Removal of Nitrate from Groundwater by Using Natural Zeolite of Nizarneshwar Hills of Western India

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*Abstract-*Nitrate contamination of the world's groundwater supply poses a serious human health threat. High nitrate levels found in drinking water have been proven to be the cause for numerous health conditions across the world. Nitrate is highly soluble in water and it is retained by the soil at a minimum rate, which makes it a major component of groundwater in formation of nitrate pollution. In this study, natural Zeolite-stilbite was used as a low-cost adsorbent to evaluate its ability to remove nitrate from groundwater by ion exchange. Experimental investigation of nitrate removal from groundwater using natural Zeolite was carried out in the laboratory. In this investigation a natural Zeolite-stilbite was used for the removal of nitrate. It was found that 92% removal of nitrates occurs at initial concentration of 80 mg NO3/l solution. The effects of various parameters such as pH, flow rate, initial concentration have been studied. The optimum pH was fou Nitrate Pollution; Natural Zeolite; Ion Exchange; Ph; Regeneration nd to be 4. The maximum removal took place at a flow of 1ml/min in a laboratory Ion Exchanger. All the findings were observed at 60 cm bed depth. When compared with the activated carbon, it was found that the natural Zeolite-stilbite was more efficient in removal of nitrate. Resins were regenerated by circulating the generating agent i.e. NaCl.

Keywords- Nitrate Pollution; Natural Zeolite; Ion Exchange; Ph; Regeneration

I. INTRODUCTION

For the drinking purpose, groundwater has been used for a long time. Its purity has made it a well-known source of potable water. Due to the huge population, the safe use of groundwater has been hampered. Nitrate (NO_3) is one of the several inorganic pollutants contributed by nitrogenous fertilizers, organic manures, human and animal waste, and industrial effluents through the biochemical activities of microorganisms.

Excessive use of nitrogenous fertilizers in agriculture has been one of the primary sources of high nitrate in groundwater (De Roo 1980; Schepers at al.1984) [1, 2]. Apart from nitrate, nitrogen is applied in ammonia (NH_4^+) and amide (NH_2^-) forms, which generates nitrate in soil system through mineralization, which is fairly rapid in tropical and subtropical soils. Due to its high solubility and low retention by soil particles, nitrate is prone to leaching to the subsoil layers and ultimately to the groundwater, if not taken up by the plants or denitrified to N_2O and N_2. The rate of leaching is governed by the soil properties and amount of water present in the soil system. The arrival of nitrate to groundwater can be enhanced by shallow groundwater table, excessive application of nitrogenous fertilizers, manures and irrigation, and abundant rain fall. Livestock feedings, barnyards, septic tanks, animal and human contaminations are the other important sources contributing high amounts of nitrate to groundwater (Stevenson, 1986) [3]. In and around areas of high urbanization and industrialization, municipal and industrial wastes may contribute high levels of nitrate to the groundwater (Handa, 1983) [4]. As and when nitrate rich groundwater is pumped out and used for the drinking, it causes a number of health disorders in humans. Different organizations and countries have set standards for NO_3 in potable water, to safe guard public health from the hazards associated with high concentration of nitrate.

The anthropogenic element in the nitrate pollution of groundwater can well be perceived from the fact that terrestrial waters in uninhabited and less polluted regions like high altitude lakes, glaciers have negligible nitrate content. In central Himalayan snow and ice, NO_3 content is about 0.5 mg/l (Lunkad, 1994) [5] while world's average river water contains 1.0 mg NO_3/lit and the ultimate sink of terrestrial waters, the oceans, on average, have 0.67 mg NO_3/lit (Mason and Moore, 1985) [6].

Handa (1975) [7] carried out a study on 2500 groundwater samples (2000 from dug wells and 500 from deep tube wells) from different parts of India. High nitrate in the samples was attributed to animal or human sources or irrigation return flows from agricultural fields, dressed with fertilizers. It was found that nitrate in deep ground water was only 1-2 mg/l whereas shallow water had up to 100 mg/l in humid areas and 1000 mg/l in arid and semiarid regions. Data from 276 groundwater samples from UP in 1978 showed that number of wells with nitrate concentration below 10, 20, 50 and 100 mg/l were 53.6%, 71.7%, 89.5% and 95.3%, respectively (Pathak, 1980) [8]. Goyal et al. (1981) [9],found high nitrate distribution was prominent, as nearby places like Faruk Nagar and Gurgaon had wide differences with respect nitrate contents. Tanwar (1981) [10] found nitrate concentration ranging from 5-60 mg/l in different regions of Haryana. In studies conducted by Gupta (1981) [11] and Gopal et al. (1983) [12], groundwater nitrate concentration in Barmer, Bikaner, Jaipur, Jaisalmer and Udaipur were studied. 74% and 40% of groundwater samples in Barmer and Jaipur respectively, were found to have more than 11.3 mg/l NO_3 N.

