

CPD GG00-2

Conceptual Process Design

Laboratory for Process Equipment
Mechanical Engineering
Delft ChemTech
Delft University of Technology

Basis of Design

3252

Subject:

BIO-Diesel from Rapeseed and BIO-Ethanol

Date:

30 October, 2000

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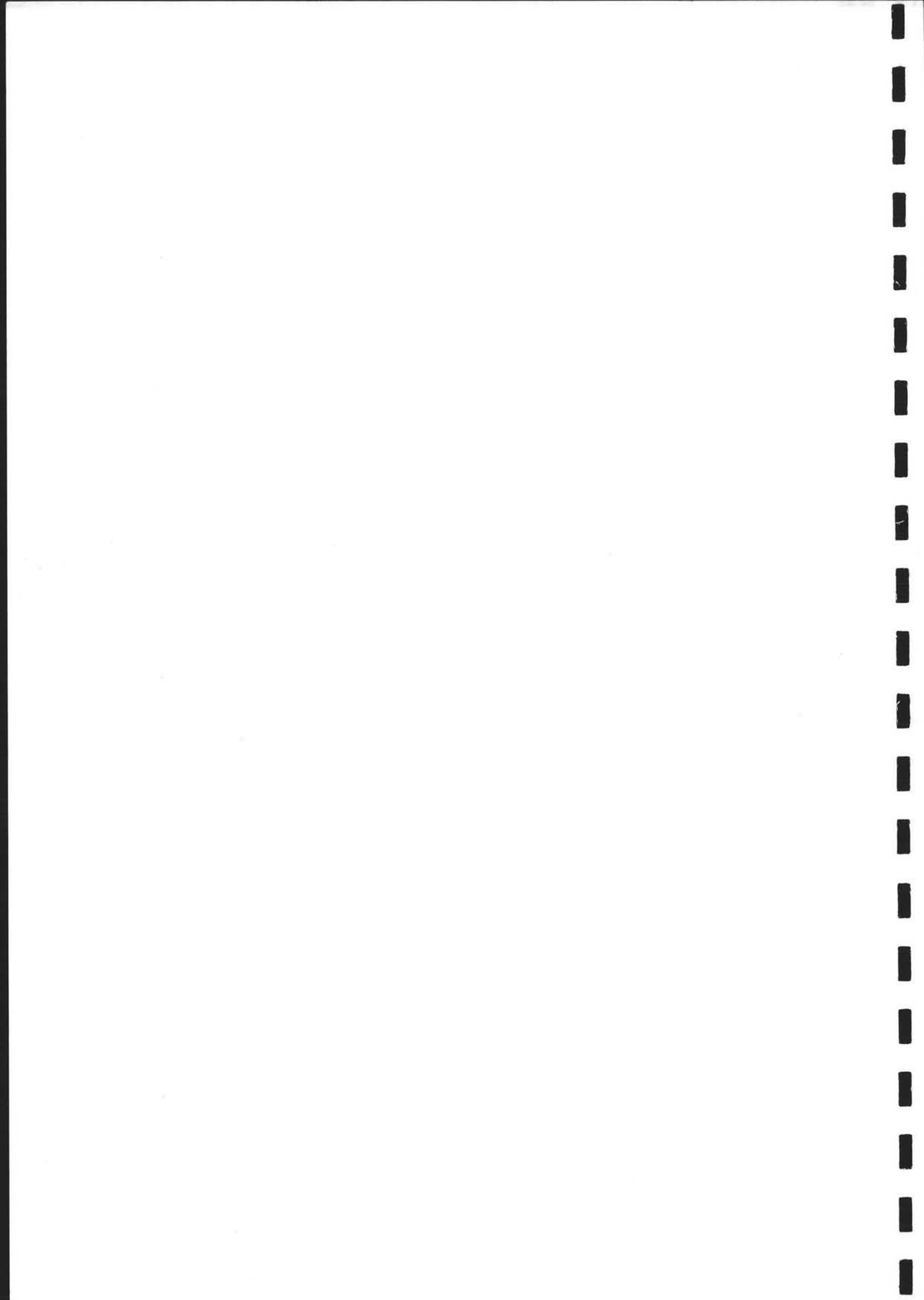
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1. Description of the design

Outline of design

The process of producing biodiesel from rape-seed and azeotropic ethanol consists of basically five steps. The first step is the production of rape seed oil (RSO) from the rape seed. This is done in unit 100. The second step, in unit 200, is to purify this RSO so that it can be added to the reactor, and the necessary impurities are removed. In unit 300 the reaction takes place. Here the purified RSO is mixed with anhydrous ethanol to produce the rapeseed ethanol ester (REE) according to the following stoichiometry;



The ethanol is produced in unit 400, where its concentration is raised from the azeotropic concentration (88.3 %mol) to the anhydrous form (99.99%mol). Finally in unit 500, the products of the reaction in unit 300 are purified into pure REE, pure glycol, and a relatively pure recycle ethanol stream.


Activities

Firstly we will do a basic engineering of the plant, determining the behavior of the chemicals involved, and the lay-out of the production and separation train.

Also, we will do a basic cost evaluation. We will take into account prices and quantities of feeds and products, utilities, and transportation costs, and decide where to locate the REE production facility.

Finally, we will do a detailed engineering of the plant, determining the specifications of the equipment and substances involved.

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Bio-Diesel from Rapeseed and Bio-Ethanol



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Introduction

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Introduction

- GG00-2
 - 10 members
 - Multi-disciplined
- TUDelft
- OBL

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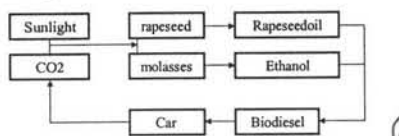
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Objective

Conceptual Process Design of a Bio-Diesel plant based on Rapeseed and Bio-Ethanol with a capacity of 100,000 t/a.

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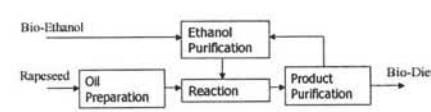
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Motivation

- Feedstocks
 - Rapeseed
 - Bio-ethanol
- Sustainability
 - CO₂



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Scheme



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Reactor	• Reactor:	Nancy Sawirjo
Product purification	• Product Purification:	Amir Abdallah
Methanol/Ethanol	• Methanol/Ethanol:	Frank Busing
Economy	• Economy:	Flip van Dijk
Conclusions	• Conclusions & Recommendations:	Bas Arntz

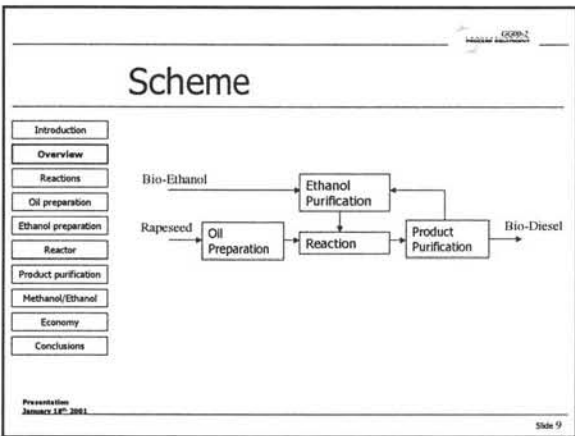
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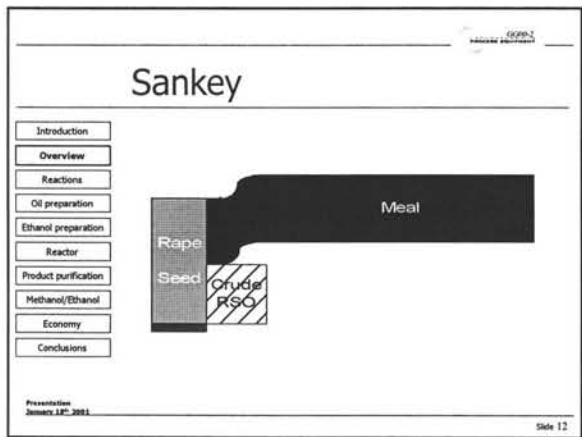
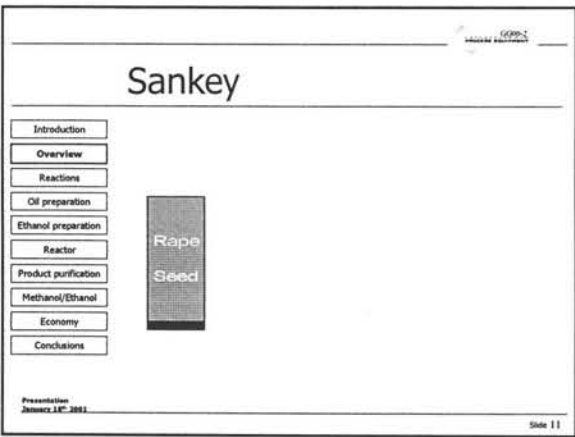
Overview

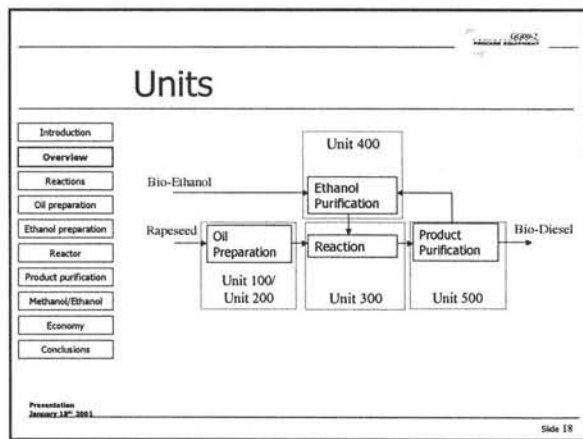
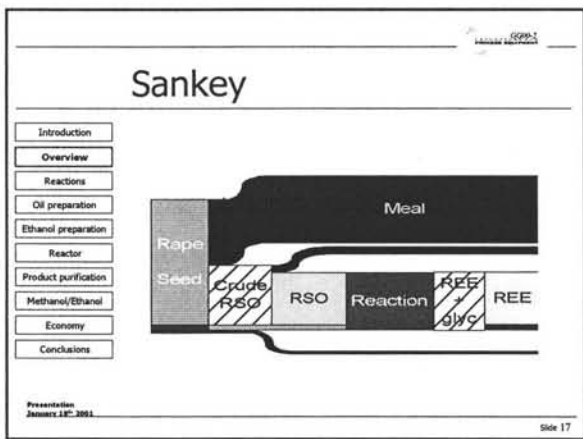
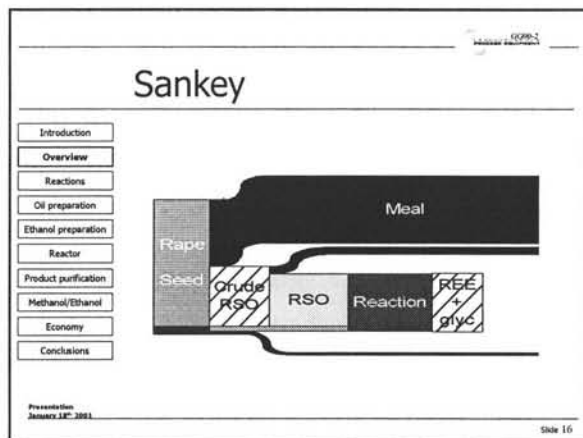
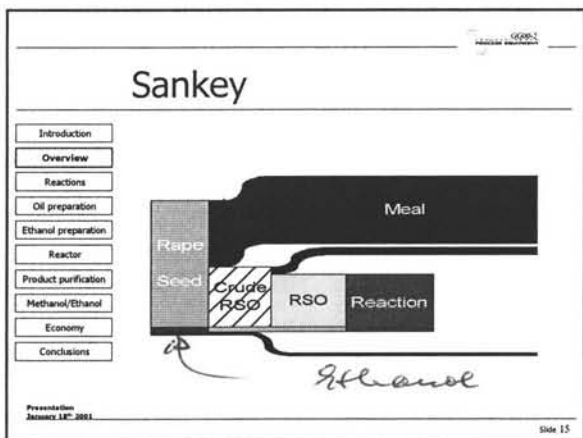
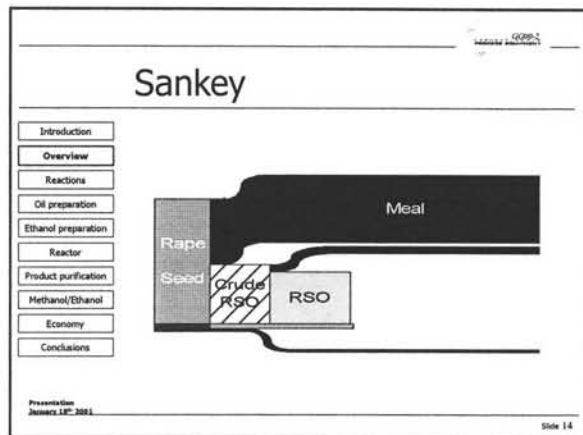
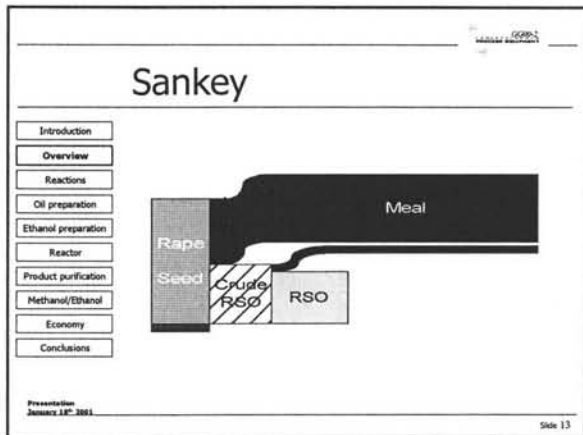
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- # Capacity
- Bio-Diesel
 - 100,000 t/a
 - 10% Dutch Diesel market
 - Equal order of magnitude to existing plants
 - Rapeseed
 - 240,000 t/a.
 - 106500 ha. of rapeseed fields
 - Utrecht: 140000 ha.
 - Ethanol
 - 13,750 t/a.
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Reactions

