ASSESSING THE IMPACTS OF AGRICULTURE ON GROUNDWATER QUALITY USING NITROGEN ISOTOPES – PRELIMINARY RESULTS ON THE "GABBROS OF BEJA" AQUIFER SYSTEM (SOUTH PORTUGAL)

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ABSTRACT
This paper intends to present preliminary results of nitrogen isotope assessment and source identification and characterization of nitrate non-point pollution in the context of official publication of "Gabbros of Beja" Vulnerable Region (Portaria 1100/2004, 3 of September).
Research is based on funding project "Assessing the impacts of agriculture on groundwater quality using nitrogen isotopes" in progress in the rural region of Beja (South Portugal), under semiarid conditions in the period 2004-2007.
Supported by previous hydrogeological characterization of "Gabbros of Beja" hard rock aquifer, seasonal groundwater chemistry and nitrate content evolution under different land use scenarios, is possible to use nitrate isotopes and oxygen isotopes to trace nitrogen origin of nitrates in groundwater and nitrogen cycle.
Stable nitrogen isotopes ($^{15}N/^{14}N$ ratios) can offer a direct way to identify the pollutant sources in groundwater systems, In the research area two major sources of nitrate were identified in agricultural areas, fertilizer and manure, which present different isotopic $\delta^{15}N$ signatures.
The relative contributions of these two sources to groundwater or surface water can be estimated by mass balance. The analysis of nitrate $\delta^{18}O$ together with $\delta^{15}N$ improves the ability to trace nitrate sources and cycling.
Preliminary results are not conclusive about the hypothesis that major source of nitrate-N in groundwater comes from agriculture. Further work is necessary under regular hydrological conditions. The intent is to have scientific proves to support political decisions, considering the sustainable development of the region and the appropriate use of nitrogen fertilizers, based on several European and national directives, namely vulnerable regions and EC Water Framework Directive.

Key-words: aquifer; diffuse pollution, nitrates, isotopes.

1. INTRODUCTION

Large-scale diffuse pollution is of great concern in most European countries. Soils and groundwater bodies show increasing nitrates and pesticides concentrations due to intensive agriculture.

This paper will focus on *Assessing the Impacts of Agriculture on Groundwater Quality Using Nitrogen Isotopes* in the rural region of south Portugal known as Alentejo, namely in the "Gabbros of Beja Aquifer System" (350 km^2) in the vicinities of Beja. This Aquifer System represents the most productive hard aquifer in the region and the best agriculture land use with direct consequences regarding nitrate diffuse pollution.

Nitrate diffuse pollution greatly concerns the scientific Portuguese community and the National Water Authorities. This situation must be changed according to the EU Nitrate Framework Directive (91/676/EEC) and EC water Framework Directive (2000/60/EC). In particular, authorities should revaluate aquifer vulnerability in the context of official publication of "Gabbros of Beja" Vulnerable Region (Portaria 1100/2004, 3 of September) and mobilize funds to promote Nitrogen-use Efficiency and Nitrogen Management, regarding Environmental Farming Proposals (91/2078/EEC) and Drinking Water Regulations (80/778/EEC).

Isotopes of nitrogen were used to distinguish pollutant sources. Nitrate-nitrogen and oxygen isotopes can facilitate the distinction between fertilizer and waste as pollutant sources Several studies have indicated how important nitrate isotopes can be in areas where there is evidence of multiple sources of pollution and nitrate is one of the most widespread pollutants of groundwater

The use of isotope techniques in hydrogeology is related with two main issues:

- improving the understanding of groundwater flow system ;
- tracing the origin and patways of a range of groundwater pollutants

Isotopic techniques were used to confirm the results obtained by hydrochemical studies and nitrate seasonal monitoring program between 1997 and 2000 (Paralta & Ribeiro, 2001). It is essential to integrate isotope techniques with conventional hydrochemistry.

