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Preliminary Engineering and Cost Analysis of Purdue/Tsao Cellulose Hydrolysis (Solvent) Process

October 1978

Prepared For:
U.S. Department of Energy
Assistant Secretary for Energy Technology
Fuels from Biomass Branch
Under Contract No. ET-78-X-01-4641

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October 1978

Prepared By:
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Chicago, Illinois
Under Contract No. ET-78-X-01-4641

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U.S. Department of Energy
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Fuels from Biomass Branch
Washington, D.C. 20545

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ENGINEERING AND COST ANALYSIS OF
PURDUE/TSAO CELLULOSE HYDROLYSIS
(SOLVENT) PROCESS

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I. SUMMARY

Using information published on the Purdue/Tsao Acid Solution Process for the Hydrolysis of Ligno-Cellulosic materials - specifically corn stovers - an engineering and cost analysis was performed for a battery limits facility to produce sufficient glucose syrup for 25 million gallons per year of ethanol.

A capital investment estimate of 59 million dollars was derived. This estimate was based on vendor quoted equipment prices and a detailed consideration of all aspects of constructing the facility.

The product transfer cost of the fermentable sugars - pentoses and hexoses - was estimated at 4.5¢ per pound.

The major factor impacting the commercial feasibility of such a facility is the price assigned to the delivered corn stover.

Although considerable development work on the process is required before it will be ready for commercialization no technical problem was uncovered to preclude this commercialization.

II. INTRODUCTION

a. Background:

The hydrolysis of cellulosic materials to produce useful end products has been evolving in recent years from the work of the 1930's, 1940's, using strong acids. Much re-interest has resulted from the enzymatic hydrolysis work by Natick Laboratories.

Recently, a Purdue University group headed by Dr. George Tsao has made what appears to be a major breakthrough in the acid hydrolysis area. The Tsao/Purdue effort directs itself to the refractory nature of native cellulose to enzymatic or mild acid treatment. This resistance to treatment had heretofore limited the yield of glucose.

The approach to this problem taken by Dr. Tsao has been to treat the cellulosic material with any one of a number of solvents in such a way as to break down the crystallinity of the cellulose and remove the protective cover of the lignin. After this treatment the cellulose is easily hydrolyzed with either acid or enzyme and the hydrolyzate yield approaches quantitative.

The overall process as conceived by Dr. Tsao and so far released to the public consists of three main operations; mild acid pretreatment to separate the pentose fraction; solvation

II. INTRODUCTION (Continued)

to effect the above mentioned modification of the cellulose/lignin structure; hydrolysis and separation of the hexose fraction.

There have been three process schematics shown by Dr. Tsao using as solvents cadoxen, CMCS (EMNN) and sulfuric acid. Other solvents according to Dr. Tsao have potential; for reasons of economics and/or potential hazards, sulfuric acid is the solvent of choice at this stage of development.

In all three process schematics the solvent is recovered and reused in the process.

b. The Acid Solvent Process Overview:

The cellulosic material to be processed, be it corn stover, bagasse, wood or other such materials, is pretreated by grinding or chipping to prepare it for easy handling and processing. Using a dilute acid stream recycled from later stages of the process, the pentosans are hydrolyzed to pentoses. These now soluble solids, the "pentose" fraction, are separated from the remaining insoluble ligno-cellulose complex, neutralized and stored ready for further processing into finished fermentation products.

II. INTRODUCTION (Continued)

In preparation for the solvation step the insoluble residue is dried to about 10% moisture in a low pH condition which reputedly aids the subsequent processing. The dried residue is intimately mixed with concentrated (70-85%) sulfuric acid (recovered from subsequent operations) which "dissolves" the cellulose. The cellulose is precipitated out by the addition of methanol.

The cellulose at this stage is amorphous and is easily depolymerized by acid or enzyme to glucose and other fermentable sugars.

To keep the economics of this process within reasonable bounds it is necessary to have good recovery of both the sulfuric acid and the methanol. To attain these recoveries the process is designed to minimize losses and maximize separation and recycle of these materials. It is necessary to control the various operations so that concentration levels are maintained at certain key steps in the process to both balance out the flows and achieve the critical yields. This must be achieved thru proper selection of the process equipment.

II. INTRODUCTION (Continued)

c. The Engineering Design:

This study is a preliminary engineering design of the Tsao/Purdue Process. The process design presented is as described in reports to the D.O.E. and supplemented by some discussions with Dr. Tsao on certain aspects relating to properties of the process streams. Most of the design basis is contained in Dr. Tsao's block flow schematic. (See Fig. 1)

This study has concentrated on selecting equipment and arranging flows so as to achieve the results implicit in the Tsao schematic. The equipment considered was essentially "shelf-type" equipment proven in applications for similar processes; e.g., wet corn process, wood hydrolysis, cane processing, etc.

Since this process has been developed only on bench scale equipment, considerable intuitive judgement was required in making the above mentioned technology transfer.

The engineering effort on this study was applied only to the basic Tsao Process of conversion of ligno-cellulose to pentose, hexose and lignin fractions. As proposed by the Tsao schematic the lignin is used as a fuel in the power plant. The two sugar fractions would serve as feed stock for other processes such as ethanol and 2, 3-butanediol as suggested by Dr. Tsao.

II. INTRODUCTION (Continued)

The capital estimate for this study was based on vendor quoted equipment prices and the construction costs were based on Arthur G. McKee's experience in estimating similar process plants.

The plant was sized to produce a dextrose feed stock for 25 million gallons per year of 190 proof ethanol. Since a large volume of pentoses are produced in such a plant the optimum size must consider very carefully the economics of the potential product mix dictated by the market place.

The alcohol volume above sets the agricultural residue input to the plant at about 600,000 dry tons per year. At 2.2 tons per acre, approximately 288,000 acres are required to supply this plant. This area is encompassed in a circle about 12 miles in radius.

This study was conducted by Chet Gunter, Process Engineer, under the supervision of E. Kirchner, Manager, Process Engineering of the Chicago Office of Arthur G. McKee.

III. PROCESS DESCRIPTION

This narrative describes the process developed by Dr. Tsao as it has been translated into process hardware.

