Abstract—Low-cost, accurate monitoring of macronutrient ions in soils, plants, and water is highly desired to improve fertilizer management for maximum profitability and minimum negative environmental impacts. Traditional ion-selective electrodes (ISEs) suffer from interference from non-target ions. This paper reports the integration of artificial neural networks (ANNs) and a miniature sensor containing an array of three ISE-based sensing elements to improve accuracy of the sensor in detecting and quantifying target nitrate (NO$_3^-$), phosphate (H$_2$PO$_4^-$), and potassium (K$^+$) ions in the environment. The sensor outputs of NO$_3^-$, H$_2$PO$_4^-$, and K$^+$ ion concentrations are used to train and optimize ANNs. The optimized neural networks are applied to classify and estimate concentrations of the target ions in the presence of interfering ions. The ANN-assisted array of sensing elements reduces cross-sensitivity between these elements. The present sensor is validated with measurements of NO$_3^-$, H$_2$PO$_4^-$, and K$^+$ ions in soil solution, plant sap, and tile drainage water from crop fields.

Index Terms—Agricultural sensor, ion-selective electrode, soil, plant, tile drainage water, ANN, cross sensitivity, machine learning.

I. INTRODUCTION

Nitrogen, phosphorus, and potassium are primary macronutrients essential for plant growth and development. Fertilizer application supplies crops with these nutrients. Under-application of fertilizer causes economic loss due to reduced yield quality, while over-application causes economic loss due to unused fertilizer. Moreover, the unused fertilizer is typically lost to the environment, where it pollutes air and water resources. Crop production is the leading cause of nutrient losses to aquatic ecosystems [1], [2]. Regionally, nitrogen and phosphorus fertilizers are the leading cause of eutrophication and hypoxia in aquatic ecosystems, which promotes algal blooms and reduces biodiversity. Locally, nitrogen fertilizers pollute drinking water supplies; the maximum contaminant level (MCL) for nitrate ions in public water supplies is 10 mg N L$^{-1}$ set by the United States Environmental Protection Agency [3]. High nitrate levels in drinking waters have been associated with a variety of negative human health outcomes. The recommended MCL of total phosphate in water streams to prevent eutrophication is 0.05 mg P L$^{-1}$ [4]. Although potassium movement in soil is relatively slow and potassium fertilizers do not contribute to air and water pollution, potassium deficiency can reduce crop yield and quality, while potassium over-application has a detrimental effect on crop quality and soil productivity [5]. To reduce the fertilizer input costs and minimize their adverse environmental and human health effects, there is a need for improving our ability to accurately monitor nutrient status, fertilizer application, and environmental impact at low cost, which is of great benefit to agricultural producers and environmental conservation practitioners [6].

Laboratory-based ion measurement methods, such as ion chromatography, spectrophotometry, and chemiluminescence, offer high sensitivity and selectivity; but, these technologies are not suitable for field application due to high cost and bulky size [7], [8]. Portable spectrophotometers are an attractive solution to environmental monitoring of ions for agricultural management [9]; however, they are still relatively expensive and often require site-specific calibrations.
for field applications. Many miniaturized nutrient ion sensors and measurement systems have been reported based on different sensing mechanisms [10], such as ion-selective electrode (ISE) [11], ion-selective field-effect transistor [12], [13], microwave resonance [14], [15], colorimetry [16], fiber-optic spectroscopy [17], electrochemical measurement [18], [19], microfluidic electrophoresis chips [20], and enzymatic sensing [21], [22]. But, much room exists to improve the sensitivity, selectivity, stability, and cost-efficiency of these miniaturized ion sensors. For example, permeable biocatalytic membranes have also been integrated into microfluidic sensors to facilitate ion movement into measurement chambers where enzymes are used to reduce specific ions for quantification [24], [25] but their selectivity and sensitivity are compromised by the instability of enzyme molecules used in the sensors.

