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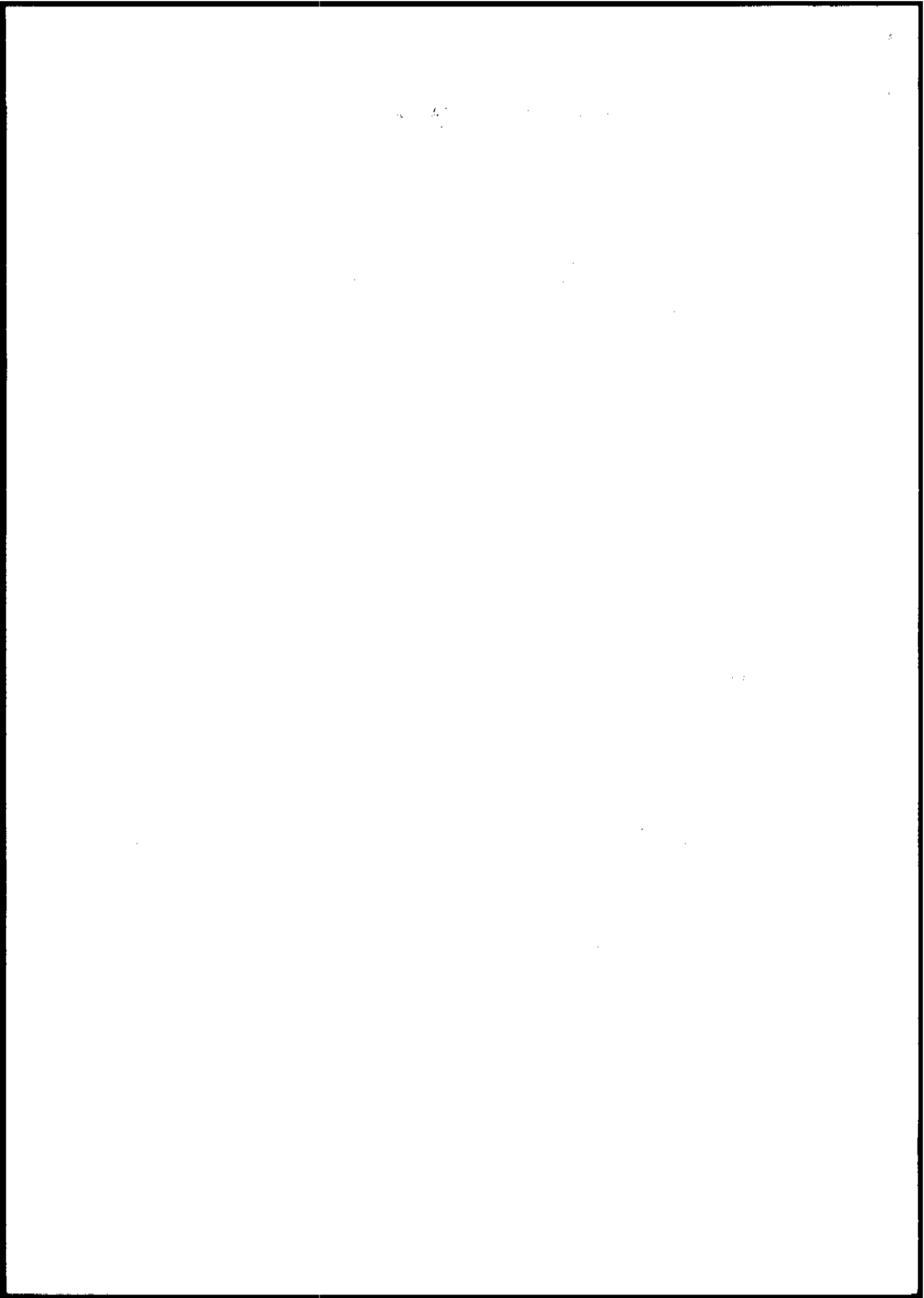
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Biological/Physical Chemical Nitrate Removal from Groundwater
Basic Design Criteria and First Pilot Results

Prepared and presented by
Mr J.-P. van der Hoek, Research Scientist, Wageningen Agricultural University
Department of Water Pollution Control
Wageningen, Netherlands



BIOLOGICAL/PHYSICAL CHEMICAL NITRATE REMOVAL FROM GROUNDWATER
- BASIC DESIGN CRITERIA AND FIRST PILOT PLANT RESULTS

J.P. van der Hoek

Wageningen Agricultural University
Department of Water Pollution Control

De Dreijen 12 - 6703 BC Wageningen - The Netherlands

Abstract

A new process is described for nitrate removal from groundwater: biological/physical chemical nitrate removal. This technique is a combination of biological denitrification and ion exchange by which the disadvantages of the separate techniques can be avoided. Nitrate is removed from the groundwater by ion exchange, but the resin is regenerated in a closed circuit with the use of a denitrification reactor. A direct contact between groundwater and denitrifying bacteria is prevented, and brine production and salt requirement for regeneration are minimal. The basic design criteria and the first results obtained with a lab-scale pilot plant are presented.

1. Introduction

To remove nitrate from groundwater several techniques are available. Some of these techniques are summarized in table 1 (1, 2). Only ion exchange and biological denitrification are considered feasible and practical for full-scale treatment of drinking water. However, both these processes have serious disadvantages.

