DESIGN AND DEVELOPMENT OF FLEXIBLE MEMS-BASED pH SENSOR FOR AGRICULTURAL AND BIOMEDICAL APPLICATIONS

By

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Abstract

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The purpose of this study is to demonstrate a novel method of using a flexible MEMS-based pH sensor for agricultural and biomedical applications. The first part of the thesis involves demonstration of the capability of our pH sensor for monitoring pH level change in the food product. The pH sensor we have developed consists of Iridium Oxide (IrOx) and Silver Chloride (AgCl) sensing electrodes. The sensor is designed to monitor and sustain long-term continuous usage, which makes it suitable for a wide variety of agricultural based applications. The electrochemical potential generated between IrOx and AgCl electrode is monitored and further correlated with corresponding pH values. In this embodiment, pH sensors are tested for in-situ continuous monitoring of spoilage pattern of fish for 40-48 hrs. The feasibility of monitoring pH values in fish meat that could further represent and identify spoilage has been demonstrated.

In the second half of the thesis, application of the flexible IrOx/AgCl pH sensor combined with a Copper (Cu) based impedance sensor is demonstrated for a biomedical application. In this study, the pH sensor and impedance sensors are used to monitor pulmonary aspiration during procedural sedation. Nowadays, for various surgical procedures, Laryngeal mask airways (LMA) are used as an effective method of ventilation. During a surgical procedure, if the patient has not fasted properly then the patient is at a high chance of pulmonary aspiration. In such a situation, gastric fluid will start flushing into the larynx and lower respiratory tract, which could have a detrimental effect on patient health. To demonstrate the use of our sensors to track gastric fluid, in-vitro experiments were conducted by embedding the sensors on LMA. Potassium Chloride (KCl) solution is flushed through the system creating reflux episodes and corresponding readings were recorded.

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PART A: AGRICULTURAL APPLICATION

CHAPTER 1

INTRODUCTION

1.1 Motivation

A definition of pH is the measurement of acidity and basicity in the solution. pH is measure on a scale of 0-14. A lower value of pH considered to be acidic, the higher value of pH is basic, and pH 7 is considered as a neutral.

pH measurement plays a vital role in multiple industrial application since it is a direct measure of [H+] ion concentration. Therefore, pH measurement plays a significant role in Wastewater quality management, oil and gas industries, food/produce and agriculture industries, environmental monitoring, research and development industries like clinics and laboratories. In this part of the thesis for demonstration purpose, the food and agriculture industry are considered. In the food industry, pH monitoring plays a really important and significant role. Due to the logarithmic nature of the measurement, even very minute change in the pH is considered to be very significant. For example, the difference between pH =6 and pH=5, represent 10 times increase in the acidity concentration. Slight variation in pH can also represent a change in flavor, color, texture, shelf life, and quality. Thus, it can further effect on the monitory value of the product.

1.2 Application of pH measurement in Food/ Produce and Agriculture industry

In case of milk and milk product pH value of 6.8 is considered to be safe [1]. Lower pH values level can represent the cattle might be having an infection. Thus, maintaining exact pH plays an important role because of the pathogen's multiplications. In quality monitoring

of spring or natural source of water pH value plays a very significant role. Monitoring the pH of water before adding it to different food processes provides a quick and easy way to check the quality of the end-food-product. In making fruit juices, the pH of sugar extracts as well as those of juices during purification and refining are checked. Also, in the case of fruit production pH of final product need to be monitored because of the water used for cultivation. In the case of liquor industry production, pH value plays a crucial role in quality monitoring. To ensure consistent quality, the pH of brewed liquor should be within the pre-decided limit. However, the taste of liquor like wine, beer also largely depends on the pH value. Value of the pH can be widely used as an indicator of the quality of meat products. It can provide vital information about the freshness of the meat product since the bacterial development inside the meat product have a significant impact on the pH of meat. Thus, by checking the pH of meat at the various level, we can predict the freshness of the meat product. Different parts of the meat have different pH values, and for example, fish head have different pH value and fish stomach will have different pH value.