ISE-based sensors are widely used in analytical chemistry due to their simple structure and low cost. But, these sensors suffer from a signal drift and instability over time because there is an issue with chloride leaching from silver/silver chloride (Ag/AgCl)-based pseudo reference electrodes (REs) due to the redox reaction occurring at these REs [26]. Also, ion-selective membranes (ISM) of the ISEs are interfered with by non-target ions. The ion exchange-induced potential \( E \) at the ISE is described as \( E = \text{constant} + (R \times T / z \times F) \times \ln \left( \alpha + \sum (k_i \alpha_i) \right) \), where \( R \) is the gas constant, \( T \) is the temperature, \( F \) is the Faraday constant, \( z \) and \( z_i \) are the charge of the target ion and interfering ion \( i \), respectively, \( \alpha \) is the chemical activity for the relevant ion, and \( k_i \) represents the selectivity coefficient [27]. It should be noted that because the potential \( E \) is logarithmic to the sum of the total activities of both target and non-target ions, ISEs exhibit a non-optimal ion selectivity under the exposure to various ion species in environments; this makes ISEs not a popular solution to detection of nutrient ions in agricultural soil solution and plant sap where dissolved ions are extremely rich. In addition, a thin water layer often develops and is trapped at the interface between the ISM and the conducting base electrode of the ISE. This thin water layer acts as not only as a barrier to the transfer of electrons to the base electrode but also a trap for water layer often develops and is trapped at the interface between the ISM and the conducting base electrode of the ISE. This thin water layer acts as not only as a barrier to the transfer of electrons to the base electrode but also a trap for water layer often develops and is trapped at the interface between the ISM and the conducting base electrode of the ISE. This thin water layer acts as not only as a barrier to the transfer of electrons to the base electrode but also a trap for water layer often develops and is trapped at the interface between the ISM and the conducting base electrode of the ISE. This thin water layer acts as not only as a barrier to the transfer of electrons to the base electrode but also a trap for water layer often develops and is trapped at the interface between the ISM and the conducting base electrode of the ISE. This thin water layer acts as not only as a barrier to the transfer of electrons to the base electrode but also a trap for water layer often develops and is trapped at the interface between the ISM and the conducting base electrode of the ISE. 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### A. Materials

\( \text{NO}_3^- \) ISM cocktail was prepared by mixing methyltriphosphonium bromide (0.25 wt %), nitrocellulose (moistened with 2-propanol (35%); 1.93 wt %), 2-nitrophenyl...
Fig. 1. (a) and (b) Photos of the fabricated multi-ion sensor containing an array of three ISE-based sensing elements to detect NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ions. (a) These sensing elements are formed on one side of a needle-shaped PCB. (b) An Ag/AgCl-based RE is shared by the three sensing elements and formed on the other side of the PCB. (c) Diagrams of building, training, and testing ANNs for improving accuracy in estimating concentrations of NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ions.

octyl ether (NPOE) (16.25 wt %), polyvinyl chloride (PVC) (5.75 wt %), tetrahydrofuran (THF) (74.3 wt %), and tridodecylmethylammonium nitrate (1.50 wt %). H$_2$PO$_4^-$ ISM cocktail was prepared by mixing tributyltin chloride (0.1 wt %), NPOE (6.55 wt %), PVC (3.27 wt %), and sodium tetraakis-[3,5- bis(trifluoromethyl)phenyl]borate (NaTFPB) (0.07 wt %) and THF (90 wt %). K$^+$ ISM cocktail was formed with valinomycin (0.2 wt %), PVC (6.56 wt %), NPOE (13.2 wt %), potassium tetraakis-[3,5- bis(trifluoromethyl)phenyl]borate (KTFPB) (0.04 wt %), and THF (80 wt %). These cocktail solutions were stirred using a magnetic stirrer for 12 hours and stored at -20 °C. Single-salt standard ion solutions were prepared by dissolving appropriate amounts of NaNO$_3$, NaH$_2$PO$_4$, or KCl in deionized (DI) water. For each ion type (NO$_3^-$, H$_2$PO$_4^-$ or K$^+$), four concentrations ($10^{-4}$ M, $10^{-3}$ M, $10^{-2}$ M and $10^{-1}$ M) were also prepared.

