Advanced monitoring of hydroponic solutions using ion-selective electrodes and the internet of things: a review

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3 authors:

Amina Richa
Université de Khemis Miliana

Meriem Fizir
Khemis Miliana University

Sami Touil
Université de Khemis Miliana

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Corresponding Author	Family Name	**Richa**
	Particle	
	Given Name	**Amina**
	Suffix	
	Division	Research Laboratory of Agricultural Production and Sustainable Development of Natural Resources
	Organization	University of Djilali Bounaama Khemis Miliana
	Address	Ain Defla, Algeria
	Phone	
	Fax	
	Email	a.richa@univ-dbkm.dz
	URL	
	ORCID	http://orcid.org/0000-0002-1353-4517
Corresponding Author	Family Name	**Fizir**
	Particle	
	Given Name	**Meriem**
	Suffix	
	Division	Laboratoire de Valorisation des Substances Naturelles
	Organization	Université Djilali Bounaâma
	Address	Khemis Miliana, Algeria
	Phone	
	Fax	
	Email	meriem.fizir@univ-dbkm.dz
	URL	
	ORCID	
Corresponding Author	Family Name	**Touil**
	Particle	
	Given Name	**Sami**
	Suffix	
	Division	Research Laboratory of Agricultural Production and Sustainable Development of Natural Resources
	Organization	University of Djilali Bounaama Khemis Miliana
	Address	Ain Defla, Algeria
	Phone	
	Fax	

Email	s.touil@univ-dbkm.dz
URL	
ORCID	

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Abstract

Hydroponic cultivation is a promising alternative that could address environmental and food issues. Hydroponic cultivation is sustainable because it allows farmers to reduce the amount of fertilizers and water. Moreover, accurate monitoring of nutrient levels allows to optimize crop growth. For that, researchers have designed ion-selective electrodes as sensors and internet of things for precise monitoring. Here we review the application of ion-selective electrodes in monitoring hydroponic solutions, with focus on parameters, sensitivity, accuracy and internet of things technologies. We found that the targeted concentrations of nitrate and potassium are successfully maintained with errors of less than 10 mg L^{-1} by using polyvinyl chloride membrane based on ion-selective electrodes. Calcium electrodes show less sensitivity in detection of calcium ions in hydroponic solution. Regular calibration sequence solved the stability issue of ion-selective electrodes. Compared to carbon nanotube-based ion-selective electrodes, the best results for sensing nitrate in hydroponic solution were obtained with polyvinyl chloride ion-selective membranes.

Keywords (separated by '-'): Hydroponic system - Nutrient solution - Monitoring nutrient solution - Internet of Things - Ion-selective electrodes

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REVIEW

Advanced monitoring of hydroponic solutions using ion-selective electrodes and the internet of things: a review

Amina Richa[1] · **Meriem Fizir**[2] · **Sami Touil**[1]

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Abstract
Hydroponic cultivation is a promising alternative that could address environmental and food issues. Hydroponic cultivation AQ1 is sustainable because it allows farmers to reduce the amount of fertilizers and water. Moreover, accurate monitoring of nutrient levels allows to optimize crop growth. For that, researchers have designed ion-selective electrodes as sensors and internet of things for precise monitoring. Here we review the application of ion-selective electrodes in monitoring hydroponic solutions, with focus on parameters, sensitivity, accuracy and internet of things technologies. We found that the targeted concentrations of nitrate and potassium are successfully maintained with errors of less than 10 mg L^{-1} by using polyvinyl chloride membrane based on ion-selective electrodes. Calcium electrodes show less sensitivity in detection of calcium ions in hydroponic solution. Regular calibration sequence solved the stability issue of ion-selective electrodes. Compared to carbon nanotube-based ion-selective electrodes, the best results for sensing nitrate in hydroponic solution were obtained with polyvinyl chloride ion-selective membranes.

Keywords Hydroponic system · Nutrient solution · Monitoring nutrient solution · Internet of Things · Ion-selective electrodes

Abbreviations

BP-ANN	Back-Propagation Artificial Neuronal Network
HPLC	High-performance liquid chromatography
KTpClPB	Potassium tetrakis(4-chlorophenyl)borate)
KTPhB	Potassium tetraphenylborate.
MQTT	Message queuing telemetry transport
NPOE	2-Nitrophenyl octylether
TDDA	Tetradodecylammonium nitrate
RMSE	Relative mean standard error

✉ Amina Richa
a.richa@univ-dbkm.dz

✉ Meriem Fizir
meriem.fizir@univ-dbkm.dz

✉ Sami Touil
s.touil@univ-dbkm.dz

[1] Research Laboratory of Agricultural Production and Sustainable Development of Natural Resources, University of Djilali Bounaama Khemis Miliana, Ain Defla, Algeria

[2] Laboratoire de Valorisation des Substances Naturelles, Université Djilali Bounaâma, Khemis Miliana, Algeria

Introduction

Hydroponics or soilless farming is cultivation technique without soil where roots are either supported with grow substrate such as perlite, rock and clay or suspended in water which is supplemented minerals nutrients (Maucieri et al. 2019; Han et al. 2020b). In this technique, if the nutrient solution is recirculated, then it is referred to as closed loop system (Mohammed 2018) (Xydis et al. 2017; Nandwani 2018). In our last review paper, we have declared that reusing hydroponic solutions which is referred to as closed system has gained significant research attention due to its positive effect on environment, e.g., prevent pollution of water and soil induced by open system and economics because in closed system the use of water and fertilizers will be reduced (Richa et al. 2020). However, reusing the nutrient solution in closed systems leads to the increase in electrical conductivity and appearance of imbalance in nutrient ratios which can decrease crops yield and affect their quality (Cho et al. 2017). Hence, accurate monitoring the concentration of the nutrients within the hydroponic solutions is needed before its reuse to optimize the composition of regenerated nutrient solution and maximize plant production (Jung et al. 2015).

Conventional methods for monitoring hydroponic nutrients have been based on controlling the pH, electrical conductivity and temperature of the nutrient solution (Ahn and Son 2011; Domingues et al. 2012). Using these approaches, the nutrient solution is delivered automatically to the hydroponic solutions tank according to the decrease in the electrical conductivity of hydroponic solutions in order to maintain its quality (Cho et al. 2018; Jung et al. 2019). The main drawbacks of this strategy are the limited information provided by electrical conductivity data because it did not provide the real concentration of individual ions, thereby limiting its capability to successfully replenish the targeted ions needed for higher crop growth (Kim et al. 2013, 2017). In this regards, increasing time use of the nutrient solution and improving efficiency of fertilizer exploitation may be possible through precise quantification of individual nutrient concentrations for automatic corrections to each deficient nutrient (Cho et al. 2017). The need for such nutrient management has led to the application of ion-selective electrode as smart sensing tools to automatically monitor hydroponic nutrients (Jung et al. 2019; Xu et al. 2019, 2020; Han et al. 2020a). Ion-selective electrodes possess several advantages over spectroscopic techniques, such as rapid response, direct measurement of the analyte, low cost, and portability (Kim et al. 2017; Cho et al. 2018). On the other hand, computerization and connectivity along with the implementation of internet of things in the farming sector facilitate and improve the monitoring process of hydroponic nutrient solution based on ion-selective electrodes (Mehra et al. 2018). Significant reduction of employment, a more robust management of the process, and using computer capabilities to make data-driven decisions are the main expected benefits of smart automation (Yanes et al. 2020). Consequently, data and analysis of hydroponic solution status could be gathered by the farmers based on the information, and they can look after the hydroponic crops through Android Smartphone (Vidhya and Valarmathi 2018). In this review, research methodology is discussed in Sects. 2 and 3 introducing the parameters such as electrical conductivity, temperature, pH and macronutrients concentrations that need to be controlled. The application of ion-selective electrodes for detection hydroponic nutrients is summarized in Sect. 4 where the sensitivity and accuracy of these devices were discussed as well. Internet of things technologies used for monitoring hydroponic solutions are reviewed in Sect. 5, while the last section discusses direction of the future contributions and concluding remarks. Monitoring strategies of hydroponic nutrients solution are summarized in Fig. 1.