Central Ground Water Board (CGWB) has carried out a rapid reconnaissance of nitrate in shallow groundwater (Handa, 1986) [13], mainly from dug wells in different states. Exceptionally high concentration of 1800 and 1620 mg/l nitrate was found in Hissar and Mahendragarh districts of Haryana. In Visakhapatanam City, nitrate in groundwater ranged from 1.12 to 28.99 mg/l (Rao and Rao, 1991) [14]. In isolated patches, affected by sewage or septic tanks, nitrate concentration was beyond the safe limit for public water supplies (20 mg/l), set by ICMR (Indian Council of Medical Research). In another study, also in Visakhapatanam City, an average concentration of 60.29 mg NO_3/lit was found at MVP colony (Kumarswamy et al., 1997) [15] and the pollution was suspected to the consequence of domestic sewage pollution.

These shows the highly variable nature of groundwater nitrate within a small geographic area and narrow time scale, indicating to the fact that local factors (like waste generation and sanitary conditions etc.) can largely influence groundwater nitrate concentration. The water in these areas is deemed unfit for drinking without proper treatment as they exceeded the maximum permissible limit for drinking water (100 mg/l) set by BIS. Few studies in India have examined the role of non-point sources in groundwater pollution. NO_3-N contents of the groundwater in and around Delhi were found to exceed the upper safe limit for potable water in a study conducted by Arora (1980) [17]. Handa (1982) [18] found a very high concentration of NO_3-N (350-700 mg/l) in groundwater of Uttar Pradesh. Singh et al. (1987) [19] found a mean concentration of 3.88 mg/l during 1982 rainy season while approximately 10% of the groundwater samples contained N concentration of 10 mg/l in highly irrigated coarse textured, highly percolating soils of Central Punjab, where 40-50 % of the applied nitrogen was lost due to leaching. High nitrate levels in the groundwater of these areas are thought to have developed due to extensive use of FYM. According to Joshi and Deb (1994) [20], occurrence of high groundwater NO_3 in isolated areas cannot be solely attributed to the use of chemical nitrogenous fertilizers only. A study by CGWB (Mehta et al., 1990) [21] hadattributed high nitrate (ranging from 3.6 to 800 mg/l) in dug well water of Ganjam district of Orissa primarily to high fertilizer application. NEERI reports that, 41% groundwater samples from villages around Nagpur Metropolitan City and 49% samples from Gulbarga district in Karnataka, which contained high levels of nitrate concentration, could be due to agricultural sources (Bulusu and Pande, 1990) [22].In Godavari basin, Karnataka, groundwater samples from dug wells showed a mean nitrate concentration of 80.91 mg/l (range 10-250 mg/l), which was attributed mainly to heavy application of chemical fertilizers (Tamta, 1991) [23]. According to Gupta (1992) [24], high nitrate in groundwater of Udaipur district (Rajasthan) could be due to five –fold increase in the use fert-N.

Comparable problems are also known from many developing countries. Water quality assessments studies carried out by by NEERI (National Environmental Engineering Research Institute, 1991) identified the States with high nitrate concentration in ground waters in India (Table 5). The widespread increases in nitrate concentrations in groundwater have been caused by an increase of the input of nitrogen into environment as a result of human activities. These nitrogen inputs have gone beyond the assimilation and elimination capabilities of the biosphere. Surface waters are predominantly polluted by nitrate from surface run-off, sub-surface flow or groundwater exchange. Intensive agricultural production, domestic and industrial wastes, sewage and atmospheric nitrogen pollution are considered to be the main sources of nitrate contamination in water.