Storage and Preparation-

Storage for thirty day operations has been provided for stover. Half of this volume is bailed, half is stored loose. The bailed stover is stacked 20 feet high on a self draining concrete pad 750 feet by 750 feet. The loose stover is piled 25 feet high on a similar pad 500 feet square. Truck delivery to the plant site is assumed. The stover is stored by two front end loaders.

During operation (see Process Flow Diagram E-03-OI-01-001 PFD 001). The stover is transferred from storage by the front end loaders or directly from incoming truck to the feeder table, 101. The action of this table cuts and spreads the stover evenly onto the conveyor 104. Tramp metal is removed and the stover is fed into the mill, 107, which grinds it to 1/4" pieces. The mill discharges into a pneumatic conveyor, 108, which moves the ground stover to the pentose extraction system.

This process handles over 100 tons per hour of bulky material. Because of the similarity to sugar cane the same type of operation and equipment is suggested here.

-Pentosan Hydrolysis-

The ground stover is collected in a cyclone separator, 110, and the exhaust air is cleaned by a dust collector, 111.

The cyclone discharges the stover into a feed bin, 112, which acts both as a surge hopper and a pretreatment bin where the stover is preheated and deaerated by direct injection steam.

The stover is metered continuously into the extraction column, 114, by the meter 123. More direct injected steam is introduced at the top of the column to quickly raise the temperature to about 250 F.

In the column the stover flows downward countercurrent to dilute sulfuric acid solution. The hot acid hydrolyzes the hemicellulose to pentoses. Because of the countercurrent flow the solids concentration of the pentose stream leaving the extraction column can be about 20%. This stream is used to preheat the feed to the methanol recovery still (PFD 007) before going to the pentose storage tank, 167 (PFD 006), where it is neutralized with lime.

The dilute sulfuric acid recovered from the methanol-acid still (PFD 007) is injected into the lower section of the extraction column. It is preheated to the operating temperature by direct injected steam.

The pentose-free residue at the bottom of the extraction column is slurried to a pumpable condition by solution from the overflow of tank 119. The slurry is forced out of the bottom of the column to tank 119.

The extraction column, 114, is similar to those used in the wood pulp industry for essentially the same type of extraction. It is felt this would be a good choice as the test work required can be reduced significantly. This selection was based on the Tsao schematic. The kinetics of this hydrolysis need further studying. Tsao's mass balance shows significant glucose in the pentose stream. It has been reported that a clean fractionation of pentoses can be achieved under proper conditions.

-Cellulose Solvation-

The residue is dewatered to about 50% moisture with roller screw presses, 126 before being sent to flash dryers (PFD 002) where the moisture is further reduced to 10%.

The drying is necessary to avoid dilution of the concentrated acid used in the solvation step. The residue is dried while still acidic as this improves the recovery of sugars.

A major portion of the energy consumption in this process is due to drying of this residue. A reduction of this requirement from that used in this design is possible.

A key step in this process is the dissolution of the cellulose by concentrated sulfuric acid. The acid is intimately mixed with the dry residue by feeding the two into two paper mill refiners, 133. These are rotating disc machines which will assure adequate wetting of the fibers with the acid. The residue is discharged into a holding tank, 134 for a mean residence time of about ten minutes. The cellulose desolves out of the lignin matrix and this mixture discharges from the bottom of the tank into the agitated cellulose precipitation tank, 140. The cellulose is precipitated by the addition of the methanol at ambient temperature.

The solvation step in the process will need study to choose the best combination of equipment. A pulp refiner has been selected to effect the wetting of the dried residue with concentrated acid because it appears that the design of the plates would assure intimate contact and also because these mills are well established process items. However, it may be necessary to recycle the mill discharge to achieve the desired results. Higher acid concentration (i.e. over 70%) may be desirable since this would facilitate solvation and there would be less of corrosion problem. This advantage would be off-set by the need for more lime to neutralize the additional acidity.

After the acid and residue are mixed the material will start to form a fluid in tank 134. The best design for this tank will require experience with larger size batches than so far used.

The slurry of cellulose-lignin in the acid-alcohol solution is pumped to solid bowl scroll centrifuges, 142, where the acid-methanol solution is separated and sent to the recovery system (PFD 007). The solids are continuously discharged to the wash tank 145. The acidity is reduced from 7% to about 2.5% to achieve the concentration needed in Dr. Tsao's pentose extraction. The reduction is done at this point in the process for two reasons. It holds down construction costs needed to accomodate the higher acid levels downstream and accomodates the purging of the gypsum precipitate formed in this process.

There is still a considerable amount of acid and methanol entrained in the residue which must be removed and recovered. This washing is done by a multi-stage hydroclone system, 147, (PFD 004) such as is used in the wet corn mill in a process for washing starch slurry. The flow through system is counter current to the flow of fresh water introduced in the final stage, which removes 99+% of the acid and alcohol from the residue of lignin and cellulose. The acid-alcohol leaves the system in the first stage and is sent to the recovery process (PFD 007).

-Acid Hydrolysis of Cellulose-

The slurry will discharge from the last stage of the hydroclone at a solids concentration which may be controlled between 30 and 40%. The slurry is acidified in tank 149 and pumped to the reactor, 155, where steam is simultaneously injected to raise the temperature to about 250°F. A residence time of about 10 minutes will hydrolyze the cellulose to glucose. An overall yield of about 90% fermentable sugars is expected.

The discharge of the reactor is flash cooled, 161, and neutralized with lime and sent to horizontal belt filters, 157, (PFD 005). The sugar solution is filtered from the lignin and the lignin is washed free of the sugar by spraying fresh water on the filter cake. The combined filtrate is the final glucose fraction which is sent to storage tank 166.

- Acid Methanol - Recovery-

(PFD 007)

The overflow from the first stage hydroclone, 147A, contains about 14% methanol and 2% sulfuric acid in water. This stream is preheated, 117, and sent to a distillation column 303, where the methanol is recovered as light ends and 99+% pure to be sent to the methanol storage tank 201 for reuse. The bottoms

from this column is a 2.5% sulfuric acid solution which is used directly in the pentose extraction column, 114. Materials of construction need to be investigated to keep costs of the column down. Vacuum distillation may be necessary to keep construction materials from getting too elaborate.

