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Mass transport modelling to estimate the efficiency of nanofiltration application for the recovery of phosphorus from sewage sludge

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ABSTRACT

The polyamide nanofiltration membrane DL has been used for experimental series investigating the filtration of sewage sludge ash eluates as well as model solutions mimicking the real eluates. It is shown that the use of synthesised model solutions makes up an useful tool to study the separation of real solutions in a practical and reproducible way. The DSPM&DE approach has been estimated from the information given in literature about the recent development in quantitative nanofiltration mass transport modelling to be a promising methodology for modelling the filtration process studied in this work. A first application of the model using a software tool described in literature using membrane parameters mainly estimated according to information from the literature was conducted. The comparison between experimental and calculated results showed that the tendency of the rejections could be predicted by the model for most of the used ions.

Keywords: Nanofiltration; Modelling; Sewage sludge ash; Phosphorus recovery

1. Introduction

Several processes using nanofiltration membranes to purify acids from metal impurenesses has been successfully applied and described in literature [1–4]. These applications are based on a high selectivity of nanofiltration membranes between the partly dissociated acid molecules and metal cations [1–4]. Furthermore, such separation technologies are getting increasingly attractive due to the recent development of highly acid resistant membrane materials [5]. The focus of this paper is the investigation of a similar separation approach, namely the separation of phosphoric acid from metal ions in order to optimise the recovery of phosphorus as a secondary raw material from waste water. Nanofiltration is aimed to be integrated in a special treatment process chain of sewage sludge or sewage sludge ash. The treatment steps applied before the nanofiltration should adjust conditions to the separation by the transfer of the phosphorus into dissolved phosphoric acid and a pH adjustment in the range of 1–3. Sludge solids respectively ash solids should be removed by decantation and ultrafiltration.

In the case of this nanofiltration process a low retention for phosphoric acid on the one hand and a high retention for metal cations is required since the nanofiltration permeate makes up the purified phosphorus containing product stream. Experimental work has already been conducted to determine the retention of nanofiltration membranes for the dissolved components of sewage sludges and sewage sludge ashes pretreated according to the above mentioned demands. The results showed a relatively high dependency of the selectivity on the concentrations of the various dissolved components and the

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selectivity was found to be significantly lower than for the purification of concentrated acids [6].

For an applicability estimation of a phosphorus recovery process from different secondary sources supported by nanofiltration this study aims to investigate the possibilities of mass transport modelling. The implementation of a mass transport simulation seems to be advantageous to enable a detailed analysis of the separation mechanisms and to study the efficiency of the phosphorus recovery with reduced experimental effort. Therefore this work gives an overview about several existing approaches and a first step in applying the Donnan steric partitioning pore and dielectric exclusion (DSPM&DE) approach on the experimental data.

2. Theoretical background of Nanofiltration modelling

Currently the literature reflects a strong effort in the field of predictive modelling of nanofiltration. Generally the approaches described in the literature can be divided into phenomenological equations basing on irreversible thermodynamics like Kedem-Katchalsky and Kedem-Spiegler model, the extended solution-diffusion model and more fundamental models basing on the Stefan-Maxwell equations and the extended Nernst-Planck equation [7] as well as the application of artificial neural networks.

In the first mentioned types of models the membrane is handled as a black box so that the predictive power is decisively constricted. The extended solution-diffusion model is applicable in case of the filtration of uncharged molecules [8]. For the transport of salt through nanofiltration membranes the use of the Stefan-Maxwell equations is the most fundamental approach and has therefore the potential for the most accurate predictive results. The basis of the Stefan-Maxwell approach is to look at the ion movement as a stationary state in which the driving forces are equal to the friction between the relevant ion and the other system immanent components [9]. Due to the complexity of this model the practical relevance especially in the case of multi-ionic system is still relatively low. The extended Nernst-Planck equation basing on statistical thermodynamics [7] is the approach with the currently most practical relevance but is according to [9] intrinsically a simplification of the generalised Stefan-Maxwell equation. Furthermore, there are several applications of artificial neural networks for the prediction of nanofiltration performance described in the literature [10–12], although the main application of artificial neural networks in the membrane sector is the modelling of fouling.

