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Chemical Engineering Journal 135 (2008) 95–102

Chemical Engineering Journal

www.elsevier.com/locate/cej

Renewable energy source—Dehydrated ethanol

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Received 23 August 2006; accepted 7 March 2007

Abstract

Ethanol produced from renewable energy sources—biomass, is the most promising future biofuel. In the article, a pervaporation membrane technique for ethanol dehydration is presented. Experimental results and semi-empirical and empirical models calculated on their basis are discussed. The data determined by mathematical models and experimental calculations are compared.

Keywords: Biofuel; Ethanol dehydration methods; Economic aspects of dehydration; Pervaporation

1. Introduction

Main objectives of the present ecological policy include reduction of energy consumption of the economy and development of branches related to renewable energy sources which include energy produced from biomass, wind, geothermal, water and solar energy. Renewable energy sources enable both improvement of environmental protection and are an important element of sustainable development. According to the experts programs of the World Committee of Energy Council, it is predicted that in 2070 the contribution of renewable energy to the total world energy balance will be about 60%. This results mainly from depletion of the traditional energy sources and general availability of unconventional energy, as well as improving environmental protection due to reduction of the emissions of carbon, sulphur and nitrogen oxides and waste minimisation [1–5].

Due to high-energy values, ethanol is the most promising future biofuel. At present it is used in fuel industry as an additive to petrol that heightens its octane number and combustibility. A small addition of ethanol to petrol significantly increases the octane number of the mixture and at 10 wt.% content the present engine structure need not be modified. Addition of ethanol to fuel means that combustion is more efficient and emission of exhaust gases is reduced. In view of the development of recoverable fuel production and ecological aspects, according to the EU recommendations, ethanol will be produced and subsidised in the period of the nearest several years [6–9].

The only barrier for a broad application of production capacities of ethanol and installations for composing petrol on the basis of ethanol is a high production cost. Despite this limitation, the market of ethanol used for fuel production increases on a global scale and in 2005 it reached ca. $3 \times 10^{10}\ \mathrm{dm}^3$. The leading world producers of ethanol are the countries of Americas, next Asia, Europe, Africa and Oceania. Beside fuel industry, the other recipients of dehydrated ethanol include food industry (mainly alcohol distilleries), pharmaceutical, paint and varnish industry, research laboratories, etc.

1.1. Methods of ethanol dehydration

In the days of world energy crisis, beside big stress on energy savings and searching for new methods of energy production, mainly from renewable sources, research is also carried out on improvement of the existing systems. Hence, a further development of traditional methods is observed and methods of reducing the costs of dehydrated ethanol production by traditional techniques are sought.

At present, ethanol can be obtained during ethanol fermentation that is much more frequently applied in industry. Raw materials in the ethanol fermentation can be all renewable products that contain simple sugars and polysaccharides after relevant technological processing. As a result of fermentation and rectification, the obtained product has about 95 vol.% ethanol. Production of anhydrous ethanol requires overcoming the barriers of a positive homoazeotrope. Now,

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doi:10.1016/j.cej.2007.03.017

Nomenclature

c	concentration (wt.%)
exp.	experimental data
J_V	permeation flux (kg/m^2 h)
mod.	model data
p	pressure (mbar)
T	temperature (°C)
u	flow rate (dm^3/h)
Greek letters	
α	selectivity coefficient
β	enrichment coefficient

the most important ethanol dehydration techniques used in the world industry include azeotropic distillation, dehydration on molecular sieves and pervaporation or vapour permeation [7,10,11].

Azeotropic distillation used in ethanol dehydration consists in adding a third component [12] which is directly connected with higher energy consumption, demand for more space for the installation, complicated process control, increased labour consumption of the ordering operations that minimise losses of the azeotropic agent and environmental pollution.

The process of dehydration on molecular sieves can adsorb much water or other compounds and remove liquid and gaseous pollutants to very low concentrations (ppm or less). For ethanol dehydration on industrial scale, potassium and sodium zeolites are used [13,14]. Ethanol dehydration on molecular sieves is connected with cyclic work in changeable conditions, consumption of big amounts of vapour under high pressure and recycling big quantities of ethanol to the process.