Biological denitrification is a process by which nitrate is converted to nitrogen gas by denitrifying bacteria. A direct contact is created between groundwater, which is generally free of micro-organisms, and bacteria. Also a C-source has to be added to the groundwater. Both aspects cause a serious risk of a bacteriological contamination of the groundwater, and to avoid this risk extensive post treatment is necessary (2,3,4,5,6).

Also the possible production of nitrite is a serious risk. With a groundwater temperature of 10-12°C the activity of denitrifying bacteria is rather low, which means that relatively large reactors are needed.

Table 1. Nitrate removal techniques

ion exchange
biological denitrification
chemical reduction
reverse osmosis
electrodialysis

Ion exchange is a physical-chemical process. By means of an anion exchange resin nitrate is exchanged for chloride or bicarbonate. A problem is the regeneration procedure of the resin. Normally this is done by using a very concentrated NaCl solution (50-100 g/l) with a flowrate of 2-4 BV/h (BV = bedvolumes) and a period of 30-45 minutes (7,8,9,10,11). So, a large excess of salt is necessary for regeneration, while a voluminous brine is produced during regeneration with high nitrate, sulphate and chloride concentrations. Brine disposal can be very difficult. Both aspects cause financial and environmental problems.

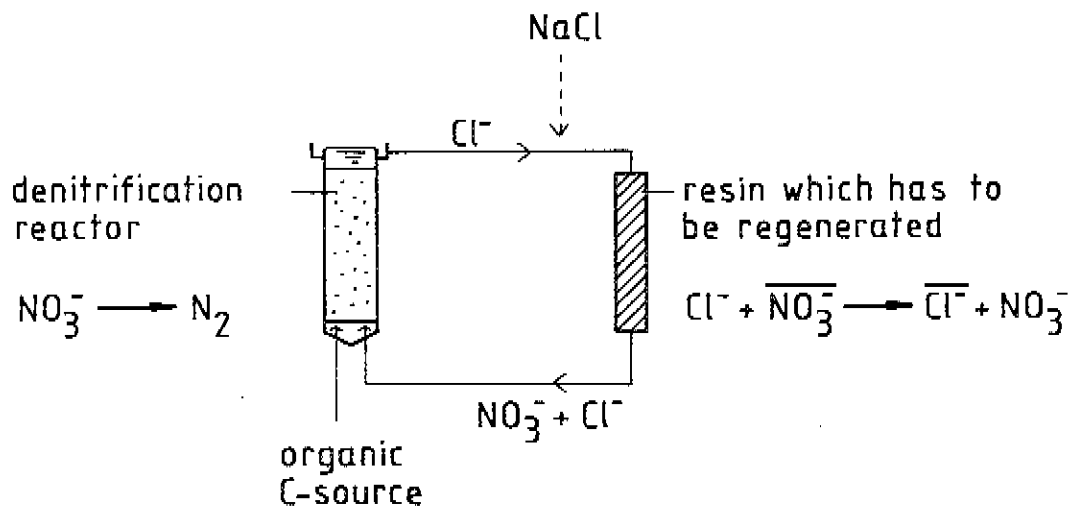


fig. 2. Regeneration of a nitrate-loaded resin to the chloride form with a denitrification reactor

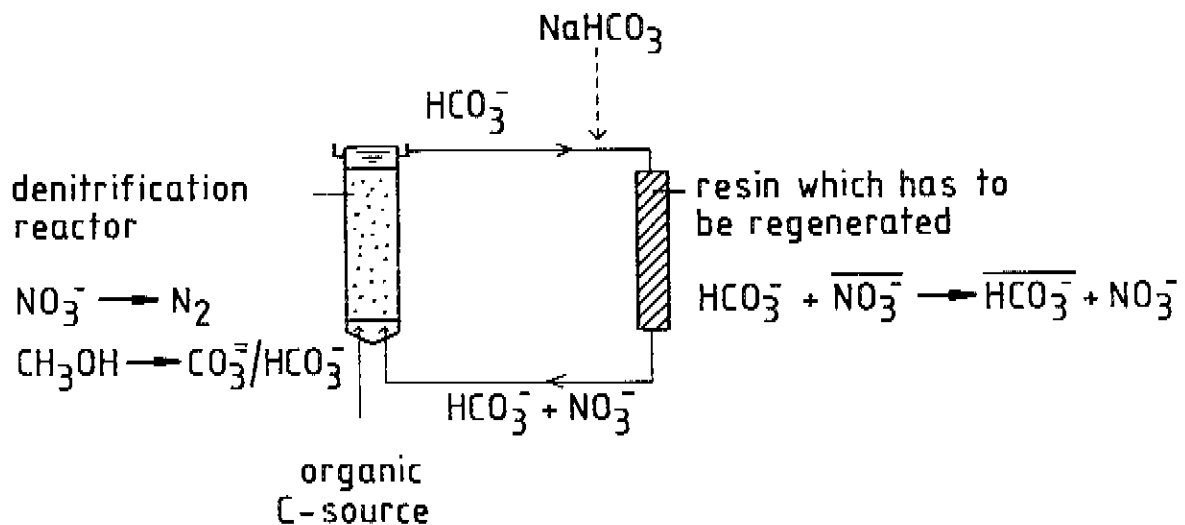


Fig. 3. Regeneration of a nitrate-loaded resin to the bicarbonate form with a denitrification reactor

Compared with ion exchange and biological denitrification the most important advantages of this new process are:

1. The regeneration is carried out in a closed system by which the production of a voluminous brine can be avoided and the salt requirement can be minimized. Using NaHCO_3 as regenerant has the advantage that the system itself produces bicarbonate because it is an endproduct of biological denitrification. When NaCl is used as regenerant, only the stoichiometric required amount has to be dosed.
2. Because the biological process is not in direct contact with the groundwater there is no risk that nitrite production will affect the water quality.
3. There is no direct contact between groundwater on the one hand and bacteria and a C-source on the other. Pollution of the resin by carry-over of suspended material from the denitrification reactor to the ion exchange column is possible, but measures against this can be taken in the regeneration circuit itself, so there is no need for an extensive post treatment.

