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SIMULTANEOUS EXTRACTION AND POTENTIOMETRIC DETERMINATION OF POTASSIUM AND NITRATE IN SOIL BY USING ION SELECTIVE ELECTRODES

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Abstract. Rapid and simultaneous determination of nitrate (NO_3^-) and potassium (K^+) ions in soil samples using ion selective electrodes (ISE) was investigated. The compatibility of ISEs with soil extraction solution is a challenging subject as various effects such as pH, ionic strength and interferences have to be considered as well as efficiency of the extraction solution. In this study, 30 agricultural soil samples of different properties were extracted with 0.01 mole.L-1 $Al_2(SO_4)_3$ and the extracts were simultaneously measured by NO_3^- - ISE and K^+ - ISE in a flow system. The results were compared with ion chromatography (IC) as the reference method, and the regression analysis between IC and ISE results was yielded high correlation ($R^2 = 0.986$ and $R^2 = 0.951$ for K^+ and NO_3^-, respectively). It was concluded that, ion selective electrodes can be used with 0.01 mole.L-1 $Al_2(SO_4)_3$ extraction solution for rapid and simultaneous determination of nitrate and potassium in soil samples.

Keywords: ion selective electrodes (ISE), soil analysis, nitrate, potassium.

INTRODUCTION

Necessary information about soil physical conditions, nutrient status, and chemical properties can only be obtained by performing appropriate soil analysis. Since nutrient levels in soils are the main criteria for fertilizer recommendations, soil analysis are important in terms of minimizing the environmental impact of fertilization practices by enabling efficient fertilizer usage. However, conventional soil testing methods which comprise of soil collection from the field and soil chemical analysis in the laboratory are costly and time consuming processes, such that numbers of samples that can be analyzed are limited and monitoring of spatial variability in soil nutrient concentrations is difficult. Drawbacks in conventional soil analysis methods have led to the investigation of fast in-field monitoring systems, which ion-selective electrode (ISE) technology is prominent. Ion selective electrodes are accurate, fast, economic and sensitive methods. They are more economic than conventional test methods such as AAS, ICP-OES, and UV/VIS. Among many advantages of ISE technology, portability is especially striking as it can enable fast and in-situ soil analysis. ISEs are compatible with on-line analysis, and many of them are commercially available for use in various matrixes and significant progress has been made in ISE technology in recent years by development of various ion-selective membranes including NO_3^-, K^+, Ca_2^+, and Na^+ which are the most important soil nutrients, and also many researches have been carried out to develop phosphate ion-selective electrodes (Tsukada et al., 1989; Carey and Riggan, 1994; Knoll et al., 1994; Xiao et al., 1995; Chen et al., 1997; Levitchev et al., 1998; Wroblewski et al., 2000; Bratovcic et al, 2009).

The selection of an extractant suitable with ISEs is often a problem in soil analysis. The extraction solution should be compatible with ISEs while extracting the target ions from soil samples efficiently. There are various extraction solutions available in literature for use in soil analysis and some of them were studied with ISEs and successful results were obtained (Wang and Scott, 2001; Wang et al, 2004; Zbiral and Nemec, 2005; Ciesla et al, 2007; Madurapperuma et al, 2008; Matula, 2009; Bortolon and Gianello, 2010; Kahveci and Atalay, 2010; Bortolon et al., 2011). However, the conventional extraction solutions such as KCl, NH_3OAc, Mehlich, $NaHCO_3$ are not suitable for use with ISEs because of their high ionic strength, high acidity or alkali. On the other hand, previous studies have shown that $Al_2(SO_4)_3$ solution can be used as a soil extractant and it can successfully work with ion selective electrodes (Poonia et al.,1974; Roth et al, 1991; Plank, 1992; Taber et al, 1993; Hartz, 1994; Smith, 1975; Kalra,1998; Baranowski et al., 2002). Efficiency of extraction depends on many process parameters and artificial neural network systems are widely used to determine optimum process conditions by mapping complex nonlinear interactions between inputs and outputs (Aitkenhead et al, 2012, Zarifneshat et al, 2012, Soares et al, 2013, Dursuna and Özden, 2014). A simple multilayer neural network model consists of an input layer, a hidden layer and an output layer. If radial basis functions are employed in the hidden layer, it is called Radial Basis Function Neural Network (RBFNN) model. RBFNN models can be used for function approximation, regularization and classification (Bishop, 1995).