It seems that an alternative to the discussed traditional methods of ethanol dehydration is pervaporation (PV). PV is a new generation of membrane separation techniques. During pervaporation, a liquid stream is separated on a semi-permeable membrane (for ethanol dehydration—hydrophilic laminated membranes from PVAL) into two streams: a gaseous permeate and liquid retentate. A permeate is enriched with a component transmitted preferentially by the membrane opposite to the retentate which has a small amount of this component. Since separation on the membrane does not much depend on liquid-vapour equilibrium (in the pervaporation a transport of water is usually preferred), the process can be used efficiently for separating azeotropes and near-boiling liquids [15].

The first and presently the broadest application of the pervaporation in the world industry is dehydration of organic solvents, including alcohols, multi-functional systems, esters, ethers and mixtures of solvents [16].

1.2. *Economic aspects of ethanol dehydration methods*

When analysing the literature on the subject, it is possible to identify concrete trends in the development of pervaporation in ethanol dehydration industry. The PV installation can be an independent, final stage of dehydration (in order to overcome the azeotropic point), a direct stage after fermentation process (to concentrate the ethanol below the azeotropic concentration) or an element of a hybrid solution combined with the presently used techniques (azeotropic distillation and dehydration on molecular sieves).

A comparison of cost for small installations (100 dm^3/day) of ethanol dehydration to 99.5 wt.% by different methods such as PV, distillation and adsorption shows PV technique as the most advantageous [17]. The cost of ethanol dehydration by distillation is twice as high, and in the case of adsorption 1.5 times higher than in PV. It enables cost reduction on the average by 1 UScent/dm^3 of the produced ethanol. Additionally, due to the modular nature of the process, costs related to pervaporation are not so sensitive to scaling up as in the case of distillation or adsorption. For PV systems operating above 100 dm^3/h the product costs grow linearly with capacity. A comparison of the costs of ethanol dehydration by various techniques in a bigger system (Table 1) indicates that operating costs of the membrane techniques are smaller by half than another dehydrating methods. The system of capacity was 30 tonnes/day $\approx$ 1580 dm^3/h. Costs were estimated in US$ per tonne of dehydrated ethanol (99.8 wt.%) [18,19]. Distillation becomes more cost-effective than pervaporation only just at the capacity of 5000 dm^3/h [20].

Pervaporation is economically justified when at the inlet water concentration in the system is less than 10 wt.% and when at the outlet we expect dehydration of the order of 100–10 ppm of its content. If still higher product dehydration is expected, then much bigger membrane surface and higher pressure reduction on the side of permeate is required. Cost of ethanol dehydration decreases with an increase of permeation flux and mass fraction of ethanol in permeate and grows with an increase of membrane cost.

Pervaporation can be applied in continuous fermentation that is related to the concentration of ethanol below the azeotropic concentration. For such a system dehydrating 21,600 dm^3/h ethanol [21], investment and operating costs become more competitive than the traditional fermentation with distillation above 0.3 kg/m^2 h. This can be obtained by using more efficient membranes or by increasing feed temperature. In the future, we should tend to develop more selective membranes and decrease the production cost. The latter constitutes 20% of the pervaporation cost (8% operating costs of the whole system) and follows from a relatively small market [22].

In literature there are many examples of hybrid processes of pervaporation with distillation [18,20,23]. Such hybrid processes enable savings of operating costs but not always of investment outlays. Lower operating costs result mainly from a lower energy demand and not from the use of additives. On the other hand, high investment outlays are a result of process complexity and high membrane prices. The development of hybrid processes of distillation-pervaporation and broad applications in industry will depend not only on high process efficiency but first of all on reduction of the membrane cost. The hybrid systems will bring about economic advantages at long-term processes but they are not profitable in the case of small ethanol dehydration systems.

Table 1
Comparison of the cost of ethanol dehydration (94 wt.%) by various techniques

Operating costs	Vapour permeation	Pervaporation	Azeotropic distillation (cyclohexane)	Adsorption on molecular sieves
Vapour pressure reduction	–	3.2	25–37.5	20
Water cooling	1	1	3.75	2.5
Electric energy	10	4.4	2	1.3
Distillation component	–	–	1.2–2.4	–
Exchange of membranes or sieves	4.75	4–8	–	12.5
Total cost	15.75	12.6–16.6	31.95–45.65	36.3

Besides building of new ethanol dehydration systems based on hybrid processes of distillation-pervaporation, producers offer also implementation of the pervaporation in the already existing installations [24]. The main aim of such a procedure is de-bottlenecking of processes and effective cost reduction. So, if a PV module is placed between the distillation and azeotropic column, this will cause reduction of the reflux in the distillation column (an increase of feed) and partial dehydration of ethanol entering the column with the azeotropic agent. Advantages of such a hybrid solution will be a double increase of efficiency and related reduction of energy cost, dehydration costs (ca. 50%), more efficient use of the existing system and a possibility to control the PV module.

