

## REVIEW

# Challenges of polymer-based pH sensing in soil

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**Abstract**

It is well established that plants need a range of soil nutrients to grow. In farming, these nutrients are generally added to the soil in the form of fertilizers. However, depending on the soil conditions (such as temperature, water content, pH, and soil type), nutrients may not be in the right form for plant uptake. Determining the availability of nutrients in the soil for plant growth is therefore critical for the yield and productivity of modern farming. A considerable amount of research and knowledge has been developed that shows the importance of the soil pH on the availability (or not) of nutrients. Furthermore, pH plays a crucial role in controlling the availability of potential toxic elements, such as aluminum and manganese. This review article discusses recent research aimed at real-time and continuous soil pH measurement in-situ. More specifically, it focuses on the development of polymer materials that will ultimately enable pH measurements for the specific application of in-ground pH sensing. Given the breadth of the polymeric sensor research field, this review has a narrowed focus on optical and electrochemical transduction methods.

**KEYWORDS**

agriculture, pH, polymers, sensing

## 1 | INTRODUCTION

Concerns regarding global food security have been growing over recent decades, with crop yield and productivity not keeping pace with the world's population growth.<sup>1</sup> Estimates in 2021 highlighted approximately 2.3 billion people or 29.3% of the global population are food insecure.<sup>2</sup> This provides significant motivation to increase crop yield and productivity – which is closely linked with maintaining healthy soil conditions in agriculture.<sup>3</sup> Soil condition monitoring assists farmers in using resources more efficiently (more outputs and less

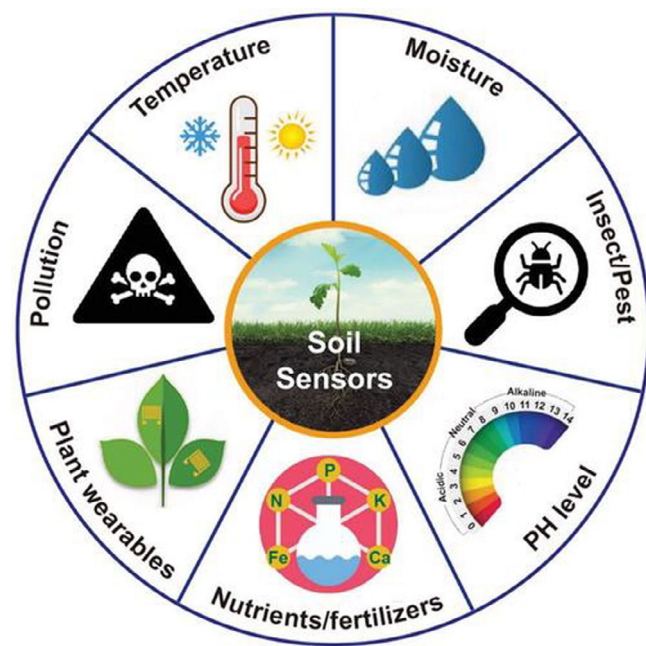
environmental side effects) by providing information for building site-specific databases about their soils. Site-specific soil databases are essential for intelligent and sustainable agricultural systems to understand the relationships between soil conditions and plant growth.<sup>4</sup> In the vast majority of cases, soil properties are measured by taking soil samples and sending them to a laboratory for analysis.<sup>4</sup> Alternatively, soil measurements can be made on-site to gather data for more rapid decision making by farmers. Soil type and properties, diseases, weeds, pests, and past land management can lead to spatial differences across a farm and/or as a function of depth within the

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soil. In some cases, certain soil characteristics such as moisture, pH, and nutrients, as well as plant diseases, can form patterns that persist over a long period of time. Weather patterns and management practices also contribute to temporal variations.<sup>4,5</sup> Conventional sampling and measurement strategies may not be able to reveal the variation at the right spatial and temporal resolution required for appropriate farm management. With the help of sensing and wireless technology, the physical, chemical, and biological attributes of soils, as shown in Figure 1, can be spatially and/or temporally monitored without the need for the farmer to be physically present or to take samples. This will enable the development of advanced smart agriculture systems that can aid in the more effective management of resources.<sup>4,6</sup>

