Final report

Project number: K112096

Aims of the project

The aim of our research was to investigate the effect of organic micropollutants and the natural components on the efficiency of membrane separation process during membrane filtration of oil contaminated ground waters, especially by examining the effect of matrix on polarization layer built up in the surface of the membrane, and type and extent of interactions occurs between the material of the membrane and the pollutants. As the main limiting factor of the use of membrane filtration in water treatment technologies is the membrane fouling phenomena, the effect of pretreatments mainly with advanced oxidation processes, and membrane modification were investigated and compared.

Scientific results of the project

According to the research plan, in the first part of the project the necessary control and preliminary studies were evaluated.

1. Evaluation of proper experimental conditions for investigating membrane fouling mitigation caused by organic micropollutants during membrane filtration of oil contaminated underground water:

- *Identification of the most common micro-pollutants, preparation of synthetic waters modelling real oil and organic content of waters and development of analytical and experimental methods for characterization of waters and membrane filtration conditions*

The main organic pollutions of thermal waters were studied and different kind of organic pollutants (crude oil (from Algyő area), petroleum, phenol, humic acid, etc) were selected and purchased for our investigations. For the preparation of oil-in-water emulsion firstly a Hielsher ultrasonic processor was applied, but based on the results of our detailed investigations it was found that the emulsion has stability problems at all the investigated conditions, which resulted in reproducibility- and analytic problems. Therefore further experiments were carried out to optimize the emulsion production method. Finally the preparation of sufficient stable oil-in-water emulsion was carried out by applying a combined method of high speed dispersion and ultrasonic homogenization. Inorganic components of thermal waters were also investigated and for the production of model thermal water 7 different kinds of salts were purchased, which were applied in calculated amounts to provide nearly real conditions for our investigations in some cases. At the final stage of experiments real underground waters were investigated, originated from a thermal well (Szeged, Szentmihály) and produced water (Szeged, Algyő).

Different types of analytic methods were investigated and optimized for the description of the performance of applied purification methods. The calibration of oil analyzer, TOC analyzer and COD measurements were carried out and spectrophotometer was also applied in some cases (e.g. to measure the amount of produced ozone, or for the description of the decomposition of some organic compounds). Gas chromatography was also applied, however in case of complex pollutants (e.g. crude oil) the separation and the identification of different components cannot be realized by the available "single" gas chromatograph. The oil emulsions were characterized by droplet size and zeta potential.

Membrane filtration experiments were carried out to investigate the optimal operational conditions of membrane filtration of crude oil containing stable oil in water emulsions (c_{oil} = 100 ppm; $d_{oil\ droplets}$ < 1.5 μm) using polyethersulfone (PES) microfiltration membrane (d_{pore} = 0.2 μm). The effects of applied transmembrane pressure (TMP), stirring speed were analyzed in detail. Removal efficiency was determined by measuring turbidity, chemical oxygen demand (COD), total organic carbon content (TOC) and extractable oil content (TOG/TPH). It was found that increased stirring speed causes much higher fluxes (at all pressures), while higher TMP test results in lower relative fluxes (at all stirring speed) due to the increased irreversible filtration resistance, and even lower purification efficiency. [i]

- *Choosing and development of a membrane test methods for identification the membrane – matrix interactions*

In order to investigate the membrane-water matrix interaction, contact angle measurements were performed with different types of membrane materials (PES, PTFE, PAN, PVDF membranes). From the water droplet volume changing experiments it was concluded that the increase of droplet volumes had no significant impact on the contact angle values. The membrane pretreatment experiments with water prewetting showed us that the pretreatment resulted in more hydrophilic membrane surface and lower fouling tendency. Based on the results of the fouled membranes experiments it can be concluded that the new membrane had the most hydrophilic surface and the fouled membranes had higher contact angles, due to the hydrophobic character of the fouling components (as oil). The wettability of the membrane surface significantly depends on the chemical nature of the filtered solution; the ozone treated oily waste water samples showed higher hydrophilicity in the fouled membrane surface due to their more polar character caused by ozonation byproducts. [ii]

In order to get more information about the membrane fouling and interactions, the surface characteristics of modified (TiO_2-covered) membranes were investigated beside the contact angle and surface free energy measurements by SEM, and AT-IR measurements. Our results showed, that the contact angle measurements are appropriate to follow the fouling and cleaning process of the membrane by following wettability of the membrane surface. [iii]

2. Membrane fouling mitigation by associated chemical methods: effects of ozone, Fenton-reaction and heterogeneous photocatalysis. Examination of fouling mechanisms at AOP's treated and natural model solutions.