The aim of this study is, first, to determine the optimum conditions for simultaneous nitrate (NO_3^-) and potassium (K^+) extraction from soil samples by using 0.01 mole.L-1 $Al_2(SO_4)_3$ solution and second, to investigate NO_3^- and K+ measurement ability of ISEs by comparing the results with ion chromatography (IC) analysis.

MATERIAL AND METHODS

Flow system

Tests of the NO_3^- and K^+ electrodes were conducted with a flow system (figure 1) that contains commercially available all solid state NO_3- and K+ ion selective electrodes and a data analyzer. It has a measuring cell into which the electrodes were inserted, containers to hold solutions (1 and 2), and a control unit that allows simultaneous collection of voltage data from the two test electrodes (K^+ and NO_3^-) measured relative to the reference electrode (R). Each test began with the flow of 0.01 mole.L-1 $Al_2(SO_4)_3$ baseline solution. The desired volume of test solution was fed to the flow line by using 3-way mini valve as shown on the scheme (figure 1) and measurements consistently continued under computer software control. As the tested solution sensed by the detector, which consists of NO_3^--ISE and K^+-ISE in this case, peaks with different heights were observed according to the ion concentrations of the solutions. Each individual test solution was introduced to the flow line when the baseline equilibrium had maintained. The electrodes were first calibrated with solutions that were prepared by dissolving 2.528 g of KNO_3 in 0.01 mole.L-1 $Al_2(SO_4)_3$ solution and diluting it several times until the final concentrations of 1.25x10-3, 2.50x10-4, 5.00x10-5 and 1.00x10-5 mole.L-1 were obtained.

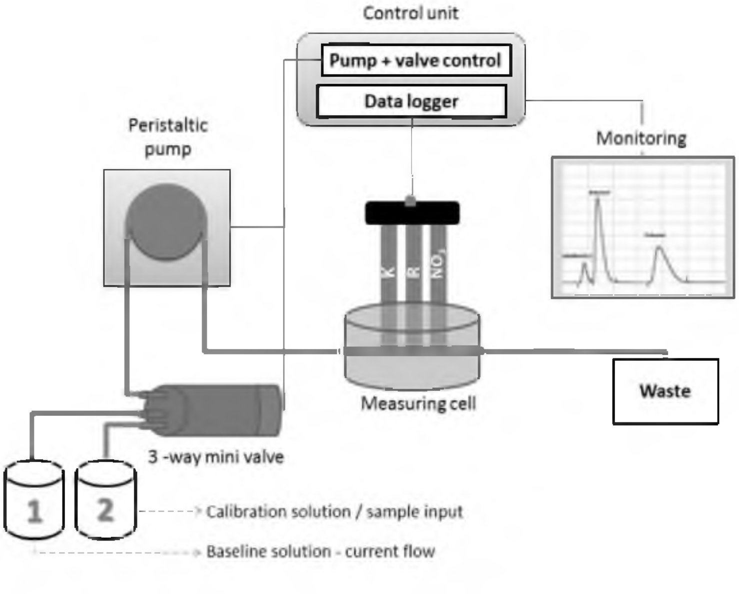


Figure 1 - The scheme of the flow system that uses NO_3^- and K^+ ion selective electrodes as detector

Optimization of extraction parameters

0.01 mole. L-1 $Al_2(SO_4)_3$ extractant, which is compatible with ion selective electrodes, was used to extract NO_3^- and K^+ ions from soil simultaneously. The parameters that effect the extraction efficiency were determined as soil: extractant ratio, shaking time and shaking speed. The soil sample which was chosen for optimization experiments had a sandy loam texture (% 63.03 sand, % 21.14 silt, % 15.83 clay) and its pH and EC were 5.80 and 0,13 dS/m (1:1 w/v). An experimental design with 3 parameters, 5 levels, 6 repeats, 2 answers (NO_3^- and K^+) was set due to the central composite design (CCD) model (tables 1 and 2).

