Preliminary Analysis of Aerosol Jet Printed Solid-Contact K^+ Sensors for Rapid Soil Extraction

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***Abstract*— Rapid monitoring of chemical species in soil is essential for environmental assessment. This work investigates potassium-selective all-solid-state ion-selective electrodes (ISEs) fabricated by Aerosol Jet Printing (AJP) as a model platform for soil contaminant and ion monitoring in rapid extraction matrices. Sensors based on a multi-walled carbon nanotube (MWCNT) solid contact and a PVC potassium-selective membrane are designed, printed by AJP and compared with commercial screen-printed K^+ ISEs. Potentiometric measurements were performed on soil extracts obtained using ammonium acetate (NH_4OAc), a challenging matrix due to high ionic strength and interfering species. With 0.1 M NH_4OAc, the commercial sensor showed a sensitivity of ~48 mV/decade, whereas the AJP-printed sensor exhibited a higher slope of ~81 mV/decade with good repeatability. Increasing the extractant concentration to 1 M NH_4OAc caused unstable responses and membrane degradation. These results demonstrate the suitability of AJP-printed solid-state ISEs for rapid soil analysis and support their potential application in the monitoring of chemically relevant species and contaminants in complex environmental matrices.**

Keywords—ion-selective sensors; potassium detection; printed electrochemical sensors; environmental applications.

I. Introduction

Monitoring soil fertility and nutrient availability is a fundamental requirement for sustainable and precision agriculture. Among the essential macronutrients, potassium (K^+) plays a central role in plant physiology, contributing to osmotic regulation, enzyme activation, stomatal control, and resistance to abiotic stress. Its availability strongly influences crop yield and overall plant health, making rapid and accurate assessment of exchangeable K^+ in the soil a priority for agronomic management [1]. Conventional analytical methods for determining potassium in soil are based on chemical extraction followed by laboratory techniques such as atomic absorption spectroscopy, flame photometry, or ion chromatography [2][3]. Although these approaches offer high accuracy, they require specialized equipment, skilled personnel, and long processing times, making them unsuitable for field or real-time monitoring. As agriculture moves increasingly toward data-driven, site-specific management, there is a growing need for portable, low-cost, rapid detection technologies that can operate directly in complex soil matrices. Ion-selective electrodes (ISEs) represent a promising solution due to their simplicity, low power consumption, and ability to provide direct potentiometric measurements [4][5]. Recent advances in solid-state ISEs have introduced nanostructured conductive materials, such as graphene, carbon nanotubes (CNTs), and PEDOT:PSS, as solid-contact transducers, significantly improving potential stability, drift, and reproducibility [6]. At the same time, the emergence of additive manufacturing techniques has opened up new opportunities for the production of miniaturized, low-cost electrochemical sensors. Technologies such as drop casting and Aerosol Jet Printing (AJP) allow precise control of film morphology, thickness, and uniformity, improving sensor performance while reducing manufacturing complexity and costs [7].

Compared to conventional screen-printing and drop-casting approaches, Aerosol Jet Printing offers several advantages for the fabrication of ion-selective electrodes. AJP enables the deposition of uniform and highly controlled thin films, with precise tuning of thickness, morphology, and material distribution. Its non-contact nature minimizes defects associated with manual deposition and allows the integration of nanostructured materials such as CNTs with improved reproducibility. Furthermore, AJP supports multilayer architectures and localized patterning on small electrode areas, enabling miniaturization and enhanced solid-contact performance compared to traditional manufacturing routes [7][10][11].

Several studies have demonstrated the feasibility of printed ISEs for detecting nutrient ions in aqueous solutions and simplified soil extracts [8] [9]. Reviews on solid-state ISEs emphasize the importance of advanced materials, micro/nanomanufacturing strategies, and miniaturization to improve sensor robustness and enable field use [10][11]. However, despite these advances, the application of printed ISEs in real soil matrices remains challenging. Soil extracts contain interfering ions (e.g., Na^+, NH_4^+) and organic compounds (e.g., urea) that can compromise membrane selectivity and distort the potentiometric response [12][13][14]. Furthermore, most existing studies focus on calibration in ideal solutions or slow extraction protocols, which do not reflect the conditions encountered in agronomic practice.