Waste from production of the explosives, fertilizers, nitro organic compounds and pharmaceuticals are sources of nitrate pollution. Additional sources of nitrate contamination include landfill leachate, leaking septic tank and municipal storm water runoff (Hiscock K. M. et.al. 1991) [11]. Nitrate is also a byproduct of many industrial processes, including paper manufacturing. Hence, effluent containing nitrate should be treated before they are discharged into the environment. When the nitrate concentration of drinking water exceeds the maximum admissible concentration, the nitrate removal becomes a technical challenge. There are biological and physicochemical methods for the removal of nitrate. Nitrate degradation processes include biological and catalytic nitrate reduction, and separation processes include distillation (Elmidaoui et. al. 2001) [29], electro analysis (Hell F. et.al. 1998) [30], Donan dialysis (Wisviewska G and Winnicki T. 1985) [31], electro deionization (Salem et. al.1995 [32]), reverse osmosis (Ismail K. 2002 [33]), electro sorption (Afkhami A 2003) [34], chitosan (Jaafari et.al.2001) [35] and aluminium oxide (Anacarvalho et. al. 2003) [36]. For removal of nitrate from groundwater, ion exchange has become one of the most economic and effective methods. The process is superior to many other methods of water reuse by virtue of its possibility of reusing the spent resins via regeneration. Thus, this process has gained considerable interest during recent years.

The objective of this work was to evaluate the potential of natural Zeolite for the removal of nitrate from groundwater. The efficiency of a natural Zeolite-stilbite for the removal of nitrate was investigated using groundwater samples collected from Loni village of western Maharashtra of India.

II. MATERIALS AND METHODS

A. Zeolite Source and Conditioning

Samples of Zeolite were collected from the Nizarneshwar Hills in Ahmednagar district of Maharashtra of Western India. The crushed original Zeolite was ground and passed through 300×600-µm sieves and was dried in an oven at 100 ± 5 C for 24 h. It was characterized by X-ray diffraction (XRD) and chemical analysis. Chemical composition of Zeolite samples was determined by the usual analytical methods for silicate materials. Standard wet chemical analysis, along with instrumental methods, was adopted (D. R. Corbin et.al.1987) [37]. Al_2O_3, Fe_2O_3, CaO, and MgO were analysed by titrimetric methods and

SiO_2 was analysed by gravimetric methods. Na_2O and K_2O were found out by flame photometry. The results of the chemical analyses are presented in Table 1.

TABLE I : CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES OF STILBITE - NATURAL ZEOLITE SAMPLE (WT%)

Chemical Composition	(%)	Physical Properties	
$SiO2$	69.31	Appearance porosity (%),	41.5
$Al2O3$	13.11	Appearance density (g/cm3),	2.27
$Fe2O3$	1.31	Weight of per unit volume (g/cm3),	1.32
CaO	2.07	Water absorption (original) (%),	31.3
MgO	1.13	Water absorption (grinding) (%),	103.7
$Na2O$	0.52	Oil absorption (g oil/100 g sample),	51
$K2O$	2.83	Whiteness (%),	68
$SO3$	0.10	Original bleaching (g sample/g tonsil),	1.95
$H2O$	6.88	Active bleaching (g sample/g tonsil),	1.92
Si/Al	4.66	pH	7.5

The activated carbon granular LR grade was supplied by SD Fine Chem (Boisar, India) in the size range 2-5 mm. It was manufactured from coconut shells, heated and then treated with acid wash before delivery. These were subsequently pulverized and sieved through 18 to 44 BS mesh to get the activated carbon particles of the desired size range. After washing the activated carbon with distilled water, it was dried in an oven at 105^0C for 72 h. After drying, it was stored in glass bottles until use. Table 2 shows the physical and chemical characteristics of activated carbon.

TABLE II : CHARACTERISTICS OF ACTIVATED CARBON.

Physical Characteristics	
BET surface area m^2/g	579.23
Specific gravity	0.92
Bulk density g/ml	0.977
Hardness	Less than 1 (in Mohr's hardness scale)
Porosity	24.43 %
Chemical Compositions (wt %)	
Carbon	95.50
SiO_2	0.47
Al_2O_3	0.06
K_2O	0.51
Na_2O	0.06
CaO	1.54
MgO	0.08
Fe_2O_3	0.07
H_2O	1.20

B. Groundwater Sampling

Nitrate-contaminated groundwater samples were collected from four locations in Loni village of Maharashtra state. Samples were collected from wells having depths from about 70–90 ft, using pre-cleaned, acid washed plastic containers. Prior to sample collection, the containers were thoroughly rinsed with the groundwater. The samples were carefully transported to the laboratory and then stored at 5°C in the laboratory until being required for analysis. In the Loni village, the nitrate concentrations ranged from 40 to 240 mg/ l. The concentration of NO_3 was determined by UV spectrophotometer (Chemito, model 2100) at 340 nm.