A set of 63 training samples was prepared and used to train ANN models, consisting of a mixture of NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ions with the concentration of each ion type varying among
C. Testing Setup
Perform the potentiometric measurement. and RE to the same medium, thus forming a closed circuit to holes were created on the PCB to allow exposing all the ISEs dried at room temperature for 10 hours. The multiple through-

holes were drop casted on the surface of the Ag/AgCl electrodes and immersed into a standard NO

C. Device Fabrication
The multi-ion sensor was manufactured on a 0.8 mm-thick PCB (OSH PARK) that had a needle shape. The needle part of the sensor was 25 mm-long and 4.2 mm-wide. The three ISEs of the sensor shared a single RE placed on the other side of the PCB. The RE was formed by screen-printing 0.2 mm thick, 1 mm diameter Ag/AgCl ink on a 0.8 mm-diameter circular copper contact pad with the help of a stencil mask, and then was dried on a hotplate at 110 °C for 2 hours. Similarly, to form the ISEs, three circular copper contact pads (0.8 mm diameter each) were coated with 0.2 mm thick, 1 mm diameter Ag/AgCl ink using the screen-printing method; next, the corresponding ISM solutions of NO

D. Optimization of ANN Architecture
Two types of ANN models were constructed. The first type (namely, individual ANN model) was built to predict a single ion species of NO

III. RESULTS AND DISCUSSION
Table II summarizes the performance of the individual and group ANN models for predicting NO

TABLE I
PARAMETERS OF THE ANN MODELS

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
<th>Definitions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hidden layer size</td>
<td>8, 10, 15, 20</td>
<td># of neurons in a hidden layer</td>
</tr>
<tr>
<td>Max. # hidden layers</td>
<td>3</td>
<td># of hidden layers</td>
</tr>
<tr>
<td>Adaptive value µ</td>
<td>0.001, 0.1</td>
<td>Controlling magnitude of weight changes based on output errors</td>
</tr>
<tr>
<td>µinc</td>
<td>1.5, 10</td>
<td>Increase factor of µ</td>
</tr>
<tr>
<td>µdec</td>
<td>0.1, 0.5</td>
<td>Decrease factor of µ</td>
</tr>
<tr>
<td>Training goal</td>
<td>10^4</td>
<td>Goal for RMSE to stop training</td>
</tr>
<tr>
<td>Max. # of epochs</td>
<td>10,000</td>
<td>Training stops if full set of the data greater than this number of times</td>
</tr>
<tr>
<td>Hidden layer transfer function</td>
<td>tansig</td>
<td>Hyperbolic tangent sigmoid transfer function that calculates hidden layer’s output from net input</td>
</tr>
<tr>
<td>Output layer transfer function</td>
<td>purelin</td>
<td>Linear transfer function for output calculation</td>
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<td>Training fraction</td>
<td>0.7</td>
<td>Training subset data fraction</td>
</tr>
<tr>
<td>Validation fraction</td>
<td>0.15</td>
<td>Validation subset data fraction</td>
</tr>
<tr>
<td>Testing fraction</td>
<td>0.15</td>
<td>Testing subset data fraction</td>
</tr>
</tbody>
</table>
Fig. 2. Calibration curves of the ISEs, showing the output voltage of each ISE as a function of concentration of target ion: (a) $\text{NO}_3^-$, (b) $\text{H}_2\text{PO}_4^-$, and (c) $\text{K}^+$. The calibration was performed with a series of ion concentrations in single-salt solutions.