While academic and industrial researchers have explored a wide range of sensors for agriculture, very few of these have transitioned into commercial products. This often relates to challenges in achieving stable measurements of a given soil parameter over extended periods of time. In the specific case of pH sensing, to the best of our knowledge no commercial sensor exists that allows for stable and continuous pH measurement in-ground and in real-time across multiple growing cycles. The following literature review focuses attention on pH sensing recently reported in the peer-reviewed scientific literature. This



**FIGURE 1** The major sensors for detecting health conditions of soil in smart agriculture. These sensors include soil moisture sensors, soil temperature sensors, soil pH sensors, soil nutrient sensors, soil pest/insect sensors, soil pollution sensors, and plant wearables. Reproduced with permission.<sup>4</sup> Copyright 2021, John Wiley and Sons.

helps identify the research gaps that need addressing on the path towards a stable and continuous pH sensor that can be used in-ground. Given that pH sensing is a broad research topic, this review will only cover the use of polymeric materials in pH sensing aiming at ultimately being deployed for extended periods in-ground. Polymers offer potential advantages for achieving stable long-term measurements such as corrosion stability albeit many other factors, such as (bio)fouling and mechanical abrasion, require addressing. Baghini and co-workers in 2013 reviewed polymer-based sensors for agriculture-related applications.<sup>7</sup> The majority of their review focused on non-pH related sensing, with the pH-relevant literature being approximately two decades old. Prior to discussion of the polymer related pH sensing literature, the following section on nutrient availability is provided to outline the motivation for developing pH sensors specific for in-ground applications.

## 2 | SOIL NUTRIENTS AND THE ROLE OF pH

An adequate supply of nutrients is essential for sustainable agricultural productivity. Seventeen elements are considered essential for plants and are shown in Table 1. They can be divided into macronutrients (more abundant) and micronutrients (less abundant). However, for plant uptake it is important that these nutrients are in appropriate chemical form in a soil solution. The amount of nutrients that are in the correct form are generally referred to as “available nutrients,” while the remainder are considered unavailable.<sup>8</sup>

Plants take up nutrients from the volume of soil surrounding, and influenced by, the roots: the rhizosphere. The rhizosphere is a complex and dynamic region, where root exudation, microbial activity, fertilization, and irrigation events can significantly alter nutrient availability in comparison to the “bulk” soil. This highlights the importance of knowing nutrient availability both spatially and temporally within the soil profile.<sup>10</sup>

Two soil properties that have been strongly linked with nutrient (and contaminant) availability are Eh (the redox potential within the soil) and pH (how acidic or basic the soil is). Eh-pH diagrams, also referred to as Pourbaix diagrams,<sup>11</sup> are composite functions of Eh and pH that depict the stability regions of different chemical species of an element in a solution based on thermodynamic rules. As can be seen in the example Pourbaix diagram for N in Figure 2, various species of N ( $\text{NO}_3^-$ ,  $\text{NH}_4^+$ ,  $\text{NH}_3$  and  $\text{NO}_2^-$ ) are stable for different pH and Eh conditions.<sup>12</sup> Soil oxidation–reduction processes are dynamic and highly complex, necessitating careful