General demands on the ability of models for membrane filtration are given in the literature and can generally be summarised as follows [9,13]:

- a physically realistic description of the relevant mechanism,
- a minimum number of assumptions,
- provision of quantitative process predictions (flux, permeate and retentate concentrations – as a function of the pressure),
- based on the physics of the separation,
- use of membrane parameters which can be obtained from a limited number of experiments with model liquids.

In this work it is additionally important to apply an approach which is able to take a multi component solution into account, since the target feed solution in this case is a system with a wide range of ions and varying concentrations. Hence it is important to be able to predict the membrane performance in dependency on the concentrations of a big number of dissolved ions.

In the literature there are recently implementations of transport modelling for multi component solutions documented mainly using the so called Donnan steric partitioning pore and dielectric exclusion (DSPM&DE) model. There are several works documented in the literature which were able to predict the retention of nanofiltration membranes reasonably well even for systems containing three and more ions. The main outline of the DSPM&DE model is presented in the following paragraphs:

This approach assumes the membrane to consist of cylindrical nanopores through which the ions are transported as solutes in a bulk solvent as proposed by Deen [14]. The transmembrane transport is divided into four phases described separately with a set of equations for each phase. As illustrated in Fig. 1 the first phase is the transport from the bulk solution through a boundary layer in which concentration polarisation takes place for all components with are retained by the membrane. The second phase is the discontinuous concentration change at the interface of the membrane which makes up the transfer from the concentration polarisation layer of the bulk phase into the membrane pore. After this the transport through the membrane pore is calculated and as the fourth phase the discontinuous transfer at the membrane interface between membrane pore and permeate bulk phase is taken into account. Transport resistances for the permeate in the porous support and the concentration polarisation layer on the permeate side are neglected in the presented model.

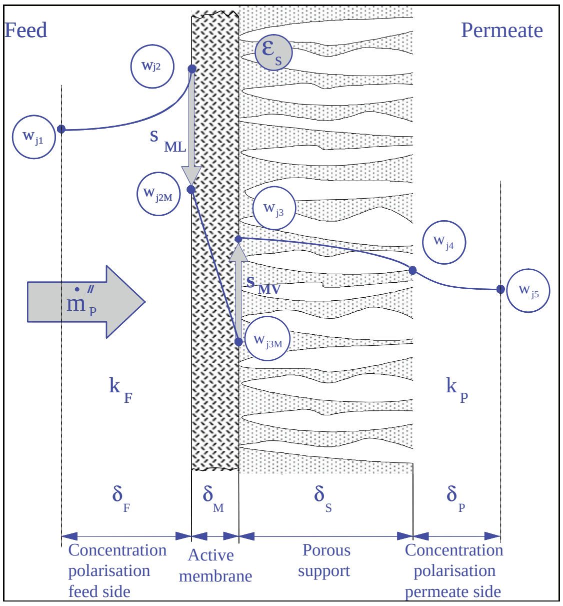

Fig. 1. Different possible phases of the membrane transport modelling [15].

There are different ideas on how to realise the description of the influence of the membrane characteristics on the transport through the membrane. The space-charge model (SCM) [16] takes into account radial and axial changing charges in the membrane pores and treats ions as point charges. The DSPM&DE based on the Theorell-Meyer-Sievers Model (TMSM) [17] assumes charges to be radially constant [18,19]. This assumption could be shown to be consistent for most nanofiltration membranes since the small pore radius is under usual concentration conditions smaller than the Debye length. This fact means that an overlapping of the charge double layers in the pore walls can be assumed and therefore an uniform radial charge distribution inside the pores [20].

