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Henrik K. Hansen[a]; Lisbeth M. Ottosen[a]; Arne Villumsen[a]
[a] TECHNICAL UNIVERSITY OF DENMARK, LYNGBY, DENMARK

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Electrical Resistance and Transport Numbers of Ion-Exchange Membranes Used in Electrodialytic Soil Remediation

HENRIK K. HANSEN,* LISBETH M. OTTOSEN,
and ARNE VILLUMSEN
DEPARTMENT OF GEOLOGY AND GEOTECHNICAL ENGINEERING
BUILDING 204, TECHNICAL UNIVERSITY OF DENMARK
DK-2800 LYNGBY, DENMARK

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ABSTRACT

Electrodialytic soil remediation is a recently developed method to decontaminate heavy metal polluted soil using ion-exchange membranes. In this method one side of the ion-exchange membrane is in direct contact with the polluted soil. It is of great importance to know if this contact with the soil causes damage to the membrane. This work presents the result from transport number and electrical resistance measurements done on four sets of ion-exchange membranes (Ionics, Inc. CR67 HMR412 cation-exchange membranes and Ionics, Inc. AR204 SXZR anion-exchange membranes), which have been used in four different electrodialytic soil remediation experiments. The experiments showed that after the use in electrodialytic soil remediation, the ion-exchange membranes had transport numbers in the same magnitude as new membranes. The electrical resistance for six membranes did not differ from that of new membranes, whereas two membranes showed a slightly increased resistance.

Key Words. Ion-exchange membranes; Electromotive force; Transport numbers; Electrical resistance; Electrodialytic soil remediation; Heavy metals

INTRODUCTION

Processes to decontaminate heavy metal polluted soil on a large scale by using a DC current as cleaning agent are at present under development (1–3).

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One of these methods includes ion-exchange membranes in the process in a way that optimizes the electric current on the heavy metals in the soil, and avoids precipitation of heavy metal salts in the soil due to alkaline conditions. This method is referred to as electrodialytic soil remediation (4, 5). Previous studies (6) showed that the ion-exchange membranes to be used in electrodialytic soil remediation were nearly ideal in the presence of Na, Ca, Zn, and Cu chloride solutions, and that it was possible to remove all ions from a simulated contaminated soil solution. A question raised when using ion-exchange membranes in electrodialytic soil remediation was: Do the ion-exchange membranes foul or decompose during the remediation experiments? This would result in deviation from ideality in permselectivity of the membranes (e.g., the transport numbers of counterions are different from 1) and in an increment of the electric resistance over the membranes during the experiments. This has special importance when extending the electrodialytic remediation principle to large-scale equipment. Here it is an obligation that ion-exchange membranes work satisfactorily even in remediation experiments of several months duration. It was discovered elsewhere that similar polymeric ion-exchange membranes have a limited lifetime due to fouling or degradation by either humic substances (7, 8), iron species (9), or colloids (10). Therefore it was regarded necessary to use new ion-exchange membranes in each remediation experiment, which resulted in high membrane purchasing costs. If the membranes worked satisfactorily after a remediation experiment, it should be possible to reuse them in further remediation experiments.

The scope with this paper was to investigate the transport numbers and electrical resistance of ion-exchange membranes after use in four different cases of electrodialytic remediation. The experiments were made on four different soils sampled at heavy metal polluted sites. The soils differed in composition (including organic content), pH, pollution (heavy metals), and water content. The remediation time, the electric current density, and the potential drop were also different in the four experiments. It was important to see if any irreversible degradation or fouling had occurred during the different remediation experiments, and knowledge of the transport numbers and electrical resistance of the membranes after the remediation experiments would help to clarify this.

BACKGROUND

Electrodialytic remediation of soil polluted with heavy metals is given in principle in Fig. 1. After remediation the heavy metals end up in compartments II and IV, the cations in II, and the anions in IV. Membranes 2 and 3 are in contact with the soil on one side of the membrane and with an aqueous solution on the other side.