The solution from the centrifuges 142, which recovered the lignin and cellulose contains about 60% methanol 27% acid and 13% water. This mixture is separated in a triple effect evaporator into 99+% methanol and 68% sulfuric acid. The latter can be brought to the desired 70% concentration by addition of make up 98% acid and reused in the solvation process.

The separation of 70% sulfuric acid and methanol is one of the more difficult design problems. It is believed that a triple effect evaporator can be used because of the vast vapor pressure differences between 70% sulfuric and methanol at the temperature at which the evaporators would operate. Research needs to be done to explore the physical characteristics of this system. Also, materials of construction need to be investigated to keep the costs of the system at a reasonable level.

-Enzyme Hydrolysis Alternatives-

Dr. Tsao has recommended the acid hydrolysis step because of better economics compared to enzymatic hydrolysis. This assumption must be confirmed considering the yields of fermentable sugars as well as future costs of enzymes. This study assumes that enzymes will be produced off-site by commercial enzyme manufacturers.

There are two basic processes in starch hydrolysis using enzymes; acid plus enzyme and all enzyme. The advantage of an acid-enzyme process with cellulose would be reduced equipment costs because acid could carry the partial depolymerization to an economical degree much faster than enzyme. The final high yield of glucose can be achieved with cellulase.

-Alternate A - Acid-Enzyme-

The cellulose/lignin slurry from tank 149 is acid converted in reactor 155 under conditions to achieve approximately 25% depolymerization. Lime is added to adjust the pH to approximately 4.5 so that enzymes from tank 178 (PFD 004) may be added by pump 181, to complete the hydrolysis. It is believed that only two hours through a continuous enzyme converter, 153, is required because the greater part of the conversion is accomplished by the acid conversion.

The enzyme converter is like those described in Alternate "B". The sugar/lignin slurry is pumped, 156, to the lignin separators 157A, B and C (PFD 005) and then the process becomes like the acid case.

-Alternate B - Enzyme -

The enzyme is mixed in the agitated enzyme tank 178 (PFD 004) and metered, 180, into the reactor surge tank 149. A conversion time of 24 hours at 50°C is assumed with a conversion yield to fermentable sugars of 95%. This conversion is accomplished in twelve, 171 A thru L, 1 thru 12, agitated compartments that overflow one to the other. These tanks are arranged in four batteries of three tanks high with transfer pumps, 175, B and C, to lift the mixture up to the next set of tanks and replace heat losses with heat from steam in exchangers 173, A, B, and C. From the last stage the dextrose solution is pumped, 177, to the lignin separators, 157 A, B, and C, and from thereon the process is exactly like the acid case.

a. Process Flow Diagrams

- Hemi-Cellulose Hydrolysis PFD E-03-0I-001
- Flash Drier PFD E-03-0I-002
- Cellulose Hydrolysis PFD E-03-0I-003
- Acid/Enzyme Hydrolysis PFD E-03-0I-004
- Enzyme Hydrolysis PFD E-03-0I-005
- Receiving & Storage PFD E-03-0I-006
- Solvent & Acid Recovery PFD E-03-0I-007

DESCRIPTION	STEAM NO.	1	2	3	4	5	6	7	8	9	10	11	12	13
FLOW	MB/HR	14.3	24.3	17.8	13.5	144.4	106.0	125.0	109.4	99.4	91.8			7.5
FLOW	GPM	11.2	19.0	13.7	10.4	112.0	82.0	96.0	84.0	76.4	69.4			5.8
WATER	MB/HR	1.5	2.5	1.8	1.3	14.4	10.6	12.5	10.9	9.9	9.1			0.6
OTHER	MB/HR	0.1	0.2	0.1	0.1	1.4	1.0	1.2	1.1	1.0	0.9			0.07
LIGNIN	MB/HR	0.1	0.2	0.1	0.1	1.4	1.0	1.2	1.1	1.0	0.9			0.07
GYPSON	MB/HR	1.1	1.4	1.0	0.7	7.4	5.4	6.2	5.4	4.9	4.5			0.3
ASH	MB/HR	0.4	0.4	0.3	0.2	4.4	3.2	3.7	3.3	3.0	2.7			0.2
FERM SUGARS			42.1	45.4	45.9	41.0	44.3	44.7	40.1	36.6	33.6			
SULFURIC ACID	MB/HR													
ENZYME	MB/HR													

UTILITIES	USE RATES
STEAM 174	7.4
HORSEPOWER (174)	15

DATE TO	1	2	3	4	5	6	7	8	9	10	11	12	13
CLIENT													
FIELD													
NO.													

EQUIPMENT MANUFACTURERS' SUPPLIERS AND SUB-CONTRACTORS COMPLIANCE WITH REQUIREMENTS OF OCCUPATIONAL SAFETY AND HEALTH ACT, RULES AND REGULATIONS HAVING JURISDICTION ON THE WORK BY FEDERAL, STATE AND LOCAL AUTHORITIES IS A MANDATORY CONTRACTUAL REQUIREMENT.

ARTHUR G. MCKEE & COMPANY

CHICAGOILLINOIS

DEPT. OF ENERGY

ENZYME SACCHARIFICATION

CONTRACT NO. GC-4967

SCALE: NONE

DATE: 10/1/68

BY: [Signature]

APPROVED: [Signature]

ENZYME SACCHARIFICATION

E 03-01-01-005

DESCRIPTION	STREAM NO.	1	2	3	4	5	6
FLOW	MPH	438.1	254.7	186.3	13.5	262.3	33.7
FLOW	GPM	870	500	370	260	520	670
WATER	MPH	85.4	215.3	76.2	0.3	262.3	33.7
METHANOL	MPH	262.3	33.7				
SULFURIC ACID	MPH	116.4	5.7	130.1	13.2		

UTILITIES	USE RATE
STEAM	HP/HR 100.0
HORSEPOWER	451
TOWER WATER GPM	17000

ISSUED FOR CONSTRUCTION

BY _____ DATE _____

DATE TO _____

CLIENT _____

FIELD _____

BY _____

DATE _____

APPROVES _____

DATE _____

EQUIPMENT MANUFACTURERS', SUPPLIERS' AND SUB-CONTRACTORS' COMPLIANCE WITH REQUIREMENTS OF OCCUPATIONAL SAFETY AND HEALTH ACT - RULES AND REGULATIONS HAVING JURISDICTION ON THE WORK BY FEDERAL, STATE AND LOCAL AUTHORITIES IS A MANDATORY CONTRACTUAL REQUIREMENT.