The central part of the filtration process, the transport through the membrane pore is according to the DSPM&DE described by the extended Nernst-Planck equation (ENPE):

$$j_i = K_{c,i} c_i V - K_{d,i} D_{i,\infty} \frac{dc_i}{dx} - \frac{z_i c_i D_{i,p}}{RT} F \frac{d\psi}{dx} \tag{1}$$

This form of the ENPE illustrates clearly that the transport in the membrane pores consists of a convective and a diffusive part which is influenced especially by electrokinetic mechanisms [15,21]. The DSPM&DE uses the hindrance factors K_c – for the convective term – and K_d for the diffusive term – which depend on the ratio of ion stokes radius and the membrane pore radius as well as on the steric partitioning factor. Additionally the hindered diffusion coefficient is used which can be calculated with the normal diffusion coefficient and the hindrance factor for diffusion.

In order to determine the equilibrium partitioning at the interfaces feed-membrane and membrane-permeate the DSPM&DE takes into account the Donnan-equilibrium, steric effects and additionally – in contrast to the DSPM – dielectric exclusion. These mechanisms are described by the following equations [13]:

$$\frac{\gamma_i c_i}{\gamma_i C_i} = \Phi_i \exp\left(\frac{-z_i F}{RT} \Delta\psi_D\right) \exp\left(\frac{-\Delta W_i}{kT}\right) \tag{2}$$

$$\Delta W_i = \frac{z_i^2 e^2}{8 \cdot \pi \cdot \varepsilon_0 \cdot r_i} \left(\frac{1}{\varepsilon_p} - \frac{1}{\varepsilon_b}\right) \tag{3}$$

The later equation originates from the Born model and expresses the phenomena of dielectric exclusion by defining the energy barrier ΔW_i to solvation of ions into the pores. This energy barrier results in the decrease of the dielectric constant ε_p inside the pores in comparison to the bulk dielectric constant ε_b.

In contrast to the modelling of a single-electrolyte solution, for which a direct solution of the relevant equations can be derived [13], the modelling of a multi-ionic solution results in a differential-equation system, which has to be solved with numerical methods [22,23]. Geraldes [20] implemented the DSPM&DE model correspondingly in a freely available computer program. In this computer program concentration polarisation is not described in terms of a fluid-dynamic model [24,25], but in terms of the Nernst-Planck equation so that no polarisation layer thickness has to be assumed. The ion solutions are treated in this model implementation as non-ideal solutions by calculation of the activity coefficients with the help of the Devies equation which has been discussed partly controversially for an application inside the membrane pores [13,20].

The DSPM&DE approach implemented according to [20] requires the pore-diameter of the membrane, the membrane charge, the membrane thickness, the dielectrical constant inside the membrane pores and the radii of the soluted molecules as parameters.

Some authors discussed approaches which would be possible to supplement this transport model. One option is the use of membrane pore size distributions instead of one average pore size diameter [13]. Furthermore, authors using an average pore size used the

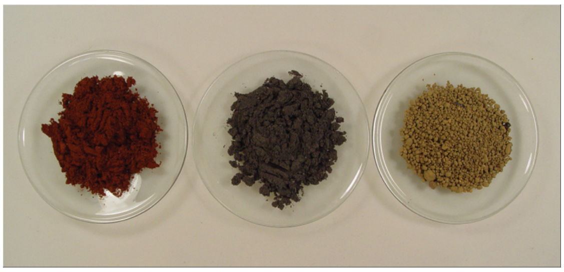

Fig. 2. Used sewage sludge ashes (A1, A2, A3).

option to treat the pores not automatically as cylindrical formed but alternatively as slit-like pores [23].

3. Experimental methods

As described elsewhere in detail [26] three types of sewage sludge ash (A1, A2, A3) have been sampled in incineration plants and treated with sulphuric acid in order to produce sewage sludge eluates of varying compositions. Fig. 2 shows the three used sewage sludge ashes which were treated with water and concentrated sulphuric acid (96%) in varying dilution rates achieving a pH of 1.5.

Before conducting the nanofiltration the sewage sludge ash eluates were filtrated with ultrafiltration membranes (UP150, Microdyn Nadir) for particle removal. The nanofiltration experiments were done with a stirring membrane test cell containing an active membrane area of 121 cm^2 of the membrane DL (GE Osmonics) placed on a sintered metal plate built of stainless steel 1,4404 with a porosity of 50 μm. The mixing was realised with a magnetic stirrer (length 7 cm, diameter 1.7 cm) placed 0.7 cm above the membrane and rotating at 230 rpm. The cell was pressurized with nitrogen gas and the temperature was adjusted to 25°C.

