EUROPEAN ROADMAP OF PROCESS INTENSIFICATION

- TECHNOLOGY REPORT -

TECHNOLOGY: Pervaporation-assisted reactive distillation

TECHNOLOGY CODE: 2.2.8.2

AUTHOR: Achim Hoffmann, University of Dortmund

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1. Technology

1.1 Description of technology / working principle

(Feel free to modify/extend the short technology description below)

The integration of membrane separation and reactive distillation combines the advantages of reactive and hybrid separation processes and thus represents a highly intensified process unit. Even though reactive distillation and membrane separation are established technologies for intensified processes, research on its combination is relatively new and only few publications can be found.

However, the working principle of this technology is very similar to that of membrane assisted distillation, as described by Kreis and Górak [2006][1]. The integration of pervaporation or vapour permeation unit, that is very selective and not limited by the vapour-liquid equilibrium, offers multiple configuration options in order to combine membrane modules and distillation columns to a single *hybrid process* (Figure 1).

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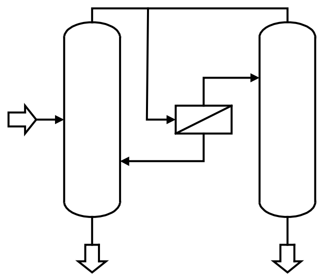

Figure 1: Optional configurations for membrane assisted distillation

Options 1 and 2 are used to separate azeotropic mixtures at the top of the column. The optimum configuration depends on the composition of the azeotropic mixture. While azeotropes with a high concentration of the target component can be separated by a membrane unit, it is often more economic to perform the final purification of an azeotropic mixtures with a considerable concentration of the permeating boiling component in a second distillation column. Option 3 can be used to assist the separation of close boiling mixtures and option 4 is well suited to remove a pure component from a side stream of the distillation column. The design of such hybrid processes with strong interactions between the two unit operations requires a distinctive process understanding and adequate models for both unit operations.

Figure 2 shows an example of an integrated process for the transesterification of methyl acetate (MeOAc) with n-butanol (BuOH) as shown by Steinigeweg and Gmehling [2004][2]. The process consists of one reactive distillation column, one pervaporation unit and one conventional distillation column for the final purification of the side product methanol (MeOH). The reactive distillation column consists of a reactive section equipped with the catalytic packing Katapak-S and two non-reactive sections above and below the reactive section. The higher boiling reactant BuOH is fed into the column above the reactive section, while the lower boiling reactant MeOAc is fed below the reactive section where the two components react towards the final products butyl acetate (BuOAc) and MeOH. An excess of MeOAc ensures a high conversion of BuOH and the product BuOAc is removed as a bottom product

[1] P. Kreis, A. Górak, Process Analysis of Hybrid Separation Processes - Combination of Distillation and Pervaporation, Chemical Engineering Research and Design, 84(A7), p. 595–600 (2006)

[2] see list of key publications (Table 6)

from the reactive distillation column. The distillate is a mixture of the side product MeOH and the non converted MeOAc which is fed to the pervaporation unit. The membrane is hydrophilic and therefore selective towards MeOH, which is removed from the feed and the resulting retentate with a low MeOH concentration can be recycled as a feed to the RD-column. The MeOH rich permeate is purified in a conventional distillation column and the side product MeOH is withdrawn as a bottom product while the distillate is recycled to the pervaporation unit. It can clearly be seen that the process layout design follows the same rules as for membrane-assisted conventional distillation since the hybrid process of reactive distillation and pervaporation is analogous to option 1 in Figure 1, which is recommended for azeotropic mixtures with a considerable concentration of the permeating component.