The transport number and electrical resistance of the ion-exchange membranes is of great importance, especially with respect to the efficiency of the

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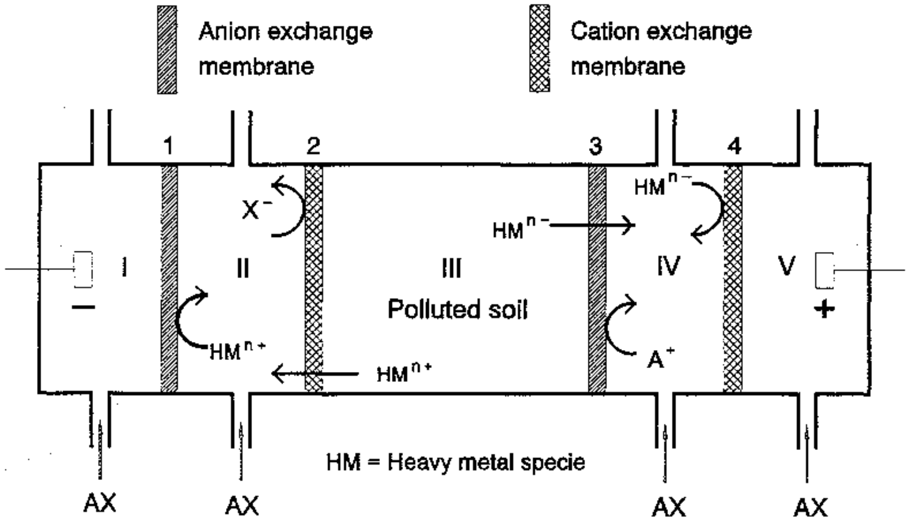

FIG. 1 The principle of electrodialytic soil remediation.

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remediation process and the energy consumption. If the membranes either foul or decompose during the remediation process, it would mean both a loss in the selectivity of the membranes (resulting in a high transport of co-ions across the membrane) and a loss in effective pore volume in the membranes (with an increased electrical resistance across the membranes as a result). The ultimate consequence would be that the membrane decomposes totally, which means free transport of ions and solution both from the soil to the electrode units or vice versa.

The parameters of the remediation process which could affect the properties of the membranes are mainly:

a) *pH*. The durability of the membranes is highly pH dependent. Ionics, Inc. gives the pH range where their membranes should work satisfactorily as 0–10 for anion-exchange membranes and 0–11 for cation-exchange membranes (11).
b) *Water content*. The water content of the soil is important, especially when it is known that in order to maintain their stability ion-exchange membranes should not be allowed to dry. In the electrodialytic soil remediation method, electroosmosis in the soil will move water toward the cathode, and electroosmosis in anion-exchange membrane 3 in Fig. 1 will drive water toward the anode, resulting in dewatering of the soil closest to this membrane.
c) *Temperature*. The temperature during the remediation process has importance for the degradation of the membranes; at higher temperatures the degradation occurs more rapidly.

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d) *Organic matter*. The content of organic matter in the soil is somewhat related to the amount of bacteria in the soil, and bacterial activity can affect the membranes. Furthermore, humic substances can foul the membranes.
e) *Heavy metal species*. The heavy metals bind differently in ion-exchange membranes, and especially cations such as Hg^{2+} or Pb^{2+} could adsorb to the exchange sites in the cation-exchange membrane pores and hinder the transport of other cations, resulting in a higher electrical resistance.
f) *Remediation time*. A long remediation time means a longer exposure to different attacks.
g) *Electrical parameters*. Depending on the such electrical parameters as current density and voltage drop over the remediation cell could lead to higher temperature in the soil/membranes, exceeding the limiting current of the membrane/soil interface and resulting in water splitting or transport of charged (or uncharged) particles toward the membranes.

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The transport numbers of a membrane can be found by measuring the electromotive force across the membrane. Using Ag/AgCl electrodes as measuring electrodes, the total potential drop (e.g., the electromotive force) between the two electrodes and across the membrane in NaCl solutions can by expressed as follows (12):

$$\mathrm{emf}_{(\mathrm{r-l})} = -\frac{\nu}{\nu_-}\frac{RT}{F}t_+\ln\frac{a_{(\mathrm{r})}}{a_{(\mathrm{l})}} \tag{1}$$

where $\mathrm{emf}_{(\mathrm{r-l})}$ is the electromotive force calculated from the right side to the left side, ν is the total number of ions formed by dissociation of an electrolyte, ν_- and ν are the stoichiometric numbers of the salt, and $a_{(\mathrm{r})}$ and $a_{(\mathrm{l})}$ are the activities of the salt on the right and left side of the membrane, respectively.

