

Solvent-Based Fatty Alcohol Synthesis Using Supercritical Butane: Flowsheet Analysis and Process Design

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ABSTRACT: The liquid-phase hydrogenolysis of fatty esters to fatty alcohols is an important step in the industrial manufacture of surfactants and detergents. High operating pressures are necessary, due to the low solubility of hydrogen in fatty esters feeds. In principle, these high operating pressures might be overcome by use of a suitable solvent, but only at the expense of large solvent recycle and cumbersome product-solvent separation. The employ of supercritical solvents may resolve these drawbacks, as an elegant solvent-product separation is possible by reverting to the subcritical regime. In the present work the hydrogenolysis of methyl palmitate in supercritical butane is investigated by simulation. Operating conditions are analyzed on the basis of vapor liquid equilibrium data and chemical equilibrium considerations. Separation and recycle problems are evaluated and discussed on the basis of a flowsheet analysis. It is demonstrated that an efficient hydrogenolysis process may be developed by using supercritical butane as solvent. A moderate operating pressure (9 MPa) and temperature (470 K) lead to high conversion levels and high product purity. A hydrogen to ester molar ratio of 4:1 in the feed is achievable, which compares favorably to existing liquid- and gas-phase processes, and allows recycle streams to be reduced.

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KEY WORDS: Conceptual design, ester hydrogenolysis, fatty alcohols, process synthesis, supercritical butane.

Liquid-phase hydrogenolysis of fatty esters to fatty alcohols is typically carried out industrially at pressures ranging from 20 to 30 MPa, in slurry or fixed-bed hydrogenation reactors (1–4). The high pressure is required (i) to overcome the limited solubility of hydrogen in methyl ester feed, (ii) to overcome chemical equilibrium limitations of the hydrogenolysis reaction, and (iii) to enhance catalyst activity. Obviously, these high operating pressures seriously affect capital investments and operating costs.

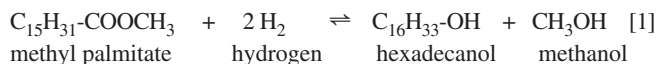
Use of a supercritical solvent may reduce the above operating pressures. A thermodynamic analysis of relevant supercritical mixtures was presented earlier (5), and several advantages of supercritical operation were identified. The aim of the present study is to assess whether hydrogenolysis of methyl palmitate by using supercritical butane is feasible from a process-design point of view. Factors analyzed are the

reaction conditions, separation efficiency, recycle flows, energy use, and product purity.

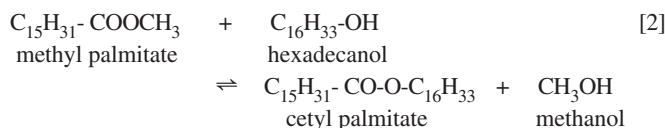
Flowsheet synthesis methodology. The development of the flowsheet structure follows the paradigm of the hierarchical methodology proposed by Douglas (6). It consists in a top-down refinement of intermediate design steps. Subsequently, we will discuss: input information, scale, input/output structure, recycle structure, separation systems, and energy integration. The procedure is general, provides a feasible and consistent modeling framework, and leads to a computationally tractable problem. Special attention is paid to the development of the separation system, which together with the reactor determines the material balance envelope, as the feasibility of the supercritical process depends critically on the systems required for product/reactant/solvent separation and recycle.

Input information. (i) Simplifications and assumptions. Depending on the feedstock, fatty acid chain lengths in the range of 12 to 20 carbon atoms are most abundant in natural fats and oils. According to Scarlet and McKinley (7), Davy McKee patented a low-pressure process applicable to smaller (C_{12} and C_{14}) esters, which have a comparatively higher vapor pressure. The challenge consists of developing a low-pressure process suitable for esters with a lower vapor pressure, such as the higher palm oil-based (C_{16} and C_{18}) feedstocks. A mixed feed of both C_{16} and C_{18} esters could have been considered, both saturated and unsaturated components. However, it is difficult to obtain reliable physical properties for these high molecular weight components. Hence, it was decided to consider only a feed of saturated C_{16} ester, for which physical properties have been accurately determined, both as a pure component and in relevant mixtures.