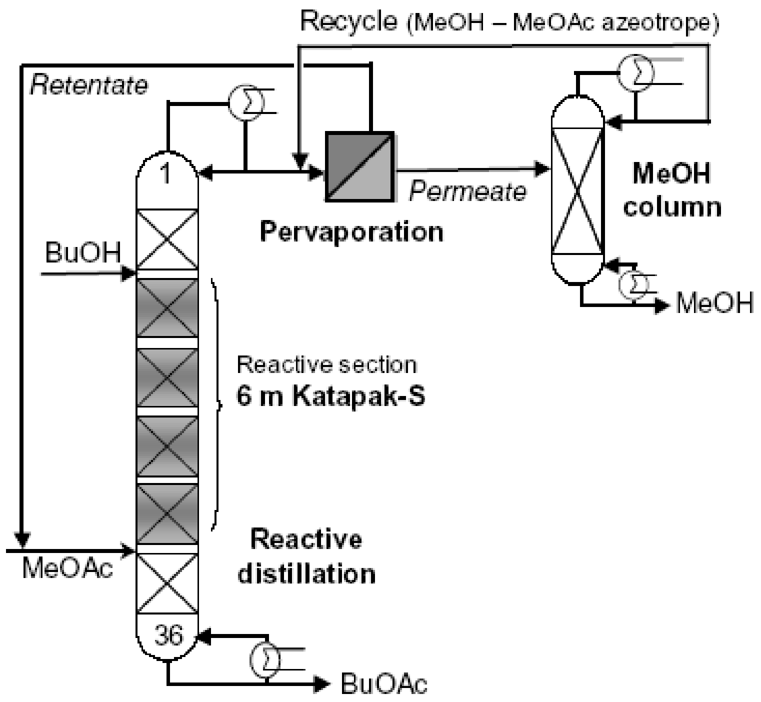

Figure 2: Flowsheet of a combined process for the transesterification of methyl acetate with n-butanol

1.2 Types and "versions"

(Describe the most important forms/versions of technology under consideration, including their characteristic features, differences and similarities)

Since membrane-assisted reactive distillation is a hybrid process that combines three different functionalities i.e. reaction, distillation and membrane separation, a large variety of process configurations are conceivable. Besides the distinction of the two different membrane processes pervaporation and vapour permeation, substantial differences can be found in the degree of integration. Hybrid processes do not require the integration of the different functionalities in one single unit but can also make use of synergetic effects by an intelligent interconnection of individual units[3] the definition of membrane-assisted reactive distillation can be broadened towards combinations of the three functionalities reaction, distillation and membrane separation with different degrees of integration.

[3] A. Górak, M. Franke, P. Kreis, J. Strube, W. Baecker, G. Olf, E. Dikow, Hybride Trenn-prozesse - Forschungsbedarf oder industriell gelöst?, GVC-Jahrestagung Karlsruhe (2004)

Even though not containing a reactive distillation unit in a strict sense, the TRIM™ process[4] shown in Figure 3 combines all three functionalities to create benefits for the production of methyl tert-butyl ether (MTBE) from a mixed C4-stream. Two alternative layouts of the process show how a combination of an organophilic membrane in a pervaporation unit can be used to significantly enhance the process performance. In the first layout the PV unit is used to decrease the methanol concentration of the reactor effluent from about 5 to 2 wt%. The methanol-rich permeate is recycled to the reactor and improves the MTBE conversion by 5% per cycle. In second layout the PV unit is used to remove methanol from a side stream of the distillation column and recycle it to the reactor. This process layout leads to an increased performance of the column by reducing the methanol concentration in the top product. The costs of the methanol recovery can thus be decreased by minimising or even eliminating the need for the methanol recovery unit. In comparison with a high conversion plant the side stream approach can save up to 20% on investment costs.