Nitrate in groundwater in both agriculture and urban areas require well-focused studies as well as dating groundwater bodies based in environmental tritium and other isotopes.

Research is based on funding project "Assessing the impacts of agriculture on groundwater quality using nitrogen isotopes" in progress in the rural region of Beja (South Portugal), under semiarid conditions for the period 2004-2007. National Research Authorities (Science and Technology Foundation - FCT) supports the project.

2. STUDY AREA OVERVIEW

2.1 Hydrogeology

The study area is located in a wide region of South of Portugal, between Ferreira do Alentejo (NW) and Serpa (SE) (see figure 1), covering an area of about 350 km^2 in Ossa-Morena geotectonic unit. A case study has been established nearby the city of Beja.

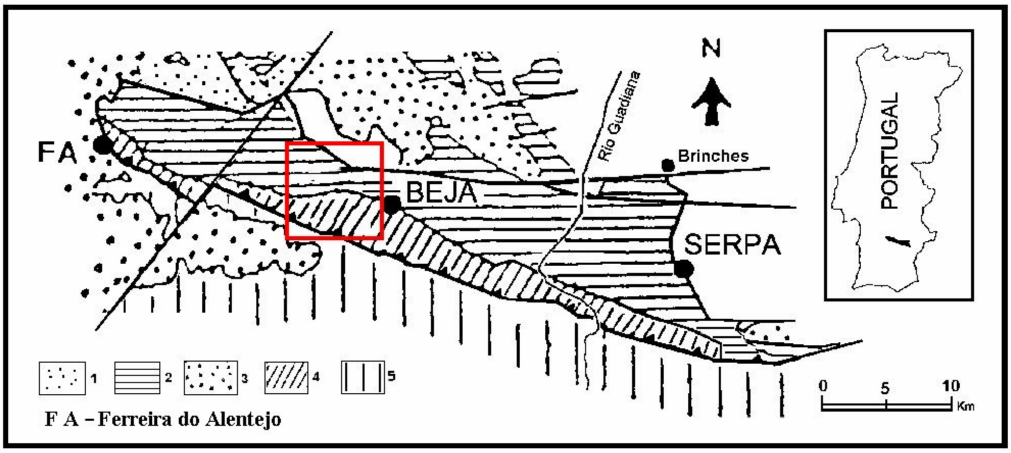

Figure 1 – Geographical location of “Gabbros of Beja” Aquifer (350 km^2) and study area (50 km^2). General geology adapted from Fonseca (1995). 1- Odivelas Volcano-sedimentary Complex. 2- Maphic and intermediate plutonic rocks (Beja`s Gabbro Complex). 3- Plutonic acid and sub-volcanic rocks (Baleizão Porphirs). 4- Beja-Acebuches Ofiolitic Complex (meta-gabbros and basic meta-volcanites). 5- Pulo do Lobo Accretion Terrain (South Portuguese Geotectonic Unit – schists).

The gabbro-dioritic shallow aquifer is one of the most productive formations of the Alentejo region when compared with other hard aquifers in this region. In the upper weathered zones, down to about 30 m deep, the aquifer is unconfined type, while after crossing unweathered rock, water circulation is mainly based on secondary porosity. Main features of Gabbros aquifer are reported in Duque (1997).
Chemical weathering of the gabbro-dioritic rocks results in the production of clay minerals, such as illite, chlorite and montmorilonite. Up to the surface, as a direct result of the semi-arid and dry climate, carbonate deposits resulting from water evaporation, called “caliços”, may occur, as a result of mobilization and re-precipitation of Ca^{2+} in the solution.