(ii) Reaction stoichiometry. The equilibrium limited hydrogenolysis of methyl palmitate is represented by:

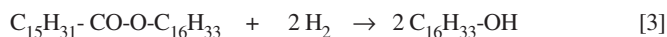


A secondary reaction produces cetyl palmitate (also referred to as wax ester) by transesterification of methyl palmitate with the product hexadecanol:

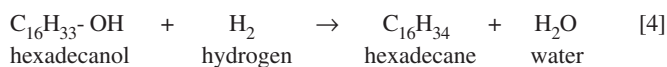


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Cetyl palmitate can be recycled and hydrogenated back to hexadecanol:



Some impurities, such as ethers, aldehydes and hydrocarbons, might also be produced as by-products. The first two categories have been neglected, as only traces are found in practice. The formation of hexadecane by the hydrogenation of hexadecanol is, however, explicitly accounted for:



(iii) *Raw materials and utilities.* Raw materials consist of methyl palmitate, hydrogen, and butane. We assume a methyl palmitate purity of 99.95%. Specifically, the absence of fatty acids is important as these are known to inhibit the catalyst. Hydrogen may be available at high purity or contain some methane. We will assume a feed of pure hydrogen. Butane is readily available as a chemical feedstock from refineries or gas plants and is also assumed to be 100% pure. The utilities used are electricity, steam ($P = 7.5$ MPa), hot oil, and cooling water (21°C).

Product specifications. We assume that hexadecanol is 99 wt%. The secondary components, hexadecane and water, are allowed a maximum of 0.5 and 0.2 wt%, respectively, in the product alcohol. As a side-product, the process delivers methanol with a purity of at least 98 wt%, suitable for the recycling to the esterification process.

(v) *Decisions on input/output structure and recycles.* To define the input/output structure, the following design decisions were made: Use of high-purity hydrogen as reactant; recycle of hydrogen and butane, if possible by a liquid recycle to save energy and capital investment for recompression; and recycle of the wax ester, with provision of a bleed stream to prevent the accumulation of heavy impurities.

(vi) *Process constraints.* The maximum allowable temperature in the process is at 543 K, because above this value the hexadecanol will decompose. Cooling will be done exclusively with water (298 K). A minimum temperature approach of 10 K is assumed for performing the heat integration by applying the Pinch Point Analysis. The maximum pressure is set at 9 MPa, while the minimum vacuum pressure is set at 0.01 MPa.

Scale. The conceptual design will be developed for a plant production of 50 Kton/year.

Input/output structure. Reactor analysis. We assume that the catalytic reactor is of a fixed-bed type, where hydrogenolysis takes place in the supercritical phase over a Cu/ZnO/SiO₂ catalyst. The reactor is taken to operate at 9.0 MPa and 473 K. The analysis is based on the equilibrium ester conversion in Reaction 1. This assumption is supported by the experimental results of Van der Scheur *et al.* (9). For methyl palmitate hydrogenolysis over a Cu/ZnO/SiO₂ catalyst, the selectivity toward hexadecanol, cetyl palmitate (wax ester), and hexadecane was reported to be 95, 4.5, and 0.5 mol%, respectively (2).

Since formation of wax esters, and particularly that of alkanes, appears to be kinetically controlled, it was decided to combine the calculated equilibrium conversion with the reported selectivity to cetyl palmitate and hexadecane. This leads to the following percentage yield of individual reactions: methyl palmitate hydrogenolysis 94.2, wax ester formation 4.464, wax ester hydrogenolysis 99.0, and alkane formation 0.494. Note that the assumptions made above imply that inert materials, such as methane, are not formed and therefore cannot give rise to complications due to accumulation. A purge of any inerts present would lead to concurrent losses in both hydrogen and butane. A sharp separation of these species from the inert material before recycling, or purification by purging, would be very costly.

Material balance boundary. Taking into account the above decisions, we can draw an abstract picture of the process, which is displayed in Figure 1. Besides reactant feeds there is a make-up stream of butane and a liquid bleed, containing mainly heavy products, which could be used as fuel for furnaces or boilers.

Recycle structure. The first elements of the flowsheet structure are introduced by examining the interaction between reactor and separation systems. Initially, all necessary separations are lumped in a large black-box unit. The synthesis of the separation systems is treated separately in the next section.

Figure 2 displays the simplest internal structure, reactor and recycles. Fresh and recycled reactants, together with the solvent, are brought to supercritical pressure and fed to the reactor at reaction temperature. The ratio of methylpalmitate/hydrogen/butane equals 1:4:35. This ratio follows from phase envelope calculations as the composition that maximizes the ester concentration and minimizes the hydrogen concentration, while keeping the critical temperature and pressure of the mixture below limits set by the reaction conditions (see also Ref. 5). A further constraint is that the molar ratio of hydrogen/ester must exceed 2 for reasons of reaction stoichiometry. A hydrogen/ester ratio equal to 4 leads to a modest recycle of hydrogen. Because the cost of gas compression is high, this ratio is favorable with regard to process economics. A feed ratio of butane to ester of 35 is approximately one order of magnitude lower than is obtained for a subcritical solvent such as octane (9). Butane is recycled and pressurized from subcritical to supercritical conditions as a liquid, at low cost.

