



Feasibility of electrokinetic nitrogen supply for soil fertilization purposes

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Abstract

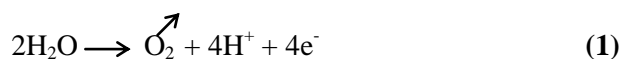
This article presents the results of the application of electrokinetic technique to fertilize an agricultural natural soil with nitric nitrogen (as NO_3^-). The injection into the soil of amounts of nitrate proportional to the applied electric current is adapted to the plant needs. This would help to reduce the nitrate pollution in waters generated in part by excessive fertilizer inputs in agricultural systems. The pilot test was realized at constant current of 2.54A (current density $6.2\text{A}\cdot\text{m}^{-2}$) on a soil mass of 46.7 kg. All results showed the injection and transport of nitrate through the soil. The electromigration is the preponderant transport mechanism. Nitrate ions were transported throughout the soil sample at contents between 208-1428 mg $\text{N}\cdot\text{kg}^{-1}$ of soil, on the ground corresponding to a nitrogen rate between 80-550 kg $\text{N}\cdot\text{ha}^{-1}$. pH is maintained at its initial value in electrolytic compartments in electrolytic compartments. the electrical energy consumed in the pilot test was about $32.9\text{Wh}\cdot\text{kg}^{-1}$.

Keywords: Electrokinetic, Pilot-scale, Nitrogen, Fertilization, Soil, Agriculture.

1. Introduction

Nitrate as a nitrogen source is necessary in soil, this is an essential constituent of living matter (proteins, chlorophyll and other vital molecules). In agricultural soils, nitrate (NO_3^-) is the predominant form of inorganic nitrogen available to plants. The nitrate soil fertilization by different organic and inorganic fertilizers has a significant effect on yield and quality of agricultural crops [1,2]. However excessive use of nitrates in the soil has caused their accumulation in aquatic environments [3,4]. The nitrate pollution is a problem that affects all waters of the globe, due to environmental and health problems it may generates [1, 4-6]. The cause is the imbalance between the massive quantities of nitrates introduced into the soil, which exceeds the real needs of the crops [4]. The contamination is facilitated by different factors such as low retention of nitrate (NO_3^-) in the soil, soil type, irrigation, plant type, etc. [6]. Action programs to limit fertilizer application in Europe at $170\text{kg}\text{N}\text{ha}^{-1}\cdot\text{year}^{-1}$ [5,7] have enabled the reduction of nitrate pollution in some countries, But for many others the problem remains unsolved [3,4].

The electrokinetic process is an innovative technology for soil decontamination and low permeability media. This technique is based on the transport of chemical species in the soil under influence of an electric field applied between two electrodes (anode and cathode). In this case, the main transport phenomena are electromigration, electroosmosis (EO) and electrophoresis. This last later one is often neglected in media with low permeability and consolidated. The other consequence of the electric current is oxidation-reduction reactions at the electrodes, dominated by the electrolysis of water [8,9]. Indeed, the ions H^+ produced at the anode (equation 1) will move through the soil in acid front to the cathode, whereas the ions OH^- produced at the cathode (equation 2) will move towards to the anode in basic front less rapid (ratio of ion mobilities H^+ / OH^- is 1.76); which will change the pH of the soil [8].



The movement motion of chemical species through the soil is influenced by parameters such as pH, the electric density current, the voltage, the hydraulic gradient and temperature [8,9]. The electrokinetic technique is effective in the saturated and unsaturated soil, a humidity content higher than 5% is sufficient [8-10]. It is potentially economical due to low electrical currents used [8,9] and the possibility of using photovoltaic electricity [11]. Studies conducted in the laboratory and pilot-scale showed an interesting efficiency for a variety of chemical species such as heavy metals, organic compounds, insecticides, radionuclides [9] and anions [9, 12,13]. The electrokinetic process was coupled in many cases with other technologies such as biological methods, to improve the decontamination of soil or sludge. In this case, the electrokinetic phenomena were used for the transport of pollutants or nutrients and the microbiological phenomena for biodegradation [9,14]. From an ecological standpoint, soil microflora plays a vital role in various activities it intervenes in the nitrogen cycle (N) and thus its natural availability in soil [15]. The effects of electric current used (a gradient of electrical potential of 0.2 to 2.0 V.cm⁻¹) are negligible on micro-organisms soil [14,16]. At the electrodes, the effects of the oxidation-reductions reactions generators of hydrogen and oxygen on the microorganisms are limited [9, 14, 16].

