Evaluation of Soil Potassium Using Miniaturized Electrochemical Sensors

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ABSTRACT

Farmers aim to increase the yield of their crops by reducing their expenses. This prompts farmers to resort to smart farming practices. These practices facilitate the measurement and detection of nutrients in soil to maximize harvest with a low-cost sensor employing electrochemical and optical techniques. Potassium is a crucial macronutrient for the growth of plants. The excess or need for potassium in the soil can pose a risk to plants and the environment. Therefore, it is imperative to conduct soil monitoring to determine the quantity of soil fertilizer. ISFETs are among the most affordable and miniaturized sensors and are specific to one target ion. Although it is not time-consuming and requires an immediate response, the ISFET requires special calibration and sample preparation. This study tested ISFET on Moroccan soil potassium using a new technique of minimal sample preparation without needing a laboratory machine to extract soil using distilled water. The results indicate that the microsensor ISFET provided accurate results on solutions prepared in the laboratory with a correlation factor of 90%, and R^2 greater than 80% when estimating potassium levels in soil extracts. ISFET can therefore be used as an alternative method for potassium estimation.

Keywords: ISFET, potassium, soil analysis, extraction method, precision agriculture, smart farming.

INTRODUCTION

Potassium is an indispensable nutrient for the development of plants. It is essential for the exchange of water, oxygen, and carbon dioxide in plants. Potassium improves drought resistance, promotes root growth, mitigates wilting from water evaporation, and is needed for photosynthesis and disease resistance promotion (Kaiser, 2018). In plants, potassium deficiency is recognized by its visible appearance. The plant leaves look brown and scorch on the edges. Furthermore, chlorosis, or yellowing, is observed between leaf veins. On the other hand, excess potassium is not visible in plants but is harmful to plants. It can run off into streams, causing algal blooms that can harm aquatic life (Prajapati, 2012). To ensure the effective management of potassium in the soil, farmers regularly conduct soil testing to assess nutrient levels and make informed decisions regarding fertilizer application. By monitoring and adjusting potassium levels, farmers can optimize crop nutrition, maximize yield potential, and sustain long-term soil health. Potassium can be found in soil in different forms; some are available for plants, while others are not (Lalitha et al., 2014). It is present in the soil in four forms as shown in Figure 1: (1) soluble potassium (available), which represents 0.1% of the total potassium in soil and is readily absorbed by plants. It is measured mainly in a water extract of soil. This form does not create any ion pairs, chelates, or complexes in the soil solution. Its concentration in soil solution is minimal, approximately 150 μM,

equivalent to 6 mg/L, which can decrease in humid soils. (2) The exchangeable form, which represents 0.1% to 2% of the total potassium in the soil. K is considered the major bioavailable form of K in the soil and typically represents 1 to 3% of the total cation exchange capacity of soils. (3) Fixed potassium, which represents 1% to 10% of the total potassium of soil, is a form with high levels of soluble and exchangeable K, where potassium is fixed by certain minerals from which it is released very slowly to the available form. (4) The unavailable mineral forms found at potassium-rich parent rock, which are 90 to 98% of the total potassium in the soil, are locked in soil minerals in different mineral forms, such as micas or illite, where potassium is liberated by plants after the destruction of minerals (Lalitha et al., 2014).

Available potassium in the soil can be quantified by different methods, but most of them are expensive, are time consuming, have large dimensions, and require laboratory preparations such as atomic absorption spectrometry, electrochemical methods, chromatographic techniques, flow injection analysis, and colorimetric methods (Han et al., 2022). Over the past few decades, researchers have targeted new simple methods based on precision agriculture (PA), specifically smart farming, where farmers can maximize yields with minimal use of resources such as water, fertilizers, and seeds using specific sensors