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Molecular Biology

Reaction Mechanism

Introduction

Overview

Reactions

Oil preparation

Ethanol preparation

Reactor

Product purification

Methanol/Ethanol

Economy

Conclusions

Transesterification:

(1)

(2)

(3)

100% Excess Ethanol

Polar Phase

A-polar Phase

Triglyceride + 3 ethanol → glycerol + 3 Rapeseed Ethyl Ester

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Unit 100 reactor Side reactions

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Molecular Biology

Catalyst

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- Active ethanol: ethoxide
- Activator: KOH
 - strong basic
 - high activity
 - homogeneous: separation problem
- Alternatives

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Molecular Biology

Side reactions

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reactions

- Water
 - de-esterification
 - forming of free fatty acids
 - soap
- Free fatty acids
 - soap

NO WATER!
NO FREE FATTY ACIDS!

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Oil Preparation

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Molecular Biology

Unit 100

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Unit 200

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- Reactor
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- Economy
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Ethanol purification

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5.

Ethanol

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

- Objective: obtain very dry ethanol (99.9%)
- Difficulty: azeotrope (at 88 mole% ethanol)
- Solutions:
 - pressure swing distillation
 - azeotropic distillation
 - hybrid distillation with membrane technology

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Azeotropic distillation

- Introduction
- Overview
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- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

- proven technology
- robust equipment:
 - two columns
 - decanter
- cyclohexane as entrainer:
 - not carcinogenic
 - used in unit 100/200

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Azeotropic distillation

U400: azeotropic distillation

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Hybrid distillation

Membranes: promising alternative

- Introduction
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- Oil preparation
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- Methanol/Ethanol
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- Conclusions

- principle of operation:
 - water passes membrane layer better than ethanol
 - result: water content reduced beyond azeotrope
- not operational yet

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Hybrid distillation

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
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U40: hybrid distillation

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Unit comparison

- Introduction
- Overview
- Reactions
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- Ethanol preparation
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- Product purification
- Methanol/Ethanol
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- Conclusions

unit cost comparison

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Unit comparison

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

Conclusions

In order to be competitive:

- Energy cost in U40 must be reduced
- Membrane investment cost must be reduced

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Unit comparison

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

reboiler duty dependence on ethanol column feed purity

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Reactor

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

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Design Criteria

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

- Heat requirement
 - Endothermic reaction
 - High reaction rate in first few seconds of reaction
 - Main design criterium
- Plug-Flow
- Continuous
- Mixing
 - Two phases
 - Reynolds

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Reaction conditions

- Pressure
 - low pressure
- Temperature
 - 75°C
- Reaction Time
- Conversion

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Reactor Design

- Three sections:
 - Different heat requirement
 - 1. Shell & Tube heat exchanger
 - 2. Shell & Tube heat exchanger
 - 3. Double pipes (20)

Introduction

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Product Purification

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Objective

- Product Purification
 - To separate the reaction mixture (incoming streams) into the main product (BIO-Diesel) and by products

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Incoming streams

- Bio-Diesel
- Ethanol
- Water
- Cyclohexane
- KOH
- Monoglycerides
- Glycerol
- Free Fatty Acids

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Product purification

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Unit 500

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Conclusions

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Conclusions

- BIO-Diesel is purified to meet the market specifications
- The ethanol is recovered
- The KOH and the monoglycerides are recycled
- Glycerol is also purified to meet the market specifications

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Ethanol/Methanol

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Properties

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Economy

Conclusions

- Diesel
 - Little differences
- Sustainability
- Toxicity

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Process consequences

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Methanol/Ethanol

Economy

Conclusions

- Conversion
 - methanol conversion meets Diesel Specs
 - Methanol no recycle needed
- Kinetics
- Catalyst

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Conclusions

	Euro
Total investment	19,000,000
Annual production costs	100,487,000
Annual income	103,386,000
Annual profit	2,899,000
1 Euro = fl 2.20371	
Rate of return	15.3%
DCFROR	59%

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Investment

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

- Calculations Purchase Cost Equipment
- Most expensive pieces of equipment
- Lang-factor
- Total investment: 19,000,000 Euro

	Euro	%
X101 Flaking machine	248,400	6.9
X102 Screw Press	1,458,000	40.6
X103 Extruder	842,200	23.3
E101 Laminated	760,900	21.4
E103 PPC	316,900	9.1
E204 Fixed bed = PPC X 1.4	443,660	12.4
E503 Total condenser = Fixed Exp	3,617,000	100.0

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Variable costs p/a

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

	Euro	%
• Rapeseed	54,946,000	74.5
• Bio-ethanol	10,908,000	14.8
• Steam	6,048,000	8.2
• Rest	1,806,000	2.5
Total variable costs	73,707,000	100

Price (€/t): 2683.6 (=fl 1.18/liter)
Amount (t/y): 26,000
Total product: 2041.54

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Production costs p/a

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

	Euro
Total variable costs	73,707,000
Total fixed costs	6,683,000
Total direct costs	80,390,000
Total indirect costs	20,097,000
Total production costs	100,487,000

Production costs per ton product (rapeseed):
937 Euro (=fl 1.75/liter)

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Annual profit

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

Income:	Euro
Biodiesel	68,965,000
Glycerol	20,384,000
Meal	14,037,000
Total	103,386,000
Total costs	100,487,000
Annual profit	2,899,000

Price (€/t): 68204 (=fl 1.20/liter)
Amount (t/y): 102,000
Total product: 11.127

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Parameter

- Introduction
- Overview
- Reactions
- Oil preparation
- Ethanol preparation
- Reactor
- Product purification
- Methanol/Ethanol
- Economy
- Conclusions

Total investment	19.0	MEuro
Annual profit	2.9	MEuro
Rate of return	15.3	%
Pay out time	6.6	Yrs
Discounted cash flow	59.0	%
Rate of return		

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Sensitivities

Changes of +10% and -10%:

- Rapeseed price
- Biodiesel price
- Price of utilities

	FCR (%)	FCR (%)	FCR (%)	FCR (MEuro)
Reactor	65.6	15.3	59.0	2.9
Product purification	1.97	54.14	148.00	9.80
Methanol/Ethanol	5.1	-19.5	71.0	-3.91
Economy				
Conclusions				
	Relative difference (%)			
+10%	-7.48	2.38	15.7	2.38
-10%	5.0	-2.35	1.20	-2.35
+10%	-2.2			

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Economical review

Introduction

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Economy

Conclusions

- Bio-ethanol price is perhaps too high
- Biodiesel price is perhaps too low
- By heat-integration the amount of steam needed can be reduced by a factor two

Biogas price per liter from £1.18 to £1.00
 Result: annual profit raises with 11.5 MEuro

Result: annual profit raises with 3.8 MEuro

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Conclusions and Recommadations

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Conclusions

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Methanol/Ethanol

Economy

Conclusions

- Technically and economically feasible
- Methanol->Ethanol, substantially different process
- Fluctuating prices / subsidies influence feasibility
- High energy consumption compared to plants already operational
- Design and construction Unit 100 and 200 can be outsourced
- Safety measurements for Unit 100 and 200
- Relatively low waste production

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Recommendations

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Product purification

Methanol/Ethanol

Economy

Conclusions

- Heat integration
- Watch developments and prices Membrane modules
- Investigate feed supply reactor in a number of stages
- Reexamine design crystallizer

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Questions

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Addendum to final report 'BIO-diesel from rapeseed and BIO-ethanol'

1. Preface

The assignment is issued by OBL, in which COSUN, CSM SUIKER and NEDALCO participate, represented by Mr. S.S. de Vries. For Delft University of Technology, Dr. Zarko Olujic represented the Laboratory for Process Equipment, Mr. C.P. Luteijn Delft Chem Tech.

2. Earning power

The earning power is calculated according to Coulson and Richardson's Chemical Engineering Volume 6. The calculation is shown in table A1. The earning power is 7.03%.

Table A1

End of year	Net cash flow (MEuro)	Cumulative cash flow (MEuro)	Discounted Cash Flow (at 15%)	Cumulative Discounted Cash Flow (at 15%)	Discounted Cash Flow (at x%)	Cumulative Discounted Cash Flow (at x%)
1	-18.99	-18.99	-16.52	-16.52	-17.75	-17.75
2	0.00	-18.99	0.00	-16.52	0.00	-17.75
3	2.90	-16.10	1.91	-14.61	2.36	-15.38
4	2.90	-13.20	1.66	-12.95	2.21	-13.17
5	2.90	-10.30	1.44	-11.51	2.06	-11.11
6	2.90	-7.40	1.25	-10.26	1.93	-9.18
7	2.90	-4.50	1.09	-9.17	1.80	-7.38
8	2.90	-1.60	0.95	-8.22	1.68	-5.70
9	2.90	1.29	0.82	-7.40	1.57	-4.13
10	2.90	4.19	0.72	-6.68	1.47	-2.66
11	2.90	7.09	0.62	-6.06	1.37	-1.28
12	2.90	9.99	0.54	-5.52	1.28	-0.00
13	4.80	14.79	0.78	-4.74	1.98	1.98
14	4.80	19.58	0.68	-4.06	1.85	3.84
15	4.80	24.38	0.59	-3.47	1.73	5.57

3. Used prices of feedstock and products

Feedstock:

Rapeseed fl 0.463 / kg
 Bio-ethanol fl 1.207 / liter

Products:

Biodiesel fl 1.204 / liter
 Glycerol fl 0.330 / kg
 Meal fl 2.850 / kg

4. Conversion, kinetics and catalyst

The article by Freedman and Pryde [23] is the basis for conversion, kinetics and catalyst in the design. The article addresses the transesterification of soybean oils, not rapeseed oils. Therefore, small but important differences in equilibrium conversion may be present. An equilibrium conversion of about 97% is assumed from the data in the article, achieved after 4 hours. In the design, the conversion achieved is 94% after 3½ minutes (217 seconds) reaction time. If it is found, i.e. from dedicated experiments, that equilibrium conversion is high enough to simplify the product purification train (U500), a better (faster) catalyst is desired.

2. Process definition

2.1. Process concept chosen

Unit 100-200

Before extracting the oil from the rapeseeds, the seeds must be cleaned, crushed/flaked and heated (pretreatment of the seeds). Three different processes for extracting oil from seeds are used in industry.

1. Direct solvent extraction
2. Direct screw pressing extraction
3. Pre-press solvent extraction

The third possibility is chosen. High capacities are possible, 1000 ton/day. The process time is relatively low and the process is efficient. Pre-press solvent extraction is the most economical process for oil extraction.

The pre-press solvent extraction process is a classical system of processing rapeseed in which the seed is initially expelled under pressure to release a percentage of the available oil. Usually hexane is used as solvent for the extraction. This method is used by many oil producers.

After the pressing step, the residue from the screw press is mixed with hexane and the oil in the residue is further extracted. The press expels 16-20% of the oil. The hexane extract 18-20% of the oil (a total amount of 40 % of oil expelling from the rapeseed is possible).

The last step in processing rapeseed oil is degumming, removal of phosphatides or gums and free moisture. The crude rapeseed oil is mixed with water or acid to hydrate phosphatides, which are then removed by centrifuging.

Unit 300

In unit 300 the reaction takes place.

Reactions:

The crude RSO contains different kinds of fatty acids that form ester bonds with glycerol. In modeling the RSO we assumed that all fatty acids are oleic acid. This assumption is made for modeling in Aspen.

Thermodynamics:

In the modeling of the mixture the ionic components are neglected. For the use of the non ionic thermodynamic models see next paragraph. With the non-ionic thermodynamic model the reactor equilibrium is calculated. The equilibrium calculation estimated a conversion of about 100 % of glycerolesters to ethylesters.