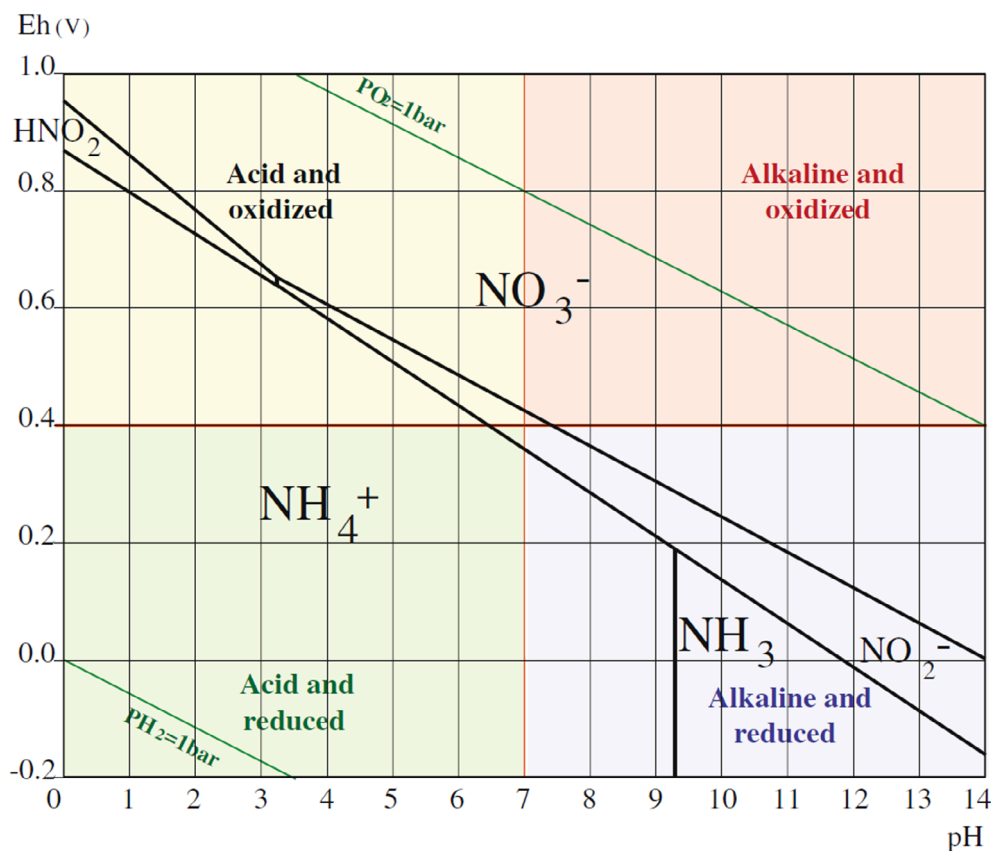
Nutrient		Concentration		
Classification	Element	Symbol	Relative <sup>a</sup> (atm)	Average <sup>b</sup> (wt.)
Macronutrient	Hydrogen	H	60,000,000	6%
	Carbon	C <sup>c</sup>	40,000,000	45%
	Oxygen	O	30,000,000	45%
	Nitrogen	N	1,000,000	1.5%
	Potassium	K	250,000	1.0%
	Calcium	Ca	125,000	0.5%
	Magnesium	Mg	80,000	0.2%
	Phosphorus	P	60,000	0.2%
	Sulfur	S	30,000	0.2%
Micronutrients	Chloride	Cl	3000	100 ppm
	Iron	Fe	2000	100 ppm
	Boron	B	2000	20 ppm
	Manganese	Mn	1000	50 ppm
	Zinc	Zn	300	20 ppm
	Copper	Cu	100	6 ppm
	Nickle	Ni	2	<1 ppm
	Molybdenum	Mo	1	<1 ppm

**TABLE 1** Essential Plant Nutrients and Their Relative and Average Concentration in Plants. Reproduced with permission.<sup>9</sup> Copyright 2020, Taylor & Francis Group.

<sup>a</sup>Based on atomic content relative to Mo being 1.

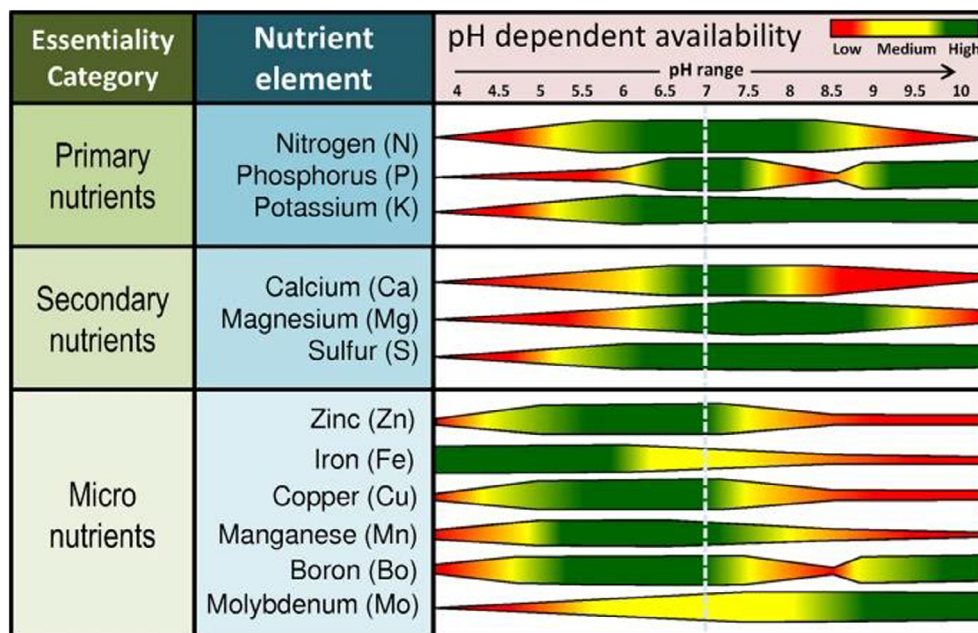
<sup>b</sup>Average concentration expressed on a dry matter weight basis.