A series of model solutions were prepared to study effects of the composition of the sewage sludge ash eluates in a systematic way. The composition of the model solutions is shown in Table 1. The cations were dissolved in deionised water as sulphates and sulphuric as well as phosphoric acid were added.

4. Results and discussion

The sewage sludge ashes were filtrated with the polyamide nanofiltration membrane to investigate the potential of an optimised phosphorus recovery with nanofiltration membranes. The aim of the separation is a phosphorus containing permeate as product stream. Therefore a low phosphorus retention is beneficial to recover as much phosphorus as possible and a

Table 1
Composition of model solutions used for the experimental work

		M1	M3	M7	M9	M10	M12	M16	M18	M19	M21	M25	M27	M28	M30	M34	M36
[mmol/L]	Al	111.19	3.71	111.19	3.71	0.74	0.74	0.74	0.74	0.74	0.74	0.74	0.74	0.74	0.74	0.74	0.74
[mmol/L]	P	3.23	3.23	258.31	258.31	3.23	3.23	258.31	258.31	3.23	3.23	258.31	258.31	3.23	2.23	258.31	258.31
[mmol/L]	Fe	0.36	0.36	0.36	0.36	1.79	21.79	1.79	21.49	0.36	0.36	0.36	0.36	0.36	0.36	0.36	0.36
[mmol/L]	Ca	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	12.48	0.50	12.48	0.50	0.50	0.50	0.50
[mmol/L]	Cu	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.06
[mmol/L]	Mg	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82	0.82
[mmol/L]	S	215.44	52.28	168.53	7.30	58.25	113.94	4.29	23.99	47.83	59.81	2.86	14.83	55.88	314.77	55.88	314.77
[mmol/L]	Na	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	37.82	486.42	121.06	805.27

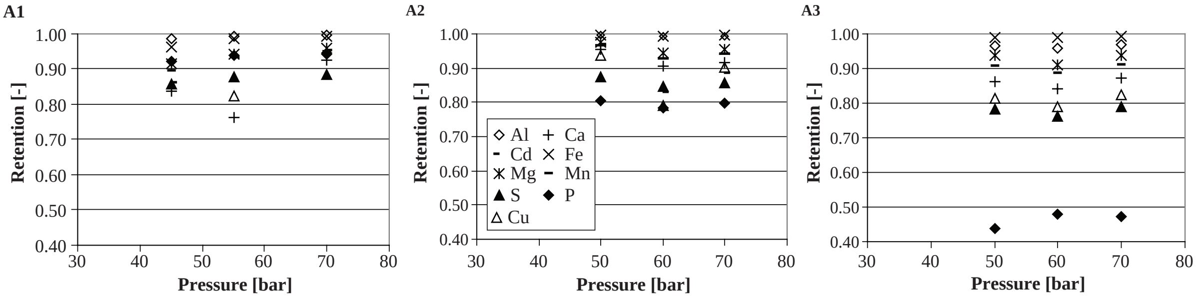

Fig. 3. Retention for several pressures A1, A2 and A3.

high rejection for metal ions to obtain a high quality phosphorus product.

Fig. 3 summarises a part of the results of the filtration of real sludge eluates of the three chosen sewage sludge ashes at three different pressures each. According to these filtration results the differences in the composition between these three ash eluates led to a significant different separation performance. Model solutions were built up to study the dependencies between the several ion concentrations and the rejection by the nanofiltration membrane. Model solution M1 was composed as a reference solution with a similar composition like the eluate of ash A1. As the rejections measured for the real ash eluate solution and the model solution were very similar to those obtained for M1 (Fig. 4) the results for the model solution study can serve for conclusions about the recovery process. Hence the approach of using synthesised solutions revealed to be a useful tool to study the separation of real solutions in a practical and reproducible way.