Review strategy

An extensive search through a diverse multidisciplinary online database and academic platforms, such as Science Direct, Springer, Google Scholar, ResearchGate and other Scopus indexed journals has been carried out in the collection of research papers that are published on monitoring hydroponic nutrient solution. In such massive research

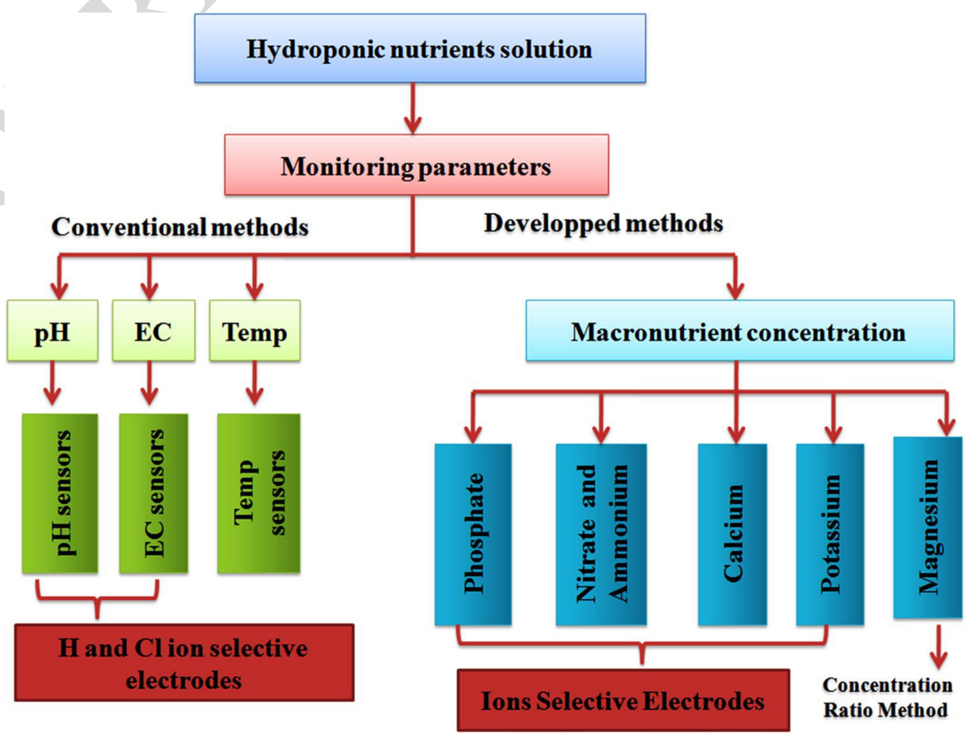

Fig. 1 Monitoring strategies of hydroponic nutrients solution. EC: Electrical Conductivity; Temp: Temperature

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platforms, we found several research papers related to monitoring and control hydroponic solution. Consequently, we have focused on recent high-ranking journals and recently published conference papers for selecting relevant papers for this review. The following keywords are used for searching articles: automated hydroponic system, monitoring hydroponic nutrient solution and internet of things. The scientific articles were selected carefully and summarized to ensure the presentation of the ideas. Figure 2 shows the number of papers published till 2020 on automated hydroponic systems using ion-selective electrodes and internet of things. Most of the selected articles were published between 2016 till date, with the years 2018–2020 recording the highest numbers of published papers cited.

Nutrient solution and monitoring parameters

A nutrient solution used in hydroponics for plant growth is composed mainly of inorganic ions dissolved in aqueous solution from soluble salts of essential elements (Kozai 2018). Nitrogen, phosphorus, potassium, calcium, magnesium and sulfur are the basic components in nutrient solution which are supplemented with the following micronutrients: iron (Fe), magnesium (Mn), zinc (Zn), boron (B), copper (Cu) and molybdenum (Mo) (Chhipa 2017; Mohammed 2018; Sharma et al. 2020). Salts like KNO_3, $Ca(NO_3)_2$, $KHPO_4$, $MgSO_4$, and a little Fe-compound are the main constitutions of the nutrient solution used to cultivate plants (Sonneveld and Voogt 2009a).

The nutrient solution in hydroponic system is usually controlled by electrical conductivity, pH, temperature and quantifying the macronutrients in cultivation fields, e.g., quantitative determination of nitrates, ammonium, phosphate, calcium, potassium and magnesium ions. In this section, each parameter is presented and its role in hydroponic systems is explained. Then, a practice used by researchers for measuring and controlling the above cited ions is explained in the following sections.

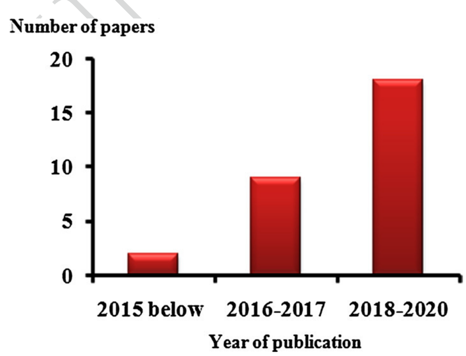

Fig. 2 Numbers of papers according to publication year

pH

In hydroponics, plants feed by ion exchange which makes the measurement of the pH of the nutrient solution essential to obtain its relative concentration of positive hydrogen ions, because in this way we will know what nutrient the crop requires (Mohammed 2018). The nutrient deficiencies symptoms will develop if the pH is higher or lower than the recommended range for individual plants (Putra and Yuliando 2015). In general, the most appropriate pH values of the nutrient solution for the growth of crops range between 5.5 and 6.5 (Tyson et al. 2007; Maucieri et al. 2019; Wada 2019). For example, pH value of the nutrient solution for the cultivation of the cucumbers, strawberries and onion ranges from 5.8–6, 5.5–6.5 and 6.5–7, respectively (Hussain et al. 2014; Martínez et al. 2017). To increase the pH, NaOH or KOH solution is recommended to be used, while H_2SO_4, H_3PO_4 or HNO_3 can be used to decrease the pH (Signore et al. 2016; Kozai 2018). AQ2

Electrical conductivity

The electrical conductivity value indicates total ionic concentration and the strength of the nutrient solution. However, conductivity data did not provide a specific ion's concentration (Kozai 2018). Along with pH measurement, electrical conductivity data in the hydroponic component can be used as an estimator of the water nutrient content (Mohammed 2018). Thus, to have insight into nutrient consumption, maintain consistency with each crop cycle, and guarantee maximization of nutrients use without excess use of fertilizers, daily measurement of electrical conductivity is necessary (Yanes et al. 2020). The crop species and environmental conditions are the factors that determine the ideal electrical conductivity. Generally, the appropriate electrical conductivity range for hydroponic systems is between range 1.5 and 2.5 ds m^{-1} (Sonneveld and Voogt 2009b; Kozai 2018). In fact, the electrical conductivity of the nutrient solution is changed due to the plants nutrient uptake from the solution. The absorption of the nutrient will be affected if the electrical conductivity is higher than the particular range. In this case, the electrical conductivity is monitored and adjusted by adding fresh water. By contrast, a determined quantity of the nutrient should be added if the electrical conductivity is too low to prevent crops yield decrease (Putra and Yuliando 2015).

Temperature

Besides pH and electrical conductivity, nutrient solution temperature also plays important role in hydroponic. The solubility of the nutrients in the solution and its adsorption by the plants root zoon is affected by the temperature (Trejo-Téllez and Gómez-Merino 2012). Plant roots absorb nutrient well when they submerged in 20–25 °C for at least 12 h. The chlorophyll level was found to be reduced when the temperature of hydroponic solution was out of the range from 15–25 °C which will impact plants growth (Kozai 2018).

Nitrogen

Nitrogen (N) is a key indispensable element for plant growth, and a constituent of amino acids, proteins and chlorophyll (Marschner 2011; Tripathi et al. 2014). Nitrogen is the major nutrient responsible for vigorous growth and luscious green coloration. Pale green color of the older leaves, growth retardation and senescence advance are the main symptoms of nitrogen deficiency (Ghorbani et al. 2008). In contrast, excess of nitrogen promotes shoot development (Ohyama 2010). The major inorganic forms of nitrogen are *nitrate* and *ammonium* (Mokhele et al. 2012).

Nitrates

Nitrate (NO_3^-) is the main form of nitrogen absorbed by plants. NO_3^- is taken up through energy expenditure and transport via transpiration (Wen et al. 2019). The roots uptake nitrates quickly (Li et al. 2013). The latter is very highly movable inside plants and also depends on the growth phase of a crop and can be stored without toxic effects (Marquez et al. 2007). Low nitrate condition led to the growth inhibition of hydroponically cultivated wheat (Jiang et al. 2017). Another experimental research on the apple leaves under low nitrate conditions showed that nitrate deficiency affects nitrogen assimilation and chlorophyll synthesis (Wen et al. 2019).