<sup>c</sup>It is taken from air.



**FIGURE 2** Pourbaix diagram of nitrogen (N) representing the various forms of N in a 100  $\mu$ M solution at 25 °C as a function of Eh (in V) and pH. Reproduced with permission<sup>12</sup> under Creative Commons CC BY 2.0.

**FIGURE 3** Effect of soil pH on nutrient availability. Colors indicate availability of the elements. Green: available; yellow: low availability; red: not available. Reproduced with permission<sup>17</sup> under Creative Commons CC BY 4.0.



consideration of their kinetics in heterogeneous and ever-changing soils. Furthermore, because of the interactions between Eh and pH and microbial activity, these factors have a profound influence on biologically-driven changes in element speciation. Conversely, redox active elements, such as Fe, N, and S, also influence soil Eh and pH.<sup>12</sup>

Measuring and interpreting Eh is challenging due to its greater variability in both space and time compared to pH. There are also difficulties in measuring Eh in soils with high oxygen levels, and the fact that Eh is dependent on pH. As a result, reproducing and comparing Eh measurements from different studies is difficult. There are still issues that must be answered regarding the oxidation–reduction process and the most reliable approach to characterize it.<sup>12,13</sup>

In the context of soils, pH ranges from 1 to 10 when transitioning from acid sulphate soils to sodic soils.<sup>14</sup> When considering agricultural soils, the pH typically varies between 5 and 8.5 (summarized in the Supporting Information of Bai et al.).<sup>15</sup> Figure 3 gives an overview of how the availability for select nutrients changes with pH. For example, acidic soils lack available Ca, Mg, P, B, and Mo, while Fe and Mn are in forms available to plants. On the other hand, very alkaline soils often have deficiencies in available P, Fe, Cu, Zn, and B. When considering the pH range of 5 to 8.5 for agricultural soils, small changes in the soil pH may result in significant changes in plants' nutrient availability where a pH of 6.5–7 results in the most nutrients available for plant uptake.<sup>14,16,17</sup> Furthermore, at low pH, common soil constituent such as Al, Fe and Mn may increase in availability to levels that are toxic to plants. Therefore, soil

amendments to increase or reduce soil pH, such as lime or elemental S respectively, are often employed. In this case, the pH adjustment can take considerable time to occur and may also show significant gradients along the soil profile. Monitoring this change over time could be of significant advantage to farmers.

In addition to chemical properties, bacterial diversity and microbial activity have been shown to also be highly dependent on soil pH.<sup>18</sup> Consequently, as stated by Thomas, “soil pH is probably the most informative measurement that can be made to determine soil properties”.<sup>19</sup> This then leads to the question about effective ways to determine pH in the soil in a continuous and real-time manner.

Some sensing technologies that have been reported for measuring soil pH value include: optical (e.g., colorimetric or photometric methods), electrochemical (e.g., conductometric and potentiometric methods), and acoustic methods.<sup>4</sup> An important point to note here is that many of the reported studies focus on ex-situ pH measurement of soil samples extracted from the ground (and often diluted into a soil-water slurry). While several publications reference in-situ measurements, they define in-situ as being in the field rather than a laboratory (not in-ground). For example, Schirrmann et al. used an electrochemical sensor attached to a machine which collects soil samples and measures their pH on-the-go.<sup>20</sup> The focus on sensors for extracted soil samples highlights the challenges of doing true in-ground sensing. While standard laboratory pH electrodes work well in that environment, they do not translate well into use in-ground for many reasons. Liu et al. point out that in-ground measurements (for soil nitrogen sensing) using new sensor