Unit 400

We will investigate two different processes to produce anhydrous ethanol from an azeotropic feed. The benchmark process will be azeotropic distillation. Ethanol-water separation using benzene is the textbook example of azeotropic distillation, so this will be used for comparison. A suitable entrainer will need to be found to replace benzene.

On the other hand, membrane separation is a very promising modern process. When designing the process carefully, a membrane separation can be included which promises lower total costs

U400 Azeotropic Distillation alternative

In azeotropic distillation, a third component is added to 'lift' the concentration over the azeotropic concentration. The standard entrainer is benzene, but a more environmentally friendly agent would be hexane. We will examine both. What happens is that the benzene-water azeotrope is more volatile than ethanol, so that ethanol can be recovered as a very pure bottoms product. The benzene-water azeotrope is then, in a decanter, separated into an organic and an aqueous phase. The aqueous phase is distilled, benzene is recycled, and water is the bottoms product. The organic phase is recycled to the first column.

Fouling is not a great problem, it can be countered by using tray columns designed to deal with fouling liquids.

U400 Membrane alternative

Considering the membrane alternative, two main concepts are applicable:

1. Pervaporation. The azeotropic mixture is supplied to the membrane unit as a liquid. The water (and some ethanol) is evaporated through the membrane layer. The driving force is the partial water vapour pressure requiring vacuum at the permeate side (~ 0.1 bar). The ethanol phase (retentate) is to be heated in stages because of heat loss in the liquid due to water evaporation.
2. Vapour permeation. The azeotropic mixture is supplied to the membrane as a (superheated) vapour, therefore no interstage reheating is required as in the pervaporation case.

Maintaining the driving force is a point of concern with the membrane. An option is the so-called product sweep: ethanol rich vapour from the retentate is recycled to the permeate side in order to lower the partial water vapour pressure. In both options the water rich permeate is supplied to an under azeotropic column where water (~ 100 ppm ethanol) is removed from the bottom and azeotrope is recycled back to the azeotropic membrane feed. In the pervaporation case the azeotropic vapour may be condensed in stages in order to reheat the liquid retentate stream.

With respect to both membrane options the ultimate retentate may directly be 99.9% pure or be 99% pure and supplied to an above azeotropic column with azeotrope as distillate and 99.9% ethanol as bottom product.

Fouling. The fresh azeotropic feed is expected to be very low in impurities. The recycle ethanol streams from unit 500 are removed as distillates and contain relatively small amounts of other components, predominantly glycol and REE. Dissolved potassium, fats and fatty acids will not be present, as they will stay in liquid bottom streams. If fouling due to glycol and REE is an issue, the recycles should be supplied after the membranes and before the above azeotropic column. These heavy components will be removed with ethanol as bottom products. The azeotropes are

in both above and under azeotropic columns removed as distillates and can therefore be recycled to the membranes virtually free of heavy components.

The water content of the reactor feed is to be kept to a minimum and therefore the hydrate water in solid fresh KOH is to be removed. The KOH can be supplied just before the above azeotropic column where KOH, being dissociated completely in ions, will go into the product stream together with the ethanol as a liquid, and will not be recycled to the membranes. If the KOH is not supplied in this way, it must be dried by heating before supplied to the dry ethanol stream.

Conclusion

Much experience has been accumulated in using azeotropic distillation for the ethanol-water mixture. This makes it a useful benchmark process. Investigation is needed to determine which entrainer to use instead of the standard entrainer, benzene.

Membranes offer an alternative to azeotropic distillation, even in the case of sensitivity to glycerol, REE, and KOH, when using an above azeotropic column. In case a virtually water free reaction feed is required this column can be used keeping the membranes free of degradation due to potassium.

In the detailed engineering study we will do a detailed engineering of both processes, and decide which is the most useful.

Unit 500

The goal of the separation process is to separate the mixture coming from the reactor in order to get a pure product (REE) and to get pure byproducts.

The stream coming from the reactor is a two-phase flow, which is separated in a decanter. Solids that are created in the reaction accumulate in the boundary layer in the decanter. This boundary layer is drained, the solids are removed by filtration and the solid free liquid is recycled into the decanter. Two options for product purification are:

1. Distillation.
2. Washing with water (the ATT process [1]).

We choose for distillation because the monoglycerides must be removed from the product phase in order to meet product specification. In the ATT process for the methyl alternative it is likely to believe that the monoglycerides content in biodiesel is within specification because of a higher equilibrium conversion (from literature [6]) than the ethyl alternative. Washing with water will not adequately remove the monoglycerides but is done in the ATT process to remove other components. In order to get operation temperature low enough to preserve product breakdown columns must be operated in deep vacuum (± 30 mbar).

Several rules of thumb [58] are taken into account to determine our distillation process:

1. Remove corrosive or hazardous components as soon as possible.
2. Remove reactive components or monomers as soon as possible.
3. Remove products as distillates.
4. Remove recycles as distillates particularly if they are recycled to a fixed bed reactor.
5. Most plentiful first.
6. Lightest first.
7. High-recovery separation last.
8. Difficult separation last.

The light phase, containing mostly apolar components, is fed to a flash drum to separate the ethanol from the feed mixture, in accordance with rule of thumb number 6. In the final column the product REE is removed as distillate and high recovery is required, in accordance with rules of thumb number 3 and 7. Although violating rule of thumb number 4, the monoglyceride recycle stream is removed from the column as a bottom product, but a minimum of impurities are expected to be present.

The heavy phase, containing mostly polar components, is fed to a distillation column where the lightest and the most plentiful component, ethanol, is distilled first, following rules of thumb number 5 and 6. The last separation is that of monoglyceride and glycerol and is the most difficult one (rule of thumb number 8).

2.2. Block schemes

See PFS' s in appendix 1.

2.3. Thermodynamic properties

The estimation of the enthalpy, entropy and Gibbs energy.

The thermodynamics can describe the equilibrium state of a system. The equilibrium calculation can be used for a concept process design. For equilibrium calculations the Gibbs-energy is fundamental. Estimation of the Gibbs-energy for pure components is possible and mixtures at every temperature and pressure if the enthalpy, entropy and the specific heat properties are available. The following formulas are used:

$$\Delta_f H(p, T) = \Delta_f H(p^0, T^0) + \int_{T^0}^T c_p dT$$

$$\Delta_f S(p, T) = \Delta_f S(p^0, T^0) + \int_{T^0}^T \frac{c_p}{T} dT - 2R \ln\left(\frac{p}{p^0}\right)$$

$$\Delta_f G(p, T) = \Delta_f H(p, T) - T\Delta_f S(p, T)$$

In the production of bio diesel, mono-, di- and tri-glycerides and ethylesters of oil acids are concerned. For these large molecules the (estimated) properties are indirectly available with group contribution methods. In this design the Benson group contribution method for ideal gas properties is used. These heavy components are not in the gaseous state. With these ideal gas properties and the knowledge that the Gibbs-energy for the gas and liquid in equilibrium are the same, the properties of liquid phase are estimable. With the following formula the Gibbs energy of gas can be transformed in the Gibbs energy for liquid.

$$\Delta_f G(p, T) = \Delta_f H(p^0, T^0) - T\Delta_f S(p^0, T^0) + \int_{T^0}^T c_p dT - T \int_{T^0}^T \frac{c_p}{T} dT + 2RT \ln\left(\frac{p^{sat}}{p^0}\right)$$

In the formula above the saturated pressure can be estimated with the Frost Kalkwarf Thodos method. The Frost Kalkwarf Thodos method uses the boiling temperature, critical pressure and temperature. These properties can be estimated with the Fedor group contribution method for critical properties.

The thermodynamic properties of linear alkanolic fatty acids are available in the literature. The estimated properties with the methods used above are correlated on the literature data. The correlation is accurate enough for the design of the bio diesel production: see appendix 6.

K-values for a vapour liquid equilibrium

The vapour liquid relation give correlation between the vapour and liquid composition (x , y) in equilibrium state.

$$\phi_i y_i P_{tot} = \gamma_i x_i \phi_i^{sat} P_i^{sat}$$

Explanation of the symbols:

- P_i^{sat} Saturated vapour pressure of component i in Pa;
- P_{tot} Total pressure in the system in Pa;
- x_i Molar fraction of component i in the liquid phase;
- y_i Molar fraction of component i in de vapour phase;
- γ_i The partial activity coefficient of component i ;
- ϕ_i Partial fugacity coefficient of component i ;
- ϕ_i^{sat} Fugacity coefficient of component i .

The saturated vapour pressure of a component can be found as a function of the temperature from the database of Chemcad. The partial activity coefficients are estimated with the UNIFAC method and the Partial fugacity coefficients are estimated with the Peng Robinson equation of state. The UNIFAC method is a group contribution method. The Peng Robinson equation of state is based on the interpolation of acentric-factor (ω), critic pressure (P_c) en temperature (T_c). This interpolation leads to a deviation from the real situation.

Normally the parameters in the vapour liquid relation are lumped in a K -value.

$$y_i = K_i x_i \quad \text{with} \quad K_i = \frac{\gamma_i \phi_i^{sat} P_i^{sat}}{\phi_i P_{tot}}$$

K-values for a liquid liquid equilibrium

For liquid liquid equilibrium the partial activity of the components in both phase are equal.

$$(\gamma_i x_i)^{\text{Phase I}} = (\gamma_i x_i)^{\text{Phase II}}$$

Explanation of the symbols:

x_i Molar fraction of component i in the liquid phase;

γ_i The partial activity coefficient of component i .

Normally the parameters in the vapour liquid relation are lumped in a K -value.

$$x_i^I = K_i x_i^{II} \quad \text{with} \quad K_i = \frac{\gamma_i^{II}}{\gamma_i^I}$$

Composition of the phases.

For the calculation of the composition of the both phases mole and equilibrium balances are drawn up.

$$F z_i = F^I x_i^I + F^{II} x_i^{II}$$

$$x_i^I = K_i x_i^{II}$$

Explanation of the symbols:

F Total amount of both phases in moles;

K Equilibrium constant or K -value of component i

F^I Amount in phase I in moles;

F^{II} Amount in phase II in moles;

x_i^I Mole fraction of component i in de phase I;

x_i^{II} Mole fraction of component i in de phase II;

z_i Mole fraction of component i in the system (both phase);

By combining the mole and equilibrium balances and introducing the factor $\beta = F^I / F$ follows the formula below.

$$x_i^{II} = \frac{z_i}{1 + \beta (K_i - 1)}$$

$$x_i^I = \frac{K_i z_i}{1 + \beta (K_i - 1)}$$

The vapor fraction (β) can be calculated by solving the following equation.

$$f(\beta) = \sum_{i=1}^n x_i^I - \sum_{i=1}^n x_i^{II} = \sum_{i=1}^n \frac{(K_i - 1) z_i}{1 + \beta (K_i - 1)} = 0 \quad (1)$$

This formula can be calculated with the Newton Rapson algorithm.

2.4. List of pure component properties

The list of pure component properties is given in Appendix 3 List of pure component properties. The properties of the glycerides and the ethylester are not available in conventional databanks and are therefore estimated. The critical temperatures (T_c) are estimated with the Fedor group contribution method [1]. The boiling point (T_b) and melting point (T_m) values of the glycerides are then estimated with the following rules of thumb:

$$T_b \approx 0.68 \cdot T_c \text{ and } T_m \approx 0.35 \cdot T_c.$$

The liquid density values of the glycerides at 20 °C are estimated with the modified Rackett technique [1]. The estimated density values are lower than expected and this should be taken account of in the design. The remaining melting point values as well as the Maximum Allowable Concentration (MAC) values are obtained from [3]. The lethal dose (LD) values are obtained from [2].

2.5. Process stream summary and mass balances

Basis of calculation

We decided to design a factory with a capacity of 100,000 tons Biodiesel a year. This is the number we based most of the calculations on. With this number in mind we calculated the conversion rate of the reaction. This allows us to determinate quite detailed what and how much products are produced in unit 300. Most of the calculations of Unit 500 are based on the qualifications set by the DIN norm for Biodiesel combined with the numbers of substances we obtained in Unit 300.

Stream list/mass balances

The mass balances are presented in a mass or molar check per piece of equipment. We didn't perform any iterative calculations yet for the recycle streams so the mass balance doesn't add to zero at every piece of equipment. Once we've modelled our design in Aspen, all mass balances should add to zero. This rough estimate is accurate enough to make basis designs though.

The stream lists are attached as appendices.

Stream 215 is based on the DIN norm for Biodiesel. The calculations for the other streams are based on stream 215 and stream 101 (the properties of rapeseed).

3. Basic assumptions

3.1. Plant capacity

If we want to design a competitive plant comparing to plants, which are built currently, we will have to design one with a capacity of 100,000 t/a. Looking at the dieselmkt in the Netherlands this amount is 20% of the diesel in 50% of the cars using diesel. This is (of course) 10% of the total use of diesel in the automobile industry.