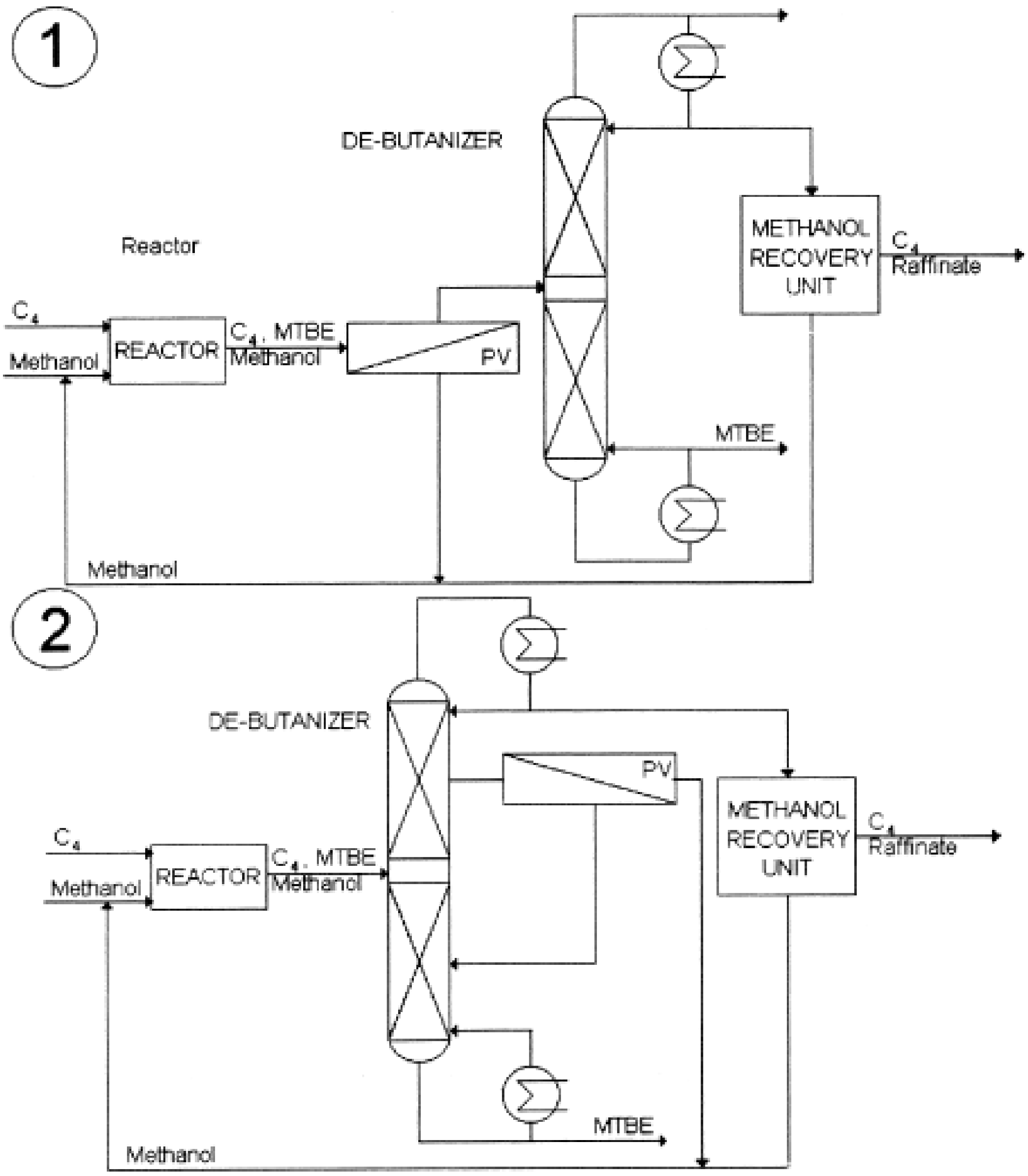

Figure 3: Process layouts of the TRIM™ process integrated in MTBE production[5]

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4 see list of key patens (Table 7)

5 see list of key publications (Table 6)

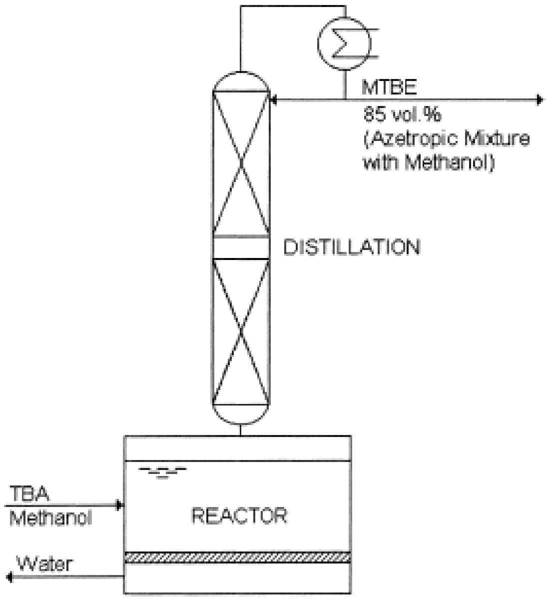

Figure 4 Hybrid process combining a membrane reactor with reactive distillation[5]

Another option to combine the three functionalities for the production of MTBE is presented by Mutouq et al. [1994][6]. MTBE is produced from tert-butyl alcohol (TBA) and methanol in a membrane reactor with a distillation column on top (see Figure 4). Since the etherification of two alcohols involves the production of water as a side product, the conversion in the reactor can significantly be enhanced by removing it from the reaction zone. This is done by using a hydrophilic PVA membrane (Mitsui Engineering and Shipbuilding) in pervaporation mode which is connected to a stirred reactor with suspended catalyst. Batch experiments on lab-scale showed no significant improvement in comparison to a conventional reactive distillation process, but it was concluded that the proposed hybrid process might be effective.

A similar apparatus was used by Yang and Goto [1997][6] for the production of ethyl tert-butyl ether from TBA and ethanol. In order to realise larger membrane areas, the pervaporation unit was operated in a pump around from the reactor. Batch experiments with and without pervaporation are compared and removing water from the reactor results in a clear improvement in conversion.

Bart and Reisl [1997][6] propose a combination of two reactive distillation columns and one pervaporation unit for the purification of an industrial mixture from the production of carboxylic esters. The pervaporation unit is connected to the bottom product of the first column to remove methanol and water and thus shift the composition across the distillation boundary. A second reactive distillation column is used for the final purification of the carboxylic ester which is withdrawn as distillate. The big advantage in comparison to a final purification with a pervaporation unit lies in the low membrane area, since it only has to produce a small change in composition to reach a different distillation region.