C. Fixed Bed Ion Exchange Column Studies

In order to test the feasibility of natural Zeolite-Stilbite as an adsorbent for the removal of nitrate from groundwater, a continuous mode of application was studied in a glass column (60 cm x 0.8 cm) which is shown in figure 1. Natural Zeolite-Stilbite was suspended in distilled water for about 10 minutes and was then used for column studies. glass wool was kept at the bottom of the column to avoid the losses of sorbent with the flow of nitrate solution. Then the sorbents was transferred onto the glass wool in the column. Nitrate solution was fed into the column at the flow rate of 10 ml/min using a peristaltic pump. To

determine exhaustive capacity, 50-ml fractions of the effluent were collected from the bottom of the column. The process was continued until the amount of nitrate in the effluent was the same as that in the feed. PH was measured using pH meter (Elico Instruments model LI-107). The effect of pH was studied in the pH range 2 to 7 by adjusting pH of nitrate solutions using dilute HCL and NaOH solutions. The effect of flow rate was studied with different flow rate between 1 to 5 mL per min. by using peristaltic pump. The effect of different initial concentration was studied with different concentration nitrate solution in the range between 40 to 240 mg/L.

After each service cycle of the exchange, a regeneration cycle took place with the solution of sodium nitrate with the concentration of 30 g/L, by passing the solution from the top of the column through the fixed bed of Zeolite. The regeneration process was stopped when the nitrate ion concentration in the effluent became <0.1 mol/L. During ion exchange process, the changes of pH value were measured in the influent and the effluent.
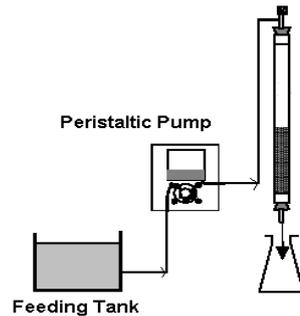

Fig.1 Experimental setup

III. RESULTS AND DISCUSSIONS

A.   *Influence of Initial Nitrate Concentration*

The effect of the initial nitrate concentration on the removal of nitrate has been examined on the Zeolite and granular activated carbon with the flow of 1.0 mL/min (Fig. 2). The amount of nitrate adsorbed increased with the increase in nitrate concentration and remained nearly constant after equilibrium time. It was observed that removal of nitrate was greater in natural Zeolite than granular activated carbon.
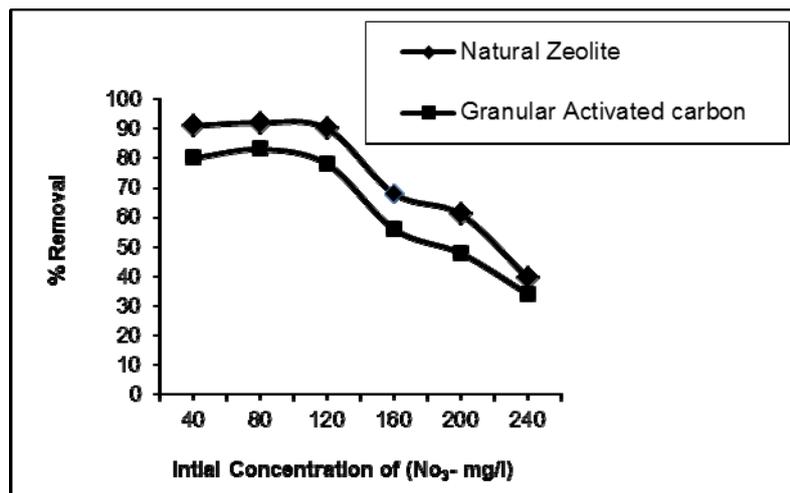

