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# Reverse Osmosis of Aqueous Organic Feed Solutions: Parametric Representation of the Separation Characteristics

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## ABSTRACT

In this study the influence of active substances on the rejection of acetic acid was investigated. Eighty-two series of experiments and the results of former studies were used to assign the observed effects to different process parameters (e.g., pressure and temperature) and to the chemical properties of the active substance (e.g., functionality, molar mass, steric aspects). Based on the results, a conceptional modified model of a microporous membrane based on the Preferential Sorption–Capillary Flow Mechanism was developed to describe and explain the effects. By means of this model a prediction of the retention of a substance is possible, based on an analysis of the feed composition solution.

## INTRODUCTION

Traditionally known as a process for seawater and brackish water desalination, today reverse osmosis (RO) is widely used for the processing of aqueous organic systems in various areas due to the rising availability of high performance composite membranes.

Many scientific publications have dealt with the creation of membrane materials. However, only a few materials are accepted in industrial applications. Additionally, the optimization of the process parameters ($p$, $T$, . . .), the module design, and the theoretical considerations concerning feed flow

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effects and transport mechanisms are important aspects of the current scientific activities.

A systematic investigation of the influence of feed characteristics on separation behavior is not available. Detailed knowledge of the reciprocal interactions of different components in the feed, their permeation capacity, and their influence on the level of rejection is scarce. However, there are some general articles which describe the effects of some of these parameters on retention behavior (1–5).

As previous publications have explained, intermolecular interactions and coupling phenomena lead to strong effects on flux and the retention of substances to be separated (6–11). With this data pool an algorithm of mass transfer was developed which condenses the data and simplifies the assessment of the retention-influencing effect of a component (7). Hence, the data collection gives support when a special separation problem is to be solved. It additionally enables insight to be gained into the separation behavior of a reverse osmosis membrane and it qualitatively describes mass transfer phenomena inside a field of parameters.

The work outlined here tries to evaluate, explain, and predict the observed effects, by creating a theoretical framework for the parameters. Based on the Preferential Sorption–Capillary Flow (PS-CF) Mechanism, and with a further series of experiments, the membrane model is explained and further developed in depth.

The overall project aim as given above could be broken down into a series of smaller subproblems which had to be tackled:

1. To determine the influence of the process parameters pressure, temperature, pH value, and flux, investigations in binary (one compound + water) and ternary (two compounds + water) solutions were carried out.
2. To investigate coupling effects and competing reactions between substances at or in the membrane. Investigations were carried out in ternary solutions and the effects on retention of the target substance were determined. Acetic acid was chosen as the target substance. The reason for this choice was the moderate rejection of 46.7% which guaranteed that an increase and a decrease of rejection could be achieved. The active substances are distinguished by at least one of the following points:
    - Functionality
    - Molar mass
    - Steric aspects
3. To relate the observed intermolecular interactions to the general influence of the parameters on retention of a particular substance.

The existing PS-CF Mechanism has to be modified to obtain the desired results. This model was developed by Sourirajan in 1963 and is well known. Details can be drawn from the literature (12–15). The work outlined focuses only on further developments as a modified concept.

## EXPERIMENTAL

The experiments were performed using an RO test rig supplied by Sempas Membrantechnik GmbH. This equipment contains a composite membrane synthesized from an aromatic polyamide (Toray: TR70-2514F). The spiral-wound module has an active membrane area of approximately 0.7 $m^2$. A rejection of 99% is reached for 0.026 $mol \cdot L^{-1}$ NaCl at 14.4 bar (1.44 MPa); see Fig. 1.

Pure solutions of the respective organic substances were prepared (see Table 1). These binary solutions were compared with other solutions which contained two organic substances, the target substance (which has to be separated: acetic acid) and the active substance (which produces the change in permeability). All substances in binary or ternary solutions were used in a 10 $mmol \cdot L^{-1}$ concentration. The feed flow was brought to a level of 2.5 $L \cdot min^{-1}$ at a hydrostatic pressure of 10 bar. The temperature was stabilized between 18 and 23°C.