(Bouhachlaf et al., 2023). Hence, farmers are beginning to understand their crops on a microscopic scale, conserve resources, and reduce environmental impacts quickly and efficiently (Ali et al., 2020). In this context, electrochemical soil sensors are one of the factors in managing smart farming. Among these sensors, the ion-selective field effect transistor (ISFET) (Ahmed et al., 2021) is a specific kind of MOSFET (metal-oxide-semiconductor field-effect transistor) (Benslimane, et al., 2023), as shown in Figure 2, based on changing the electrode metal of the gate by a selective membrane (8) placed on a thin layer of gate oxide (5) composed of inorganic materials such as Ta_2O_5 to improve sensitivity when in contact with the insulating resin (7), and the reference electrode can be used as the gate of the FET (6) (Bermejo, 2013). The ISFET does not require a sophisticated laboratory; it ensures simple, specific, and inexpensive soil monitoring (Burton et al., 2020). In the 1980s, researchers conducted experiments to test the combination of the ISFET with flow injection analysis (FIA) for the purpose of detecting potassium ions. This particular combination proved to be successful. The resulting curves for potassium detection exhibited a high coefficient of determination (R^2) of 99.96% and a slope of 54 mV/pK. The researchers employed a carrier solution consisting of 25 mM Tris buffer in 0.14 M NaCl (Bermejo, 2013).
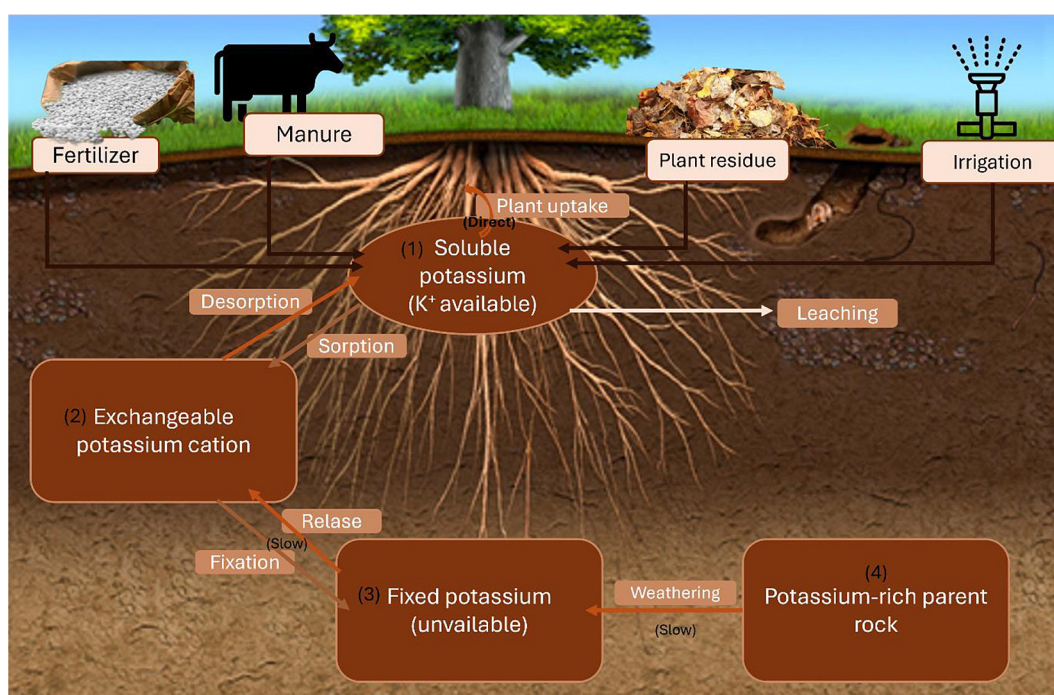

Figure 1. Different potassium forms in the cycle of potassium

1: Si Substrate
2: Channel
3: Source
4: Drain
5: Gate oxide
6: Metal electrode gate/ Reference
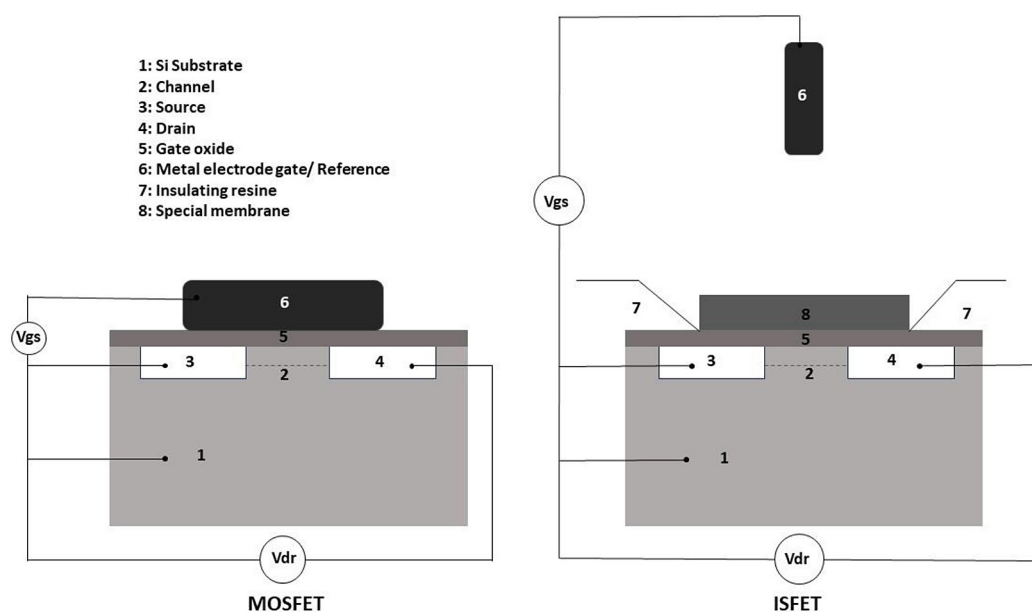7: Insulating resine
8: Special membrane