Ammonium

Low quantity of ammonium (NH_4^+) can be uptake by plants root. By contrast, high quantities storage of this element exert toxic effects (Li et al. 2013). Increasing the proportion of NH_4^+ in hydroponic nutrient solution depressed dry matter accumulation, shoot extension, and branching of avocado plants (Lobit et al. 2007). Similarly, cabbage growth in hydroponic system was reduced by 87% when the proportion of ammonium to nitrate in the nutrient solution was more than 75% and it was suggested that the NH_4^+/NO_3^- ratio of 0.5:0.5 is the best for shoot development in cabbage plants (Zhang et al. 2007). Same findings were reported with tomato plants (Ganmore-Neumann and Kafkafi 1980). Therefore, the precise determination of nitrate and ammonium concentration in hydroponic solution is necessary for providing an equilibrium set of nutrients to plants within an appropriate range.

Phosphate

The phosphate ion (PO_4^{3-}) is considered as one of the primary macronutrients (Ryan et al. 2001; García y García et al. 2003; Rowley et al. 2012; Morgan and Connolly 2013) which means one of the most necessary macronutrient (Geilfus 2019; Mahmoud et al. 2020). The formation of high-energy compounds required for plant metabolism is assured by the availability of phosphate in nutrient solution (Moonrungsee et al. 2015). This component stimulates roots growth, the rapid development of buds and flower amount. Low substrate temperature, e.g., less than 13 °C, or high pH value, e.g., more than 6.5, leads to the decrease in the absorption of phosphate which can lead to deficiency signs (Han et al. 2020c). For example, supply the wheat plants with a low phosphate level led to reduce the growth rate and decrease the phosphate concentration in the shoots (Barrett-Lennard et al. 1982). Phosphate-deficiency treatment on tomato growth inhibited biomass growth of all organs except the roots and the leaf photosynthesis and diameter of fruit and stem were depressed (Fujita et al. 2003). According to the above outcomes, monitoring PO_4^{3-} of hydroponic nutrient solution is crucial to achieve a higher plant production.

Potassium

Potassium (K) is regarded as an essential element to all plant life (Tripathi et al. 2014). Potassium is a regulator of several vital functions such as resistance to disease. In addition, physiological processes such as photosynthesis needed a determined quantity of potassium in nutrient solution (Zlatev and Lidon 2012). Yellowish spots that very quickly necrotize on the margins of the older leaves is the first symptoms of deficiency (Geilfus 2019; Tran et al. 2019). Several researches have reported a reduction in leaf area under low K^+ concentration in hydroponic nutrient solution (Hafsi et al. 2014). On the other hand, potassium deficiency resulted in crops yield slow-down. However, after potassium supply resumption, the plants could recover a growth pattern (Pujos and Morard 1997). Photosynthetic gas exchange and pigment contents of *Sulla carnosa* plants were found to be affected by low K^+ conditions and growth of vegetative organs was significantly reduces by 50% (Hafsi et al. 2016). Hence, the management of potassium in hydroponic nutrient solution is of great importance to maximize the crop yield.

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Calcium

Calcium (Ca) plays an important role in plant life cycle (Sajid et al. 2020). The plant development is considerably influenced by the concentration of calcium ions within hydroponic solution (Tripathi et al. 2014). Membrane permeability, cell wall formation, cell division and extension which are growth plant activities needed a determined quantity of calcium. In addition, plant resistance to fungal attacks and bacterial infections is increased by the availability of calcium ions in nutrient solution (Maucieri et al. 2019). Imbalanced concentrations in the nutrient solution can result in reduced plant growth (Bamsey et al. 2014). Recently, various researchers found that blossom-end rot in tomato was primarily associated with calcium deficiency (Jae-Won et al. 2018; Hagassou et al. 2019; Tran et al. 2019).

Magnesium

Magnesium (Mg) is the most abundant divalent cation in cytosol of plant cells and plays a significant role in numerous physiological processes (LI et al. 2018). Magnesium plays a main function in plant photosynthesis because it is a central atom of chlorophyll (Tripathi et al. 2014). Consequently, sings of deficiency are yellowing between leaf veins and internal chlorosis of the basal leaves due to the degradation of the chlorophyll content (Maucieri et al. 2019). In additions, magnesium deficiency restricts plant growth and dry matter partitioning between shoots and roots (Guo et al. 2016). However, an adequate supply of Mg makes the plant healthy (Hermans et al. 2010).

The overall view of the above-mentioned facts bring evidence that management of nutrient solution in hydroponic system is of high importance to maximize the crops growth and to prevent the nutrient deficiency symptoms. The aforementioned target ions source used in nutrient solution is depicted in Table 1.

Monitoring and management of nutrient solution technologies

Conventional electrical conductivity-, pH- and temperature-based nutrient management systems

In hydroponic cultures, the pH, electrical conductivity and temperature of the nutrient solution should be measured every day. Several studies had been made by researchers to monitor these parameters for different crops. Electrical conductivity and pH of the solutions are usually controlled to assess the nutrient status of hydroponic solution used in plant production (Cho et al. 2018). Basic system for controlling the nutrient solution in closed hydroponics is presented in Fig. 3.

Typically, electrical conductivity and pH sensors use analog electrical conductivity meter and pH meter, respectively (Wada 2019). The effect of the temperature of the root zone on the growth and chlorophyll levels of plants in hydroponic systems was studied by various researchers. For example, in hydroponically lettuce cultivation, the optimal absorption of nutrients by the roots occurs at a solution temperature between 15 and 25 °C. Beyond this range, there is a risk to reduce the level of chlorophyll (Ginting 2008).

The conductivity and pH were monitored automatically throughout 24 h during the whole cycle of hydroponic

Table 1 The source of monitored ions in hydroponic nutrient (De Marco and Phan 2003; Kim et al. 2013; Rius-Ruiz et al. 2014; Jung et al. 2015; Cho et al. 2017, 2018; Jung et al. 2019; Xu et al. 2020)

Target ions	Ions salts
Nitrates, NO_3^-	Calcium nitrate, $Ca(NO_3)_2{\cdot}4H_2O$ Potassium nitrate, KNO_3 Ammonium nitrate, NH_4NO_3 Sodium nitrate, $NaNO_3$
Ammonium, NH_4^+	Ammonium nitrate, NH_4NO_3 Ammonium sulfate, $(NH_4)_2SO_4$ Ammonium dihydrogen phosphate $NH_4H_2PO_4$
Phosphate ion PO_4^{3-}	Sodium dihydrogenphosphate, NaH_2PO_4 Monopotassium phosphate, KH_2PO_4
Potassium K^+	Potassium nitrate, KNO_3 Potassium sulfate, K_2SO_4
Calcium, Ca^{2+}	Calcium nitrate tetrahydrate, $Ca(NO_3)_2{\cdot}4H_2O$ Calcium dichloride, $CaCl_2$
Magnesium Mg^{2+}	Magnesium sulfate heptahydrate, $MgSO_4{\cdot}7H_2O$ Magnesium nitrate hexahydrate, $MgNO_3.6H_2O$ Magnesium chloride, $MgCl_2$

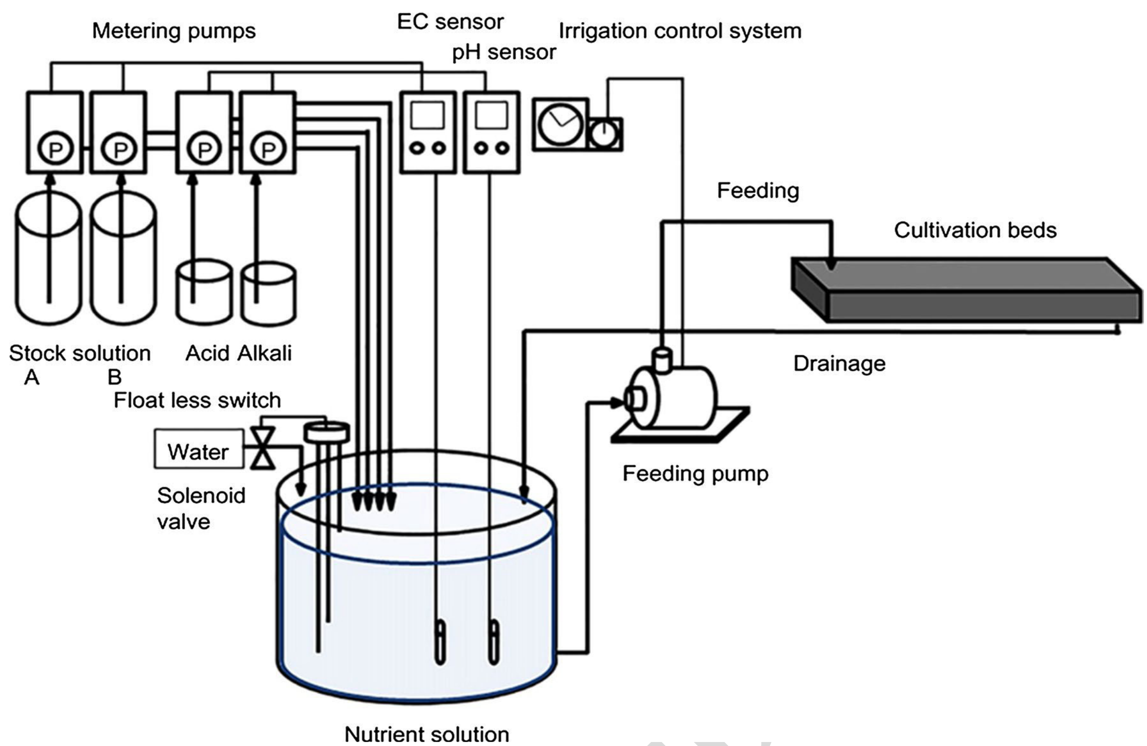

Fig. 3 Basic system for controlling the nutrient solution in closed hydroponics. EC sensor: electrical conductivity sensors. Reprinted with permission of Elsevier from (Wada, 2019)