ARTHUR Q. McKEE & COMPANY

CHICAGO ILLINOIS

U.S. DEPT. OF ENERGY

PURDUE/ISAQ PROCESS

P.O. E-1-78-X-01-4641

SCALE _____ BY _____ CONTRACT NO. GC-4967 R. H. NO. _____

DATE 6-30-78 BY _____

APPROVES _____

DATE _____

SOLVENT & ACID RECOVERY

E-03-01-007-

b. Equipment List

MCKEE EQUIPMENT LIST-TSAO PROCESS

P. L OF 16

ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
101	FEED TABLE	Designed to evenly feed baled agricultural residue of 13#/ft. ³ or 6#/ft. ³ at a rate of 130TPH on the stover conveyor, hydraulically driven.	CS Rubber	30	56,000	\$136,000	
102	STOVER FEED CONVEYOR	A drag chain design 6 ft. wide by 50 ft. long to transport 130 TPH agricultural residue 6#/ft. ³ , hydraulically driven.	CS	75	62,000	\$148,000	
103	FEED TABLE KNIVES	To reduce 6 ft. long by 1-2" dia. at 130TPH baled agricultural residue to 1 ft. lengths. Bulk density of bail 13#/ft. ³ , loose 6#/ft. ³ .	CS	75	Part of 101	Part of 101	
104	STOVER CONVEYOR & MAGNET	Constructed to receive agricultural residue from Item 101 at the rate of 130TPH at 6#/ft. ³ to transport residue over the magnet and separate tramp iron larger than 1/4"Ø from the residue. Belt conveyor to be 6 ft. wide by 30 ft. long. Magnet to be slider type covered by 316SS wear table over electro magnetic coils.	CS	30		\$50,000	
107	STOVER PULVERIZER	Designed to process 130TPH agricultural residue 12" long x 1-1/2"Ø with bulk density 6#/ft. ³ in mat 6' wide by approximately 3' high of approximately 60% fiber and 40% moisture and reducing it to sheds not thicker than 1/4".	CS	2000		\$90,000	

MCKEE EQUIPMENT LIST

P 2 OF 16

ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
108	STOVER BLOWER	Rotary positive displacement blower for pneumatic transport of 20 #/ft. ³ material at 130 TPH thru cyclones and bag collectors 180 ft. 1 ft., 50 ft. run, with two 90° elbows.	CS	600		\$142,333	
109	STOVER FEEDER	Rotary feeder 130 TPH at 20 lbs./ft. ³	CS	20		See 108	
110	STOVER SEPARATOR	Cyclone separator with replaceable lining for 130TPH residue at 20 lbs/ft. ³	CS			See 108	
111	DUST COLLECTOR	Approximately 1800 ft. ² filter area, with rotary lock discharge and exhaust fan.	CS	160		\$26,250	
112	EXTRACTOR FEED BIN	5,000 ft. ³ , 28'-8" H x 17' Ø.	CS			\$7,300,000 Turn-Key	
113	FEED LOCK	Rotary valve 130TPH 6#/ft. ³ material 15 psi P	Incoloy 825	2		See 112	
114	EXTRACTION COLUMN	120' H 19' Ø with steel supports, platforms.	CS acid brick Hastelloy "B"			See 112	
115	EXTRACT PUMP	500 GPM 200' TDH centrifugal pump with mech. seals.	Durmet 100	50		See 112	

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McKEE EQUIPMENT LIST

P 3 OF 16

ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
116	DISCHARGE PUMP	2,000 GPM 100' TDH centrifugal pump with mech. seals.	Durimet	125		See 112	
117	COUNTER CURRENT EXCHANGER	Shell and tube 600 GPM @ 250°F. to 170°F. 450 GPM @ 100°F to 200°F. sulfuric acid	Graphite FRP lined CS			\$60,000	
118	DISCHARGE COOLER	Shell and tube 2,000 GPM acid at 190° to 160°F.	Graphite			\$117,400	
119	DEWATER SURGE TANK	26' H X 20'Ø - 50,000 gal. flat bottom cone roof.	CS Rubber lined			\$290,000 Turn-Key	
120	SURGE TANK AGITATOR	Side entering propellor with double mech seals.	316 L SS	125		See 119	
121	DEWATERING PUMP	2,000 GPM 100' TDE centrifugal pump with mech. seal.	CD4M	125		\$6,300	
122	BIN FEEDER	Rotary valve - 130 TPH 25 lbs./ft. ³ material.	CS & CI	15		See 112	
123	BIN METER	Rotary vane - 130 TPH 25 lbs./ft. ³ material.		15		See 112	

McKEE EQUIPMENT LIST

P 4 OF 16

ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
126A & B	RESIDUE PRESS	Compaction rolls 80,000 lbs./hr.	316 SS	110 ea.		\$3,536,000	\$707,200 ea. five required. Manufacturer will not make the advertised larger equipment.
129A & B	RESIDUE DRIER	Flash type 80 TPH feed 30 TPH water evap. 800°F. Air temperature incl. cyclone and fan, and feeders.	CS	533 ea. Drier		\$628,000	\$314,000 ea.
132A & B	DRIER FURNACES	Combustion chambers for 123 A & B 84 x 10 ⁶ Btu/hr., oil fired with comb air fan and oil pump.	CS	1 ea.		\$200,000	\$100,000 ea.
133A & B	REFINER-DEFIBERIZER	45 TPH fiber feed 20 lbs/ft. ³ material. 10 mesh product discharge.	Carpenter 20 & CS	1000 ea.		\$320,000	\$160,000 ea.
134	RESIDENCE TANK	7,000 gal. 12' H X 10' Ø flat bottom vertical.	CS Rubber lined			\$12,000	
136	RESIDENCE TANK AGITATOR	Slow speed paddle bottom sweep - stuffing box.	CS Rubber lined	25		\$6,000	

