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MEASUREMENT SYSTEM FOR ISE & ISFET SENSORS

Master Thesis

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PhD

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TALLINNA TEHNIKAÜLIKOOL
Infotehnoloogia teaduskond

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ISE & ISFET SENSORITE MÕÕTMISSÜSTEEM

Magistritöö

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Author’s declaration of originality

I hereby certify that I am the sole author of this thesis. All the used material, references to the literature and the work of others have been referred to. This thesis has not been presented for examination anywhere else.

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30.04.2019

Abstract

Fast determination of key chemicals in the human body helps medical professionals diagnose major diseases. ISE and ISFET sensors are used in detecting many of these chemicals. These sensors are also effective in finding impurities in water and soil i.e. they have many applications. But they have limitations with no reliable measurement method outside a laboratory environment.

This thesis gives design and development of a measurement system for the ISE and ISFET sensor which is accurate and reliable when used both indoors and outdoors, with the help of a mobile application. The problem involves two vital issues: one is to develop a potentiometry circuit which is portable and secondly to reduce the noise which will be produced outside a galvanized box.

A low voltage high impedance amplifier is used for the potentiometric measurement and the analogue current is converted using an ADC. For noise reduction Savitzky-Golay algorithm is used which can filter the noise without losing the signal structure. Raspberry Pi controller reads the data from the sensor using the potentiometric circuit and sends it to mobile application for processing.

The results suggest the measurement method is accurate and reliable when comparing to the lab results and it can be developed further to use commercially for medical and environmental diagnostics.

This thesis is written in English and is 46 pages long, including 8 chapters, 19 figures and 2 tables.

Annotatsioon

ISE & ISFET Sensorite Mõõtmissüsteem

Inimkehas olevate peamiste kemikaalide kiire määramine aitab meditsiinitöötajatel diagnoosida põhilisi haigusi. ISE ja ISFET andureid kasutatakse paljude nende kemikaalide tuvastamiseks. Need andurid on tõhusad ka vees ja pinnases lisandite leidmisel, st neil on palju rakendusi. Kuid neil on piiranguid, kuna ei ole usaldusväärseid mõõtmismeetodeid väljaspool laborikeskkonda.

Selles töös on toodud disain ja arendus ISE ja ISFET anduri mõõtesüsteemile, mis on mobiilirakenduse abil täpne ja usaldusväärne nii siseruumides kui ka väljas. Probleemil on kaks olulist komponenti: esiteks välja töötada potentsiaalomeetria vooluahel, mis on kaasaskantav ja teiseks vähendada müra, mis tekib väljaspool galvaniseeritud kasti.

Potentsiomeetriliseks mõõtmiseks kasutatakse madalpinge suure impedantsi võimendit ja analoogvoolu muundatakse ADC abil. Müra vähendamiseks kasutatakse Savitzky-Golay algoritmi, mis filtreerib müra ilma signaalstruktuuri kaotamata. Raspberry Pi kontroller loeb anduri andmeid potentsiomeetrilise ahela abil ja saadab selle töötlemiseks mobiilirakendusele.

Tulemused näitavad, et mõõtmismeetod on laboritulemustega võrreldes täpne ja usaldusväärne ning seda saab edasi arendada kommertskasutuseks meditsiinilise ja keskkonnadiagnostika alal.

See töö on kirjutatud inglise keeles ja on 46 lehekülge pikk, sisaldades 8 peatükki, 19 skeemi ja 2 tabelit.

List of abbreviations and terms

DPI	Dots per inch
TUT	Tallinn University of Technology
ISA	Institute of Analytical Sciences
UT	University of Tartu
ISE	Ion Selective Electrode
ISFET	Ion Sensitive field effect Transistor
SRS	System Requirement Specifications
ICI	Independent component analysis
RPI	Raspberry Pi
IOT	Internet of Things
ADC	Analogue to Digital Convertor
EIS	Electrochemical impedance spectroscopy
FR	Functional Requirements
NFR	Non-Functional Requirements
SVD	Singular value decomposition
FFT	Fast Fourier Transformation
I/O	Input and Output
uA	Micro Amperes
nA	Nano Amperes
USB	Universal Serial Bus

Keywords

Electrochemical sensor, ISE sensor, ISFET sensor, Internet of Things, Remote chemical analysis, Noise Filtering

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1 Introduction

Medical professionals increasingly rely on fast determination of key chemical species such as dissolved oxygen, carbon dioxide, ionized calcium and potassium in the blood, saliva, breath and other biological fluids. Many of the common ions found in biological fluids (Sodium, Potassium, Calcium etc) are determined by Ion-selective electrodes (ISEs) and Ion Sensitive Field Effect Transistor (ISFET) sensors. As the understanding of the impact of these chemical species on human wellbeing and health, improves the quantification of these chemical species receives continuously more attention. At the same time sensing technology is improving and affordable solid-state sensor are available. Intensified by the success of IOT and smart sensors, real time analysis of certain valuable biomarkers through reliable biosensors can play an important role in the early diagnosis of diseases.

ISE and ISFET sensors are widely used and a lot of research is carried out on the development of these sensors now a days. But the use of these sensors is limited to the Laboratory environment because of large apparatus and unstable reading caused by electrical noise if used outside a galvanised box.

In this work, we develop a simple and accurate measurement system for ISE and ISFET sensors, which can be used outside a Laboratory Environment. The system is based on interfacing the sensors with a raspberry pi which work as a controller and reads the data from the sensors. Raspberry pi sends the readings from the sensor to an Android application through Bluetooth. The android application processes the data, reduce the noise and display the real time measurements from the sensor.

1.1 Research Goals

The goal of the research is to develop an application which can measure the ISE and ISFET sensors in real world environment. Find a Noise Filtering Algorithm suitable for reducing the Electrical noise produced outside a galvanised box. Develop an Embedded system which interface the sensor and the mobile application both.

1.2 Working of ISE and ISFET sensors

ISE and ISFET sensors are chemical sensors which detects the concentration of a chemical dissolved in a solution.

The working of ISE sensor is based on the potential difference created between the electrode containing the membrane of a selective chemical and the reference electrode, whose potential remains constant, when the sensor is dipped in a solution. This potential difference changes with the change in the concentration of the solution. If we measure this potential difference, we can calculate the concentration of the specific chemical in the solution. The following figures show an ISE sensor.

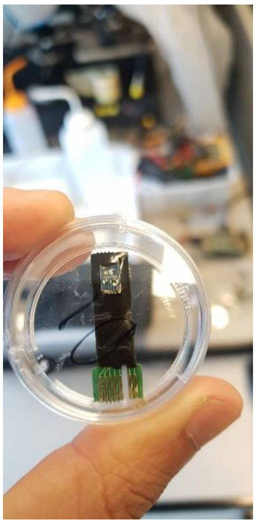


Figure 1: ISE Sensor

ISFET sensor works as a transistor with current flowing from the source to the drain when it is dipped in a solution. The amount of the current flowing from source to drain changes as the concentration of the solution changes. The concentration of the solution can be calculated by measuring this current flowing from source to drain. Following figure shows an ISFET sensor.