Figure 2. Differences between MOSFET and ISFET structures

After that, many studies began to use the ISFET for soil nutrient detection, some of which tested potassium in soil solutions using a valinomycin membrane with different plasticizers in four extractant solutions: DI water, Bray P1, Kelowna, and Mehlich III. In DI water, a linear Nernstian response with a slope ranging from 54.6 to 58.2 mV/Dec is obtained at concentrations ranging from 10^{-5} to 10^{-1} M; below these concentrations, the slope decreases, but the concentration still decreases, and the detection limit might be under 10^{-6} M. In contrast to Kelowna and Bray P1, the slope decreases to 46–52 mV/Dec and 41–42 mV/Dec, respectively, for potassium detection at concentrations ranging from 10^{-4} to 10^{-1} M, where $1{,}7.10^{-4}$ and $2{,}6.10^{-4}$ M are the detection limits, respectively. For Mehlich III, the slope is considerably reduced to 20–25 mV/Dec in the range of detection from 10^{-3} to 10^{-1} M, with 10^{-1} M as the limit of detection (Kim et al., 2006). J. Artigas et al. (2001) used potassium ISFETs in soil directly and worked with photocurable membranes. They found that this membrane is more adhesive to the surface than the polyvinyl chloride (PVC) membrane; it also has a long lifetime period of approximately seven months, and its signal in the soil is relatively stable. Before the in-soil test, they tested the system in aqueous solutions of potassium and obtained a sensitivity of 55.7 mV/Dec in the range of 7.10^{-5} to 10^{-1} M with 4.10^{-5} M as the limit of detection. In the soil, the sensitivity decreased to 32 mV/Dec in

the first month, which was considerably lower in the second month. Compared with the standard methods, the in-soil results did not exhibit good accuracy but exhibited a high correlation (Artigas et al., 2001). Another study used the same ISFET type in this work: an ISFET with a valinomycin membrane and a PVC polymer. They worked on three types of Indian soil, with an extraction of 20% from the soil by 1 N neutral ammonium acetate and used a flame photometer as the conventional method. The sensitivity of the calibration was approximately 60 mV/dec in the range of 7.10^{-5} to 10^{-1} M, and the R^2 of potassium in the soil extract was 63% (Bhagat et al., 2020). Potassium ISFETs can also be used in horticulture, as mentioned in this study (Gieling et al., 2005), or as hydroponic substrates (Riedel et al., 2024).

This study aimed to achieve three primary objectives: (i) to analyze potassium (K^+) levels in Moroccan soils utilizing an ion-sensitive field effect transistor (ISFET) with a selective polyvinyl chloride membrane; (ii) to evaluate the efficacy of different soil-to-water ratios as extraction methods; and (iii) to propose a novel and straightforward extraction technique. The investigation involved the examination of soils obtained from diverse regions in Morocco, with the obtained results being compared to those obtained using the standard automatic continuous flow analyzer method (SKALAR) as the conventional reference method.

MATERIALS AND METHODS

K-ISFET

The potassium in this study was analyzed by K-ISFET, as shown in Figure 3, procured from Microsens-SA, a Swiss company specializing in miniature semiconductor sensors and systems. The materials were tested at the Digital & Smart Microelectronic Devices Laboratory in MAScIR (Moroccan Foundation for Advanced Science, Innovation, and Research). The USB amplifier powers this K-ISFET with a drain voltage of 0.5 V and a drain current fixed at 0.1 μA. The data were connected to a graphical interface used to display the results as chronopotentiometric graphs on a laptop (Benslimane et al., 2023). The potassium concentration in an aqueous solution was determined by a polyvinyl chloride (PVC) membrane ship, as demonstrated in Figure 3, based on valinomycin, Tretrakis (4-chlorophenyl) borate as an ionophore, bis(2-ethylhexyl) sebacate (DOS) as a plasticizer, and cyclohexanone as a solvent, using an integrated Ag/AgCl reference electrode as shown in Figure 3.