During the investigations of pressure and temperature dependence, the pressure was varied between 2 and 10 bar and the temperature rose from 12 to 26°C.

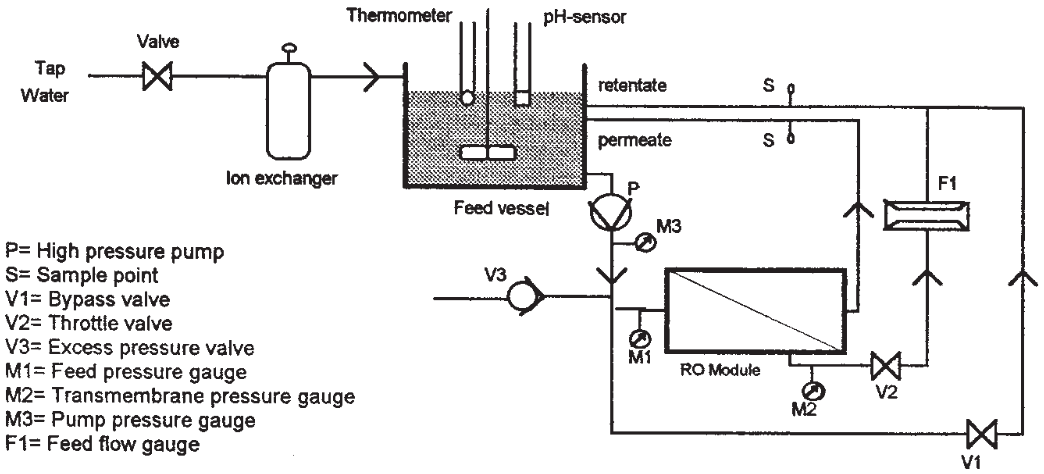

FIG. 1 Reverse osmosis test rig (internal recirculation).

TABLE 1
Overview of the Substances Used in This Investigation

| Organic acids | Alcohols | Esters | Sugars |
|---|---|---|---|
| Acetic acid | Benzyl alcohol | Ethyl acetoacetate | D-(−)-Fructose |
| 2-Ethylbutyric acid | Pyrocatechol | Acetomethyl acetate | L-(−)-Fructose |
| Glutaric acid | Hydroquinone | Acetylaminodiethyl malonate | D-(+)-Xylose |
| Adipinic acid | *cis*-2-Butene-1,4-diol | | L-(+)-Xylose |
| Itaconic acid | L-(+)-Ascorbinic acid | | L-(−)-Sorbose |
| *trans*-Aconitic acid | D-Sorbitol | | Lactose |
| 3-Methylcrotonic acid | L-Sorbitol | | |
| Benzoic acid | L-Mannitol | | |
| 4-Hydroxybenzoic acid | | | |
| 3-Hydroxybenzoic acid | | | |
| 2-Phenylbutyric acid | | | |
| Phenoxyacetic acid | | | |
| *o*-Acetylsalicylic acid | | | |
| Citric acid | | | |
| Benzene-1,2,4,5-tetracarboxylic acid | | | |
| | Amino acids and derivates | Ketones | Others |
| | Alanine | Acetonylacetone | Maleic anhydride |
| | *N*-Acetyl-L-cysteine | 5-Hexene-2-on | Urea |
| | *N*-Acetyl-glycine | | L-(+)-Ascorbinic acid |
| | *n*-α-Acetyl-L-glutamine | | Ethylenediaminetetraacetic acid (EDTA) |

When a steady state was reached, samples of the permeate ($C_P$) and the retentate ($C_R$) were taken. The different concentrations were analyzed by HPLC. The following equation was used to calculate the rejection for each data group:

$$R = 1 - \frac{C_P}{C_R} \cdot 100\% \qquad (1)$$

Five $R$-readings were found for each combination, and these were used to determine the median. By using the U-Test of Wilcoxon, Mann, and Whitney (16), a check was made to see whether the median of the target substance was significantly better or worse than that of this substance in any of the other solutions. Pearson's correlation coefficient to the second power ($R^2$) was used to describe the exactness of the linear regression (17).

