SUST Journal of Engineering and Computer Science (JECS), Vol. 16, No. 2, 2015

Temperature Compensation in pH meter-A Survey

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Received:24.04.2014
Accepted:12.06.2014

ABSTRACT - The measurement of pH has a great significance in many applications, ranging from water quality management through the agricultural to the biomedical applications. Most of those require an accurate measurement of pH. Unfortunately the accuracy of pH measurement is affected by the temperature of the measurement sample, which leads to an error in the measured pH value. To eliminate or reduce this effect, the temperature change must be compensated or avoided. The purpose of this paper is to describe the ways that the temperature can affect the measured pH value as well as how they are to be compensated or avoided. From the sensor point of view, the sensors that are used in pH measurement have attracted researchers' attention so as to develop and enhance those sensors which have a capability to meet an application requirement. Since the first pH sensor (glass electrode) failed to meet some application requirements, the developers have proposed a new electrode based on CMOS technique known as ISFET. This paper also aims to highlight the works which were done based on glass electrode and ISFET with concentration on advantages and disadvantages of each technique.

Keywords: Temperatuer Compensation, pH meter, Glass Electrode, ISFET, Embeded Systems.

المستخلص ـ إن قياس درجة حامضية وقاعدية السوائل من الأهمية بمكان في العديد م التطبيقات التي تمتد من إدارة المياه مرورا بالتطبيقات الزراعية والحيوية. أغلب تلك التطبيقات تحتاج إلي قياس درجة الحامضية والقاعدية بدقة عالية. تتأثر دقة تلك القياسات بدرجة حرارة السائل المقاس مما يؤدي لخطأ في القيمة المقاسة. لتقليل أو تفادي هذا التأثير الحراري لابد من تعويضه أو تجنبه. إن غرض هذه الورقة هو وصف كيفية تأثير درجة الحرارة علي قياسات الحامضية والقاعدية للسوائل وكيفية تعويض هذا التأثير أوتجنبه. من ناحية تقنية الحساسات, نجد أن الحساسات المستخدمة في قياس درجة الحمضية والقاعدية تنال اشد الإهتمام من قبل الباحثين لتطويرها وتحسين ادائها لمقابلة مطلوبات التطبيقات المختلفة.وحيث أن حساس درجة الحمضية والقاعدية من النوع الزجاجي لم يلبي مطلوبات بعض التطبيقات, قام الباحثون بإقتراح وتطوير حساس من أكاسيد أشباه الموصلات من النوع المكمل والذي يعرف بحساس الأيون مقحل تأثير الحقل. أيضا تهدف هذه الورقة مراجعة بعض الأدبيات التي تستخدم حساس أكاسيد أشباه الموصلات من النوع المكمل وحساس النوع الزجاجي في تصميم مقاييس درجة حامضية وقاعدية السوائل مع التعرف لمزايا كل نوع.

INTRODUCTION

PH stands for power of hydrogen ions. It is a measurement of the degree of acidity or alkalinity of aqueous solution; the acidity of solution indicates the concentration of the hydrogen ions $[H^+]$ while the alkalinity indicates the concentration of hydroxide ions $[OH^-]$. It is also mathematically defined by Danish biochemist, Soren Sorensen in 1909 as the negative base 10 logarithm of the molar concentration of the hydrogen ion in an aqueous solution, and it is expressed as: $pH = -\log[H^+]$. In other words the ions concentration for mol/liter is equal to 10^{-pH}.

The pH covers the range from 0 to 14. pH 0 is considered as strong acid while pH 14 is considered as strong base and the midpoint between them (i.e. pH=7) is considered as a neutral point. At pH =7 the solution is neutral because the hydrogen ions $[H^+]$ become equal to the hydroxide ions $[OH^-]$ (equal to 0.0000001 mol/liter for both) [1-8].

pH measurements can be performed conventionally or electronically. The pH test strips (pH paper) and colorimetric method (pH indicator) are the conventional methods which are not accurate. Therefore they are often limited to differentiate between basic and acidic solutions. The electronic pH meter is the most popular method nowadays due to its high accuracy and resolution [9, 6]. To perform this electronic measurement, a pH electrode connected to an electronic meter that measures the potential difference between the measuring and reference electrodes, converts it into corresponding pH value and displays it [7, 10].