Figure 2: ISFET Sensor

The difference between the two sensors is that ISE sensor is selective in nature and it only measures the concentration of the chemical for which the membrane of the electrode inside is made for. On the other hand, ISFET is generic and it measures the concentration of the solution overall.

The limitation of using these sensors is that they are highly sensitive to electrical noise since the sensor read a very low potential difference and any change in the environment nearby can produce magnetic or electric field which can affect the measurement.

1.3 System Description

The application is developed to measure the ISE and ISFET sensors, both indoor and outdoor, accurately. The application receives the sensor data from the controller which is Raspberry pi. Raspberry pi is interfaced with the sensor using a circuit board we designed. It reads the voltage from the sensor and send it to the mobile application through Bluetooth.

The Mobile application is paired with the raspberry pi and exchange the sensor data continuously. Mobile application runs the noise reduction algorithm on the sensor data and try to produce stable and accurate result. Based on the potential difference after reducing the noise the concentration of the solution is calculated.

The user needs to select which sensor is going to be used. The user then needs to calibrate the sensor before using it for the first time. After the calibration process the system ask for the name of the chemical being measured and then the user can start measuring the concentration of the specified chemical. Following figure shows Raspberry pi connected with the circuit designed.

Figure 3: Raspberry Pi with the Developed Circuit

2 Background and Related Work

2.1 Chemical Analysis Using Mobile Application

As the literature review on different research suggest a significant progress on using the smart phones and devices as a remote chemical analysis tool and have huge application in the field of medicines in detecting major disasters, but also the growing number of these mobile applications produce a need to put a check on the quality of the services as there can be life depending on the results of the applications; whether they are working real time or just as an analysis tool.

(Hongying Zhu, 2013) developed a cost effective and rapid blood analysis system on a cell phone which measures the density of red and white blood cells using a blood sample of only 10uL. The circuit designed is attached to the camera of the mobile to take microscopic images of blood sample and process the data using custom designed application and measure the haemoglobin concentration in less than 10 second. The data can then share on a cloud with a laboratory for further analysis.

(Hooman Hafezi, 2015) developed an ingestible sensor for measuring medication adherence. This paper presents a novel sensor for detecting the ingestion of pharmaceutical tablets and capsules. According to the paper 30 to 50 percent of the drugs prescriptions are never taken which results in complications of patient’s health. The sensor developed can be integrated with every tablet and capsule manufactured and on digestion it detects the chemicals in the stomach and send a signal to a mobile application of the intake.

(AntonioGarcía, 2011) present mobile phones as a platform for portable chemical analysis. This platform uses the built-in camera of mobile phone. A potassium sensor is used which is based on ion exchange equilibrium. The application developed to analyse the hue change of the sensor by taking the images and processing them to analyse the concentration.

(ZafarIqbal, 2011) presents analysis of water and sand sample using a mobile phone. The screen of the mobile device was used as a light source and the front camera was used as a detector. The reflected intensities of the RGB and white light were used to analyse and classify different components present in the water. Water impurities can cause major risks of different diseases. Detecting impure water using a potable mobile device is helpful in a common person's life.

(M. O. Salles, 2014) presented the research on explosive colorimetric discrimination using a smartphone. In their paper they introduced an approach of using a colorimetric paper sensor array for detecting and discriminating five different explosives. The colorimetric sensor gives different colour for each explosive and thus can be classified on the basis pf using a mobile application made specifically for the purpose.

(Ali Kemal Yetisen, 2014) discuss the regulation of mobile medical applications in a paper published in Lab on a chip in 2014. They mention the guidance issued by U.S food and drug administration to regulate these applications and protect consumers by minimising the risk of unintended use. According to the paper awareness and incorporation of the guidelines into product development can hasten the commercialization and market entry process.

2.2 Literature Review on ISE and ISFET

(N. Ramanathan, 2006) describe a procedure for calibration of ISA sensor. They designed a tool to use in field, to detect potential faulty data and suggest remedies. As in the real time, sensing the data is very small and small fault in a reading can give wrong results. The author discusses the calibration of ISE sensor with standard concentrations. For ISEs, the electric potential relates linearly with the logarithm of the concentration. The R2 value of the resulting linear regression gives a measure of the confidence to be placed in concentration values translated from voltage readings. According to the author the in-field tools subsequently designed will allow to interact with future deployments and greatly improve the confidence in the quality of data collected.

(Matti Kaisti, 2016) describe a hand-held sensing system using a transistor-based platform and a detector that connects electrochemical information wirelessly to a smartphone. It is based on ISFET technology. They compare the sensing system with commercially available Potentiometers, and it was proven to be more effective than those.

(Poonia, 2018) present a prototype for a wireless system that sense NPK from the soil and farmers can check the information in the android application. The nutrients Nitrogen Phosphorous and Potassium are very important for plant growth and the deficiency of these in soil can cause be harmful for crops. The authors used ISE and ISFET sensor to detect these in soil and send the information wirelessly to the mobile application, so the farmers don't have to go to the laboratories for the process.

2.3 Related Work on ISE and ISFET sensor

Hamdi Ben Halima a PhD student at Institute of Analytical Sciences, Lyon works on detection of different proteins including Tumor necrosis factor alpha (TNFa), Interleukin 10 (IL 10), N-terminal prohormone of brain natriuretic peptide (NT-Pro BNP) and Cortisol using ISFET sensor under the supervision of Prof Errachid who is also the supervisor of this report. Quantity of these proteins in human body can help diagnose and predict heart failures. There are several instrumentations to detect these proteins which require time and are not accurate enough. Hamdi works on detection of these proteins using ISFET sensor, which is cheaper, fast and reliable. Following figure shows the characterization of ISFET sensor using different pH by Hamdi Ben Halima.

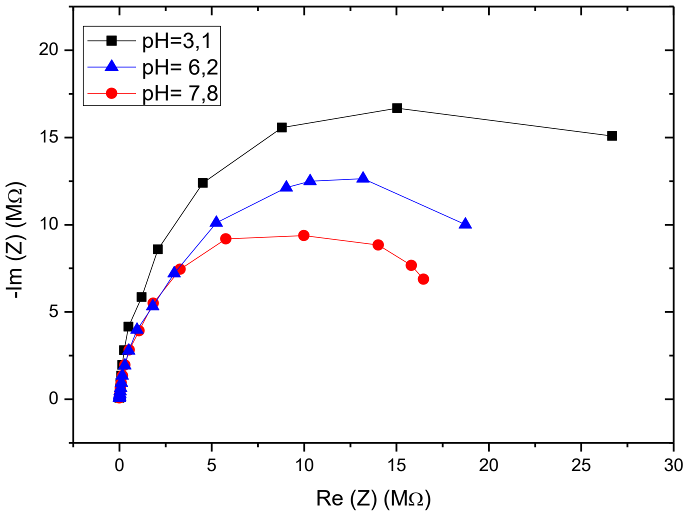


Figure 4: Characterization of ISFET using different pH.