A selection of the substances used in these investigations is shown in Table 1.

## RESULTS AND DISCUSSION

Results of former studies (6–10) were used to develop the modified model assumptions. A complete list of the results can be requested from the authors.

### Process Parameters

#### *Pressure*

The influence of the hydrostatic pressure on the separation behavior is well known and has been described before (e.g., in Refs. 18–20). It has been found that retention decreases with rising pressure while the permeate flux increases.

#### *Temperature*

Temperature has a strong influence on separation behavior.

The retention of 47 ternary solutions was measured in the 18 to 22°C temperature range of Fig. 2 shows three series of experiments.

The complete 235 samples were used to develop an equation to describe the change of acetic acid retention in relation to the temperature. It was found that an increase in temperature of 0.391°C results in a loss of acetic acid retention of 1.3341% on average.

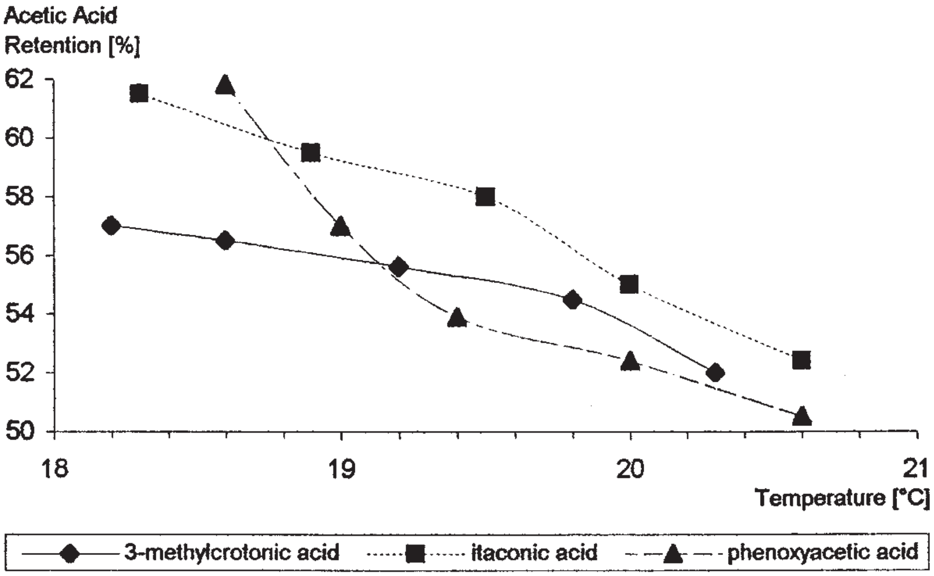

FIG. 2 Retention of acetic acid in ternary solution vs temperature.

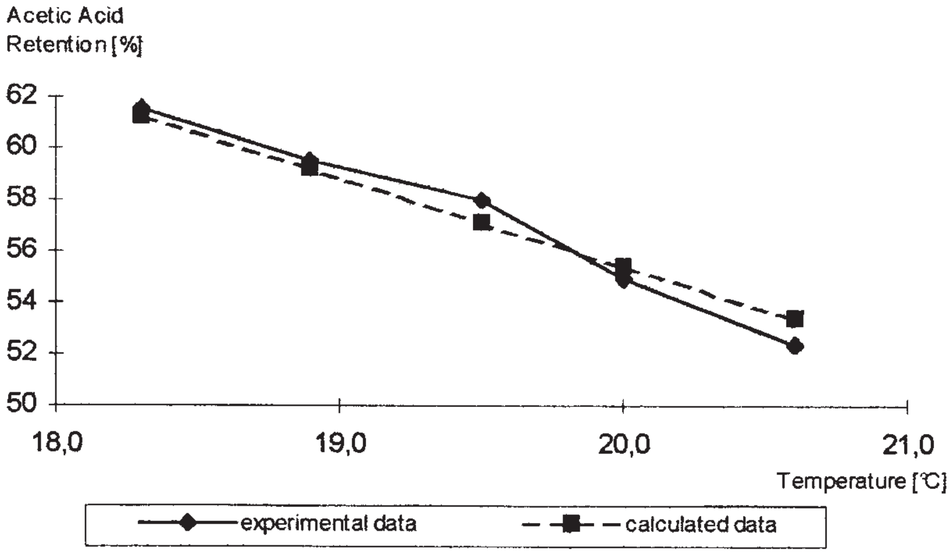

FIG. 3 Retention of acetic acid combined with itaconic acid vs temperature (comparison between experimental and theoretical data).