Table - Coded values of central composite design (CCD) (Liu et al, 2006)

Parameters	**Coded Parameters**	**Coded Levels**				
		-2	-1	0	1	2
Soil: Extractant ratio (w/v)	x_1	3	4	5	6	7
Shaking time (min)	x_2	5	7.5	10	12.5	15
Shaking speed (rpm)	x_3	140	170	200	230	260

Table - Central composite design (CCD) (Liu et al, 2006)

Experiment No	Coded Parameters			Actual Parameters		
	x_1	x_2	x_3	Soil: Extractant ratio (w/v), x_1	Shaking time (min), x_2	Shaking speed (rpm), x_3
1	-1	-1	-1	4	7.5	170
2	-1	1	1	4	12.5	230
3	1	-1	1	6	7.5	230
4	1	1	-1	6	12.5	170
5	-1	-1	1	4	7.5	230
6	-1	1	-1	4	12.5	170
7	1	-1	-1	6	7.5	170
8	1	1	1	6	12.5	230
9	-2	0	0	3	10.0	200
10	2	0	0	7	10.0	200
11	0	-2	0	5	5.0	200
12	0	2	0	5	15.0	200
13	0	0	-2	5	10.0	140
14	0	0	2	5	10.0	260
15	0	0	0	5	10.0	200
16	0	0	0	5	10.0	200
17	0	0	0	5	10.0	200
18	0	0	0	5	10.0	200
19	0	0	0	5	10.0	200
20	0	0	0	5	10.0	200

Each experiment was carried out three times and the mean values of the results were used in training of RBFNN models. Every set of training data was composed of three parameters (soil:extractant ratio, shaking speed and shaking time) as input vector and their respective ISE response as output. Each extraction parameter was scaled between -1 and 1 to improve training efficiency; likewise ISE responses were normalized between 0 and 1. Since ISE response is proportional to extracted element concentration from soil, optimization of extraction process is equivalent to optimization of ISE response. A symmetrical Gaussian distribution function was used as radial basis in RBFNN models. Spread constant for Gaussian distribution function was determined by testing different spread constant values between 1 and 5. Error is calculated by taking mean square of difference of actual extraction rate and predicted extraction rate by RBFNN model. Leave-one-out cross validation method is used for optimization of neural network structure.

Extraction

All reagents were prepared by using deionized water purified with a Milli Q system (Millipore Direct Q-UV3). Chemicals were purchased commercially from Merck (Merck KGaG, Darmstadt, Germany). Soil samples were extracted according to the procedures described in table 2 in an orbital shaker (Stuart SS11), 0.01 M $Al_2(SO_4)_3$ extractant.

Comparison of ISE with IC

The new extraction method needed to be tested with a wide variety of soils to represent the extreme features of agricultural soils in Turkey. For this purpose, 30 soil samples were provided from Soil, Fertilizer and Water Resources Central Research Institute in Ankara. The properties of 30 soil samples are given in table 3.

Table 3 - Chemical and physical characteristics of 30 soil samples used this study

ID	pH	EC , dS/m	Textural class	ID	pH	EC , dS/m	Textural class	ID	pH	EC , dS/m	Textural class
1	8.25	0.43	Heavy Clay	11	8.55	0.29	Sandy	21	7.71	0.49	Loamy
2	8.02	0.45	Clay	12	7.85	0.58	Loamy	22	7.88	0.41	Loamy
3	7.98	0.47	Silty Clay	13	8.22	0.94	Loamy	23	6.78	0.36	Loamy
4	7.87	0.39	Silty Clay Loam	14	8.17	0.38	Clay Loam	24	7.70	0.33	Clay
5	7.82	0.64	Clay Loam	15	8.05	0.44	-	25	4.82	0.08	Sandy
6	7.85	0.86	Loamy	16	8.10	0.51	-	26	6.41	0.40	Clay Loam
7	8.01	0.55	Sandy Clay	17	7.83	0.62	Loamy	27	7.32	2.61	Heavy Clay
8	7.96	0.42	Sandy Clay Loam	18	8.00	0.51	Loamy	28	7.72	0.41	Loamy
9	7.9	0.62	Sandy Loam	19	7.75	0.67	-	29	8.03	0.28	Loamy
10	7.33	0.34	Loamy Sand	20	7.6	0.62	Sandy	30	8.14	0.42	Loamy

NOTE: pH and EC measurements; 1:1 w/v, 25 ^{0}C.