Fig. 2. Removal Efficiency Vs Intial Concentration

B. *Influence of PH*

It is well known that pH of the medium is one of the most important factors that influence the ion exchange process. As the pH increases, the removal of nitrate also increases. Figure 3 has shown the change in the rate of adsorptions due to the change in PH. At pH 4, maximum removal is possible. It was noticed that removal of nitrate was greater in natural Zeolite than granular activated carbon at pH 4.
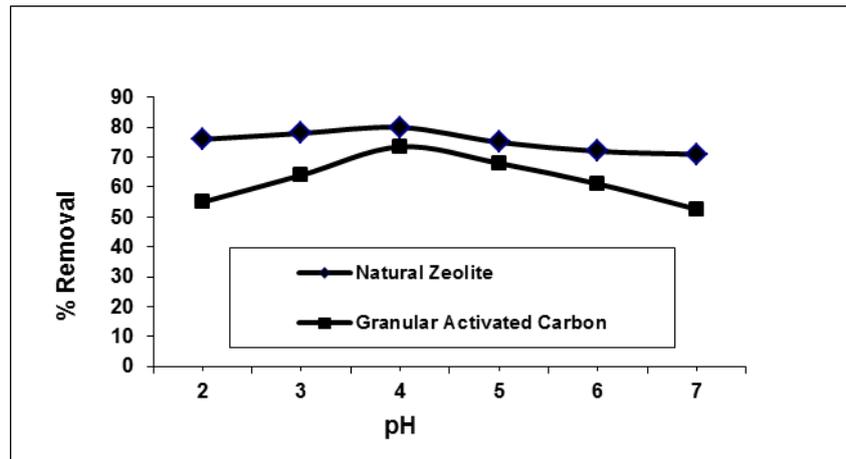
Fig. 3.Removal Efficiency Vs Ph

C. Influence of Flow Rate

Fig. 4 shows the effect of flow rate on nitrate removal for Zeolite and initial concentration of 80 mg/L. When the flow through the column increases, the removal of nitrate decreases. At 1 ml/min flow rate there was 98 % nitrate removal by natural Zeolite and 91% nitrate removal by granular activated carbon.
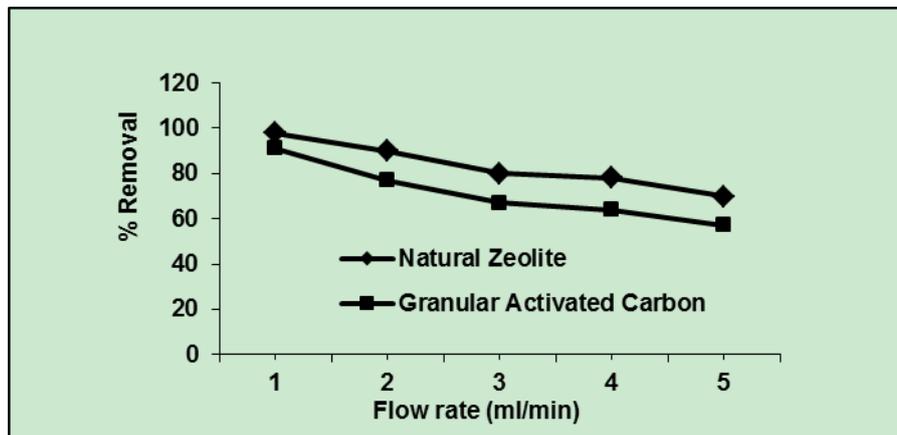

Fig. 4.Removal Efficiency Vs Flowrate

D. Fixed Bed Column Study for Nitrate Removal

The column study was conducted having initial concentration of 200-mg/lit and pH 8. The breakthrough curve obtained is shown in figure 5. The breakthrough and exhaustion (ultimate) capacities of the natural zeolite-stilbite were calculated as 6.5-mg/lit and 7.4-mg/l and for granular activated carbon, it was 7 mg/l and 7.5 mg/l.
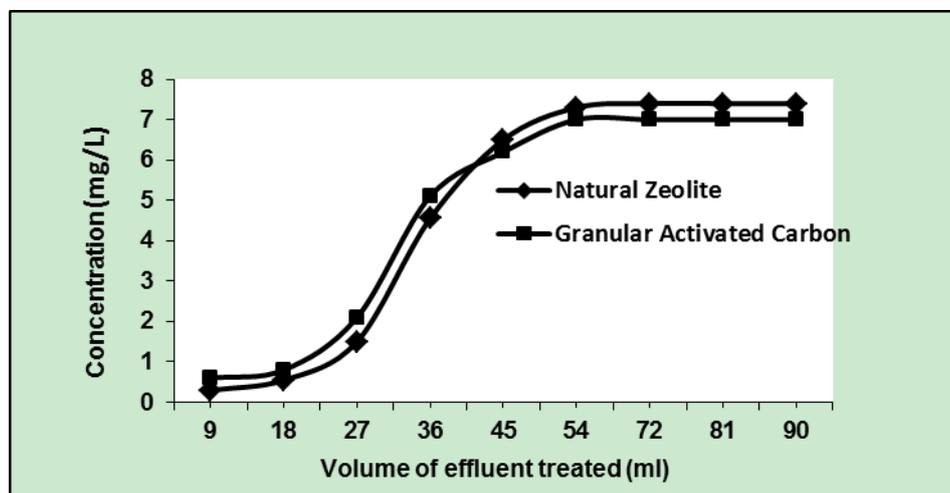