lettuce production in greenhouse by using online system-based software and webcam where the pH and conductivity of the nutrient solution were fixed efficiently (Domingues et al. 2012). A Microsoft Office Excel SOLVER tool was efficiently calibrating electrical conductivity with 1.5 dS m^{-1} of nutrient solutions in the nutrient film technique hydroponic cultivation of lettuce (de Azevedo et al. 2018). The effect of different electrical conductivity levels in nutrient solution on yield and fruit quality of tomatoes was investigated by Eltez et al. (2000). The authors reported that the growth and average fruit weight reduced with increasing salinity and the highest total yield were obtained at electrical conductivity of 2.0 dS m^{-1}. However, fruit number was not affected by salinity (Eltez et al. 2000). Similar findings were found by Signore et al. (2016). Similarly, an experimental research about the effect of electrical conductivity on the growth of Gerbera cultivated in soilless culture showed that higher electrical conductivity level increased nutrient uptake and plant development (Paradiso et al. 2003). The electrical conductivity of the nutrient solution used to grow blueberries in hydroponics was maintained at 0.8–1.0 dS m^{-1}, and pH was automatically controlled between 4.5 and 4.8. In this research, the sulfuric acid solution was used to adjust the pH of the nutrient solution (Voogt et al. 2012). But, using water low in HCO_3 is suggested to avoid acidification process. More recently, electrical conductivity monitoring of nutrient solution used for the cultivation of saffron enhanced corm yield by a 20% and the highest number of corms·m^{-2} was associated with an electrical conductivity of 2.5 dS m^{-2} (Salas et al. 2020).

From the above facts, we can conclude that monitoring the electrical conductivity of nutrient solution is of high importance to optimize the crop production. However, such conventional system cannot provide sufficient details about ion imbalances induced by nutrient uptake in plants and the drainage ratio in plant cultivation beds because electrical conductivity provides the total composition of nutrient ions (Bamsey et al. 2012a). As proof of concept, tomato hydroponic research outcomes indicated that the variation in the dosage of NO_3^-, SO_4^{2-}, Mg^{2+}, Ca^{2+}, and K^+ in nutrient solution was equal to the electrical conductivity variation. However, in the cases of PO_4^{3-}, Na^+, Cl^-, dissolved Fe and Mn, Cu^{2+}, and Zn^{2+}, variation did not correspond with that of electrical conductivity (Lee et al. 2017).

To manage the imbalances in nutrient ratios in electrical conductivity-based systems, periodical adjustment of recycled nutrient solutions was suggested, and adequate range was determined. For example, for paprika plants cultivated in rockwool in a closed system, the nutrient solution is renewed every 4 to 8 weeks and electrical conductivity with pH should be adjusted every 3 days (Ko et al. 2013, 2014). For sweet pepper, proper renewal period was found to be

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each 4 weeks (Ahn and Son 2011). The nutrient solution to grow strawberry was replaced every 8 weeks and 2 weeks for basil with daily adjustment of electrical conductivity and pH (Sarooshi and Cresswell 1994; Solis-Toapanta et al. 2020). The dependence of the adjustment period on crop varieties limited the application of the above strategy. Consequently, ion-selective electrode technology along with electrical conductivity and pH sensors which are able to constantly control the plant's nutrient uptake and ideally readjust the needed nutrient in the case of deficiency so that the system is never imbalanced is a very well suited approach for nutrient solution monitoring (Knight and Lefsrud 2017).

Ion-selective and cobalt electrodes-based nutrient management systems

Most of the electrochemical techniques used to determine hydroponic nutrient levels are based on the use of an ion-selective electrode (Bamsey et al. 2012b). Electrodes are any substance that is good conductor of electricity (Richter et al. 2008). Ion-selective electrodes are relatively simple membrane-based potentiometric tools that are able to precisely measure the concentration of ions in nutrient solution. In other words, the term "Ion-selective electrodes" means that ion-selective electrodes are capable of differentiating between ions (Mikhelson 2013) where a voltage or current output in response to the activity of selected ions can be generated by ion-selective electrodes (Kim et al. 2009).

The nature and membrane materials compositions used to construct the electrode are the selectivity keys of these devices. Electrodes sensors in Table 2 generally incorporate the polymer membrane. This later is embedded with an Ionophore, along with an anionic site. The anionic site balance charge within the polymer matrix and the ionophore part possess high selectivity toward target ion of interest (Koryta 1990). The comparison of the electrical potential indicated at the solution/sensor interface with that indicated at a reference electrode is the principal of electrode function (Bamsey et al. 2012b). At present, the sensor and reference electrodes are placed within one sensor body which is referred to as *modern electrodes*. The sensor and reference relationship is illustrated in Fig. 4. Typically, when the sensor device is immersed into a bulk nutrient solution containing specific ions, the ion-selective materials will selectively bind the desired ions until an electrochemical equilibrium is attained. At this point, a charge which is proportional to the amount of ionophore develops at the interface between the ion-selective material and the internal electrolyte solution of the sensor electrode. Linking the sensor or working electrode with the reference electrode which is commonly made from Ag/AgCl creates a small electric current generated by an electrochemical cell. This later can be measured using a sensitive meter. As the electrical potential at the reference electrode is relatively constant and the sensor responds selectively to desired ions, the electrical potential at the solution and sensor interface can be correlated with the activity of the analyte in the nutrient solution (Grieshaber et al.

Table 2 Summary of ion-selective electrodes components in the literature

Ion-selective electrodes	Ionophore[a]	Plasticizer[b]	Matrix[c]	Additive[d]	Inner solution[e]	References
NO_3	TDDA	NPOE	PVC	-	0.01 M $NaNO_3$ + 0.01 M NaCl	(Jung et al. 2015; Kim et al. 2017; Cho et al. 2018; Fukao et al. 2018)
	TDDA	DBF	PVC	KTpClPB	-	(Vardar et al. 2015)
K	Valinomycin	Dos (Bis)	PVC	KTpClPB	0.01 M KCl	(Jung et al. 2015; Cho et al. 2017, 2018; Kim et al. 2017)
	Valinomycin	NPOE	PVC	KTPhB	0.1 M KCl	(Xu et al. 2019)
H	Hydrogen ionophore II	NPOE	PVC	KTpClPB	pH 5.6 (1 M citric acid + 2.73 M NaOH + 0.01 M NaCl)	(Kim et al. 2017)
Ca	Calcium ionophore II	NPOE	PVC	KTpClPB	0.01 M $CaCl_2$	(Kim et al. 2013)
Mg	Magnesium Ionophore I	NPOE	PVC	KTpClPB	0.01 M $MgCl_2$	(Erne et al., 1980)
	Magnesium Ionophore III	Chloroparaffin	PVC	KTpClPB	0.01 M $MgCl_2$	(Müller et al. 1988)

[a]*TDDA* tetradodecylammonium nitrate

[b]*DOS* bis(2-ethyhexyl) sebacate, *DBF* dibutyl furmarate, *NPOE* = 2 nitrophenyl octylether

[c]*PVC*: high-molecular-weight polyvinyl chloride

[d]*KTpClPB*: potassium tetrakis(4-chlorophenyl)borate), *KTPhB*: potassium tetraphenylborate