The product from the reactor is depressurized to subcritical conditions to allow for product-solvent separation. Follow-

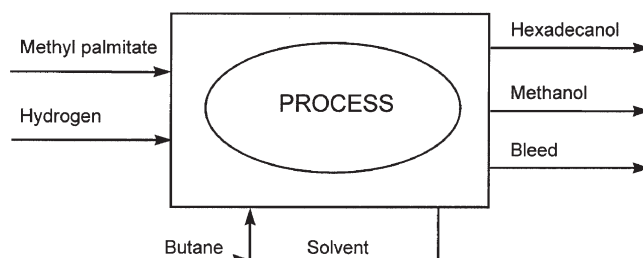


FIG. 1. Input/output structure.

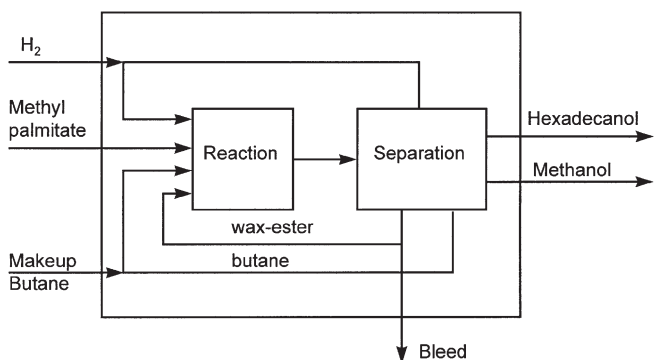


FIG. 2. Recycle structure.

ing depressurization, the pressure should be under 3.8 MPa, which is the critical pressure of butane, but not too low in order to minimize (re-)compression costs. The lower temperature limit is 308 K, if cooling water is to be used. Because of high single-pass conversion (about 95%), the amount of recycled ester is small. However, the wax ester formed must be recycled in order to improve the overall yield. This will create the second liquid recycle. Other impurities will not be recovered, but eliminated by a bleed stream.

Synthesis of the separation system. General structure. Figure 3 displays the general superstructure of the separation system. The first separation split is an essential design step. The supercritical stream leaving the reactor (9.0 MPa and 473.2 K) is depressurized to 3.5 MPa, cooled, and fed to flash FL1, where a first separation takes place. A sharp split is created between hydrogen and *n*-hexadecanol. Indeed, 99.5% of the hexadecanol can be recovered in the liquid phase at 418 K and 3.5 MPa, and 98% of the hydrogen will then end up in the gas phase. However, roughly 80% of the methanol and butane also ends up in the vapor phase, requiring further separation.

Gas-separation system. To prevent too much of the butane and the methanol ending up in the gas phase, a second flash is applied to recover as much as possible of the methanol and butane as a liquid. This is schematically shown in Figure 4. Hydrogen dissolved in the liquid streams leaving the flashes is recycled with the butane. The vapor effluent from flash FL1 is depressurized at 3.0 MPa and cooled from 415 K to 308 K and split again in flash FL2. In this way, about 99% of the

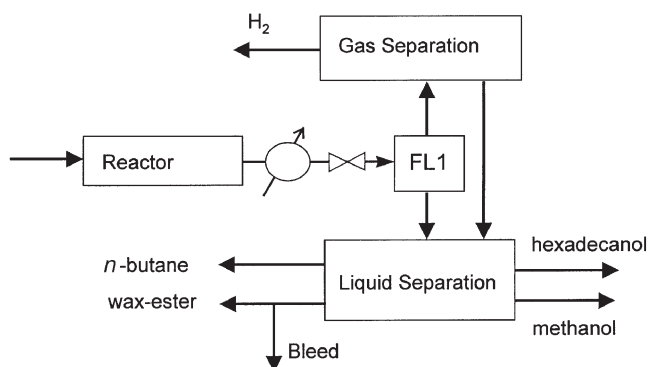


FIG. 3. General superstructure of the separation system.