Similarly, the PV module can be connected to the already existing dehydration on molecular sieves. Addition of the PV unit before the sieves will cause an increase of process efficiency and the quantity and quality of water removed, reduction of product recirculation degree and energy consumption.

Just this last application that consists in placing PV between distillation and adsorption on molecular sieves, can bring in the future the biggest economic benefits in the process of ethanol dehydration.

The authors of the article carry out research on ethanol dehydration by pervaporation [25]. They present experimental results and propose on this basis a semi-empirical and empirical description. Then data determined by mathematical models and experimental data are compared.

2. Experimental

The aim of study was to determine the effect of process variables such as flow rate, alcohol concentration, inlet stream temperature and pressure on the low-pressure side of the membrane, on transport and separation properties of the tested membrane. Additionally, a mathematical description of the process was proposed using a quick PV analysis of process and economic aspects.

2.1. Materials

PERVAP 2210 is a commercially available hydrophilic membrane provided by Sulzer Chemtech. It is used to dehydrate organic solvents such as ethanol or isopropanol.

The membrane of active surface 0.0177 m^2, was placed in a flat module. A binary mixture of 80–95 wt.% ethanol was prepared from pure industrial ethyl alcohol supplied by an industrial plant. Liquid fed to the module was thermostated at the selected temperature. Due to a small membrane surface, temperature drop on the module was neglected.

2.2. Pervaporation experiments

Fig. 1 shows a schematic diagram of the pervaporation equipment. From a feed tank 2 dm^3 in volume, the mixture was directed to the membrane module, the liquid washed the membrane surface and returned to the tank.

Flow rate was controlled by a flow meter. A maximum flow rate was 100 dm^3/h.

Under the module there was a permeate tank immersed in liquid nitrogen. This system was connected to a vacuum pump to achieve the desired reduction in the pressure. Permeate was collected in the cold trap at determined time intervals (1 h), then it was thawed and weighed. Based on this and alcohol concentration in the permeate a permeation flux was calculated. The feed and permeate compositions were analysed by gas chromatography. A Thermo Finnigan chromatograph was equipped with a FID detector and 30 m Quadrex Corporation column, model BTR-CW-30V-1.0F 0.53 mm and 1.0 μm thick.

3. Results and discussion

The permeation flux J_V and separation coefficient β are two vital parameters that determine transport and separation abilities of a membrane. In the study, the effect of operating parameters: feed concentration, temperature, flow rate and pressure changes on J_V and β was analysed.

The process of pervaporation was carried out according to a three-level factorial design for the temperatures 65, 70 and 75 °C, ethanol concentrations in the feed 90, 92.5 and 95 wt.% and three flow rates: 20, 40 and 60 dm^3/h.

To compare the effect of pressure on process efficiency, experiments were performed for pressure values 3 and 30 mbar on the low-pressure side of the membrane. The process was carried out in steady-state conditions.

Experimental results were used to mathematically model the process. An empirical method was developed at Lodz Technical University using the Excel program and a semi-empirical method with the use of commercial Aspen Customer Modeler. A detailed description of procedures assumed in the empirical model is given in papers [26,27] and a mathematical description of the semi-empirical model is provided in Ref. [27].