A pH electrode is an electrochemical sensor which produces a voltage output proportional to the pH value; it usually consists of a measuring electrode (indicator electrode or glass electrode) and reference electrode (standard electrode). Two measuring electrode types depending on a pH sensing element can be recognized: glass electrode and ion sensitive field effect transistor (ISFET) electrode.

The pH measurement is significantly affected by the temperature variation, which is the most common parameter that causes an error in measurement of pH. The temperature variation affects the measured pH in two ways: the pH of an aqueous solution itself can change due to the effect of temperature, and the temperature affects the pH sensor, which is the source of the error. To perform an accurate pH measurement it is most important to take this effect into account, and it should be compensated or avoided. This study is intended to review the ways this temperature can affect the pH measurement and how it can be compensated or avoided.

In electronic engineering, the problem that is facing many system designers is the selection of appropriate components that are suitable to satisfy the design requirements. It is most critical in embedded system design since it is constrained by many requirements such as: power consumption, size, performance and reliability. This study will also helps any researcher who wants to design an electronic pH meter in terms of the most suitable pH electrode that should be used for a specific design. Besides, it highlights the two designs; those based on the glass electrodes or the ISFET electrodes.

The importance of pH measurement

Since; all living things depend on aqueous solution; they need a proper pH level to sustain life. The human beings and animals have an internal mechanism to maintain the pH level of their blood, the foodstuffs must be produced under a certain value of pH; in agriculture the optimum growth of crops also depends on the soil pH value. The measurement of pH is not only important for living things, but it is an important parameter that must be measured for any product that contains aqueous solution. During product production, the pH value must be controlled to ensure that it satisfies the predefined pH measurement as well as product cost efficiency. The pH measurement is important for other applications such as: wastewater treatment, environmental monitoring, chemical and life sciences research and electronics production etc. The table below contains pH value of some foodstuffs [1, 2, 4, 5, 11, 12].

Table 1: The pH Value of Some Foodstuffs

Foodstuffs	Corresponding pH value	
Lemon Juice	2.3	Acidic
Cola Beverages	2.8	Acidic
Vinegar	2.9	Acidic
Fruit Vinegar	3.2	Acidic
Orange Juice	4.3	Acidic
Coffee	5.0	Acidic
Cheese	5.3	Acidic
Corn	6.2	Acidic
Milk	6.6	Acidic
Distilled Water	7.0	Neutral
Egg White	7.8	Basic

The pH electrode is an electrochemical sensor used in many applications. The pH electrode usually consists of a reference electrode, sometimes called a standard electrode, and a measuring electrode; also called indicator electrode or glass electrode. It is just like a battery source, so it produces a voltage output in millivolts for the glass electrode- which is proportional to the pH value [4, 13, and 14].

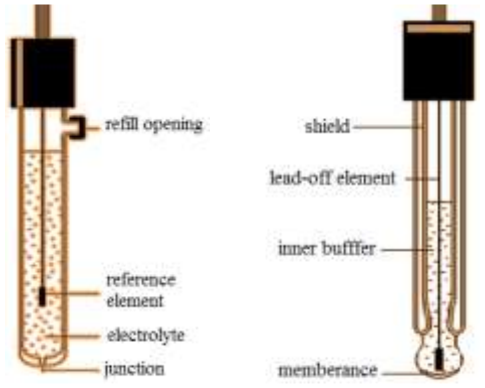
The reference and measuring electrodes can be separated or combined in a single design (see Figure 1 and Figure 2(a)). The combination electrode is the popular pH electrode today, however; the combination electrode with a built-in temperature sensor (known as three-in-one electrodes) is the most popular one (see Figure 2(b)). Since the temperature can affect the pH value, the temperature compensation can be easily done with one probe [15, 11].