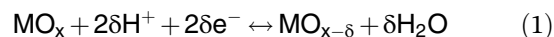
technology have rarely been attempted, as lab-based validation is unsuitable for practical implementation.<sup>21</sup> For the broader soil and plant sensor market, Yin et al. acknowledge the “immature reality of the sensor development and implementation” as one impediment to in-ground real-time sensing.<sup>4</sup> The ability to fabricate non-invasive, low cost, and reliable sensors is paramount to their use in-ground – which could be addressed through appropriate materials selection. Reliability of the sensor material needs to take into consideration stability to corrosion (or inability to corrode),<sup>22</sup> minimal interference from foreign analytes,<sup>23</sup> non-fouling (as considered for filtration membranes<sup>24</sup>), and mechanically robust to withstand the abrasive soil particles during installation, to name but a few. Polymers as the sensing material may address some of these challenges/limitations.

### 3 | APPROACHES TO SENSING pH IN SOIL USING POLYMERS

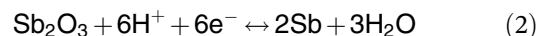
Soil is a harsh environment for materials, where processes such as corrosion and fouling inhibit their stability and performance. Polymers are one class of material commonly used in industrial applications due to their resistance to (some forms of) degradation – such as corrosion.<sup>25</sup> Polymers that can also reversibly and predictably respond to pH are thus attractive sensing materials. Conversely, polymers are relatively soft when compared to metals and ceramics and are therefore likely to be easily abraded by soil particles. In the following discussion, pH measurement will be discussed in the context of the two predominant sensor transduction methods; optical and electrochemical. Despite the value proposition for polymer-based soil pH sensors, there is limited peer-reviewed research in the space. A systematic search of the peer-reviewed literature yields 318 articles in the Web of Science Core Collection for the keywords of “agriculture”, “soil”, “pH” and “sensor”. When “polymer” is added to the search terms this reduces to just six articles. This section will provide a critical discussion about the challenges for using polymers in soil pH sensing.

Electrochemical pH sensors are the predominant type found in the general pH sensor literature. Conductometric and potentiometric methods are the most used techniques in this area. A potentiometric soil pH sensor consists of a sensing half-cell and a reference half-cell.<sup>4</sup> The sensing half-cell is typically an ion-selective electrode whose potential is directly related to the pH. The sensor design requires two separate electrodes, one coated with a pH-sensitive material and the other made of a material with a known electrochemical potential (such as Ag/AgCl) to act as a reference electrode. The pH values are obtained from the potential difference between the two electrodes.

The state-of-the-art in electrochemical solid-state pH sensors utilizes metal/metal oxide electrodes<sup>26</sup> that undergo the following general reaction as a function of pH<sup>27</sup>;



where  $\delta$  is an integer number related to the magnitude of reduction that occurs for a specific metal oxide;  $\text{MO}_x$  is the higher valence metal oxide and  $\text{MO}_{x-\delta}$  is the lower valence metal oxide (or pure metal when  $\delta = x$ ). Several publications explored the use of Sb as an electrode, where the redox equilibrium between Sb and  $\text{Sb}_2\text{O}_3$  is defined by the local pH.<sup>27,28</sup> This equilibrium is defined as,



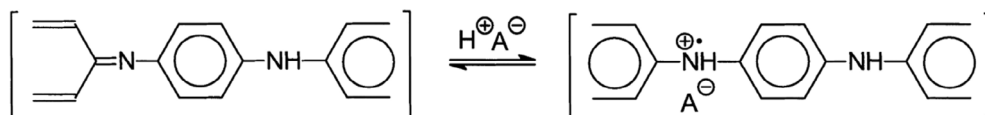
Comparison of Equations (1) and (2) reveals  $\delta = 3$  for the Sb/ $\text{Sb}_2\text{O}_3$  system. The redox equilibrium of Equation (2) can be monitored by measuring the potential of the Sb/ $\text{Sb}_2\text{O}_3$  electrode (E) relative to a known reference electrode ( $E_0$ ). The relative potential ( $E - E_0$ ) is then related to the pH via a simple linear relationship where the denominator of 59.16 mV/pH is the Nernst slope at room temperature (25 °C);