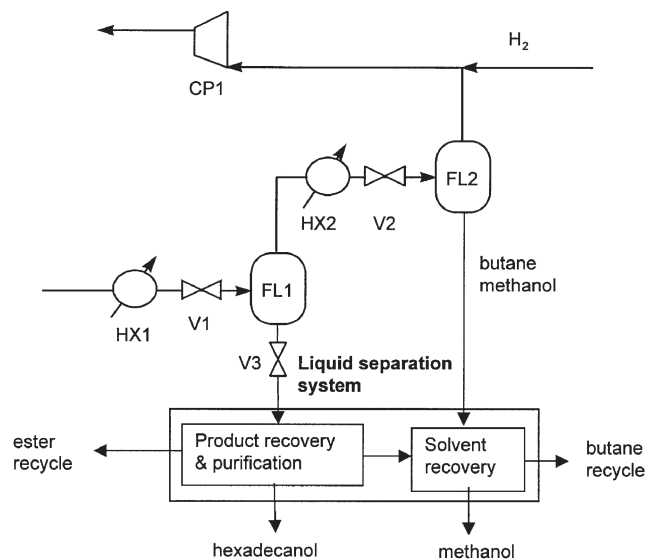


FIG. 4. Gas separation system. FL, flash; HX, heat exchanger; CP, compression; V, valve.

methanol and butane are recovered in the liquid phase, and sent to the liquid-separation system. Note that the streams from the flashes FL1 and FL2 are fed in different locations of the liquid-separation system, as will be discussed later.

The gas phase contains approximately 90 mol% hydrogen—the rest being butane—and is recycled to the reactor. Fresh hydrogen is compressed from 20 to 50 bar. After mixing with recycled hydrogen, the pressure of the gas mixture is further increased to 90 bar.

Liquid separation system. Figure 5 shows a possible solution for separation of the liquid streams leaving the gas separation section represented in Figure 4. The liquid effluent

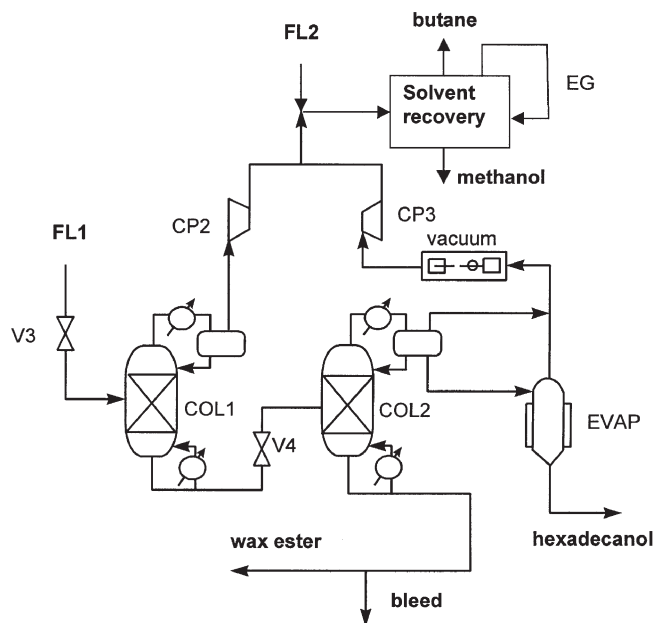


FIG. 5. Liquid separation system. COL, distillation column. EG, ethylene glycol; see Figure 4 for other abbreviations.

from FL1 contains virtually all hexadecanol, but contains also a large amount of butane. First, the pressure is reduced from 35 to 4 bar. After cooling to 308 K, this stream is fed to the top of a reboiled stripping column COL1. Simulations indicate that only a few trays are required to separate the two key components. The top vapor product recovers almost quantitatively the methanol and butane. The bottom product contains the hexadecanol and the wax ester. To prevent thermal decomposition, a small amount of butane (below 10% mol) is allowed, which limits the reboiler temperature below 540 K. A better separation in this column at lower pressure would require cooling by a refrigeration system.

Product purification. The product, hexadecanol, is recovered by vacuum distillation in COL2 as a top liquid distillate, while butane, methanol, and residual lights are removed as vapor distillate. The top-stage temperature in COL2 is 507.3 K. Only four theoretical trays are required. The bottom product contains mainly wax ester that is recycled. To keep the bottom temperature below 543 K to prevent thermal decomposition, a residual pressure of about 0.01 MPa is necessary. The yield of hexadecanol production is of 99%, with better than 98 wt% purity. More advanced product purification is achieved in a downstream evaporator. The vapor distillate is compressed at 10 bar, cooled, and mixed with recycled butane from hexadecanol purification.

To prevent the accumulation of impurities, a very small bleed stream of 0.1 wt% from the recycled wax ester is withdrawn. The recovered butane and methanol are compressed and mixed with the stream from the COL1 for further recovery.

Solvent recovery. For optimal energy efficiency, butane is recycled as a liquid, so no compression is required. However, a small amount of methanol, approximately 3 mol%, is present. Equilibrium calculations show that the presence of 6 mol% methanol in butane would lead to a decrease in the

equilibrium conversion to 98%. Hence, accumulation of methanol must be prevented. Another complication arises from the minimal boiling point azeotrope of butane and methanol (Fig. 6). At 3.0 MPa the azeotrope contains about 25 mol% methanol (10). This azeotrope is not pressure sensitive. Hence, simple distillation is not possible.