The Measuring Electrode

The measuring electrode, which acts as first half-cell of the galvanic cell, is responsible for producing a potential signal that depends on the changes in the pH of a measured sample [16]. The measuring electrode can be a glass electrode (see Figure 1) or solid state electrode (ISFET). The ion sensitive part of a measuring electrode is a glass membrane and membrane material (*e.g.* aluminum oxide or silicon nitride) for glass and ISFET electrode respectively [17].

The Reference Electrode

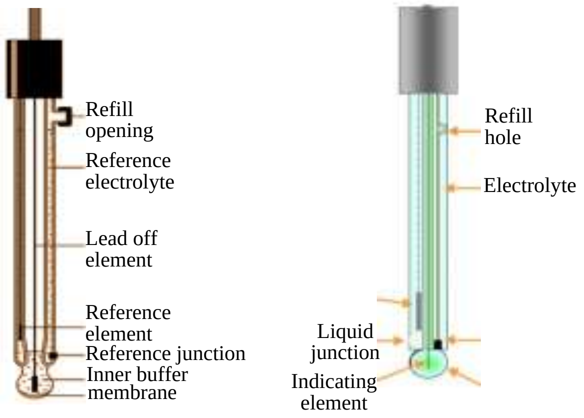
The reference electrode, which acts as the combined second half-cell (see Figure 3), will be of a constant and stable potential. It is independent of the measured sample solution for accuracy [4, 14].



(a) (b)

Figure 1: (a) Refference Electrode Structure (b) Measuring Electrode (Glass Electrode) Structure [11]

The most common reference electrode is a silver wire coated with silver chloride (Ag/AgCl), as a consideration to the environmental effect. The reference electrode can be a single junction or a double junction. In many applications, the single junction can satisfy the application requirement, where the sample contains sulfides, proteins, or any material that can interacts with silver ions, erroneous reference signals beside a short service life result; in this case the double junction reference electrode must be used because it is designed to overcome the silver ions reaction problem [13, 18].



(a) (b)

Figure 2: (a) Combination Electrode (b) Combination Glass Electrode with Built in Temperature Sensor [11,18]

Sensing Element

Ion Sensitive Field Effect Transistor (ISFET)

An ISFET is a solid state pH sensor device which is used as sensing component to measure the pH of the solution to be analyzed. It differs from a known Metal Oxide Semiconductor Field Effect Transistor (MOSFET) in the way that its metal gate is replaced by the Ion Sensitive membrane material (such as aluminum oxide or silicon nitride) [17, 19]. Moreover ISFET is a suitable component in contrast with a breakable glass electrode which leads to unacceptable safety hazards. Also the small size leads to the development of an inexpensive, low power consumption and small size system [17].

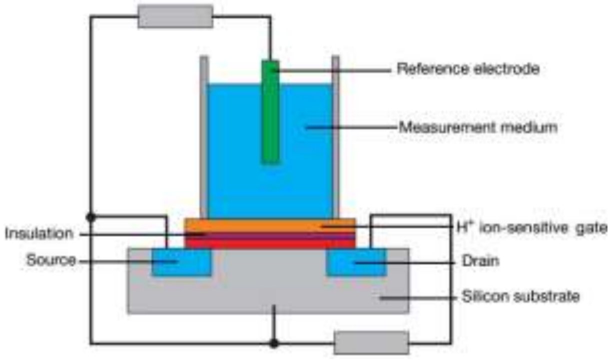


Figure 3: The ISFET Combination Electrode Structure [34]

The first ISFET was proposed as an alternative to the fragile glass electrode by Bergveld in 1970. It was based on ion sensitive solid state device (field effect transistor). ISFET development was based on CMOS (Complementary Metal Oxide Semiconductor) technology, so it became a base for many biochemical sensor devices [20, 21]. ISFET has become available commercially in 1990. The gap between proposed and commercial availability (about 20 years) was spent to overcome the few practical limitations such as threshold voltage drift, temperature and light sensitivity effect, reference electrode minimization and encapsulation [22]. The ISFET is gaining interest due to its superiority over a glass electrode in many aspects such as: stiffness and durability, easily clean, low cost so it is suitable for disposable application, can be stored dry, has an extreme pH range with a less acidic and alkaline error, has a wide temperature range, can be fabricated massively using VLSI (Very Large Scale Integration) technology, has low output impedance, which can directly interface to a low input impedance data acquisition system, has a fast response time and high sensitivity, has a micro size and can be integrated in an on-chip circuit and has an excellent shelf life [17, 21-23].