The objective of this study was the use of electrokinetic technique to fertilize an agricultural natural soil with nitrogen (as NO₃⁻), virgin of any amendment or fertilizer. This technique has served to inject quantities of nitrate in the soil as a function of applied electric current, as a substitute for other conventional spreading techniques. During this pilot trial, different parameters were monitored such as NO₃⁻ concentration, pH, voltage, the electroosmotic flow (EOF), etc.

2. Experimental

2.1. Soil analysis

The studied soil was collected from a farming area, situated in the locality of "Boumaiza" at 40 km southeast of Annaba north-eastern Algeria. The sample was taken as a surface area of 3 m² and 0.01 m depth, the chosen plot is not yet cultivated, virgin of any amendment or fertilizer with a bare surface (no plants or plant debris). A soil sample of ~ 200 kg underwent a preliminary drying in open air for 48 h followed by sieving to 2 mm particle size. While all parameters that may be altered, were rapidly analyzed, in accordance with conservation rules and standardized methods of analysis [17,18]. The soil structure was carried out by X-ray diffraction (XRD, Siemens Munich, Germany). Particle size analysis (wet process) was performed on a apparatus Laser-Malverne Mastersizer. The specific surface area was measured in a BET apparatus Micrometrics ASAP 2010. Carbon and nitrogen are identified after combustion "Flash" at the temporary temperature of 1800°C on apparatus NA2100 CHN (Thermoquest Analyser). The organic carbon was determined according to Afnor standard (NF X31-109) [19]. The total concentration of exchangeable cations of basic character (Ca²⁺, Mg²⁺, K⁺, Na⁺) and cationic exchange capacity or CEC were carried out according to the Afnor standard (NF X31-130) [19]. The other elements (Fe, Al, Ti, Si) are analyzed chemically according to standardized conventional protocols (NF X 31-151) [19]. The total porosity is determined by formulating Duchofour from the real and apparent densities [20]. With respect to analyzes of moisture, pH (pH meter scientific IQ 240), they are realized from Afnor standardized techniques [17]. The state of the soil surface charge was established by measuring the zeta potential, about 20 mg of soil powder contained in 50 ml of distilled water (Zeta-meter, 3000 HS from Malvern Instrument). To study the capacity of the soil to adsorb NO₃⁻, solutions of 50 ml of NO₃⁻ at 2 mmol.l⁻¹ were mixed with different masses of soil in sealed vials air and stirred for 24 hours. The nitrate ions are analyzed by ion chromatography (Dionex DX 500, USA) in the interstitial fluid of the soil. This liquid is extracted by centrifugation (8000 revolutions per min for 15 min) and vacuum filtration (0.45µm filter).

2.2. Equipment and operating conditions

The study conducted at the laboratory scale showed the close relationship between the applied electric current and the amount of NO₃⁻ (or nitrogen) electrokinetically injected into the soil [21]. It also enabled the optimization of various experimental parameters for the implementation of the pilot test, based on the choice of electrolytic solutions anodic and cathodic and their flow rates, as well as the electric current which was selected on the basis of the report between the nitrate ions injected into the soil and energy minimum consumed. The