1.3 Importance of pH monitoring in Seafood industry

In the case of fishing industries, Post-Harvest Fish Losses (PHFL) is a major concern and occur in most fish distribution chains throughout the world [2]. PHFL are often caused by biochemical and microbiological spoilage changes that occur in fish after the death. When the fish is alive, it has its defense mechanism that helps in preventing spoilage by attacking the spoilage bacteria generated inside. However, once a fish dies, its defense mechanisms stop, and enzymatic, oxidative and microbiological spoilage begins that results in the quality deterioration in the fish quality [2].

Based on the average level of seafood consumption in the United States (US) the 2010 Dietary Guidelines Encourages the citizens to double their seafood intake to improve the health of their diets, since the fish meat is a major source of the proteins and omega-3 fatty acids. The growing global population and advancing ecological threats such as climate change are placing increasing demand and constraints on the US and global seafood supplies. For the years 2009-2013, it is estimated that 40-47% of the edible US seafood supply went un-eaten in this period [3]. The greatest portion of this loss occurred at commercial supply chain and distribution levels. Based on the estimates, this loss of seafood represents 208 billion grams of proteins. This loss of seafood could provide the total yearly target protein for 10.1 million men or 12.4 million women [3]. Therefore, there is a need to monitor pH during the supply chain of the fish to reduce the wastage of fish. Since the perishable fish item, it becomes important to monitor the bacterial level of fish by monitoring pH levels.

1.4 Objective:

In this part of the thesis to show the capability of our flexible IrOx/AgCl pH sensor for agriculture application, the fish has been used for demonstration purpose. Among all of the agricultural product fish is a most easily perishable product. Additionally, various environmental factors and handling factors contributed to the quality of the fish. Here the main goal of this part of this thesis is to simulate the entire supply and distribution chain from the ocean to the table of the consumer.

Figure 1 : Image showing wastage of seafood at different stages during supply chain

Since 40-47% of the wastage of quality fish happens during this period it become important to investigate the effect of significant stages of spoilage process on bacterial growth inside the fish meat and in what way it affects the pH of fish meat. Further to demonstrate how the IrOx/AgCl pH sensor can be used for the detection purpose of the spoilage.

CHAPTER 2

FABRICATION AND WORKING OF SENSORS

2.1 Fabrication of Iridium Oxide (IrOx) working electrode

Our iridium oxide working electrode was fabricated in 4 major steps. First fabrication of substrate, a second deposition of thick iridium oxide sensing layer, third thermal treatment of sensor, and final step of sample cutting.

In the first stage of fabrication process, 7 nm thick layer of Cr was deposited on a piece of polyamide substrate, followed by 0.1µm thick layer of Au. In the next step, the thick iridium oxide layer was deposited on a substrate by dip coating of the substrate at 2.0 cm/min withdrawing using Sol-gel solution. A procedure of making Sol-gel solution [4],[5] is explaining in section 2.3. In the third step of fabrication procedure, sample undergoes thermal treatment with heating profile starting at 25ºC for 1 h, 125ºC for 1h, 225ºC for 1h, and 325ºC for 4h. The hot-plate was then cooled down to room temperature. Once the heating treatment is completed, then the sample is cut into small electrode shapes.

2.2 Fabrication of Silver Chloride (AgCl) reference electrode

The silver chloride-based reference electrode was fabricated by applying AgCl paste on top of the Kapton sample. Then in the next step sample was passed through the thermal treatment at for 10 mins. At 120ºC. Once the heating treatment is completed then the sample is cut into small electrode shapes matching to the shape and size of the reference electrode.

The dip coating process and low-temperature treatment not only provide simpler and economical fabrication approach, but also allow the fabrication based on micro-scale, and

the polymer substrate. Both working and reference electrodes were connected with a metal line to probing contact using silver epoxy paste.

2.3 Sol-gel solution making procedure

The Sol-gel solution [4] was prepared by dissolving one gram of anhydrous iridium chloride (IrCl4) in 42 ml of ethanol (C2H5OH) and 10 ml of acetic acid (CH3COOH). Addition of acetic acid avoids the unnecessary chemical reactions. The coating solution was the stirred with a magnetic rod for 1 h.