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McKEE EQUIPMENT LIST

P 5 OF 16

ITEM NO.	N A M E	D E S C R I P T I O N	MAT'L CONST.	HP.	WEIGHT	P R I C E	R E M A R K S
139	CELLULOSE PRECIPITATION TANK AGITATOR	Flat blade turbine with mech. seal.	Rubber lined CS	15		\$12,000	
140	CELLULOSE PRECIPITATION TANK	20,000 gal. 20'H x 13' Ø - sloping bottom.	Rubber lined CS			\$21,000	
141	CELLULOSE TRANSFER PUMP	1,100 GPM - 100' TDH centrifugal with mech. seals.	Alloy 20	75		\$9,000	
142A & B	CELLULOSE SEPARATORS	40 IPH - Solid bowl centrifuge 15% solids feed 60% solids discharge.	Alloy 20 Rubber lined CS	250 ea.		\$1,000,000	\$500,000 ea.
144	WASH TANK AGITATOR	SAMES AS 139	See 139	15		\$12,000	
145	WASH TANK	20,000 gal. 20' H x 13' Ø	Rubber lined CS			\$21,000	

McKEE EQUIPMENT LIST

P 6 OF 16

ITEM NO.	N A M E	D E S C R I P T I O N	MAT'L CONST.	HP.	W E I G H T	P R I C E	R E M A R K S
146 A thru L	WASH TANK PUMP	1,000 gpm @ 260' TDH centrifugal pump with mech. seal.	CD4M	100		\$84,000	\$7,000 ea.
147 A thru L	DORRCLONE WASH SYSTEM	450 gpm 34% solids feed, 40% solids discharge, wash 2.5X. solids.	316 SS			\$350,000	\$29,167 ea.
148	REACTOR SURGE TANK AGITATOR	SAME AS 139.		15		\$12,000	
149	REACTOR SURGE TANK	20,000 gal. 25' H X 12' ϕ	FRP			\$11,000	
150	REACTOR SURGE TANK PUMP	600 gpm 100' TDH centrifugal with mech. seals.	CD4M	40		\$7,000	
151	ROTARY SCREEN	450 gpm - 10 mesh.	316 SS	5		\$4,100	
153	ENZYME CONVERTER	62'L x 12' ϕ - 12 baffled compartments 24 HR ₂ . hold up. 500 gpm.	FRP			\$113,000	

McKEE EQUIPMENT LIST

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ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
154	REACTOR AGITATOR	10 - 3 blade propellers.	Inconel	20		\$578,000	
155A	DEXTROSE REACTOR	20' H x 3.5' ϕ with 10 baffled compartments.	Inccnel			See 154	
155B	HOLDING COIL	2' ϕ x 36'L four pipes in series.	Inccnel			See 154	
156	SEPARATOR FEED PUMP	500 gpm @ 100' TDH centrifugal pump with mech. seals.	316 SS	25		\$3,100	
157 A, B & C	LIGNIN SEPARATORS	Belt type filter 500 gpm with vac. pumps and separators.	316 SS	127 ea.		\$519,000	\$173,000 ea.
158 A & B	EJECTOR	150 psig. steam supply 100 lbs./hr. air cap. 4" Hg ABS.	316 SS			See 154	
159	SEAL WATER SURGE TANK	2,000 gallon.	316 SS			\$3,000	
160	SEAL WATER PUMP	200 gpm at 100' TDH		10		\$2,000	
161 A & B	FLASH COOLER	7,000 gal. 4" Hg ABS. int press and ATM 13' ϕ and 10' ϕ . with TFE mist eliminator.	316 SS			See 154	
162 A & B	BAROMETRIC CONDENSER		CS			See 154	

McKEE EQUIPMENT LIST

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ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
163	DEXTROSE NEUTRALIZER PUMP	500 gpm 100' TDH centrifugal pump with mech. seals.	316 SS	40		\$3,100	
166 A & B	DEXTROSE STORAGE	50,000 gal. 24' H x 19' ϕ .	CS			\$48,000	\$24,000 ea.
167 A & B	PENTOSE STORAGE	SAME AS 166.	CS			\$48,000	\$24,000 ea.
168	DEXTROSE TRANSFER PUMP	500 gpm 100' TDH centrifugal pump with mech. seals.	CS	25		\$2,000	
169	PENTOSE TRANSFER PUMP	SAME AS 168	CS	25		\$2,000	
170	LIGNIN CONVEYOR	Rubber belt conveyor 15 TDH 40 lb./ft. ³ material 300'L 2'W covered.	CS	100		\$60,000	
171 A thru L	ENZYME CONVERSION TANKS	12 ft. Dia. by 62 ft. long partitioned FRP tanks to convert cellulose to dextrose via cellulose enzymes in 24 hrs. at a temperature of 122°F (max) to 116°F (min). Tanks are to be compartmentalized with individual agitators and over flow from one compartment to the next. Tanks are to be arranged on a superstructure in two batteries of six tanks each.	FRP			\$960,000	\$80,000 ea.