pilot test was performed under an electric current imposed of 2.54A (electric current density of 6.2Am^{-2}) in a cylindrical reactor made of high density polypropylene (Fig. 1a). It consists of three distinct parts: two electrolyte compartments anodic and cathodic with a volume of 4.6 liter each containing the electrodes and the measuring elements, they are located on either each side of the reactor. The central cylinder with a diameter of 72 cm and a length of 7 cm is intended to contain the soil. The electrodes titanium-iridium to 8% has the same section as the central cylinder, the inter-electrode distance is $L_{Pilot} = 0.07$ cm. A hydrophilic nylon membrane with pore sizes from $0.8\ \mu\text{m}$ was placed between the electrodes and the central compartment containing the soil to prevent the migration or dispersion of soil particles in the anodic and cathodic compartments. In this central portion, the soil mass introduced was 46.7 kg. The cell lying laterally on one of the compartments, is filled with dry soil, by compaction and successive weighings up soil consolidation; the saturation is carried out with demineralized water. The device is then raised upright and left several hours before its use. The supply of saline solutions (fresh) is ensured continuously in the compartments anode (NaOH (Merck) $100\ \text{mmol.l}^{-1}$) and cathodic (KNO_3 (Merck) à $106\ \text{mmol.l}^{-1}$, adjusted at $\text{pH}=2.2$ with HNO_3 (Merck)) 2 and 3 liters / hour respectively, through a system located on each side of the electrolytic compartments. This system consists of a recirculation circuit (RC) at high-speed solutions (180 liters/hour) provided by a vane pump calibrated (VEM-Motors Thurn, GmbH) and synchronized electrovalves whose opening and closing enable the introduction of fresh solutions and the extraction of electrolyte solutions. In this system, the recirculation (RC) provides; a perfect mix in the compartments and enables evacuation of the gases produced in the compartments. Introduced electrolytic solutions, allow constantly renew the nitrate solution in the cathodic compartment, retrieve ions NO_3^- at the anode and neutralize the ions produced by the electrolysis of water (H^+ in the anode, OH^- at the cathode according to the equations 1 and 2). The electrical circuit includes a power supply (DC, Aim Thurlby Thandar-Ins, England) and two multimeters (Metrix MX 579, France): one in series (amperemeter A) and the other in parallel (voltmeter, V). During the experiment, automatic samples of the solutions are carried out at regular time at the overflow tray (TP), of each of the anode and cathode compartments to be used to analyze nitrate. Flows are measured at the outlet of the overflows tray by balances (OHAUS to $\pm 5\text{g}$) placed under the recovery containers of solutions. These flow rates are used to experimentally determine the electroosmotic flow (EOF). The acquisition over time of different measures (voltage V, current I, anode and cathode flow rate) is performed directly on a computer. At the end of experiment soil samples are taken at different heights "P"; Top ($\text{P} = \text{T}$); Middle ($\text{P} = \text{M}$); Bottom ($\text{P} = \text{B}$) (Fig. 1b). Each sample is then cut into seven identical sections numbered from the anode (S_{P-1} to S_{P-7} , Fig. 1b) to be used to analyze nitrate and pH (Consort C834, Belgium).

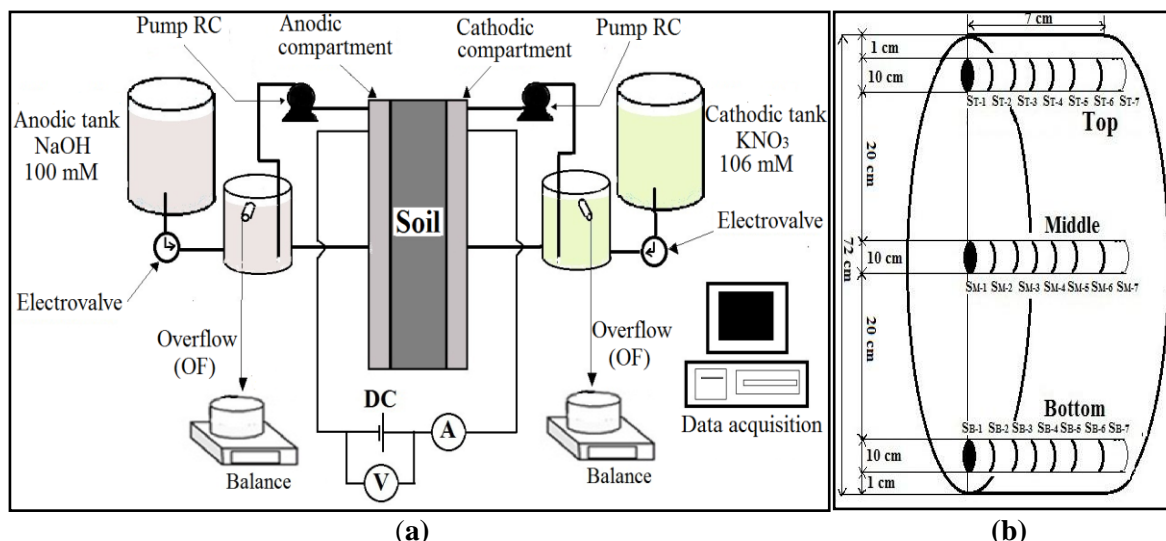


Figure 1: Schematic of experimental setup and sampling positions: (a) Schematic diagram of experimental set-up, (b) Central compartment (Soil): Samples and sections.

