1953

PROBLEM

As a recently hired engineer in the alcohol-process section of a large synthetic-detergent manufacturer, you have been asked to determine the maximum capacity of a new sodium-reduction unit for converting coconut oil to fatty alcohol for sodium alkyl sulfate manufacture and to submit a finished report on your calculation for discussion at a management meeting. For planning purposes, management is interested in the maximum possible production rate of distilled alcohol from the unit as limited only by the equipment and by safety requirements.

Since the plant will not be operating on stream before the meeting date, it will be impossible to determine the maximum rate by actual operation of the entire system. Therefore, equipment specifications; data from tests performed to date on this and other units; and general information about the process which should enable you to calculate the maximum capacity of the existing drying, reduction, hydrolysis, and distillation steps as pounds of distilled fatty alcohol per hour have been furnished to you from the factory technical service and the research departments. It is recognized that the rate which you calculate may not be the most efficient operation of the system with regard to cost per pound of distilled alcohol, but maximum production rate is the primary objective of these calculations that you have been asked to make.

Information from an article in Industrial and Engineering Chemistry of a few years back has been furnished you to acquaint you with the general picture of the entire operation, including equipment and chemical reactions. In conjunction with the article you have been cautioned to use only the process data furnished you on the new unit, since it differs somewhat from the one discussed in the article. Comments which should answer many of the questions that might occur to someone new to the process have been added to the data sheets given you.

So that the details of your calculation will follow the standard report form of the company for this type of presentation, a general outline has been included in your data brochure.

Attachments:

1. Information from I. & E. C. article
2. Proposed outline for report
3. Plant equipment specifications
4. Miscellaneous plant data
5. Laboratory and literature data
6. Safety requirements

SPECIAL REPORT OUTLINE

1. Brief summary of calculations, indicating the limiting factor to capacity in present equipment and the calculated maximum capacity in pounds of distilled alcohol per hour.
2. Index of report.
3. Detailed calculations. (Break down into sections, each headed by a sentence or short paragraph indicating the phase of the calculations covered in that section.)
4. Summary and conclusions.

PLANT-EQUIPMENT SPECIFICATIONS

Ejector for Single-stage Vacuum Drier for Coconut Oil

Performance Data

Capacity, (lb. water vapor/hr.)	Pressure, mm. Hg abs.
0	2.5
50	12.5
90	25.0
120	38.0

Heat Exchanger for Coconut Oil Drier

Heating capacity	400,000 B.t.u./hr. maximum
Design	Internal, G-fin, steam heat exchanger (Assume over-all heat transfer coefficient to be essentially constant over range of flow rates to be considered)
Heating medium	150 lb./sq. in. gauge steam

Reduction Reactor

Maximum usable working volume	260 cu. ft.
Diameter	5.5 ft.
Turbine agitator diameter	2.0 ft.
Turbine speed	120 rev./min.
Agitator motor horsepower	5

(Heat cannot be removed effectively through walls; consider all latent heat to be absorbed completely by the reflux system.)

Condenser capacity	4,000,000 B.t.u./hr. maximum
Feed line - maximum rate	1,000 lb./min.
Sodium drop line - maximum rate	200 lb./min.
Toluene drop line - max. rate	500 lb./min.

Hydrolysis Tank

Maximum usable working volume	300 cu. ft.
Condenser capacity	4, 000, 000 B.t.u./hr. maximum

(Heat cannot be removed effectively through the walls; consider all latent heat to be absorbed completely by reflux system.)

Drop line from reactor - maximum rate	1, 000 lb./min.
Pump-off pump capacity	100 gal./min. maximum
Heating capacity of coil in tank	30, 000 B.t.u./min.
Water pump-in capacity	100 gal./min.