The soil samples were extracted with 0.01 mole.L-1 $Al_2(SO_4)_3$ solution at optimum conditions, which were previously determined by CCD and RBFNN model approach as 5.1 soil:extractant ratio (w/v), mixing at 245.6 rpm for 10.5 minutes in orbital shaker. The extracted samples were filtered with Whatman No. 42 filter paper. Every extract was analyzed with NO_3^--ISE and K^+-ISE in the flow system, and with ion chromatography (IC) as a reference method. The IC analyses were held in Soil Water Laboratory in Atatürk Soil, Water and Agricultural Meteorology Research Station Manager.

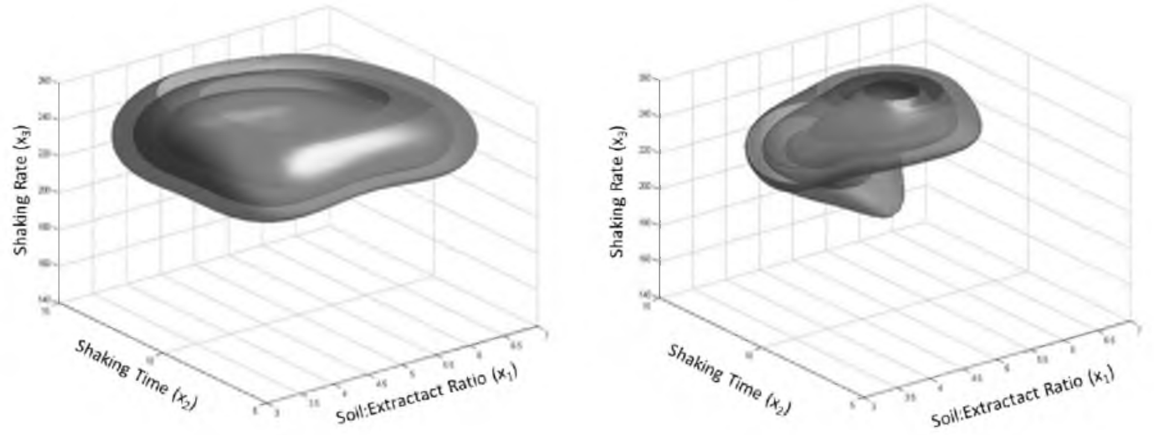
Analytical grade (Merck) chemicals without any further purification were used in all experiments. Aqueous solutions were prepared with deionized water (Millipore Direct Q-UV3). Soil samples were extracted in Stuart SS11 orbital shaker.

RESULTS AND DISCUSSION

Optimization of extraction parameters

Optimum conditions for the extraction process were deduced to get maximum simultaneous extraction of NO_3^- and K^+. Two different RBFNN models were constructed with different spread constants and fitted with the experimental results of CCD. For NO_3^-, a RBFNN model with 1.3 spread constant provided lowest relative mean squared error. Similarly for K^+ prediction, a RBFNN model with 1.9 spread constant value was used. For each of 3 extraction parameters, uniformly distributed 40 data points were chosen between the lowest and highest values in CCD. Thus 403 input vectors were formed. Corresponding ISE response was predicted for each input vector by utilizing trained RBFNN model. Since there were 3 independent variables (extraction parameters) and one dependent variable (ISE response), a 3D isosurface plot was appropriate for visualization of results. In figure 2a and 2b, three axes of plot denote extraction parameters. Surface was specified by connecting input vectors that have a specified isovalue (here, ISE response). Isosurface plot is similar to contour plot where contours are specified by elevation value instead of isovalue. In figure 2a and 2b, isosurfaces with ISE responses 0.7, 80 % and 90 % are given. As ISE response increases, volume occupied by isosurface shrinks towards to the point where maximum extraction ratio is achieved.