- *Effects of ozone pretreatment on membrane fouling*

Membrane filtration was applied for the purification of ozone-pretreated crude oil containing oil in water emulsions (c_{oil} = 100 ppm; $d_{oil\ droplets}$ < 1.5 μm) using polyethersulfone (PES) microfiltration membrane (d_{pore} = 0.2 μm). The effect of ozonation on the size of oil droplets and on zeta potential was determined by dynamic light scattering measurements. Results pointed out, that short pre-ozonation causes increased fluxes and decreased resistance without notable change in the purification efficiency in case of low transmembrane pressure (TMP) (0.1 MPa). However longer pre-ozonation or higher transmembrane pressure results in increased irreversible resistance, lower permeate fluxes or even lower purification efficiency[(i)].

- *Effects of coagulation-flocculation and Fenton pretreatment on membrane fouling*

Before the investigation of the effect of iron-salt using Fenton-preteratment, as control experiments the same model emulsion was treated by simple coagulation/flocculation, combined with membrane separation. For the chemical treatment iron (III) chloride, polyaluminum-chloride as coagulant and anionic polyelectrolyte as flocculant were applied. It was found that the pretreatment does not affect the purification efficiency (it is above 90% in all cases), but the flux considerably increases with the pretreatment.[iv] In further experiments Fenton reaction, UV/H_2O_2 combined treatment and photo-Fenton reaction were combined with membrane separation. Among the investigated advanced

oxidation processes, photo-Fenton treatments showed emergent purification efficiency during the pre-treatment period. It also was found that after the treatment with photo-Fenton reaction, adjusting the pH to 7, iron-hydroxide was precipitated, which by adhering the residual oily contaminants caused increased flux during the microfiltration of the waste water.[v] **The Fenton-type and coagulation/flocculation pretreatment in all cases results in decreased membrane fouling; both reversible and irreversible fouling decreased**.

- *Effects of membrane surface modification and heterogeneous photocatalytic pretreatment on membrane fouling, evaluation of fouling models*

To investigate the effect of hydrophilicity on membrane fouling different type of membrane materials (PAN[vi], PVDF[iii], PES[vii]) were modified by titanium-dioxide coating, achieving a more hydrophilic membrane surface. It was found that the hydrophilic character of the surface is proportional with the amount of the titanium dioxide. The investigation of the fouling propensity of the neat and modified membranes showed that the more hydrophilic surfaces are less prone to be fouled than the neat membranes. The fouling mechanism is highly determined by the nature of the membrane and the foulant, but although in case of oil emulsions the reversible fouling is more determinant[(iii, vi)] while in case of protein-containing waste waters[viii] the irreversible fouling is higher, the increased hydrophilicity results in membrane surfaces which are less prone to be fouled. In addition, the **coating of the membrane by a photocatalyst makes it possible to clean the fouled membrane by UV irradiation, without using any chemical cleaning agents**.

In another series of experiments beside TiO_2 carbon nanotubes (CNT) were applied to prepare modified membranes. Applying either solely TiO_2 or solely CNT coating resulted in significant reduction of total filtration resistance in both cases, however the combination of them (TiO_2 and 1% CNT) resulted in by far the highest flux and lowest resistance; moreover enhanced photocatalytic efficiency of the composite also was achieved. [ix]

As a result of these experiments, **description of the membrane-hydrocarbon pollutant interactions was evaluated**. The wettability of neat, modified, fouled by oily contaminants and UV-cleaned membranes give us information about the membrane-hydrocarbon interactions. The result show that the hydrocarbons have a relatively strong bonding to the neat membranes, the relative fluxes considerably decrease during filtration, and after the filtration the oil layer cannot be eliminated, the surface remains highly hydrophobic due to the hydrophobicity of the oil layer. In case of modified hydrophilic membranes, the surface "repels" the oil layer, the flux remains high, after a slight rinsing and UV-cleaning the original properties of the membrane can be recovered. On the other hand, the wettability of the membrane alone does not provide enough information about the membrane-foulant interactions. To obtain more information about the interactions and membrane surface properties, SEM images were obtained and these images confirm the results that the membrane hydrophilicity is proportional to the coverage of the membrane. Beside the contact angle measurements, surface free energies also were measured of different types of membranes, showing that in some cases the surface free energy gives us more information about the fouling propensities than wettability.