A particularly relevant gap concerns rapid extraction methods, such as with ammonium acetate (NH_4OAc), widely used in soil science to quantify the exchangeable fraction of potassium. Although fast and effective, NH_4OAc introduces high concentrations of NH_4^+, a strong interferent for valinomycin-based K^+ membranes, making the measurement environment significantly more complex. To date, the behavior of printed ISEs in NH_4OAc soil extracts has not been systematically studied, and no direct comparison with commercial sensors under these conditions has been reported.

In this context, this work focuses exclusively on the evaluation of potassium-selective electrodes in rapid soil extracts obtained with NH_4OAc. The purpose of this study is: to fabricate potassium-selective electrodes using AJP; to compare their performance with that of commercial screen-printed ISEs; to evaluate their response, stability, and feasibility in NH_4OAc soil extracts; to assess the suitability of AJP-printed sensors for future in situ soil monitoring applications. By addressing the challenges presented by rapid extraction matrices, this work contributes to the development of portable, fully printed sensing platforms for precision agriculture, enabling faster and more efficient assessment of soil nutrient availability.

II. Materials and Methods

A schematic overview of the experimental procedure, including soil sampling, potassium extraction, and electrochemical measurements, is shown in Figure 1.

The workflow shown in Fig. 1 has been structured into six numbered phases to improve clarity: (1) soil sampling in the field; (2) preparation of the extractant solution (NH_4OAc); (3) mixing of soil and extractant; (4) filtration of the suspension; (5) incremental addition of the extract onto the electrode surface; (6) potentiometric acquisition and data processing. These phases reflect the complete sequence from sample collection to electrochemical measurement.

A. Chemicals and reagents

Potassium chloride (KCl, analytical grade) was used for the preliminary calibration of potassium-selective electrodes in order to verify their electrochemical response prior to soil analysis. Ammonium acetate solutions (0.1 M and 1 M) were prepared in deionized water. These solutions were used as extractants for soil potassium. The soil potassium measurements refer to exchangeable potassium already present in the soil samples, without external potassium enrichment. The reagents used for the preparation of the potassium-selective PVC membrane included high molecular weight polyvinyl chloride (PVC), plasticizer, potassium ionophore, lipophilic ionic additive, and tetrahydrofuran (THF) as a solvent. All chemicals were analytical grade and were used as supplied.

B. Description and architecture of the ion-selective sensors

In this study, two types of potassium ISEs were used to measure the potassium concentration in soil samples, with the aim of comparing the performance of commercial sensors with that of sensors developed in-house using Aerosol Jet Printing technique.

The first type consisted of commercial screen-printed electrodes (DRP-110KION, Metrohm, Switzerland), characterized by screen-printed carbon electrodes coated with a potassium-selective membrane based on PVC containing conductive additives (e.g., PEDOT, CNT, as reported by the manufacturer).

The second type of sensors was fabricated in the laboratory from commercial screen-printed carbon electrodes (DRP-C110, Metrohm). These electrodes were functionalized by depositing a nanostructured conductive layer of multi-walled carbon nanotubes (MWCNTs, Multi-Walled Carbon Nanotubes ink, Nink 1000, NANOLAB, Waltham, MA, USA), followed by the deposition of a potassium-selective membrane. Both layers were deposited using AJP, which allows precise control of the morphology and thickness of the functional layers.

Table I summarizes the main characteristics of the sensors used in this study, including the type of conductive substrate, the presence of conductive additives, the membrane deposition, and the manufacturing method.

The printed potassium ISEs consist of a multilayer structure built on a screen-printed carbon electrode. A nanostructured layer of MWCNTs was deposited on the working electrode (WE) to act as a solid contact, improving electrical conductivity and signal stability. A potassium-selective membrane was then deposited as the ion-selective layer.