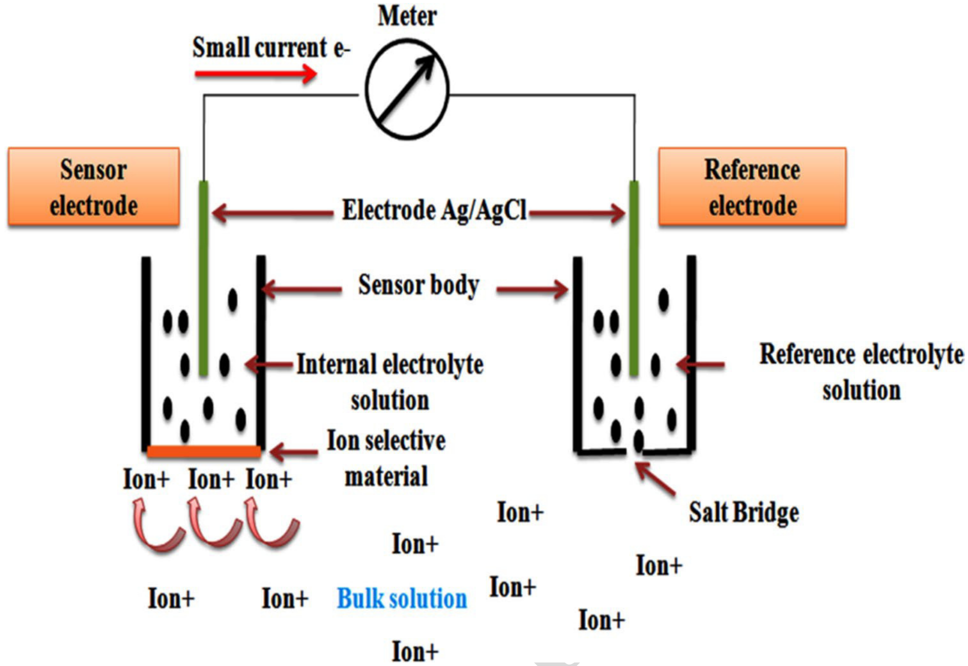

Fig. 4 Illustration of the standard components of an ion-selective electrode system used for activity measurements in solution

2008; Bamsey et al. 2012b). The potentiometric ion-selective electrode for an ion (i) is given by Nikolsky Eisenman equation (Bailey et al. 1988). This semiempirical equation is exploited to define.

The potentiometric selectivity coefficients which are used to express, generally, the ability of the electrode to distinguish between the desired ion (i) and the interfering one (j) (Deyhimi 1999).

Advantages of ion-selective electrodes over other spectrophotometric techniques include uncomplicated process, direct quantification of analyte, small size, sensitivity, and low cost (Kim et al. 2011). Liquid and polymer membranes allow for the selective quantification of different ions, e.g., NO_3^-, Na^+, K^+, Ca^{2+} and Ag^+ (Meyerhoff and Opdycke 1986). On the other hand, as an alternative to polymer membranes-based ion-selective electrode for phosphate sensing, cobalt-based electrodes have been prepared and have exhibited sensitive responses to phosphate ions and satisfactory selectivity (Kim et al. 2007a). Several key examples of successful applications of these electrodes in monitoring of hydroponic nutrient solution are reviewed in this section and presented in Table 3. As proof of concept, an economic automatic nutrient control system without any computers and pumps for a hydroponic system comprised by a programmable logic controller, potentiometers, a data logger and nitrate, phosphate, and potassium selective electrodes was designed by Xu et al. (2020). The system can monitored all the tested ions by ion-selective electrodes and maintain them to the desired concentration by using solenoid valves connected with the programmable logic controller. The authors stated that in the concentration range of 10^{-6} to 10^{-5} M, the response time of the NO_3^- and K selective electrodes was within 5 s and the system can be stably used for more than 30 days continuously. However, phosphate selective electrodes were 10 times longer than other tested ion-selective electrodes (Xu et al. 2019, 2020). It seems reasonable that a K and N-ion sensor-based membrane would have high potential for commercialization due to their lifetimes of more than one month.

Bailey et al. (1988) investigated the performance of commercially available ion-selective electrodes such as NO_3^-, Ca_2^+, K^+, Na^+, H^+ and Cl^- in conjunction with a flow cell installed in a nutrient film technique system used for tomatoes cultivation. The sensitivity of the sensors was found to be decreased over time for all of the tested electrodes. To address this drawback, regular calibration was suggested. Another commercially NO_3^-, K^+, Ca^{2+} and Cl^- four ion-selective electrodes-based carbon nanotubes integrated computer-operated liquid handling system was tested for monitoring of vertical tomato hydroponic nutrient solution where two-point calibration methods and frequent measurement of NO_3 and K ions were suggested. Change in each specific nutrient was automatically calculated using an excel tool and instantly adjusted to desired values manually by adding nutrient solution (Rius-Ruiz et al. 2014). However, the system has a drawback that ion-selective electrodes need to be daily calibrated before a sample measurement. In addition, the analytical performances of the ion-selective electrodes indicated that the stability of the Ca and K selective electrodes was higher than those of NO_3 selective electrodes. The different elements composing this automated system are illustrated in Fig. 5.

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Table 3 Examples of ion-selective electrodes used in monitoring hydroponic nutrient solution

Ion-selective electrodes	Plant	Calibration method	Remarks	References
K	Brassica rapa	One-point method	Excellent selectivity in the presence of other cation	(Xu et al. 2019)
NO_3, PO_4 and K	Brassica rapa	One-point method	The nutrients concentrations were kept almost constant during plant growth	(XU et al. 2020)
NO_3, Ca, K, Na, H and Cl NO_3, K, Ca and Cl NO_3, K, Ca and Cl	Tomato (nutrient film system) Tomato Lettuce	Two-point method Two-point method One-point method	The life of polyvinyl chloride electrodes used for nitrate, potassium and calcium were short (less than 4 months) Higher stability of K^+ and Ca^{2+} selective electrodes than NO_3^- and Cl^- electrodes The level of tested ions concentration was maintained according to the plant requirement with in errors of less than 21 mg L^{-1}	(Bailey et al. 1988) (Rius-Ruiz et al. 2014) (Pan et al. 2017)
NO_3, Ca and K	Without crops	One-point method	Ca electrode exhibited low sensitivity	(Heinen and Harmanny 1991)
NO_3, Ca and K	Tomato (Nutrient film system)	One-point method	Ions uptake by plant was monitored successfully	(Morimoto et al. 1991)
PO_4	Paprika	One-point method	PO_4^- concentrations were found to be comparable to those obtained with standard laboratory techniques, R^2 equal to 0.80	(Kim et al. 2011)
NO_3, K, Mg and Ca	Paprika	Two-point normalization method	Ca-selective electrodes showed reduced sensitivity and poor selectivity	(Kim et al. 2013)
NO_3, Ca K	Lettuce (ebb-and-flow system)	Two-point normalization method	K-selective electrodes showed less sensitivity The level of tested ions concentration was maintained according to the plant requirement with in errors of less than 30 mg L^{-1}	(Jung et al. 2015)
NO_3, Ca and K	Lettuce (ebb-and-flow system)	Two-point normalization method	The level of tested ions concentration was maintained according to the plant requirement with in errors of less than19 mgL^{-1}	(Cho et al. 2017)
NO_3, Ca and K	Paprika (greenhouse)	Two-point normalization method	The level of tested ions concentration was maintained according to the plant requirement within errors of less than 20 mg L^{-1}	(Cho et al. 2018)
NO_3, Ca and K	Cucumber and tomato (greenhouse)	One-point method	Ions uptake by plant was monitored successfully same amounts of calcium were taken by cucumber and tomato, cucumbers constitute more potassium than tomato, and tomato was consumed less amount of nitrate than cucumber	(Vardar et al. 2015)
NO_3, Ca, K and PO_4	Lettuce (ebb-and-flow system)	Two-point normalization method	The level of NO_3, Ca and K concentration was maintained according to the plant requirement with in errors of less than 26 mg L^{-1} The cobalt electrodes yielded a relative mean standard error result of 10.9% from a comparison of the electrode process and standard analysis	(Jung et al. 2019)

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Table 3 (continued)

Ion-selective electrodes	Plant	Calibration method	Remarks	References
NO_3	Brassica rapa	One-point method	NO_3 selective electrodes showed a satisfactory sensitivity	(Fukao et al. 2018)
NO_3, K, and H	Without crops	Two- and three-point normalization methods	Strong linear relationships between the sensors and the standard instrument methods	(Kim et al. 2017)
K, Ca, NH_4 and NO_3	Nutrient film system without crops	Three-point normalization methods	The system successfully measured the nutrient concentration	(Ban et al. 2020)

Recently, a multi-channel detection platform with an ion-selective electrodes array combined with a complex data processing by back-propagation artificial neuronal network (BP-ANN) exhibited acceptable outcomes for monitoring NO_3^-, K^+, Ca^{2+}, Cl^- and pH during the lettuce growing. In a concentration range of 150 to 300 mg L^{-1} NO_3–N and 160 to 400 mg L^{-1} K, there was a significant linear relationship as high as 0.84–0.93 between the ISEs and spectrometer methods within errors of less than 10 mg L^{-1} for NO_3^- and K^+. However, insufficient detection precision of the calcium ions with an errors of less than 21 mg L^{-1} was reported due to the complex interference in nutrient solution (Pan et al. 2017). Nutrients concentration in an automated nutrient film technique system without crop was measured using membrane-based NO_3 and Ca-selective electrodes and glass-based K selective electrode. As the electrode response depends on the temperature of nutrient solution, these devices provided more accurate data when temperature was maintained constant. In this system, daily calibrations were required to maintain usability over long periods. In addition, Ca electrode exhibited low sensitivity (Heinen and Harmanny 1991). In tomato hydroponic research, the uptake of the NO_3^-, Ca_2^+, K^+ ions was monitored by the above-mentioned ion-selective electrodes (Morimoto et al. 1991).