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ITEM NO.	N A M E	D E S C R I P T I O N	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
172A thru L 1 - 12	ENZYME TANK AGITATORS	To be mounted on tanks 171A thru L on flanges to mix compartments 5 ft. long by 12 ft. dia. dextrose at pH 4.5 and 122°F. (max.) viscosity approximately 100 cps and sp. gr. 1.1.	316 SS	1 ea.		See 171	144 H.P. Total
173 A, B, C	HEAT EXCHANGER	Shell and tube 11C,000 btu.hr. steam (Shell) to fluid in 122°F. out 250°F.	Graphite & C.S.			\$75,000	\$25,000 ea.
174	DEXTROSE SURGE TANK	20,000 gal. 13' ϕ x 20' S.S.	C.S. Rubber lined			\$21,000	
175	DEXTROSE SURGE TANK AGITATOR	For 174.	See 174	15		\$12,000	
176A, B, C	TRANSFER PUMPS	500 gpm 75' TDH centrifugal w/mech. seal.	LD4M	15		\$9,300	\$3,100 ea.
177	TRANSFER PUMP	500 gpm 75' TDH centrifugal w/mech. seal.	CD4M	15		\$3,100	
178	ENZYME TANK	6,000 gal. 10' H x 10' ϕ jacketed 50 psi jacket press nozzles manholes, agitator flange ASME code.	316 SS			\$24,000	
179	ENZYME TANK AGITATOR	Propellor type 3 ft. ϕ 4 blade.	316 SS	2		\$3,000	72

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McKEE EQUIPMENT LIST

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ITEM NO.	N A M E	D E S C R I P T I O N	MAT'L CONST.	HP.	WEIGHT	PRICE	R E M A R K S
180 & 181	ENZYME PUMP	Piston type - metering pump variable flow 0-5 GPM.	316 SS TFE	1 ea.		\$2,500	
182 A thru L	ENZYME CONVERTER AGITATORS	Flange mt'd propellor type mech. seals.	316 SS	1 ea.		See 153	

McKEE EQUIPMENT LIST

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ITEM NO.	N A M E	D E S C R I P T I O N	MAT'L CONST.	HP.	WEIGHT	P R I C E	R E M A R K S
201	METHANOL STORAGE TANK	250,000 gal. flat bottom.	CS			\$71,000	
202	METHANOL TRANSFER PUMP	600 gpm 100' TDH centrifugal with mech. seals.	CS	15		\$2,000	
203	METHANOL UNLOAD PUMP	500 gpm 100' TDH centrifugal with mech. seals.	CS	25		\$2,000	
210 A & B	98% SULFURIC ACID STORAGE	250,000 gal. 32'H 37' Ø flat bottom with foundation	CS			\$120,000	\$60,000 ea.
211 A & B	ACID PUMPS	25 gpm 100' TDH centrifugal with mech. seals.	CS	3 ea.		\$2,000	\$1,000 ea.
212	SULFURIC ACID UNLOAD PUMP	500 GPM 100' TDH centrifugal with mech. seals.	CD4M	25		\$3,000	
215	20% LIME TANK	3,000 gal. 8' H X 8' Ø	CS			See 218	

McKEE EQUIPMENT LIST							
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ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
216	LIME TANK AGITATOR	3 blade propeller type.	CS	5		\$3,000	
217 A & B	LIME PUMPS	100 gpm 100' TDH centrifugal stuffing box.	CS	10 ea		\$3,000	\$1,500 ea.
218	LIME STORAGE BIN COLLECTOR	Bag filter 160 ft. ² cloth area.	CS			\$203,500 Complete lime set	
219	LIME BIN DISCHARGER	Vibrating bin bottom.	CS	3		See 218	
220	LIME SURGE BIN	1,200 ft. ³ - 24' H x 9' Ø	CS			See 218	
221	LIME TRANSFER BLOWER	Rotary positive displacement blower 5 TPH 300 ft. run, 100 ft. L. ft. approximately 500 CFM.	CS	40		See 218	
222 A & B	LIME STORAGE SILO	21,310 ft. ³ bolted construction 72' H x 21' Ø mounted on weigh cell pads.	CS			See 218	
223 A & B	LIME SILO DISCHARGER	Bin bottom vibrator.	CS	10 ea.		See 218	

McKEE EQUIPMENT LIST

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ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	K.P.	WEIGHT	PRICE	REMARKS
224	LIME SILO COLLECTOR	Approximately 160 ft. ² cloth area reverse air cleaning dust collector.	CS			See 218	
225	LIME UNLOADING BLOWER	15 TPH approximately 700 CFM. Rotary positive displacement blower.	CS	40		See 218	
226	LIME CAR AEREATION BLOWER	Rotary positive displacement blower approximately 200 CFM. for car air pads.	CS	20		See 218	
227 A & B	LIME TRANSFER FEEDERS	Rotary valve 5 TPH 125 lb./ft. ³ mat'l.	CS	1 ea.		See 218	
228	LIME FEEDER	SAME AS 227.	CS	1		See 218	
230 A & B	FRONT END LOADERS	To handle 130 TEH ea. of 6#/ft. ³ corn stover with 5.5 ton grab.	CS			\$248,120	\$124,060 ea.
231	RAIL SCALE		CS			\$67,000	
232	TRUCK SCALE		CS			\$20,000	

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ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
301	DISTILLATION FEED TANK	100,000 gal. 24' H x 27' ϕ .	Rubber lined C.S.			\$70,000	
302	DISTILLATION FEED PUMP	100 ft. TDH 500 GPM centrifugal mechanical seals.	CD4M	20		\$3,000	
303	DISTILLATION COLUMN	Bubble cap trays - to separate 2.5% H_2SO_4 solution from methanol. Methanol 99+ $\%$ in overs and bottoms 99+ H_2SO_4 .	FRP & Inconel			1.4×10^6	
304	REBOILER	Shell and tube thermo syphon a reboiler.	Graphite & CS			See 303	
305	CONDENSER	Shell and tube.	316 SS			See 303	
306	EXTRACTION PUMP	400 GPM @ 100' TDH centrifugal w/mech. seal.	CD4M	20		\$3,000	
310	FIRST EFFECT HEATER	To transfer 8.0×10^7 Btu/hr. using 25 psig steam to heat methanol - 70% sulfuric acid solution at 2,500 GPM to approximately 240°F.	Graphite & C.S.			\$24,800	

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McKEE EQUIPMENT LIST							
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ITEM NO.	N A M E	D E S C R I P T I O N	M A T ' L C O N S T.	H P.	W E I G H T	P R I C E	R E M A R K S
311	FIRST EFFECT EVAPORATOR	To be operated at approximately 2ATM and approximately 184°F to separate approximately 100,000PPH methanol 2300GPM liquid. TFE entrainment from separator included.	Hastelloy B			\$30,000	
312	SECOND EFFECT HEATER	To transfer 4×10^7 Btu/hr. using approximately methanol vapors and liquid at approximately 150°F.	Graphite FRP lined CS			\$89,000	
314	THIRD EFFECT HEATER	Same as 312 except vapor at approximately 150°F and liquid at approximately 120°F.	See 312			\$97,000	
313	SECOND EFFECT EVAPORATOR	Same as 311 at atmospheric pressures.	FRP			\$35,000	
315	THIRD EFFECT EVAPORATOR	Same as 311 at approximately 360mm Hg absolute.	FRP			\$65,000	