TABLE I
TYPES OF SENSORS USED IN THE STUDY

Sensor type	Conductive substrate	Conductive editing	PVC membrane	Deposition method
Commercial ISE K^+ (DRP-110KION)	Carbon	Conductive additives (e.g., PEDOT/MWCNTs, not declared)	Provided by the manufacturer	Screen printing
DRP-C110 + AJP	Carbon	MWCNTs (printed by AJP)	Developed in laboratory and printed by AJP	Aerosol Jet Printing

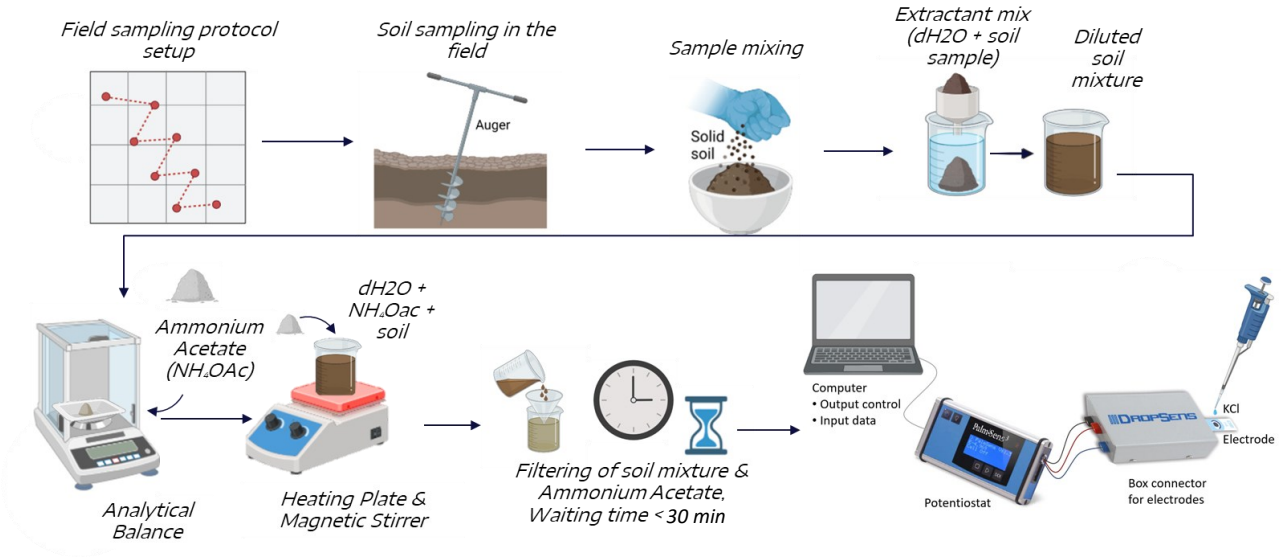

Fig. 1. Schematic representation of the experimental workflow adopted in this study, including soil sampling, potassium extraction using ammonium acetate, sample preparation, and potentiometric measurement using potassium ion-selective electrodes and data acquisition through a potentiometric system

The reference (RE) and counter (CE) electrodes remained unchanged from the original commercial substrate. Figure 2 illustrates the sensor fabrication steps, showing (A) the commercial electrode, (B) the electrode modified with MWCNTs using AJP, and (C) the final device with the PVC membrane.

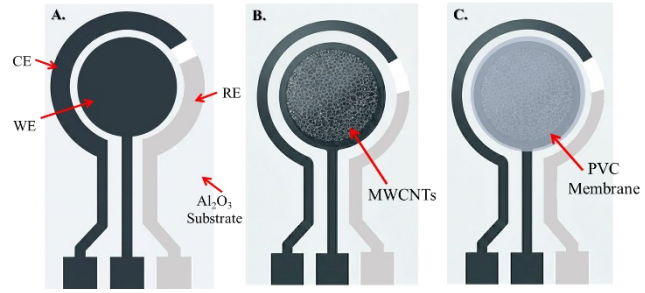

Fig. 2. A. Commercial sensor; B. Modified sensor with MWCNTs in AJP; C. Modified sensor with MWCNTs and PVC membrane in AJP.