The amounts of bio-ethanol and rapeseed, which we will need to produce 100,000 t/a, are large, but they are within the production-capacity of modern factories. The area we need to cultivate the rapeseed is enormous, but we think there will be enough land for example in Eastern Europe, Russia, USA or Brazil.

The factory will be producing REE 350 days per year, 24 hours per day. Per year it will be active for 8400 hours per year (96% of a full, continuous year). In the other 15 days there is time for cleaning, repairing and to solve unexpected problems. The economical plant lifetime is 15 years.

3.2. Location

The principal factors to consider are:

1. Location, with respect to the marketing area. You want to produce the bio-diesel as close as possible to their primary market (refineries), because otherwise the transport costs become too high. The Dutch market is the main customer.
2. Raw material supply. A bio-ethanol producer has to be nearby. You want the rapeseed producer not too far away.
3. Transport facilities. The rapeseed is transferred by boat, so we need a (little) dock. Building the factory close to an industrial area has a couple of advantages. For example the infrastructure and nearby deliverers of products we need in the process like steam and bio-ethanol
4. Availability of labour. To keep the costs of our plant low, we want to locate the plant in a country where the labour costs are as low as possible. The labour cost is price of labour times labour productivity.
5. Availability of utilities. You need steam, but you don't want to produce it yourselves. So also a steam-producer has to be close to the plant.
6. Availability of suitable land. Sufficient suitable land must be available for the proposed plant and future expansion.
7. Environmental impact, and effluent disposal. The disposal of harmful effluents will be covered by local regulations.
8. Local community considerations. The proposed plant must fit in with and be acceptable to the local community. A safe location without significant additional risk to the community.
9. Climate. Adverse climatic conditions at a site will increase costs.
10. Political and strategic considerations. In some European countries (e.g. Italy, France, Germany, Sweden, Austria and Czech Republic) there are some special, lower taxes for bio-diesel.

The location of the plant will be in the 'Rijnmond'-area, Rotterdam, the Netherlands. A bio-ethanol producer is nearby, rapeseed can easily be transported to the plant, the refineries are close to the factory, the road and water infrastructure is excellent, all the utilities are nearby, there is still enough suitable land. Conclusion: A good place to locate the plant.

3.3. Battery limit

Inside the battery you will find the next aspects:

- U100 (the crushing-process of rapeseed)
- U200 (the pre-treatment of rapeseed oil)
- U300 (the reaction/the reactor)
- U400 (the purification of bio-ethanol)
- U500 (the separation-steps to purify the REE and the glycol)
- Storage facilities

Outside the battery we keep the following:

- Bio-ethanol production
- Steam production
- Electricity production
- Control room
- Harbor
- Canteen, change house and offices
- Parking places
- Fire stations
- Laboratories

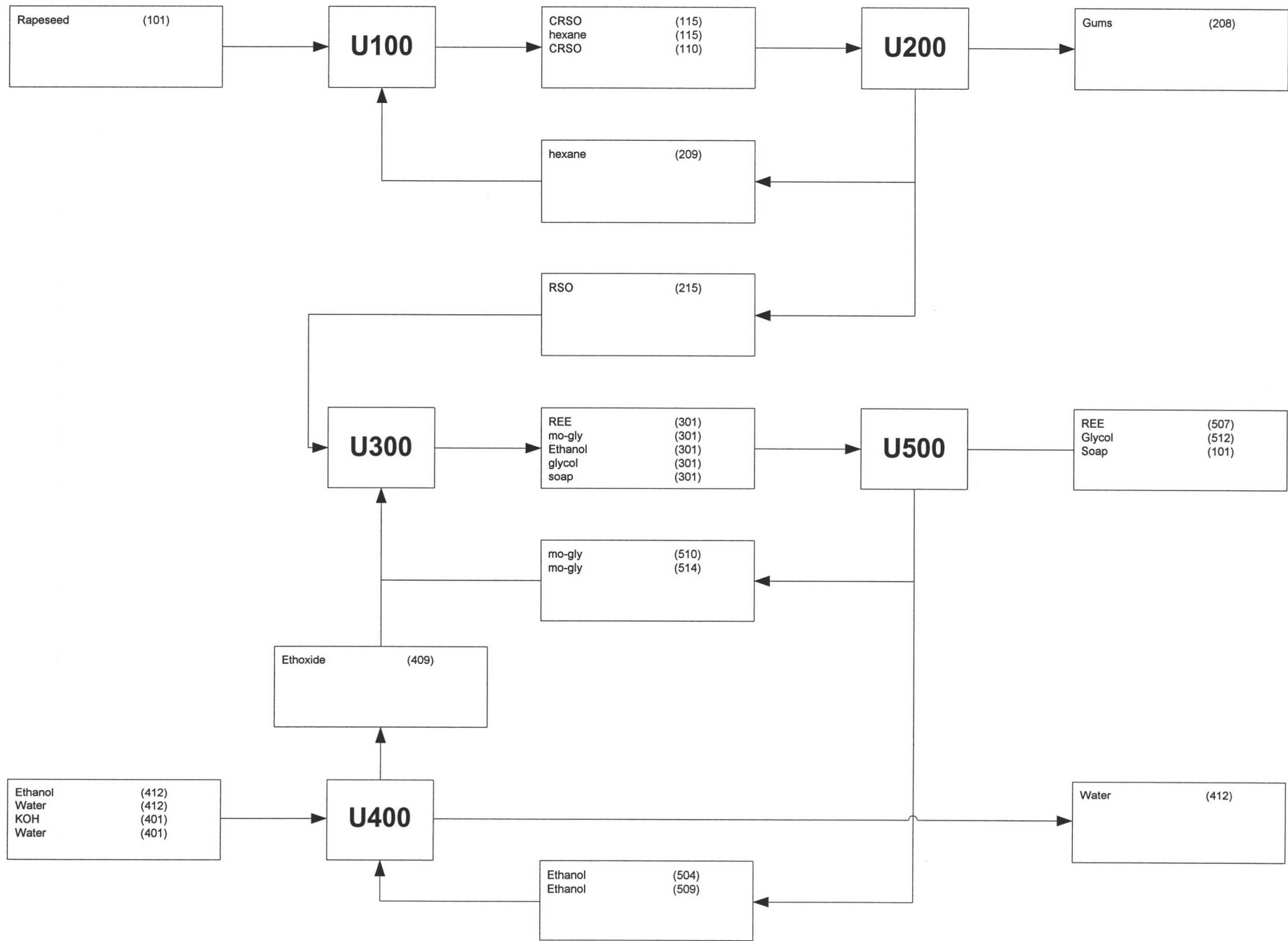
3.4. Battery limit streams

See appendix 5.

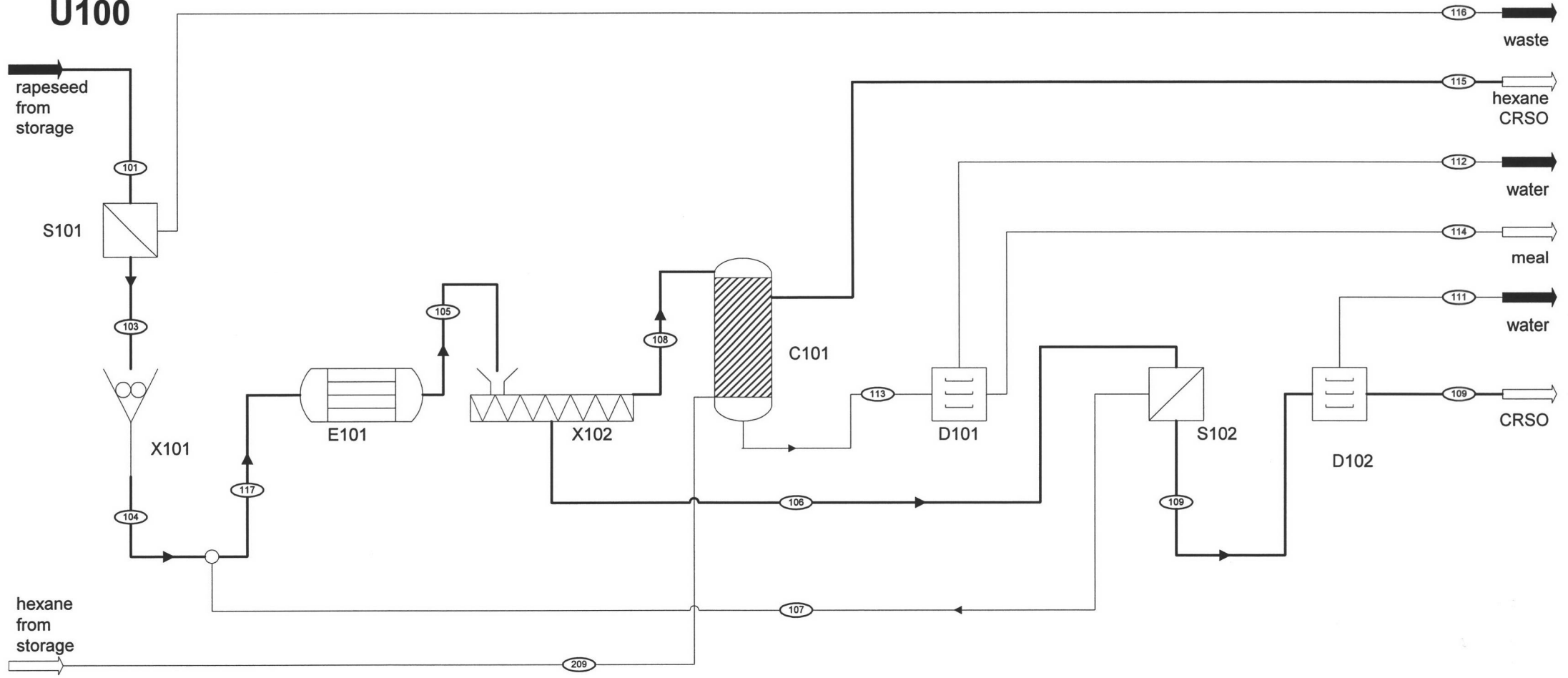
Appendices

1. Process Flow Schemes
2. Streamlists
3. List of Pure component Properties
4. Components Flows
5. Battery limit streams
6. Thermodynamic properties

Appendix 1, helicopter view



U100



Process Equipment Summary

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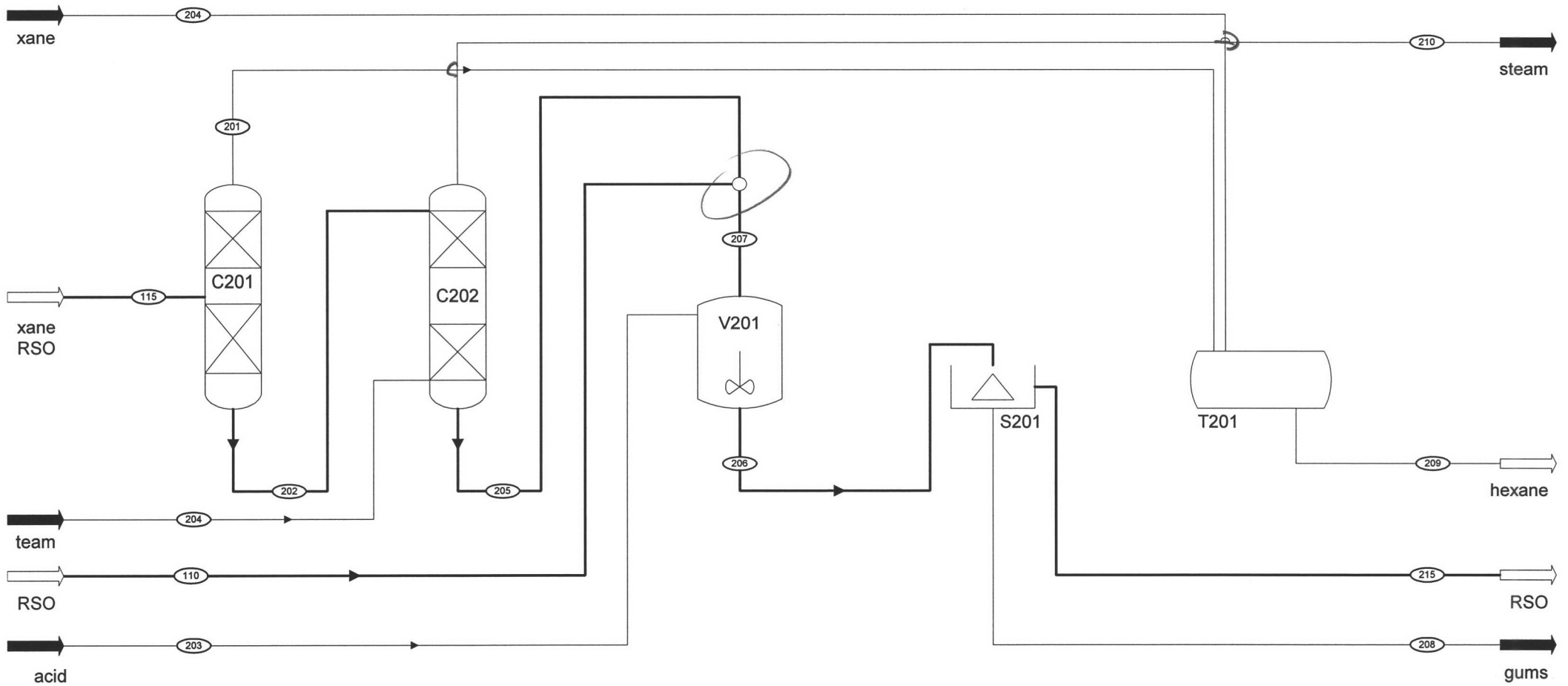
Designers