2.4 Noise Reduction Techniques

There are many real time applications on signal processing especially in biomedicine and Nano-biotechnology. These applications make use of biosensors where each sensor electrode read the data with a mix of useful and noisy information. Noise cancellation is a subject of wide interest and there are several methods proposed in literature review for noise reduction and filtration. The techniques include band-pass filtering, the fast Fourier transform, autocorrelation, autoregressive modelling, adaptive filtering, Kalman filtering, and singular value decomposition (SVD). (Sergiy Vorobyov, 2002) discuss a noise reduction technique using independent component analysis ICA and subspace filtering. In their approach they apply subspace filtering not to the raw data but to a mixed version of data obtained by ICA. Finite impulse response filters are employed whose vectors are parameters estimated based on signal subspace extraction. ICA allows to filter independent components. After the noise is removed, they reconstruct the enhanced independent components to obtain clean original signals, so they project the data to the sensor level.

(HEATH, 2017) In Band pass filtering technique, there is a circuit deigned which allows signal of certain frequency to pass through it and does not allow the rest to pass. With band pass filter anything higher or lower than the frequency range will be blocked. This technique can be used to reduce high electrical noise and sudden peeking of a signal with the environmental changes around.

Fast Fourier Transformation technique works by dividing N point time domain signal into N signals and find the Spectrum of each N domain signal and synthesize an N frequency spectrum into a single frequency spectrum. FFT is widely used in noise reduction but the limitation of the technique is that it requires high processing power and memory for recursive division of N points.

Autocorrelation is correlation of a signal as a delayed copy of itself. The analysis of this autocorrelation can help finding noise in a signal. (Farahani, 2017) works on autocorrelation-based noise subtraction method with smoothing.

An adaptive filter is a digital filter that is self-adjusting. It adjusts the filter coefficient automatically to adapt the input signal via an adaptive algorithm. (Ed. Vijay K. Madisetti, 1999) By choosing an adaptive filter structure, one specifies the number and type of parameters that can be adjusted. The adaptive algorithm used to update the parameter values of the system can take on a myriad of forms and is often derived as a form of optimization procedure that minimizes an error criterion that is useful. The adaptive filter is very commonly used in noise reduction and echo filtering.

Kalman Filter is one of the most important and common estimation and filtering algorithms. T[1]he Kalman Filter produces estimation of hidden variables based on inaccurate and uncertain measurements. As well, the Kalman Filter provides a prediction of the future system state, based on the past estimations. The filter is named after Rudolf E. Kalman. In 1960, Kalman published his famous paper describing a recursive solution to the discrete-data linear filtering problem. The Kalman filter is used in tracking location and navigation systems, control systems, computer graphics and much more.

[1] https://www.kalmanfilter.net/default.aspx

3 System Requirement Specification

A real time system has been implemented for the measurement of the chemicals in a solution. The sensor is calibrated with the system by dipping it into two known concentration solutions after the sensor is calibrated the solution of unknown concentration can be measured. The system constitutes of two independent parts the mobile application developed on android and the Raspberry pi application developed using Python and interfaced with the circuit designed to interact with the sensors.

3.1 Functional Requirements

Following table shows the functional requirement for the system which is developed.

Table 1: Function Requirements for the System

Reference No.	Description of Function	Category
Req-1	System should be able to turn on the Bluetooth of the device on which application is active.	Joint I/O
Req-2	System should be able to turn off the Bluetooth of the device on request.	Joint I/O
Req-3	System should show the already paired devices.	Output
Req-4	System should generate the list of available devices within Bluetooth range including Raspberry pi.	Output
Req-5	System should be able to successfully pair with the Raspberry pi as its controller.	Performance
Req-6	System should allow the user to calibrate the sensor.	Joint I/O
Req-7	System should allow the user to enter the concentration data for the two solutions of the known concentrations one by one.	Input
Req-8	System should ask the user to dip the sensor in two solutions of the known concentrations one by one.	Output
Req-9	System should send the signal to Raspberry pi.	Output

Req-10	System should then fill the fields with the sensor readings obtained from Raspberry pi.	Joint I/O
Req-11	System should ask user to dip the sensor in the solution of unknown concentration.	Output
Req-12	System should be able to read the potential difference from the designed circuit.	Performance
Req-13	System should use an amplifier to amplify the potential difference.	Performance
Req-14	System should convert the analogue potential voltage to digital voltage using the connected Analogue to Digital Converter (ADC).	Performance
Req-15	System should read the digital voltage from the ADC using Adafruit library of Raspberry pi.	Input
Req-16	System should ask for the chemical name for which the concentration has to be measured.	Joint I/O
Req-17	System should be able to run the noise reduction algorithm and reduces the noise.	Performance
Req-18	System should calculate the concentration of the solution using the potential difference.	Performance
Req-19	System should then successfully produce stable and accurate results.	Performance
Req-20	System should be capable of generating real time graphs between time and voltage.	Performance

3.2 Non-Functional Requirements

The following table shows the Non-Functional Requirements for the system under development.

Table 2: Non-Function Requirements for the System

Reference No.	**Description**
NFR-1	The system will be available 99% of the time.
NFR -2	Application is user-friendly.
NFR -3	The average response time of the system is around 0.5 seconds.
NFR -4	The system gives an accuracy of 0.1 pH for ISFET sensor.
NFR-5	The system Gives an accuracy of 0.0001 Moles for ISE sensor.

3.3 Use-Cases

Following are some use cases for the system:

<table>
<tr><th>Use-Case</th><th colspan="2">Description</th></tr>
<tr><td rowspan="6">Turn on Bluetooth</td><td>Actors</td><td>User</td></tr>
<tr><td>Overview</td><td>The system will allow the user to turn the Bluetooth on or off using the app.</td></tr>
<tr><td>Priority</td><td>Low</td></tr>
<tr><td>Preconditions</td><td>Application should be installed and launched.</td></tr>
<tr><td>Success Guarantee</td><td>The Bluetooth will be turned on.</td></tr>
<tr><td>Typical Course of Events</td><td>• User select the button to turn on the Bluetooth.
• System ask the permission to turn on the Bluetooth.
• User grant the permission to turn on the Bluetooth.
• System turn the Bluetooth on.</td></tr>
</table>