Recharge is calculated between 10 % and 20 % (some places more) of average annual rainfall (500 mm/yr) according to recent recharge models, and occurs mainly between January and March/April (Paralta 2001, Paralta *et al.* 2003, Paralta & Oliveira 2005).
In the study area well productivity's range from 1 to 15 L/s with most frequent average values around 5 L/s and unproductive drills less than 20 %. Transmissivity most frequent values are around 100 m^2/day. Hydrochemical characterization indicates that these waters are mineralized (with TDS reported to range from about 400 mg/L to 900 mg/L). The main facies are calcium-magnesium bicarbonate and magnesium-calcium bicarbonate.
A hydrochemical monitoring was carried out between July 1997 and July 2000 to assess spatial and temporal variability of nitrate contents in the aquifer due to seasonal fertilization and rainfall episodes. A large range of values was recorded but more frequently classes were 50-60 and 70-80 $mgNO_3$/L.
The intra-annual analysis has a large range variation reaching 100 mg/l and median range from 53 mg/L to 86 $mgNO_3$/L.
Spatial structural analysis of diffuse pollution was performed using geostatistc techniques namely indicator variography and indicator kriging. Probability maps of exceeding specific cut-off of 50 $mgNO_3$/L represent stochastic simulations and therefore equally probable images of seasonal nitrate contamination useful in environmental management and municipal policies (Paralta & Ribeiro, 2001).
Assess diffuse pollution index based in the structure/de-structuration analysis of indicator variogram meaning continuity/non-continuity of contamination pattern shows that "maximum" diffuse pollution in July 99 was around 60 $mgNO_3$/L.
Frequency distribution was highly skewed (Lognormal) and the coefficient of variation ranges from 37 % to 63 % with median around 47%.
The extreme variability of time-space nitrate diffuse pollution makes it difficult to establish accurate predictive models for environmental management.
Outputs such as diagrams, hydrogeochemical indexes and risk/probability maps could be of major importance in groundwater management and also in the implementation of quality monitoring network systems (Nunes *et al.,* 2004).

2.2 Land Use – Agriculture

Beja region is influenced by the Mediterranean climate, with large temperature intervals between summer and winter and cyclical droughts. The annual average temperature is 16 ºC and average rainfall is about 500-600 mm/year. There are two distinct periods. The warm and dry period occurs between June and September and the wet period occurs between October and March, with 75 % of total annual rainfall.
Increasing concentrations of nitrate in groundwater supplies for Beja municipality was recognized early in the 1940s. The actual situation is the long-term consequence of major changes in agricultural cultivation in Alentejo during 1930-1940 directed to increase national grain production. The following decades will lead to substantial increase in application of inorganic fertilizers to sustain more continuous cereal cultivation.
Nearby Beja land use is mainly cereal (wheat) and sunflower or corn as alternative crops. Under intensive cereal crops fertilizer application in the range of 100-150

kgN/ha/yr is common. The sunflower and corn crops usually do not need fertilizer but groundwater pumping for irrigation is important in the range of 4000-5500 m^3/ha/yr. For an average nitrate content of 50 $mgNO_3$/L irrigation flux represents 45 kgN/ha/yr to 62 kgN/ha/yr.
Seasonal rainfall and variation of nitrate leaching at the shallow water-table were monitored between 1997 and 1999. Results show a slightly increase trend of nitrate contents in the beginning of spring and a large range variation, reaching 100 mg/L.
The relationship between nitrogen leaching, agriculture practices, crops and seasonal rainfall is not evident, due to climate irregularity and alternative crops, but annual variations are significant.
The nitrate ion content in groundwater is thought to be due mainly to processes of natural nitrification, decomposition of organic material and human pollution, namely agriculture related to the use of nitrogenous fertilizers in farming.