2.4 pH sensing mechanism of Iridium Oxide Electrode

Between two oxidation stages of iridium oxide there are three possible mechanisms were responsible for redox intercalation equilibrium.

$Ir_2O_3 + 6H^+ + 6e^- \leftrightarrow 2Ir + 3H_2O$ (1)

$IrO_2 + 4H^+ + 4e^- \leftrightarrow Ir + 2H_2O$ (2)

$2IrO_2 + 2H^+ + 2e^- \leftrightarrow Ir_2O_3 + H_2O$ (3)

governing equation for calculating redox potential

$$E = E^0 - 2.303 \frac{RT}{F} pH \quad (4)$$

$$E = E^0 - 0.05916\, pH \quad (5)$$

where, E = redox potential

E^0 = standard electrode potential

F= Faraday's constant = 96487 coul/equiv at 25^0C

T= Temperature

R = Gas constant = 8.314 J/^{0}

In equation (4), E^{o} is the standard electrode potential, for standard hydrogen electrode the value of E^{o} is 926 mV and 577 mv for standard Ag/AgCl reference electrode.

For standard values of gas constant and Faraday's constant at 25ºC, the value of RT/F will be 25.688. Therefore, according to Nernstian Response, the pH potential sensitivity will be -59 mV/pH if assuming all changes were formed on the electrode [6], [7].

Since the Nernstian response of the IrOx/AgCl sensing film electrodes depends on the surface quality of the electrode, surface microstructure, and the oxidation states of the films. Thus, due to the above-mentioned reasons, we should consider that the sensitivity of the pH sensor is expected to vary between Nernstian and near- Nernstian response depending upon the accuracy of the fabrication procedure.

2.5 Calibration

In the stage of calibration fabricated were tested on with standard pH buffer solution. After dipping the sensor into the standard buffer solution, the electrochemical potential corresponding to the pH level will get generated. The IrOx electrodes generate an electrochemical potential about the reference electrodes potential that varies according to the pH solution as discussed above.

Here each IrOx and AgCl electrodes were connected to the NI DAQ USB-6211 card and synchronized with the LabView based program for potential recording. The

electrochemical potential was displayed, and data was recorded in a computer simultaneously.

CHAPTER 3

FISH BIOLOGY

3.1 Post Harvest Fish Loss:

Post-Harvest Fish Loss (PHFL) is the major concern and occurrence in most of the fishing industries supply and distribution chains throughout the world. Generally, PHFL often caused by the biochemical and microbiological spoilage changes that occur in the fish after death. Spoilage is caused by the action of enzymes, bacteria, and chemicals present in the fish. When the fish is alive fish has its defense mechanism that counteracts with the internal bacterial spoilage activity. But, immediately after fish is dead the oxygen supply to the muscle and tissues of the fish stops. This increase in the biochemical and microbiological activity which leads to the spoilage in the fish. The nature and type of bacteria present in a fish depend upon the water from where it was caught, and methods used for handling of the fish after its catch.

The softness of muscles changes in fish color, odor, texture, a color of gills, a color of the eyes of the fish are few of the characteristics [8] that can be monitored and observed in spoiled fish. Following are the some of the factors contribute to spoilage of fish:

- High moisture content
- High fat content
- High protein content
- Weak muscle tissue
- Ambient temperature

- Unhygienic handling

3.2 Major steps involved in the Fish Spoilage Process [9]:

pH of freshly killed fish is in the range of 6.5-6.8 [10]

3.2.1 Rigor Mortis:

Rigor Mortis means stiffening of the muscles of the fish after death. Immediately after the fish is dead the muscles of the fish are soft and limp, this time the flesh of fish is said to be in pre-rigor condition. Eventually, the muscles of the fish begin to stiffen and harden, at this stage animal is said to be in rigor mortis period. During this step, the, pH value of the fish decreases. This happens due to the lack of oxygen inside the dead muscles and tissue of the fish, and glycogen presents in the muscles and tissues of the fish starts to decompose into lactic acid [9]. Also, the Adenosine Tri-Phosphate (ATP) is also hydrolyzed to be phosphoric acid. Therefore, the pH level of the muscles/tissues will decrease. Generally, speaking the rigor mortis period will last for 10-22 Hrs. if the fish is not kept in ice. There are several other factors which have impact on the rigor mortis period of the fish for example, type of fish, physical condition of the fish at the time of death, temperature at which it is kept after the death, handling of the fish after death, and quality of the water the where fish was harvested.