3. Results and discussion

3.1. Soil analysis

The physicochemical characteristics and properties of natural soil studied are summarized in Table 1. This soil is silty clay type. The examination of the diffractogram, has shown that it is essentially composed of : Quartz, Illite, kaolinite, montmorillonite. Particle size analysis was used to determine the coefficients of the

permeability K and the uniformity U of the soil. From the approximation taken by Letourneur and Michel [22] on clay soils, the value of K shows that the studied soil is low permeable. As for the uniformity coefficient U, it indicates according to convention of Castany [23], that the soil particle size is varied. Further, the low value of the specific surface, the chemical composition with high content of SiO₂ and Al₂O₃, confirm more the clayey nature of the sample. The presence of iron in important proportion gives to this sample a fixer power towards some elements such as phosphorus. The soil has a low content total carbon and organic carbon C_{Organic}, (estimated at 0.23%). The low content of organic matter would probably have a negative influence on the storage of nitrogen in the soil, this can be observed in the report C_{Organic}/N_{Total}=2.6 which shows the high mineralization of nitrogen. This would increase the risk of leaching of nitrate nitrogen which thus be lost for plants [15, 20, 24]. An intake of nitrate nitrogen by conventional methods of spreading would only increase the risk of nitrate pollution, conversely the electrokinetic technique will provide the nitrogen at the long-term depending on the needs of the plants. The total porosity value found equal to 32%, expresses a slightly compacted soil, difficult to till which makes its fertilization difficult by conventional techniques. The negative value of the zeta potential is probably due to the ionization of surface groups clay in the presence of determinants ions of the potential (OH⁻). This is confirmed by the basic pH of the soil. Indeed this tight binding of the state of the surface charge of a clay material and its pH was often evidenced [25]. All spectroscopic analyzes in this characterization have an average error of about ± 2% and ± 6% for other analyzes. The results of the adsorption tests have shown that nitrate ions are not adsorbed on soil. The absence of nitrate adsorption on the clayey soil is often cited; it is attributed to the overall negative surface charge of the clay particle creating a repulsion to any anion which will bind to its surface. The origins of this surface charge drift the negative parts that come from substitutions Si⁴⁺ by Al³⁺ ions [15, 26].

Table 1: Soil characteristics.

Parameter	Value	Chemical composition	(%)
Real density (g/cm ³)	2.84	SiO ₂	55.82
Apparent density (g/cm ³)	1.93	Al ₂ O ₃	25.85
Porosity (%)	32	MgO	4.93
Humidity at 105 °C (%)	8.20	TiO ₂	0.97
pH _{Eau}	8.50	CaO	2.93
pH _{KCl}	6.73	Na ₂ O	0.92
BET specific surface area (m ² /g)	17	K ₂ O	1.90
Uniformity coefficient (U)	6.0	Iron total	5.67
Permeability coefficient (K)	0.72	C Carbon	0.27
Zeta potential (mV)	-6.5	N total nitrogen	0.09
Cation Exchange Capacity or CEC (meq / 100 g soil)	22.5	N – NO ₃ ⁻ (liquid soil)	0.004
Conductivity (μS / cm)	230.66	CaCO ₃	0.39

3.2. Electrokinetic transport of nitrate in the soil

3.2.1. Evolution of pH

The continuous addition of NaOH (pH_{initial} = 12) at the anode and KNO₃ (pH_{initial}=2.2) at the cathode during all test (Fig. 2) kept the soil pH in the central compartment at its average initial value and thus avoids the appearance of fronts acid (H⁺) and basic (OH⁻) directed towards the electrodes of opposite charge. This pH control would avoid the influence of this parameter on soil microflora. Generally the pH tolerated by the microorganisms is between 5 and 9 [14, 15].

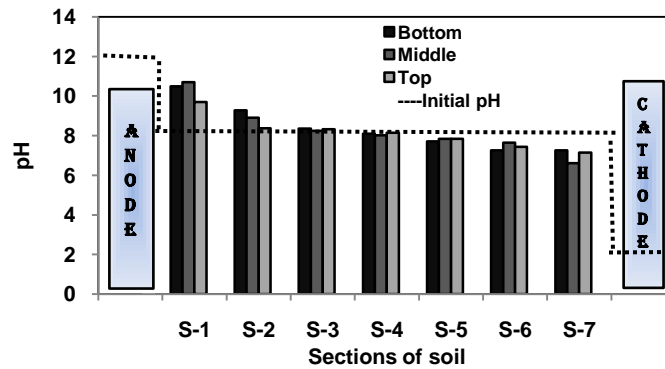


Figure 2: Soil pH at the end of the test.