2.3 Aquifer Vulnerability

The vulnerability maps constitute a set of tools that were developed with the aim of preventing groundwater pollution. These maps define spatially the degree of protection of an aquifer to anthropogenic and natural pollution.
Regarding "Gabbros of Beja" Aquifer System several comparative studies based in different methods were tested (Paralta *et al.* 2005).
Aquifer vulnerability assessment was made using DRASTIC (Aller *et al*. 1987), GOD (Foster 1987) and AVI (Van Stempvoort *et al.* 1993).
A new method named Susceptibility Index (SI) by Ribeiro (2000) was also tested, considering an additional parameter: the land use and all the associated impacts. SI is the weighted sum of five parameters: Depth to the water table (D); annual recharge (R); aquifer lithology (A); topography (T) and land use type (LU). Weights were inferred on the basis of a Delphi panel of Portuguese specialists.
Comparative results for intrinsic methods show clearly that DRASTIC dominant class is 100-119 and DRASTIC PESTICIDE class is 120-130, meaning low and low to average vulnerability index. For AVI index, the results are in the range of 1 to 2.7, meaning extremely high risk. For GOD, results are between 0.2 to 0.4 meaning low to moderate vulnerability.
Using SI and considering land use and related practices, dominant class is 65-75 %, meaning average-high vulnerability.

3. ISOTOPIC TECHNIQUES

This paper documents the development of appropriate and innovative techniques to enable isotope measurements in order to discriminate nitrogen source in groundwater of unconfined shallow aquifer near Beja (South Portugal) recently declared as vulnerable area to nitrate diffuse pollution.
Water analyses were performed in Instituto Tecnológico e Nuclear (ITN-Portugal) regarding $\delta^{18}O$ and $\delta^{2}H$ and in Instituto de Ciência Aplicada e Tecnologia (ICAT-Portugal) regarding $\delta^{15}N$. All the isotopic determinations ($\delta^{2}H$, $\delta^{15}N$ and $\delta^{18}O$) were carried out using a mass spectrometer for light isotopes.

Pollution associated to agriculture has had a direct and indirect effect on groundwater recharge and on aquifer biogeochemistry rates and composition. Direct effects include dissolution and transport of excess quantities of fertilizers (Bohlke, 2002). Using the mass spectrometry methodology, isotopic differences are found in most terrestrial materials has $\delta^{15}N$ compositions between -20 and +30 $^{o}/oo$ (Kendall & McDonnell, 1998).

The dominant source of nitrogen is the atmosphere ($\delta^{15}N=0$ $^{o}/oo$). Many plants fix nitrogen and organisms cycle this nitrogen into the soil. Other sources of nitrogen to watersheds include fertilizers produced from atmospheric nitrogen with compositions of 0 to 3 $^{o}/oo$ and animal manure with nitrate $\delta^{15}N$ values generally in the range of +10 to +25 $^{o}/oo$. Two factors control the $\delta^{15}N$ values of any N-bearing compound in the subsurface: (1) variations in the $\delta^{15}N$ values of inputs (sources) and outputs (sinks) of the compound in the subsurface, and (2) chemical, physical and biological transformations of materials within the soil or groundwater that produce or remove the compound.

The analysis of both $\delta^{18}O$ and $\delta^{15}N$ of nitrates provides excellent separation of nitrate sources. The nitrates formed in waters with $\delta^{18}O$ values in the range of -25 to -5 $^{o}/oo$ should have $\delta^{18}O$ values in the range of -9 to +4 $^{o}/oo$. The $\delta^{15}N$ of atmospheric nitrate has a moderate range of composition around 0 $^{o}/oo$ (Kendall *et al*. 1996).

Under ideal circumstances, stable nitrogen isotopes offer a direct mean of source identification, because the two major sources of nitrate in many agricultural areas, fertilizer and manure, have isotopically distinct $\delta^{15}N$ values. The relative contributions of these two sources to groundwater or surface water can be estimated by mass balance. However, soil-derived nitrate and fertilizer nitrate commonly have overlapping $\delta^{15}N$ values, preventing their separation using $\delta^{15}N$ alone, but the analysis of nitrate $\delta^{18}O$ together with $\delta^{15}N$ improve the ability to trace nitrate sources and cycling.