Table II

<table>
<thead>
<tr>
<th>Parameters of the Optimal ANNs for Predicting $\text{NO}_3^-$, $\text{H}_2\text{PO}_4^-$ and $\text{K}^+$ Ion Concentrations in the Absence of Interfering Ions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type of ion</td>
</tr>
<tr>
<td>-------------</td>
</tr>
<tr>
<td>Individual ANN model</td>
</tr>
<tr>
<td>NO3-</td>
</tr>
<tr>
<td>H2PO4-</td>
</tr>
<tr>
<td>K+</td>
</tr>
<tr>
<td>Group ANN model</td>
</tr>
<tr>
<td>NO3-, H2PO4- and K+</td>
</tr>
</tbody>
</table>

0.30, respectively. The slopes of the linear fitting curves for the ANN-predicted $\text{NO}_3^-$, $\text{H}_2\text{PO}_4^-$ and $\text{K}^+$ ion concentrations are 0.934, 0.927, and 0.943, respectively, indicating a considerable correlation of the predicted ion concentrations to the prepared ones.

Further, the optimal individual ANNs were used to predict the concentrations of $\text{NO}_3^-$, $\text{H}_2\text{PO}_4^-$ and $\text{K}^+$ ions in the samples containing not only $\text{NO}_3^-$, $\text{H}_2\text{PO}_4^-$ and $\text{K}^+$ ions, but also $\text{Cl}^-$ and $\text{Ca}^{2+}$ interfering ions. The prepared concentrations of $\text{NO}_3^-$, $\text{H}_2\text{PO}_4^-$ and $\text{K}^+$ ions were set as the targets of the output layer of each individual ANN, while the sensor-measured concentrations were set as the three inputs of the input layer of the ANN. The $\text{Cl}^-$ and $\text{Ca}^{2+}$ ion concentrations were treated as the unknown input values for the ANN. The scatter plots in Fig. 4a-4c show that, for measuring the concentrations of $\text{NO}_3^-$, $\text{H}_2\text{PO}_4^-$ and $\text{K}^+$ ions, the sensor measurements provide the RMSE values of 0.48, 0.55, and 0.66, respectively; while the ANN models result in the RMSE values of 0.35, 0.46, and 0.43 with the slopes of the linear fitting curves at 0.968, 0.889, and 0.925, respectively. Therefore, for a given target ion, the ANN prediction exhibits a smaller RMSE value than the sensor measurement, indicating that the incorporation
Fig. 3. Scatter data plots showing the ion concentrations of (a) NO$_3^-$, (b) H$_2$PO$_4^-$, and (c) K$^+$ measured by the sensor and predicted by the optimal individual ANNs (Y-axis) with respect to the prepared ion concentrations (X-axis) in training-samples without interfering ions.

<table>
<thead>
<tr>
<th>Type of ion</th>
<th># of neurons in hidden layer 1</th>
<th># of neurons in hidden layer 2</th>
<th>RMSE</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>NO$_3^-$</td>
<td>20</td>
<td>0</td>
<td>0.35</td>
<td>94%</td>
</tr>
<tr>
<td>H$_2$PO$_4^-$</td>
<td>8</td>
<td>10</td>
<td>0.46</td>
<td>95%</td>
</tr>
<tr>
<td>K$^+$</td>
<td>8</td>
<td>15</td>
<td>0.43</td>
<td>95%</td>
</tr>
</tbody>
</table>

Table III summarizes the ANN settings and the obtained RMSE and $R^2$ values for predicting NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ion concentrations in the presence of Cl$^-$ and Ca$^{2+}$ ions. The RMSE values become higher, and the $R^2$ values become lower with Cl$^-$ and Ca$^{2+}$ ions, compared to those obtained with no interfering ions (Table II).