	Alternative Course of Events	• User select the button to turn on the Bluetooth. • System ask the user to allow the application to turn on the Bluetooth. • User rejects the permission. • System will not turn the Bluetooth on.
Discover new device	**Actors**	User
	Overview	The system will allow the user to discover new Bluetooth devices nearby
	Priority	High
	Preconditions	Application should be installed and launched. Bluetooth is turned on.
	Success Guarantee	User will see a list of all the available devices nearby.
	Typical Course of Events	• User select the button to discover new devices nearby. • System start searching for nearby devices. • System displays a list of all the nearby devices.
	Alternative Course of Events	• User select the button to discover new devices nearby. • System ask the user to allow the application to turn on the Bluetooth. • User grants the permission to turn on the Bluetooth. • System start searching for the nearby devices. • System displays all the available Bluetooth devices nearby.
Pair a device	**Actors**	User
	Overview	The system will allow the user to pair with the raspberry pi after showing a list of available devices nearby

	Priority	High
	Preconditions	User should have a list of all the nearby Bluetooth devices
	Success Guarantee	When pairing is complete the device name will appear in the list of paired devices
	Typical Course of Events	• User select one of the nearby available Bluetooth devices. • System try to authenticate pairing from the device. • System successfully pair with the selected device if authentication is success.
	Alternative Course of Events	• User select one of the nearby available Bluetooth devices. • System try to authenticate pairing from the device. • Device rejects the pairing request.
Calibrate the Sensor	**Actors**	User
	Overview	The system will allow the user to calibrate the sensors before using it.
	Priority	High
	Preconditions	The application should be connected to Raspberry pi using Bluetooth and the sensor is ready to use.
	Success Guarantee	The sensor will accurately show the concentration of the solution whose concentration is unknown.
	Typical Course of Events	• User select the option to calibrate the sensor. • System ask the user to dip the sensor in a solution of known concentration and input the concentration of that solution. • System measure the potential difference of the known concentration solution.

		• System asks the user to dip the sensor in another solution of known concentration and input the concentration value. • System will again measure the potential difference from the known concentration solution. • System will ask the user to dip the sensor in any unknown concentration solution. • System will display the concentration of the solution which is unknown.
	Alternative Course of Events	
Measure Concentration	**Actors**	User
	Overview	The system will allow the user to keep measuring the concentration of solutions after the calibration.
	Priority	High
	Preconditions	The sensor is calibrated.
	Success Guarantee	The sensor will accurately show the concentration of the solution whose concentration is unknown.
	Typical Course of Events	• User select the option to display the concentration of solution. • System ask the user to the dip the sensor in a solution of unknown concentration. • System measure the concentration of the solution and display it.
	Alternative Course of Events	• User select the option to display the concentration of solution. • System ask the user to calibrate the sensor first.

<table>
<tr><td rowspan="7">Real Time Graph</td><td>Actors</td><td>User</td></tr>
<tr><td>Overview</td><td>The system will allow to see the real time graph of the voltage transformation with time and concentration.</td></tr>
<tr><td>Priority</td><td>High</td></tr>
<tr><td>Preconditions</td><td>The application is connected to the Raspberry pi using Bluetooth.</td></tr>
<tr><td>Success Guarantee</td><td>The sensor will show a real time graph of voltage over time without electric noise.</td></tr>
<tr><td>Typical Course of Events</td><td>• User select the option to display the real time graph.
• System sends the request to the Raspberry pi app to start sharing the sensor data over Bluetooth.
• System starts showing the voltage value over time.</td></tr>
<tr><td>Alternative Course of Events</td><td>• User select the option to display the real time graph.
• System detects that Bluetooth in the application is disconnected from Raspberry pi.
• System warns the user and ask to connect again.</td></tr>
</table>

4 ISE Sensor Measurement Methodology

The working of ISE sensor is based on the potential difference created between the electrode containing the membrane of a selective chemical and the reference electrode, whose potential remains constant, when the sensor is dipped in a solution. This potential difference changes with the change in the concentration of the solution. If we measure this potential difference, we can calculate the concentration of the specific chemical in the solution.

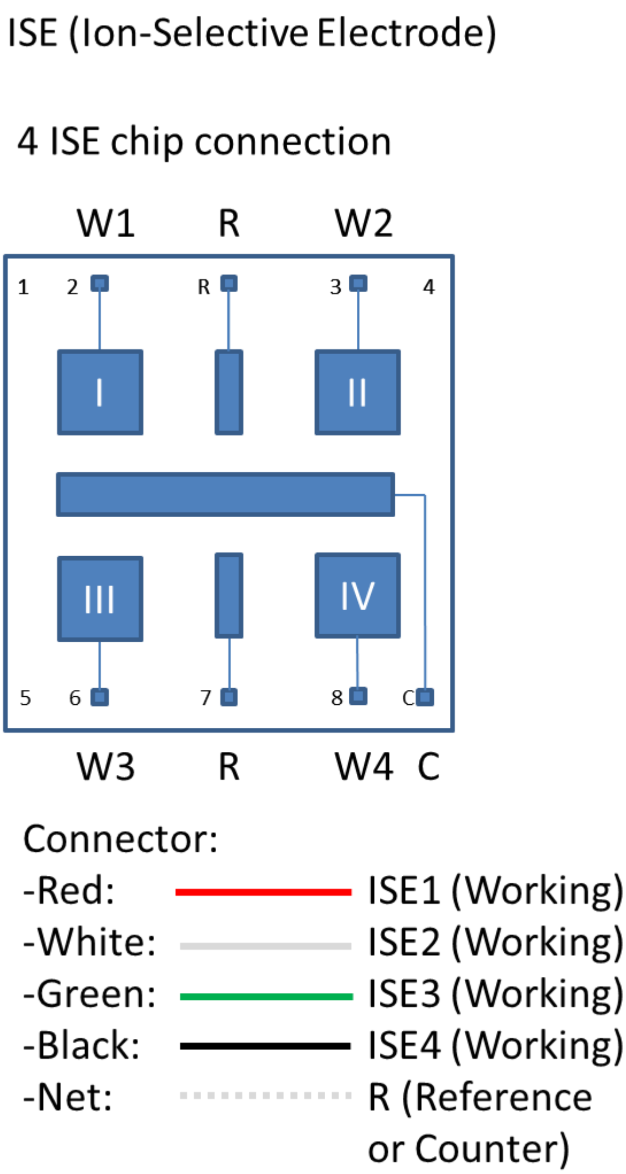


Figure 5: ISE CHIP

4.1 ISE Hardware

For potential measurement there are number of techniques that can be implemented. In Amperometry we either use a commercially available AFE like LMP91000 or build a potentiostat circuit. The benefit of LMP91000 is that the entire AFE is completely integrated into a single chip which consumes ~40 μW. (Pechlivanidis, Papadimitriou, Evans, Vasilakis, & Prodromakis, 2017) report current resolutions for their Bluetooth based biosensors of 400 pA and 122 PA, respectively.

Electrochemical impedance spectroscopy (EIS) is typically measured by applying a small sinusoidal voltage stimulus between electrodes and measuring the magnitude and phase of the resulting current signal at multiple frequencies. Due to the typical frequency range 1-100 kHz and the low 5-mV peak amplitude of the stimulus signal, EIS tends to be the most power consumptive measurement technique since it needs to accurately measure both magnitude and phase accurately from a small current signal at all frequencies within the spectrum.