$$R_{s_2} - R_{s_1} = 0.013341 \cdot \frac{T_{s_2} - T_{s_1}}{0.3391^\circ\mathrm{C}} \tag{2}$$

Mean square error = 0.0746%, $R$ = retention (%), and $s_1/s_2$ = Sample 1/ Sample 2.

In Fig. 3 the experimental results are compared to results calculated with Eq. (2). It can be seen that Eq. (2) leads to a good approximation.

In binary solutions the retention decreases with rising temperature. This effect is known from other studies (21, 22).

## pH Value

The increase of the pH value caused by the addition of sodium hydroxide results in an increase of acetic acid rejection, as shown in Table 2. The sodium acetate concentration was calculated by the Henderson–Hasselbalch Equation:

$$\mathrm{pH} = \mathrm{p}K_s + \lg \frac{C_{\mathrm{Na(CH_3COO)}}}{C_{\mathrm{CH_3COOH}}} \tag{3}$$

As shown in Table 2, the sodium acetate concentration depends strongly on the pH value.

Due to the fact that pure sodium acetate is 99.5% rejected, the increase of acetic acid rejection is caused by an increase of the sodium acetate concentration.

TABLE 2
Dependence of $Na(CH_3COO)$ Concentration and Retention on the pH Value

| Active substance | pH value | $Na(CH_3COO)$ concentration [$mmol \cdot L^{-1}$] | Retention [%] (median) |
|---|---|---|---|
| Acetic acid, pH 3.3 | 3.3 | 0.342 | 46.2 |
| Acetic acid, pH 4.2 | 4.2 | 2.199 | 65.2 |
| Acetic acid, pH 5 | 5.0 | 6.401 | 82.2 |
| Acetic acid, pH 6 | 6.0 | 9.468 | 92.3 |
| Acetic acid, pH 7 | 7.0 | 9.944 | 97.4 |

In ternary solutions the influence on rejection depends solely on the chemical properties of the second active substance if there is no adjustment with NaOH.

If the active substance is able to react with NaOH, this reaction occurs prior to possible interactions between the active and the target component acetic acid in the solution. Therefore, intermolecular interactions which result in higher rejections will decline. Substances which are not able to react with NaOH exhibit no observable effect on acetic acid retention.

### Flux

The permeate flux depends on a combination of several parameters. In general, an increase in permeate flux results in decreasing rejection. Based on the PS-CF Mechanism and the conceptional assumptions described below, a lower permeate flux effects a longer contact of the substances with the membrane. Hence the pores will be covered and the acetic acid will be excluded by the membrane.

## Chemical Properties of the Feed Solution

### Functionality

Figure 4 shows that the functional group of the active substance is not the only parameter which influences the separation behavior of acetic acid. Substances of four different functional groups effect a decrease, and substances of all functional groups effect an increase of acetic acid rejection in ternary solutions. Hence it can be concluded that other parameters may exhibit stronger effects on the retention.

### Molar Mass

The results show that the molar mass has an influence on retention behavior. A consideration of all results shows no clear tendency, but inside a homolo-