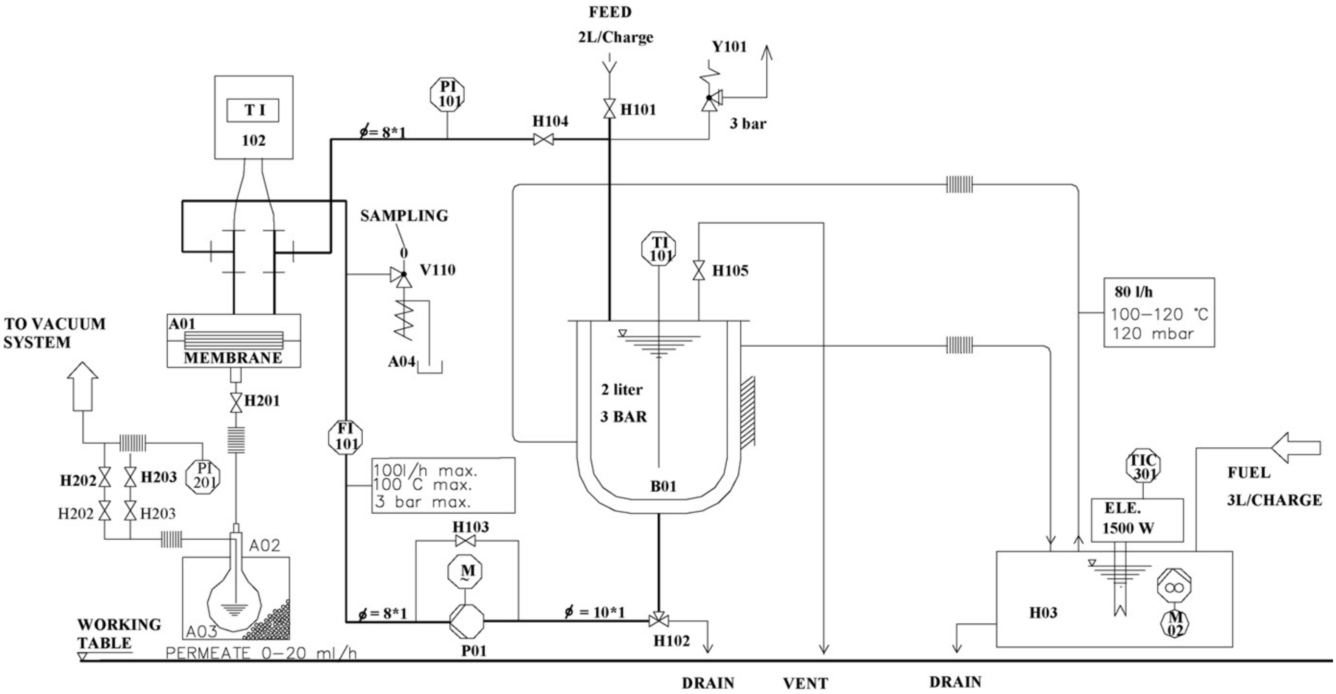

Fig. 1. Schematic diagram of the pervaporation equipment.

Using the empirical model assumptions and experimental data, model coefficients were determined. Figs. 2 and 3 give a comparison of experimental data and calculations of the flux from the empirical model for the pressure 3 and 30 mbar, respectively.

Taking into account results shown in the above diagrams, it was found that the empirical model fairly well described the experimental data and could be used in further calculations (correlation coefficients were $R_3 = 0.90$ and $R_{30} = 0.92$).

Similar calculations were made for the semi-empirical model. It follows from a simulation that the model accurately describes the experimental data and results are comparable with results of the empirical model. The semi-empirical model enabled analysis of the effect of polarisation layer on PV process efficiency (Fig. 13). Such effects could not be analysed with the use of the empirical model.

In studies of the pervaporation process at $p = 3$ mbar, the permeation flux was in the range from 0.2 to 1.8 kg/m^2 h, while for $p = 30$ mbar from 0.15 to 0.37 kg/m^2 h. Hence, it follows that lower pressure at the permeate side given bigger flux, and as a consequence higher efficiency. Fluxes obtained on the PERVAP 2210 membrane are comparable with the fluxes obtained for composite chitosan membranes [28]. Similar results were obtained for the PDMS membrane, where the flux was from 0.52 to 0.90 kg/m^2 h [29].

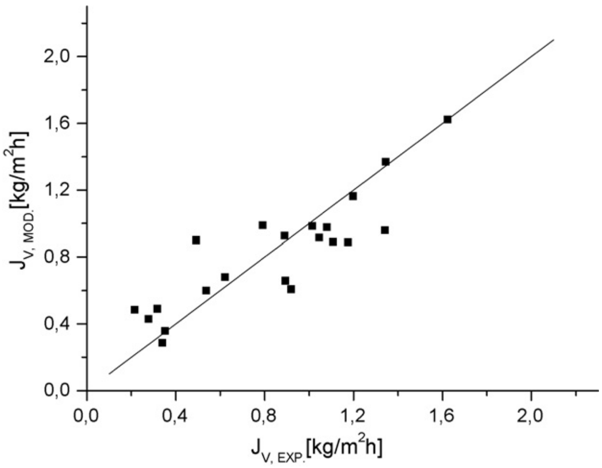

Fig. 2. Comparison of flux calculated from the model with experimental data for pressure $p = 3$ mbar.