Soil sampling and extraction method

Soil samples with different textures were collected from different regions of the country (Northern, Eastern, and Southern Morocco) (Reda et al., 2021). Each of the ten collected samples was air-dried, sieved through a 2 mm sieve, and stored in plastic bags (ISO 11464, 2006). Soil extracts were made from each sample. Three soil–water extraction methods (standard and two simple extraction methods) were tested on some soils, and three soil–water ratios (1:5, 1:2.5 and 1:1) were tested on others. Standard extraction at a ratio of 1:5 was performed by mixing 10 g in 50 ml of distilled water, mixing 1H30 in a shaker, settling overnight, and filtering through a Whatman #42 filter (Allison et al., 2006). The 1:2.5 and 1:1 ratio were made by the same extraction steps but with the soil quantity changed to 20 g and 50 g, respectively. Both simple extractions are hand extractions with no need for large laboratory machines, respecting the same standard extraction steps. The first simple extraction was performed by liquefying 8 g of soil in 40 ml of distilled water, mixing it by hand four times for one minute every 30 minutes, allowing it to settle for several minutes, and then analyzing it as described by Sonmez et al. (Sonmez et al., 2008). The second one was made with identical soil and water quantities but with different shaking methods. During this extraction, the mixture was shaken by hand for 10 minutes continuously and then allowed to decant for at least 10 minutes. Finally, the extract was analyzed by ISFET. All
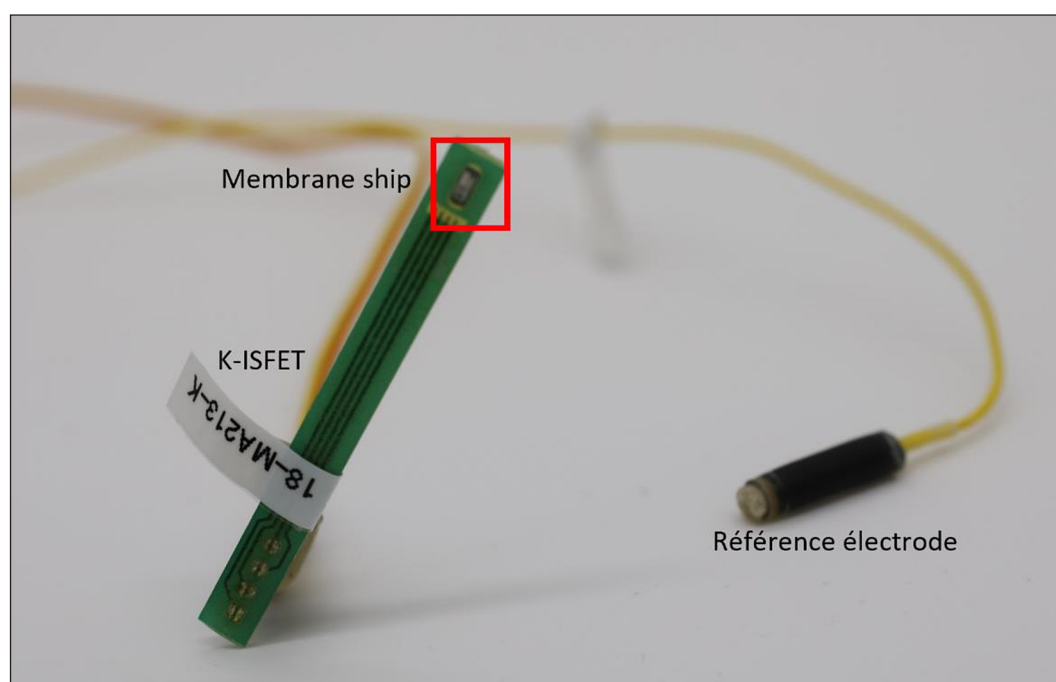

Figure 3. K-ISFET identifying potassium by a PVC membrane using a Ag/AgCl reference electrode

these soil extracts were then measured by the K-ISFET and compared to the continuous flow auto-analyzer (SKALAR) measurement, which is used as a conventional method.

ISFET preprocessing

Calibration is an essential step that needs to be performed before utilizing each sensor. In the case of the ISFET, conditioning and calibration are crucial to ensuring accurate baseline values and optimal performance. Conditioning is recommended prior to the initial use of the ISFET and following extended periods of dryness. This involves immersing the ISFET in a conditioning buffer containing 10 mM potassium chloride (KCl) for two to five hours, while the reference electrode is submerged in a saturated KCl solution for a period of half to two days. To account for baseline drift, The calibration of the potassium ISFET was performed by immersing the sensor into five KCl standards with known concentrations, covering the entire detection range of the ISFET (5.10^{-4} to 10^{-1} M). Each standard solution was supplemented with an ionic strength adjuster (ISA) of NaCl (sodium chloride) as a background solution to ensure the ionic strength equilibrium of the solutions. This calibration process enables accurate potassium detection and compensation for any deviations in the baseline values of the ISFET.

Solutions analyzed by potassium ISFET

The potassium concentrations are predicted by soaking the potassium sensor in the desired solution (soil solution or standard) to obtain the results in approximately five minutes. This sensor has good selectivity for interferent ions in the range of 5.10^{-4} M to 10^{-1} M of potassium and a sensitivity of 50 ($\pm$ 5) mV/decades, with an accuracy of 3 mV.

RESULTS AND DISCUSSION

Statistical study

The collected soil samples, along with their corresponding textures, are presented in Table 1 for reference. The samples exhibit an average value of 15.727 ppm and a standard deviation of 3.87 ppm, indicating considerable variability in the potassium content among the samples.

Table 1. Statistical analysis of the texture of the ten soils

Soil	Soil texture	Potassium concentration (ppm)
1	Clay	21.67
2	Sandy clay loam	20.6
3	Sandy loam	19.63
4	Silty clay loam	15.76
5	Sandy loam	15.7
6	Sandy loam	14.78
7	Clay loam	14.32
8	Sandy clay loam	12.67
9	Sandy loam	12.59
10	Clay loam	9.55
Standard deviation (Sd)		3.87 ppm
Mean		15.727 ppm
Range		9.55 ppm; 21.67 ppm

Furthermore, the chosen concentrations demonstrated substantial variation, effectively representing the diverse potassium levels observed in Moroccan soils. This selection ensures comprehensive coverage of the potassium concentration, enabling a robust analysis of the soil samples and facilitating accurate characterization of the potassium content within the regions.