Information on an industrial scale membrane-assisted reactive distillation plant for the production of trimethylborate (TMB) is given by Maus and Brüschke [2002][6] (see Figure 5). In an upstream reactor methanol is reacted with boric acid to trimethylborate (TMB). The reaction is completed in a reactive distillation column, and the side product water with traces of non-reacted acid is withdrawn from the bottom of the column. At the top of this column an azeotropic mixture of methanol and TMB is obtained. Removing methanol by washing the mixture with water is not possible since this ester is very sensitive against hydrolysis. Therefore, the azeotropic mixture is fed at a pressure of about 2.5 bar to a vapour permeation system where the methanol content is reduced from about 30% to a concentration of 3–5%. After expansion this vapour is led into the second distillation column at the bottom of which the pure ester is obtained. The methanol permeated through the membrane of the vapour permeation unit is condensed and recycled to the reactor as well as the nearly azeotropic top product of the purification column. The plant comprises about 300 m² of membrane area, installed in plate modules, which are housed in two vacuum vessels.

[6] see list of key publications (Table 6)

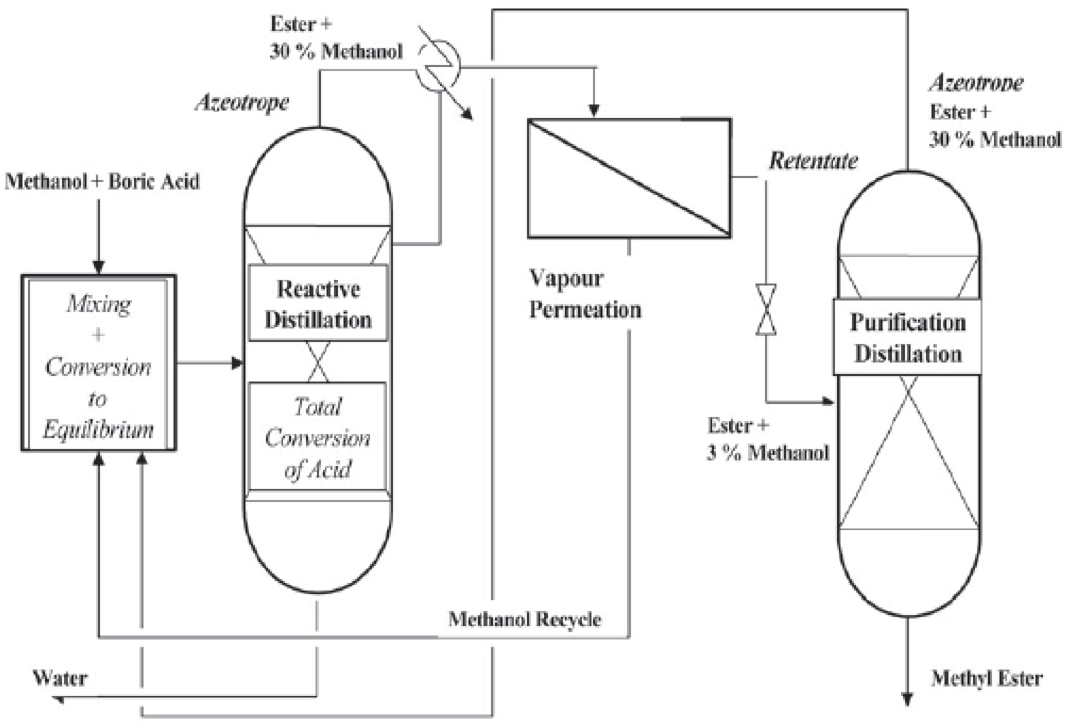

Figure 5: Hybrid process for trimethylborate production

Lab-scale experiments for the production of tert-amyl ethyl ether (TAEE) by an esterification of tert-amyl alcohol with ethanol have been carried out by Aiouache and Goto [2003][7]. They have combined all three functionalities in a small reactive distillation column with a height of only 50 cm and a diameter of 5 cm by inserting a zeolithe tube membrane in the middle. Even though the apparatus allows to remove water from all sections of the column and thus to eliminate its inhibitory effect on the reaction rate, it will be difficult to implement this complex concept in an industrial scale process.

A second hybrid process that has been scaled up to industrial application is presented by von Scala et al. [2005][7]. The process is used for the continuous esterfication of fatty acids for use in the cosmetic industry. The process layout for an esterification of myristic acid (MA) with isopropyl alcohol (IPA) is shown in Figure 6. The process mainly consists of one pre-reactor (R-1), one reactive distillation column (C-1), a pervaporation unit (M-1) and a conventional distillation column (C-4). The two reactants are continuously fed to the prereactor where they react to the equilibrium conversion and the reactor effluent is then fed to the reactive distillation column which enables a higher conversion due to the removal of products from the reaction zone. Water is removed from the distillate by pervaporation and the dry retentate consisting of IPA and the side-product diisopropyl ether (DIPE) is recycled to the pre-reactor. IPA is removed from the bottom product with a flash (C-2) unit and the remaining stream of ester and fatty acid is purified to the desired specifications in the distillation column. The fatty acid can be recycled to the reaction step. The new hybrid process was developed within two years and prodcution was started in April 2003.

