etc. Feedstock characteristics for these materials are summarized in Table 7. A range of synthesis gas compositions possible for these materials (at various operating conditions) is given in Table 8. The adjustments are not independent. Thus, for example, the  $\rm H_2/CO/CO_2$  trends were normally dictated by water-gas shift reaction effects.

The most promising areas for product yield improvement are:

- 1. Improvement of synthesis gas yields, (particularly with respect to olefin content). The use of an appropriate catalyst in the fluidized bed would appear to be the best approach. Note that the conversion system is already configured to continuously regenerate a fluidized catalyst.
- 2. Improvements in the liquefaction catalyst, particularly with regard to acceptivity away from noncondensible gases (methane, ethane, etc.).

Operational Reliability. The system, as configured in Figure 1, would be potentially sensitive to problems in the following primary areas: (1) solice feedings, (2) hot solids transfer, (3) tar condensation, and (4) catalyst activity maintenance. Problems were encountered and addressed in all of these areas over the course of the project, particularly in the early stages. All the solid handling steps were contained in the gasification system. Solids feeding problems were minimized when sufficient funds were available to purchase a reliable system. The system was operated to minimize tar formation and transfer lines were maintained above condensation

### Table 6 Feedstock List

		TENT	e a reedatock t		
1)	Tuphorbia lathyria Euphorbia lathyria	3\$)	Tall goldenrod Solidage altinoine	69)	Sryarbuah Abus trilebata
<b>2</b> 1	Contolilla baganne Exportia antisypailities	36)	Samefres Samefres albidus	10)	Terba-samta Eriodictyon angustifolis
3)	Nav guejule Parthesium argentatum	37)	Coral borry Symphoricarpos orbiculatus	713	Fourving salthumb Striplos camescome
4)	Susyale resime Parthenium argustatum	38)	Vilé bergamet Menaréa fistulosa	72)	Fetleaf backberry
5)	Guayalo bagasso Farthenium argentatum	39)	Russian thistle Salsels Hali	73)	Catelay minosa Minosa biuncifera
()	Guayalo care Parthemium argontatum	10)	Veter hyecistm Myscistbus spp.	74)	Sevage studge
73	Gressowed Sarrobatus vermiculatus	41]	Counce miliwood Asclopian syriage	75)	Core stares
•>	Jajoba sval Simmadsia chimensia	•2)	Swamp milkwemi Asclepiam incarnata	763	Coal
1)	ilacad bulla Termisalia catappa	43)	Peat Spagawa app.	773	Polyethylens
10)	Alsond shelig Terminalio savagea	20)	Portuguese sek cork	-87	Palyprapylane
113	Almond prunings Terminalia catappa	45)	Silver ample	791	Lignia
123	Sugareane tagasse Saccarum officizarum	44)	Tollowiesf sliktersel Sarrys Clavescom	301	Saw fung
13)	Yheat straw Triticum aestivum	47)	Sweet sorgawa Sorgawa seccueratwa	81)	Pager chiga
14)	Tresete busa Larrea tridentata	481	Pale Indian Siantala Caralia atripitesfolia.	12)	Hog fuel
.21	Fir bark Pseudotsuga senziesti	+91	Tali telifiower Tampanula americana	93)	Mesquite Presepts torreyase
16)	inizena lypnesa Turnessus anizonica	501	Theory elseagnus Classgous multiflors	14)	Caletrepia Caletropia procura
17)	Pringle sarzanita Arciestashyles Pringles	\$13	Grass leaved goldenred Solitage graminifolia	15:	Rice nulls
183	Vright silviasol Carrys urightii	52)	Compen elder Cambucum canademsia	16)	Vhoried mirveed taclepias verticilists
15)	Pointlesf canzenits Protostasnylos jungens	13)	Taraca vildrye Elyava manadens <u>ia</u>	37)	Stiff leaves goldenmes Solidago migida
10)	Suercus turateella	56)	field inistle firstum fiscolog	383	Trenweed Termenta missurica
21)	Service pure pressions	557	lew injaile Ionenus planaceus	59)	Toother spurge Supproofs contate
22)	7tab juniper Juniperus saceosperas	563	Ioneses plant Eliphium laciniatum	30)	Weeping william Talim recylonics
23)	Piryon pine Pinus edulis	572	Tanazgre roots Pumes tytenosepalus	31)	Carpenter's square Compositoria marijancica
	Velvet mesquite/fromosis	583	Sut leaf teasel Signerus laciniatus	72,	lapem Populum
	ECO Fuel II 'municipal preprocessed refuse;	-	five teasel Dipaccus sylvestris	931	locust lesizis
	Yew colp		American germander Teucrium canadense	94)	leresizated feruatored grayule
	felp residue		doody milweed dacuspids app.	9 <b>5</b> )	Flag snive
	failing russe Thus repailing		Totund-leaf milewood Tocolpias spp.	76 )	Whole joins Cummontais intrensis
	Isooth susse thus glears		Totton seed seat Tossipium thurberi	<b>;</b> *:	Perchany solute
	Yed tatarian toneysucwie lanters tatarica		Delinted rotton seed Cossyptum thurters	36 ?	Pecan inells
31	Zient reguesed Introxis intfids		Tation reed line Commypilm thursers		
•	Poseweed Phytolaces Emericana Tall coneses		Tottop reed se <u>el</u> Tossyppum tounters		
131	Tall toneses Eupathorium Altisateum		Tatton se <b>ed</b> bull <b>a</b> Trasyphia inurberi		
<b>]%;</b>	Posin weed		Totion gim irasm	Appre	eviational var. / variety isp. / ipecies