3. Basic design criteria

3.1 Salt concentration of the regenerant

The salt concentration of the regenerant is determined by two aspects. On the one hand, very high salt concentrations can have an inhibiting effect on the biological denitrification but on the other the salt concentration must be high enough to obtain a sufficient regeneration of the resin within reasonable time.

Claus et al. (12) demonstrated that 20 g NaCl/l has no effect on autotrophic denitrification. Denitrification has also been observed in marine sediments (13,14). The effect of NaCl and NaHCO₃ on the capacity of denitrifying sludge, with methanol as C-source, is shown in fig. 4. It is clear that with NaHCO₃ concentrations of 25-30 g/l and NaCl concentrations of 10-15 g/l the denitrification capacity is still present for about 80%.

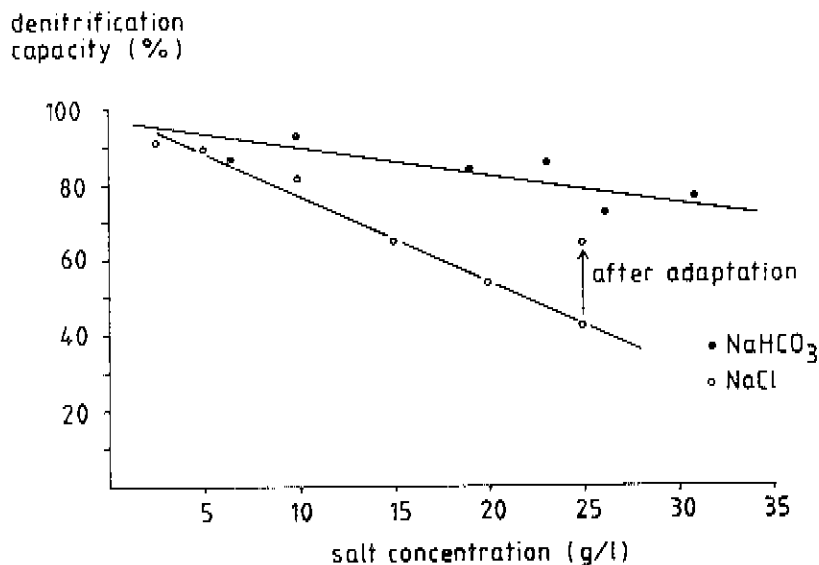


Fig. 4. Effect of high NaCl and NaHCO₃ concentrations on denitrification

From fig. 5 it can be concluded that regeneration of a nitrate loaded resin is possible with a solution containing 30 g NaHCO₃/l (357 mequiv/l). Compared with the normal regeneration procedure using 50-100 g NaCl/l, a flow rate of 2-4 BV/h and a period of approximately 30-45 minutes (7,8,9,10,11) it takes a longer time and a larger flowrate. However, with 30 g NaHCO₃/l and a flowrate of 10 BV/h almost complete regeneration is possible in 3.5 h.

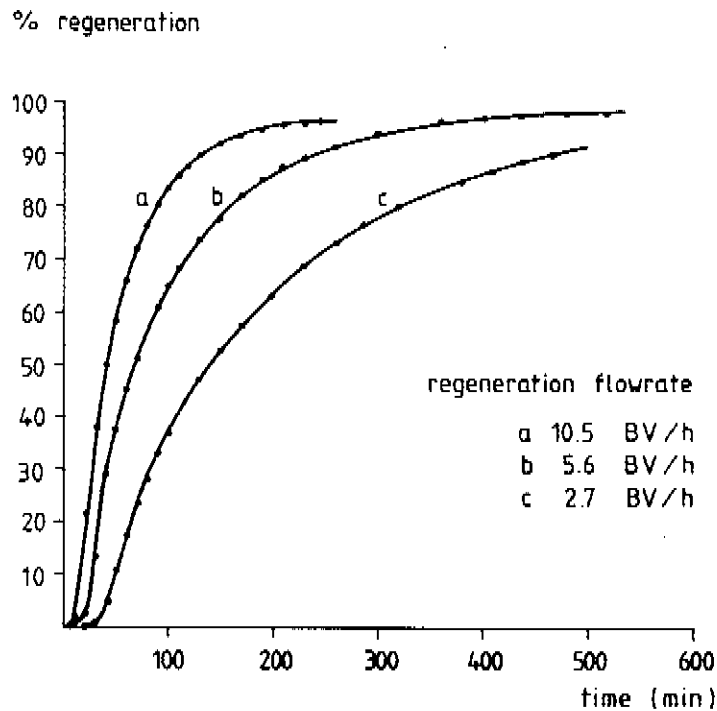


Fig. 5. Regeneration of a nitrate-loaded resin (Duolite A161) with a solution containing 30 g NaHCO₃/l (regeneration percentage = regeneration as percentage of total exchange capacity)