Glass Electrode

The glass electrodes are the most common sensing element utilized by pH meter designer. The principle of potential measurement was discovered by Max Cremer in 1906. Although it was discovered since 1906, it became available commercially in 1950. Since then, the glass electrodes are developing over time to meet the needs of application requirements [24].

The glass electrode has advantages over ISFET electrode such as: not sensitive to direct sunlight, simplicity and convenience use especially for learning purposes, gives more accurate results, can be used in solutions which are colored, turbid and even colloidal.

The effect of temperature in pH measurement

The most common parameter that causes an error in pH measurement is the temperature. The temperature can affect the pH measurement in two ways: firstly the pH of an aqueous solution can change due to the effect of temperature. The temperature causes the molecule dissociation of weak acids and bases beside the molecules of water itself. This is known as the solution temperature coefficient and is measured by (ΔpH/°C); it depends on the composition of the solution itself [5, 25].

The temperature variation of standard pH buffers is usually printed on their bottles. While the measured sample temperature coefficient is unknown because any sample solution has a unique pH value versus temperature relationship, so a solution temperature correction cannot be done for measured sample, but it must be compensated when performing a calibration by using standard pH buffers in order to achieve an accurate measurement. Moreover the change in pH value due to the solution temperature coefficient for the measured sample is considered as a true value of pH rather than an error [18, 25-26].

To avoid misunderstanding it is preferred to report the temperature value at which the measurement of pH is done, or perform a pH measurement and meter calibration under a constant temperature (*e.g.* at 25°C) [26]. Secondly the temperature affects the pH sensor itself in many ways: it affects the slope of pH electrode, affects membrane resistance, can affect the isothermal intersection points, affects response time of the electrode., and affects the difference in response of pH electrode and temperature sensor.

Effect of Temperature on Slope of pH Electrode

The electrode slope or sensitivity will change with temperature variations; the slope of pH electrode is determined by the Nernst equation [18, 25-27]

$$\begin{aligned} E &= E_0 + \left(2.303RT/nF\right)\log[a_{H^+}] \\ &= E_0 - \left(2.303RT/nF\right)pH \end{aligned} \tag{1}$$

where E ≡ total potential produced between the sensing and reference electrode in mV, E_o ≡ standard potential of the electrode, R ≡ gas Law constant, T ≡ temperature in degrees Kelvin, n ≡ the charge of the ion, F ≡ Faraday constant, and a_H^+ ≡ activity of the hydrogen ion in solution.

As seen in equation (1) above the relationship between potential of a pH electrode and temperature of the pH electrode is linear [28]. The factor $(2.303RT/nF)$ is the slope of the straight-line, which depends on temperature (T). By substitution the Gas constant (R), Faraday's constant (F), the charge of the ion (n) and temperature is 25°C, it is produce the slope equals: -59.16 mV/pH unit, which is considered as 100% theoretical Nernst slope [18, 25, 27]. This slope will be changed as temperature deviates away from 25 °C; the estimated value of this change is about 0.1984 units for every degree Celsius, Table 2 illustrates the slope of pH electrode at different temperatures.

Table 2: The Effect of Temperature Changes on the Nernst Slope for a Perfect Electrode

Temp. °C	Nernst slope (mV/pH)	Temp. °C	Nernst slope (mV/pH)
0	-54.20	50	-64.12
10	-56.18	60	-66.10
20	-58.17	70	-68.09
25	-59.16	80	-70.07
30	-60.15	90	-72.06
40	-62.14	100	-74.04

As illustrated in equation (1) the slope is multiplied by the pH value, this leads to increase the error as pH value is increased. This is illustrated in Figure 6. In order to perform an accurate pH measurement this change in slope must be compensated. It may be compensated either manually or automatically by automatic temperature compensation (ATC) [26]. Automatic temperature compensation is the automatic determination of pH value from the measured potential and temperature, using a slope that is adjusted to the measured temperature. Most of the modern pH measurement systems have the feature of automatic temperature compensation.