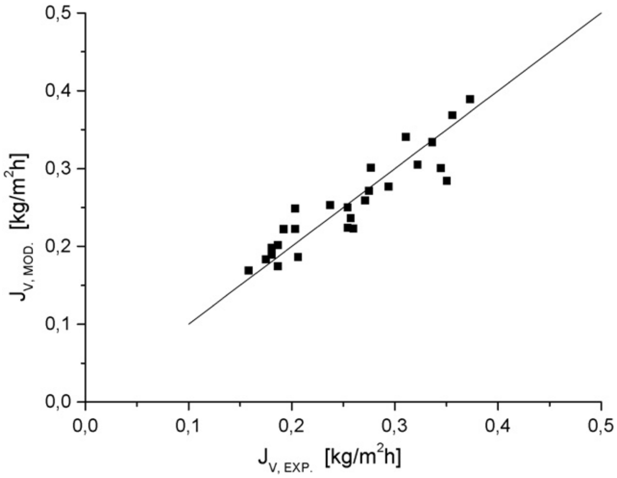

Fig. 3. Comparison of flux calculated from the model with experimental data for pressure $p = 30$ mbar.

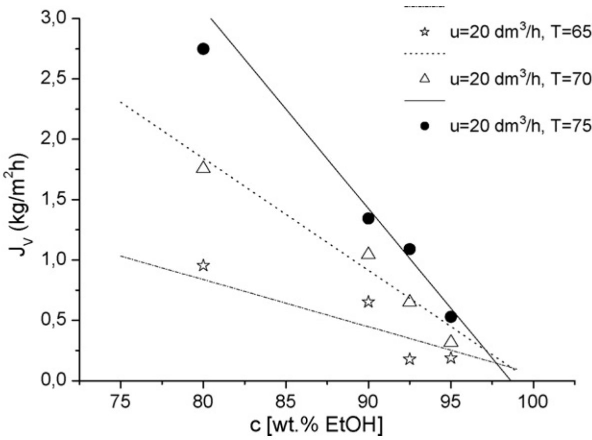

Fig. 4. Dependence of permeation flux on ethanol concentration in the feed for $p = 3$ mbar (line, model data; symbols, experimental data).

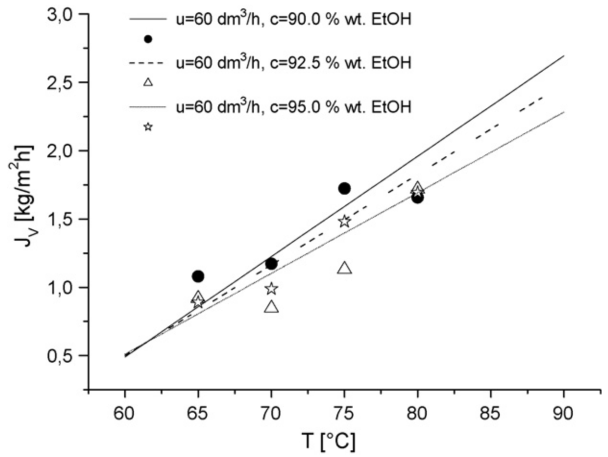

Fig. 6. Dependence of permeate flux on temperature at $p = 3$ mbar (line, model data; symbols, experimental data).

3.1. Effect of feed concentration

Figs. 4 and 5 show the effect of feed concentration on the permeation flux for two considered values of pressure. The smallest effect on the permeation flux is observed for high ethanol concentrations in the feed. For a concentration level of 95 wt.%, the fluxes determined at each of the three temperatures were almost the same. This can be a result of transition of the ethanol–water mixture through the azeotropic point.

3.2. Effect of temperature

Figs. 6 and 7 show the effect of temperature on the permeation flux for the PERVAP 2210 membrane. According to the experimental results, we observe that the flux increases with temperature increase. A higher mixture temperature has a favourable effect on transport through the membrane, just like lower pressure on vacuum side [30].