4.1. Modelling

To understand the transport phenomena of the membrane in a better way and to obtain more filtration data with a reduced experimental effort it is aimed to apply a predictive mass transport model approach. As a first step in achieving predictive calculations for the retention of nanofiltration membranes the experimentally obtained retention for the two model solutions M1 and M3 were compared with calculations using the implementation of the DSPM&DE approach by Geraldes [20] described in the theoretical part of this paper. As first assumptions a Sherwood-relation described by [27] and a calculated Reynolds number of 132,065 has been used to take the concentration polarisation inside the stirred cell into account. Furthermore, data for the required parameters pore size (r_p = 0.45 nm [28]) and equivalent membrane active layer thickness ($?x_e = ?x/A_k = 2.54$ μm [28]) has been documented in the literature for the membrane type DL. The dielectric constant inside the pores has been described in the literature for similar membranes mostly with values between 42 and 63 [23]. Here, 60 was used being in the range of the mostly used values. The membrane charge has been fitted for the filtration experiment with model solution M1 resulting in 132.267 mol/m^3 as the best fitting value.

Comparisons between the experimental results for the filtration of the model solutions M1 and M3 and the calculated values are shown in Figs. 5 and 6. According to the degree of accurateness in the estimation of the

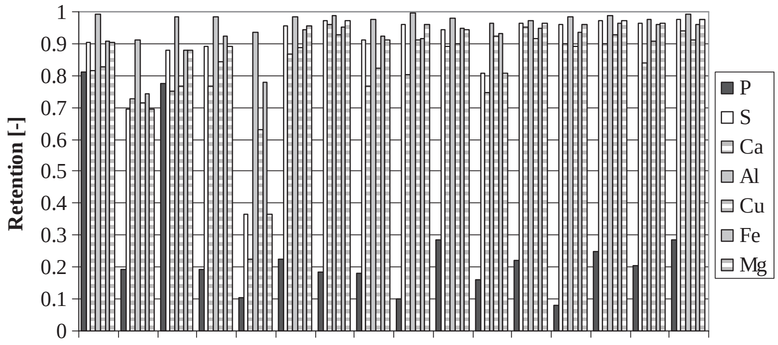

Fig. 4. Retention obtained for the filtration of model solutions.

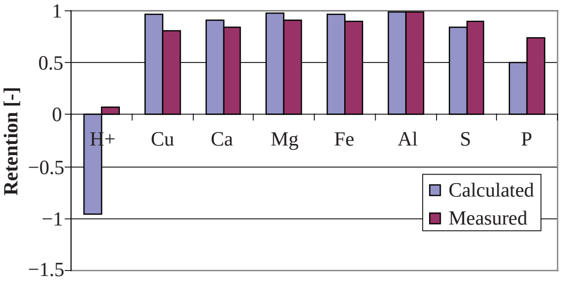

Fig. 5. Comparison of experimentally obtained retention for the model solution M1 and calculated retention using a membrane charge of 132.27 mol/m^3.

input data for the model the calculation presented in this work has the character of a first orienteering study. Nevertheless, it is shown that the data produced based on a relative large amount of assumptions reflects roughly the tendency of the experimental results. For both model solutions the rejections of multivalent cations are slightly overestimated. In the case of sulphuric and phosphoric acid the rejection is underestimated in the case of M1 and overestimated in the case of M3. The difference between the measured and calculated values for the H$^+$-retention is the biggest for both model solutions. This can be explained by the fact that the H$^+$ ion is the most mobile ion and will therefore taken by the model somehow into account for the provision the electroneutrality. For M1 in reality a part of the multivalent cations permeated through the membrane being therefore able to balance the negative charges of the relative small amount of sulphuric and phosphoric acid permeating. According to the modelled rejections there are less multivalent cations on the permeate side and more anions than measured. The therefore relatively big amount of negative charges on the permeate side are counterbalanced by a large amount of H$^+$-ions leading to a very low H$^+$-rejection. According to an analogous argumentation for M3 the underestimation of the anion-rejection could lead to the conclusion of a higher H$^+$-rejection than experimental measured.