3.2.2 Autolysis

After completion of rigor mortis period, stiffens of the muscles starts to decrease gradually. During this period the pH of the fish starts to increase, ending up in softening of the muscles. Autolysis is defined as the breakdown of the muscles and tissues into smaller molecules using the enzymes. Thus, autolysis can be described as an internal breakdown

of the structure of the proteins and fats by the complex series of the reactions by enzymes results in the formation of amines.

Autolysis steps start after the rigor mortis (post-rigor) and create favorable conditions for the growth of the bacteria. Enzymatic action in fish also leads to belly bursting. Fish have a large content of digestive enzymes in digestive tracts. This will lead to very quick degrade and spoilage in the fish.

3.3.3 Spoilage

The third step is the final spoilage of the fish. During this step the pH value of starts to increase rapidly. The free amino acid pool in the muscle of fish is readily utilized by spoilage organism by the process of deamination resulting in the formation of ammonia which is primary chemical formed during decomposition of the fish. Fish contains the small percentage of the odorless Trimethylamine N-oxide (TMAO) which is reduced to an offensive smelling Trimethylamine (TMA) by bacterial action [8]. Also, in some fish the high concentration the urea in the flesh of the fish starts to degrade into ammonia by microorganisms. The formation of ammonia is accompanied by an offensive odor.