3.2.2. Distribution of the voltage

Figure 3 shows that the voltage starts at 6 volts and then it increases slightly until 7 volts, after 22 hours, the values of voltage decreases to return at the end to about 6 Volts. The decrease in voltage, would result from the decrease in electrical resistance within the soil cell caused by dissolution of the salts initially associated to soil (Table 1) and to the increasing the concentration of the charged species. These species would come from migration of anions NO_3^- and the majority cations which would cross the soil toward the oppositely charged electrodes [9,21,27,28]. The voltage measured during the test would have negligible effects on soil microflora, because the voltage gradient used is less than 100 V.m^{-1} [14,16].

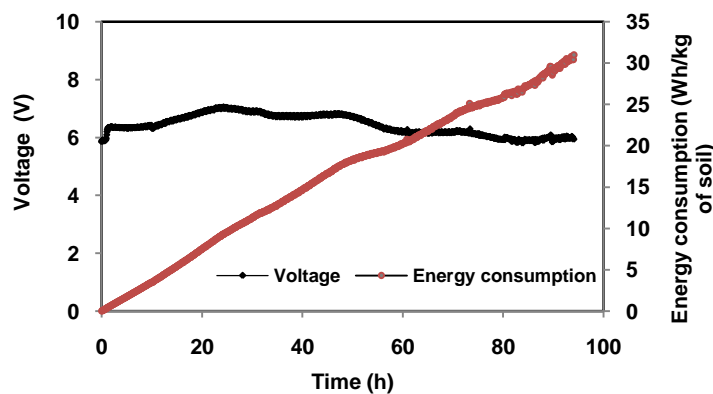


Figure 3: Voltage and energy consumption during the test

3.2.3. Energy consumption

The Energy consumption W is a very important factor for the application of electrokinetic treatment [29-31], It increases with increasing treatment time (Fig. 3).

$$W = \frac{\Phi I t}{m} \quad (3)$$

W : energy consumption (Wh.kg^{-1}); I : electrical current (A)
 Φ : Voltage (Volt); t : time (h); m : mass of soil (kg).

The results obtained from Equation 3 [30], show that at applied current of 2.54A, the energy consumption was 1535 Wh (32.9 Wh.kg^{-1}). This value is less than that measured in the test conducted in the laboratory under the same current density with an inter-electrode distance for the laboratory test $L_{\text{Laboratory}}$ equal to 11 cm [21].

According to Al Shawabkeh et al. [31] and Costarramone et al. [30] the inter-electrode distance L is a very important factor, Indeed the report of the inter-electrodes distances $\frac{L_{\text{Pilot}}}{L_{\text{Laboratory [21]}}} = \frac{7\text{cm}}{11\text{cm}} = 0.64$ approximately equals to the ratio of energy consumption $\frac{W_{\text{Pilot}}}{W_{\text{Laboratory [21]}}} = 0.69$, so $\frac{L_{\text{Pilot}}}{L_{\text{Laboratory [21]}}} \approx \frac{W_{\text{Pilot}}}{W_{\text{Laboratory [21]}}}$. Electrical energy can be supplied from renewables energies [11].

3.2.4. Evolution of electro-osmotic flow (EOF)

The electroosmotic flow (EOF) was collected at the cathode compartment, in accordance with the negative value of the zeta potential (Table 1). The electroosmotic flow rate is relatively higher at the beginning of the test, then from about the 6th hour he begins to decrease rapidly to attain 0.04 liters/hour. Increasing the ionic strength due to the introduction of ion into the interstitial fluid of the ground causes the compression of the thickness of the diffuse double layer, which results in a decrease in the electroosmotic flow [8,27,28]. This can be observed on the curve (Fig. 4) of concentrations of nitrate recovered in the anode compartment, where the characteristic time taken by the nitrate ions from the cathode compartment to cross the soil and go out again the anode side coincides with the time of the fall of electro-osmotic flow.