C. Aerosol Jet Printing (AJP) process

AJP is a non-contact, and additive manufacturing technique in which functional inks are atomized into aerosol droplets and transported to the nozzle by a carrier gas. The MWCNT layer and PVC membrane were deposited using an aerosol jet printer (AJ-300 UP, Optomec Inc., USA). The printing parameters were optimized on glass substrates. Given the viscosity of the inks, a pneumatic atomization mode was chosen (Figure 3). A spiral deposition pattern was designed to evenly cover the 4.0 mm working electrode area, ensuring controlled overlap between adjacent lines and promoting the formation of continuous, homogeneous layers. The printing parameters used for depositing the MWCNT layer and the PVC membrane are shown in Table II.

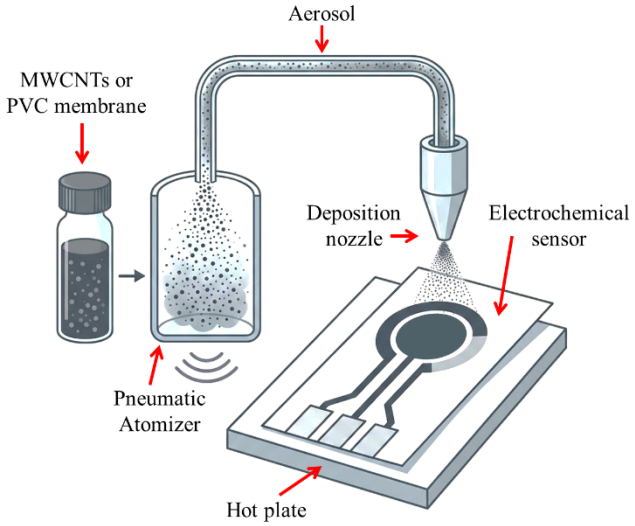

Fig. 3. Aerosol Jet Printing for potassium ion-selective sensors.

D. Rapid soil extraction and calibration procedure

To evaluate the performance of potassium-selective electrodes in real matrices, soil samples collected in the Cassino area (Italy) were analyzed. Soil sampling followed a standard agronomic protocol: approximately 10 subsamples were taken from the top 20 cm using a stainless-steel auger, homogenized in a clean container, air-dried at room temperature, and sieved at 2 mm. All sampling operations were performed under dry weather conditions to minimize moisture-related variability.

Before soil measurements, the electrodes were preconditioned for 15 minutes in a 30 mM KCl solution prepared in 0.1 M ammonium acetate (NH_4OAc). Standard potassium solutions were then prepared and used for calibration by incremental additions (20 µL every 100 s), allowing for greater sensitivity due to the small volume

increments. Calibration curves were obtained by plotting the electrode potential as a function of log[K^+].

TABLE II
AJP PARAMETER FOR PRINTING THE SENSORS LAYERS USED IN THE STUDY

Parameter	*MWCNTs*	*PVC membrane*
Print speed (mm/s)	3	1
Sheat flow (SCCM)	600	1500
Atomizer flow (SCCM)	850	1300
Exhaust Flow (SCCM)	650	1800
Refrigerator (°C)	45	0
Number of depositions	2	2

To accelerate potassium extraction from the soil, ammonium acetate (NH_4OAc) was used as an extractant, since NH_4^+ effectively replaces K^+ at cation exchange sites, allowing quantification of the exchangeable (bioavailable) potassium fraction. A stock solution of 1 M NH_4OAc was prepared in distilled water and diluted to 0.1 M when needed.

For extraction, 10 g of dried soil was mixed with 100 mL of NH_4OAc (soil:solution ratio = 1:10) and shaken for 30 minutes. The suspension was then filtered through 0.45 µm syringe filters, and the extract obtained was used directly for potentiometric measurements.