Potentiometric measurement circuitry typically only requires an amplifier with a large input impedance to measure the voltage from an ion selective electrode known for its high resistance (10 MΩ–1 GΩ). (Zhang, Yang, Yang, Liu, & Gu, 2015), a USB powered non-inverting amplifier with a input current of ~20 nA is used and can achieve a LOD of α-amylase of 89 nM [14]. There also exists a commercial potentiometric measurement chip, i.e. LMP91200, which is essentially an ultra-low leakage buffer (600 fA), that has been used in audio-jack biosensors.

The sensor measurement will be performed using the well-known potentiometric method. For the implementation of this method we used following configurations.

Configuration for the Ion Selective Electrode (ISE) sensors, is based on one operational amplifier (op-amp) to acquire the potential difference between the sensor and the reference. In this case the op-amp is configured as a non-inverting voltage follower or buffer with a RC filter in the output. Following figure shows the schematic of the configuration:

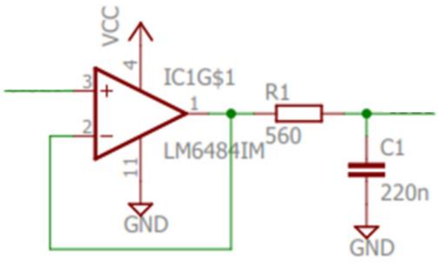


Figure 6: Non inverting voltage follower for ISE measurements.

In this non-inverting circuit configuration, the input impedance increases to infinity and the feedback impedance is reduced to zero. The output is connected directly back to the negative inverting input, so the feedback is 100% and the input voltage is exactly equal to the output voltage giving it a fixed gain of 1 or unity.

4.1.1 Amplifier

The amplifier used for the purpose is LM6484 (TexasInstruments, LMC6484 CMOS Quad Rail-to-Rail Input and Output Operational Amplifier, 2015). Low voltage characteristics and low power dissipation makes this amplifier well suited for the battery-operated system and in this case as it is powered by a Raspberry pi with 5.0 voltage. The LMC6484 has the high input impedance and circuits designed with it can reject a larger range of signals than most in-amps. This makes LMC6484 an excellent choice for (electric) noisy environments. The amplifier has four channel output and hence this system can measure four ISE electrodes using it. Following figure shows the input and output pins for LMC6484.

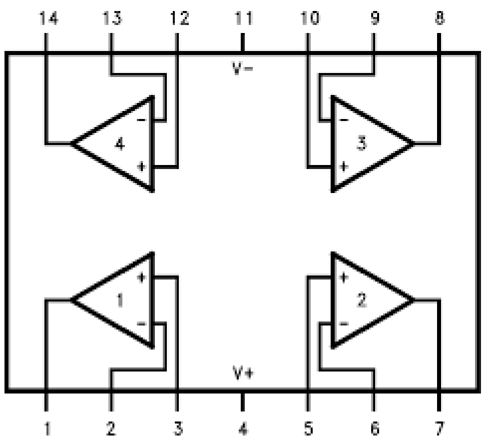


Figure 7: Pin out for LMC6484.

4.1.2 Analogue to Digital Converter

MCP3208 is a 12 bit Analogue to Digital convertor used in the circuit to convert the output analogue voltage from the LMC6484 amplifier to digital values which can be read by the Raspberry pi controller, since Raspberry pi cannot read the analogue values. The devices are capable of conversion rates of up to 100 kbps. The MCP3208 operate over a broad voltage range (2.7V - 5.5V) ideal for our circuit and it has 8 channels (Microchip, 2007). The first four channels of MCP3208 are being used for the four electrodes potentiometric analogue values provided by the amplifier. The following figure shows the schematic of the ADC.

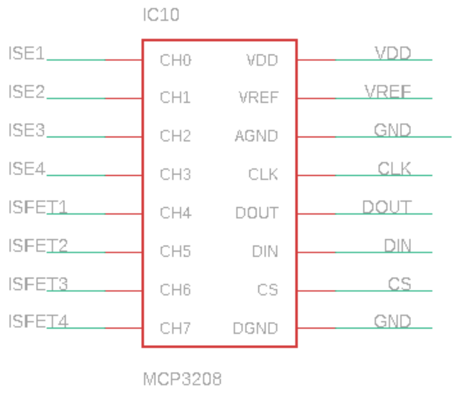


Figure 8: Schematic for ADC MCP3208

4.1.3 Sensor Interfacing

The sensor is connected to the circuit with 6 pins to an ISE jack port. Four pins represent the four electrodes of ISE sensor along with a pin for the reference electrode. The VDD is 2.5 volts current for the differential voltage circuit.

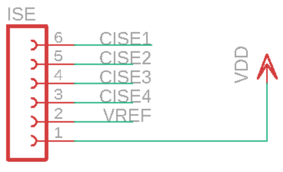


Figure 9 Schematic for ISE pin interface

4.2 ISE Software

ISE software works on two platforms. There is a python script in Raspberry pi responsible for reading the data from the MCP 3208 Analogue to Digital convertor and then connects with the Mobile application when requested. The mobile application is responsible for processing the data sent by the python script and calculates the concentration.

4.2.1 Python

The Python code open the serial port RFCOMM 0 for Bluetooth communication with the mobile application. RFCOMM is a simple transport protocol, which provides emulation of RS232 serial ports over the L2CAP protocol. [1] The RFCOMM protocol supports up to 60 simultaneous connections between two Bluetooth devices. For the purposes of RFCOMM, a complete communication path involves two applications running on different devices (the communication endpoints) with a communication segment between them. And that serves our purpose of continuous communication between Raspberry pi and the android application.

Python script uses the Adafruit Python MCP3008 library which is deprecated but is still usable for research purpose. Using the library, we read the 8 channel MCP3208. The read value is converted to voltage value. [2]The ADC assumes 5V is 1023 and anything less than 5V will be a ratio between 5V and 1023. We use the following equation to convert the read value to voltage in the python script.

$$\frac{1023}{5} = \frac{ADC\ Reading}{Analog\ Voltage\ Measured}$$

Python script keeps running in threads waiting for the connection with the mobile application. With a successful connection it waits for signal by the application to start sending the voltage data over the serial port.

[1] https://www.amd.e-technik.uni-rostock.de/ma/gol/lectures/wirlec/bluetooth_info/rfcomm.html#RFCOMM%20Overview/Service

[2] https://learn.sparkfun.com/tutorials/analog-to-digital-conversion/relating-adc-value-to-voltage

4.2.2 Mobile Application

The mobile application starts with the page for Bluetooth connection with the Raspberry pi and without that measurement or any action cannot be proceeded. For connecting with the Bluetooth on serial we use Bluetooth socket class and Bluetooth adapter in Java. Once the socket is created and the device is connected input stream and output stream is used in thread to read and send data continuously.