MCKEE EQUIPMENT LIST

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ITEM NO.	NAME	DESCRIPTION	MAT'L CONST.	HP.	WEIGHT	PRICE	REMARKS
316	THIRD EFFECT CONDENSER	Vapcr at approximately 120°F cooling water at 85°F for 4×10^7 Btu/hr.	Graphite & CS			\$49,000	
317	STEAM EJECTOR	To evaluate 100#/hr air at 360mm Hg absolute pressure constructed of C.S. with 316 SS nozzle.	CS			\$1,100	
318	FIRST EFFECT PUMP	2500 GPM at 100' TDH constructed of Alloy 20 mechanical seal NEMA 7 460V/3PH/60HZ.	Alloy 20	125 HP		\$12,000	
319	SECOND EFFECT PUMP	SAME AS 318.	Alloy 20	125 HP			
320	THIRD EFFECT PUMP	SAME AS 318.	Alloy 20	125 HP			
321	EVAPORATOR FEED PLMP	600 GPM @ 75 TDH Alloy 20, mechanical seal, NEMA 7 460V/3PH/60HZ.	Alloy 20	30 HP		\$5,000	
322	EVAPORATOR FEED TANK	100,000 gal. 27'Ø x 24' H CS with rubber lining flat bottom cone top.				\$70,000	

c. General Arrangement of Equipment

PLANT DESCRIPTION

The site plot plan shown incorporates not only the hydrolysis plant (the subject of this study) but also, the process areas for the fermentation products.

The total site encompasses almost 60 acres, about one third of which is used for one month stover storage. The area required for hydrolysis is seen to be less than one-quarter of the total.

IV. COST ANALYSIS

a. Capital Costs:

The summary of the capital costs for the hydrolysis of corn stover to pentose and hexose sugars by the Purdue/Tsao Process is given in Table I.

Equipment costs are based on actual vendor quotations for these engineered equipment selections. The balance of the capital costs were developed by Arthur G. McKee's Estimating Department based on long experience in the design and construction of similar process plants. Consideration was given to the total requirements for process piping and utility services for this plant layout. The estimate assumes that the plant is on a site with good soil bearing qualities and no special piling, drainage or stabilization requirements, and it allows for the foundation and structural requirements for the types of equipment and buildings specified.

For convenience in evaluating various process section contributions to the total capital requirements the estimate has been divided into eight process sections.

The total estimate is for a "battery limits" plant for the processing of corn stover to fermentable sugars assumed to be feed stock for such products as ethanol and 2, 3 butanediol.

IV. COST ANALYSIS (Continued)

The ethanol process has been well reported recently. The butanediol process is less well known. Since the bulk of the waste will be from these sections of an operating plant no capital for waste treatment is included in the hydrolysis section.

Utility capital has been allocated to the sections of the process approximately according to each section's requirements for steam and electricity.

The direct capital is summarized thus:

	<u>\$ Million</u>	<u>% Dir. Cap.</u>
Feed Stock Preparation	3.2	7.8
Pentosan Hydrolysis	10.0	24.3
Dehydration	10.8	26.3
Cellulose Solvation	6.6	16.1
Hexosan Hydrolysis (Acid)	2.5	6.3
Lignin Separation	1.4	3.4
Chemicals Storage	.9	2.2
Acid/Alcohol Recovery	<u>5.6</u>	<u>13.6</u>
TOTAL DIRECT CAPITAL	41.1	100
Engineering & Construction	6.2	
Contingency (15% Dir. Cap.)	<u>6.2</u>	
TOTAL FIXED	53.4	
Start Up	2.5	
Working Capital (2 month mat'l @ cost)	3.0	
TOTAL CAPITAL INVESTMENT (Land not Included)	59.0	

IV. COST ANALYSIS (Continued)

A 25 million gallon alcohol facility may add about 15 million dollars while a butanediol section another 20 million dollars so with additional utilities the total complex may reach 100 million dollars.

TABLE I

PURDUE/TSAO HYDROLYSIS CAPITAL COST ESTIMATE
 (\$1,000's)

Facility Construction
 August 1978

	Feed Prep.	C-5 Hydrolysis	Dehydration	Solvation	C-6 (Acid) Hydrolysis	Lignin Separation	Chemicals Storage	Acid/ Alcohol Recovery	Total
Equipment	841	4,640	4,364	1,851	685	587	410	1,954	\$15,332
-Installation	308	2,700	2,200	962	350	240	213	1,018	7,991
Instruments	50	50	405	230	175	75	25	315	1,325
Piping	67	371	873	370	208	88	41	586	2,604
Electrical	897	137	386	1,149	45	155	63	133	2,965
Building	-	-	925	392	175	124	-	-	1,616
Site	171	941	885	375	167	119	83	396	3,137
Utilities	900	1,200	800	1,300	650	30	20	1,200	6,100
TOTALS	3,234	10,039	10,838	6,629	2,455	1,418	855	5,602	\$41,070

Indirect

Engineering	3,286
Construction Management	1,643
Fee	1,232
Contingency	6,160

TOTAL FIXED CAPITAL \$53,391

Manufacturing Costs

The manufacturing cost for producing the two fractions of fermentable sugars are detailed in Table II. Since this study is confined to a battery-limits facility these costs are actually the transfer costs to the finishing processes.