Fig. 5.Breakthrough Curve

E. Regeneration

In order to examine the effect of regeneration on the ion exchange, after each service cycle a regeneration cycle took place with the sodium chloride solution with the concentration of 30 g/L, and the flow of 1 mL/min. The results are shown in elution curves as concentration in the effluent relative to the regeneration solution volume flowed through the bed. Regeneration achieves elution of bound nitrate ions and restoration of zeolite for the next service step. As the same elution curves were obtained for all regeneration cycles, Fig. 6 shows the curves for only first cycle. The curves shows a rapid increase in the nitrate concentration in the exit solution, followed by a rapid decrease, until the nitrate concentration in the effluent reaches <0.1 mmol/L. Only 160–200 bed volume is needed for complete regeneration of the zeolite bed.
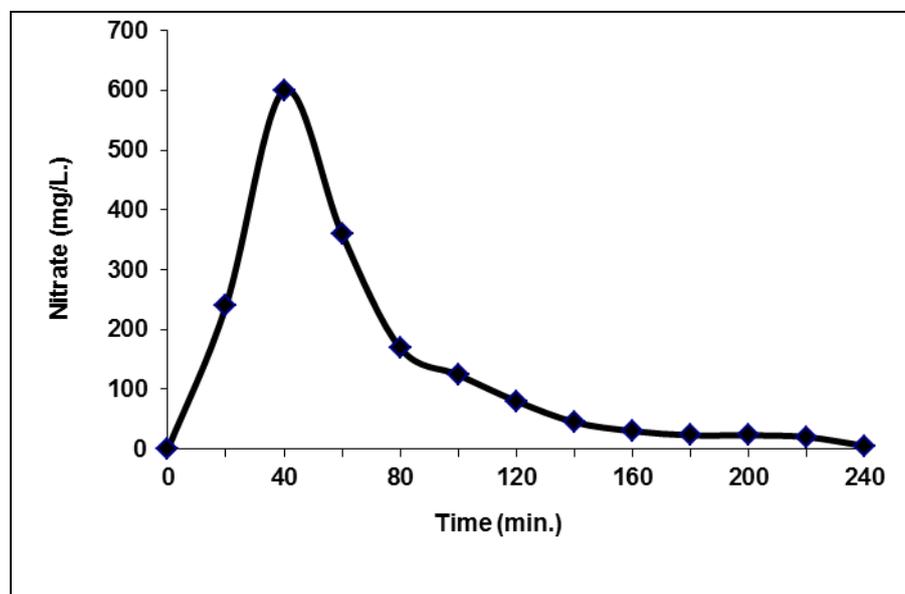

Fig. 6. Regeneration Curve

IV. CONCLUSIONS

Nitrate can be removed from groundwater very successfully by means of the column exchange process on natural Zeolite-stilbite with successive service and regeneration cycles. Already after the first cycle of regeneration with the sodium chloride solution, the sample is transformed to near homo ionic sodium form, which results in the increase of capacity that remains unchanged with successive regenerations. This makes it possible to reuse the same Zeolite sample several times. The removal decreases with the increase of nitrate concentration in the initial solution, at the same flow. When the flow increases for the solutions of the same nitrate, removal of nitrate decreases. The column experiment has shown that the best efficiency is obtained at the initial nitrate concentration of 80 mg/L and the flow of 1 mL/min. In this study, natural Zeolite-stilbite has been shown to be more effective for the removal of nitrate from ground water than granular activated carbon.

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