The aim of the project also was describing the fouling mechanisms of AOP's treated samples. During the evaluation of filtration experiments generally two types of models were used to obtain information about the fouling mechanisms: the resistance-in-series model, calculating the reversible and irreversible filtration resistances[x]; and the classic Hermia models, which make it possible to determine the exact fouling mechanism (complete blocking, intermediate blocking, standard blocking, and cake layer formation or their variations) by fitting models to measured data. It was found that during the filtration the mechanism may change; even if at the beginning of the filtration pore fouling may occur, at the stage of the stationary filtration in most cases the cake-layer formation is determinant due to the coalescence of the oil droplets. The short time oxidation of other types of pollutants (e.g. humic materials[xi] or proteins[xii]) may cause microflocculation and result in decreased irreversible fouling due to changing of the main fouling mechanism from pore blocking to cake layer formation. On the other hand, the longer oxidation degrades both oil droplets, and other types of pollutants to small molecules, causing is some cases more severe pore fouling (increasing irreversible resistance during filtration).

Thus, it can be concluded that the degree of oxidation is a crucial parameter of the pre-treatment which must be optimized during development of an oxidation pretreatment method[i, xiii].

3. Effect of inorganic components of the water matrix on the reactions during advanced oxidation processes, modelling the effect of inorganic components on membrane filtration prameters

The effects of the salt content on the properties of the oil emulsions, as oil droplet size and zeta potential were investigated to determine the characteristics of the different emulsions. It was found that increasing the salt content of the model wastewaters, oil-in-water emulsions, increased the zeta potential and the size of the droplets. The zeta potential of crude oil droplets dispersed in saline water, are dependent on the pH and ionic strength. The matrix effect was investigated in case of ozone treatment and heterogeneous photocatalysis, as advanced oxidation processes.

- *Matrix effect in case of pre-ozonation*

In case of ozone pre-treatment oil in water emulsions (coil = 100 ppm; $d_{oil\ droplets} < 2$ μm) and in some cases 20 ppm humic acid containing oily waters were ozonated in distilled water and model thermal water matrix. It was found that the matrix significantly affected the size distribution and adherence ability of oil droplets onto the membrane surface, therefore fouling mechanisms also were strongly dependent on the matrix. In case of low salt concentration, the total resistance was caused mainly by reversible resistance, which could be significantly reduced (eliminated) by pre-ozonation. In case of model groundwater matrix, nearly twice higher total resistance was measured, and irreversible resistance was dominant, because of the higher adhesion ability of the oil droplets onto the membrane surface. In this case, pre-ozonation resulted in much lower irreversible, but higher reversible resistance. Increased duration of pre-ozonation raised the total resistance and reduced the elimination efficiency (due to fragmented oil droplets and water soluble oxidation by-products) in both cases. The chemical oxygen demand increased by short term ozone treatment, probably due to ozonide formation, while further treatment decreased the COD; the oxidation rate was higher in model thermal waters, due to the reactions of the radicals (mainly hydroxyl radicals) formed from degradation of ozone in alkaline solutions. This also was confirmed by the results of ozonation of humic acid containing oily thermal waters; it was found that in alkaline solutions the COD diminishing rate is lower in presence of humic acid, while in acidic solutions the presence of humic acid did not affected on the oxidation rate. Summarizing these results it can be concluded that **short pre-ozonation can be recommended both from economic and performance aspects**.[xiii, iii, xi)]

- *Matrix effect in case of heterogeneous photocatalysis combined membrane filtration*

The heterogeneous photocatalytic experiments were carried out in the following way: after oil-in-water emulsion filtration, the membrane cleanability by means of photocatalysis (without any additional chemicals) was investigated. The fouled membranes were taken out of the cell and rinsed with distilled water to remove the oil layer if possible. Then, the membranes were irradiated with UV light. During UV irradiation the surface hydrophilicity was increased, and nearly total flux recovery was achieved, showing that the fouled membranes can be effectively cleaned with UV irradiation. It also was observed that the relative flux recovery was the most effective in case of the 2500 mg L^{-1} salt concentration. **The efficiency of the heterogeneous photocatalysis is determined by** several factors and these factors affect on the degradation efficiency in a very complex way: beside the **effect of water matrix (ionic strenght, pH, inorganic salt content**), the degradation is determined by **the adsorption of the pollutants on the catalyst surface**; in case of 2500 mg/L salt concentration more oil was adsorbed on the TiO_2 surface (according to the contact angle and filtration resistance results) which may have been more easily available for photocatalytic degradation. The chemical changes during cleaning were followed and characterized by ATR-IR.[iii]