Moreover, the incorporation of the ANN model with the three-element sensor improved the accuracy in determining NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ion concentrations in various real samples from agriculture croplands, maize stalk sap, soil solution, and drainage (i.e., 'tile') water. For in-situ measurement of NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ions in maize stalks, plants (genotype: B73) were grown in the greenhouse. The sensor measurement was conducted at the V8 growth stage [43]. The sensor was directly inserted into the stalk at ~8 cm above the soil surface. The sensor was then left in the stalk until the signal became stable. Next, the sensor was used to measure NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ions in soil solution. The soil was placed on a cellulose filter paper (Whatman) in a funnel and became water saturated by adding 500 ml water that contained NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ions of $10^{-3}$ M concentration each. To validate the machine learning-assisted sensor in measuring NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ions in agricultural tile drainage water, water samples were collected from drainage systems on the Iowa State University research farm (Ames, Iowa). The sensor was dipped into the collected sample for direct measurement. For comparison, we also performed traditional measurements of NO$_3^-$, H$_2$PO$_4^-$ and K$^+$ ions in the fluids squeezed from the stalk at the inser-
tile drainage water samples. The result indicates that the individual ANN model can improve the measurement accuracy over the direct sensor measurement. For \(\text{NO}_3^-\), \(\text{H}_2\text{PO}_4^-\), and \(\text{K}^+\) ions, the RMSE values with the sensor alone were found to be 0.68, 4.85, and 0.90, respectively; however, the ANN-assisted estimation helped lower the RMSE values to 0.28, 0.69, and 0.70, respectively. Also, for \(\text{NO}_3^-\), \(\text{H}_2\text{PO}_4^-\), and \(\text{K}^+\) ions, the \(R^2\) values of the ANN-predicted result were 92%, 71%, and 81%, respectively, while the \(R^2\) values with the sensor alone increased to 71%, 59%, and 79%, respectively. Therefore, compared to the sensor alone, the incorporation of the ANN with the sensor improved the measurement accuracy substantially for \(\text{NO}_3^-\) and \(\text{H}_2\text{PO}_4^-\) ions (Fig. 5a-5b), but only slightly for \(\text{K}^+\) ion. The difference in the efficacy of the ANN model for predicting the three ion species may be associated with the difference of the ISEs in selecting the corresponding target ions. As shown in Fig. 5c, the sensor-measured data for \(\text{K}^+\) ions appeared more disperse than those for \(\text{NO}_3^-\) and \(\text{H}_2\text{PO}_4^-\) ions, indicating the lower selectivity of the ISM for \(\text{K}^+\) ion compared to the other two ISMs for \(\text{NO}_3^-\) and \(\text{H}_2\text{PO}_4^-\) ions.

The present sensor includes three ISE-based sensing elements with different sensing characteristics. These elements exhibit differential interactions with analytes. These readings from these elements are analyzed by the machine learning algorithm to detect and quantify target ions. There is much room to improve the accuracy of the ANN-assisted sensor for estimating different ion species in environments. Numerous ion species exist in real samples and interfere measurement accuracy of the sensor. The bias in the determination of the target ion species is affected by these interfering ions but can be reduced by increasing the number of the ISE-based sensing elements, where each ISE uses an ISM responsible for a target ion ideally. Also, by increasing the data set size of the calibration for the sensor, it is possible to improve the prediction accuracy of the neural network. We believe that by improving the selectivity of each sensing element to its target species, the response diversity across multiple sensing elements of the device will be increased, which, in turn, will aid in discrimination and quantification for the target species from the background interferences in the environment. Other future efforts include expanding the detection capability of the present sensor system from \(\text{NO}_3^-\), \(\text{H}_2\text{PO}_4^-\), and \(\text{K}^+\) to not only other agriculturally important nutrient ions such as \(\text{NO}_2^-\), \(\text{SO}_4^{2-}\), \(\text{NH}_4^+\), \(\text{Cl}^-\), \(\text{Mg}^{2+}\), \(\text{Ca}^{2+}\) and \(\text{Na}^+\), but also physical parameters such as temperature, moisture, and water...
Fig. 5. Photos of the sensor for measuring NO$_3^-$, H$_2$PO$_4^-$, and K$^+$ ions in different samples. Upper left: the sensor was inserted into the stalk of a maize plant; upper right: the sensor was placed in the soil; lower: the sensor was placed in the tile drainage water collected from the crop field. The traditional analytical instruments were used to test the following samples, including the squeezed fluids from the stalk using a juice squeezer (upper middle), the drained water from the soil in the funnel (upper right), and the same tile drainage water collected from the farm. Scatter data plots show the concentrations of (b) NO$_3^-$, (c) H$_2$PO$_4^-$, and (d) K$^+$ ions measured using the sensor and predicted using the ANN (Y-axis) with respect to the results obtained using the traditional analytical instrument (X-axis).