$$\text{pH} = -(E - E_0)/0.05916 \quad (3)$$

In general, metal oxides have been researched for pH sensing due to their unique properties like insolubility, stability, mechanical strength, and the ability to be made into miniature electrodes. However, metal-oxide pH sensors are limited by the interference caused by oxidizing and/or reducing agents in the sample solution. Most metal oxides are sensitive to redox species like ferricyanide ions present in the solution, which impacts on the pH measurements and leads to substantial errors, thus rendering them impractical for detecting low levels of analytes.<sup>29</sup>

In contrast to the metal/metal oxide approach, polymers such as poly(aniline) (PANi) that are responsive to pH undergo a protonation (deprotonation) reaction that typically leads to a formal positive (neutral) charge on the polymer.<sup>30</sup> In order to balance the positive charge, counterions (anions) would typically migrate into (out of) the polymer structure, leading to Schematic (1). Zea et al. exploit this for PANi and polypyrrole multilayer doped with polystyrene sulfonate (PSS) to yield a printed flexible pH sensor.<sup>31</sup> The concept was validated using commercial pH buffer solutions (with added KCl as an interfering salt) from 3 to 10, and compared to a conventional glass electrode pH sensor.

When considering Scheme 1, on a molecular level the specificity of the anion is unimportant other than its



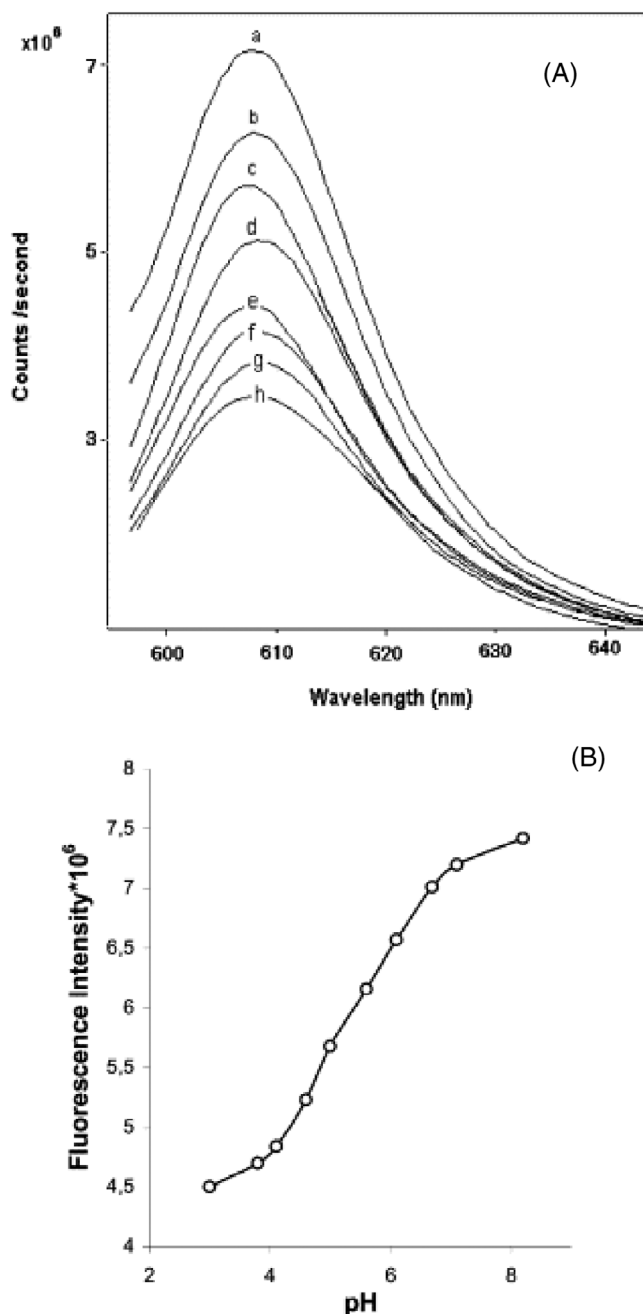
**SCHEME 1** Scheme of the protonation of PANI base with acids. Adapted with permission.<sup>32</sup> Copyright 1998, American Chemical Society.

valence. However, when considering a thin film or bulk volume of PANi consisting of crystalline and amorphous regions, the specific anion may influence the resultant PANi properties through morphology changes.<sup>33</sup> This highlights the potential for interference of anions in the sensing of pH changes.