These costs are summarized thus:

	<u>\$1,000</u>	<u>¢/lb.F.S.</u>	<u>%</u>
Raw Materials	\$21,080	2.39	53.2
Utilities	7,620	0.87	19.3
Other Variable Costs	3,893	0.44	9.8
Factory Overhead	1,680	0.19	4.2
Depreciation	<u>5,339</u>	<u>0.61</u>	<u>13.5</u>
TOTAL TRANSFER COSTS	\$39,621	4.50	100.0

As can be seen the major contribution to these costs are the raw materials. Seventy-eight percent (78%) of the raw materials is due to the cost of the stover which is priced at \$30 per dry ton. However, only about 80% of this price is charged into raw materials as the balance was charged to fuel as contribution of the lignin to the utilities costs. A ten dollar per dry ton change in stover costs will change the cost per pound of sugars by 0.7¢.

TABLE II

MANUFACTURING COST ESTIMATE

C-5 AND C-6 FERMENTABLE SUGARS

FROM PURDUE/TSAO ACID HYDROLYSIS PROCESS

BASIS: Corn stover to produce 25 million
 GPY ethanol. 7920 hr. per yr. operation.

PRODUCTS: 327.7 E6 lb. "Hexose"
 554.4 E6 lb. "Pentose"

	<u>Unit Cost</u>	<u>Units Per Yr.</u>	<u>\$1000 Per Yr.</u>	<u>¢/lb. F.S.</u>
<u>Raw Materials:</u>				
Stover	\$24.30/Ton*	.62MM	15,012	1.702
Sulfuric Acid	\$ 0.02/lb.	106.90MM	2,138	.242
Methanol	\$ 0.08/lb.	20.80MM	1,666	.189
Lime	\$ 0.02/lb.	98.20MM	1,964	.223
Misc. Chemicals	-	-	300	.034
Total Raw Materials			\$21,080	2.39
<u>Conversion Costs:</u>				
Operating Supplies	-	-	154	.018
Maintenances (M&L)	6% Fixed Cap.		2,460	.280
Operating Labor	\$12.11/Hr.	105.6MM	1,279	.145
Sub Total			\$3,893	.441
<u>Utilities:</u>				
Purchased Fuel	\$0.340/Gal.	10.63MM	3,614	.411
Purchased Electricity	\$0.035/Kwh	11.88MM	416	.047
Generated Energy	\$1.500/MM Btu	1.93MM	2,892	.329
Process Water	\$0.350/M Gal.	.42MM	147	.017
Cooling Water	\$0.040/M Gal.	14.00MM	560	.064
Total Utility			\$ 7,629	.868
Total Conversion Costs			\$11,522	1.306
Total Variable Manufacturing Cost			\$32,602	3.70

*19% of Stover Cost allocated to Lignin Fuel

Manufacturing Costs (Continued)

The balance of the raw materials costs is distributed among the cost for makeup sulfuric acid and methanol and for lime used to neutralize the acidic sugar streams before storage.

The makeup methanol is estimated at 1% of the flow to the recovery system. Even at this low estimate the contribution to the transfer cost is 0.2¢ per pound. The loss of methanol during process or the need for a bleed stream to purge impurities can have a significant effect on the economic viability of this process.

The production labor requirements for this operation has been estimated at eleven shift workers plus seven day laborers including shift supervision. Factory overhead charged to this portion of the operation is estimated at about 40% of operation labor and maintenance costs.

There is not enough lignin to supply all of the fuel requirements of this process so it is used to fire the steam generating boiler which needs to have a small supplement of fuel oil. The two large residue dryers use fuel oil exclusively. Boiler stack gases are sufficient to supply only half the needs of one of these dryers; this was not incorporated into these costs. Co-generation of fuel was used with a net cost for the resulting steam and electricity of \$1.50 per million Btu's. Since the utility cost represent 20% of

Manufacturing Costs (Continued)

the total transfer costs energy conservation in the final design of such a facility should be given considerable attention. Since drying of the residue is the largest single energy consumer it should get first attention.

Depreciation charges are based on a 10 year project life.

The total energy input to this process is 2800 Btu per pound of stover.

V. CONCLUSIONS

Although the subject engineering design is based on preliminary bench scale work for the purposes of capital investment and manufacturing cost the results obtained appear reasonable.

This conclusion is reached because the potential for technology transfer seems good. This is not to say that substantial pilot scale testing is not required. Such testing may suggest other equipment than used in this design but probably would not adversely change the capital estimate. There appears to be a number of ways to improve some of the operations which would reduce costs, however, they would not significantly affect the conclusions drawn from the present estimates.

The major question in the manufacturing cost estimate is the cost of the stover which in this estimate Tsao's figure of \$30 per dry ton was used. Even though about 20% of this \$30 is assigned to fuel costs the contribution to the variable manufacturing cost from stover alone is about 46%. Even with co-generation using the lignin as fuel the utilities contribute 19% to the variable cost.

This report has not addressed the problems of harvesting and storing the corn stover or the effect of removal from the land.

c. Product Value Analysis

The estimate transfer cost of 4.5¢ per pound of fermentable sugars appears marginally competitive to cane molasses. However, five carbon sugars (xylose) are not fermentable to the same products as the six carbon sugars (glucose). Since the 5 carbon sugars are recovered utilizing both less capital equipment and energy the transfer costs could be allocated according to such useages between the two type sugar fractions. There appears to be no general agreement in the literature of the relative proportions of cellulose and hemi-cellulose; the actual numbers used would of course effect the derived costs.

V. CONCLUSIONS (Continued)

From a manufacturing process view, a major problem is the low bulk density of stover. Close to 20 acres of storage space are required at the plant site just for one month's supply.

How the process will be effected by variation in quality of the stover as it ages through the season is unknown. The fact that the moisture of the stover changes substantially during this period will have a significant effect.

On the basis of an average yield of 2.2 ton/acre-year it has been calculated that 288,000 acres would be required to supply a plant of this size. Possibly 3 to 4 times this area would be required in a practical sense. However, this expanded distance for delivery to the plant site may create an economic problem in its operation. A smaller plant means less product whose revenue must support the operation. This optimization must be made eventually.

This study has not given attention to the possible problem of a reaction between the menthanol and sulfuric acid. At the conditions in this process it is not immediately obvious that there would be such a problem. However, consideration should be given to the fact that these materials will be in contact for a very long period so that even extremely slow reaction kinetics could eventually create a problem.