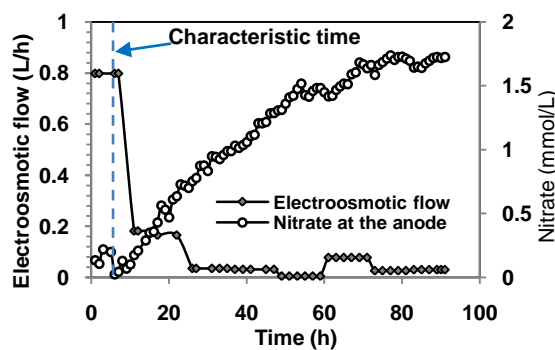


Figure 4: concentration of nitrate at the anode and electroosmotic flow (EOF) during the test.

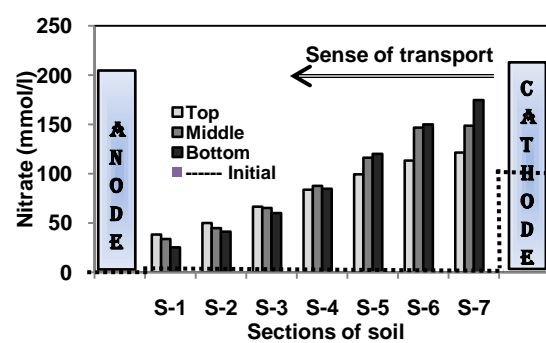


Figure 5: Profile of the nitrate ions in the soil at end of test.

3.2.5. Nitrate

Figure 5 shows the distribution of nitrate ions in the soil at the end of the pilot test which lasted 93 hours and under an electric current of 2.54A imposed. The progression of nitrate ions is fronting on cathode compartment toward the anode compartment in the different sections of soil (S_{P-1} to S_{P-7}) for the different tested samples ["P" (high (H), medium (M) and low (B))]. The nitrate concentration in the interstitial fluid of the soil is multiplied on average by ≈ 13 times in the more remote sections of the cathode (S_{B-1} , S_{M-1} et S_{H-1}) and by ≈ 60 times in sections adjacent to the cathode (S_{B-7} , S_{M-7} et S_{H-7}). The results of the material balance of nitrate ions expressed in mg of nitrate nitrogen (Table 2) show that of the initial concentration of the nitrogen fed continuously to the cathode (501 000 mg) for 93 hours, it is recovered 1280 mg N – NO_3^- to the anode or 0.3% and the equivalent of 50.5% nitrogen are present in the soil. A preliminary test carried out under similar experimental conditions, but in the absence of electric current gives no significant result of the presence of nitrogen in the soil. These results justify the transportation and injection of the nitrogen (as NO_3^-) into the ground by electromigration. In the case of anions NO_3^- , the electroosmotic flow (EOF) directed towards the cathode is opposed to the transport by electromigration. But this flow (FR) is very low (Fig. 4) in this case and according to Acar et al. [32] the electromigration is the most significant mechanism in the transport of present chemical species. In soil the Nitrogen forehead (N – NO_3^-) advances towards the anode. For example, in the sample "high" cut into seven sections (Table 2), the nitrogen content is 1155 mg N – $\text{NO}_3^- \cdot \text{kg}^{-1}$ and 320 mg N – $\text{NO}_3^- \cdot \text{kg}^{-1}$ into the sections S_{H-7} and S_{H-1} respectively, the difference between these two sections is 835 mg N – $\text{NO}_3^- \cdot \text{kg}^{-1}$ (a deviation of 72% compared to S_{H-7}). In the sample "down" the content is 1428 mg N – $\text{NO}_3^- \cdot \text{kg}^{-1}$ into the section S_{B-7} and 208 mg N – $\text{NO}_3^- \cdot \text{kg}^{-1}$ into the section S_{B-1} , a difference between these two sections is of 1220 mg N – NO_3^- (a difference of 85% compared to S_{B-7}) on ground that corresponds to a variation from 550 to 80 kg N.ha⁻¹. This divergence was also observed at the laboratory scale [21]. The second irregularity into the nitrogen content in

the soil cannot be observed only at pilot scale (Table 2, Fig. 5), in the various samples taken "high", "medium" and "low" concentrations are not always similar, example: in the same section S_{B-7} , S_{M-7} et S_{H-7} nitrogen contents are respectively 1428, 1255 et 1155 mg N – NO₃⁻.kg⁻¹. The difference is less important because it appears only in sections $S_{.6}$ and $S_{.7}$ of soil. According to Zhou et al. [29] and Kim et al. [33] the cause may be linked at the liquid of soil, which may concentrate more on the bottom than at the top due to the gravity and evaporation, entailing with him the ions not retained by the soil such as NO₃⁻. According to Basso et al [2] substantial variability in the content of nitrogen applied in a soil might influence yield, but with less effect on the quality of cereal crops. This would apply probably in the first case of discrepancies observed in this study where the deviations between the sections $S_{.1}$ and $S_{.7}$ may be up to 85%. For the second divergence studied the phenomenon is less important, it would have less effect.