Interestingly, a similar problem exists in the synthesis of MTBE, where excess alcohol in reaction must be recovered and recycled. Patent literature reports the use of glycols as extraction solvents (10). Ethylene glycol (EG) seems attractive as it is hardly soluble in butane, and methanol readily dissolves in it. Methanol can be separated from EG by simple distillation. An extraction process for removal of 5% methanol is described in Reference 10.

Liquid activity coefficients were predicted using the UNI-FAC model. The column for the extraction of methanol from butane with EG requires 10 theoretical stages, for a molar ratio glycol/butane of 1:14, and a methanol split fraction in extract of 92.3%. The extraction column works at 30 bar and an almost constant temperature of 306 K. Although predictive L-L equilibrium calculations are not necessarily very accurate, the results are in good agreement with those reported by Chase *et al.* (10). The raffinate phase, containing butane and a small amount of methanol (below 10% as split fraction), is recycled to the reactor. The extract phase goes to a distillation column for the separation methanol/EG (COL3 in Fig. 7), requiring six theoretical trays. Here methanol is obtained as a top product, with small amounts of butane, while the bottoms are recycled to the extractor. In COL3 the top stage is at 416.2 K and the bottom at 540.6 K. Makeup of both butane and EG is necessary to compensate for losses.

The above elements can be assembled to yield a process flowsheet that is shown in Figure 7.

Evaluation of supercritical conditions. For reliable operation, it is required that the reaction mixture remains supercritical, since properties like viscosity and diffusion coefficients change dramatically near the critical point. From part 1 of this study (5), it became clear that the initial reaction mixture entering the reactor is supercritical at 473 K and 9.0 MPa. This is also indicated in Figure 8, where the phase envelopes of the reactor inlet and outlet streams are shown, after the convergence of recycles.

Energy integration. Energy conservation is implemented by integrating process streams in such a way that the amount of heat exchanged is maximized, thereby avoiding an excessive number of heat exchangers. Given the selected minimum approach temperature ΔT_{\min} of 10 K, and after supplying all stream data in our design, a Composite Curve was developed as shown in Figure 9. The Pinch-Point is located between 349 and 339 K. We recall that the Pinch indicates the region where the heat recovery problem is the most constrained. Two subsystems are generated, above and below the Pinch, where heat recovery can be analyzed and solved independently. Moreover, no cold utility is allowed above the Pinch, as well as no cold utility below the Pinch. Minimal energy requirements and heat exchanger network area can be determined by a

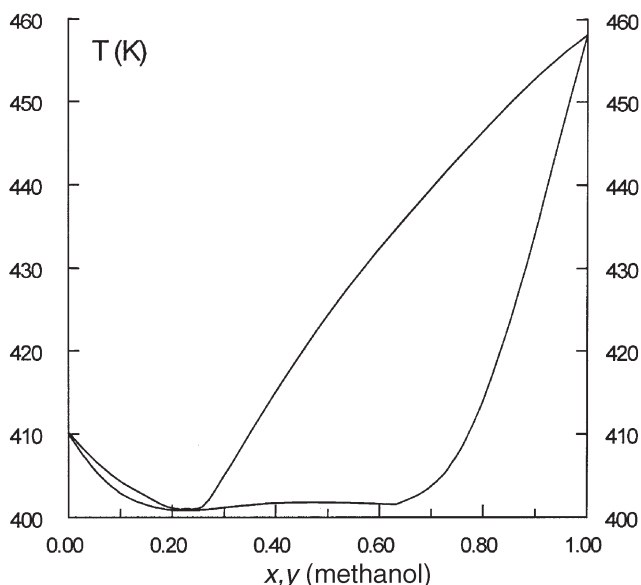


FIG. 6. Liquid (x) and vapor (y) equilibrium compositions of methanol-butane mixture at 3 MPa.