[7] see list of key publications (Table 6)

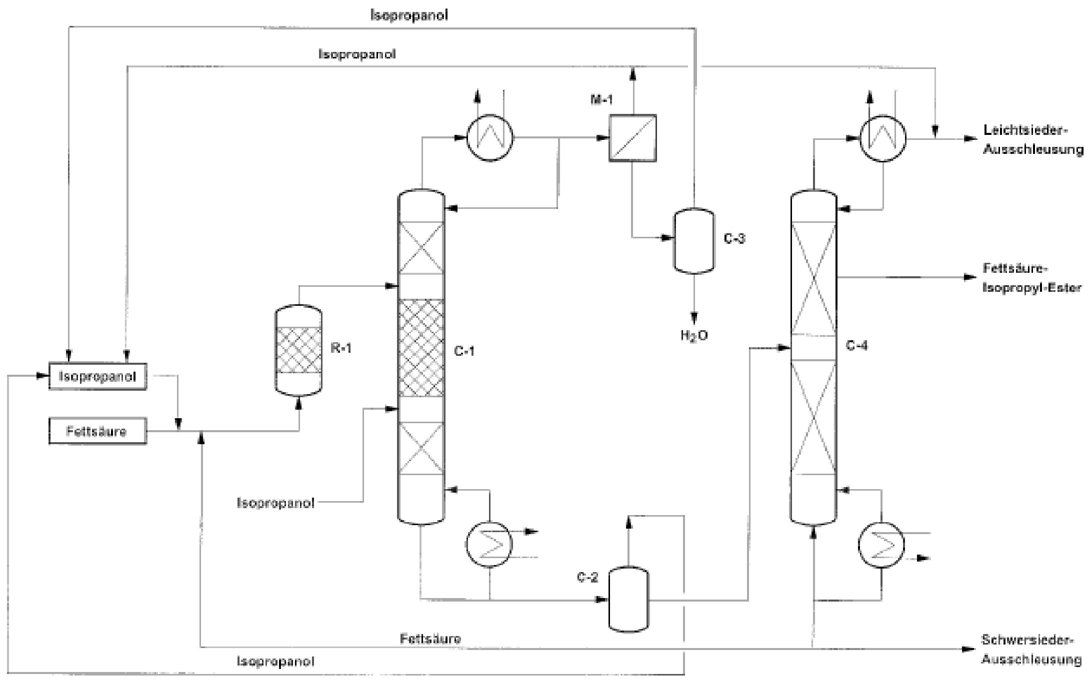

Figure 6: Hybrid process for the esterification of fatty acids

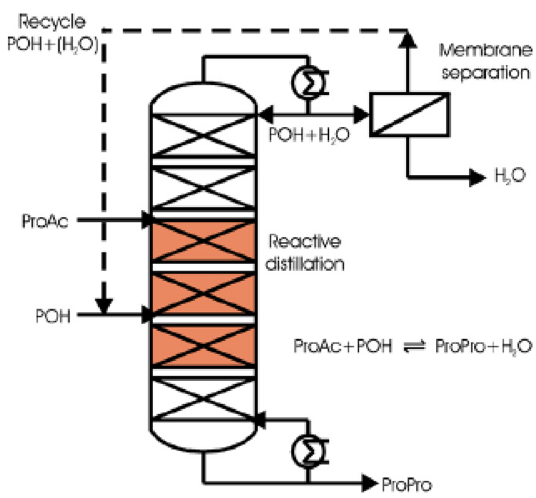

Figure 7 Hybrid process for the production of propyl propionate

A relatively simple process for the production of production of propyl propionate has been presented recently by Buchaly et al. [2007][8]. The process shown in Figure 7 consists of a reactive distillation column where the two reactants propionic acid (ProAc) and 1-propanol (POH) are fed above and below the catalytic section. The ester is removed as a bottom product while the distillate is consists for POH and the side product water. This mixture is separated by pervaporation with a hydrophilic membrane to remove the water from the process and recycle the POH rich retentate to the reactive distillation column. So far both unit operations have been experimentally investigated to provide reliable model parameter and validate a state-of-the-art process model which has been used for a detailed process analysis. Experimental work on the combined process and a process optimisation with an evolutionary algorithm are announced.

[8] see list of key publications (Table 6)

1.3 Potency for Process Intensification: possible benefits

(In Table 1 describe the most important documented and expected benefits offered by the technology under consideration, focusing primarily on energy; CO_2 emission and costs, providing quantitative data, wherever possible. Add other benefits, if needed).