Alternatively, doped conducting polymers such as PEDOT:PSS carry a formal negative charge due to the excess PSS (that is used to stabilize PEDOT:PSS dispersions in water).<sup>34,35</sup> This material has been studied for its ability to transport cations and shown in application for ion pumping (transport of cations such as  $\text{Ca}^{2+}$ ).<sup>35,36</sup> In this context, PEDOT:PSS has been considered as an individual candidate material for pH sensing,<sup>37</sup> or in a multilayer combination with PANi,<sup>38</sup> in wearable pH sensing applications. From these limited studies aspects of analyte interference was not investigated – where pH buffer solutions were used to observe the polymer's pH response. More generally PEDOT and other conducting polymers are known to interact with a wide range of ions in solutions, and hence highlighting their likely interference in the sensing of pH.

As an alternative to changes in electrical properties, several polymers are known to undergo morphology or structural changes based on pH. This is commonly described as a swelling or densification upon changes in pH, normally associated with the uptake or expulsion of water. Shrinkage of a hydrogel sensing layer results from a reaction to the analyte, leading to a change in electrical properties. A conductometric pH sensor was produced by coating an interdigital electrode with a pH-sensitive hydrogel membrane.<sup>4</sup> The reversibly pH-dependent swelling hydrogel can be used in such a model to fabricate reusable pH sensors. However, swelling based hydrogel requires a permanent aqueous environment which is not always the case in agricultural soils therefore requiring a soil solution to be prepared.

Optical fiber sensor technology, including for chemical sensing, has seen significant growth as a result of the rapid advancement, and widespread availability, of components developed for the telecommunications industry.<sup>39</sup> Fiber optic sensors have some advantages over electrochemical sensors, such as their light weight, small size, and corrosion resistance. They also have low attenuation, allowing for remote detection, and have no electrical power carrying requirements along the cable. Their



**FIGURE 4** (A) Dependence of emission spectrum of Schiff base III on pH in different pH buffers, [a] pH = 8.2, [b] pH = 7.1, [c] pH = 6.7, [d] pH = 6.2, [e] pH = 5.6, [f] pH = 5.0, [g] pH = 4.2, [h] pH = 3.2; (B) emission based sigmoidal response of Schiff base III to pH. Reproduced with permission.<sup>42</sup> Copyright 2004, Elsevier.

key differentiating functionality is the ability to perform multi-parameter and multi-point sensing, such as using time-domain detection for distributed sensing.<sup>40</sup>

Optical fiber pH sensors can be fabricated by chemically or physically immobilizing thin and proton-permeable layers that change the optical properties in response to pH. Moreover, some organic dyes have been reported to be used as absorbent or fluorescent pH indicators. Bargrizan et al. utilized bromocresol purple and phenol red dye as optical probes for the pH of soil-water slurries (with or without 10 mM CaCl<sub>2</sub>) with good correlation to the typical glass electrode pH instruments used in laboratories.<sup>41</sup> pH sensitive molecules and compounds can then be incorporated/encapsulated in polymer-matrix hosts. Hazneci et al. used plasticised poly(vinyl acetate) matrices to host Schiff bases that give an optical (fluorescent) response when they (de)protonate upon pH change.<sup>42</sup> As shown in Figure 4, the resultant polymer composite demonstrated repeatable optical pH measurement over a range of 3.0 to 7.8 (covering the expected range of soil pH in agriculture; see Section 2). The concept of measuring optical response with changing pH can be extended to the polymers themselves. Based on absorption both in the visible and near IR regions, certain conductive polymers can be used as pH sensing layers.<sup>43</sup> These conducting polymers are the same as those discussed above in regards to electrochemical pH sensing, relying on the fact that the electrical and optical properties are inherently linked via their distribution of density of states and electronic band structure.<sup>44</sup> An electropolymerized composite of PANi and PEDOT was shown to be sensitive for the pH range of 3.8–7.4, having optical response times of <100 s in the visible spectrum.<sup>45</sup> How studies such as these can be translated into the complex soil environment is the next challenge faced by researchers.