Potentiometric measurements were performed using a high-impedance acquisition system (PalmSens4, PalmSens BV). The electrodes were operated in drop-mode configuration, where 20 µL aliquots of soil extract were added every 100 s onto the working electrode area. The potential was recorded after stabilization (typically within 5–10 s), and all measurements were conducted at room temperature (22 ± 1 °C).

III. EXPERIMENTAL RESULTS

The soil extract obtained using ammonium acetate (NH_4OAc) was analyzed using the drop-mode configuration, which provided the most stable and interpretable potentiometric response. Rapid extraction allowed the release of the exchangeable potassium fraction, enabling direct comparison between commercial sensors and sensors printed with AJP.

A. Potassium extraction with 0.1 M NH_4OAc

Figure 4 shows the potentiometric response of the commercial KION sensor in soil extracts obtained using 0.1 M NH_4OAc. In Figures 4 and 5, the variable "C" on the x-axis refers to the potassium concentration expressed as logarithmic percentage increments of the soil extract added in drop-mode configuration.

The commercial sensor showed a slope of approximately 48 mV/decade, reflecting the combined effects of ionic strength and NH_4^+ interference. In contrast, the AJP sensor showed a higher slope of ~81 mV/decade, indicating greater sensitivity but also a stronger influence of the extraction matrix. The sensitivity obtained for the commercial KION sensor (~48 mV/decade) is consistent with the values typically reported for screen-printed valinomycin-based K^+ electrodes operating in high-ionic-strength matrices [1], [4]. The reduced slope compared to the ideal Nernstian value is expected in the presence of NH_4^+, which is a known interferent for valinomycin membranes. Ammonium competes with potassium at the membrane interface, altering the phase boundary potential and effectively lowering the apparent sensitivity, as widely reported in the literature on solid-contact ISEs [10][11].

For the AJP-printed sensors, the rapid extraction matrix based on 0.1 M NH_4OAc produced a clear and well-defined potentiometric response, with a steeper slope (~81 mV/decade) compared to the commercial device. This behavior reflects both the higher electroactive surface area of the MWCNT solid-contact layer and the stronger contribution of NH_4^+ to the measured potential. The incremental additions of soil extract introduce simultaneous variations in K^+ and NH_4^+ activity, and the AJP membrane – being freshly printed and highly permeable – responds more markedly to these changes. Despite the complexity of the matrix, the AJP sensors maintained stable potential steps and good repeatability, confirming their suitability for rapid-extraction conditions.

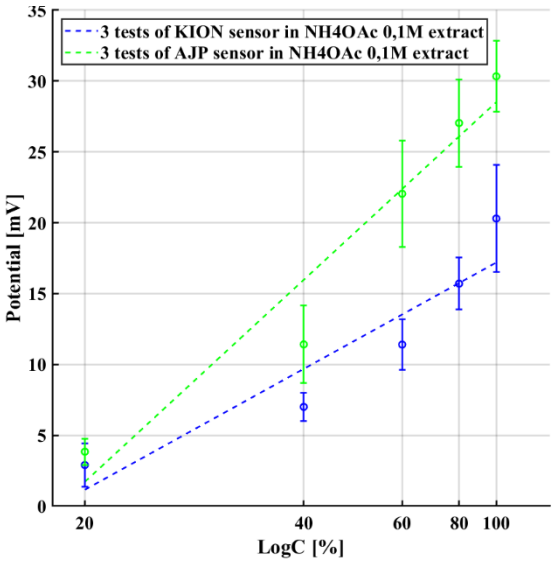

Fig. 4. Comparison between the commercial KION sensor (blue) and the AJP sensor (green) using extraction with 0.1 M NH_4OAc.

Despite this, the AJP sensor demonstrated good reproducibility and a clear potential change following incremental additions of soil extract.

Table III shows the calibration parameters for both sensors.