The report of the inter-electrode distance (L) $\frac{L_{Pilot}}{L_{Laboratory} [21]} = \frac{7cm}{11cm} = 0.64$ is approximately equals to the reports between the nitrogen concentrations in the laboratory test [21] $[N - NO_3^-]_{S-i}^{Laboratory}$ ($i=1 \text{ à } 7$) and those of the pilot test $[N - NO_3^-]_{SP-i}^{Pilot}$ (or P = H, M, B et $i = 1 \text{ à } 7$).

Example:

$$\frac{[N-NO_3^-]_{S-1}^{Laboratory}}{[N-NO_3^-]_{SP-1}^{Pilot}} = 0.60 ; \quad \frac{[N-NO_3^-]_{S-4}^{Laboratory}}{[N-NO_3^-]_{SP-4}^{Pilot}} = 0.40 ; \quad \frac{[N-NO_3^-]_{S-5}^{Laboratory}}{[N-NO_3^-]_{SP-5}^{Pilot}} = 0.46 ; \quad \frac{[N-NO_3^-]_{S-6}^{Laboratory}}{[N-NO_3^-]_{SP-6}^{Pilot}} = 0.57 ;$$

$$\frac{[N-NO_3^-]_{S-7}^{Laboratory}}{[N-NO_3^-]_{SP-7}^{Pilot}} = 0.66 ; \text{ for } \frac{[N-NO_3^-]_{S-2}^{Laboratory}}{[N-NO_3^-]_{SP-2}^{Pilot}} \text{ and } \frac{[N-NO_3^-]_{S-3}^{Laboratory}}{[N-NO_3^-]_{SP-3}^{Pilot}} = 0.25.$$

In this case, there would be a relationship between the tests results from pilot and laboratory $\frac{L_{Pilot}}{L_{Laboratory} [21]} \approx$

$$\frac{[N-NO_3^-]_{S-i}^{Laboratory}}{[N-NO_3^-]_{SP-i}^{Pilot}}.$$

Table 2: Mass balance for nitrogen (N – NO₃⁻).

Nitrogen introduced to the cathode		501×10 ³ mg	100 %	
Nitrogen injected into the soil		253×10 ³ mg	50.5 %	
Nitrogen recovered at the cathode		246×10 ³ mg	49.2 %	
Nitrogen recovered at the anode		1280 mg	0.3 %	
Samples P		Top (P=T)	Middle (P=M)	Bottom (P=B)
Nitrogen injected into the soil (mg N – NO ₃ ⁻ .kg ⁻¹) P = H, M, B et $i = 1 \text{ à } 7$	Section S_{P-1}	320	286	208
	Section S_{P-2}	432	378	342
	Section S_{P-3}	617	550	526
	Section S_{P-4}	794	739	714
	Section S_{P-5}	969	980	994
	Section S_{P-6}	1116	1237	1241
	Section S_{P-7}	1155	1255	1428

Conclusion

The use of the electrokinetic technique a fertilization medium an agricultural natural with nitrate was realized in a pilot scale. The results obtained demonstrate the injection and moving of anions NO₃⁻ through the soil. The absence of hydraulic convection and the appearance of a very low electro-osmotic flow implies that electromigration is the dominant mode of transport. Nitrate ions were displaced into the ground in decreasing front from the cathode to the anode, creating a difference in the nitrogen contents which varies from 1428 to 208 mg N – NO₃⁻.kg⁻¹ soil, on ground that corresponds to a variation from 550 to 80 kg N.ha⁻¹, this difference could possibly have an influence on the Cereal crop yield. The control of pH in the anode and cathode compartments allowed the introduction of nitrate ions without perturbs the soil pH. The pilot test has consumed

electrical energy of 32.9 Wh.kg^{-1} . The report of the inter-electrode distances: $\frac{L_{\text{Pilot}}}{L_{\text{Laboratory}}}$ can help predict the results obtained in this test. Further studies are needed for application on ground where certain operating conditions must be reviewed. The use of renewable energy to provide electricity for the process will be considered.

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