Once the application is connected with the Raspberry pi we can start measuring the sensor. The application gives the user option to measure the ISE sensor of ISFET sensor. In case the user selects the ISE sensor the application proceeds to the main page of ISE sensor. For calibrating the ISE sensor, we take two known concentrations from the user and measure the respective voltage with them. Using the following equation, we can measure any unknown concentration afterwards.

$$c_3 = c_2 - ((e_2 - e_3)/(e_2 - e_1) * (c_2 - c_1))$$

where c3 is the unknown concentration and c2 and c1 are the known concentrations input by the user. E1 is the potential difference voltage measured against c1 and e2 is the voltage measured against the concentration c2 while e3 is the voltage measures when the user dips the sensor in the solution of unknown concentration. The application plots a graph of all the points once the sensor is calibrated.

Figure 10: App showing graph points

The other option for the user is to see the real time graph of the ISE measurement. This is based on ISE standard addition and sample addition methods. In this case the application shows live voltage to time measurement graph. The following equation has been used to calibrate the sensor manually.

$$C_n V_n = C_{n+1} V_{n+1}$$

$$C_n = C_{n+1} V_{n+1} / V_n$$

The equation is used to find out the next concentration of solution we need to add. Where C_n is the concentration of the current solution, we can calculate the volume and concentration of next solution.

Following image shows the main page of ISE sensor measurement.

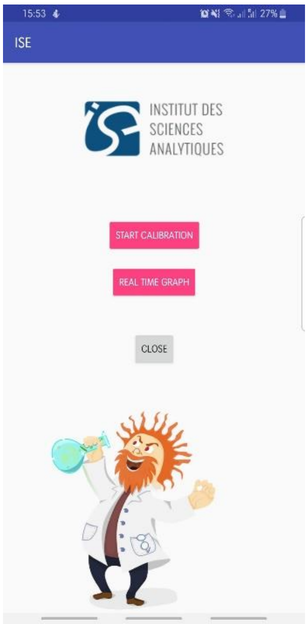


Figure 11: ISE sensor application interface

5 ISFET Sensor Measurement Methodology

ISFET sensor works as a transistor with current flowing from the source to the drain when it is dipped in a solution. The amount of the current flowing from source to drain changes as the concentration of the solution changes. The concentration of the solution can be calculated by measuring this current flowing from source to drain.

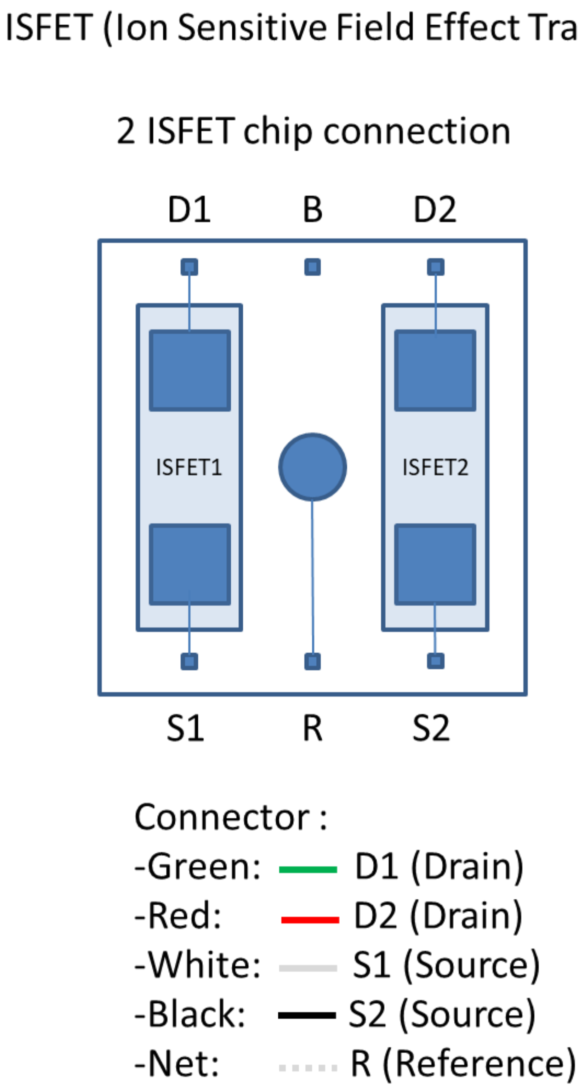


Figure 12: ISFET CHIP

5.1 ISFET Hardware

The configuration implemented for the Ion Sensitive Field Effect Transistor (ISFET) sensors, is the source and drain follower, which consist of a pair of op-amps and a resistor that fix the drain source voltage across the ISFET. One current source (S2) provides a constant current to the source terminal of the ISFET. This current only flows through the ISFET because the source terminal is connected to the noninverting terminal of an op-amp (OA2) that has very high input impedance as can be seen in the following figure:

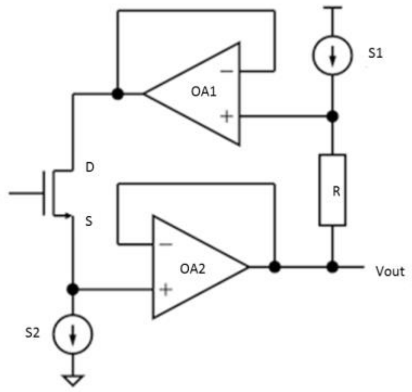
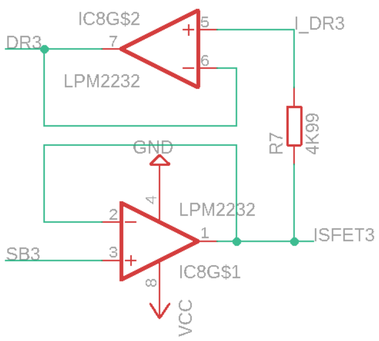


Figure 13: Source and Drain for ISFET measurement

The upper op-amp (OA1) output the source voltages to the upper terminal of the resistor (R). One current source (S1) draw a fixed current though the resistor, developing a potential drop. The lower op-amp (OA2) output the potential at the lower terminal of the resistor to the drain terminals of the ISFET. This circuit path ensures that a constant drain source potential difference is maintained. Hence, the output voltage is directly proportional to the variations that are represented by a change in the threshold voltage of the ISFET.

5.1.1 Amplifier

The amplifier used is LMP2232 Dual Micropower. The reason for using this amplifier is that it is designed for battery powered applications between the range of 1.5 to 5.5 volts and since the system is powered by the circuit using the Raspberry pi with 5.0 volts current.



(TexasInstruments, LMP2232 Dual Micropower, 1.6V, Precision, Operational Amplifier with CMOS Input, 2013) The LMP2232 is part of the precision amplifier family. The high impedance CMOS input makes it is ideal for instrumentation and other sensor interface applications. The input current noise of the LMP2232 is so low that it will not become the dominant factor in the total noise unless source resistance exceeds 300 MΩ, which is an unrealistically high value.