The permeation flux in the ethanol–water system for the tested membrane ranged from 0.8 kg/m^2 h at the temperature 65 °C to 1.7 kg/m^2 h at the temperature 80 °C. At pressure 30 mbar, the flux was from 0.18 to 0.45 kg/m^2 h, while for the 1060-SULZER membrane [31] these values ranged from 0.6 to 2.2 kg/m^2 h, and were comparable for the membrane at 3 mbar. Slightly smaller fluxes were obtained for the Celfa CMG-OM-010 membrane [31]: 0.3–1.5 kg/m^2 h.

3.3. Separation coefficient

On the basis of the proposed empirical and semi-empirical models we also studied the effect of process parameters on enrichment coefficient β defined as a ratio of mass fraction of a component transferred preferentially in the permeate—y_A to mass fraction of this component in the feed x_A.

In Figs. 8 and 9 the experimental points are grouped in three clusters depending on alcohol concentration in the feed. Coefficient β increases with alcohol content in the feed; and at $c = 90$ wt.% ethanol $\beta = 9.0$–9.8, at $c = 92.5$ wt.% $\beta = 12.0$–12.9, while at $c = 95$ wt.% enrichment coefficient $\beta = 17.5$–18.2.

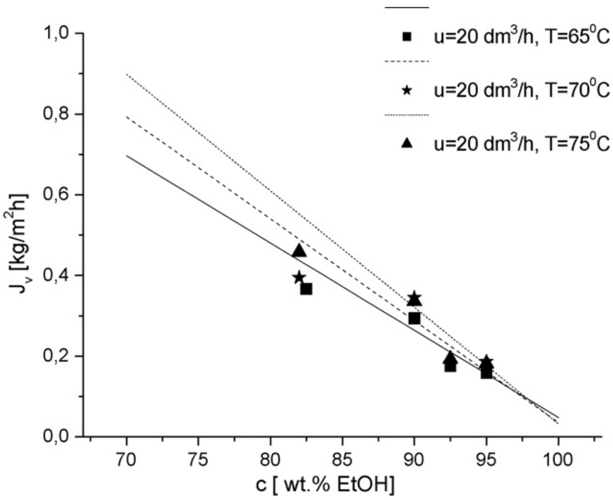

Fig. 5. Dependence of permeation flux on ethanol concentration in the feed for $p = 30$ mbar (line, model data; symbols, experimental data).

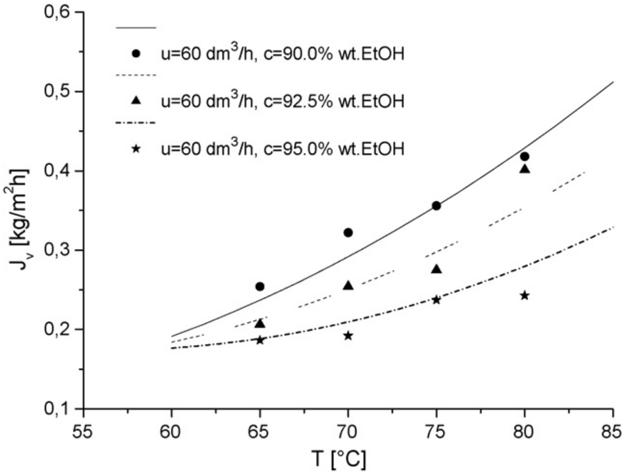

Fig. 7. Dependence of permeate flux on temperature at $p = 30$ mbar (line, model data; symbols, experimental data).

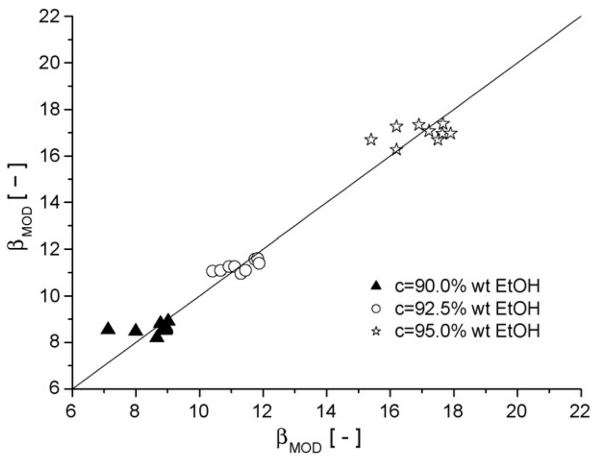

Fig. 8. Comparison of enrichment coefficient β obtained experimentally and calculated from the model for $p = 3$ mbar.