GG00-2

Process Flow Diagram

Project: Biodiesel from Rapeseed and Bio-ethanol
 Unit: 100
 Proj.ID: CPD

U200



Process Equipment Summary

Designers

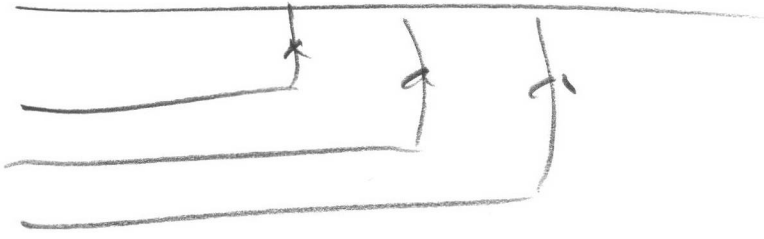
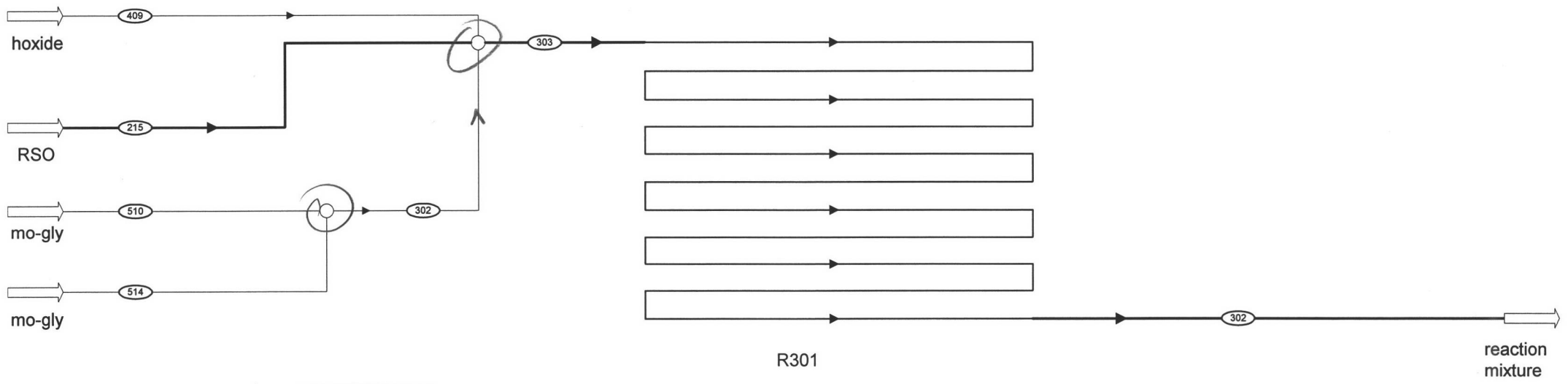
GG00-2

Process Flow Diagram

Project: Biodiesel from Rapeseed and Bio-ethanol

Unit: 200

Proj.ID: CPD

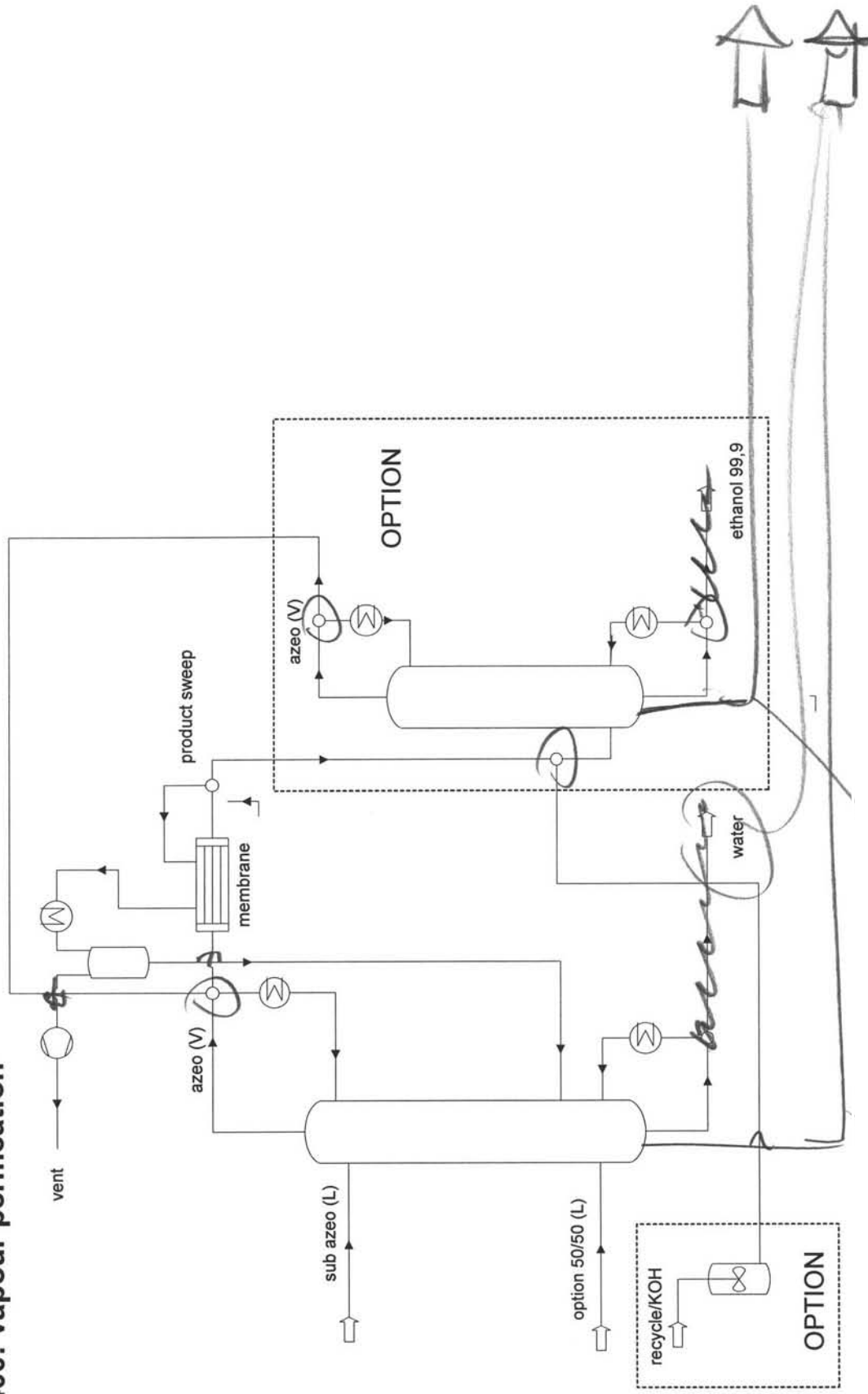


Process Equipment Summary	

Designers
GG00-2

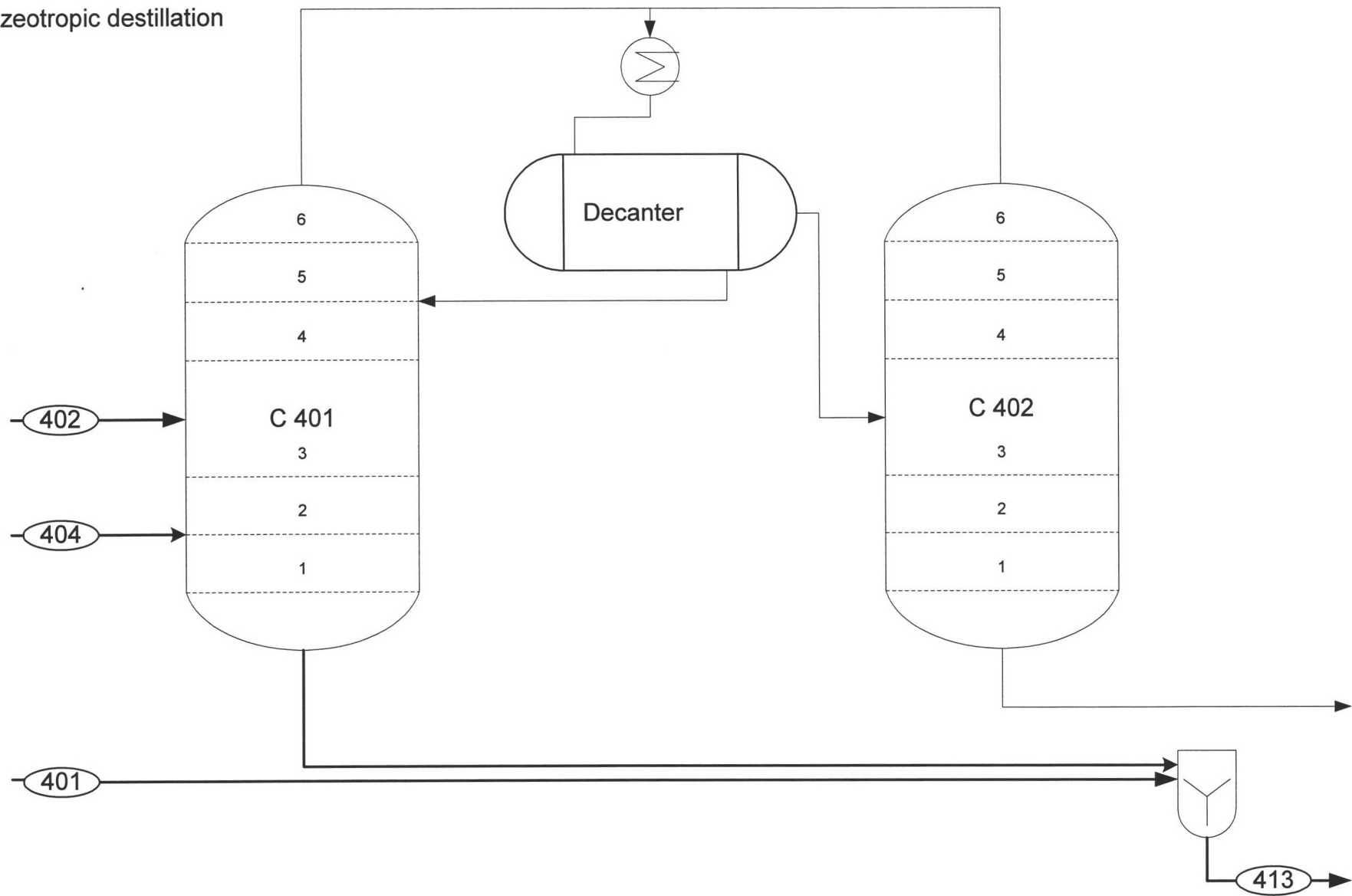
Process Flow Diagram	
Project:	Biodiesel from Rapeseed and Bio-ethanol
Unit:	300
Proj.ID:	CPD