In hydrology studies $\delta^{15}N$ are used also as a tracer in the identification of aquifer mixtures. According to Joseph *et al.* (1987) the relationships between various aquifers that are linked in succession along a general flow gradient, the nitrate concentrations and the different isotopic $\delta^{15}N$ content on groundwater allow the identification and quantification of the interconnection between different groundwater systems.

The methodologies and techniques proposed in this study may be obtained through the bibliography. The critical analysis of the methods, with the necessary rigor, would occupy space that isn't available in the format of this paper.

4. RESULTS AND DISCUSSION

Field sampling was carried out during December 2004 and 16 water samples were collected. One sample represents city sewage (sample nº 1), 9 samples were collected from shallow wells (up to 10 m deep), corresponding to samples 2 to 10. Also 5 boreholes with depths around the 30 to 40 m were sampled (samples n. 11 to 15) and 1 spring (sample n. 16). See sample points in figure 2.

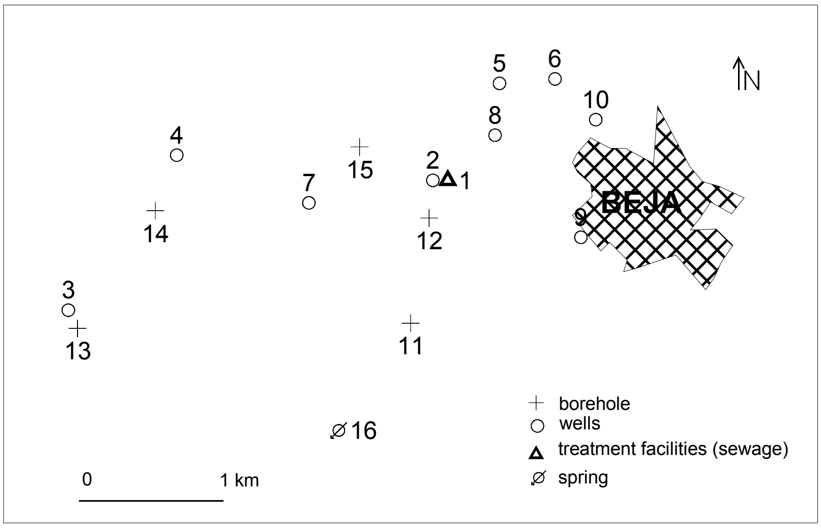

Figure 2 – Study area and sampling locations.

The results obtained from the physical-chemical analyses are presented in table 1.
Waters are medium mineralized, with electrical conductivity (EC) between 300 and 1300 µS/cm and calcium-magnesium facies.
Nitrates in groundwater are in the range of 2 to 66 mgNO_3/L with most common records between 35 and 60 mgNO_3/L.
Water sample from wastewater treatment facilities (sample n. 1) shows the highest mineralization (EC= 2370 µS/cm) and low NO_3 and N-NO_3 content (less than 2 mg/L).
The isotopic composition of the groundwater samples collected in the shallow wells, boreholes, spring and sewage were plotted in a $\delta\ ^2H$ *vs* $\delta\ ^{18}O$ diagram (Figure 3). Also in this figure the Local Meteoric Water Line (LMWL) have been represented, estimated from the isotopic composition of precipitation in Beja meteorological station (ITN database; monthly samples from 1988-2001). The equation of the LMWL is $\delta^2H = 7.67\ \delta^{18}O + 8.93$.
From the diagram it is possible to observe that the spring is plotted relatively closer from the LMWL, as well as the majority of the borehole water samples. The shallow groundwater samples and sewage present a deviation from this line, most probably related with isotopic fractionation due to evaporation processes. Also from the diagram a good relation between the isotopic composition of the sewage and the isotopic composition of the shallow groundwater samples (wells) can be observed.