Solvent Still

- 30-plate bubble-cap fractionation column
  - 6-ft. column diameter
  - 2-ft. spacing between plates
  - 1-in. liquid seal on bubble caps
- 3, 500, 000 B.t.u./hr. condenser (maximum)
- Dowtherm-fired reboiler (5, 000, 000 B.t.u./hr. capacity)
- Reflux returned to plate 30 (top)
- Solvent mixture withdrawn on plate 25
- Feed inlet from still feed tank on plate 20
- Return of recirculated bottoms from reboiler on plate 1
- Feed to alcohol still is also withdrawn from bottom of column
- Water is withdrawn from the separator after the condensation of the overhead stream

Alcohol Still

- 4-plate bubble-cap column (plates used for entrainment separation only)
- 7-ft. column diameter
- 2-ft. spacing between plates
- 1-in. liquid seal on bubble caps
- 1, 400, 000 B.t.u./hr. condenser (maximum)
- Dowtherm-fired feed preheater (1, 000, 000 B.t.u./hr. capacity)
- Reflux is returned to top plate
- Feed inlet to pot below bottom plate
- Bottoms line from pot connects through the recirculation pump to the feed line to the still ahead of the preheater
- Bottoms are withdrawn from the recirculation line just after the recirculation pump

General

The layout of the new plant equipment differs from the I. & E. C. flow chart in that a single-stage vacuum drier for drying the CNO and a flash-type still for alcohol distillation are used. All auxiliary equipment not specified herein may be considered to be of adequate capacity.

MISCELLANEOUS PLANT DATA

Coconut Oil (CNO)

Temperature from storage	100°F.
Free fatty acid	0.5%
Moisture	1.0%
Coconut oil	98.5%

Solvents

Toluene for sodium slurry from toluene gauge tank	
Moisture and impurities	nil
Temperature	225°F.
Toluene - MIC* solvent mix from solvent still	
Impurities	nil
Moisture	0.05%
Temperature	170°F.

*Methyl isobutyl carbinol or methyl amyl alcohol

Sodium

Impurities nil

One pound of toluene is used to slurry each pound of sodium in the reduction reactor

Temperature 240°F.

Reduction

Normal minimum lost time during reduction cycle 5 min.

Hydrolysis

- Normal minimum settling time for phase separation after boiling = 5 min.
- Normal minimum lost time during quench cycle = 5 min.
- Temperature of water for hydrolysis = 80°F.
- Minimum allowable Na_2O concentration in aqueous layer is 16% owing to the limitations of the process equipment utilizing this by-product.

Solvent Still

- Feed temperature = 170°F.
- Water in feed = 1.5% by weight
- Solvent ratio in overhead (azeotrope) to condenser (lb. toluene/lb. MIC) = 5.7
- Operating pressure on top plate = 760 mm. Hg abs.
- Pressure drop per plate = 2 mm. Hg
- Solvent mixture overhead is supercooled in condenser to 170°F. (At this temperature part of the water separates from the azeotrope.)
- Solvent content in water withdrawn from separator is insignificant.
- Reflux ratio (lb. returned to plate 30 from separator/lb. solvent withdrawn from plate 25) = 2.0
- Water in reflux to top plate = 1.5% by weight
- MIC or water content in bottoms is insignificant.
- Maximum temperature of bottoms from Dowtherm reboiler is 620°F.

Measured maximum capacity of recirculation system for bottoms at 450°F. is 150 gal./min.
Heat losses are minimized by tracing and insulation.

Alcohol Still

Feed temperature essentially the same as the bottoms from the solvent still
Operating pressure on top plate = 4 mm. Hg abs.
Pressure drop per plate = 2 mm. Hg
Reflux ratio (lb. returned to top plate/lb. alcohol product withdrawn) = 0.25
Maximum feed temperature possible from preheater = 620°F.
Measured maximum capacity of recirculation system for bottoms at 400°F. is 20 gal./min.
Heat losses are minimized by tracing and insulation.
Ejector capacity is adequate for all possible rates.

Alcohol condensate temperature = 200°F.
Losses = 1 lb. alcohol/lb. of soap in still feed (Laboratory tests have shown that this loss is due to thermal decomposition of alcohol catalyzed by the soap. The decomposition products are essentially nonvolatile.)