U400: vapour permeation

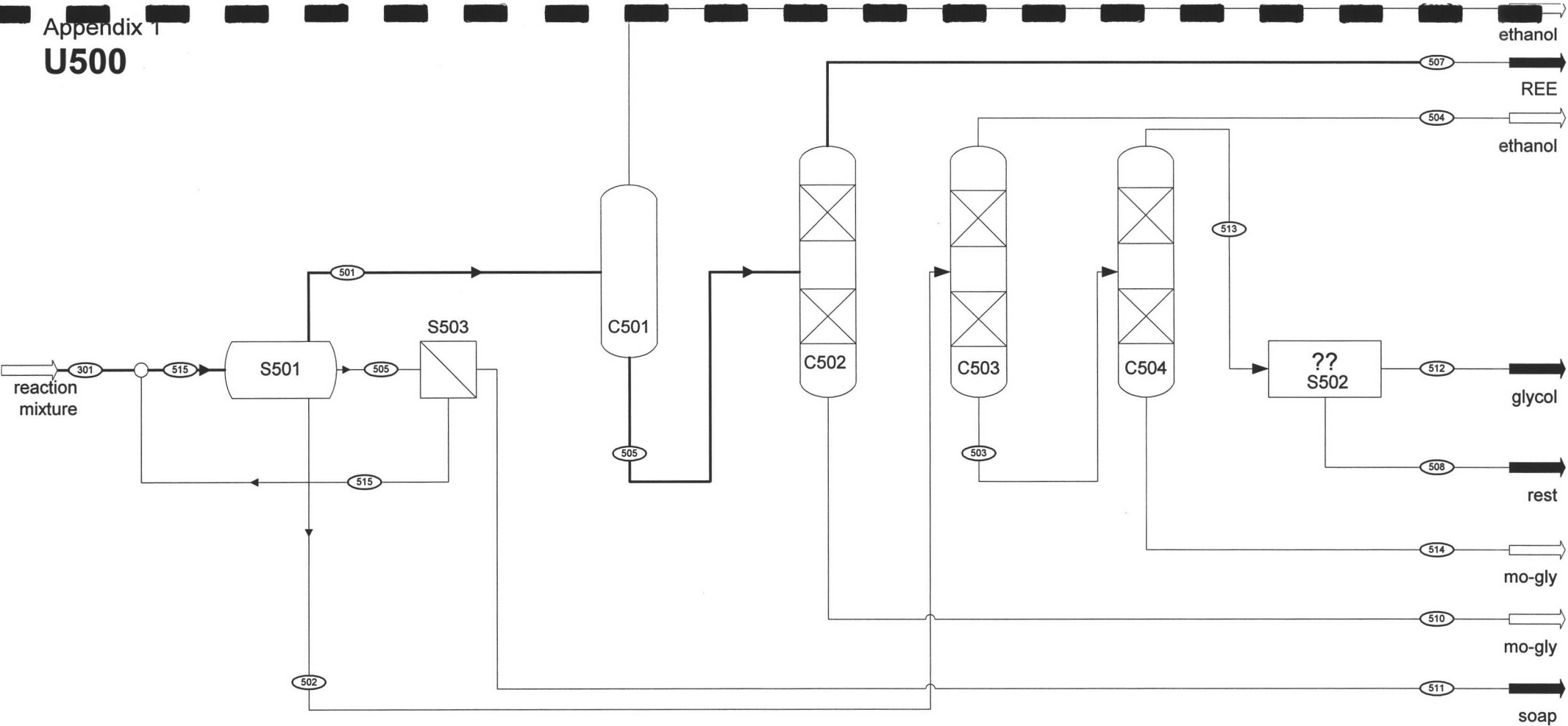


Appendix 1

U400; Azeotropic distillation



ethanol
 REE
 ethanol



Process Equipment Summary

Designers
GG00-2

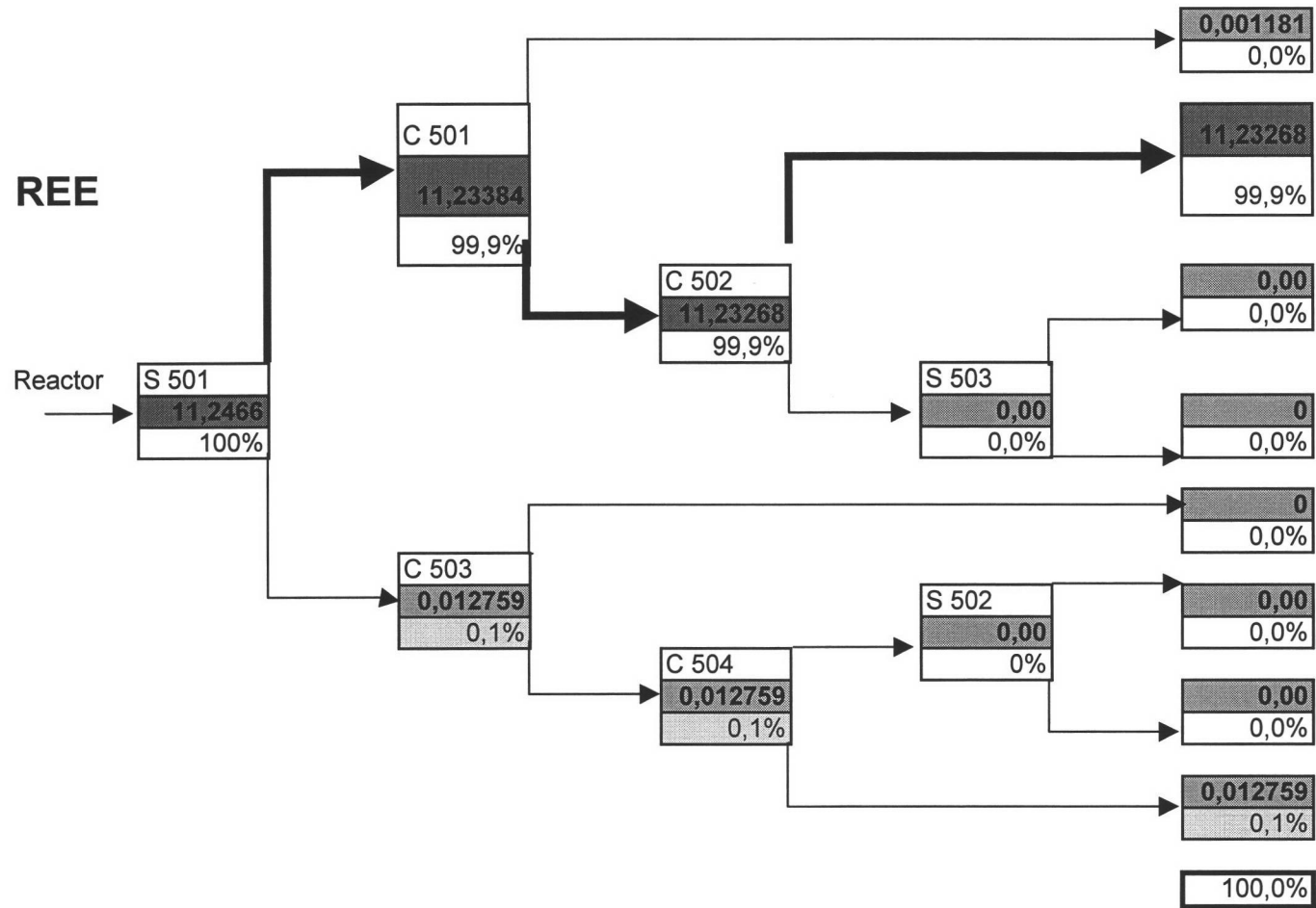
Process Flow Diagram	
Project:	Biodiesel from Rapeseed and Bio-ethanol
Unit:	500
Proj.ID:	CPD

Appendix 3 List of pure component properties

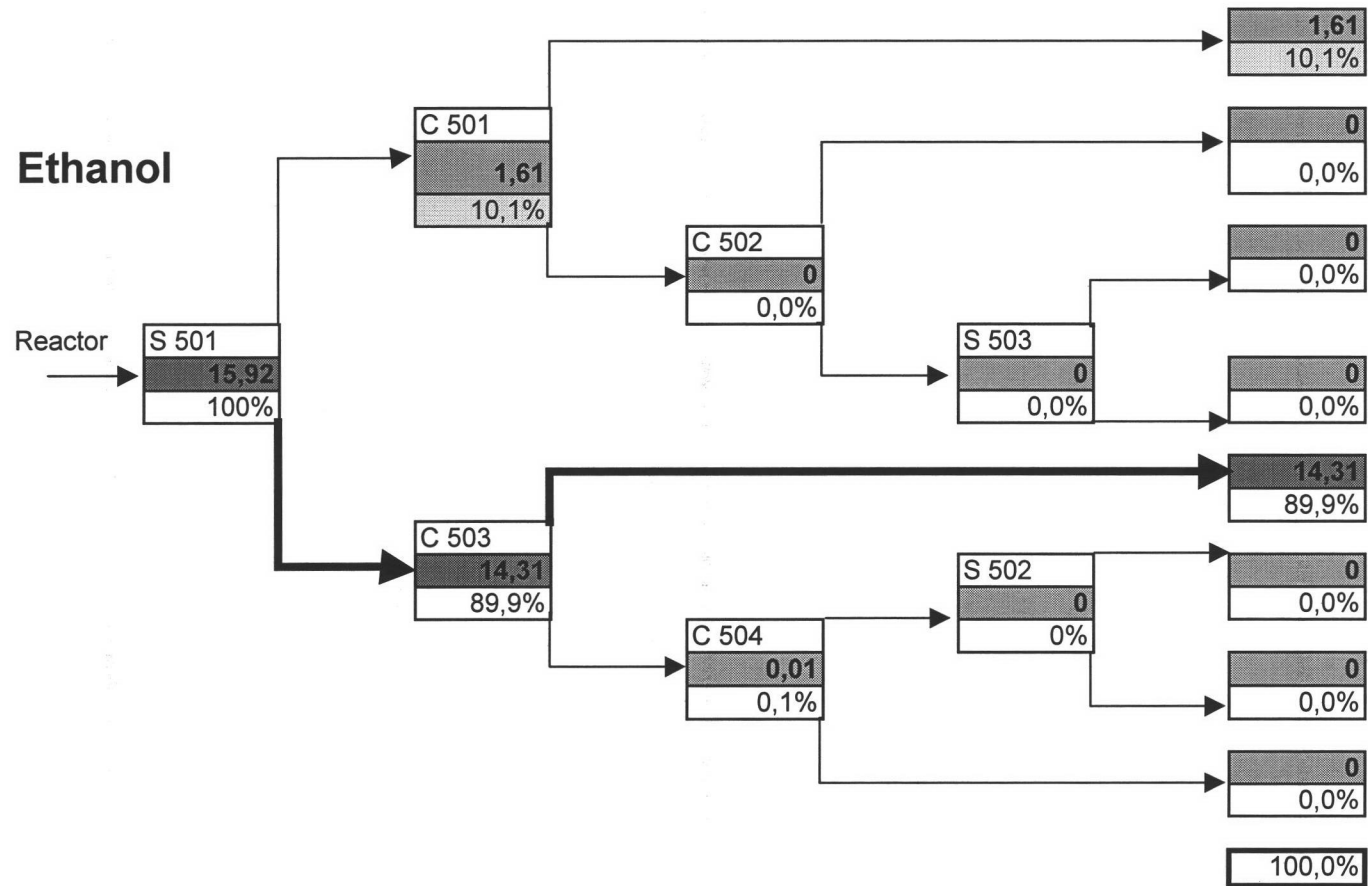
PURE COMPONENT PROPERTIES									
Component Name		Technological Data					Medical Data		Notes
Design	Systematic	Formula	Mol.	Boiling	Melting	Density	MAC	LD ₅₀	
			Weight	Point	Point	of Liquid	value	Oral	
		g/mol		(1) °C	(1) °C	(2) Kg/m ³	mg/m ³	(4) g/kg	
Hexaan	Hexaan	C ₆ H ₁₄	86.2	69.0	-95.0	698.6	90	n.a.	(3)
Ethanol	Ethanol	C ₂ H ₆ O	46.1	78.0	-117.0	800.0	1000	5-15	
Water	Water	H ₂ O	18.0	100.0	--	998.0	n.a.	n.a.	
Citric acid	2-hydroxy-1,2,3-propaantricarbonsuur	C ₆ H ₈ O ₇	192.1	200.0	153.0	1537.0	n.a.	n.a.	
Fosforzuur	Orthofosforzuur	H ₃ O ₄ P	98.00	200.0	42.0	1896.2	1	n.a.	
REE	Ethyl-9-octadecenoaat	C ₂₀ H ₃₈ O ₂	310.5	273.4	8.20	391.2	n.a.	n.a.	
Fosfor	Phosphorus	P ₄	123.9	280.0	44.0	1796.4	0.1	< 0.0005	
Glycerol	Glycerol	C ₃ H ₈ O ₃	92.1	290.0	18.0	1261.0	10	> 15	
Monoglyceride	Propaan-1,2,3-triol-1-[(z)-9-octadecenoaat]	C ₂₁ H ₄₀ O ₄	356.5	321.3	32.8	415.4	n.a.	n.a.	
Oliezuur	9-octadeceenzuur	C ₁₈ H ₃₄ O ₂	282.5	360.0	4.0	883.7	n.a.	>15	
Diglyceride	Propaan-1,2,3-triol-1,2-di-[(z)-9-octadecenoaat]	C ₃₉ H ₇₂ O ₅	621.0	389.0	67.7	501.1	n.a.	n.a.	
Triglyceride	Propaan-1,2,3-triol-1,2,3-tri-[(z)-9-octadecenoaat]	C ₅₇ H ₁₀₄ O ₆	885.4	436.3	92.0	615.2	n.a.	n.a.	
KOH	Potassiumhydroxide	KOH	56.1	1320.0	360.0	1996.0	2	n.a.	
Notes :									
(1)		At 101.3 kPa							
(2)		Density at 20 °C, unless specified otherwise							
(3)		Estimated densities are lower than the expected values (800-900 kg/m ³). When dealing with the pure components, this should be taken account of in the design.							
(4)		Oral in g 's for a male of 70 kg's weight							