TABLE III
SENSITIVITY AND STANDARD DEVIATION FOR COMMERCIAL AND PRINTED SENSORS (0.1 M NH4OAC)

Sensor	C range	Sensitivity (mV/dec)	SD (mV)
KION 0.1M	0.5 dec	48.46	1.5; 1.0; 1.8; 1.8; 3.8
AJP 0.1M	0.5 dec	80.95	0.9; 2.7; 3.8; 3.1; 2.5

The slightly higher uncertainty observed for the AJP sensors (Table III) can be attributed to the intrinsic variability of freshly printed membranes and to the higher sensitivity of the MWCNT solid-contact layer to small fluctuations in the extraction matrix. Since the AJP electrodes exhibit a steeper slope, even minor variations in the NH_4OAc/K^+ ratio produce proportionally larger potential changes, resulting in a higher SD compared to the commercial sensor. Nonetheless, the SD values remain within an acceptable range for printed solid-contact ISEs operating in complex matrices [10][11].

B. Potassium extraction with 1 M NH_4OAc

To evaluate the possibility of increasing the amount of potassium extracted through the use of ammonium acetate, the concentration of ammonium acetate was increased to 1 M and the tests were repeated.

Figure 5 shows the response of the sensors in soil extracts obtained using 1 M NH_4OAc. The commercial sensor showed a slope of ~67 mV/decade, while the AJP sensor showed a significantly lower slope (~31 mV/decade). These results are summarized in Table IV. The different behaviour of the sensors is due to the degradation of the membrane.

All the sensors after an optical analysis show degradation of the membrane. Prolonged exposure to 1 M NH_4OAc caused visible degradation of the ion-selective membrane, as shown in Figure 6. Due to this instability and the strong interference introduced by the high NH_4^+ concentration, the 1 M extraction method was excluded from further analysis, and only the results obtained with 0.1 M NH_4OAc are discussed in detail.

The results obtained with 0.1 M NH_4OAc indicate that both sensors are capable of detecting potassium released by rapid extraction, but with different sensitivities. The commercial sensor shows a reduced slope due to NH_4^+ interference, while the AJP sensor has a greater slope and more pronounced potential variations.

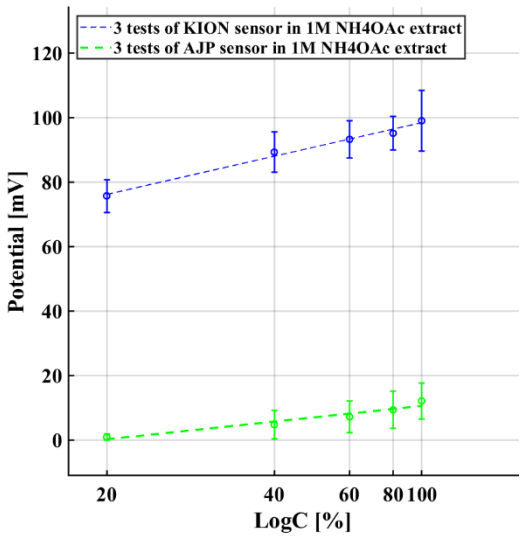

Fig. 5. Comparison between the commercial KION sensor (blue) and the AJP sensor (green) using extraction with 1 M NH_4OAc.

TABLE IV
SENSITIVITY AND STANDARD DEVIATION FOR COMMERCIAL AND PRINTED SENSORS (1 M NH_4OAc)

Sensor	C range	Sensitivity (mV/dec)	SD (mV)
KION 1M	0.5 dec	67.31	5.1; 6.2; 5.8; 5.2; 9.5
AJP 1M	0.5 dec	31.28	0.9; 4.5; 4.8; 5.8; 5.5

Fig. 6. Degradation of the ion-selective membrane after exposure to 1 M NH_4OAc.