In table 2 the selectivity coefficients $K_{NO_3}^{Cl}$ and $K_{NO_3}^{HCO_3}$ of some strong base anion exchange resins are shown. The coefficients are defined as

$$K_{NO_3}^A = \frac{[\bar{A}^-] \cdot [NO_3^-]}{[NO_3^-] \cdot [A^-]}$$

with $[\bar{A}^-]$, $[\bar{NO}_3^-]$ = concentration of A⁻ and NO₃⁻ on the resin (eq/l resin)

$[A^-]$, $[NO_3^-]$ = concentration of A⁻ and NO₃⁻ in solution (eq/l)

Because $K_{NO_3}^{Cl}$ is about two times $K_{NO_3}^{HCO_3}$ it is possible to use a NaCl

solution as regenerant with a concentration which is only half of the NaHCO₃ concentration. So, regeneration can also be carried out in 3.5 h with 10.4 g NaCl/l (178 mequiv/l) and a flowrate of 10 BV/h.

Table 2. Capacity and selectivity coefficients $K_{NO_3}^{Cl}$ and $K_{NO_3}^{HCO_3}$ of strong base anion exchange resins

anion exchange resin	capacity (equiv/l)	$K_{NO_3}^{Cl}$	$K_{NO_3}^{HCO_3}$
macroporous resin			
Duolite A161	1.11	0.30	0.16
Duolite A162	1.19	0.24	0.12
Duolite A165	1.19	0.35	0.17
Bayer Lewatit MP500	1.09	0.36	0.18
Bayer Lewatit MP600	1.14	0.30	0.15
Amberlite IRA 996	1.01	0.11	0.04
gelresins			
Bayer Lewatit M500	1.36	0.33	0.18
Bayer Lewatit M600	1.29	0.35	0.18

3.2 Selection of denitrification reactor type

The denitrification reactor in this process must fulfill a number of conditions:

1. hydraulically it must fit in the process: this means that the flowrate through the denitrification reactor must equal the regeneration flowrate through the ion exchange column, otherwise a bypass would be necessary
2. the reactor must be capable to treat solutions with very high nitrate concentrations without recirculation, because nitrate concentrations in the regenerant can be expected up to 700 mg NO_3^- -N/l (15)
3. it must be possible to develop and maintain a high sludge concentration in the reactor. By this a constant high volumetric capacity can be obtained and the reactor dimensions can be small
4. sludge washout must be minimal to prevent organic fouling of the ion exchange resin
5. maintenance and process control must be minimal

In most experiments concerning denitrification of potable water fluidised bed reactors (16,17) and fixed bed reactors (18,19) are used. Considering the above mentioned conditions it is clear that they are not fulfilled with these reactors.

The flowrate through a fluidised bed reactor is not in accordance with the flowrate through the ion exchange column, and this type of reactor is not capable to treat high nitrate concentrated water without recirculation. Also it needs a good control and balancing of the flowrate to avoid washout of sludge/sand particles. The drawbacks of fixed bed reactors are that backwashing is necessary to avoid clogging (18,19,20) and that the volumetric capacity is low compared with fluidised bed reactors (20).

The best suited denitrification reactor in this process is the Upflow Sludge Blanket (USB) reactor. With this type of reactor much experience has been obtained the last years, both in the field of denitrification (21,22) and anaerobic treatment of wastewater (23). In this type of reactor the biomass is not present on a carrier material as in the fluidised bed reactor and fixed bed reactor, but the biomass grows in pellets due to chemical, physical and biological conditions. In the case of denitrification pellet formation (2-3 mm) is promoted by precipitation of CaCO_3 which is a result of the rise in pH due to biological denitrification. In this way a sludge concentration up to 30-40 g VSS/l can be maintained (15) with superior settling characteristics, and superficial velocities as high as 2-4 m/h are possible (22,23).

With the USB denitrification reactor the above mentioned conditions can be fulfilled. Hydraulically it is possible to use the same flowrate through the USB reactor and the ion exchange column. A USB denitrification reactor is capable to treat water with very high nitrate concentrations without recirculation, and a volumetric capacity of 400-500 g N/m³.h is possible (15,22). Sludge washout is very low due to good settling characteristics and can be minimized when the reactor is equipped in the upper part with a gas-solids separator as shown in fig. 6. Operation of this reactor is very simple and it doesn't need backwashing.

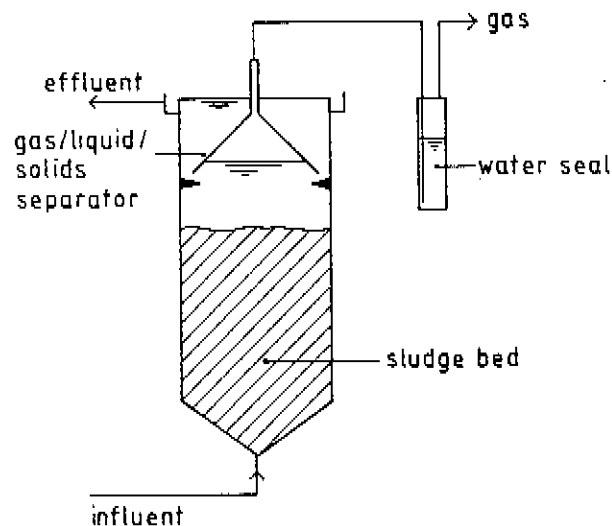


Fig. 6. Schematic diagram of a USB denitrification reactor

3.3 Influence of sulfate and selection of resin type

The normal strong base anion exchange resins are more selective for sulfate than for nitrate (7,24,25). Most groundwaters contain both sulfate and nitrate, and sulfate in the groundwater influences the process in two ways:

- firstly, the effective nitrate capacity of the resin decreases when the groundwater contains more sulfate (11)
- secondly, sulfate is easily removed from the resin during regeneration into the regeneration circuit (15). By this sulfate will accumulate in the regeneration circuit, and it may be possible that after several regenerations the resin will remain partly loaded with sulfate due to the high sulfate concentration in the regenerant.

This also decreases the effective nitrate capacity of the resin. Treating a Dutch groundwater with 19.2 mg NO_3^- -N/l and 29.5 mg SO_4^{2-} /l no problems were encountered with a normal resin, Duolite A165 (see chapter 4.2). However, when a groundwater has to be treated with a much higher sulfate concentration problems are to be expected.

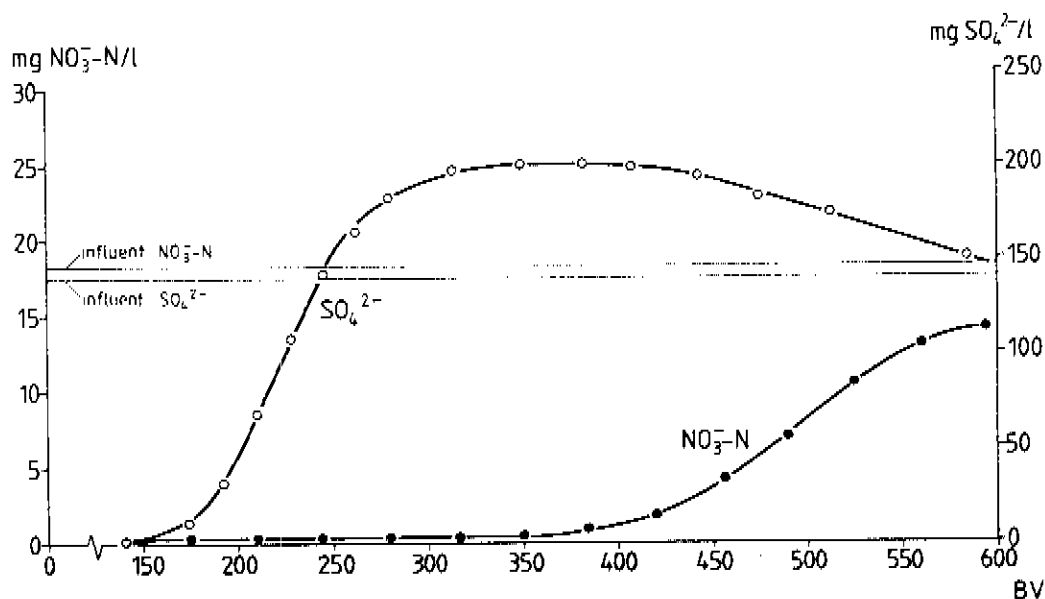


Fig. 7. Break-through profile of Amberlite IRA996. Influent concentrations 18.1 mg NO_3^- -N/l and 139.4 mg SO_4^{2-} /l, flowrate 35 BV/h, resin in HCO_3^- form

Recently some nitrate selective resins are developed (7,26). In our department in Wageningen a resin from Rohm & Haas, Amberlite IRA996, was tested. From the break-through curve in fig. 7 it is clearly visible that this resin is nitrate selective, even with a very high sulfate concentration of 139.4 mg SO_4^{2-} /l.

When this resin was regenerated several times with a solution containing 30 g NaHCO_3/l and additional sulfate varying from 0 g $\text{SO}_4^{2-}/\text{l}$ up to 18.4 g $\text{SO}_4^{2-}/\text{l}$ (regeneration flowrate 10 BV/h, regeneration time 3.5 h), it appeared that the nitrate capacity (expressed as percentage of total exchange capacity) of this resin during running with groundwater after each regeneration is almost independent of the sulfate concentration in the regenerant, as can be seen in fig. 8 (groundwater composition 18 mg NO_3^-/l , 30 mg $\text{SO}_4^{2-}/\text{l}$, 25 mg Cl^-/l and 58 mg HCO_3^-/l ; flowrate 35 BV/h, runtime 17 h).