There have been several reasons for the limited success of past designs for optical pH sensors.<sup>46</sup> One of the crucial factors in achieving an optimal sensor is the use of appropriate pH indicators and their efficient immobilization. These factors play a significant role in determining the sensor's lifespan and signal stability which have not been successfully achieved in previous designs. Research has demonstrated that ineffective immobilization of the dye can cause the dye to leach, resulting in the calibration of the probe drifting over time. This phenomenon ultimately leads to a decline in the probe's ability to accurately sense and measure pH level. Nguyen, Sun and Grattan highlight that covalent binding should result in robust and stable pH sensors when compared to absorption, entrapment, or layer-by-layer (LbL) electrostatic self-assembly.<sup>47</sup> Covalent binding often requires specialized indicator molecules containing silane functional groups to provide strong attachment to the substrate and hence stop desorption during normal operation.

The distributed or multi-point pH sensing using optical fiber technology has great potential for achieving soil depth profile monitoring. This can be achieved using time-domain sensing, where a pulse of light is sent into an optical fiber coated with a fluorescent indicator sensitive to pH and the return time for the pulse directly correlates to the position along the optical fiber.<sup>48</sup> In this case the spatial resolution is limited by the fluorophore's lifetime, corresponding to approximately 1 m. Improved spatial resolution with millimeter scale can be achieved, at the expense of system complexity, using frequency-domain optical measurements, such as demonstrated by Lu et al. with a sol-gel coated optical fiber pH sensor.<sup>49</sup> In all cases the number of sensing points was limited to less than 10, likely due to optical losses, which would be even more challenging in a soil monitoring system. The demonstrations have primarily been limited to sol-gel coatings or covalent attachment to the optical fiber,<sup>50</sup> but these techniques can be equally applied to polymer-based coatings. For example, Lopez Aldaba et al. dip-coated PANi onto a tilted fiber Bragg grating for pH sensing.<sup>51</sup> They measure the transmitted amplitude in the 1510 to 1590 nm wavelength range from the fiber as a function of PANi thickness and pH between 2 and 12. The pH of a NaCl solution was modified by using HCl or NaOH. While being preliminary in nature, this serves as a starting point towards a multi-point sensing for spatial sensing of soil pH.

## 4 | OUTLOOK

Efficient management of agricultural land is critical to ensuring that the increasing global population has secure food supply. One aspect of maintaining fertile and productive agricultural land involves monitoring soil conditions to keep farmers well informed. This puts sensors at the core of mechanisms to help farmers improve yield and productivity of their crops. The soil pH is one parameter that has great impact on crops, yet real-time in-ground pH sensors for continuous operation do not commercially exist. A challenge to realizing this comes from the harsh environment the soil provides – namely wet, salty conditions with various biological material, to name a few. This means that the materials used to fabricate a sensor need to be stable and robust within the soil for extended periods of time. Polymers present as viable options for use in pH sensors because they do not corrode. Polymers that can respond to pH are therefore of interest to researchers looking to develop soil pH sensors. The response to pH may be electrical or optical and will depend on the chosen transduction method for the final sensor.

On the path towards deployable sensors there are two identified areas of research: (i) investigating polymers in simulated soil environments, and (ii) studying other

properties of the polymers. In the context of (i), there is a need to understand the interference of common ions found in the soil on pH sensing. Many of the research publications for pH sensing utilize buffer solutions or simple acid/base solutions to undertake their studies. As highlighted in Table 1 there is a wide range of other elements/ions that may reside in the soil – and could interfere with the pH sensing mechanism. Once this has been addressed there are other polymer properties that become important. To address (ii), aspects such as (bio)fouling, abrasion resistance, and temperature stability become important. These aspects will influence the long-term performance/drift of the sensor, which is critical for agricultural applications where the sensing is remote and routine calibration is difficult.

Researchers will need to take a wholistic view to developing polymers for pH sensing in soil. This requires a concerted effort to develop the polymers for testing within realistic soil environments and explore how the polymer responds to other conditions of the soil.

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