

**Original Research Paper** 

# **Removal Techniques for Nitrate : A Review**

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Nitrate is a colourless odorless and tasteless compound. Nitrate can be expressed as either NO<sub>3</sub>- (nitrate) or NO<sub>3</sub>-N (nitratenitrogen). Nitrate levels above the EPA maximum contaminant level of 10mg/L NO<sub>3</sub>-N or 45 mg/L NO<sub>3</sub>- may cause methemoglobinemia in infants. Proper management of fertilizer, manures and other nitrogen sources can minimize contamination of drinking water supplies. Nitrate is a problem as a contaminant in drinking water (primarily from ground water and wells) due to its harmful biological effects. High concentrations can cause methemoglobinemia, and have been cited as a risk factor in developing gastric and intestinal cancer. This review article is aimed at providing precise information on efforts made by various researchers in the field of nitrate removal from drinking water.

KEYWORDS	EPA, Contamination, Nitrate, Concentration and Methemoglobinemia etc.

## INTRODUCTION

Nitrate contamination of ground water resources has increased in Asia, Europe, United States and various other parts of the world. This trend has raised concern as nitrates cause methemoglobinemia and cancer. Several treatment processes can remove nitrates from water with varying degrees of efficiency, cost and ease of operation. Available technical data, experience and economics indicate that biological denitrification is more acceptable for nitrate removal than reverse osmosis and ion exchange. Here we review the developments in the field of nitrate removal processes which can be effectively used for denitrifying ground water as well as industrial water. Many technologies for the removal of nitrate from water have been adopted based on scientific developments. A brief overview of all techniques is presented here. Nitrate is a stable and highly soluble ion with low potential for coprecipitation or adsorption. Thus conventional treatment technologies cannot be used. Here we review various techniques in terms of their effectiveness, ease of operation and cost.

These are following -

- A. Chemical Denitrification
- B. Reverse Osmosis (RO) for Denitrification
- C. Electrodialysis (ED) for Denitrification
- D. Catalytic Denitrification
- E. Electrocatalytic Reduction for Denitrification
- F. Ion Exchange Process
- G. Denitrification Using a Membrane Bioreactor
- H. Combined Ion Exchange & Membrane Bioreactor for Denitrification
- I. Denitrification Using Nanofiltration
- J. Adsorption
- K. Biological Denitrification

# A. Chemical Denitrification

Nitrate reduction can be induced under basic pH according to the following reaction :

No<sub>3</sub>- + 8Fe (OH)<sub>2</sub> + 6H2O → NH<sub>3</sub> + 8Fe(OH)<sub>3</sub> + OH-

Experimental result showed that a Fe: NO3- ratio of about 15:1 was required in the presence of copper catalyst for the reaction to proceed. This process generated a large quantity of iron sludge and formed ammonia removal by air stripping (1). The process was associated with high costs.

In chemical denitrification by powdered aluminum ammonia was found to be the principal reaction product (60-95%) at pH of 10.25, which was removed by air stripping (2-3). The

denitrification was explained on the basis of the following reactions:

2NO<sub>2</sub>-+2AI+4H<sub>2</sub>O ----N2 + 2AI(OH)<sub>3</sub> + 2OH-

Aluminum reacted with water as per the following equation:

 $2AI + 6H_2O \longrightarrow 2AI(OH)_3 + 3H_2$ 

It was shown that 1.16g of aluminum was required for the reduction of 1g of nitrate. Catalytic reduction of nitrate with Pd and/Cu catalysts was another removal technique (4). It was found that Pd-Cu combined catalysts at a ratio of 4 can maximize the nitrate reduction into nitrogen; above 80% total nitrate removal efficiency was realized. 84% denitrification efficiency was achieved at ambient temperature and pressure using zero-valent magnesium [Mg(0)] for Mg(0): NO<sub>3</sub>-N molar ratio of 5.8 and pH of 2(5).

## B. Reverse Osmosis (RO) for Denitrification

Nitrates could be removed by reverse osmosis cells under pressures ranging from 300 to 1,500 psi to reverse the normal osmotic flow of water. Membranes used were made of cellulose acetate, polyamides and composite materials. Problems associated with reverse osmosis membranes included fouling, compaction and deterioration with time. These problems resulted from deposition of soluble materials, organic matter, suspended and colloidal particles, and other contaminants, pH variations and chlorine exposure; thus the reverse osmosis process required pretreatment. A 15-gpm spiral wound cellulose acetate reverse osmosis system was tested for 1,000 h and up to 65% nitrate separation was observed for influent NO<sub>3</sub>- concentrations ranging from 18 to 25mg/L(6).

Reverse osmosis using both polyamide and cellulose triacetate membranes were tested and sulfuric acid and sodium hexametaphosphate were added to feed water to prevent scaling. Polyamide membranes were more effective than cellulose triacetate membranes (7).