These first results of a mass transport shows that the chosen approach turned out to be promising but requiring optimisation. An important method for an improvement is to achieve a better estimation of the membrane parameters. A better understanding of the meaning of the dielectric constant could lead to a more exact determination. The pore size and active layer thickness could possibly be estimated more exactly by applying, e.g. high performance REM measurements or by the conduction of an elaborate experimental series including the filtration of uncharged molecules like glucose.

The exact determination of the membrane charge is complicated and discussed controversial in literature. The membrane charge density is in many cases determined by fitting to experimental results or by measurements of the zeta potential by the conduction of tangential streaming potential measurements. Often the electrolytes used in the fitting experiments respectively the streaming potential measurements differ from the composition of the solution the rejection should be predicted by the mass transport modelling. As described in literature [23] it is therefore difficult and still in a process of research how to obtain exact and consistent values for the membrane charge density.

Another possibility to improve the previous performed modelling is to verify the exact dissociation behaviour of phosphoric and sulphuric acids under the circumstances during the membrane filtration. So far the dissociation behaviour of ideal solutions has been assumed which had to be corrected partly for electroneutrality. This correction has as a first approximation been relatively arbitrarily by changing the ideal dissociation factors of phosphoric acid and sulphuric acid equable until electroneutrality was achieved. A physically consistent simulation of dissociation could improve the quality of the modelling decisively.

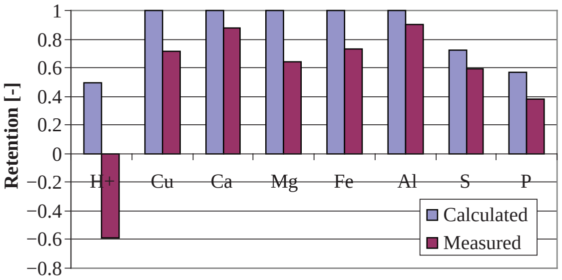

Fig. 6. Comparison of experimentally obtained retention for the model solution M2 and calculated retention using a membrane charge of 132.27 mol/m^3.

5. Conclusion

There are recently theoretical approaches for a mass transport modeling in nanofiltration models described in literature which are capable of taking into account multi component solutions and giving reasonable good predictions about the rejections for all included ions. The DSPM&DE approach was therefore identified of making up a promising possibility for a modeling of the filtration process using pretreated sewage sludge ash eluates adjusted to a relatively low pH of around 1.5. A first application using approximately estimated membrane parameters showed that the tendency of the rejection for most ions could be predicted for an experiment using model solutions representing the real sewage sludge ash eluate. It is expected that an optimised determination of the membrane parameters pore size, equivalent active layer thickness, membrane charge density and pore dielectric constant as well as an advanced simulation of the dissociation behavior of sulphuric and phosphoric acid could decisively improve the prediction quality.

Nomenclature

A_k	membrane porosity
c_i	concentration in membrane of ion i
C	concentration in the bulk phase
e	electronic charge
F	Faraday constant
D_i	diffusion coefficient in the membrane of ion i
$D_{\infty i}$	diffusion coefficient at infite solution of ion i
DSPM&DE	Donnan steric partitioning pore and dielectric exclusion
ENPE	extended Nerst-Planck equation
j_i	ionic flux of ion i
k	Boltzmann constant
K_{ci}	Hinderence factor for convection of ion i
K_{di}	Hinderence factor for diffusion of ion i
M	molecular weight
p	pressure
r_i	Stokes radius of ion i
r_p	membrane pore radius
R	universal gas constant
SCM	space-charge model
T	temperature
TMSM	Theorell-Meyer-Sievers model
v	convective flow in the membrane
V	total volume flux through membrane
$\tilde{V}$	partial molar volume
x	axial position within the membrane
z	valence of ion
ε_0	permittivity of free space
ε_b	bulk dielectric constant
ε_p	pore dielectric constant
$\Delta\psi_D$	Donnan potential
ΔW	Born solvation energy barrier
γ	activity coefficient of ion i within the pore
ψ	electric potential
Φ	uncharged solute steric partition coefficient
Δx_e	equivalent membrane active layer thickness
Δx	effective membrane active layer thickness
Indizes	
b	bulk
p	pore
P	permeat
i	ion i
s	solvent

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