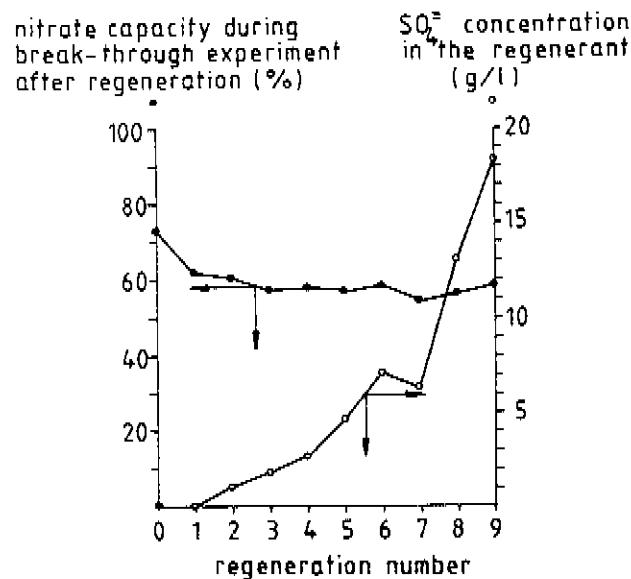


Fig. 8. Effect of sulfate in the regenerant on nitrate capacity of the nitrate selective resin Amberlite IRA996 (nitrate capacity expressed as percentage of total ion exchange capacity)

Both aspects mean that the process is also suitable for groundwater with high sulfate concentrations when nitrate selective resins, such as Amberlite IRA996, are used.

3.4 Use of a sandfilter and a disinfectant in the process

Although sludge washout can be minimized with a USB denitrification reactor still the resin can become polluted by carry-over of suspended solids from the denitrification reactor to the ion exchange column. Also humic and fulvic acids, which can accumulate in a closed regeneration circuit with a biological process, can cause organic fouling of the resin (27,28,29,30,31). This pollution of the resin has no serious effect on resin capacity: after 9 regenerations of a resin (Duolite A165) with effluent of a USB denitrification reactor the capacity decreased only 8%, and this decrease was already reached after the third regeneration, after which the capacity stabilized (32).

However, it can affect the bacteriological quality of the treated water, because after regeneration the resin is in contact with drinking water. For this reason two things can be done. First, a sandfilter can be placed in the regeneration circuit between the USB denitrification reactor and the ion exchange column to remove suspended solids from the regenerant before it reaches the resin. Secondly, a disinfectant can be used in the process. After regeneration the ion exchange column is rinsed with water. It is possible to use a disinfectant during the first minutes of this rinsing. Especially peracetic acid is often used for disinfection of ion exchange resins (33,34).

In a pilot plant for nitrate removal from groundwater the ion exchange columns are used 9 h for potable water production, after which they are regenerated 3.5 h and rinsed 1 h (see chapter 4.1). In a laboratory experiment this was simulated by contacting two resins for 3.5 h with effluent from a USB denitrification reactor containing vary many bacteria (see table 3) and then rinsing them for 1 h. One resin was rinsed with water containing 0.15% peracetic acid during the first 15 minutes. After rinsing, both resins were contacted with sterilized water for 9 h. In this water the number of bacteria was measured (colony counts on glucose yeast extract at 22°C, number of bacteria per ml). This procedure was repeated several times. In table 3 the results are presented. It seems possible that a bacteriologically reliable water can be produced when 0.15% peracetic acid is used the first 15 minutes during rinsing.

Table 3. Colony counts (number of bacteria per ml) in biological regenerant, in water treated with a not disinfected resin and in water treated with a disinfected resin

	colony counts
regenerant (effluent from USB denitrification reactor)	100.000 - 1.000.000
water treated with a not disinfected resin	50.000 - 100.000
water treated with a disinfected resin	0

4. First experiments with a lab-scale pilot plant for nitrate removal from groundwater

4.1 Design of the pilot plant

Very schematically the pilot plant is shown in fig. 9. It consists of three ion exchange columns, filled with resin Duolite A165, a sand filter and a USB denitrification reactor. Methanol is used as substrate for the denitrification reactor.

Two ion exchange columns are used for production of potable water and have a run time of 9 h each. They work 4.5 h out of phase. The third ion exchange column is connected with the denitrification reactor, and is regenerated for 3.5 h. For the next hour this ion exchange column is rinsed with water which contains a disinfectant (peracetic acid) the first 15 minutes. During rinsing water is recirculated through the denitrification reactor by means of a by-pass. In this way every 4.5 h a regenerated ion exchange column is put into service for nitrate removal from groundwater. The pilot plant is controlled by a Programmable Logic Controller (PLC).

In table 4 the dimensions of the plant and groundwater composition are shown. During the experimental period NaHCO_3 was used as regenerant. No disinfectant was used and the sand filter was only used temporarily.

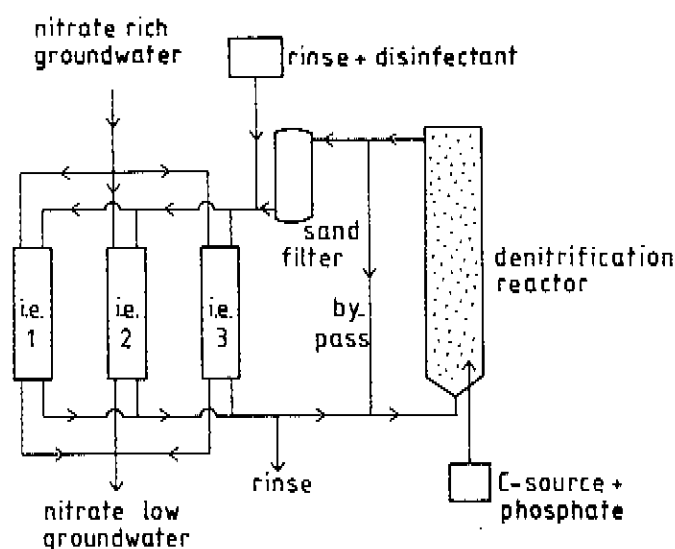


Fig. 9. Lab-scale pilot plant for nitrate removal from groundwater, using the biological/physical chemical process