The potential benefits of membrane-assisted reactive distillation are resulting from the combination of a membrane unit and a reactive distillation column. In cases where this combination allows for applying reactive distillation to new processes, benefits listed for the use of reactive distillation in general can be attributed to this technology. In other cases, the use of a membrane separation unit leads to further process improvements which can be compared with the benefits expected from membrane assisted conventional distillation.

Table 1 contains both aspects documented benefits for industrial scale applications (italic letters) and potential benefits resulting from the integration of a membrane separation unit in a (reactive or non-reactive) distillation process.

Table 1: Documented and expected benefits resulting from technology application

Benefit	**Magnitude**	**Remarks**
Energy saving	The potential for energy savings is significant (>50%) but largely depends on the process and the performance of the membrane unit	Energy savings are mainly effected by the selective removal of a side component that can lead to ▪ an improved performance of the reactive distillation column at a lower reflux ratio ▪ a higher selecitvity and conversion causes a reduced effort for final purification ▪ a reduced number of energy intensive separation steps *The energy demand for the esterfication of fatty acids was cut in half in comparison to a conventional batch process.*
Reduced CO_2 emissions	No data available	See remarks concerning energy savings above.
Cost savings	up to 25%	Significant cost savings can be reached by reduced investment and operation costs. Savings have to be balanced with additional costs for the regular replacement of membranes. *Two distillation columns were replaced by a pervaporation unit for the separation of isopropyl alcohol and water in the esterification of fatty acids.*
Improved sustainability	No data available	*Waste streams were significantly reduced by the use of a heterogeneous catalyst for the esterification of fatty acids since catalyst neu-tralisation, separation and recycle is avoided*
Catalyst savings	No data available	Longer catalyst life-time can be expected due to the removal of inhibitory components from the reaction zone
Better product quality	No data available	The selective removal of components can be used to prevent undesired side-reactions and thus to improve the product quality. *Traces of LiCl in the ester fraction which are present in the conventional TMB process are avoided by the hybrid process and a high purity product is obtained.*

1.4 Stage of development

Membrane-assisted reactive distillation can be classified as an available technology since it combines of two known technologies that have already made their way into commercial application. However, technical limitations are mainly caused by the availability of membrane materials with a special selectivity towards organic compounds and reasonable flux at moderate operating conditions. Therefore, existing applications are so far restricted to the separation of water and methanol as a small and polar molecule that can also be separated by hydrophilic membranes.

In addition the complex interactions which occur in the hybrid process require suitable tools for process modelling and simulation that allow for exploiting the full potential of the new technology.

2. Applications

2.1 Existing technology (currently used)

(Describe technology (-ies) that are conventionally used to perform the same or similar operations as the PI-technology under consideration)

State-of-the-art technology which can be replaced by membrane-assisted reactive distillation cannot clearly be specified since the new technology can be used to replace different process steps and unit operations. However, the use of membrane units is considered as beneficial for the selective removal of components which meet one or more of the following criteria:

- have a relatively low fraction in the feed stream if removed as permeate
- are difficult to remove by conventional techniques or require an extensive use of energy
- exhibit a significant difference in molecule size, structure (e.g. alkane vs. alkene) or component behaviour (e.g. hydrophilic vs. hydrophobic) in comparison to other components in the feed stream

For this reason, membrane separation is a favourable technique to remove minor components from the process and especially to replace complex and often energy intensive technologies for the separation of azeotropic mixtures like azeotropic distillation or pressure swing adsorption.

2.2 Known commercial applications

(Is the technology broadly applied on commercial scale? In which process industry sectors is the technology most often applied: large volume chemicals – specialty chemicals & pharma – consumer products – ingredients based on agro feedstocks? What is the estimated number of existing applications? In Table 2 provide the most prominent examples of realized applications and provide their short characteristics)

So far, only two commercial application of membrane-assisted reactive distillation are know and reported in the table below.

Table 2. Industrial-scale applications of the Technology (existing and under realization)

Sector	Company - Process/Product name/type	Short characteristic of application	Production capacity /Plant size	Year of application	Reported effects
Not known	Not known	Production of tri methyl borate	Not known	1997	• Savings in investment of 13% • Energy savings of 120,000 €/year • Operating costs reduced by 111,000 €/year • improved product quality by avoidance of LiCl traces in product
Cosmetic industry, Malaysia	CONFIDENTIAL (Process and Equipment design from Sulzer Chemtech AG)	Production of fatty acid esters	100 – 1,000 t/year	2002	• energy savings of 45% • improved product quality due to continuous operation • avoidance of rotating equipment

2.3 Known demonstration projects

(Are there any demonstration projects known related to the technology under consideration? In which process industry sectors are those projects carried out: large volume chemicals – specialty chemicals & pharma – consumer products – ingredients based on agro feedstocks? In Table 3 provide the short characteristics of those projects.)