The transport number of the cation, t_+, is then derived as

$$t_+ = -\frac{\nu_- F\,\mathrm{emf}_{(\mathrm{r-l})}}{\nu RT\ln\dfrac{a_{(\mathrm{r})}}{a_{(\mathrm{l})}}} \tag{2}$$

The transport number of the anion, t_-, can be found from the definition

$$t_+ + t_- = 1 \tag{3}$$

EXPERIMENTAL

Membranes

The cation-exchange membranes used were CR67 HMR412 (Ionics, Inc., USA), and the anion-exchange membranes AR204 SXZR 3679 (Ionics, Inc., USA).

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TABLE 1
Initial Soil Conditions

Remediation experiment	Soil	Pollutants	Organic matter (%)	pH	Water content (%)
1	Sand	Zn (705 ppm) Pb (290 ppm) Cu (490 ppm)	4.4	6.8	12
2	Sand	Hg (1200–1900 ppm)	0.5	8.6	9.8
3	Sandy loam	Cu (1300 ppm) Pb (700 ppm)	1.8	6.4	15
4	Loamy sand	Cu (310 ppm) Cr (855 ppm) As (770 ppm)	0.7	6.1	30

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Chemicals

The chemical used was Merck pro analysi sodium chloride, used without any further purification.

Description of the Remediation Experiments

The remediation conditions are given in Tables 1–4 for the four cases. The membranes used were all in direct contact with soil during the remediation experiments, and would therefore be directly exposed to chemical or microbiological influence. The remediation cell was a cylindrical, closed glass cell of 8 cm inner diameter, and 15 cm length. Details on electrodialytic remediation experiments on polluted soil can be found elsewhere (4, 5, 13, 14). Table 1 gives the initial soil conditions, Table 2 the electrolyte solution parameters

TABLE 2
Conditions of the Electrolytes during Electrodialytic Remediation

Remediation experiment	Electrolytes in compartment II and IV	pH in compartment II and IV
1	0.01 M $NaNO_3 + HNO_3$ (II and IV)	1.5–11 (II) 1.5–2 (IV)
2	0.01 M $NaNO_3 + HNO_3$ (II and IV)	2–3 (II and IV)
3	0.01 M $NaNO_3 + HNO_3$ (II and IV)	2–3 (II and IV)
4	0.01 M $NaNO_3 + HNO_3$ (II and IV)	1.5–10 (II) 1.5–2 (IV)

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TABLE 3
Final Conditions of the Soil

Remediation experiment	Soil closest to the cathode: Water content (%)	pH	Pollutants	Soil closest to the anode: Water content (%)	pH	Pollutants
1	10.2	7.0	Zn (720 ppm) Pb (300 ppm) Cu (360 ppm)	14.5	3.6	Zn (300 ppm) Pb (250 ppm) Cu (200 ppm)
2	13.6	4.2	Hg (1500 ppm)	5.6	3.4	Hg (1350 ppm)
3	14.6	6.0	Cu (1200 ppm) Pb (650 ppm)	6.9	3.9	Cu (600 ppm) Pb (550 ppm)
4	25	4.0	Cu (220 ppm) Cr (780 ppm) As (450 ppm)	17.5	3.0	Cu (75 ppm) Cr (780 ppm) As (660 ppm)

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during electrodialysis. Table 3 the final parameters of the soil closest to the membranes 2 and 3 (see Fig. 1), and Table 4 the parameters during the remediation experiments.

Apparatus

Figure 2 shows the measuring cell, which consisted of two separable polyacryl compartments, used for both transport number and electrical resistance measurements. The membrane was placed between the compartments, and the membrane area was 1.0 cm^2. The measuring cell was connected with two conical flasks each of 500 cm^3 volume for transport number determination or 1 L for electrical resistance determination, where the solutions were placed and recirculated by Eheim 1041 pumps. The pumps were regulated so that they had

TABLE 4
Parameters during Remediation

Remediation experiment	Voltage drop range (V)	Current range (mA/cm^2)	Remediation time (days)	Temperature (°C)
1	5–90	0.2	39	20–25
2	20–135	0.06–0.08	54	20–25
3	10–50	0.1–0.2	30	40
4	20–90	0.2	65	20–25