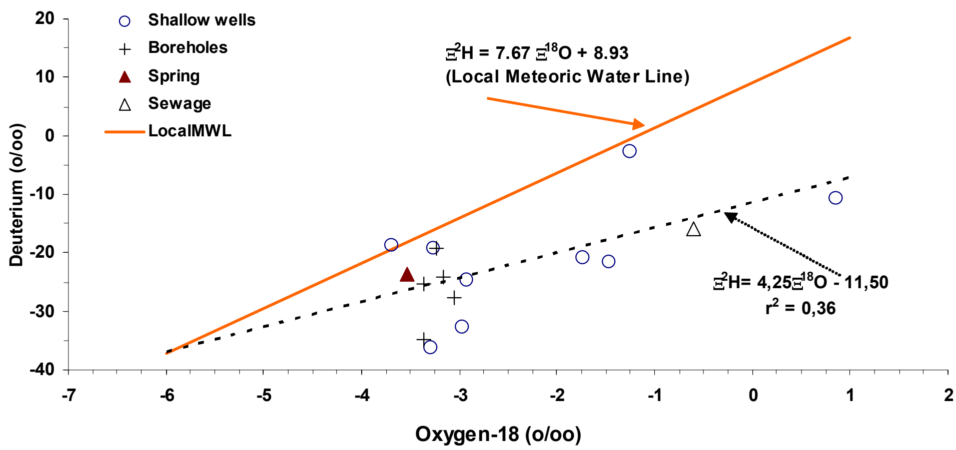

Figure 3 - δ^{2}H *vs* δ^{18}O for groundwater samples from the study area.

Previous data and field practice indicate that samples nº 2, 4 and 9 could have domestic/sewage contribution to nitrate content and that other locations (samples nº 3, 5, 6, 7, 8, 10, 11, 12, 13, 14, 15 and 16) should be influenced by fertilizers, since wells are located in the open field.

Table 1 – Physical and chemical composition of water samples (December 2004).

Code	Sampling point	pH	EC (uS/cm)	Temp (ºC)	Ca (mg/L)	Na (mg/L)	Mg (mg/L)	K (mg/L)	HCO_3 (mg/L)	SO_4 (mg/L)	Cl (mg/L)	N-NO_3 (mg/L)	NO_3 (mg/L)	SiO_2 (mg/L)
1	Sewage	7.48	2370	9.5	92.00	238.00	35.00	19.90	428.0	87.99	509.0	0.29	1.28	18.80
2	Well	8.07	1295	13.7	101.00	51.00	60.00	0.32	267.5	69.48	251.0	8.80	38.97	28.85
3	Well	8.55	618	11.4	28.20	37.50	33.25	0.06	223.0	82.30	45.0	8.53	37.78	30.28
4	Well	7.97	726	14.3	76.00	38.00	18.75	0.08	241.5	83.66	38.0	13.94	61.73	27.93
5	Well	8.24	796	9.1	63.00	41.00	35.50	1.19	275.0	86.85	88.0	4.27	18.91	28.78
6	Well	7.63	695	15.4	87.50	12.00	23.25	0.14	226.0	44.42	37.0	14.97	66.30	34.29
7	Well	7.46	723	12.6	77.75	30.50	24.25	0.05	283.0	78.55	32.0	11.94	52.88	34.41
8	Well	7.40	1054	17.2	100.00	37.50	43.75	0.12	296.0	79.26	119.0	13.11	58.06	37.58
9	Well	7.25	1228	18.5	139.00	34.00	45.75	0.54	329.5	76.98	167.0	12.14	53.76	41.22
10	Well	7.62	371	10.6	45.00	14.00	10.25	3.12	210.0	7.19	27.0	0.60	2.66	22.94
11	Borehole	7.52	672	12.5	70.75	23.00	26.50	0.20	228.5	81.59	43.0	9.25	40.96	30.37
12	Borehole	7.51	615	19.3	61.25	22.50	24.00	0.07	267.5	43.14	39.0	9.11	40.34	30.09
13	Borehole	7.23	796	14.4	74.25	32.50	31.75	2.62	322.0	73.00	49.0	10.37	45.92	29.08
14	Borehole	7.32	780	17.0	76.50	43.00	24.00	0.08	264.5	79.07	50.0	12.67	56.11	34.50
15	Borehole	7.53	792	17.1	73.75	29.00	31.50	0.06	239.0	76.03	82.0	9.41	41.67	34.15
16	Spring	7.53	675	18.5	52.50	23.00	41.50	0.06	288.0	37.03	37.0	9.37	41.50	38.07