For the deeper explanation of the mechanism of the filtration, the fouling mechanisms were modelled by using Hermia filtration laws (such as complete-, internal- and intermediate pore blocking and cake layer formation). It was found that in these systems fouling cannot be described by only one given fouling model, since different mechanisms occur simultaneously. In case of model groundwater very different fouling mechanisms were found. Without pre-ozonation, the cake filtration was dominant in accordance with the observed thick contaminant cake layer. In parallel with the duration of pre-ozonation, the pore blocking models could be fitted more reliably. In case of long-term pre-ozonation, internal- and intermediate pore blockings showed the best fits. These observations are in good agreement with the measured intensive fragmentation of the oil droplets during pre-ozonation in case of the model groundwater matrix, where smaller than 100 nm oil droplets were also generated during the pre-treatment. However, in spite of the decreasing role of the cake layer formation, the reversible resistances increased, which means that beside fragmentation, the electrostatic repulsive interactions determine the fouling propensity of the membrane. Independently from the water matrix, microfiltration of slightly pre-ozonized oil emulsions can be described mostly by cake layer formation model, but in case of model groundwater, longer pre-ozonation caused the blocking of the pores due to the intensive fragmentation of the oil droplets, which can be attributed to the advanced oxidation ability of OH• radicals, which can be produced at the higher pH value (~8.2) of model groundwater.[xiii]

In case of TiO_2 modified membranes the effect of salt content on the filtration resistances was found to be contradictory; while in case of neat PVDF membrane, the salt content decreased the irreversible fouling, in case of TiO_2 covered surfaces it was observed that the increased salt content increased the irreversible fouling. Although decreased fouling could be expected due to higher magnitude of the zeta potential of the emulsion at higher salt content, the slightly increased irreversible (non-washable) resistances can be explained by the DLVO theory, as increase in ionic strength lowers the energy barrier and hence favours. In case of TiO_2 covered surfaces the lower salt concentration resulted in lower filtration resistances, probably due to increased surface zeta potential resulting in increased repulsive electrostatic forces. Furthermore, at higher salt concentrations the magnitude of zeta potential of the surface may be decreased, as earlier studies showed, leading to increased role of the attractive van der Waals interactions, and thus the adhesion.[iii]

- *Determination of the optimum parameters of AOP's at different matrices and modeling of the fouling mitigation effect of the oxidation*

The optimum parameter determination of oxidation pretreatments were based on model calculations. In case of ozone pretreatment it was found that short (1-5 min) pre-ozonation enhanced the fluxes in all investigated matrices due to the higher negative surface charge of the oil droplets, resulting increased repulsive force between the negatively charged membrane surface and the oil droplets. Parallelly, notable reduction of the total resistances was observed in both cases, while further pre-ozonation decreased the fluxes and increased the irreversible resistance and therefore the total resistance as well due to the production of smaller and less hydrophobic oil droplets, which were adhered on the membrane surface. Moreover, while short pre-ozonation significantly increased the negative surface charge of the droplets, further ozonation caused only a slight increase in the zeta potential values. However, pre-ozonation significantly reduced the adherence ability of the contaminants (due to increased zeta potentials), longer pre-ozonation increased both the irreversible and reversible resistances due to the intensive fragmentation of the oil droplets. [I, xiii]

To compare the performance of different AOPs, Oxygen-equivalent Chemical-oxidation Capacity (OCC, kg O_2/m^{-3}) also was used to quantify the oxidants used in the ozone treatment and Fenton process. It was found that the effect of ozone treatment and Fenton-treatment is different in the case of initial normalised flux. Not only Fenton pretreatment but the addition of reagents without hydrogen peroxide has coagulation-flocculation effect, resulted in enhanced initial flux. In case of Fenton reaction this effect is independent from $[H_2O_2]$:[Fe] ratio. The fouling coefficient also changes by addition of oxidants, but in this case the tendency is more likely depends on OCC than on the applied AOP method. At lower oxidation capacities the fouling coefficient decreases resulted in lower fouling than in non-treated solutions, however at higher oxidation grade fouling coefficient is increasing.[xiv]