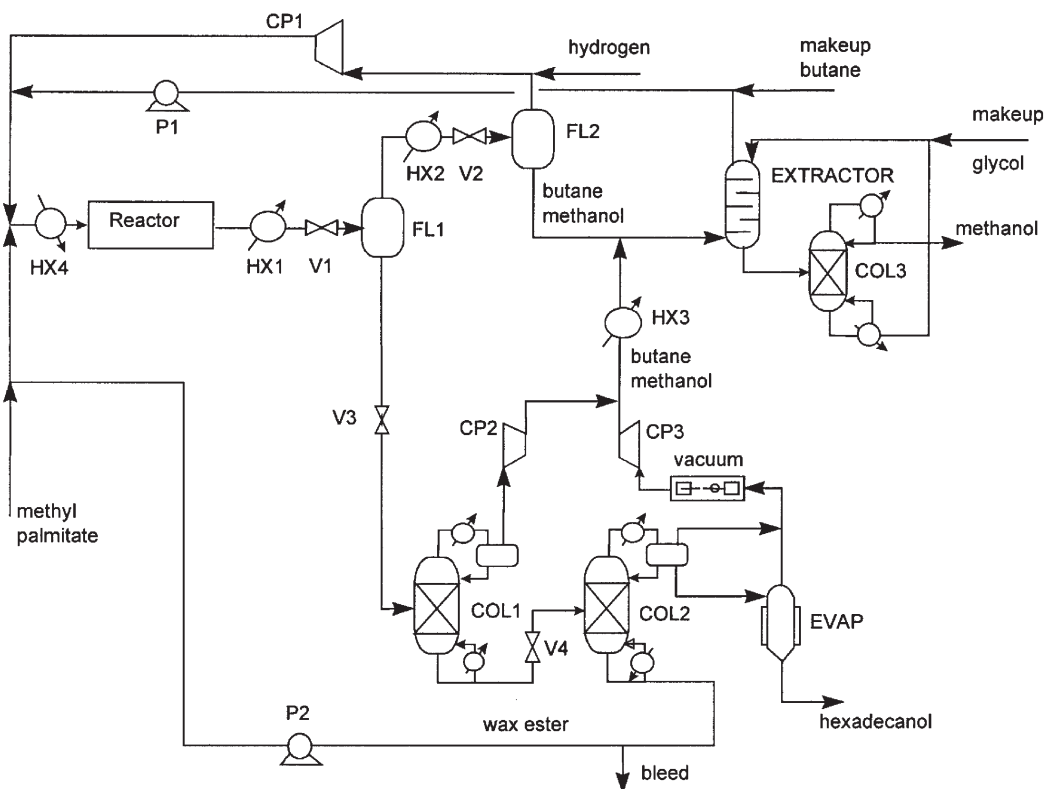


FIG. 7. Complete flowsheet of the fatty ester hydrogenolysis using supercritical butane. EVAP, evaporator; for other abbreviations see Figures 4 and 5.

targeting procedure, before any detailed calculation of the heat exchangers.

A composite curve (Fig. 9) reveals good opportunities for heat integration, because the hot and cold streams can be matched over a large interval of temperature (Part II). The heat balance is closed with 2.793 MW cold utility (Part I), and 2.957 MW hot utility (Part III).

Figure 10 displays the Grand Composite Curve, where the difference in enthalpy between hot and cold stream is displayed against a conventional temperature scale obtained by

shifting the temperature of hot and cold streams with $\Delta T_{\min}/2$. In this diagram the optimal use of utilities can be assessed. In this case several levels of steam temperature may be used, from low-pressure to superheated steam. Table 1 presents the minimal energy requirements and the optimal for $\Delta T_{\min} = 10$ K.

The construction of a minimal-cost or minimal-energy heat exchanger network is beyond the scope of the present investigation. However, with three heat exchangers, as placed on the flowsheet given in Figure 7, already over 90% of the target heat can be recovered.