A pilot plant using spiral wound modules with composite membranes was operated with a 2 m<sup>3</sup>/h capacity, at an operating pressure of 14 bar. Influent pretreatment consisted of passing the water through 5 µm cartridge filters and acid dosing to avoid scaling. The result showed high levels of denitrification (8).

## C. Electrodialysis (ED) for Denitrification

In ED ions are transferred through membranes from a less concentrated to a concentrated solution by application of direct electric current. ED treated the water by selective removal of undesirable ions through a semi permeable membrane. An electrodialysis system required a supply of pressurized water [50-75 psi (345-578 kPa)] with pretreatment.

In the electrodialysis reversal (EDR) process, the polarity of the electrodes was reversed two to four times an hour to alter the direction of ion movement. The EDR process reduced scaling and chemical usage compared with conventional ED and was used for the production of drinking water from nitrate rich water. The nitrate removal efficiency of ED and RO processes was almost the same (9).

A selective nitrate removal process was developed, NitRem, based on ED (10). The process was effective in reducing nitrate concentrations from 50 mg/L or more to less than 25 mg/L. An attractive feature of the process was that it included the removal of nitrate without the addition of any chemicals.

It was observed that for nitrate reduction from 100 to 50 mg  $NO_3$ -/L the costs of EDR and RO were about the same (8). It was expected capital and maintenance cost of membrane filtration to reduce with time (11).

# D. Catalytic Denitrification

A catalytic process was developed for removal of nitrite and nitrate from water (12). Palladium – alumina catalysts were effective in reducing nitrite to nitrogen (98%) and ammonia in the presence of hydrogen. The lead (5%), copper(1.25%), Al2O3, catalyst were found to completely remove nitrate from water having an initial nitrate concentration of 100mg/L. The reaction was completed in 50 min. The process operated effectively at a temperature of 10°C and pH 6-8.

# E. Electrocatalytic Reduction for Denitrification

An electrocatalytic reduction process was used to remove  $NO_3$ from ground water (13). A commercially available carbon cloth with a 30% surface coated Rh (rhodium) (1µg cm<sup>-1</sup>) was tested at an applied potential of 1.5 V versus standard calomel electrode (SCE) with a Pt auxiliary electrode. The result suggested that electrocatalysis reduced  $NO_3$ - concentrations in ground water from 73 to 39 mg/L on a timescale range of 40-60 min.

# F. Ion Exchange Process

The ion exchange process involved passage of nitrate water through a resin bed containing strong base anion (SBA) exchange resins on which nitrate ions were exchanged for chloride or bicarbonate ions until the resin exhausted. The exhausted resin was regenerated using a concentrated solution of sodium chloride or sodium bicarbonate (14). Fifteen ion exchange plants used in the United States reduced nitrate from 18 to 6.8 mg/L (15, 16). Addition of bentonite clay to the backwash water helped restore the resin, which was regenerated with 1 N NaOH and HCI. Nitrate removal capacity of the resins was reduced by Silica and iron precipitates (17). An ion exchange process was tested for the removal of nitrate from ground water containing 16-23 mg NO<sub>3</sub>-/L at flow rates of up to 31.5 L/m<sup>2</sup> (18). Treatment of sulfate water with resins is difficult as the nitrate removal capacity of the resin is reduced by the sulfate ions. It was observed that sulfate selectivity was reduced by increasing the distance between ion-exchange sites and nitrate selectively can be increased by increasing the matrix and functional group hydrophobicity (19). Triethyl amine resins showed an increase in the bed life by 62% when treating water containing 1.5 meq/L nitrate and 6.5 meq/L sulfate. Regenerant usage decreased by 25-50%, thus the operating cost of the ion exchange process reduced (18). The 0.043 m<sup>3</sup>/s ion exchange plant in California (USA) used a non-nitrate-selective resin, which was regenerated using a saturated brine solution (20). The total treatment cost was reported to be 24.2 cents/1,000 gal. The energy requirements of the plant were 0.244 kWh per 1,000 gal (18). A pilot-scale study was conducted to evaluate nitrate removal from drinking water by ion exchange, reverse osmosis

(RO) and electrodialysis (ED) (7). The raw water contained 10-25 mg/L, 43 mg/L sulfate and 530 mg/L total dissolved solids (TDS). All processes were able to reduce nitrate concentration below 10 mg/L. Ion exchange process was found to be five times more economical in comparison to RO process (20). A process was developed in which regeneration and exhaustion were performed in the same direction and reduced nitrate concentrations from 15.8 to 5.7 mg/L (21). The Carbon dioxide regenerated ion exchange resins (CARIX) process for removing nitrate, sulfate, and hardness from water was based on ion exchange principles (22-24). The exhausted exchange resins were regenerated through contact with a concentrated carbon dioxide solution. A CARIX pilot plant (0.047 m<sup>3</sup>/s) was constructed in Germany which was effective in reducing nitrate concentrations from 90 mg/L to less than 5.7 mg/L. The consumption of carbon dioxide amounted to 0.35kg/m<sup>3</sup> of treated water.