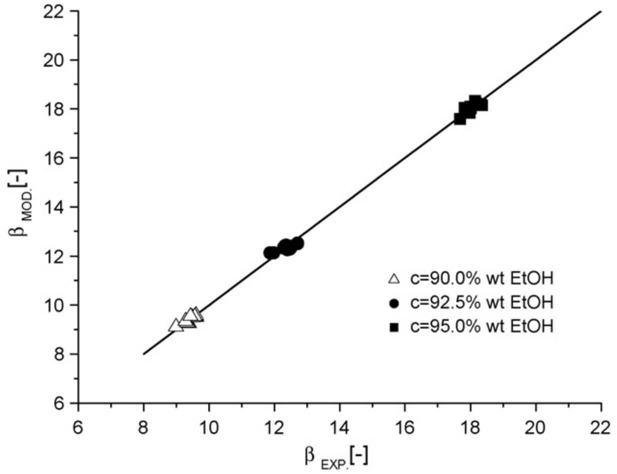

Fig. 9. Comparison of enrichment coefficient β obtained experimentally and calculated from the model for $p = 30$ mbar.

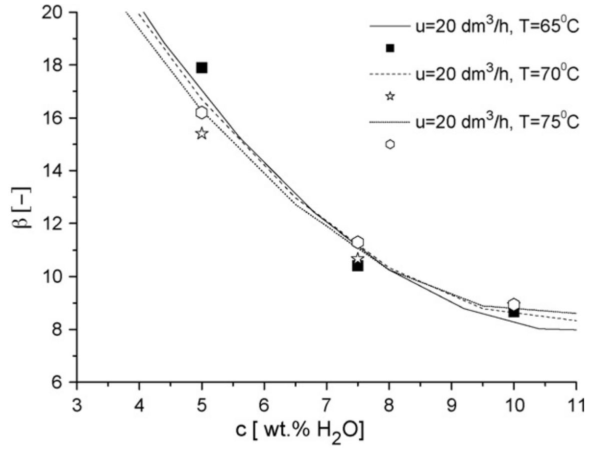

Fig. 10. Dependence of enrichment coefficient β on water content in feed at $p = 3$ mbar (line, model data; symbols, experimental data).

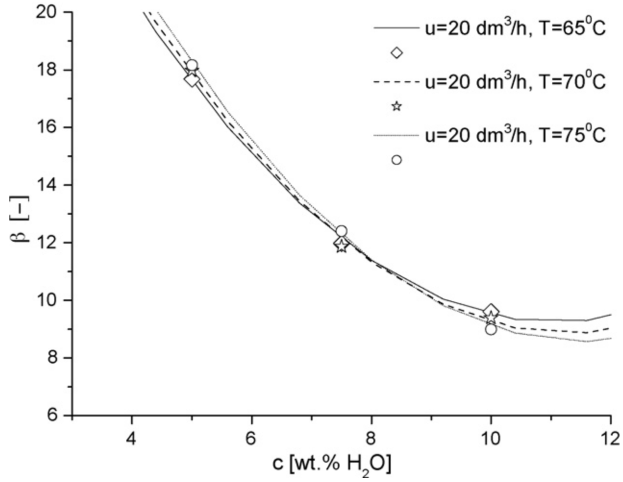

Fig. 11. Dependence of enrichment coefficient β on water content in feed at $p = 30$ mbar (line, model data; symbols, experimental data).

Figs. 10 and 11 show the dependence of enrichment coefficient on water content in the feed for three temperatures and $u = 20\,\mathrm{dm}^3/\mathrm{h}$.

With an increase of water content in the feed, enrichment coefficient β decreases which is confirmed in literature. Taking into account the definition of enrichment coefficient β, mass fraction of water in the permeate can be determined.

An additional coefficient that describes the process of pervaporation is separation coefficient defined as $\alpha = (y_A/y_B)/(x_A/x_B)$ For the considered membrane the coefficient ranged from 40 for 80 wt.% ethanol in the feed to 140 for 95 wt.%, at pressure 3 mbar and from 80 to 250 at $p = 30$ mbar. For membranes from aromatic polyamides [32] coefficient α was 60 for 90 wt.% ethanol in the feed, while for the commercial membrane GFT-PVA [33] $\alpha = 40$, and for the composite membrane $\alpha = 35$. Chen et al.'s research [34] confirms that selectivity increases with an increase of ethanol concentration in the feed.