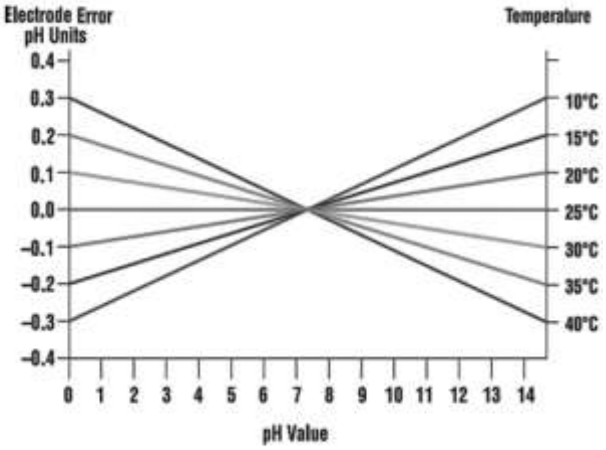


Figure 6: Error in pH Unit Versus Temperature for Glass Electrode [35]

Effect of Temperature on Membrane Resistance

Glass electrode membrane is an ionic conductor; the resistance of membrane is decreased with temperature increase. This results in slower response of the electrode that leads to an error. It also limits the operation temperature range of the electrode. The resistance change due to temperature varies and depends on membrane shape and membrane glass formulation. To avoid this error, it is recommended to perform the measurement at room temperature [25].

Effect of Temperature on the Isothermal Intersection Points

The isothermal intersection point of an electrode is defined as the intersection point of calibration lines that specify the relationship between pH value (horizontal axis) and a potential difference of electrode (vertical axis) at different temperatures. The isothermal intersection point of an ideal electrode is represented at the zero point (0 mV) with pH=7 for different temperatures. For real electrodes the individual potentials which contribute to the overall potential output have different temperature coefficients of variation. So the isothermal intersection point hardly ever coincides with the zero point of the electrode Figure 7 represents an ideal and real isothermal intersection points. This variation in an isothermal intersection point due to the temperature effect causes an error. This error increase when the temperature difference between measurements sample and calibration buffer increase. To reduce this error it is better to perform the calibration and measurement at the same temperature [25].

Effect of Temperature on Response Time of the Electrode

When the temperature of measuring solution change rapidly during measurement or when pH electrode exchange between different measuring solutions with different temperature, the pH electrode will drift until the temperature of the electrode and the measuring solution become equal. This leads to slower response of the electrode. To reduce this effect it is proposed to use symmetrical lead-off pH electrode instead of conventional pH electrode. Figure 8 shows the difference between symmetrical lead-off pH electrode and conventional pH electrode [11].

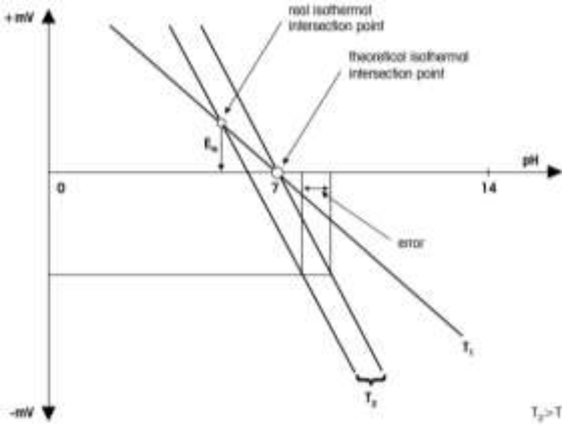


Figure 7: Ideal and Real Isothermal Intersection Points With Temperature Effect [11]

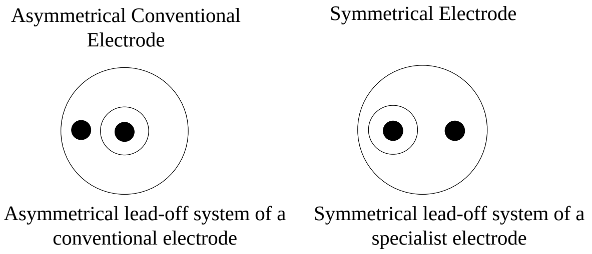


Figure 8: Sectional View of Asymmetrical and Symmetrical Combination Electrode [11].