LABORATORY AND LITERATURE DATA

Reactants (General)

Molecular weights

Coconut oil (CNO)	659	(triglyceride)
MIC	102	
Toluene	92	

Usages
- 5% excess sodium over theoretical for CNO reduction
- 5% excess MIC over theoretical for CNO reduction

Specific Gravities

	Temperature, °F.									
	50	100	150	200	210	225	250	300	350	390
CNO	--	0.92	--	--	--	0.87	--	--	0.82	
CNO-fatty alcohol	0.85	--	0.80	--	--	--	0.75	--	0.70	
Sodium (liq.)	--	--	--	--	0.93	--	--	0.91	--	0.89
MIC	0.81	--	--	0.74	--	--	--	--	0.67	
Toluene	0.87	--	--	0.80	--	--	--	--	0.73	

Mean specific gravity of aqueous layer in hydrolysis tank = 1.23
All volumes are considered to be additive

Alcohol still bottoms

	Temperature, °F.			
	420	500	580	660
Specific gravity	0.814	0.782	0.750	0.718

All specific gravities referred to water at 39.2°F.

Heat Capacities, B.t.u./(lb.)(°F.)

Toluene	mean = 0.45
MIC	mean = 0.70
Sodium	mean = 0.33
Hydrolysis-tank solvent layer	mean = 0.80
Hydrolysis-tank aqueous layer	mean = 0.82
Nonvolatile fraction of alcohol still bottoms	mean = 1.20

	Temperature, °F.					
	100	200	300	400	500	600
CNO	0.48	0.54	0.60			
CNO fatty alcohol	--	0.65	0.83	1.08	1.32	1.60

Latent Heats of Vaporization, B.t.u./lb.

	Temperature, °F.					
	100	200	300	400	500	600
MIC	185	171	154			
Toluene	152	141	124			
CNO fatty alcohol	145	139	130	120	110	100

Vapor Pressure, mm. Hg abs.

	Temperature, °F.								
	122	158	194	230	266	338	410		
Toluene	92	203	410	735	-	3,310	7,310		
MIC	22	64	171	380	685	3,150	9,440		
	280	320	360	400	440	480	520	560	600
CNO fatty alcohol	10.5	28	70	150	300	550	980	1,800	3,000

Boiling-point Data @ 760 mm. Hg abs.

	Boiling points, °F.
MIC	271
Toluene	232
CNO fatty alcohol	505

All boiling solutions can be considered ideal.
The hydrolysis-tank mixture boils at approximately 210°F., and the b.p. variation with composition is insignificant.

Azeotropes

	B.P. @ 760 mm. °F.	% of Water by weight
Toluene	183	13.5
MIC	202	44.4

Viscosity

Final reaction mass (reduction reactor) = approximately 20 centipoises @ 250°F. for solvent ratios of 1.3 to 2.3

Alcohol still bottoms -

	Temperature, °F.		
	500	600	700
Viscosity, centipoises	50	12	3.2

Coconut Oil Equilibrium Moisture

% H_2O @ equilibrium	Pressure, mm. Hg abs.	Temperature, °F.
0.016	10	350
0.018	10	300
0.024	10	250
0.025	25	300
0.025	50	350
0.032	50	300
0.034	25	250
0.042	10	200
0.045	50	250
0.064	25	200
0.090	50	200

Reduction Reaction Data

Effect of time and solvent ratio on crude alcohol yield in reduction reactor

Pounds crude alcohol/100 lb. dry, fatty-acid-free CNO fed	Feed time, min.	Solvent ratio, lb. toluene /lb. MIC
68.0	100	2.3
74.0	100	1.3
74.5	60	2.3
78.0	100	2.1
78.3	60	1.3
80.9	60	2.1
81.0	20	2.3
81.0	100	1.5
82.6	20	1.3
82.8	60	1.5
83.0	100	1.9
83.8	20	2.1
84.0	100	1.7
84.1	60	1.9
84.6	20	1.5
84.7	60	1.7
85.2	20	1.9
85.4	20	1.7

Side-reactions products can be considered to be only high-molecular-weight ethers formed from two fatty alcoholate molecules per molecule of ether, except for the soaps formed from fatty acids and from unreduced CNO equivalent to the moisture present.