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4. Organic micropollutant elimination from real waters by membrane separation

- *Organic micropollutant elimination from real waters by membrane separation*

Produced water (pre-cleaned by traditional techniques) were used to comparing membrane filtration efficiency of PTFE, PES and PVDF ultrafiltration membranes, and PVDF membranes modified with TiO_2 or TiO_2+CNT coating. Investigation of available fluxes, filtration resistances ad flux recovery and flux reduction ratios showed that the highest fluxes could be achieved using PVDF membranes modified with photocatalyst nanoparticles. The lowest filtration resistances evolved in case of modified membranes, while the flux recovery ratios were the highest in case of PES and modified membranes. It also was proved that the modified membranes were cleanable with UV irradiation due to their photocatalytic activity.

- *Membrane fouling mitigation by associated chemical methods in real underground waters*

To compare the performance of different AOPs during waste thermal water treatment, Oxygen-equivalent Chemical-oxidation Capacity (OCC, kg O_2/m^{-3}) was used to quantify the oxidants used in the ozone treatment and Fenton process. It was found that the effect of ozone treatment and Fenton-treatment is different in the case of initial normalised flux. Not only Fenton pretreatment but the addition of reagents without hydrogen peroxide has coagulation-flocculation effect, resulted in enhanced initial flux. In case of Fenton reaction this effect was found to be independent from $[H_2O_2]$:[Fe] ratio. The fouling coefficient also changes by addition of oxidants, but in this case the tendency is more likely depends on OCC than on the applied AOP method. At lower oxidation capacities the fouling coefficient decreases resulted in lower fouling than in non-treated solutions, however at higher oxidation grade fouling coefficient is increasing.[xiv] As the thermal water had very high ion content (mainly HCO_3^- and Cl^-), and relatively high organic content (phenols, hydrocarbons and humic substances), this composition of waste water resulted in a very complicated reaction system for advanced oxidation processes; where inorganic anions may inhibit the oxidation reaction of ozone and also the Fenton-like reactions, while aromatic compounds may catalyze them. In this system the photo-Fenton reaction was found to be the most effective method for degradation of organic pollutants due to its high hydroxyl radical generation ability. The investigation of the effect of different light sources (UV tubes with λ_{max} = 360 or 254 or 254/180 nm and visible light emitting tube) also revealed, that both the purification efficiency, and the COD or TOC elimination "quantum yield" of photons increases with the energy of photons due to their hydroxyl radical producing reactions.

Comparison of filtration of produced water with ozone-pretreatment and photocatalyst modified membrane filtration revealed that higher flux enhancement could be achieved with modified membranes. Although ozone pretreatment increased the purification efficiency (87-91%), it did not achieved the purification efficiency of TiO_2 or TiO_2+CNT modified membranes (97,5-99%).

- *Cost estimation for treatment of underground waters by combined processes*

Evaluation of the cost estimation of advanced oxidation processes alone were based on the data of a real thermal water treatment, while the cost estimation of combined processes was based on measurements of model waste waters. As AOP's, ozone, Fenton and photo-Fenton treatments were compared on the basis of OCC, while the cost estimation of membrane filtration processes were based on ultrafiltration of pretreated waters and the costs of pretreatments. During calculations 1000 m^3/day feed volume were assumed. In case of AOP's, the target value was 500 mg/L COD decreasing and total phenol elimination. In case of combined methods the costs of pretreatments were considered to be proportionally lower, due to lower chemical oxidation capacity demand. Amortization was assumed to be 10 years. Taking into consideration the capital investments and operational costs, the total cost of 1 m^3 waste water purification was calculated. (Table 1.)[xv] The results obtained from the calculations based on model and real waste waters were similar, showing validity of our model waters.

Table 1. Estimated costs of investigated

	Method	Cost (HUF/m^3 waste water)
AOP's alone	Ozone (pH=8)	25000
	Fenton	1138
	photo-Fenton	902
Membrane filtration	Ultrafiltration (UF)	54
Combined methods	ozone pretreatment +UF	228
	UF+TiO_2	18
	UF+TiO_2+CNT	9
	Fenton + MF	17
	photo-fenton + MF	14

Summary of the results

During the research period the results were presented in international and national conferences; 20 presentation in English, and 8 presentation in Hungarian. 14 original research articles were already published (IF=15.332), and four further article is under publication. Beside the scientific publications 12 diploma thesis, and one PhD thesis were related to these topics.

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