Calibration diagram

According to the recommendations of international standards (Allison et al., 2006), five standard solutions of KCl were prepared to calibrate the system, and three different concentrations of NaCl (1 M, 0.1 M, and 5 M) were added to these standards as ISAs to adjust the ionic strength of the solution, as clarified in Table 2. This table shows the sensitivities (mV/decades) and the coefficients of determination (R^2) of the calibration diagrams for different ISA concentrations. The highest ISA concentration gives better ionic strength stability than the best results, with an excellent coefficient of determination of 99.79%.

Figure 4 presents the calibration diagram with 5 M NaCl as the ISA; this figure shows an R^2 above 99% with a sensitivity of 48 mV/Dec. In line with the findings of a similar study conducted by N. Bhagat et al. in 2022 (Bhagat et al., 2020), this study employed the same type of ISFET sensor for potassium detection. However, different Ionic Strength

Table 2. Sensitivity (mV per decade) and coefficient of determination (%) of standard curves with different concentrations of NaCl as the ISA solution

Medium	Sensitivity (mV/dec)	R² (%)
KCl	19	94.42
KCl + 0.1 M sodium chloride (NaCl)	46	95.14
KCl + 1 M sodium chloride (NaCl)	38.5	79.85
KCl+ 5 M sodium chloride (NaCl)	37	99.99

Adjuster (ISA) solutions have been utilized and compared in their study. While Bhagat et al. opted for ammonium acetate as their ISA solution, we chose to use NaCl. Interestingly, these study results align with their findings, demonstrating a high coefficient of determination (R^2) of 99.99% and a sensitivity of 32.5 mV/dec when employing ammonium acetate as the ISA solution. However, we observed a slight decrease in R^2 when NaCl was used as the ISA solution, albeit with an increase in sensitivity. These parallel outcomes emphasize the impact of the choice of ISA solution on the potassium detection performance of the ISFET sensor. This indicates that the selection of the ISA solution should be carefully considered to achieve optimal sensitivity and accuracy in potassium analysis using the ISFET technique.

Evaluation of ISFET sensitivity

To assess the sensitivity of the ISFET sensor, a series of nine solutions with varying concentrations of KCl spanning the entire detection range of the ISFET were employed. The resulting curve presented in Figure 5 illustrates an exceptional correlation between the actual values of the prepared solutions and the potassium estimations made by the ISFET sensor, yielding an R^2 of 99.37%, which can be defined as excellent R^2. This outcome confirmed the accurate performance of the sensor in accurately measuring potassium levels. To further validate the reliability of the ISFET sensor, diverse solutions containing interferent ions were introduced at certain concentrations, as illustrated in Figure 6. These interferent ions included 1 M ammonium nitrate (NH_4NO_3), 3 M NaCl, and 0.1 M sodium acetate ($C_2H_3NaO_2$). Remarkably, even in the presence of these interferent ions, the sensor demonstrated a high degree of accuracy, with an R^2 of approximately 98% and a correlation coefficient of approximately 99%. These results highlight the sensor's robustness of the sensor and its ability to accurately detect potassium ions, even in the presence of potential interfering substances.

The findings obtained from laboratory solutions indicate that the ISFET sensor is highly efficient for potassium ion detection, underscoring its suitability for accurate and reliable potassium analysis in various applications.