# G. Denitrification Using a Membrane Bioreactor (MBR)

Immersed heterotrophic membrane bioreactor (MBR) produced high quality water when  $NO_3$ - contaminated water was made to flow through the lumen of tubular microporous membranes (25).  $NO_3$ - diffused through the membrane pores (26). Denitrification took place on the shell side of the membranes. The MBR achieved over 99%  $NO_3$ - removal at an influent concentration of 200 mg  $NO_3$ -/L.

# H.Combined Ion Exchange & Membrane Bioreactor for Denitrification

The IEMB concept combines dialysis and simultaneous biological degradation of nitrate in small concentrations (27, 28). The IEMB process operated with hydraulic retention times ranging from 1.4 to 8.3 h in the water compartment, proved to remove nitrates effectively, while preserving the water composition with respect to other ions, thus avoiding secondary contamination of the treated water.

# I. Denitrification Using Nanofiltration (NF)

During the last decade, nanofiltration (NF) made a breakthrough in drinking water production for the removal of nitrate (29). For the removal of nitrates the membranes NF70, NF45, UTC-20 and UTC-60 have been experimentally studied. The result showed that only a small fraction on nitrate was removed for most membranes, except for NF70 where a 76% nitrate removal was obtained.

# J. Adsorption

Adsorption has been found to be better than other techniques for water purification in term of ease of application, cost, simplicity of design and feasibility for in situ treatment of underground and surface water. Moreover adsorption does not need skilled maintenance and equipment intensive processes and can thus be applied in rural areas (30, 32, 38). Different adsorbents were used for water purification from nitrate such as agar, benonite, mustard, slag, impregnated almond shell activated carbon by zinc and zinc sulphate, charcoal prepared from bamboo, zinc chloride activated carbon from coconut coir pith, wheat and mustard straw (30-32). Nitrate ions were also removed from water using carbon cloth, sepiolite and activated carbon, protonated cross-linked chitosan Zeolite (30-41).

# K. Biological Denitrification

Many bacteria belonging to different genera can grow anaerobically by reducing ionic nitrogenous oxides to gaseous products. Nitrates or nitrities served as the terminal electron acceptors instead of oxygen and resulted in generation of ATP (42). Such denitrification was dissimilatory nitrate reduction (43). When electrons are transferred from the donor to the acceptor, the organism gains energy which was applied for the synthesis of a new cell mass and the maintenance of the existing cellmass. The enzymes associated with denitrification are synthesized under anaerobic or partially aerobic conditions (44). Nitrate reduction to nitrogen gas occurred as :

 $No_3 \rightarrow No_2 \rightarrow No \rightarrow N_2 o \rightarrow N_2$ 

Each step was catalyzed by an enzyme system. Dissimilatory reduction of nitrate to nitrite was important for most bacteria, since the process involved energy conservation by increased substrate level phosphorylation reaction (45). Since denitrification was a respiratory process, an oxidisable substrate was needed as an energy source. Limitation of biological denitrification was bacterial contamination and presence of residual organics (46-50).

#### Heterotrophic Denitrification

Most denitrifying bacteria are heterotrophic and utilize complex organic substances as oxidisable substrates such as methanol, ethanol, methane, carbon monoxide and acetic acid for the conversion of nitrate to nitrogen (51-55). Pilot scale studies heterotrophic denitrification were conducted using packed and fluidized columns (56). The reactors required a start-up period of two weeks to establish sufficient bacterial populations. The highest denitrification rate per unit reactor volume was observed for the fluidized sand bed (160 g N/m<sup>3</sup>.h at 10°C) and lowest for the packed bed reactors (12 g N/m<sup>3</sup>.h at 10°C). Nitrate concentrations were reduced to approximately 45 mg/L (57,58). Denitrification by immobilized Pseudomonas denitrifications cells was studied using a sodium alginate polymer and ethanol as the carbon source (59). The nitrate concentration reduced from 104 to 0.1 mg/L. The limitations of the process being that the low rate of diffusion of substrate and reaction products through the alginate matrix and the short life span of the alginate matrix. To overcome these problems a membrane immobilized biofilm reactor was developed in which denitrifying bacteria and carbon energy source were segregated from the water to be treated (60, 61).