Difference in Response for pH Electrode and Temperature Sensor

For the purpose of automatic temperature compensation, the temperature sensor must be used. When the pH electrode and temperature sensor are immersed into a measuring solution that varies significantly in temperature, the measurements can drift. This is due to the fact that temperature response of the pH electrode and temperature sensor may not be similar and the measuring solution may not have a uniform temperature. So the pH electrode and temperature sensor are responding to different environments [26, 27]. To overcome this effect it is useful to use a pH electrode with built-in temperature sensor.

pH Measurement Data Acquisition System

There are various researches dealing with pH data acquisition such as [1, 2, 29-31]. Ramya et al [1] have proposed the pH data acquisition and data logging system using a peripheral interface controller (PIC) microcontroller without using external memory to log the data. The system including the power supply subsystem to convert AC (Alternating current) to DC (Direct current) with step down transformer. However, the transformer has a large weight and size which increase the size and the weight of the device; also a direct connection to an AC source limits the portability of the device.

In addition the proposed system does not take into account the temperature effect compensation.

Chaitanya et al [29] proposed the pH data acquisition and data logging using Renesas M16C/62P microcontroller with a built in 10-bit analog to digital converter (ADC), a 4 Kbyte on chip flash memory on chip electrical erasable programmable read only memory (EEPROM). The proposed system used a timer to take the readings at the regular intervals and has the capability to set the time to control the power consumption. Besides, this system’s design is based on a lookup table which is recorded in the system.

The limitation of this design is that it is implemented based on a lookup table that reduces the accuracy of the measurements; besides this system is suitable only for processes that need to take a readings frequently at regular intervals. It does not include a mechanism for temperature effect compensation.

Saleha *et al* [2] have proposed an embedded-based soil pH meter which used the PIC18F4550 microcontroller connected to a personal computer (PC) or laptop through a serial or universal serial board universal serial bus (USB) interface to transfer the digital data of the measurements performed in a PC using a MATLAB program as host program. This method has many advantages such as it can be used to control process parameters in production of chemicals, fertilizers and food. The disadvantage of the above proposed solution depends on the PC that limits its portability and that it may not satisfy real-time constrains needed to perform measurement. This system also does not take into account the temperature effect compensation.

Paul *el al* [30] has designed an on-chip digital pH meter identical to the Motorola one in its functionality. This system is designed to be used in wireless diagnostic capsule application. The system is divided into two subsystems (digital subsystem and analog subsystem). The Motorola MC68HC05 was selected as a controller; it has a relatively simple architecture and small instruction set, 2 Kbyte on chip static random access memory (SRAM), and it uses an ISFET as a sensing element of the analog subsystem. The ISFET will only work with pH meter, which are adapted to the ISFET technology and is not interchangeable with meters using conventional pH glass electrode. Moreover there is no temperature compensation mechanism.

Gaytri et al [31] have described the experimental setup that must be taken to perform measurement in pH measurement system. The proposed system is in-house low-cost and based on ADu814 microcontroller and ISFET sensor. The system utilized a radio frequency (RF) modem (Zigbee) interfaced to micro controller by RS232 and connected to PC to perform continuous monitoring. Moreover, the system can be monitored by PC or locally by liquid crystal display (LCD). It takes into account the temperature effect compensation. However an ISFET sensor has many disadvantages compared with pH glass electrode as mentioned above.

pH Electrode calibration

Calibration is performed to compensate the potential change between the measuring and reference electrodes. To compensate for the pH electrode performance degradation over time, it must be recalibrated from time to time. This time period depends on the application and its unique conditions [4]. Always the pH electrode is characterized by its zero point (at pH=7.0) and its slope, and a two point calibration is chosen rather than a one point calibration for greater accuracy [11].