In order to determine the optimum extraction conditions, predictions of two RBFNN models for each element (K^+ and NO_3^-) was summed and over this total ISE response, optimum conditions for extraction were determined as 5.1 soil:extractant ratio (w/v), 10.5 minutes shaking at 245.6 rpm in orbital shaker.



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(b)

Figure 2 - Isosurface plots corresponding to isovalues of 0.7, 0.8, 0.9 normalized ISE response for soils extracted with 0.01 mole.L-1 $Al_2(SO_4)_3$, (a) NO_3^- and (b) K^+

Comparison of ISE with IC

The goal of this research was to develop a NO_3^- and K^+ ions measuring method based on ion selective electrodes (ISE), so the newly developed soil extraction procedure was applied to the sensor system and the results were compared with those obtained from IC. Regression analyses were applied to the results of ISE and IC measurements and high correlation of $R^2 = 0.986$ for K^+ and $R^2 = 0.951$ for NO_3^- were obtained (figure 3). The NO_3^- and K^+ ISEs used in conjunction with 0.01 mole.L-1 $Al_2(SO_4)_3$ extractant provided results in close agreement with IC analysis.

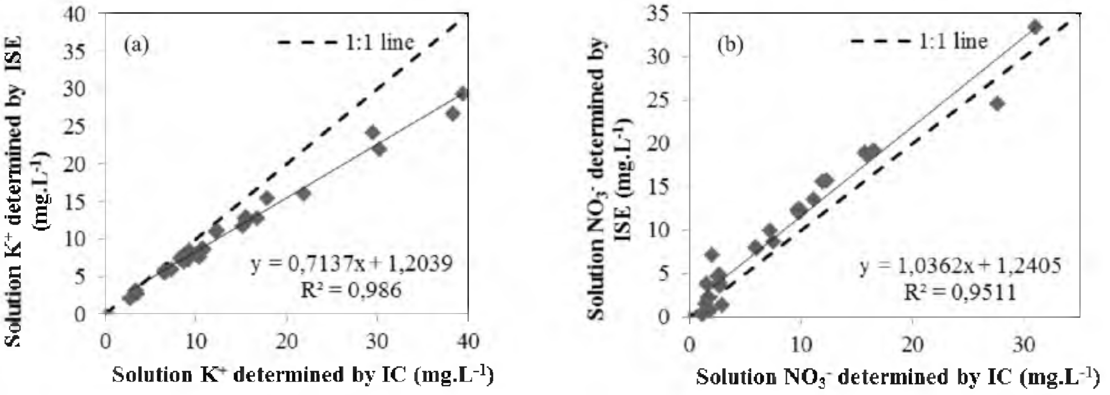

Figure 3 - Relationship between soil extract K^+ and NO_3^- determined by ISE and IC, (a) K^+ - ISE vs. IC, (b) NO_3^- - ISE vs. IC

CONCLUSION

In this study, a flow system with two different ISEs was evaluated for the simultaneous determination of NO_3^- and K^+ ions in soil. 0.01 mole.L-1 $Al_2(SO_4)_3$ was chosen as soil extractant and the ability to extract NO_3^- and K^+ was investigated. A new extraction procedure was determined by optimizing the extraction conditions with RBFNN model approach using central composite design (CCD) data. The newly developed soil extraction procedure was tested with 30 different agricultural soils from Turkey. The relationships between the concentrations of the soils obtained with ISEs and with IC were investigated. The NO_3^- -ISE results showed high correlation ($R^2 = 0.951$) and almost 1:1 relationship with IC readings. The K^+-ISE results also yielded high correlation with IC readings ($R^2 = 0.986$); however they were not as close to 1:1 line as NO_3^- - ISE. This may be caused by the potential drift in K^+ -ISE. The results show that the use of ion selective electrode technology for simultaneous NO_3^- and K^+ measurement can be performed with the developed flow system in conjunction with 0.01 mole.L-1 $Al_2(SO_4)_3$ extractant, at least for 30 soils included in this study. Additional researches and tests with a broader range and number of soils are needed to further validate the applicability of these results.