C. Measurement uncertainty analysis

The uncertainty values reported in Figures 4 and 5 and in Tables III and IV correspond to the standard deviation of repeated measurements performed under the same conditions (repeatability). For each sensor type, five consecutive measurements were carried out using independent incremental additions of soil extract. The standard deviation was computed from the potential values recorded at each concentration step. Reproducibility, defined as the variability between different sensor batches, was not evaluated in this preliminary study and will be addressed in future work. The number of digits used to express the standard deviations has been reduced to two, in accordance with metrological guidelines.

Sensor sensitivity was determined from the slope of the linear regression of potential vs $\log[K^+]$. The uncertainty associated with the sensitivity corresponds to the standard error of the regression slope. The correlation coefficients (R^2) were 0.987 for the commercial sensor and 0.992 for the AJP sensor in 0.1 M NH_4OAc, confirming the linearity of the calibration.

IV. CONCLUSION AND FUTURE DEVELOPMENTS

Experimental evaluation of commercial and AJP-printed potassium-selective electrodes in rapid soil extracts highlights the critical influence of the extraction matrix on potentiometric performance. The use of ammonium acetate (NH_4OAc) allowed the release of the exchangeable potassium fraction but introduced significant challenges due to the presence of NH_4^+, a known interferent for valinomycin-based membranes.

The behavior observed with 1 M NH_4OAc further emphasizes the importance of matrix optimization. Although the commercial sensor exhibited a high sensitivity (~67 mV/decade), the AJP sensor showed a significantly reduced response, and both devices suffered membrane degradation due to the high ammonium concentration. The visible detachment of the ion-selective membrane confirms

that 1 M NH_4OAc is not compatible with PVC-based ISEs and is therefore not suitable for reliable potentiometric measurements. For this reason, only the results obtained with 0.1 M NH_4OAc were considered valid for comparison.

When 0.1 M NH_4OAc was used, both sensors showed measurable and stable responses, albeit with different sensitivities. The commercial sensor showed a reduced slope (~48 mV/decade), reflecting the combined effects of ionic strength and NH_4^+ interference. In contrast, the sensor printed with AJP showed a steeper slope (~81 mV/decade), indicating higher sensitivity. This behavior highlights the effectiveness of the printed MWCNT solid contact layer, whose nanostructured morphology improves charge transfer efficiency and increases the effective electroactive surface area. Comparison with the commercial sensor also suggests that additive manufacturing, and in particular AJP, enables the fabrication of solid contact layers with improved electrochemical performance compared to conventional screen-printing techniques. Moreover, despite the complexity of the matrix, the AJP sensor demonstrated good repeatability, given the low SD for both 0.1 M and 1 M.

Future work will focus on optimizing the formulation of the AJP-printed membrane to improve its homogeneity, selectivity, and long-term stability. Further efforts will be directed toward integrating printed sensors into portable measurement platforms for field monitoring, evaluating their performance under varying environmental conditions, and extending the approach to other nutrient ions to enable multi-ionic detection. The development of fully printed sensor arrays represents a promising direction for the creation of scalable, low-cost analytical tools for precision agriculture.

Overall, this study demonstrates that AJP-printed potassium-selective electrodes can function effectively in rapid soil extracts, offering greater sensitivity and good repeatability compared to commercial screen-printed sensors, even in the presence of interfering species. Focusing exclusively on the rapid extraction protocol, which is particularly relevant for agronomic applications requiring rapid assessment of nutrient availability, this work confirms that 0.1 M NH_4OAc represents an adequate compromise between extraction efficiency and sensor compatibility. The results further highlight the potential of additive manufacturing approaches for the development of next-generation ion-selective electrodes tailored for field and in-situ soil analysis.

ACKNOWLEDGMENT

"Funded by the European Union - Next Generation EU, Mission 4 Component 1 CUP D53D23001390006 PRIN 2022 - project code 2022P3JY7N - PE7 - New sensors and measurement techniques for underground monitoring and mapping. CUP D53D23001390006 using PRIN 2022 funds financed by the European Union - Next Generation EU mission 4 Education and Research, component 2, investment 1.1.

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