The changes in potential between measured electrode and reference electrode can be caused by one or more of the following factors [4, 11]: electrolyte depletion, reference junction blockage, chemical attack of the Ag/AgCl wire, contamination of glass bulb or sensitive gate of ISFET, and contamination of the reference electrolyte solution

In fact the calibration of the pH electrode is performed using a standard buffer solution. Since the buffer solution is also affected by temperature, its effect must be taken into account. It can be compensated for using a tabular method by storing the standard buffer solution values at different temperatures in meter's memory in order to select the buffer value that corresponds to the measured temperature or using polynomial interpolation by applying the equation that represent the relationship between the buffer solution and temperature.

pH meters designed based on ISFET electrode

The glass electrode is used nowadays in environment monitoring specially in wastewater treatment. However, the ISFET is the best one to be used due to contamination of wastewater that affects the response of the glass electrode. Cecilia Jimenez-Jorquera *et al* performed a comparison between ISFET and glass electrode in wastewater treatment and found that the ISFET is the most suitable for environmental application such as wastewater treatment [22]. In some medical applications, the meter is disposable and requires a small size such as gastric acidity monitoring. In such applications, the glass electrode is not suitable. The use of ISFET is preferred due to its small size and low cost. Ph. Duroux *et al* recommend using ISFET in the ambulatory pH monitoring for such gastric acidity monitoring [21]. Dhiraj *et al* [32] were performing design and fabrication of ISFET for bio-medical application. The pacemaker company Cordis in Roden, in the period 1975-1985, has designed a catheter with built-in pH sensor based on pH ISFET chip [33]. The ISFETs are also suitable for application that needs a small size of sensor such as system on-chip pH meter which can be integrated on-chip.

In the field of wireless communication the ISFETs are preferred, while the wireless system is always constrained by low power consumption and small size. Paul *et al* [30] have designed a system on-chip digital pH meter used in wireless diagnostic capsule based on ISFET. Gaytri *et al* [31] have suggested a microcontroller based pH measurement system with wireless communication using ISFET sensor.

In the portable or pocket pH meter the size is important parameter. The ISFET can be a small in size. Hashim *et al* [19] have proposed a microcontroller-based portable digital ISFET pH meter in the field agriculture using wireless sensor network (WSN). The ISFET is preferred where it can be adapted to measure the nutrient solutions. Kshitij *et al* [17] proposed a low power pH sensor based on ISFET for wireless sensor network node suitable for used in automatic wireless chemical monitoring in aagricultural applications.

pH meters designed based on glass electrode

As mentioned above, the glass electrodes are used in many applications; due to their simplicity, which are greatly used by researchers that develop a prototype of their proposed system. *Stephen Kenneth* [24] designed an automated pH monitoring system to be used in greenhouse agriculture. This design was based on a glass electrode and its system requirements are fast response time, robust and accurate, since the ISFET offer robustness and fast response time. The ISFET is most suitable for this system although the glass electrode offers higher accuracy. The glass electrode is also used in some designs in the field of embedded data acquisition. SalehaBegum *et al* [2] have implemented an embedded instrumentation based on soil pH measurement system by using a glass electrode. Chaitanya *et al* [29] have designed a pH data acquisition and logging device by using epoxy electrode.

Ramya and Palaniappan [1] also used a glass electrode to design and develop a prototype of embedded pH data acquisition and logging. The ISFET is preferred in these designs; due to its small size, which is an important criterion in an embedded system design.

CONCLUSIONS

The measurement accuracy of pH is affected by the temperature in different ways, some of them can be compensated and the others cannot be compensated; however, it can be avoided. The dominant one is the effect on the slope of pH electrode that can be considered as a significant source of error when pH measurement is done without temperature compensation. From the sensors point of view, the ISFET is the most pH electrode suitable for the enormous applications today such as: wastewater treatment, disposable application, portable pH meter, food processing and agriculture; due to its advantages. However, it becomes less selected if the does not need to minimize the size. Although the glass electrode is convenient, more accurate and simple to deal with, however, the ISFET promises and becomes the most economical and most convenient pH sensor in future.

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