Equation	y = a + b*x
Slope	47,99843 ± 2,393
R-Square (COD)	0,9926
Adj. R-Square	0,99013
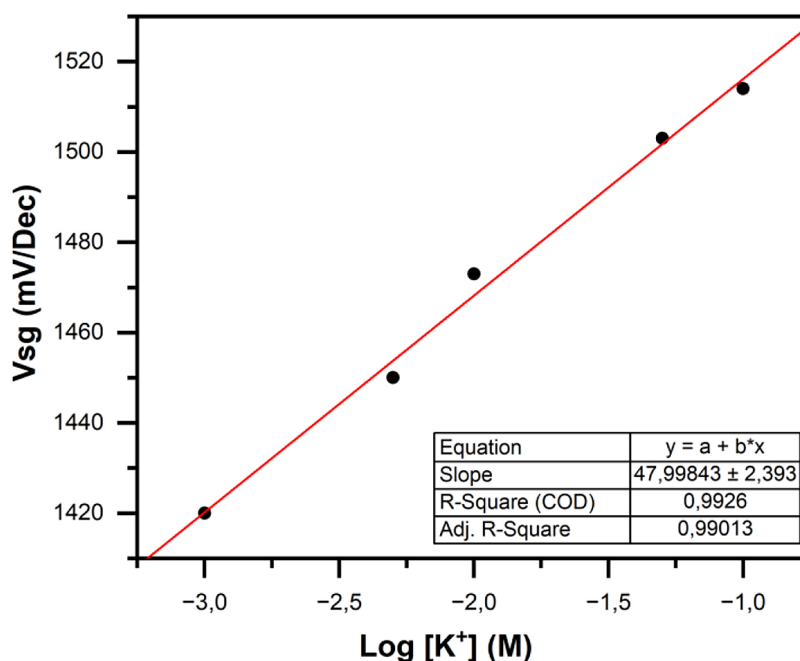
Figure 4. Potassium standard diagram using KCl with 5 M sodium chloride (NaCl) as the ISA
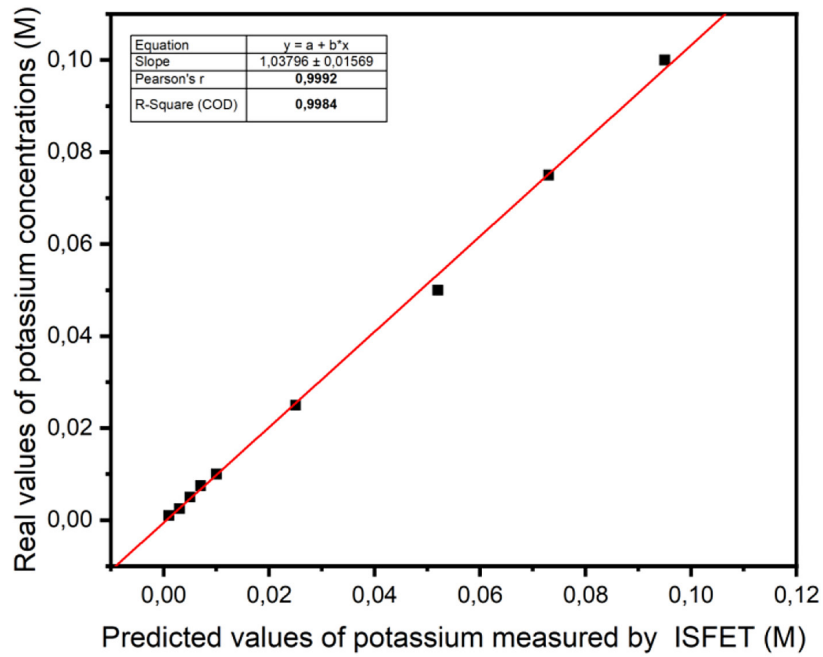
Figure 5. Potassium prediction using ISFETs of different solution concentrations prepared in the laboratory
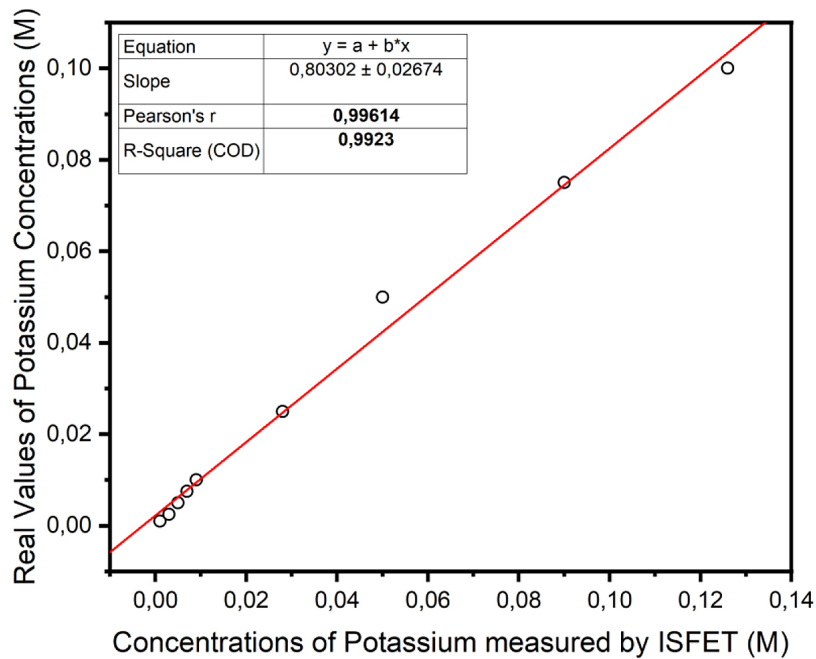

Figure 6. The concentration of potassium measured by the ISFET of different solutions of KCl with added interferent ion solutions