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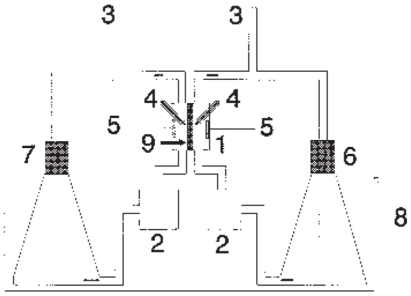

FIG. 2 Experimental setup. 1: The membrane measuring cell. 2: Eheim No. 1046 recirculation pumps. 3: Thermometers. 4: Measuring electrodes. 5: Current electrodes. 6: Solution on the right side of the membrane. 7: Solution on the left side of the membrane. 8: Thermostatic bath (25°C). 9: Membrane to be measured.

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the same flow rates in order to avoid any addition of streaming potential to the total potential difference measured. The conical flasks were placed in a thermostatic bath kept at 25°C (−0.1°C). The temperature (25°C) was measured in the solutions immediately after leaving the two half-cell compartments as shown in Fig. 2. Ag/AgCl electrodes (produced in the laboratory by electrochemical chloridification of a Ag-rod in HCl, see Ref. 12) were used for determination of the potential difference for the transport number determination, and Radiometer calomel electrodes (REF402) for the electrical resistance determination. Platinized titanium electrodes (BAC, Denmark) were used as current electrodes for the electrical resistance measurements. A Fluke 37 Multimeter with an internal resistance of 20 Mohm was used as the voltmeter, and a Hewlett-Packard E3612A power supply generated DC current for the measurement of the electrical resistance.

Procedures

Transport Number Determination

The membrane to be analyzed was placed in a 1 M NaCl solution for 24 hours in order to have the membrane on either the Na-form (cation-exchange membrane) or the Cl-form (anion-exchange membrane).

The membrane was thereafter washed with deionized water until no change in conductivity of the washing water could be detected, and then was placed in the cell at 1 in Fig. 2. Approximately 500 mL 0.001 M NaCl solution was recirculated on the right side of the membrane (**6**) and 500 mL 0.1 M NaCl solution was recirculated on the left side of the membrane (**7**) for approximately 1 hour to equilibrate the system of solutions and membrane.

The solutions were removed, and approximately 500 mL new solution with the same concentrations was filled in the system on the right side (0.001 M

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NaCl) and on the left side (0.1 M NaCl) of the membrane, and were recirculated. After approximately 15–20 minutes the potential difference reached a constant value, which was noted and used for transport number determination.

Electrical Resistance Measurements

After the transport number measurements the same membrane piece was used for determination of the electrical resistance. The two Ag/AgCl electrodes were exchanged with two calomel electrodes, and two platinized titanium electrodes were placed in the cell (at **5** in Fig. 2), and the 500 cm^3 flasks were exchanged with two 1 L flasks. 300–400 cm^3 0.1 M NaCl was placed in each flask for equilibrating the system, and recirculation was started. After 30 minutes the solutions were removed and 1 L 0.1 M NaCl was placed in each flask. A current density (I) of 2.00 mA/cm^2 was applied across the membrane, and the potential difference between the calomel electrodes was noted (U(m)). The same was done with current densities of 5.00 and 10.00 mA/cm^2. The used current densities were far under the limiting current density for the membranes. The pumps were stopped, the membrane piece was removed, and the recirculation was started again. The potential difference between the current electrodes was measured without the membranes (U(O)) for current densities of 2.00, 5.00, and 10.00 mA/cm^2. The area-specific electrical resistance (R) could now be estimated from the difference in potential drop between the calomel electrodes with and without the membrane for the three current densities using:

$$R = \frac{U(m) - U(0)}{I} \tag{4}$$

and an average was calculated. The concentration in the solutions could be regarded as constant during the experiment due to the low current densities and the large volume of solution. The distance between the electrodes was 1.00 ± 0.05 cm, meaning that the membrane thickness (approximately 0.06 cm) was negligible in comparison with the whole distance.