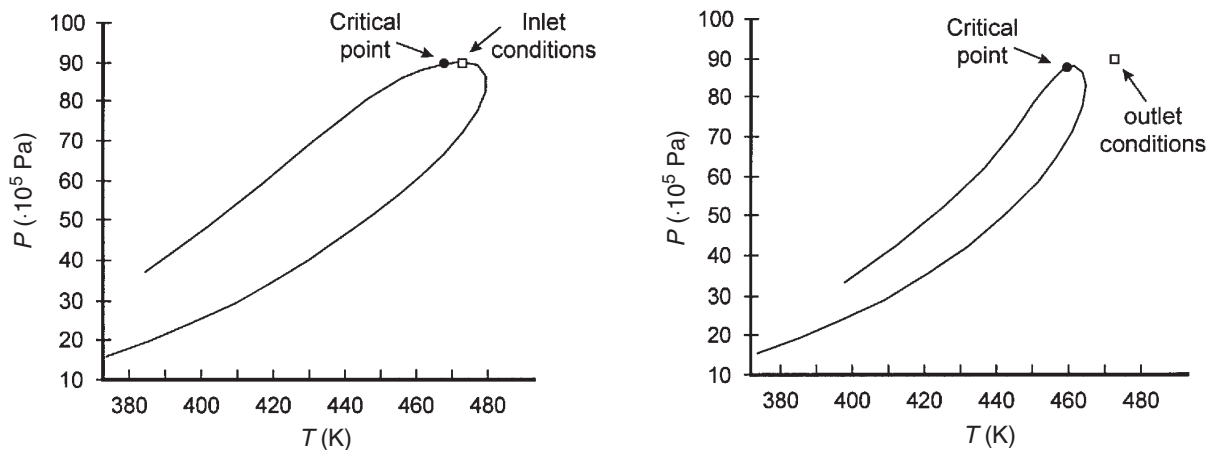


FIG. 8. Pressure-temperature envelopes for the feed flow (left) and product flow (right) to/from the reactor, together with the critical points and process conditions of both flows.

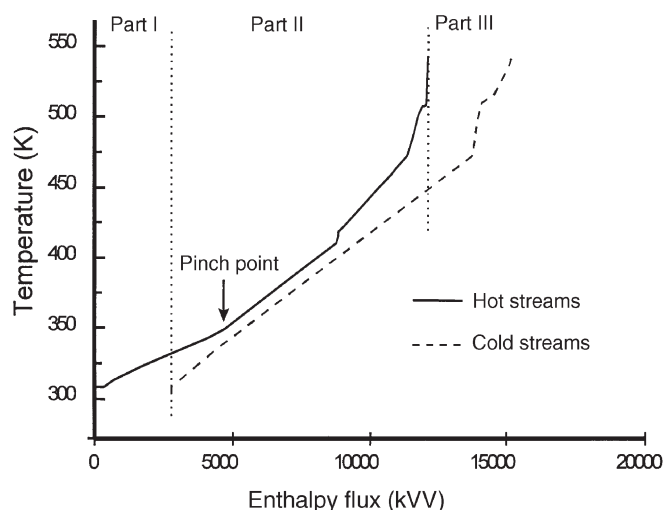


FIG. 9. Composite curve of all hot and cold streams.

DISCUSSION

The flowsheet presented in Figure 7 is the result of the conceptual design approach. The process design for the hydrogenolysis of methyl palmitate under supercritical conditions and using butane as a solvent has been evaluated for a reaction temperature and pressure of 473 K and 9.0 MPa, respectively. Under these conditions, and a feed consisting of 2.5 mol% methyl palmitate and 10 mol% hydrogen butane, an equilibrium conversion of 99.2% is obtained. The overall material balance, including reactant, solvents, product with specifications, as well as main recycle streams, is shown in Table 2.

The amount of hydrocarbons (hexadecane, butane) in the product lies within limits of commercial fatty alcohol specifications (11,12). The other significant impurity consists of unreacted methyl palmitate, which could be removed by saponification when required.

The butane impurity level of the methanol stream, to be recycled to the esterification process, is quite high. This is un-

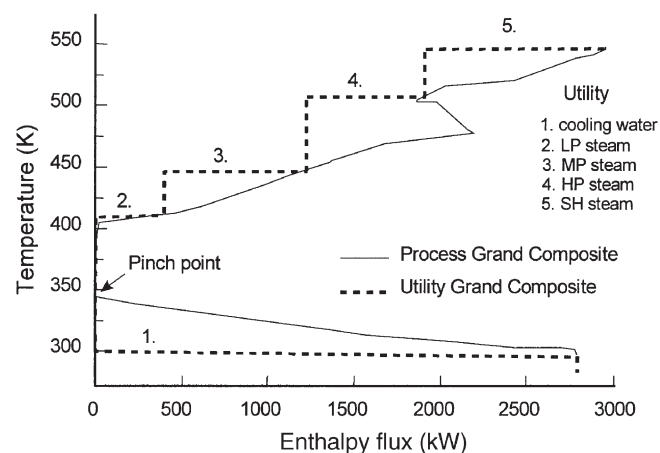


FIG. 10. Grand composite curve for process and utility streams. LP, low pressure; MP, medium pressure; HP, high pressure; SH, superheated.

TABLE 1
Utilities for a 50 kton per year Fatty Alcohols Plant
Using a Supercritical Solvent^a

Stream	Cold/ hot	Utility name	Supply T (K)	Target T (K)	Duty (kW)
1	C	Cooling water	293.2	298.2	2793.0
2	H	LP-steam	417.2	417.1	401.4
3	H	MP-steam	453.2	453.1	823.0
4	H	HP-steam	513.2	513.1	694.7
5	H	Superheated steam	563.2	553.2	1037.7
Total cold utilities					2793.0
Total hot utilities					2956.8

^aLP, low pressure; MP, medium pressure; HP, high pressure.

likely to influence the esterification reaction to any significant extent.