Effect of the soil-to-water ratio on the system accuracy

To determine the optimal method for potassium extraction from soil using water, three different soil-to-water ratios were investigated (1:1, 1:2.5, and 1:5). The results, as depicted in Figure 7, indicate that the 1:5 soil ratio in graphical "a" yields the highest level of accuracy and has an excellent R^2 = 99%. Consequently, the 1:5 soil ratio is considered the most suitable ratio for potassium estimation using the ISFET sensor. In contrast, the 1:2.5 and 1:1 soil ratios mentioned in graphs "b" and "c", respectively, did not demonstrate a strong correlation between the predicted and actual
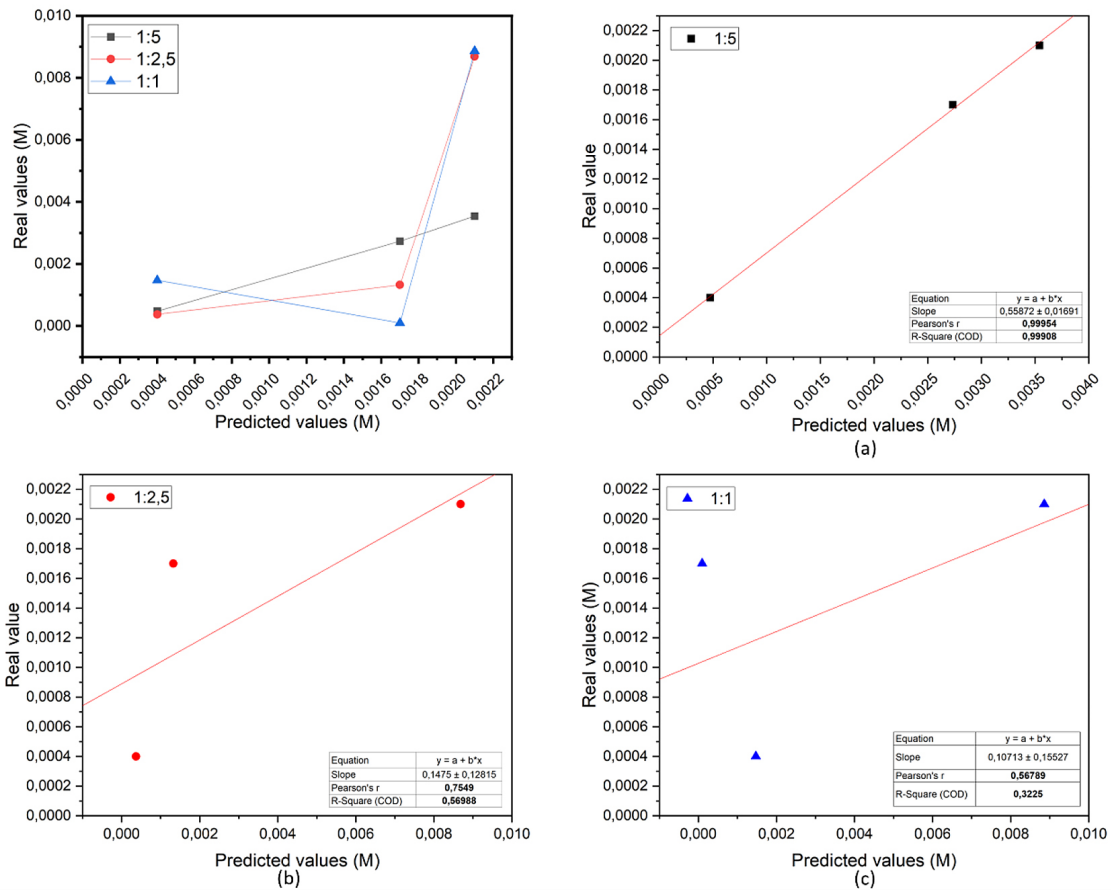
Figure 7. The correlation between the predicted and real values of the three extraction soil ratios (1:1, 1:2.5, and 1:5)

potassium contents. Therefore, these ratios are not recommended for accurate potassium extraction using the ISFET technique. Thus, the 1:5 soil ratio emerged as the standard and recommended ratio for potassium extraction using water. This ratio ensures a robust correlation between the potassium levels estimated by the ISFET sensor and the actual potassium content in the soil (Allison et al., 2006).

Effect of soil extraction methods on system accuracy

To simplify the extraction process and eliminate the need for large amounts of laboratory equipment, two simplified extraction methods were evaluated and compared to the standard extraction method, as shown in Figure 8. The first method was adapted from Sonmez et al. [22],
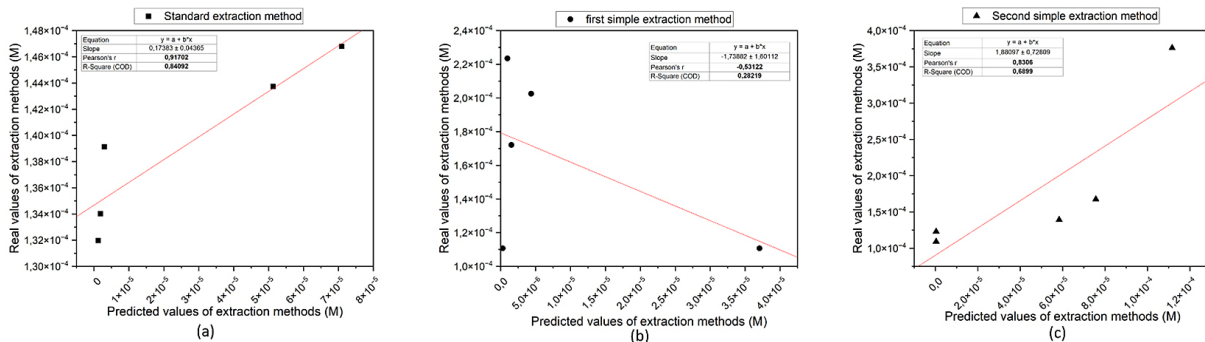