Table 7. Feedstock Characteristics (dry basis)

	Ranges	
Heating value, Btu/lb	7,400 - 12,700	
Ash, wt≸	0.1 - 35.9	
Protein, wt%	0.1 - 25.3	
Polyphenol, wt \$	0.1 - 20.2	
Oil, wt \$	0.03 - 9.20	
Hydrocarbons, wt\$	0 - 10.4	
Suberin, wt%	0.5 - 26.6	
Lignin, wt#	7.8 - 28.8	
Cellulose, wt \$	17.7 - 46.7	
Lipids, wt \$	5.1 - 14.9	
Elemental analysis, wt%		
C	37.7 - 60.9	
H	4.7 - 8.8	
ŋ	28.9 - 54.4	
N	0.3 - 1.7	
S	<0.01	

Table 8. Synthesis Gas Composition (mole %)

	Range	Typical	
Hydrogen	10 - 53	30	······································
Carbon Monoxide	6 - 60	30	
Olefins	5 - 39	10	
Paraffins	6 - 33	15	
Carbon Dioxide	4 - 26	15	

temperatures up to the inlet of the scrubber. Clinker formation problems were essentially eliminated with proper choice of fluidized solid and proper fluidization control. At the termination of the contract, the integrated system was essentially an unattended operation with the exception of loading of the feed hopper and dumping of the product tank. Continuous run lengths were never scheduled, however, for more than a five day period. Long term effects (erosion, tar buildup, catalyst activity etc.) remain for future work.

Environmental Compatibility. The areas to consider in the process with regard to compliance with environmental regulations are as follows:

- (1) particulates (from feedstock preparation and storage, ash removal).
- (2) air emissions (from regenerator vent and liquid product storage),
- (3) liquid effluents (from scrubber water processing), and
- (4) noise control.

At the laboratory scale, none of the above were studied in depth other than to achieve a safe working environment for operating personnel. Thus, the scrubber systems were considered to be well oversized to achieve sufficient dilution before disposal, the laboratory was continuously vented during runs with an exhaust system, appropriate masks were worn when handling feedstocks, etc. Compatibility with environmental regulations is not considered to be a major hurdle, however. Pyrolysis type technology lends itself to minimal air emissions control problems. The major stream of concern is the scrubber effluent. On a commercial scale, separation of organic condensibles from the water stream would probably be implemented with disposal of the organics in the gasification regenerator and recyle of the water to the process distribution system. Scrubber effluent samples were retained for all documented runs performed in the laboratory. Analysis

were performed on selected samples (5,6). The project was terminated before any detailed correlation of feedstock type and processing conditions vs. effluent composition could be developed.

Throughput. The continuous, integrated laboratory system was considered to be commercially realistic with regard to processing steps, operating modes and procedures. However, the configuration at the end of the contract period was limited with regard to throughput (defined as quantity of feedstock processed/volume of equipment) by the regenerator size and/or efficiency. Thus, as feedstock flow rates were increased, eventually it would be impossible to maintain desired pyrolysis temperatures. Equipment height in the laboratory was limited by the laboratory roof. Recognizing that a taller regenerator would be required to optimize throughput, a major renovation was funded in the laboratory to accommodate the planned throughput study. Unfortunately, although the renovation was completed, so was the project budget. Thus, this aspect remains for future study.

Simplicity/Automation. It is recognized that the process technology is analogous to that utilized in a typical chemical plant or refinery. As limited by delivered feedstock costs, any facility processing cellulosic wastes will be relatively small, however, thus limiting the advantages of economy of scale with respect to operating labor requirements. Thus, process simplicity and/or reliable, vendor supported automation is considered an absolute necessity for commercial implementation of the technology. This was addressed throughout the project. A thorough discussion regarding automation of the process can be found elsewhere (10, 19, 28, 38). A major effort to simplify the process via a staged conversion

system design was also performed (11), but not implemented by the project termination date.

Scaleup. A detailed design of a nominal 10 ton/day pilot plant was performed based on data supplied by the project laboratory. This study has been reported separately (9).

Economics. Several economic studies were performed externally on the process (7-9). Cost figures for the 10 ton/day design were based on vendor quotations. Using the pilot plant numbers as a base, an economic model using a chemical plant simulator package was under development at the end of the contract period. The intent of the model is to accurately project process costs at various scales based on individual units rather than overall scale factors. The model will also enable one to tailor the design to specific site and feedstock requirements.

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