5.1.2 REF200

Ref 200 is used for sensor excitation and low voltage reference in the circuit. (Instruments, 2015) The REF200 device combines three circuit building-blocks on a single chip. Also, because the current sources are two terminal devices, they can be used equally well as current sinks. The performance of each section is individually measured to achieve high accuracy.

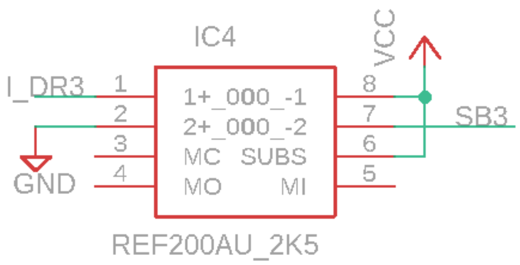
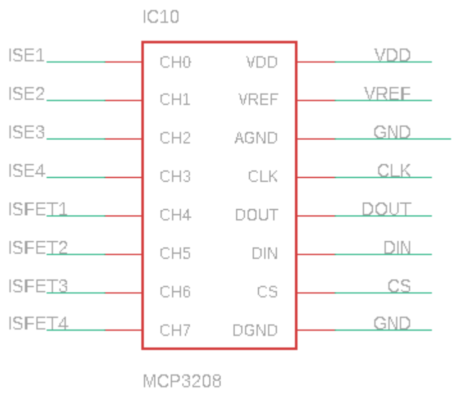


Figure 14: Schematic for Ref 200

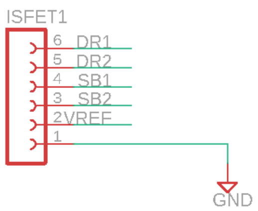
5.1.3 Analogue to Digital Conversion

MCP3208 a 12 bit Analogue to Digital convertor used in the circuit to convert the output analogue voltage from the amplifier to digital values which can be read by the Raspberry pi controller, since Raspberry pi cannot read the analogue values. The ADC mentioned before in the ISE sensor section 4.1.2 is used as both the sensors are using the same ADC. The last four channels of MCP3208 are being used for the four ISFET transistors. The following figure shows the schematic of the ADC with ISFET1, ISFET2, ISFET3 and ISFET4 connected to the last four pins of ADC.



5.1.4 Sensor interface

The ISFET sensor is interfaced with the circuit using 6 pins connector. There are two drains and two sources representing two ISFET sensors. Moreover, there is the reference pin and a ground.



5.2 ISFET Software

ISFET sensor is the second sensor the software measure. It receives the data on Bluetooth from the Raspberry pi, process and show the measurements in the mobile application.

5.2.1 Python

The python script for connecting to the Bluetooth is already discussed in section 4.2.1. When the app is connected the python, script wait for the option from the application to select while to go for ISE measurement or ISFET. Either selected value of the Raspberry pi will read the voltage from the respective pins of the ADC. In case of ISFET which is pins 4 to 7.

5.2.2 Mobile Application

The mobile application connects to the Bluetooth as discussed in section 4.2.2 of ISE sensor. If the user selects the ISFET sensor on the main page the applcation will send the appropriate message to the Raspberry pi python script to start sending the data from the ISFET sensor.

The ISFET measures the pH of the solution based on Voltage received from the sensors. Measurement is done based on 50 mV difference per pH value. The application shows the pH and voltage graph of the solution while characterization of ISFET sensor. Following figure shows a real time graph of voltage over time.

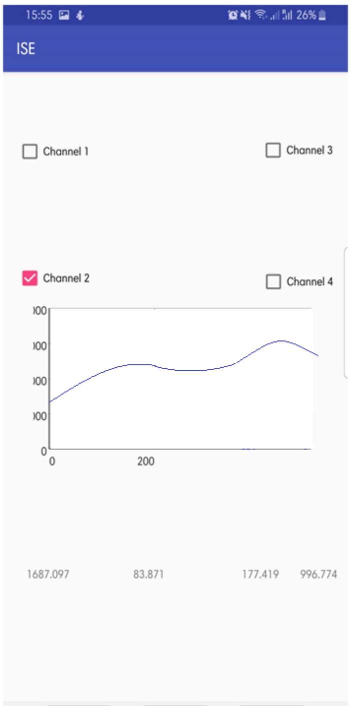


Figure 15: Real time graph for ISFET Sensor

6 Noise Reduction

The primary output of a measurement is information which measures phenomenon under observation. Along with this information, there are some error values which is called noise. This noise can be produced by a number of factor thermal noise because of heat, electric noise because of electromagnetic field around etc. Various algorithms in this research has been studied for noise reduction while measuring ISE and ISFET sensors. Savitzky-Golay algorithm proposed in 1964 is the one which is used for making the solution for the measurement.

6.1 Savitzky-Golay algorithm

(A. Savitzky, 1964) Savitzky and Golay proposed a method of data smoothing based on local least-squares polynomial approximation. 1Input points are fitted in a polynomial and the resulting polynomial is evaluated at a single point. These lowpass filter obtained by using this method are called Savitzky-Golay filters. Savitzky-Golay show that least squares smoothing reduces noise while maintaining the shape and height of waveform peaks. 2In the algorithm a set of integers (A-n, A-(n-1) …, An-1, An) could be derived and used as weighting coefficients to carry out the smoothing operation. These convolution integers are exactly equivalent to fitting the data to a polynomial, as just described and it is computationally more effective and much faster. Following is the equation for the algorithm

$$(y_k)_s = \frac{\sum_{i=-n}^{n} A_i y_{k+i}}{\sum_{i=-n}^{n} A_i}$$

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[1] https://inst.cs.berkeley.edu/~ee123/fa12/docs/SGFilter.pdf

[2] http://195.134.76.37/applets/AppletSmooth/Appl_Smooth2.html

Where $(y_k)_s$ is the smoothed data point obtained and $A_{i=-n} \ldots A_{i=n}$ are the set of integers derived and used as weighing coefficients. Y_{k+I} are the variable points from the noisy data being filtered out.

Smoothing effect of this algorithm is not so aggressive like other filters where a good amount of signal is lost while reducing the noise. In Savitzky-Golay Algorithm the shape of the signal remains same which is very important in our real time graph detection to detect change in voltage while increasing the concentration of solutions. The following figure shows smoothing of data using Savitzky Golay Algorithm[1].