RESULTS AND DISCUSSION

The transport numbers of the four cation-exchange membranes are shown in Table 5, where the transport number of a new cation-exchange membrane (C5) also is given. The numbers C1 to C4 refers to the cation membranes used in remediation experiments 1 to 4 as given in Tables 1–4. The transport numbers are given as an average (including the standard deviations) of three different pieces of one membrane used in the remediation experiments. In Table 5 the transport numbers of the four anion-exchange membranes are also listed together with the transport number of a new anion-exchange membrane (A5)

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TABLE 5
The Transport Numbers of Cation- and Anion-Exchange Membranes after Electrodialytic Soil Remediation

Membrane	Transport number (t_+)	Membrane	Transport number (t_-)
C1	0.97 ± 0.01	A1	0.95 ± 0.02
C2	0.96 ± 0.02	A2	0.96 ± 0.02
C3	0.98 ± 0.01	A3	0.95 ± 0.01
C4	0.97 ± 0.01	A4	0.96 ± 0.02
C5	0.98 ± 0.01	A5	0.96 ± 0.01

calculated after the principle given earlier. Here it could be expected that the uncertainty of the measuring method would be larger because the electromotive force measured should be close to zero.

Furthermore, these values can be compared with data given by the manufacturer (Ionics, Inc. (11)). Here the current efficiency is given as the fraction of current carried by the cation only and was 0.99 (in 0.01 M NaCl) and 0.94 (in 0.1 M NaCl) for the cation-exchange membrane CR 67 HMR-412, and $>$0.99 (in 0.01 M NaCl) and 0.97 (in 0.1 M NaCl). It can be seen that the transport numbers for the membranes used in electrodialytic remediation do not vary from the values of the new membranes.

The area-specific electrical resistances for the four sets of cation- and anion-exchange membranes used in soil remediation together with the electrical resistance of new membranes are given in Table 6. The values are taken as an average of three pieces of the same membrane used in the remediation experiments. The standard deviations are given as well.

The electrical resistance given by the manufacturer is 6 ohm·cm^2 for the cation-exchange membrane and 6.0 ohm·cm^2 for the anion-exchange mem-

TABLE 6
The Area-Specific Electrical Resistance of Different Membranes

Membrane	Area-specific electrical resistance (ohm·cm^2)	Membrane	Area-specific electrical resistance (ohm·cm^2)
C1	6.5 ± 0.5	A1	5.9 ± 0.4
C2	5.6 ± 0.6	A2	6.6 ± 0.5
C3	6.7 ± 0.4	A3	6.6 ± 0.4
C4	8.5 ± 0.8	A4	8.1 ± 0.7
C5	6.2 ± 0.4	A5	6.3 ± 0.3

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brane in 0.1 M NaCl (11). A visual evaluation of the membranes found that the membranes had a brownish color in all cases. In two cases (A1 and A4) the membranes had small, insoluble particles attached to the surface, which could be removed by cleaning with a wet tissue cloth. This could indicate that small colloidal particles were moved by electrophoresis toward the anode, but that they could not pass the anion-exchange membranes due to their size, which was larger than the pore diameter. The small particles could also be some precipitation products. The particles did not seem to have any influence on the transport properties of the membrane after the remediation experiments, but could have had a negative effect during remediation, e.g., by covering a part of the membrane and hindering anions from passing the membrane.

Elevated temperature did not seem to influence the electrical membrane properties. The transport numbers and the electrical resistance for the membranes after 1 month at 40°C were the same as for the new membranes.

The limiting current densities for the anion-exchange membranes were exceeded in all four remediation experiments, as demonstrated by the lowering of pH in the soil closest to the anode. However, the water splitting resulting from these current densities did not affect the electrical properties of the anion-exchange membranes.

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CONCLUSIONS

It was found from the experiments that all the ion-exchange membranes used for electrodialytic soil remediation kept their permselectivity after remediation experiments of approximately 3 months duration with direct contact to soil. This indicated no degradation of the membranes resulting in holes through which cations and anions could be transported equally.

The electric resistance across the membranes did not increase for six of the membranes, and a slightly increased resistance over the membranes was observed in the experiment of longest duration (3 months). This shows that no important fouling of the membranes had occurred.

The color of the membranes turned brown in each case, indicating some change in the membrane structure/surface, but this had no effect on transport through the membranes. The pH ranged between 1.5 and 11, the temperature between 20 and 40°C, the current densities exceeded the limiting current densities for the anion-exchange membrane, and a voltage drop greater than 100 V did not affect the electrical properties of the membranes.

It can be concluded that all the membranes investigated could be reused in further remediation experiments, although the remediation time could be of crucial importance due to the increased electrical resistance in the experiment of longest duration.

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