The proposed empirical model can be used to simulate the pervaporation process for the ethanol–water mixture.

On the basis of the experiments the process of pervaporation was simulated using a program proposed by the authors and a commercial Aspen Customer Modeler. A detailed form of the semi-empirical model and applied procedures are presented in study [27]. Figs. 12 and 13 show a comparison of experimental data and calculated results for selected experiments.

Changes of flux with temperature, alcohol concentration in the feed and flow rate were the same as for the empirical model. Additionally, using the semi-empirical model, the effect of polarisation layer on the permeation flux was determined. Polarisation concentration phenomenon accompanied all membrane separation processes. This process is disadvantageous because it causes an increase of feed concentration in the near-membrane region thus inducing a decrease of the permeate flux. The final conclusion is that fluxes of permeate calculated with and without reference to polarisation did not differ significantly (Fig. 13).

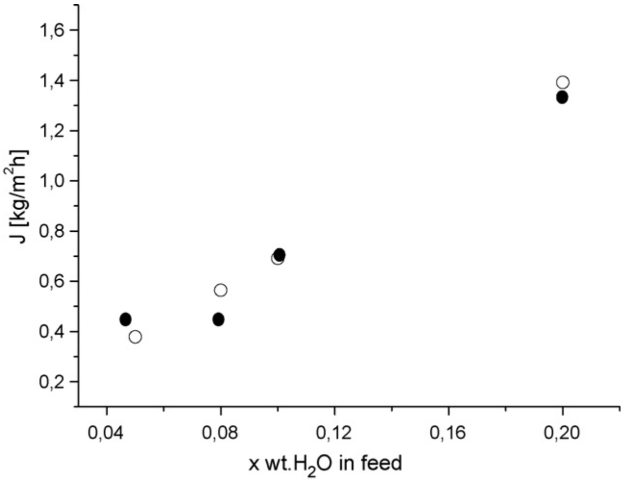

Fig. 12. Comparison of model and experimental data $u = 20\ \mathrm{dm^3/h}$ and $T = 65\ ^\circ\mathrm{C}$ ((●) experimental data, (○) model data).

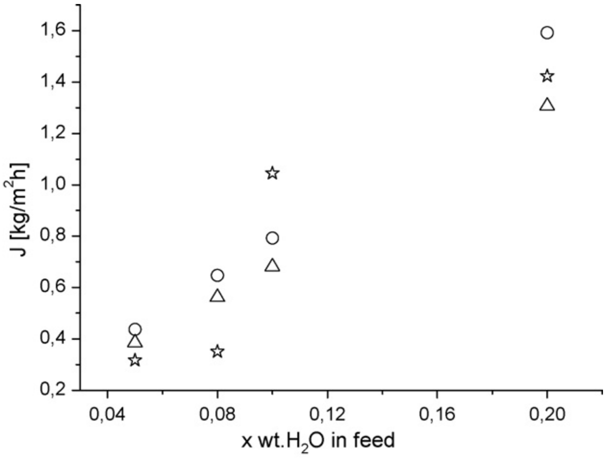

Fig. 13. Comparison of experimental data and those calculated for $T = 70\ ^\circ\mathrm{C}$ and $u = 20\ \mathrm{dm^3/h}$ ((☆) experimental points, (○) model data, (△) with reference to polarisation layer).

4. Conclusions

It follows from the experiments and mathematical analysis that pressure on the low-pressure side of the membrane has the biggest effect on permeation flux, and hence on the pervaporation efficiency (the lower is the vacuum, the bigger is the permeation flux). Formation of low vacuum requires, however, higher energy input, and consequently higher costs.

A conclusion is drawn from the experiments, that with an increase of alcohol content in the feed the permeation flux decreases. Taking into account the effect of temperature on process efficiency we observe that with temperature growth the permeation flux also increases. It might be useful to carry out research for high temperatures, i.e. maximum temperatures for a given membrane material.

Using results of the experiments and simulation it was found that polarisation layer (analysed in the semi-empirical model) had no effect on process efficiency.

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