Evaluation. Little information on energy use and process economics of fatty ester hydrogenolysis plants has been published. For the Lurgi direct fatty acid hydrogenation process, however, energy and cooling water use have been published for a 29 kton/yr fatty alcohol plant. It should be mentioned that this process is based on hydrogenation of fatty acids rather than fatty acid esters. However, it gives an indication with regard to the energy and compression duties of a typical hydrogenolysis plant. One of the expected advantages of a solvent-based process over a regular high-pressure or gas-phase process is the lower compression duties, as here a much smaller hydrogen/ester ratio is employed.

The hydrogen/ester ratios used in industrial processes are shown in Table 3. It is clear from Table 3 that the largest excess of hydrogen is used in the gas-phase process. A solvent-based, and especially a supercritical butane-based process, allows for a much lower excess of hydrogen.

The amount of electric power, hot utilities, and cooling for the slurry phase and the present supercritical process are shown in Table 4. As expected, the present supercritical process requires less compression power. Although the separation section of the supercritical process is somewhat more complex, the amount of heat required is not higher than indicated for the Lurgi slurry process. As indicated in Table 3, for trickle-phase hydrogenolysis processes, a higher hydro-

TABLE 2
Overall Material Balance

Components	Reactant and solvents	Product ^a	Subproducts ^b	Recycles ^b
Methyl palmitate	56.06	—	—	—
Hydrogen	0.835	—	—	—
Butane makeup	0.04	<800 ppm	—	1.60
Glycol	Negligible	<10 ppm	—	0.1
Methanol	—	<100 ppm	6.22	7.0
Hexadecanol	—	98.6%	0.0006	—
Hexadecane	—	0.5%	—	—
Cetyl palmitate	—	Trace	0.0022	—
Flow rate	56.93	50.71	6.22	—

^aComposition in wt%.

^bFlow rate in kton/year.

TABLE 3
Hydrogen/Ester Feed Ratios of Several Processes

Process	Gas-phase	Trickle-phase	Slurry-phase (Lurgi)	Solvent (octane)	Solvent (supercritical)
H ₂ /Ester ratio (mol/mol)	600 ^a	100 ^a	20 ^b –50 ^a	6–30	4

^aTaken from References 11.

^bTaken from Reference 2.

gen/ester ratio is common and therefore higher compression duties are expected in that case.

Another important further advantage of the presently developed supercritical process lies in the lower investment and operating costs as arise from the reduction of the reactor operating pressure to 9.0 MPa as compared to 20.0–30.0 MPa for commercial hydrogenolysis processes.

A moderate-pressure process for fatty methyl ester hydrogenolysis was developed, based on the use of supercritical butane as solvent. The overall process appears feasible and has important advantages over either high-pressure liquid or vapor-phase operation.

The limited solubility of hydrogen in the fatty methyl ester feed in case of conventional liquid-phase ester hydrogenolysis processes can be overcome by using supercritical butane as solvent. As compared to subcritical solvents, the attainable methyl palmitate concentration in the reactor increases by one order of magnitude.

Reaction equilibrium calculations reveal that in supercritical butane high conversion levels—in excess of 99%—can be obtained at a smaller than usual hydrogen/ester ratio, closer to the stoichiometric ratio. Hence, both hydrogen and solvent recycles are reduced. The process can be operated at a moderate pressure of 9.0 MPa, vs. 20–30 MPa for conventional liquid-phase processes. Final product purity is better than 99%.

Due to the lower operating pressure of the currently proposed process, capital as well as operating expenses are expected to be considerably lower. Estimations of the energy consumption indicate similar values as for conventional hydrogenolysis plants.

TABLE 4
Utilities for the Supercritical and the Lurgi Slurry Processes (4)

Utility	Supercritical	Lurgi slurry	Unit
Electric power	115	130	kWh
Energy for heating	1.59 ^a	1.65 ^b	GJ
Cooling water	35	35	m ³

^aTotal steam consumption.

^bCalculated from energy required for hydrogen-preheating and total steam consumption. Basis: 1 ton of fatty alcohols.

The energy efficiency of the separation section may be improved by reducing the amount of solvent. However, severe constraints are set by the fact that the critical temperature of the reaction mixture increases quite dramatically for high methyl palmitate concentrations. For example, a fourfold increase in this concentration would lead to a critical temperature of the mixture going up from 470 K to about 570 K. Such high-reaction temperatures result in a drop in selectivity, due to overhydrogenation to alkanes. Thus, further improvements for the supercritical butane-based hydrogenolysis process would await new catalysts, with superior selectivity at high-operating temperatures.

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