Isotopic data was used to confirm this conceptual model and the magnitude of source contribution. Results of isotopic analyses are show in table 2.

Table 2 – Isotope data analyses (December 2004)

Code	**Sampling point**	**δ ^{15}N** ($^{o}/_{oo}$)	**δ ^{18}O** (H_2O) ($^{o}/_{oo}$)	**δ ^{2}H** (H_2O) ($^{o}/_{oo}$)
1	Sewage	16.14	-0.60	-15.90
2	Well	18.22	-1.46	-21.60
3	Well	12.42	-1.26	-2.60
4	Well	…..	-2.93	-24.60
5	Well	26.17	0.86	-10.80
6	Well	3.69	-3.27	-19.20
7	Well	3.07	-3.29	-36.30
8	Well	13.38	-3.69	-18.70
9	Well	13.63	-2.96	-32.60
10	Well	11.95	-1.73	-20.90
11	Borehole	8.12	-3.37	-25.30
12	Borehole	26.12	-3.37	-34.80
13	Borehole	…..	-3.17	-24.20
14	Borehole	3.50	-3.24	-19.30
15	Borehole	…..	-3.05	-27.60
16	Spring	…..	-3.53	-23.60

Nitrogen isotope ratios on groundwaters show a range in δ^{15}N from +3 ^{o}/oo to +26 ^{o}/oo. For sewage sample, δ^{15}N is +16.14 ^{o}/oo.
Theses results overlap between the δ^{15}N values of both animal and septic tank wastes (Figure 4).

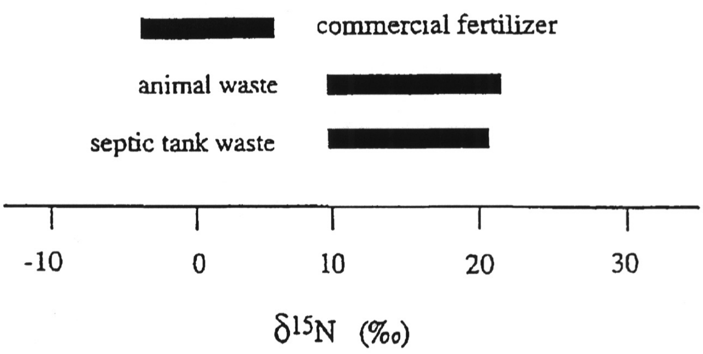

Figure 4 – Range of $\delta^{15}N$ values for the major sources of nitrates into groundwaters (Wassenaar 1995).