potential [44]. From an application perspective, in addition to improving fertilizer management [45], the machine learning-assisted array of sensing elements can be modified to detect many other biochemical compounds of environmental concern such as pesticides [46], volatiles [47], and greenhouse gas emissions (e.g., nitrous oxide, carbon dioxide, and methane).

IV. CONCLUSION

A machine learning-assisted multi-ion sensor was developed to improve the identification and quantification of NO$_3^-$, H$_2$PO$_4^-$, and K$^+$ ions in various samples in agriculture. The ANN model was optimized to reduce cross-sensitivities between the three ISE-based sensing elements integrated on the sensor. The prediction for a single ion species by an individual ANN was more accurate than the prediction for all three ions together using a single ANN. Three optimized individual ANNs were used to estimate the concentrations of NO$_3^-$, H$_2$PO$_4^-$, and K$^+$ ions, respectively; the result shows that they provided the prediction accuracy of 96% across the three ions based on the R$^2$ value that examined the linearity between the predicted and actual ion concentrations. Further, the ANN-assisted multi-ion sensing approach exhibited an improved ability to measure the target ions in the presence of interfering ions. Lastly, this method was validated in detecting these nutrient ions in the stalk, soil, and tile drainage water in agriculture.

ACKNOWLEDGMENT

The authors would like to thank Dr. Patrick Schnable, Dr. Baskar Ganapathysubramanian, Hussam Ibrahim, Koushik Nagasubramanian, and Raufur Rahman Khan for insightful discussions.

REFERENCES


Yuncong Chen received the B.S., M.S., and Ph.D. degrees from Iowa State University in 2015, 2017, and 2021, respectively, all in electrical engineering. His research interest includes low-cost and miniature sensors for environmental monitoring.

Zheyuan Tang received the B.S. and M.S. degrees in electrical engineering from Iowa State University in 2019 and 2021, respectively.

Yunjiao Zhu received the B.S. degree in agricultural water conservancy engineering from Hohai University, China, in 2018. She is currently pursuing the Ph.D. degree in soil sciences with the Agronomy Department, Iowa State University.

Michael J. Castellano received the Ph.D. degree in soil science from The Pennsylvania State University in 2009. He is the William T. Frankenberger Professor of Soil Science with the Agronomy Department, Iowa State University. His research focuses on maximizing sustainable productivity of crop systems. He has particular interest in nitrogen because nitrogen is a costly input and often limits crop production, but is easily lost to the environment where it becomes an economic loss that degrades air and water quality.

Liang Dong (Member, IEEE) received the Ph.D. degree in electronic science and technology from the Institute of Microelectronics, Tsinghua University, China, in 2004. From 2004 to 2007, he was a Postdoctoral Research Associate with the University of Wisconsin-Madison. He is the Vikram L. Dalal Professor with the Department of Electrical and Computer Engineering, Iowa State University. His research interests include MEMS, sensors, micro-fluidics, micro-optics, and advanced materials and manufacturing and their applications in agriculture, environment, plant and animal science, and biomedicine. He serves as the Editor-in-Chief of Sensors and Actuators A: Physical, an Associate Editor for Micro & Nano Letters, and a Topic Editor for Biosensors.