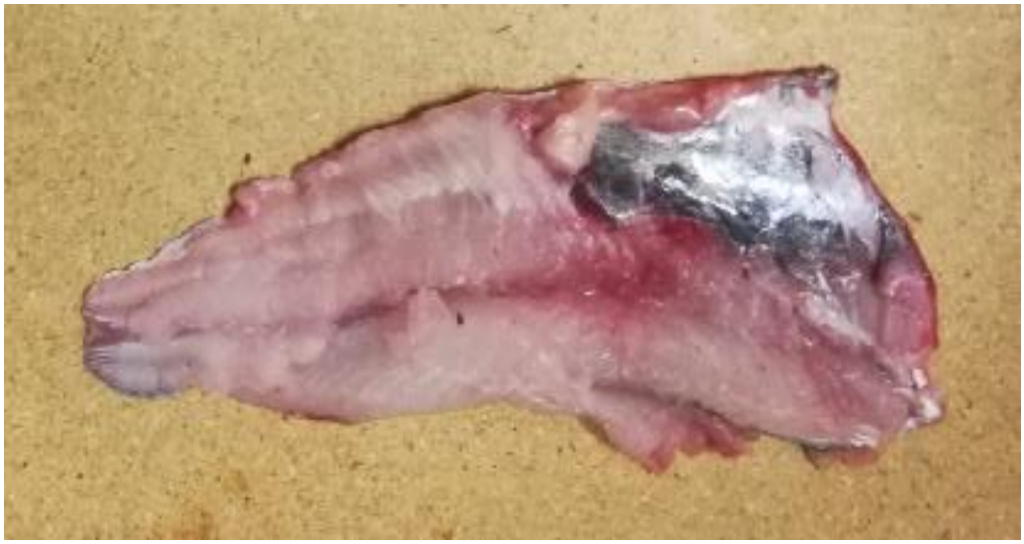

Figure 2: Image showing freshly killed fish after cutting

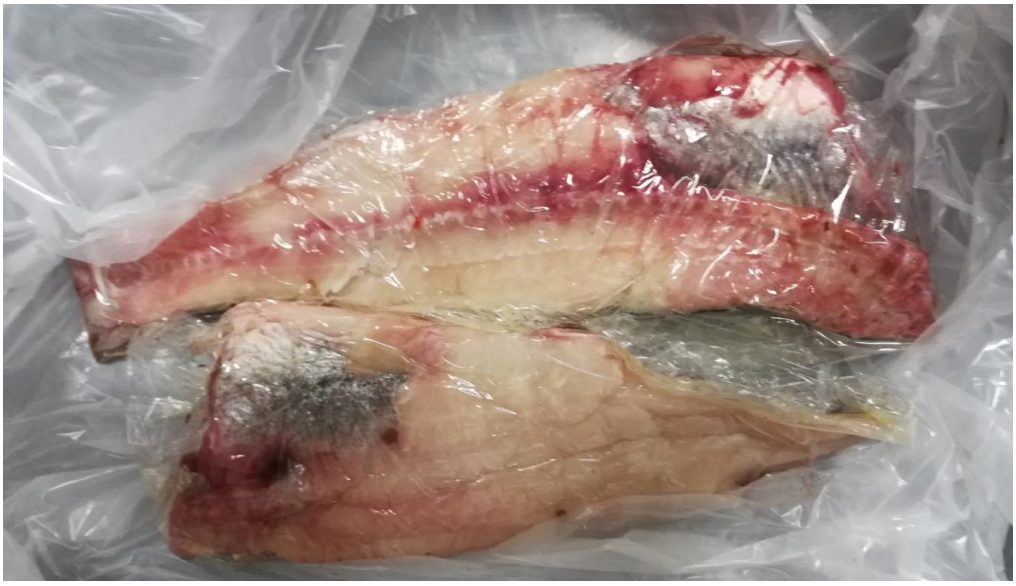

Figure 3: Image showing fish after the span of experimentation (41 h)

CHAPTER 4

EXPERIMENTS

4.1 Experimental Set-up and data acquisition procedure

In this part of the thesis for the experimentation purpose, Tilapia and Catfish were used. The time between the time of the death h of the fish and start of the experiment was maintained at half an hour for all of the experiments. Each fish was cut into symmetrical section of the head, stomach, fillet, etc., depending upon the requirement of the experiment.

Each of this fish sections were placed on the flexible IrOx/ AgCl sensor for the monitoring. Then each section of the fish was neatly wrapped using thin plastic film to contain moisture inside the fish. Doing so will also reduce the spreading of the foul odor. Based on the requirement of the experiment, the Styrofoam boxes were used to keep the fish inside to maintain the temperature at a certain level using the blue ice packs. In this part of the thesis, the experimentation time for all of the experiments was maintained at 40-48 hrs. Depending upon situations.

Once the wrapping of fish and placement of the sensors was done, then the connecting end of each sensor was connected to the NI-DAQ card using connecting wires for the measurement of the voltage at a specific interval of time. LabView based programs were used to monitor and record the values of the voltage at an interval of 10 seconds each.

Upon completion of the experiments, the voltage values were converted into the pH using the calibration curve.

4.2 Experiment 1

In this study, Tilapia fish is used for experimentation. Here the fish is divided into four sections. Each head and stomach of the fish was divided into individual symmetrical sections (as shown if figure 5). In this experiment, the guts of the fish were kept inside the fish to demonstrate the effect of the guts on the spoilage process.

One pair of head and stomach was individually kept inside the Styrofoam box with blue ice packs. The temperature inside the ice box (figure 6) was maintained at 0ºC-4ºC and constantly monitored using a thermometer. Total testing time for the experiment was 41 Hrs.