Table 4. Dimensions of the pilot plant and groundwater composition

volume ion exchange columns	0.95 l
volume denitrification reactor	5 l
groundwater flowrate	65.7 l/h (2 x 32.9 l/h)
regeneration flowrate	9-12 l/h
rinse flowrate	9-12 l/h
groundwater composition	
NO ₃ ⁻ -N	19.2 mg/l
SO ₄ ²⁻	29.5 mg/l
Cl ⁻	26.1 mg/l
HCO ₃ ⁻	98.3 mg/l
pH	7.8
regenerant NaHCO ₃	10.5-23.7 g/l

4.2 Results obtained with the pilot plant

In fig. 10 the nitrate concentration in the treated groundwater is shown. All measurements are related to the process-cycle of 4.5 h.

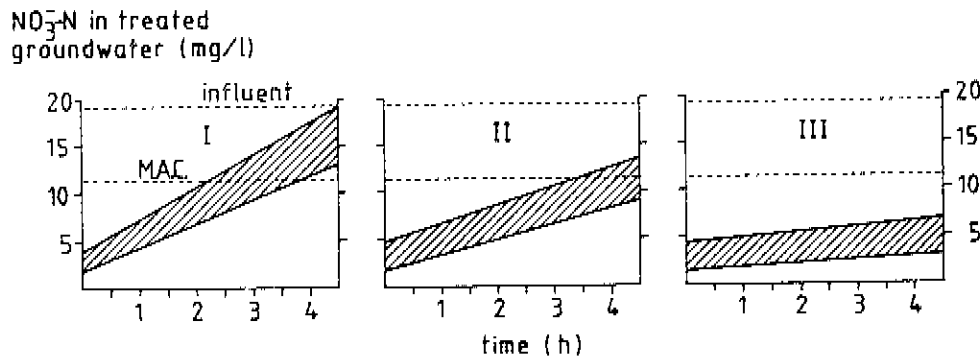


Fig. 10. Nitrate concentrations in the treated groundwater
 I denitrification reactor capacity 525 mg N/h
 II denitrification reactor capacity 625 mg N/h
 III denitrification reactor capacity 840 mg N/h
 (influent concentration 19.2 mg NO₃⁻-N/l; maximum admissible concentration = 11.3 mg NO₃⁻-N/l)

During the experimental period three different periods could be distinguished, each with a different denitrification reactor capacity: period I with a capacity of 525 mg N/h, period II with a capacity of 625 mg N/h and period III with a capacity of 840 mg N/h. A higher denitrification reactor capacity results in a better regeneration of the resin, which means that a lower nitrate concentration in the treated water can be reached.

During all periods clearly a sort of break-through profile was visible in the 4.5 h process-cycle. This is caused by the fact that at the start of every 4.5 h process-cycle one ion exchange column is just put into service for water production, while the other is already 4.5 h in service. At the end of the 4.5 h process-cycle one ion exchange column has been 4.5 h in service and the other 9 h, resulting in a higher nitrate concentration in the treated water.

In period I sulfate was present in the treated water ranging from 3.4 to 5.8 mg $\text{SO}_4^{2-}/\text{l}$. In period II and III only occasionally sulfate was present in the treated groundwater in very low concentrations. Chloride concentrations in the treated water varied between 4.4 and 39.7 mg Cl^-/l . Bicarbonate concentrations in the treated water were always higher than influent concentrations due to a NaHCO_3 regenerant. The highest measured concentration was 238 mg HCO_3^-/l . The pH ranged from 7.70 to 8.60.

To control and prevent sulfate accumulation in the regenerant, the regenerant was renewed every six days. In fig. 11 the course of sulfate concentrations in the regenerant during these six-days periods is shown.

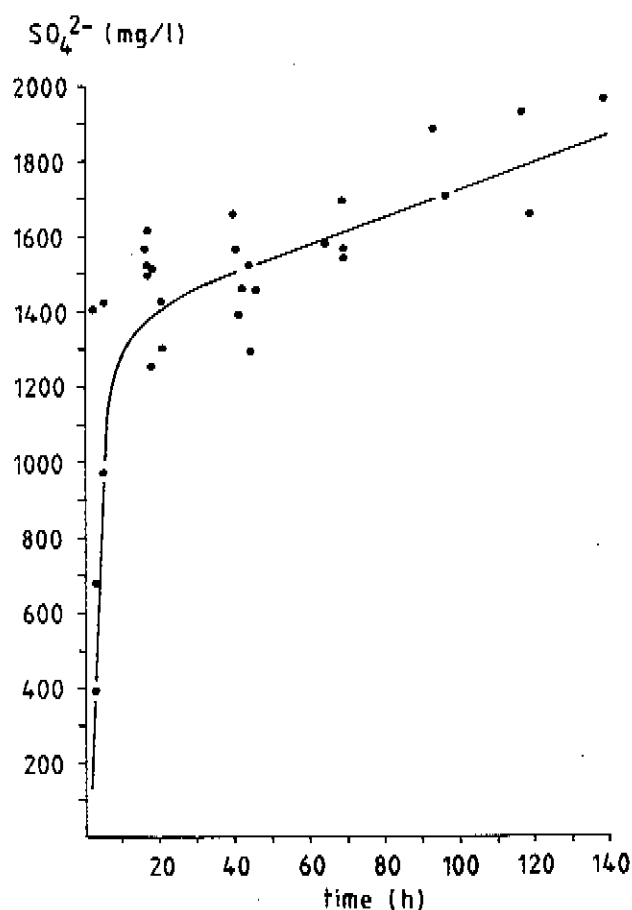


Fig. 11. The course of sulfate concentrations in the regenerant

The brine volume produced by renewing the regenerant every six days is only 13-20% of the brine which would be produced when the ion exchange columns are regenerant in the conventional way without a closed system.