No demonstration projects on membrane-assisted reactive distillation are known.

2.4 Potential applications discussed in literature

(Provide a short review, including, wherever possible, the types/examples of products that can be manufactured with this technology)

Even though the literature on membrane-assisted reactive distillation is unelaborate, new applications can be foreseen in dependence on the availability of suitable membrane materials for the selective removal of individual components from the reactive distillation unit. For indications on favourable process conditions for the use of membrane separation please refer to section 2.1.

3. What are the development and application issues?

3.1 Technology development issues

(In Table 4 list and characterize the essential development issues, both technical and non-technical, of the technology under consideration. Pay also attention to “boundary” issues, such as instrumentation and control equipment, models, etc.) Also, provide your opinion on how and by whom these issues should be addressed)

Technology development issues can only be listed separately for the two process units membrane separation and reactive distillation.

Table 4. Technology development issues

Issue	Description	How and by whom should be addressed?
Membrane separation units	Development of new membrane materials which can be tailored to the process requirements in terms of: • Selectivity towards specific components • High flux at favourable operating conditions • Long lifetime at process conditions	Universities and research institutes in collaboration with equipment manufacturers
	Improved simulation strategies for membrane separation units that provide a high accuracy for a wide range of operating conditions with a reduced experimental effort for membrane characterisation	Universities and research institutes
	Development of improved module concepts that facilitate an easy installation and maintenance of the membrane separation unit and avoid leakage currents between feed and permeate side	Equipment manufacturers
Industrial scale reactive distillation units	Development of new column internals which facilitate an easy or even continuous exchange of exhausted catalyst	Universities and research institutes in collaboration with equipment manufacturers
	Establishment of reliable methods for the scale-up of reactive distillation columns with catalytic packings	Universities and research institutes in collaboration with end users

3.2 Challenges in developing processes based on the technology

(In Table 5 list and characterize the essential challenges, both technical and non-technical, in developing commercial processes based on the technology under consideration. Also, provide your opinion on how and by whom these challenges should be addressed)

Challenges in developing new membrane-assisted reactive distillation processes are mainly caused by the strong interactions of both process units and the variety of possible combinations. The development of new membrane materials with high stability, solvent resistance and long lifetime is another issue that requires close collaboration with material scientists.

Table 5. Challenges in developing processes based on the technology

Challenge	Description	How and by whom should the challenge be addressed?
Design of optimised processes	The performance of the hybrid process depends on an optimum process design and optimised operating conditions for the three steps reaction, distillation and membrane separation. Thus, the operating window for the complete process is limited an the optimum of the process is not in agreement with the optimum operating conditions for the single units	Powerful simulation and optimisation tools have to be developed by universities, R&D departments and software providers
Process control	The complex interactions require a sophisticated process control strategy that considers the mutual influence of the different units	Development of model-based process control strategies for complex hybrid processes by universities and process control departments
Start-up strategies	Side components which are removed by the membrane separation unit can lead to a significant variation of the reaction rate for main and side reactions and thus have a very strong influence on the column profile	Suitable start-up strategies for hybrid processes have to be developed by universities and R&D departments

4. Where can information be found?

4.1 Key publications

(Provide the list of key publications in Table 6)

Table 6. Key publications on the technology

Publication	Publication type (research paper/review/book/ report)	Remarks
M. Matouq, T. Tagawa, S. Goto, Combined process for production of methyl tert-butyl ether from tert-butyl alcohol and methanol, J. Chem. Eng. Japan. 27, p. 302-306 (1994)	Research paper	Batch reactor with distillation column on top and membrane module in pump around
H.-J. Bart, H.-O. Reisl, Der Hybridprozess Reaktivdestillation / Pervaporation zur Herstellung von Carbonsäureestern, Chemie Ingenieur Technik 69(6), p.824-827 (1997)	Research paper (German)	Process design and simulation studies for a combination of two RD-columns and one pervaporation unit
B.-L. Yang, S. Goto, Pervaporation with Reactive Distillation of the Production of Ethyl tert-Butyl Ether, Separation Science and Technology, 32(5), p. 971-981 (1997)	Research paper	Batch reactor with distillation column on top and membrane module in pump around
F. Lipnizki, R. F. Field, P.-K. Ten, Pervaporation-based hybrid process: a review of process design, applications and economics, Journal of Membrane Science 153, p. 183-210 (1999)	Review	Review on different combinations of reaction, distillation, and membrane separation