Heats of Reaction

Reduction of ester to alcoholate	2,070	B.t.u./lb. sodium to form alcoholate
Ether formation from alcoholate	334	B.t.u./lb. Na_2O formed from alcoholate
Saponification of CNO	49	B.t.u./lb. CNO saponified
Neutralization of CNO fatty acid	90	B.t.u./lb. fatty acid

Note: All reactions are exothermic, and variations of heats of reaction with temperature are negligible in the range under consideration.

Hydrolysis Data

Maximum soap concentrations to prevent stable emulsion formation in hydrolysis tank

% Na_2O in aqueous layer	Maximum % soap permissible in solvent layer
17	1.74
20	1.26
25	0.60
28	0.36

Consider these data to define the breakpoint between no emulsion or completely stable emulsion.

Heats of Hydrolysis

Free sodium	2,600 B.t.u./lb. Na (includes heat of dilution of NaOH)
Alcoholates	400 B.t.u./lb. Na (includes heat of dilution of NaOH)

Note: Both reactions are exothermic and variations in heats of reaction with temperature are negligible.

Unreacted CNO is saponified during hydrolysis.

All soap formed may be considered to go to the solvent layer in the hydrolysis tank upon settling if a stable emulsion is not formed.

Distillation

A 30-plate solvent column is needed for the fractionation at the reflux conditions given under plant data.
Plates are needed in alcohol distillation for entrainment separation only.

SAFETY REQUIREMENTS

Reduction Reactor Operating Pressure
800 mm. Hg abs.

Reduction Reactor Agitation
Minimum agitation time after all feed is added = 2 min.

Heats of Reduction and Hydrolysis
The latent heats generated must be completely absorbed by the reflux systems.

Hydrolysis Boil Time
Minimum boil time after drop from reduction reactor is complete = 1 min.

Distillation of Alcohol
Maximum alcohol permitted in final bottoms = 1.0% by weight

SOLUTION

Raymond M. Cartier
Polytechnic Institute of Brooklyn

Proctor and Gamble Company
Cincinnati, Ohio

Dear Sirs:

In reply to your request that I investigate fully the productive capacities of all the pieces of equipment used in the manufacture of alcohol from coconut oil by the sodium reduction process; I have prepared the enclosed report.

The investigation was authorized by representatives of the American Institute of Chemical Engineers.

The form of the report will adhere strictly to the form suggested. In addition, to facilitate the readability of the material, visual interpretations of all results will be made through the use of graphs and flow sheets.

I trust that you will find this report to your complete satisfaction.

Respectfully yours,

BRIEF SUMMARY OF CALCULATIONS

In order to obtain maximum production with the present equipment it is suggested that management select a solvent ratio of 1.70 lb. of toluene/lb. MIC in order to conduct operations.

It was found that this solvent ratio gave us a maximum production rate for the limiting piece of equipment. The following tabulation represents a summary of capacities for all the pieces of equipment currently in operation.

Equipment	Capacity in lb. CNO fatty alcohol/hr.
Reduction reactor	2,390
Quench tank	2,420
Solvent still	
1. Column alone	5,250
2. Recirculation pump	19,100
3. Dowtherm reboiler	2,520
4. Overhead condenser	2,440
Alcohol still	
1. Column alone	6,250
2. Overhead condenser	3,360
3. Recirculation pump	31,200
4. Preheater	None required

The limiting factor to capacity in present equipment is the reduction reactor. The calculated maximum capacity in pounds of distilled alcohol per hour is 2,390. An important outcome of the calculations, in addition to the items specified above, is the so-called capacity-improvement curve, which enables management to make an accurate estimate of the situation at a single glance. A thorough analysis of this curve is presented in the section Summary and Conclusions.

DRIER

This section will be devoted to determining the relation between a dry acid-free CNO throughput and exit equilibrium moisture. This relation is important because not only will it determine drier capacities but in addition will serve as a guide to our calculations in the study of succeeding pieces of equipment.

As a prerequisite a graph must be constructed which will relate the heating capacity of the heat exchanger to the temperature difference between the 150 lb./sq. in. gauge saturated steam and the bulk fluid in the drier. This single-stage drier may be considered as an evaporator; i.e., the entering fluid immediately attains the temperature of the bulk fluid. The amount of heat transferred to the bulk fluid at any time may be determined from the relation