After an experimental period of 44 days and 111 days the capacity of the resin in the three columns was measured. From the colour of the resins it was visible that they had become fouled. Probably this organic fouling is caused by humic acids and fulvic acids which can accumulate in a closed system containing a biological process. These acids can be absorbed by anion exchange resins (27,28,29,30,31).

The results are presented in table 5. The capacity is expressed as percentage of the capacity of an unpolluted resin, which is 1.19 equiv/l in the case of Duolite A165. Although the resins had become more polluted from day 44 to day 111 (visible by eye) the resin-capacity did not decrease in this period. Compared with the loss of capacity after 3 regenerations (8%, see chapter 3.4) the decrease is very small. Already Harries et al. (35) stated that there is no apparent link between the deterioration in resin performance and the degree of organic fouling of the resin.

Table 5. Loss of resin capacity due to organic fouling of the resin

resin in column nr.	relative capacity (%)	
	after 44 days	after 111 days
1	90.1	91.0
2	90.1	90.9
3	89.2	90.6

APPENDIX. Economic aspects of the new process

As already mentioned in paragraph 2 the combined biological/physical chemical nitrate removal process has important financial advantages compared with ion exchange as a result of minimal salt requirement and brine production.

To have an indication of the financial implications of the new process some rough cost calculations were made. Three alternatives were compared for nitrate removal from groundwater were compared:

- conventional ion exchange (I)
- biological/physical chemical nitrate removal (II)
- ion exchange with partial regeneration (III)

Biological denitrification was not included for the reasons mentioned in paragraph 1.

Calculations were made for a plant with the following dimensions:

- capacity $75 \text{ m}^3/\text{h} = 1800 \text{ m}^3/\text{d} = 394200 \text{ m}^3/\text{y}$
- nitrate removal from $90 \text{ mg NO}_3^-/\text{l}$ to $0-10 \text{ mg NO}_3^-/\text{l}$
- sulfate concentration of the groundwater $100 \text{ mg SO}_4^{2-}/\text{l}$

The calculations were made with several assumptions:

- ion exchange costs were based on data of the Environmental Protection Agency
- construction costs only include manufactured equipment. Housing, excavation and side work, and pipe costs will be approximately equal for the three alternatives. Manufactured equipment is the major part of construction costs.
- costs of brine disposal Hfl. $20/\text{m}^3$
 - regeneration salt Hfl. 200/ton
 - methanol Hfl. $700/\text{m}^3$
 - disinfectant Hfl. 2500/ton
 - energy Hfl. 0.30/kWh
- estimation of the costs of biological/physical chemical nitrate removal (II) from the costs of conventional ion exchange (I) with the following assumptions:
 - . manufactured equipment 50% more
 - . maintenance and labor 20% more
 - . energy: energy for pump in regeneration circuit (flowrate $17 \text{ m}^3/\text{h}$)

5. Conclusions

The described biological/physical chemical process is a very attractive technique for nitrate removal from groundwater. Compared with ion exchange brine production is very low and salt requirement for regeneration is minimal. Compared with direct biological denitrification of groundwater the production of bacteriologically reliable drinking water seems possible by means of simple measures. Also groundwater with a high sulfate concentration can be treated with this technique when a nitrate selective resin is used, for example Amberlite IRA996.

Acknowledgement

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- . brine disposal 13% of brine disposal compared with conventional ion exchange
- . salt requirement 10% of salt requirement compared with conventional ion exchange
- . methanoldosage 2.4 kg/kg NO_3^- -N removed
- . disinfection 0.1% for 15 minutes in regeneration circuit
- estimation of the costs of ion exchange with partial regeneration (III) from the costs of conventional ion exchange (I):
 - . manufactured equipment 25-30% more due to 25-30% lower resin capacity in connection with partial regeneration
 - . regeneration salt 50% less in connection with 150% instead of 300% excess
 - . brine disposal 50% less

	I	II	III
manufactured equipment	Hf1. 226000	Hf1. 339000	Hf1. 290000
operation and maintenance costs			
. energy	Hf1. 6480/y	Hf1. 7700/y	Hf1. 6480/y
. maintenance and labor	Hf1. 21700/y	Hf1. 26040/y	Hf1. 21700/y
. brine disposal	Hf1. 80000/y	Hf1. 10400/y	Hf1. 40000/y
. regeneration salt	Hf1. 165000/y	Hf1. 16500/y	Hf1. 82600/y
. methanol		Hf1. 16800/y	
. disinfectant	_____*	Hf1. 13250/y+	_____*
	Hf1. 273180/y	Hf1. 90690/y	Hf1. 150780/y

Table 1. Cost calculation of three alternatives ($75 \text{ m}^3/\text{h}$).

- I. conventional ion exchange
- II. biological/physical chemical nitrate removal
- III. ion exchange with partial regeneration