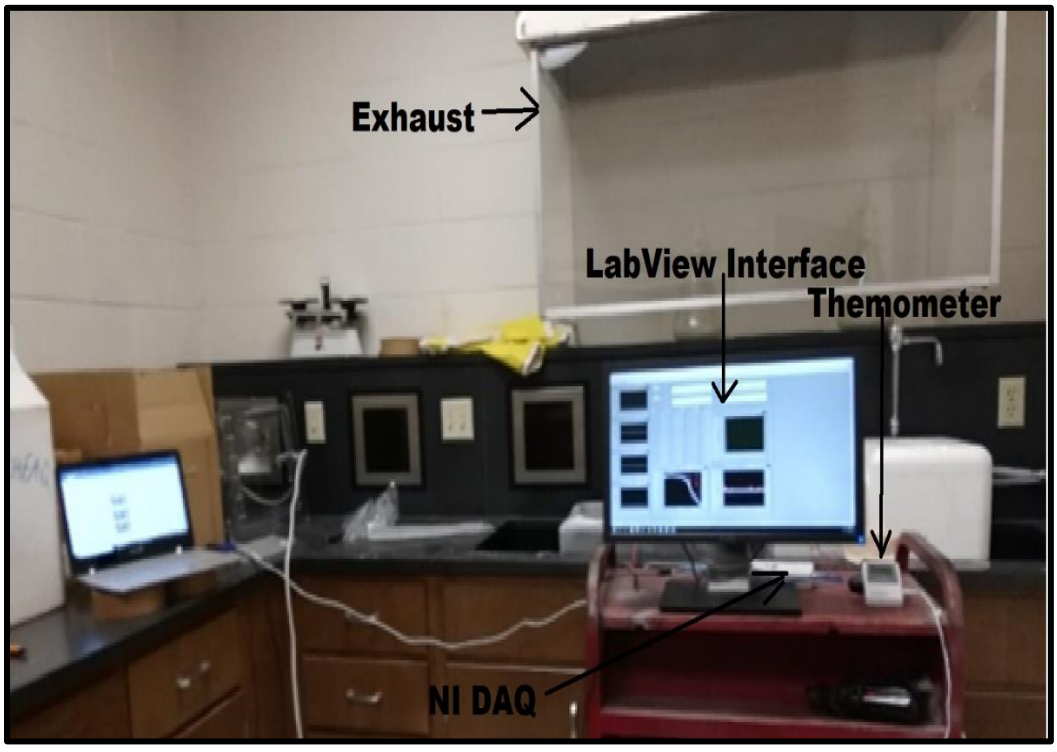

Figure 4: Image showing the experimentation set-up

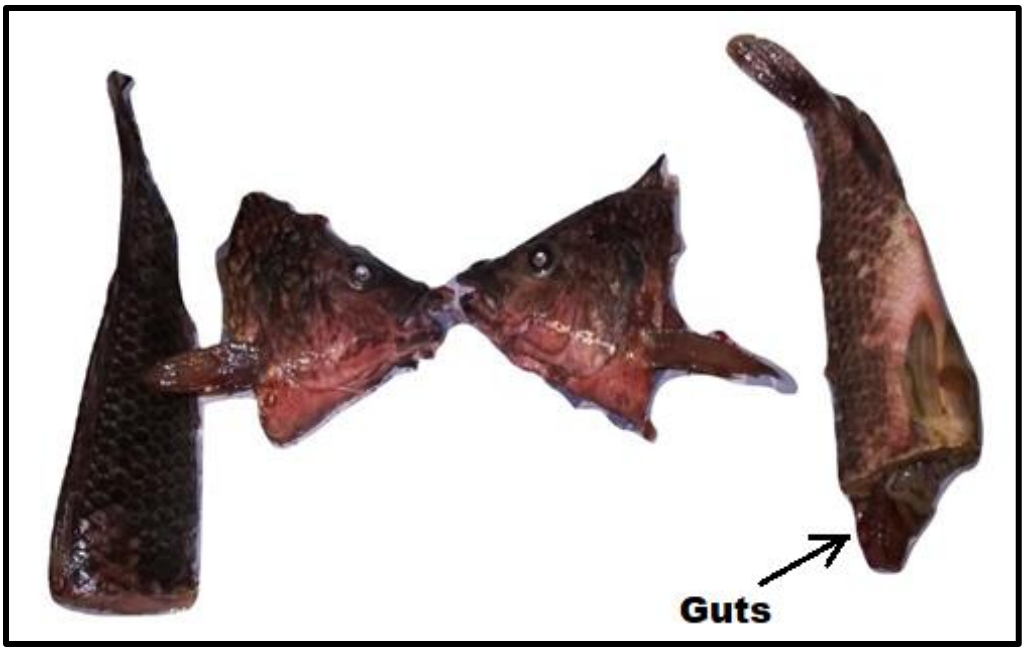

Figure 5: Image Showing head and stomach sections of the fish

Figure 6: Image showing arrangement of fish sections for experimentation