Project ID Number :	GG-002
Completion	Dec.2000
Date :	

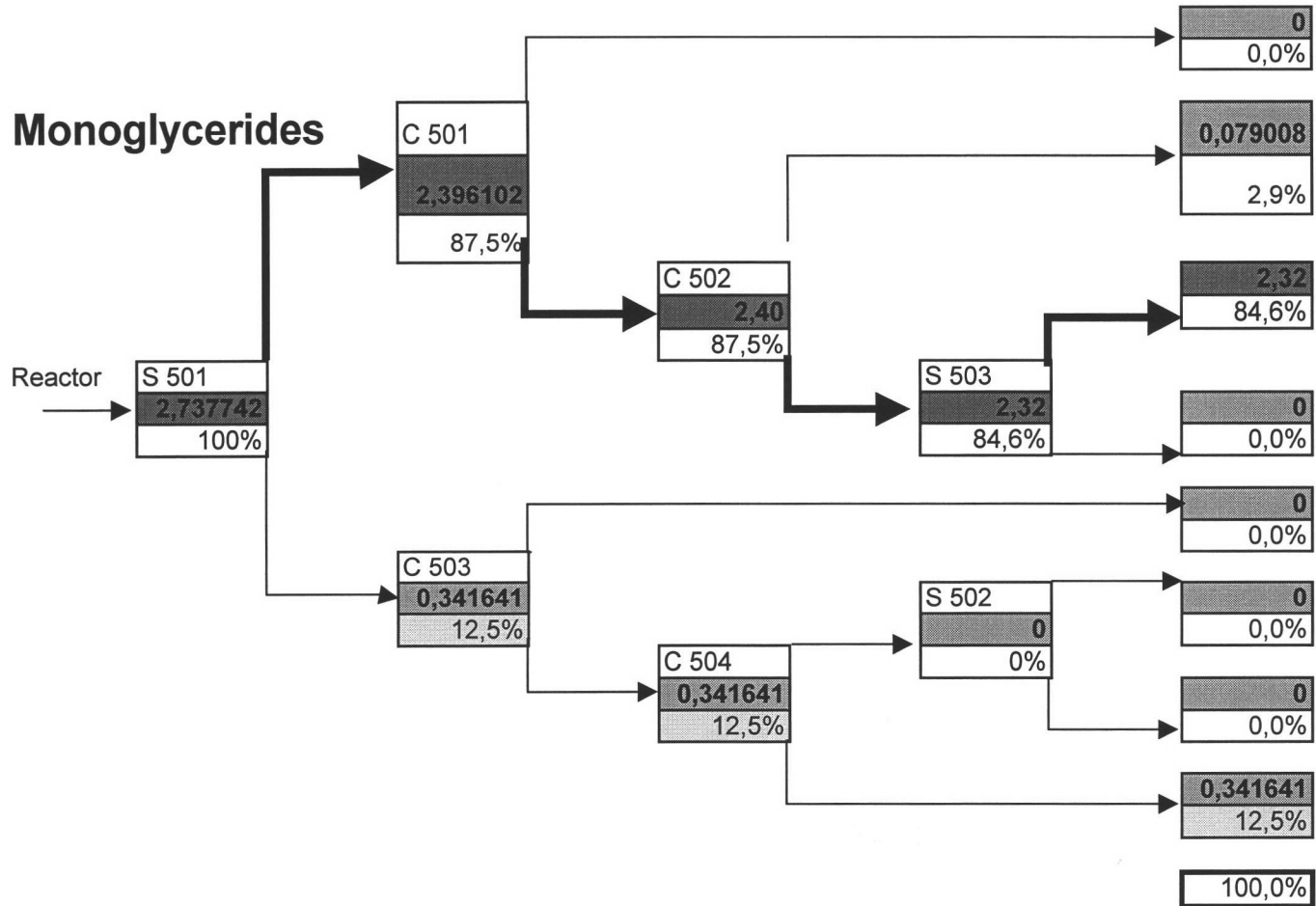
Streams



Streams



Streams



Appendix 5

Ingoing Streams:

- 1,1 Rapeseed
- 1,2 Bio-ethanol
- 1,3 Hexane
- 1,4 Citric acid
- 1,5 Potassium hydroxide
- 1,6 Steam
- 1,7 Electricity

1.1. Rapeseed

The rapeseed will be transported to the plant by boat. A boat can carry approximately 800 tons. So each day a boat will be emptied and the content will be processed.

Stream Name :		Rapeseed					
Comp.	Units	Specification			Notes	Additional Information (also ref. note numbers)	
		Available		Design			
Meal	%wt	52		52,0	(1), (2)	(1) First rapeseed will be separated into meal and rapeseed oil. The next step is the further separation of the oil. The meal will be sent to the farmers. (2) See stream 101 (3) The farmers uses the meal as food for their animals. The protein in the meal is healthy for them. (4) See stream 109/207 (5) In the degumming-process the percentage of phosphor will reduce to less them 0,008 wt%. (6) See outgoing stream 111/112	
Protein	%wt		15,4128		(3)		
Rest	%wt		36,5872				
Rapeseed oil	%wt	40		40,0	(1), (4)		
Tri-glyceride	%wt		28				
Di-glyceride	%wt		5				
Mono-glyceride	%wt		3,17		(2)		
Oleic acid	%wt		0,82				
Phosphor	%wt		0		(5)		
Rest	%wt		3,01		(3)		
Water	%wt	8	8	8,0	(6)		
Total		100	100	100,0	(4)		
Process Conditions and Price							
Temp.	oC	20		20			
Press.	Bara	1		1			
Phase	V/L/S	S		S			
Price	Nfl/ton	475		475	(6)		

1.2. Bio-ethanol

The bio-ethanol will be delivered 95%wt pure. Because of the large volume the ethanol is transported to the plant by boat.

Stream Name :		Bio-ethanol			
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
Ethanol	%wt	95	95,0	(1)	(1) See stream 402. Azeotropic ethanol.
Methanol	%wt	0,5	0,5		
Water	%wt	4,5	4,5		
Total			100,0		
Process Conditions and Price					
Temp.	oC	20	20		
Press.	Bara	1	1		
Phase	V/L/S	L	L		
Price	Nfl/ton	1508	1508		

1.3. Hexane

The hexane will be delivered at 95% purity.

Stream Name :		Hexane			
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
Hexane	%wt	95	100,0	(1)	(1) See stream 204
Rest	%wt	5			
Total			100,0		
Process Conditions and Price					
Temp.	oC	20	20		
Press.	Bara	1	1		
Phase	V/L/S	L	L		
Price	Nfl/ton	1073	1073		

1.4. Degumming acid

The citric acid is transported in 50-kilo drums by trucks.

Stream Name : Degumming acid					
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
Degumming acid	%wt	100	100,0	(1)	(1) See stream 203
Total			100,0		
Process Conditions and Price					
Temp.	oC	20	20		
Press.	Bara	1	1		
Phase	V/L/S	L	L		
Price	Nfl/ton	2000	2000		

1.5. Potassium hydroxide

The potassium hydroxide is delivered to the plant by car or truck on pellets with 110-lb drums.

Stream Name : Potassium hydroxide					
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
KOH	%wt	85	100,0	(1)	(1) See stream 401
Water	%wt	15	0,0		
Total		100	100,0		
Process Conditions and Price					
Temp.	oC	20	20		
Press.	Bara	1	1		
Phase	V/L/S	S	L		
Price	Nfl/ton	10864	10864		

1.6. Steam

The steam which is used at the plant is produced by another company outside the battery limits and it enters our battery limit by pipeline.

Stream Name : Steam					
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
Water	%wt	100	100	(1)	(1) See stream 204
Total		100	100		
Process Conditions and Price					
Temp.	oC	220	220		
Press.	Bara	10	10		
Phase	V/L/S	V	V		
Price	Nfl/ton	32,5	32,5		

1.7. Electricity

Stream Name : Electricity					
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
					(1) We need a tree-phase AC with high voltage.
Total					
Process Conditions and Price					
Voltage	kV	3-10	5	(1)	
Price	kWh	0,22	0,22		

Appendix 5

Outgoing

Streams:

- 2,1 REE
- 2,1 Glycerol
- 2,3 Meal
- 2,4 "Soap"
- 2,5 Steam
- 2,6 Rapeseed waste
- 2,7 Gums/lecithine
- 2,8 Water

2.1

REE

Our main product. Transported by boat or in vessel by truck to the consumers.

Stream Name :		REE				
Comp.	Units			Notes	Additional Information (also ref. note numbers)	
		Available	Design			
REE	%wt	98,6	98,0065	(1)	(1) Main product (stream 507)	
Mono-glycerine	%wt	0,8	0,8	(2)	(2) The rest products in our main product (REE) must be less then the amounts in this table according to the DIN-norm.	
Di-glycerine	%wt	0	0,4			
Tri-glycerine	%wt	0	0,4			
Free fatty acids	%wt	0,1	0,02			
Phosphor	%wt	0	0,001			
Alkali (Na+K)	%wt	0	0,0005			
Water	%wt	0	0,03			
Sulphur	%wt	0	0,01			
Ash	%wt	0	0,03			
Glycerol	%wt	0,02	0,02			
Ethanol	%wt	0,3	0,3			
Contamination	%wt	0	0,002			
Total			100,0200			
Process Conditions and Price						
Temp.	oC		20			
Press.	Bara		1			
Phase	V/L/S		L			
Price	Nfl/ton		1268			

2.2 Glycerol

The purity at which it can be sold is 96%wt. It will be transported in tanks by trucks and boats.

Stream Name :		Glycerol			
Comp.	Units			Notes	Additional Information (also ref. note numbers)
		Available	Design		
Glycerol	%wt	97		(1)	(1) See stream 512
Water	%wt	0,2			
Ethanol	%wt	2			
KOH	%wt	0,8			
Total			100,0		
Process Conditions and Price					
Temp.	oC		20		
Press.	Bara		1		
Phase	V/L/S		L		
Price	Nfl/ton		3212		

2.3 Meal

The meal will be transported in the same boats as the ones which delivered the raw rapeseed.

Stream Name :		Meal			
Comp.	Units			Notes	Additional Information (also ref. note numbers)
		Available	Design		
Meal	%wt	100	100,0	(1)	(1) Stream 114
Total		100	100,0		
Process Conditions and Price					
Temp.	oC		20		
Press.	Bara		1		
Phase	V/L/S		S		
Price	Nfl/ton		330		

2.4 "Soap"

This stream is full of different components. And is for sure not the same as the soap you have at home.
 This waste stream will be transported in tanks by truck to a waste processor, for example the AVR in Rotterdam.

Stream Name : "Soap"					
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
KOH	%wt	10		(1)	(1) Stream 511
Free fatty acids	%wt	10			
Glycerol	%wt	80			
Total		100	100,0		
Process Conditions and Price					
Temp.	oC		20		
Press.	Bara		1		
Phase	V/L/S		S		
Price	Nfl/ton		-500		

2.5 Steam

Steam will be returned to the producer and will be transported outside battery limit by pipeline.