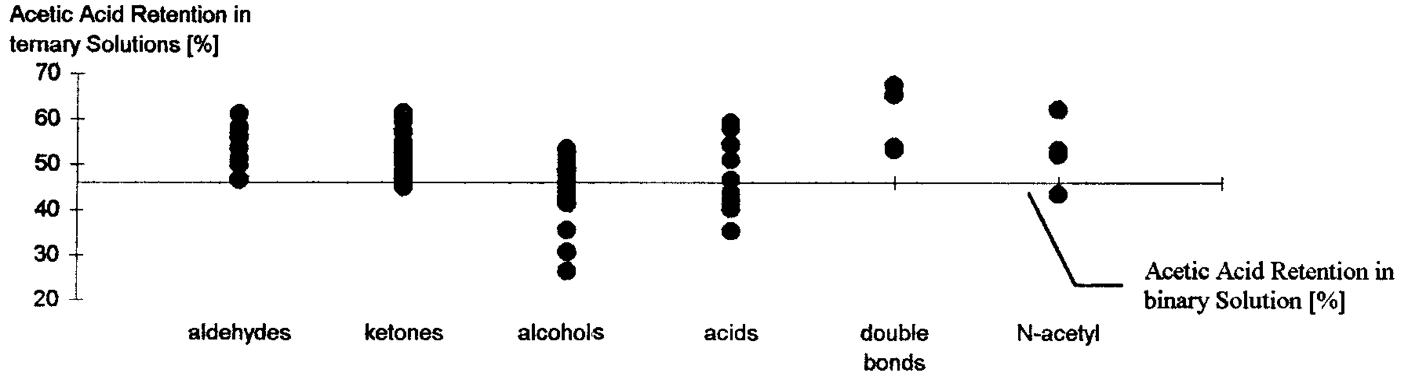

FIG. 4 Acetic acid retention vs functional groups of the active substance.

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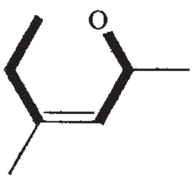
gous series of chemicals the retention increases with rising molar mass. The only exeption is the bivalent acids which show decreasing retention with rising molar mass. Figure 5 exhibits three different homologous series: aldehydes, diketones, and diols.

Table 3 presents statistical data of the dependence of acetic acid retention on the molar mass of the active substance. The $R^2$ values show that there is no clear linear connection between molar mass and acetic acid retention.

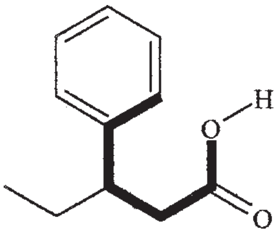
## Steric Consideration

The substances which exhibit the highest influence on the rejection of acetic acid are summarized in Table 4. A general connection between the influence of an active substance and its structure was found.

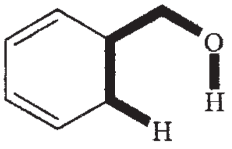
This common factor is neither the molar mass nor the functional groups. The structures presented here show that there is a common element, a kind of ‘‘bracket’’ which contains four to six atoms and at least one oxygen or nitrogen atom.



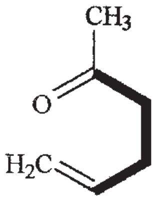
**mesityl oxide**



**2-phenylbutyric acid**



**benzylalcohol**



**5-hexene-3-on**

It is presumed that this ‘‘bracket form’’ is the reason for the strong influence. This conformation of the active substance explains the differences between the influences on acetic acid rejection. This consideration is demonstrated by the example of Table 5: The bracket of pyrocatechol contains six atoms, including two oxygen atoms, whereas the bracket of hydroquinone is build up of four atoms including only one oxygen. The para position in hydroquinone creates a distance between the oxygens of 5.57 Å and a O—HO