To avoid the continuous control of sample preparation, ion-selective electrodes calibration, and data collection which reduce variability among numerous electrodes during replicate measurements, use of a computer-based automatic measurement approach was suggested to improve accuracy and precision in the measurements of nutrient concentration (Kim et al. 2007b, 2011). In this regards, cobalt-based electrode used in conjunction with a laboratory-made automated test stand was applied for quantification of PO_4^- in paprika hydroponic solution. The developed system showed sensitive responses to phosphate and adequate selectivity (Kim et al. 2011). Computer-controlled measurement system combined with polyvinyl chloride membrane-based ion-selective electrodes for automatic sampling and electrode rinsing was developed by Kim et al. These sensors were fabricated with tetradodecylammonium nitrate-2 nitrophenyl octylether (TDDA–NPOE), valinomycin, and calcium ionophore II and have been evaluated in terms of sensitivity and selectivity by measuring NO_3–N, K, Mg and Ca concentrations in paprika hydroponic solution. In mixed solutions, responses of the NO_3 and K ion-selective electrodes provided a linear calibration models with high coefficients of determination, R^2 equal to 0.87. However, Ca^{2+} sensor had standard error of 20.8 mg L^{-1} and the R^2 of the calibration regression line was just 0.58 because it is affected by both Ca and K ions. Monitoring tests in the paprika hydroponic solution showed that in a concentration range of 3–300 mg $L^{-1}$$NO_3$–N and 3–700 mg L^{-1} K, there was a significant linear relationship, R^2 equal to 0.85 between the ion-selective

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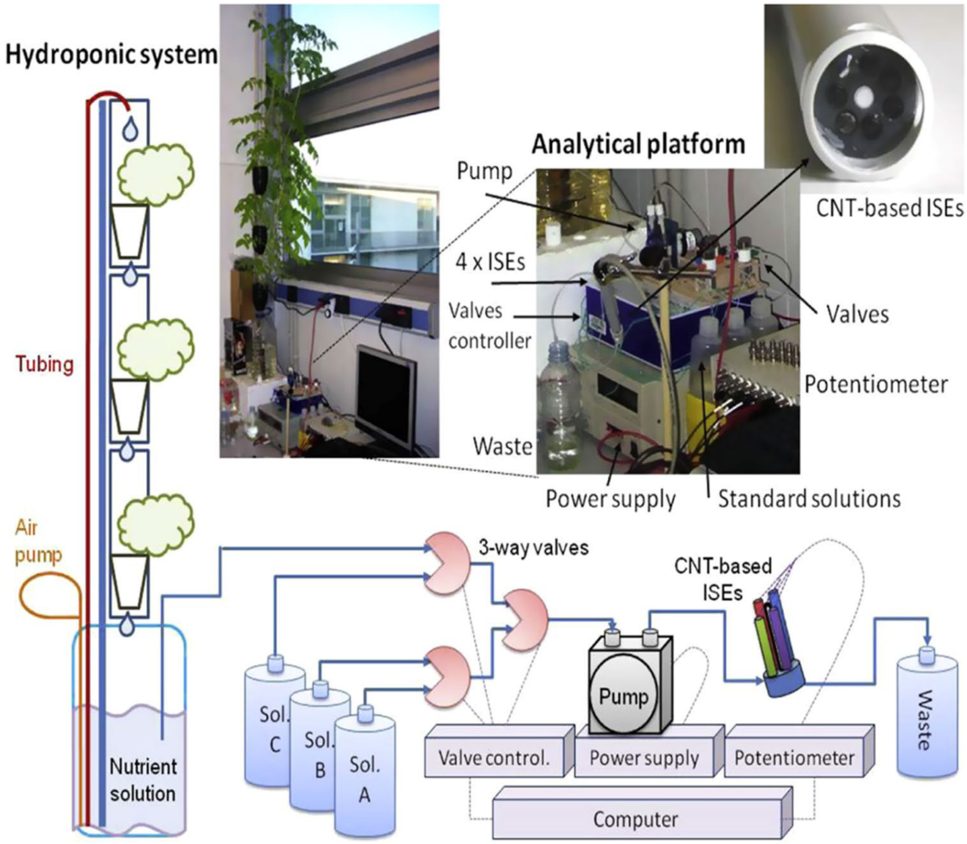

Fig. 5 Photographs and scheme of all the elements composing the computer-operated analytical platform for the determination of nutrients in hydroponic systems. CNTs-based ISEs: carbon nanotubes-based ion-selective electrodes. Reprinted with permission of Elsevier from (Rius-Ruiz et al. 2014)

electrodes and laboratory methods (Kim et al. 2013). The author suggested developing a new Ca and Mg membrane with high sensitivity to address the issue of the poor selectivity for Ca in hydroponic solution. The aforementioned ion-selective electrodes have been evaluated for nutrient control in lettuce hydroponic solutions in closed systems where a dosing algorithm to maintain target concentrations of NO_3, K, and Ca ions during the period of lettuce cultivation was implemented. The system was able to formulate different concentrations of NO_3 and Ca ions analogous to the target concentrations in spiked test, showing almost no differences between the actual and target values. However, K sensor showed less sensitivity. This system was able to manage the studied ions to attain target concentrations within errors of less than 30 mg L^{-1} (Jung et al. 2015). However, the relatively high cost of the proposed system including hardware, software and multiple commercial data acquisition limited its practical use in real greenhouses. To maintain the target concentrations of specific nutrients necessary for crop more efficiently with inexpensive materials, an embedded system that includes a micro-control unit-based embedded nutrient controller and ion-selective electrodes in combination with an aforementioned supposed fertilizer dosing algorithm was designed. In lettuce cultivation experiment, the level of NO_3, K, and Ca concentration was maintained according to the plant requirement with in errors of less than 19 mg L^{-1} (Cho et al. 2017), which is more accurate than previous developed system (Jung et al. 2015; Mishra et al. 2020). Based on these encouraged results, real-time measurement of NO_3, K, Ca, and P ions in different real hydroponic solutions samples, e.g., kale, paprika, basil and beach silvertop-based embedded system, has been constructed. The test results showed a high linear relationship between the standard analyzers and embedded system for the quantitative determination of the concentrations of NO_3 and K where R^2 was found to be equal to 0.99. The accuracy error of the measurements for these ions was 3.7 to13.2%, and 1.0 to 14.0%, respectively. As for P ions, cobalt electrode showed a stable measurement results (Han et al. 2020a). However, in both above embedded systems, Ca-selective electrode showed low sensitivity (Cho et al. 2017; Han et al. 2020a). The predictive capability of the above suggested systems was not validated in hydroponic solutions in real greenhouses.

The test results using paprika hydroponic solution in real greenhouse indicated that the previously designed system showed a high sensitivity and accuracy toward measurements of nitrate concentrations where the results were comparable to the results of ion chromatography, slope of 0.99 and R^2 of 0.99. However, K and Ca measurements showed some deviations with slopes of 1.17 and 0.75, respectively. Same results were found with K selective electrodes used in previous reports (Mishra et al.

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2020). In this test, the level of the above ions concentration was maintained according to the plant requirement with in errors of less than 20 mg L^{-1} over the range of 40–1200 mg L^{-1}. In addition, the temporal changes in ionic concentrations were monitored efficiently and with high sensitivity (Cho et al. 2018). The previously developed ion-selective electrodes showed acceptable selectivity and sensitivity for measuring NO_3^-, K^+, and Ca^{2+} concentrations in cucumber and tomato hydroponic nutrient solutions (Vardar et al. 2015). However, the ion interference effect has been considered in their studies.