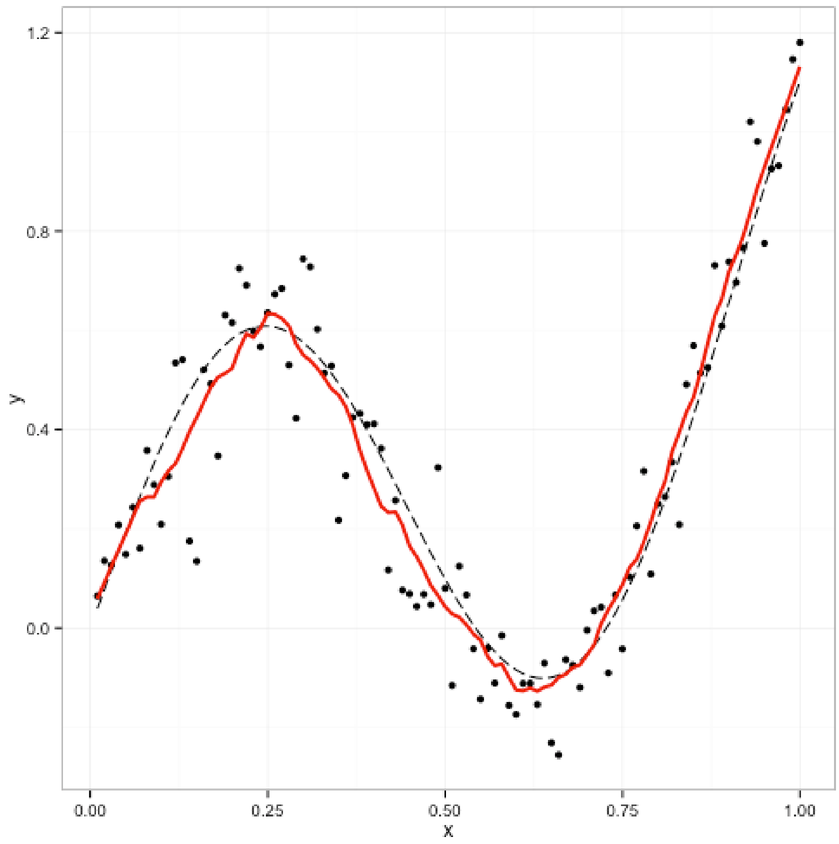

Figure 16: Smoothing data with Savitzky Golay Alorithm

[1] http://wresch.github.io/2014/06/26/savitzky-golay.html

In the figure the dotted points are the noisy data obtained. The dotted line shows the moving average algorithm applied to data which simply obtain the average and the signal shape is not maintained. The red line shows the smoothing by Savitzky-Golay algorithm and the signal shape is maintained. Small changes in the signal can be seen which is important when measuring a variable which is changing slowly which in our case is the voltage and concentration of the solution.

6.2 Results

The data received without using the algorithm was noisy. The following figure show the sensor voltage value over time.

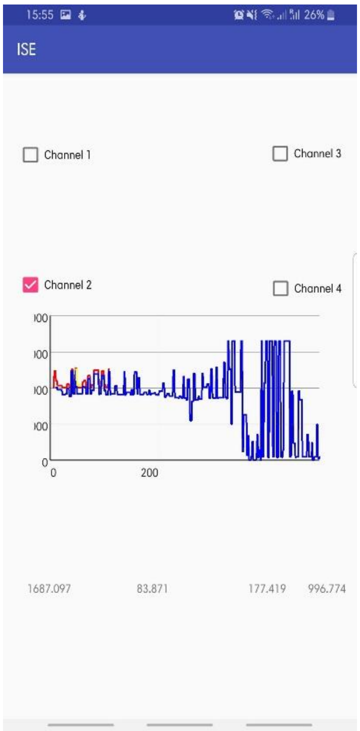

Figure 17: Noisy data from the sensor

After applying the filtering algorithm, the noise is reduced, and the signal value is clear. The sudden peak of voltage with the movement in environment are now eliminated. Following figure show the sensor data from ISFET measure over 4.6 pH and 2.2 pH value.

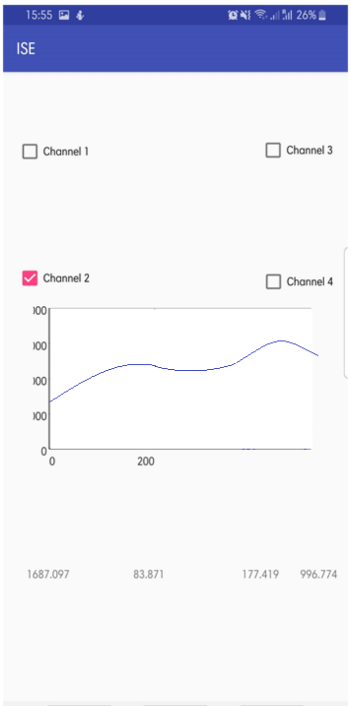

Figure 18: Real time graph with Filtering

Compared to a lab environment the potentiometry approach with the filtration of noise is very effective. Following figure show the reading of ISE sensor in the Lab with a potentiometer and compare to that the application with filtration has less noise fluctuations and more accurate data.

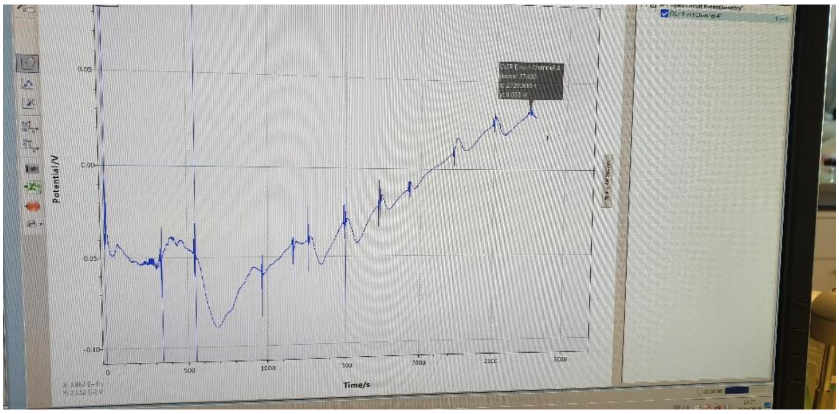

Figure 19: ISE measurements in Lab

7 Conclusion

7.1 Summary

The ISE and ISFET sensor are state of art technology with a lot of applications but they have limitation of use because of different environmental constraints. By using the potentiometry technique and noise reduction algorithm the application developed, reduce these hazards faced in the ISE and ISFET measurements. Further development on the circuitry and application design can improve the measurement and this ISE and ISFET measurement method can be used commercially.

7.2 Future Development

In future the design of the circuit developed can be reduced for remote use. The microcontroller used has big size also which can be replaced with smaller controllers.

Another idea is to do the potentiometry reading using the mobile USB port. Further research is undergoing to see if one can make use of the mini USB port of the mobile to detect the potential difference from the amplifier directly or with the use of a small microcontroller. In the case, a small pencil size circuit can be designed, which can connect with a mobile device anywhere and used as a remote measuring system using a manually developed mobile application.

Specialize application for measurement in soil and other maters can be developed using the same measurement system we have presented.

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