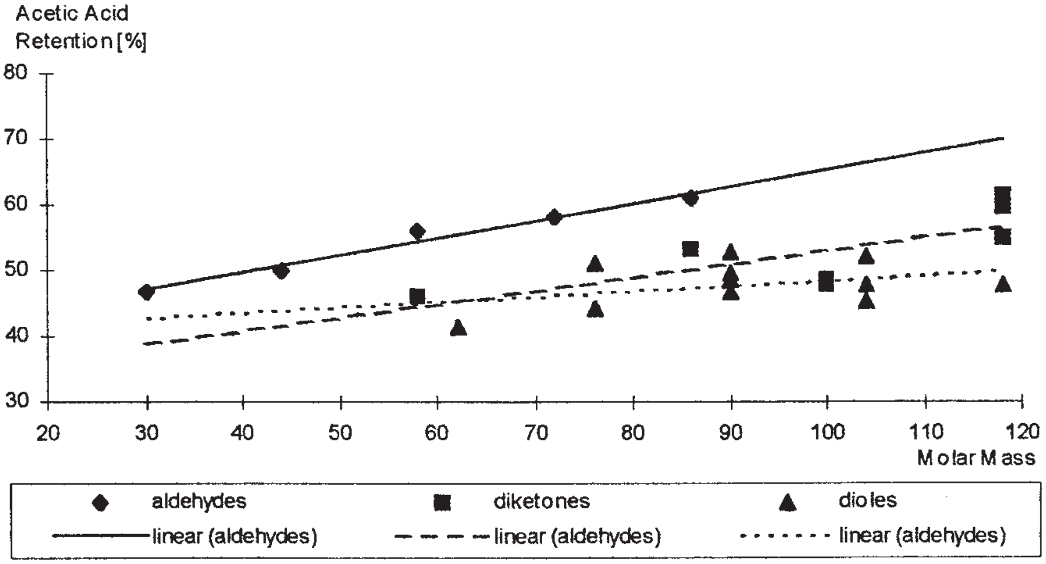


FIG. 5 Molar mass vs acetic acid retention modified from Refs. 10 and 13.

distance of 6.07 Å. On the other hand, in pyrocatechol the O—O distance is 2.78 Å and the O—HO distance is 3.72 Å. Hence, the bracket of pyrocatechol is superior to the bracket form of hydroquinone. This results in a stronger effect on the retention of a target substance.

## Concentration Polarization

Attention is drawn to concentration polarization which is an important phenomenon of reverse osmosis experiments. It shows significant effects on the *real retention* of both acetic acid and active substances in every experi-

TABLE 3
Dependence of Acetic Acid Retention on Rising Molar Mass of Active Substances

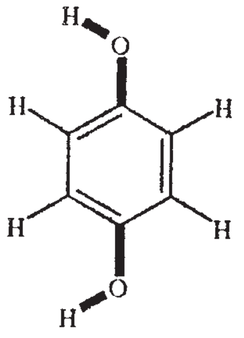
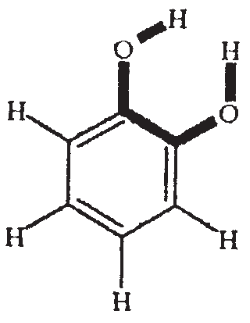
| Chemical series | $R^2$ |
|---|---|
| Aldehydes | 0.972 |
| Ketones | 0.2138 |
| Diketones | 0.5659 |
| Alcohols | 0.4327 |
| Diols | 0.1366 |
| Carboxylic acids | 0.3871 |
| Dicarboxylic acids | 0.5482 |

TABLE 4
Substances with a Strong Influence on Acetic Acid Retention

| Substance | Formula | Molar mass [g·mol$^{-1}$] | Functional group(s) | Increase of retention (percentage) |
|---|---|---|---|---|
| 3-Hydroxybenzoic acid | $C_7H_6O_3$ | 138.12 | COOH, OH | +10.0 |
| β-Alanine | $C_3H_7NO_2$ | 89.09 | COOH, $NH_2$ | +16.1 |
| 5-Hexene-2-on | $C_6H_{10}O$ | 98.75 | CO, double bond | +23.4 |
| Benzyl alcohol | $C_7H_8O$ | 108.14 | OH | +19.7 |
| 2-Phenylbutyric acid | $C_{10}H_{12}O_2$ | 164.20 | COOH | +13.3 |
| Mesityl oxide[a] | $C_6H_{10}O$ | 98.14 | CO, double bond | +18.8 |
| *o*-Acetylsalicylic acid | $C_9H_8O_4$ | 180.15 | COOH, OCOR | +23.0 |
| Lactose | $C_{12}H_{22}O_{11}\cdot H_2O$ | 360.32 | 8 OH, 3 ROR | +15.8 |
| Cyclohexanecarboxylic acid[b] | $C_7H_{12}O_2$ | 128.17 | COOH | +12.7 |

[a] From Ref. 13.
[b] From Ref. 14.