Figure 8. The correlation between the predicted and real values for the standard extraction and the two simple methods
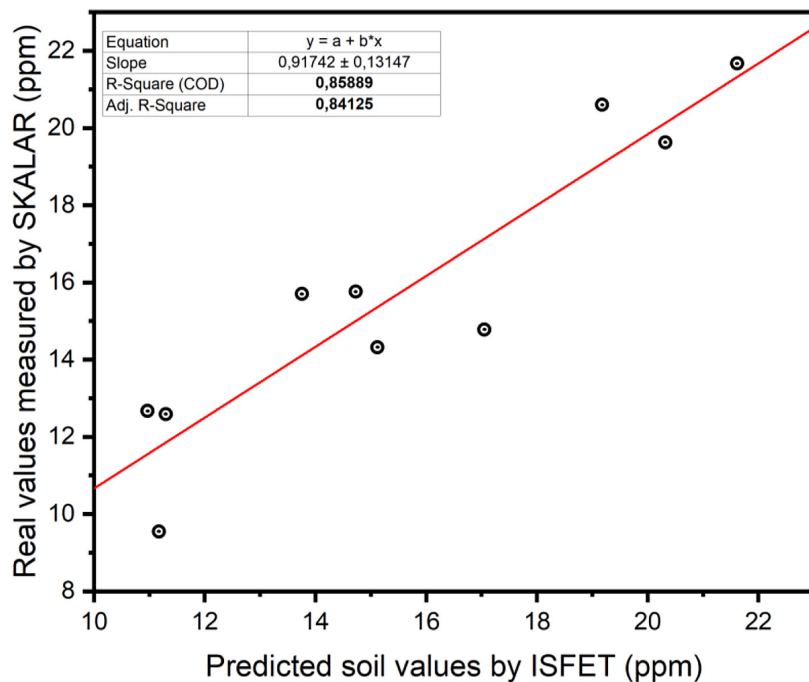
Figure 9. The correlation between predicted and real values of potassium using ISFET and Skalar (Continuous Flow AutoAnalyzer) in Moroccan soils

while the second method was developed in the MAScIR Laboratory. Interestingly, the second simple extraction method demonstrated promising results, as shown in the graphical "c" of Figure 8, with an R^2 value of approximately 70%, which was close to the R^2 value of 84% obtained with the standard extraction method, as presented in the graphical "a" of Figure 8. This suggests that the second simplified method has potential for accurate potassium extraction. In contrast, the Sonmez et al. (Sonmez et al., 2008) method did not yield favorable results in this study, as mentioned in the graphical "b" of Figure 8. This discrepancy may be attributed to variations in soil type and differences in the performance of the sensors used. The obtained results highlight the importance of considering specific soil characteristics and the compatibility of the extraction method with the sensor employed. Further research and refinement of simplified extraction methods are warranted to optimize the accuracy and applicability of potassium extraction without the need for complex laboratory equipment.

Soil potassium estimation by ISFET

The analysis of soil potassium content was conducted on ten different sample types using a standard extraction method, employing a soil-to-water ratio of 1:5. The results illustrated in Figure 9, demonstrate a reasonably acceptable R^2, even when applying a correction factor. However, it should be noted that the correlation between the predicted and actual values is not precise, primarily due to the complex nature of the soil matrix, which can alter the ionic strength of the extracted soil.

Figure 9 shows an R^2 value of 86%, indicating a moderate correlation between the estimated and actual potassium values. This level of accuracy may still be considered acceptable. This suggests that the ISFET sensor has the potential to serve as an alternative method for measuring potassium in soil. It is important to acknowledge the influence of the soil matrix and its impact on the accuracy of potassium measurements. Further research and refinements in the extraction and analysis techniques are necessary to improve the precision and reliability of potassium detection using the ISFET sensor in soil samples. Nonetheless, these preliminary findings demonstrate the viability of utilizing the ISFET sensor as an alternative method for assessing soil potassium content.

CONCLUSIONS

Using only water as the reagent and employing a straightforward extraction method, this study measured potassium levels in Moroccan

soils using ISFET technology, achieving a correlation coefficient of approximately 86% compared to conventional methods. This approach enables farmers to enhance their yields with minimal expense. Consequently, this prompts us to explore additional avenues for our research, including the feasibility of an in-situ, multi-sensor study that would allow farmers to obtain real-time results.

Acknowledgement

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