Stream Name : Steam					
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
Steam	%wt	100		(1)	(1) See stream 210
Total		100	0	100,0	
Process Conditions and Price					
Temp.	oC		100	100	
Press.	Bara		1	1	
Phase	V/L/S		V	V	
Price	Nfl/ton		0	0	

2.6 Water

Stream Name :				Water		
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)	
		Available	Design			
Water	%wt	100	100,0	(1)	(1) See stream 111,112	
Total		100	0	100,0		
Process Conditions and Price						
Temp.	oC		20	20		
Press.	Bara		1	1		
Phase	V/L/S		L	L		
Price	Nfl/ton		0	0		

2.7 Lecithine

Stream Name :				Lecithine		
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)	
		Available	Design			
Lecithine Phosphor	%wt	100	100,0	(1)	(1) See stream 208	
Total		100	0	100,0		
Process Conditions and Price						
Temp.	oC		20	20		
Press.	Bara		1	1		
Phase	V/L/S		L	L		
Price	Nfl/ton		0	0		

2.9 Rapeseed waste

Stream Name :		Rapeseed waste			
Comp.	Units	Specification		Notes	Additional Information (also ref. note numbers)
		Available	Design		
Rapeseed waste	%wt	100	100,0	(1)	(1) See stream 116
Total		100	0	100,0	
Process Conditions and Price					
Temp.	oC		20	20	
Press.	Bara		1	1	
Phase	V/L/S		L	L	
Price	Nfl/ton		0	0	

Appendix 6 Thermodynamic properties

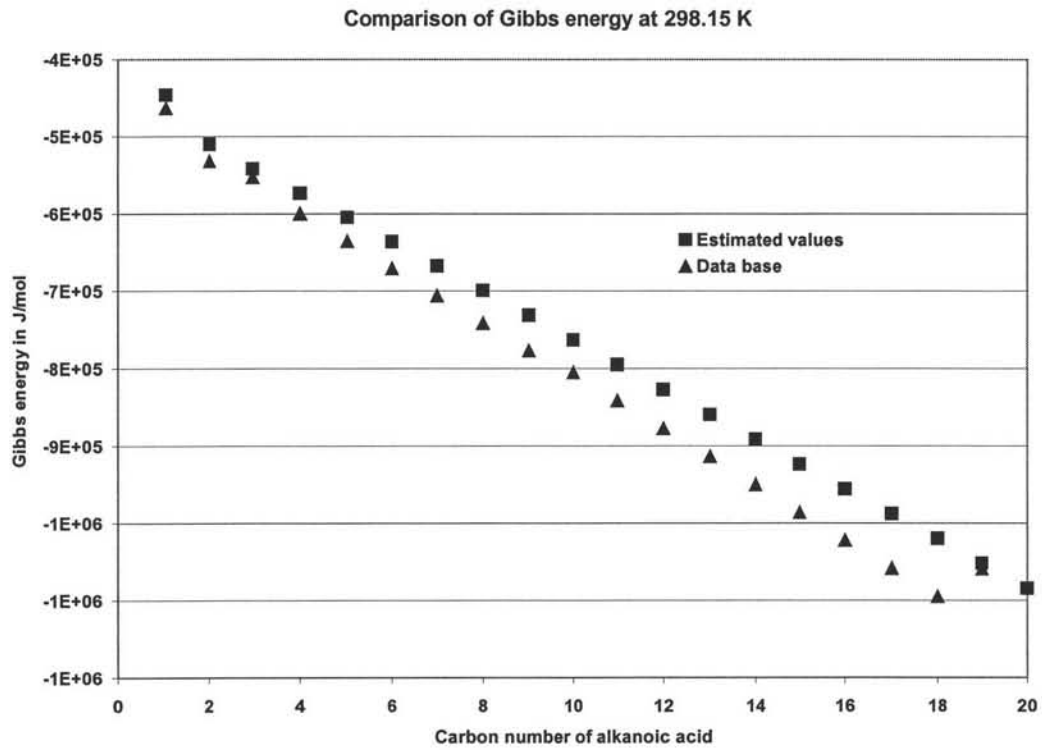


Figure 1 Comparing the estimated values with the literature data of linear alkanolic fatty acids

Appendices

Thermodynamics

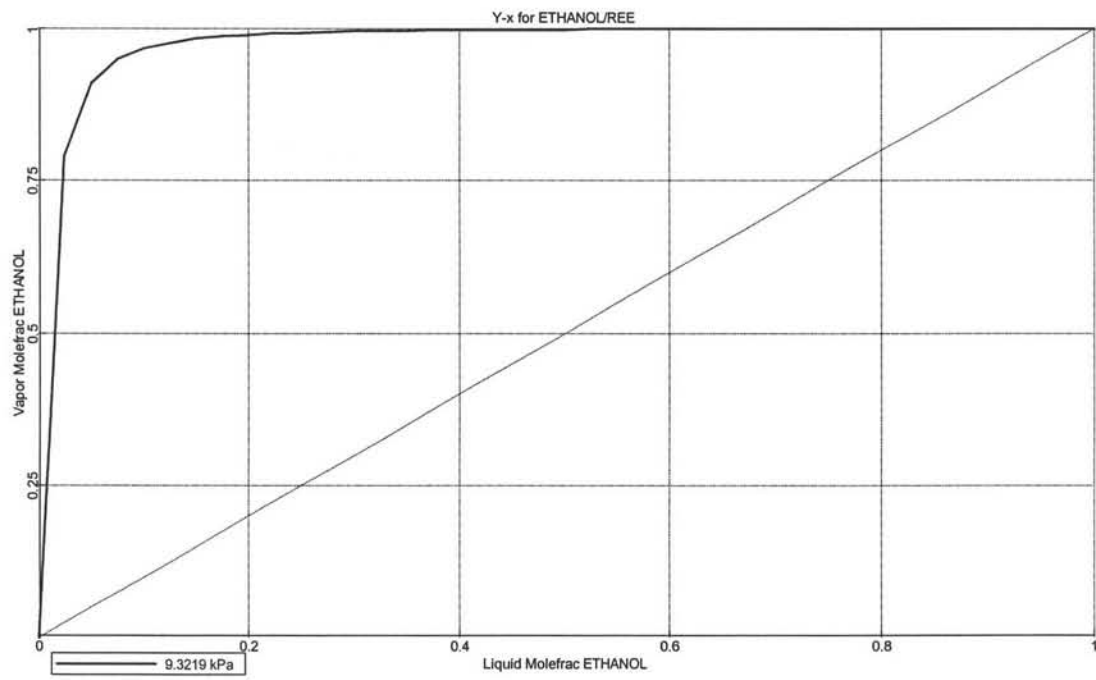


Figure 2 YX plot for Ethanol/REE

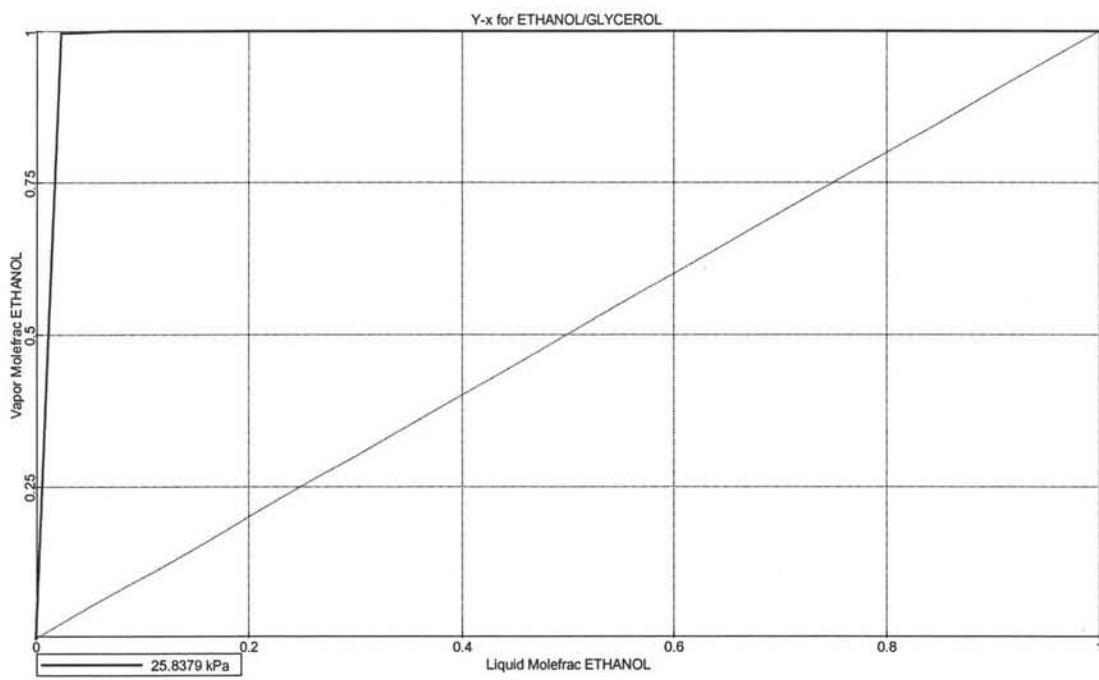
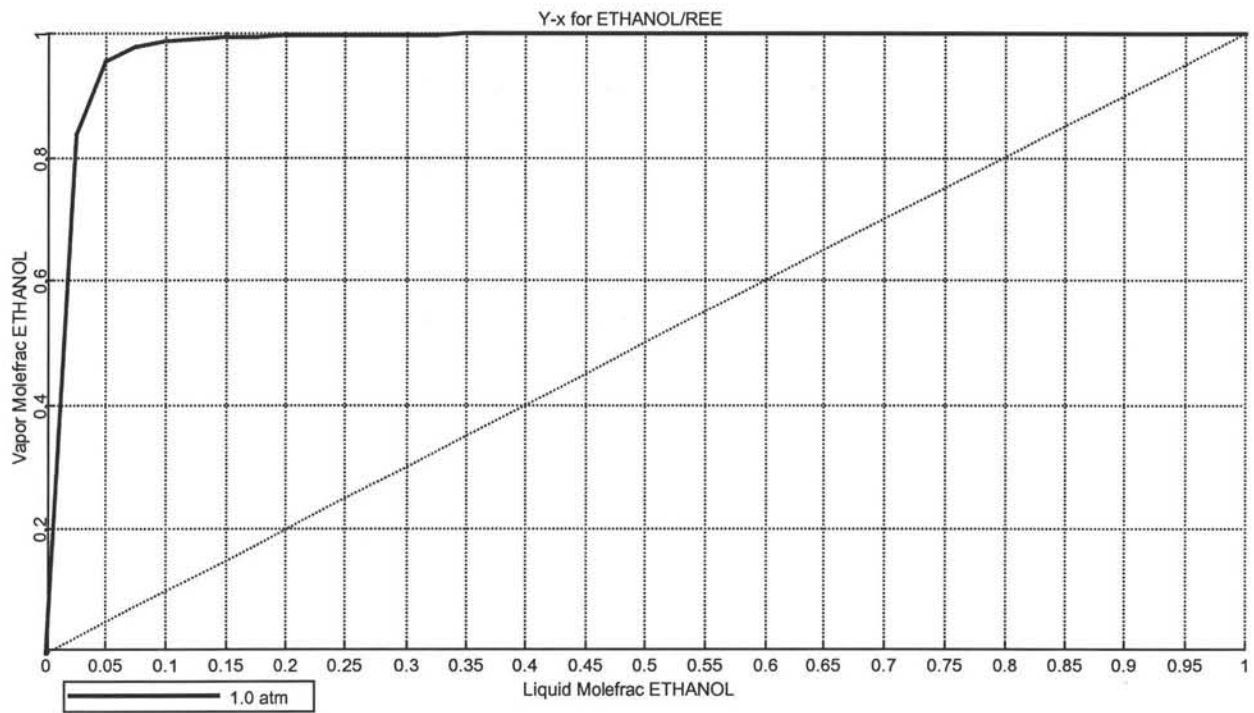


Figure 3 YX plot Ethanol/glycerol

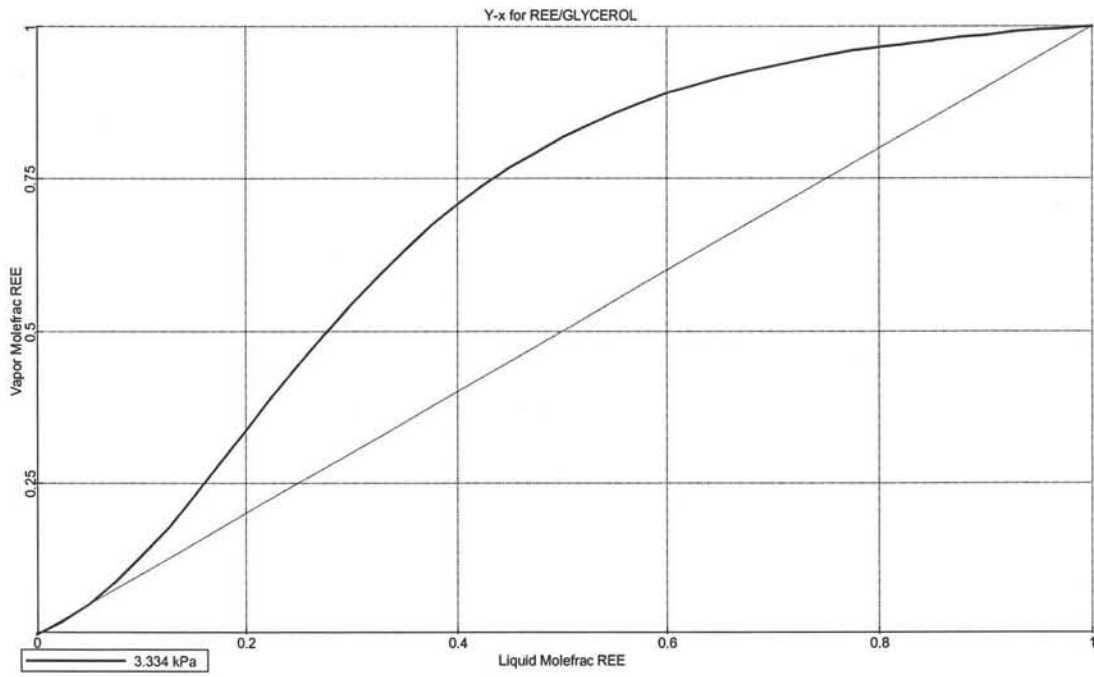


Figure 4 YX plot REE/glycerol