More recently, real-time quantification of NO_3^-, K^+, and Ca^{2+} and the application of a concentration ratio method to replenish Mg^{2+} and PO_4^{3-} were investigated in hydroponic lettuce solution. However, this method was not useful in monitoring the concentrations of PO_4^{3-}. Based on these outcomes, the applicability of a cobalt rod-based electrode phosphate sensing in nutrient solution was investigated and the cobalt electrodes yielded a relative mean standard error (RMSE) result of 10.9% from a comparison of the electrode method and standard analysis (Jung et al. 2019). Hence, the combination of the cobalt electrode to ISEs device would be feasible for hydroponic nutrient solution management. The above designed system is presented in Fig. 6.

The ion interference effect has been considered in the above suggested systems. They all declared that in higher density solutions, the increase in error will be greater between selective electrodes values and theoretical values.

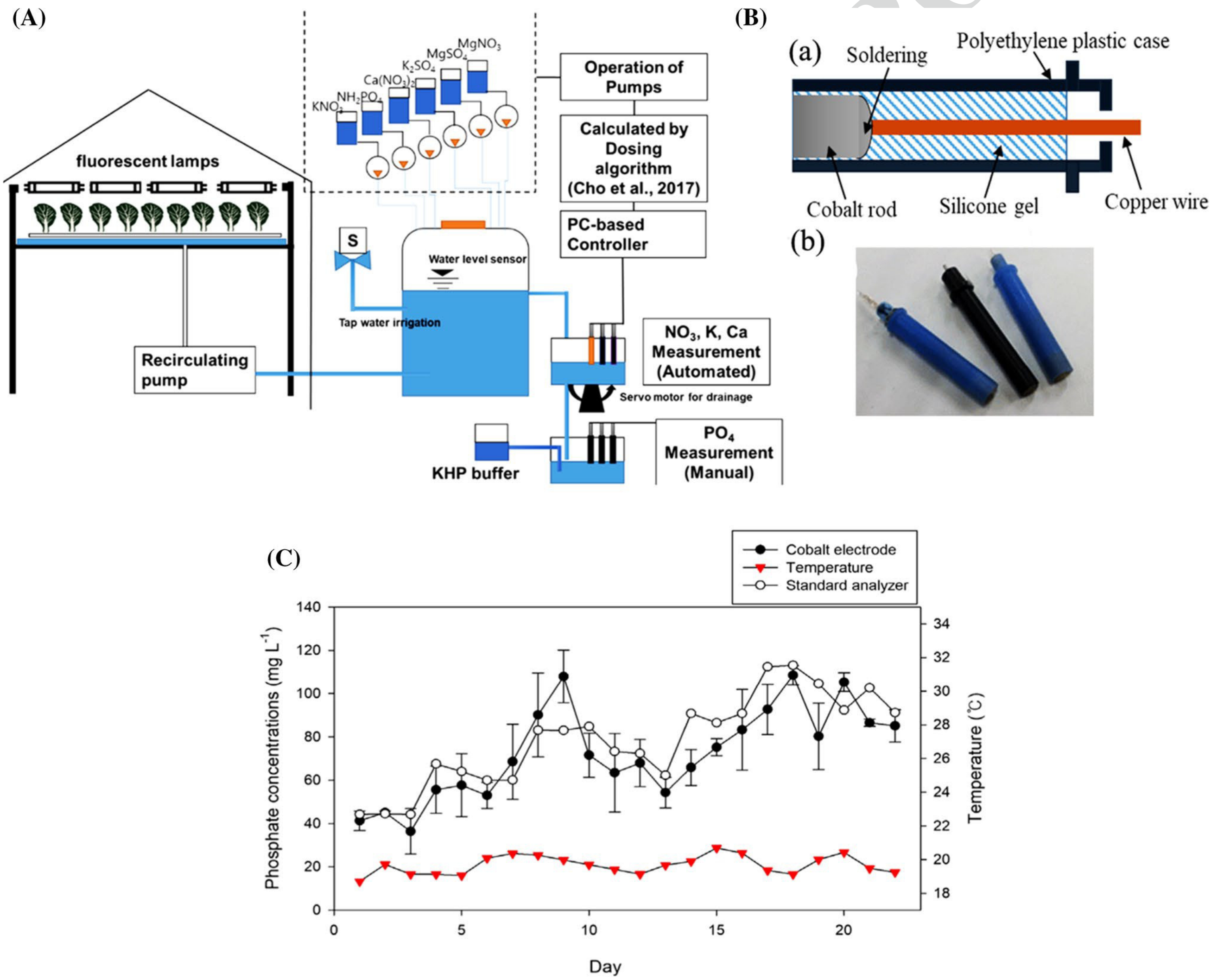

Fig. 6 Schematic of the automated lettuce cultivation system based on simultaneous measurement of NO_3, K, and Ca ions in conjunction with a fertilizer dosing algorithm (**A**). A section view of showing the internal components of the cobalt electrode (**B**), (**a**), and a photographic image of the cobalt electrodes (**b**) prepared in the study. Comparison between PO_4 concentrations was determined by standard analysis and by cobalt electrodes and a change in solution temperature over time (**C**). Reprinted with permission of Elsevier from (Jung et al. 2019)

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Consequently, flow injection potentiometric determination of phosphate in nutrient solution was carried out using cobalt-wire phosphate ion-selective electrode. Flow injection potentiometric is the type of continuous flow analysis that utilizes an analytical stream, unsegmented by air bubbles, into which highly reproducible volumes of sample are injected (Ranger 1981). This technique provides a fast response to PO_4^{3-} and is comparatively free from common anion interferences. Cobalt-wire phosphate flow injection potentiometric method yields a comparable accuracy to the laboratory methods, e.g., spectrophotometric and ion-pair high performance liquid chromatography methods (HPLC) (De Marco and Phan 2003).

In order to avoid the presence of interference ions such as K and Cl, NO_3 sensors prepared from polyvinyl chloride membrane were modified by changing the electrolyte of the inner and the salt bridge between the sample solution and reference electrode. A stable electrochemical response of this electrode was noted for more than 30 days with low limit of detection of 10^{-6} M (Fukao et al. 2018).

In measurement with ion-selective electrodes, an embedded portable analyzer integrated with NO_3, K, and H electrodes array-based polyvinyl chloride membrane which was calibrated using two-point normalization methods and three-point calibrations method, respectively, enabled direct analysis of paprika hydroponic nutrient solution without the need to dilute samples and exhibited strong linear relationships with the standard instrument methods where R^2 was found to be higher than 0.96 (Kim et al. 2017). With the high sensitivity of valinomycin and tetradodecylammonium nitrate-based membranes to potassium and nitrates in hydroponic solution and their lifetimes of more than one month, it seems reasonable that a K and NO_3 ion sensor-based membrane would have high potential for commercialization and worldwide application in the large-scale hydroponic system.

More recently, a machine-learning based algorithm has suggested to be applied on automated fertilization system in vertical smart farm for removal of ion interference effect by readjusting the electrodes signals for restoring its accuracy up to 98%. In this research, conductivity, pH, K^+, Ca^{2+}, NH_3^+ and NO_3^- sensors which calibrated using three points normalization methods are installed in a container of nutrient solution where it includes also water level sensor. The developed process successfully quantified and readjusted the nutrient concentration of each reservoir (Ban et al. 2020).

Hydroponic nutrient control system based on internet of things

The internet of things is the interconnection between the internet and objects. These forms of connections make it possible to bring together new masses of data on the network and therefore new forms of knowledge (Tan and Wang 2010). At present, numerous heterogeneous devices or systems can be connected using internet of things concept (Wortmann and Flüchter 2015; Verdouw et al. 2016). Each industrial sector needs internet of things due its ability to communicate industrial machinery with each other and supply a framework where data-driven decisions can be taken automatically (Tzounis et al. 2017).

By using internet of things in agriculture sector (Srivastava et al. 2018), data and analysis of hydroponic solution status could be gathered by the farmers based on the information, and they can look after the hydroponic crops through Android Smartphone (Vidhya and Valarmathi 2018). Generally, the microcontroller ensures the supply of the nutrient solution (Saaid et al. 2013). Data from the sensor are transmitted through the Arduino board. Based on this later, management of hydroponics nutrient solution can be done automatically even if the farmer is in long-distance (Sihombing et al. 2018). By contrast, with WiFi, data transmitted only up to a certain distance (Vidhya and Valarmathi 2018). Nowadays, the network capacity is improved by the 5G technologies which expand the feasibility of internet of things implementations and integration of complex communication systems into hydroponics (Yanes et al. 2020). In this section, we will review the internet of things technologies used for monitoring hydroponic solution. An example of system architecture of internet of things based hydroponics is illustrated in Fig. 7.