Publication	Publication type (research paper/review/book/ report)	Remarks
E. Maus, H.E.A. Brüschke, Separation of methanol from methylesters by vapour per-meation: experiences of industrial applications, Desalination 148, p. 315–319 (2002)	Review on process examples	Industrial scale process for trimethylborate production.
F. Aiouache, S. Goto, Reactive distillation–pervaporation hybrid column for tert-amyl alcohol etherification with ethanol, Chemical Engineering Science 58, p. 2465-2477 (2003)	Research paper	Process design, simulation and experiments with lab scale equipment
S. Steinigeweg, J. Gmehling, Transesterification processes by combination of reactive distillation and pervaporation, Chemical Engineering and Processing 43, p. 447-456 (2004)	Research paper	Separate experiments for RD and pervaporation. Simulation studies for the hybrid process
C. von Scala, P. Fässler, J. Gerla, E. Maus, Kontinuierliche Herstellung von kosmetischen Fettsäureestern mittels Reaktivdestillation und Pervaporation, Chemie Ingenieur Technik 77(11), p. 1809-1813 (2005)	Review on process example (German)	Description of process layout and design procedure
C. Buchaly, P. Kreis, A. Górak, Hybrid separation processes - Combination of reactive distillation with membrane separation, Chemical Engineering and Processing (46), p. 790-799 (2007)	Research paper	Separate experiments for RD and vapour permeation, Simulation studies for the hybrid process

4.2 Relevant patents and patent holders

(Provide the list of relevant patents in Table 7. Under "remarks" provide, where applicable, the names/types of products targeted by the given patent.)

Table 7. Relevant patents

Patent	Patent holder	Remarks, including names/types of products targeted by the patent
US 4,774,365 (1988) Pervaporation Process for Separation Alcohols from Esters	Air Products and Chemicals Inc.	TRIM™ process
EP 1 424 115 (2002) Verfahren zum Verestern einer Fettsäure	Sulzer Chemtech AG	membrane separation is only mentioned as an option to remove water from the process
US 7,091,367 Method for the Esterification of a Fatty Acid	Sulzer Chemtech AG	Same invention aspects covered as in EP 1 424 115

4.3 Institutes/companies working on the technology

(Provide the list of most important research centers and companies in Table 8)

While research institutes working on reactive distillation and/or membrane separation are numerous, the investigation of the membrane-assisted reactive distillation is limited to a few institutions listed in Table 8.

Table 8. Institutes and companies working on the technology

Institute/Company	Country	Remarks
Carl von Ossietzky Universität Oldenburg (Prof. Jürgen Gmehling)	Germany	Design and experimental evaluation of membrane-assisted reactive distillation processes
Universität Dortmund (Prof. Andrzej Górak)	Germany	Design, modelling and experimental evaluation of membrane-assisted reactive distillation processes
Sulzer Chemtech AG	Switzerland	Development of industrial scale processes and dedicated process equipment

5. Stakeholders

5.1 Suppliers and developers

(Provide the list of key suppliers/developers in Table 9)

The only know commercial developer and provider of membrane-assisted reactive distillation technology is Sulzer Chemtech AG but processes can also be designed on the basis of technologies from different key suppliers. A selection is therefore listed in table 9.

Table 9. Supplier and developers

Institute/Company	Country	Remarks
Bayer Technology Services GmbH	Germany	Development of reactive distillation and membrane processes
CDTECH, Houston, Texas	US	Development of reactive distillation processes
Dow Haltermann Custom Processing	US	Process development and custom manufacturing for reactive distillation
GKSS Forschungszentrum	Germany	Membrane development
Julius Montz GmbH	Germany	Hold-up trays for long residence time in a reactive distillation
Mitsui & Co. Ltd.	Japan	Design and equipment for membrane separation processes

Institute/Company	Country	Remarks
Sulzer Chemtech AG	Switzerland	Design and equipment for membrane-assisted reactive distillation processes
Whitefox Technologies Ltd.	UK	Development of membranes and membrane processes

5.2 End users

(Describe the existing and potential end-users, other than those already listed in Table 2)

Since the use of membrane-assisted reactive distillation is not limited to a special class of products, the potential end users cannot be specified. The area of application includes the following industrial sectors:

- Bulk chemicals
- Fine chemicals
- Cosmetics
- Consumer care
- Alternative fuels

6. Expert's brief final judgment on the technology

(maximum 5 sentences)

Membrane-assisted reactive distillation can be considered as a developed technology since it consists of two units that are already applied on a commercial scale. The selective removal of components from the reactive distillation column offers a significant potential for process intensification by improving the reaction conditions or simplify the separation of complex mixtures. However, the availability of efficient membrane materials has to be improved to extend the potential of this technology to new applications. The integration of a pervaporation or vapour permeation unit requires a detailed process analysis and optimisation to find the best process configuration and tackle the full potential of the technology. Even though large saving in terms of energy demand or equipment costs have already been shown, membrane-assisted reactive distillation is expected to remain a niche application until membrane separation achieves a real breakthrough in terms of versatility, efficiency and durability.