TABLE 5
Comparison of Two Molecules of Different Conformations

| | Hydroquinone | Pyrocatechol |
|---|---|---|
| Total formula | $C_6H_6O_2$ | $C_6H_6O_2$ |
| Acetio-acid retention (%) | 48.5 | 53.5 |
| Structural formula |  |  |

ment. In every one-component solution the effect is always the same because of the equimolar concentrations which can thus be neglected. Concentration polarization leads to rising osmotic pressure of the bulk solution near the membrane, followed by a selectivity drop based on the decreased flux of water (18).

It was determined that the described effect is not only compensated for but even reversed by the addition of certain active substances. Since concentration polarization is solely dependent on the mole fraction at the membrane surface, which is connected with the concentration in the bulk solution, the addition of another substance increase the polarization and hence decrease the selectivity (23–25). This assumption was disproved by the experiments.

Hence it can be concluded that the effect of concentration polarization is of *no importance* for the observations mentioned, keeping in mind that the received data are exclusively qualitative.

## Modification of the Preferential Sorption—Capillary Flow Mechanism

The PS-CF Mechanism can be described as follows: The membrane is assumed to be microporous, which means the diameter of a pore is less than 20 Å. The preferential permeating component is also preferential sorbed by

TABLE 6
Summary of the Observed Effects

| Process parameters | Effect on the retention of acetic acid |
|---|---|
| Pressure | Rising pressure leads to a decrease of retention |
| Temperature | A decrease of temperature leads to an increase of retention. A quantitative connection could be found. |
| pH value | An increase of the pH value results in an increase of retention caused by an increase of sodium acetate concentration |
| Flux | In general, a rising permeate flux leads to decreasing retention |
| **Chemical properties** | **Effect on the retention of acetic acid** |
| Functionality | Acetic-acid retention changes with different functional groups, but other parameters exhibit stronger effects |
| Molar mass | An increase of molar mass leads to an increase of retention in homologous series |
| Structure | The common factor among the substances which shows the highest influence on the retention of acetic acid is a structural element, a ‘‘bracket form’’ which contains four to six atoms and at least one oxygen or nitrogen atom |