The ion-selective sensors described in previous section create massive flows of data that should then be analyzed in order to be adequately exploited (Sambo et al. 2019). Machine learning algorithm which is defined as an application of artificial intelligence that enables a system to learn from examples and practice without explicit programming has helped in automating the plant growth through self-calibrating, managing the parameters of hydroponic solutions based on sensors data and ensuring the system will improve over time to better respond to conventional variations in the system (Knight and Lefsrud 2017; Mupangwa et al. 2020). Recently, various machine learning algorithms such as fuzzy logic (Yolanda et al. 2016), k-nearest neighbors (Herman et al. 2019) and deep neural network (Mehra et al. 2018) to monitor the hydroponic nutrient solution based on the multiple input parameters gathered to improve the performance of the automated hydroponic systems. For example, fuzzy logic control algorithm is executed using embedded Arduino microcontroller ATMega328 which is device that receives and sends information or command to the respective circuit with I/O hardware to monitor pH and electrical conductivity of nutrient solution where the data were shown to internet of things web (Badamasi 2014; Yolanda et al. 2016). Similarly, the uses of fuzzy logic and internet of things for monitoring lettuce and bok choy hydroponic system have been proposed

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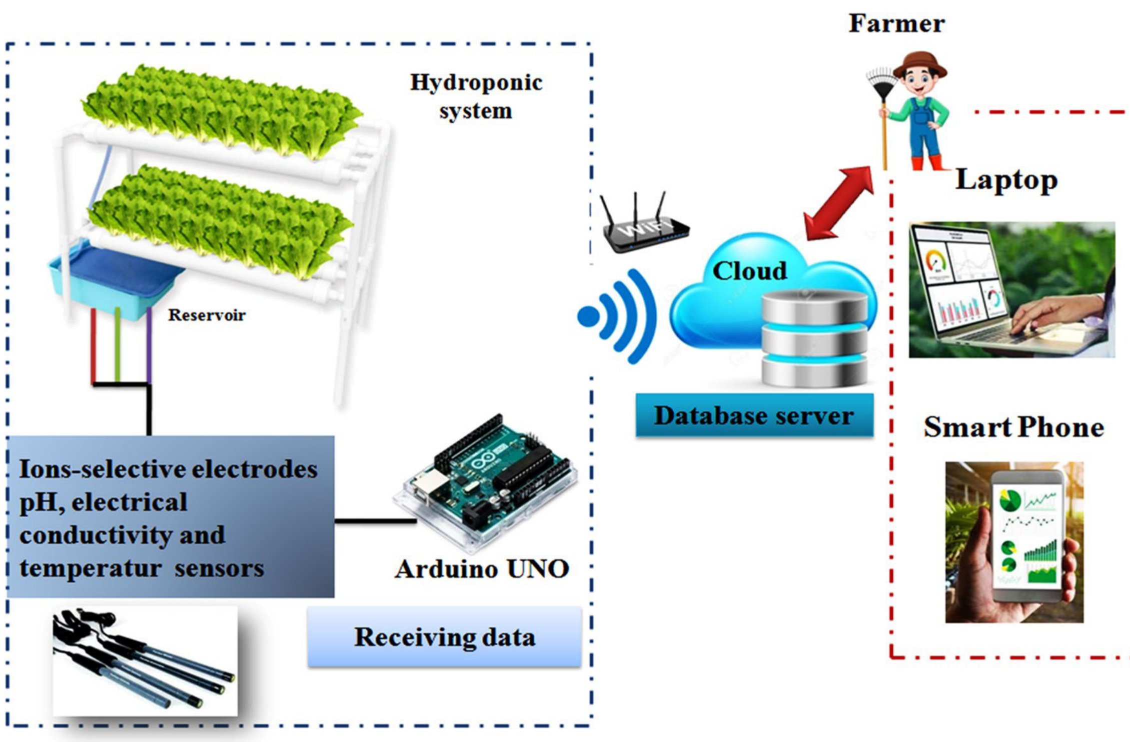

Fig. 7 System architecture of internet of things-based hydroponics

by Herman and his coworkers. Internet of things devices are used to monitor plant conditions and water needs. Meanwhile, fuzzy logic is used to manage accurately the supply of water and nutrition (Surantha 2019). The k-nearest neighbor algorithm was applied to predict the classification of nutrient conditions, and then the prediction outcomes are used to offer a command to the microcontroller to turn on or off the nutrition controller actuators simultaneously at a time (Herman et al. 2019). In another tomato hydroponic research, internet of things is combined with deep neural network to predict nutrient control. This algorithm predicts the label based on table control, which has eight labels. The sensor value was shown by the system output which predicts the control labels with the prediction of accuracy percentage (Mehra et al. 2018). In measurement with smart hydroponics, Node Red for wiring together hardware devices, message queuing telemetry transport (MQTT) to transfer data, raspberry pi 3 and sensors for monitoring water level, pH, temperature of nutrient solution and room humidity have been exploited to design an automated smart lettuce hydroponics system. By using this system, the farmers could receive an email and SMS as well, to be informed about the situation of their implemented hydroponics. In addition, the sensor hub when connected to the WiFi, the sensors data could be shown in the farmer's Smartphone via secure cloud (Lakshmanan et al. 2020). In another research, STM32 microcontroller has been used as device to mix hydroponic solution according to nutrients needed automatically (Yapson et al. 2018).

The reviewed literature researches use micro-controllers, such as Raspberry Pi and Arduino, in hydroponic systems. The most basic control methodology can be understood as a local approach with external communication where the information is analyzed and gathered wirelessly through cloud servers. The data were sent wirelessly to the control center where the data are stored, then processed, and sanded to a remote server. Once this later stores the information, farmers were able then to examine the information and make decisions in regard to the state of the nitrates, phosphate, pH, conductivity and temperature and so forth. From an environmental point of view, the use of new technologies such as internet of things and ion-selective electrodes seems to be a better alternative to be adopted.

Concluding remarks and perspectives

The current researches conducted on the monitoring of hydroponic nutrient solution by using ion-selective electrodes and internet of things have been detailed and discussed in this review. The performance of the aforementioned systems in terms of stability, sensitivity and its limitations is concluded as follows:

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1. pH and electrical conductivity measurements alone do not provide detailed information to allow growers or farmers to realize optimal plant production from an effective management of the nutrients.
2. An ion-selective electrode utilizes the ion-selective features of specialized materials to generate an electrical signal that can be detected and quantified. In this case, the behavior of ion-selective materials is due to ion-selective components that are included in their design which can also be influenced by environmental conditions. For example, the temperature variability of the nutrient solution has not always been taken into account in several studies.
3. Most of the tested electrodes exhibited strong linear relationships with the standard instrument methods except for Ca electrodes which show less sensitivity. Hence, future research needs to focus more in enhancing the sensitivity of Ca selective electrodes at high concentration by using new materials.
4. The target concentrations of NO_3, K, and Ca were successfully maintained at desired levels of concentration with in errors of less than 19 mg L^{-1} through using polyvinyl chloride membrane-based ion-selective electrodes and cobalt rod-based electrode provides rapid response to hydroponic phosphate. However, measuring few plant macronutrients at a time did not supply the farmers a complete picture of the elements' availability in the nutrient solution. In addition, selective electrodes for the determination of plant micronutrients, such as Fe, Cu, and Zn, have not been designed or applied yet.
5. AQ3 Despite of regular calibration sequence was implemented to solve the stability issue of ion-selective electrodes. However, temperature sensitivity, lifetime and the requirement of a reference electrode are the main limitations that selective electrodes technologies are facing now. Hence, new generation of these sensors will likely continue to overcome the aforementioned technical drawbacks such as simultaneous macro- and microelements measurements and sensitivity and might represent a useful device for the timely monitoring of nutrient solution, with the aim of satisfying the nutritional requirements of crop plants for optimal yield.
6. Overall, compared to carbon nanotubes based ion-selective electrodes, the best results for sensing nitrate in hydroponic solution were obtained with polyvinyl chloride ion-selective membranes prepared with quaternary ammonium compounds, such as tetradodecylammonium nitrate.
7. On the other hand, the emergence of internet of things has allowed growers to automate the hydroponic system. Monitoring of conductivity, pH, temperature and detection of macronutrient in hydroponic cultivation bed can be done, and they can be regulated by use of internet of things.
8. In terms of economic, the relationship between costs and benefits of smart automated hydroponic systems should be analyzed and quantified in the future researches. In addition, life cycle assessment of this technology could be substantial and warrants further research in future studies.

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