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РЕЗЮМЕ

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ОДНОВРЕМЕННОЕ ИЗВЛЕЧЕНИЕ И ПОТЕНЦИОМЕТРИЧЕСКОЕ ОПРЕДЕЛЕНИЕ КАЛИЯ И НИТРАТОВ В ПОЧВЕ С ПОМОЩЬЮ ИОНОСЕЛЕКТИВНЫХ ЭЛЕКТРОДОВ

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Было исследовано быстрое и одновременное определение нитратов (NO_3^-) и ионов калия (K^+) в почвенных образцах с использованием ионоселективных электродов (ИСЭ). Совместимость ИСЭ с раствором экстракции почвы является сложным объектом, так как нужно рассматривать различные эффекты, такие как pH, сила иона и помехи, а также эффективность экстракционного раствора. В данном исследовании, были взяты 30 образцов почвы сельскохозяйственного предназначения с различными свойствами с 0,01 моль. L-1 $Al_2(SO_4)_3$ и экстракты одновременно измеряли при помощи NO_3^- - ISE и K^+ - ISE в проточной системе. Результаты были сопоставлены с помощью ионной хроматографии (IC) в качестве эталонного метода, а регрессионный анализ результатов ИС и ISE показал высокую корреляцию ($R^2 = 0{,}986$ и $R^2 = 0{,}951$ для K^+ и NO_3^-, соответственно). Был сделан вывод, что, ионоселективные электроды могут быть использованы с экстракционным раствором 0.01 mole. L-1 $Al_2(SO_4)_3$ моль для быстрого и одновременного определения нитратов и калия в образцах почвы.

Ключевые слова: ионоселективные электроды (ИСЭ), анализ почвы, нитрат, калий.

ТҮЙІН

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ИОНДЫҚ ІРІКТЕМЕЛІ ЭЛЕКТРОДТАРДЫҢ КӨМЕГІМЕН ТОПЫРАҚТАҒЫ КАЛИЙ МЕН НИТРАТТАРДЫ ҚАТАР АЛУ ЖӘНЕ ПОТЕНЦИОМЕТРЛІК АНЫҚТАУ

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Иондық іріктемелі электродтарды (ИІЭ) пайдалана отырып топырақ үлгілерінен нитраттар (NO_3^-) мен калий иондарын (K^+) тез және қатар анықтауға зерттеу жүргізілді. ИІЭ-ніц топырақ экстракциясы ерітіндісімен үйлесімділігі күрделі объект болып табылады, өйткені pH, ион күші мен кедергілер секілді түрлі әсерлерді, сондай-ақ экстрациялық ерітінді тиімділігін қарастыру қажет. Бұл зерттеуде 0,01 мольден L-1$Al_2(SO_4)_3$ бастап түрлі қасиеттері бар ауылшаруашылыққа арналған топырақтыц 30 үлгісі алынды, және сығындылар бір мезетте NO_3^- - ISE и K^+ - ISE көмегімен ағынды жүйеде өлшенді.

Нәтижелер эталондық әдіс ретінде ионды хромотография (IC) көмегімен салыстырылды, ИС и ISE нәтижелерінің арегрессиялық талдауы жоғары өзара байланыстылықты көрсетті ($R^2 = 0,986$ и $R^2 = 0,951$ K^+ және NO_3^-). Иондық іріктемелі электродтар топырақ үлгілеріндегі нитраттар мен калиді тез және бір мезетте анықтау үшін 0.01 mole.L-1$Al_2(SO_4)_3$ моль экстракциялық ерітіндімен пайдалануға болады деген қорытынды жасалды.

Кілтті сөздер: иондық іріктемелі электродтар (ИІЭ), топырақ талдауы, нитрат, калий