Oxygen isotope $\delta^{18}O$ data are in the range of -3.7 $^{o}/oo$ to + 0.86 $^{o}/oo$ for groundwaters and is -0.6 $^{o}/oo$ for sewage.
Figure 4 shows the normal range of $\delta^{18}O$ and $\delta^{15}N$ values for dominant sources of nitrate. Nitrate derived from ammonium fertilizer, soil, organic matter and animal manure have overlapping $\delta^{18}O$ values; for these sources $\delta^{15}N$ is a better discriminator. In contrast, nitrate derived from nitrate fertilizer or atmospheric sources are readily separable from microbial nitrate using $\delta^{18}O$, even though the $\delta^{15}N$ values are overlapping. The dual isotope method has proved quite useful for source identification is some surface-groundwater studies (Kendall, 1996).
Nitrification of ammonium and/or organic-N in fertilizer, precipitation, and organic waste can produce a large range of δ values. Soil waters tend to have higher NO_3-$\delta^{18}O$ values and a larger range of NO_3 - $\delta^{18}O$ values than groundwaters due to higher $\delta^{18}O$ values of O_2 and/or H_2O in soils.
Oxygen isotope from the NO_3 determinations indicates that fertilizers do not provide a source of nitrate to groundwater. The combination of both $\delta^{15}N$ and $\delta^{18}O$ data indicate that signatures observed in groundwater are the result of ammonia nitrification originating from either animal wastes or septic tank wastes, with no significant influence from commercial fertilizers.
Samples 6, 7, 11 and 14 could have influence of soil nitrogen and others are more related with manure and septic waste (Figure 5).
For samples 1 and 2 related with sewage facilities the dual use of $\delta^{15}N$ and $\delta^{18}O$ provide positive identification of nitrogen source.

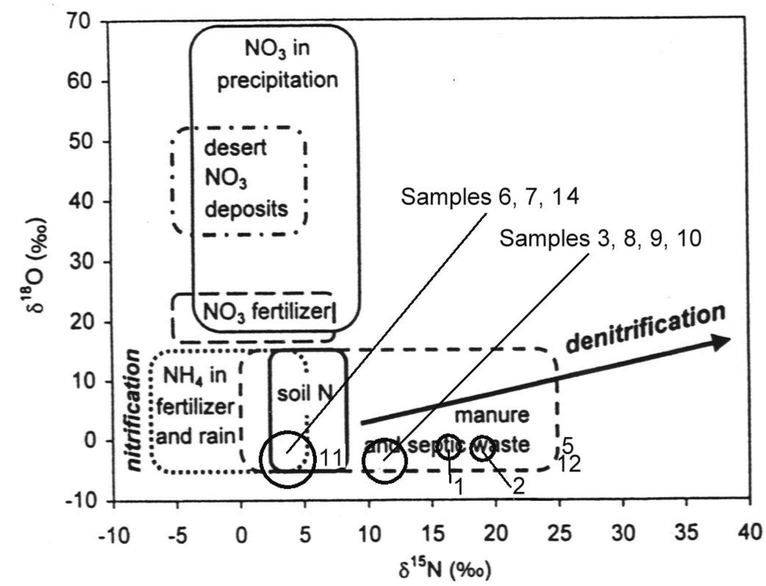

Figure 5 – Schematic of typical ranges of $\delta^{18}O$ and $\delta^{15}N$ values of nitrate from various sources (in Kendal & McDonnell, 1998) and sample plot.

5. CONCLUSIONS

Progress has been made in Portuguese research centers to allow determination of $\delta^{18}O$ and $\delta^{15}N$ in nitrates. Further work is required to ensure robust techniques with sufficient precision accuracy.

Using the current techniques it is possible to discriminate different sources of anthropogenic contamination into ground and surface systems.

According field practice and previous monitoring, conceptual model of non-point agriculture pollution in the rural area of Beja indicates that major cause of pollution comes from fertilizers.

However, preliminary results are not conclusive about the possibility that major source of nitrate-N in groundwater comes from agriculture. Further work is necessary under regular hydrological conditions, since sampling period in December 2004 was under severe drought.

There are several factors in the ecosystems than can significantly modify de $\delta^{15}N$ values. Mixing of point and non-point sources along shallow flowpaths makes determinations of sources, extent and denitrification processes very difficult to identify.

For samples 1 and 2, related with sewage facilities, the dual use of $\delta^{15}N$ and $\delta^{18}O$ provide positive identification of nitrogen source.

For other samples, it seems that they are contaminated with NO_3 from fertilizer origin. Results show relation with manure and septic waste.

Work will go on in order to confirm or not the real origin of nitrate in “Gabbros of Beja” aquifer system and source contribution.

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