#### Autotrophic Denitrification

Some bacteria from the genera Paracoccus Thiobacillus, Thiosphaera and others can accomplish denitrification autotrophically using hydrogen or various reduced sulphur compounds such as S<sup>0</sup>, S<sup>2</sup>, S<sub>2</sub>O<sub>3</sub><sup>-2</sup>, S<sub>4</sub>O<sub>2</sub><sup>-2</sup> or SO<sub>3</sub><sup>-2</sup> as energy sources. Bacteria from the genera Ferrobacillus, Gallionella, Leptothrix and Sphaerotillus can utilize ferrous iron as an energy source for autotrophic denitrification. Under autotrophic growth conditions, carbon dioxide or bicarbonate was used as carbon source for microbial cell synthesis. Thiobacillus denitrifications was used to reduce nitrate concentrations from 24 to 1 mg/L in packed bed reactors using elemental sulfur as an electron source (62). Schippers described denitrification using limestone filtration (63). Lewandowski encapsulated autotrophic denitrifiers in calcium alginate beads containing sulfur and calcium carbonate for autotrophic denitrification (64). Nitrate concentrations were reduced from 27 to 6 mg/L in seven hours. A process known as DENITROPUR was developed using hydrogenotrophic denitrification (65). The process does not require the post treatment that heterotrophic denitrification requires. The reproduction rate of autotrophic bacteria is low, therefore, less biomass sludge was generated.

#### L. Emerging Treatment Technologies

Several drinking water treatment technologies for nitrate are being developed but are still primarily in the experimental stage or do not have published information on the effectiveness of largescale applications. Some of the emerging technologies include the following-

#### 1. Chemical Denitrification

Kapoor and Viraraghavan (2007) and Shrimali and Singh (2001) reviewed and provided general information on several laboratory studies that have been conducted on the use of metals for the chemical reduction of nitrate to other nitrogen species (66-67). Chi et al. (2005) demonstrated that a 50% reduction in nitrate from an initial concentration of 1500 mg NO<sub>3</sub>-N/L (equivalent to 6, 650 mg NO<sub>3</sub>-/L) could be achieved using metallic iron when water was acidified to a pH of 5 (68). Luk and Au-Yeung (2002) reported a maximum nitrate removal of 62% to achieve treated water concentrations of 8.3 mg NO<sub>3</sub>-N/L (equivalent to 37 mg NO<sub>3</sub>-/L) using 300 mg/L of aluminum powder and a pH of 10.7 (69). Seidel et al. (2008) conducted piot-scale testing of sulphur-modified iron for the chemical reduction of nitrate (70). Result indicated that the

highest nitrate removal, from approximately 15 mg  $NO_{\rm 3}\text{-}N/L$  to 10 mg NO<sub>3</sub>-N/L (equivalent to 66 to 45 mg NO<sub>3</sub>-/L), occurred at a pH of 6.0 and an empty bed contact time of 30 minutes. The authors noted that a treated water goal of 8 mg  $NO_3$ -N/L (35 mg  $NO_3$ -/L) was not achieved consistently during the pilot testing.

#### 2. Catalytic Denitrification

Research studies have also examined the chemical denitrification of nitrate in the presence of catalyst (Reddy and Lin, 2000; Chen et al., 2003) (71,72). Reddy and Lin (2000) conducted laboratory tests of catalytic denitrification using three catalysts : palladium, platinum and rhodium. Rhodium was the most effective catalyst for nitrate removal. The result demonstrated that addition of 0.5 g of the rhodium per litre of water could decrease nitrate concentrations from 9 mg NO<sub>3</sub>-N/L to 3 mg NO<sub>3</sub>-N/L (equivalent to 40 to 13 mg NO<sub>3</sub>-/L) at a redox potential of -400mV. Chen et al. (2003) found that a 4:1 palladium-copper combined catalyst maximized nitrate reduction to nitrogen gas. An initial nitrate concentration of 22.6 mg NO<sub>3</sub>-N/L (equivalent to 100 mg NO<sub>3</sub>-/L) was reduced to less than 1 mg NO<sub>3</sub>- N/L (equivalent to 5 mg NO<sub>3</sub>-/L) after 20 minutes to reaction time.

#### 3. Polyelectrolyte-enhanced Ultrafiltration

Zhu et al. (2006) demonstrated greater than 90% removal of 60 mg NO<sub>3</sub>-N/L (equivalent to 266 mg NO<sub>3</sub>-/L) using polyelectrolyteenhanced ultrafiltration (73). The percentage of nitrate removed depended on the types of chelating polymers and the ultrafiltration membrane that was used in the study.

#### CONCLUSION

The four treatment processes that have been applied full-scale for nitrate removal include ion exchange, biological de-nitrification, reverse osmosis and adsorption. The other methods discussed have limited potential for full-scale application. Removal of nitrates from drinking water is an important and developing area of research. Technology development has occurred in this area, but further optimization of current technologies is required.

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