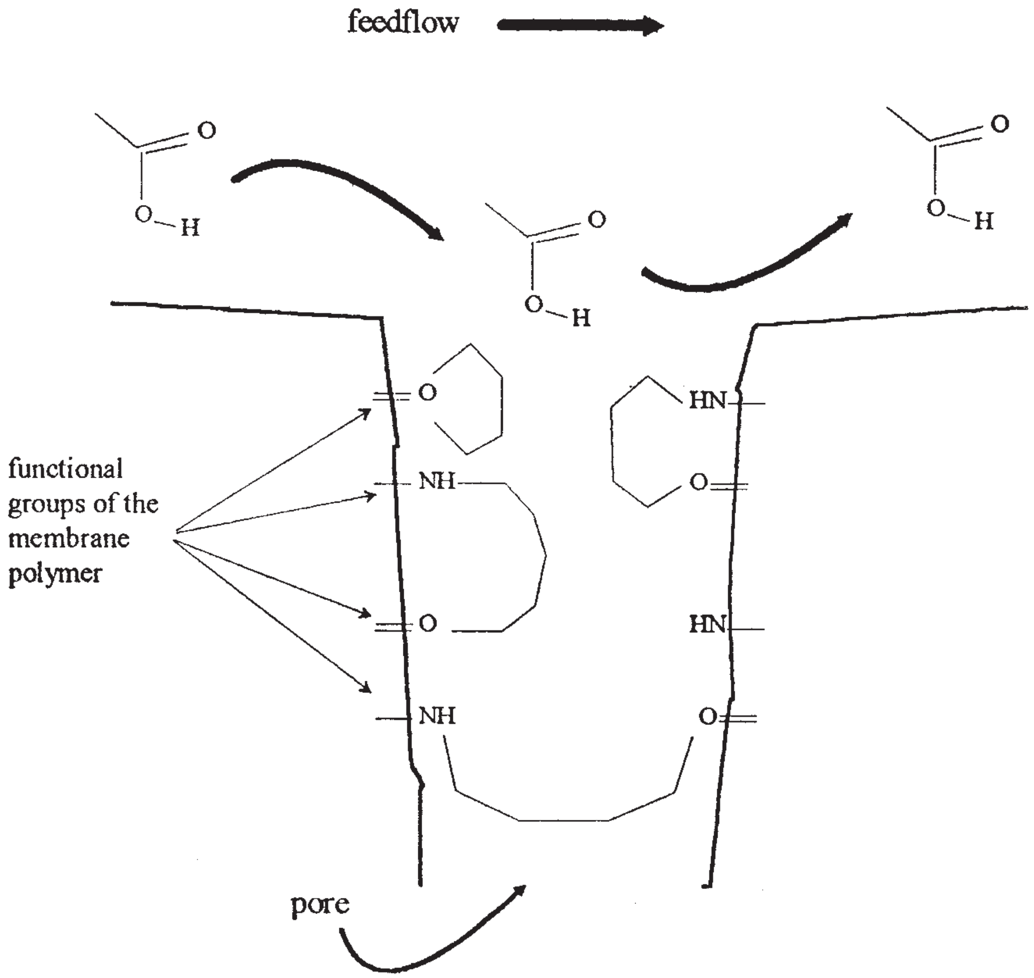


FIG. 6 Model of a membrane pore.

the membrane surface. Transport through the membrane takes place by migration of the different substances at the pore wall (12, 15, 26).

The PS-CF Mechanism was developed for binary (two compound) solutions, especially for salt–water mixtures. The effects of additional substances as described above can not be explained by this mechanism. The following modifications of the PS-CF Mechanism should describe and explain the influence of an active substance on the separation of acetic acid from water.

All effects influencing the retention of acetic acid in three-component solutions are summarized in Table 6. Due to the heteroatoms, interaction between the active substance and the membrane material is possible. These interactions can be electrostatic interactions, London forces, or hydrogen bonds. Figure 6 describes the possible arrangements of the active substance emerging inside the membrane. The active substance is enriched inside the pore and competes with acetic acid for sorption on the pore wall. The pore diameter decreases and, as a result, the acetic acid is pushed back into the feed.

Hence, the retention of acetic acid depends on the strength of interaction between the membrane material and the active substance and its resulting steric hindrance to the target substances.

The influence of the other process parameters can also be explained by this theory:

- A higher temperature causes a higher kinetic energy of the molecules. This effect impairs interactions and increases acetic acid permeation.
- Rising hydrostatic pressure results in similar effects. A decrease of pressure causes an increase of residence time, and interactions between the membrane material and different substances will be intensified.
- The effect of pH depends on the acetate anion concentration. Ions are preferential repulsed by a polyamide membrane, and therefore retention decreases while the pH value increases.
- The size and the shape of the molecular residuary of the active substances has a decisive influence on rejection of acetic acid. Due to the increase of molecular mass within a homologous series of chemicals, membrane pore diameter decreases and acetic acid rejection increases.

## CONCLUSION

The following conclusions can be drawn:

1. The process parameters pressure and temperature have the strongest influence on retention behavior. These results are partially confirmed by other studies.
2. The influence of the pH value is dependent on the components of the feed. A clear connection can be found between the retention of the target substance and the acetate concentration.
3. The size of the molecular residuary depends on the molecular mass; the pore diameter of the membrane will be reduced with rising molecular mass. Hence, increasing molecular mass results in an increasing effect on acetic acid retention.
4. A clear connection was found between the stereochemistry of the active substance and the retention of the target substance. These observations lead to a modification of the Preferential Sorption–Capillary Flow Mechanism to explain the described effects.

A qualitative prediction of the separation behavior of a particular substance in a mixed system can be achieved by means of these modifications. Further studies are planned to confirm and to consolidate this theory.

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