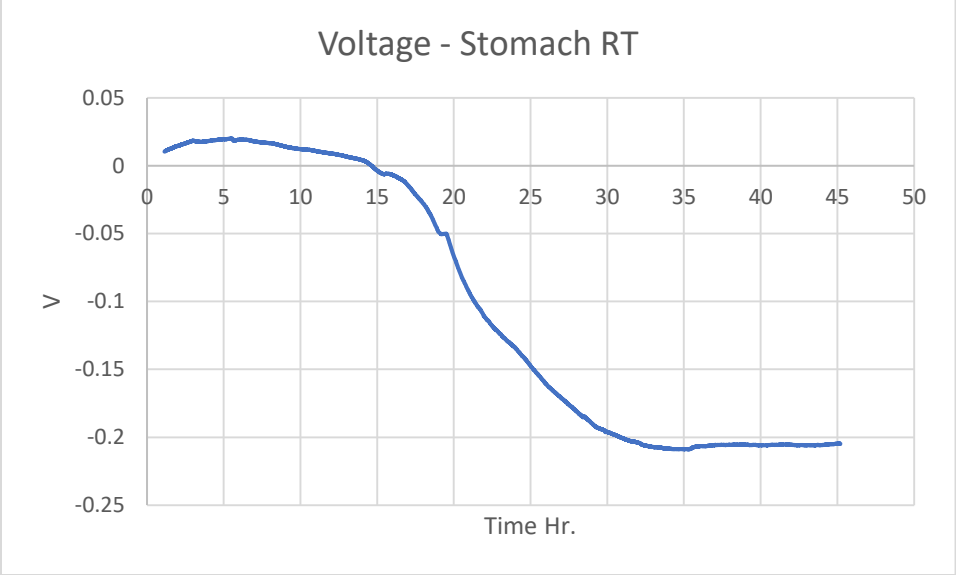

Figure 7: Graph indicating voltage recorded by sensor from Stomach kept at room temperature

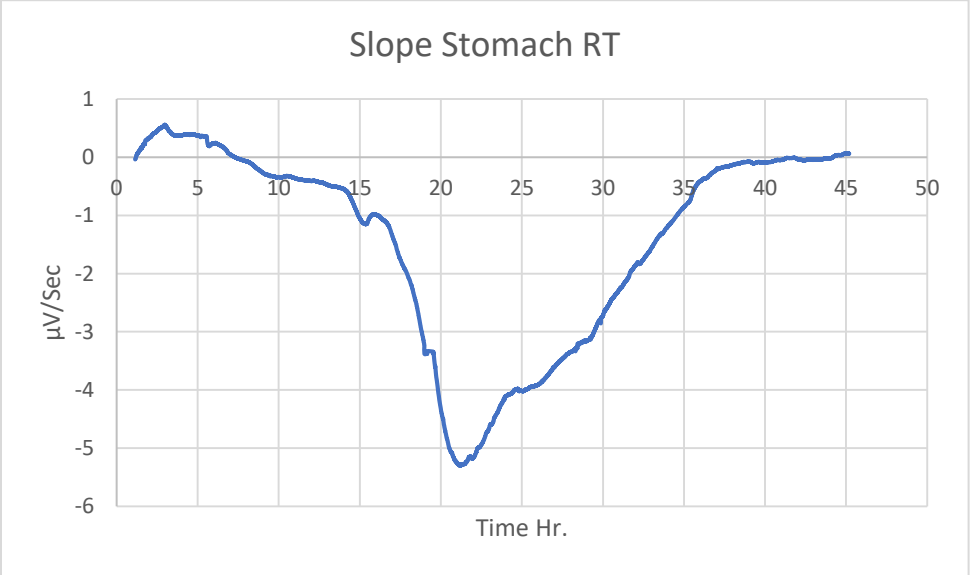

Figure 8: Graph showing slope of Voltage-Stomach RT Curve

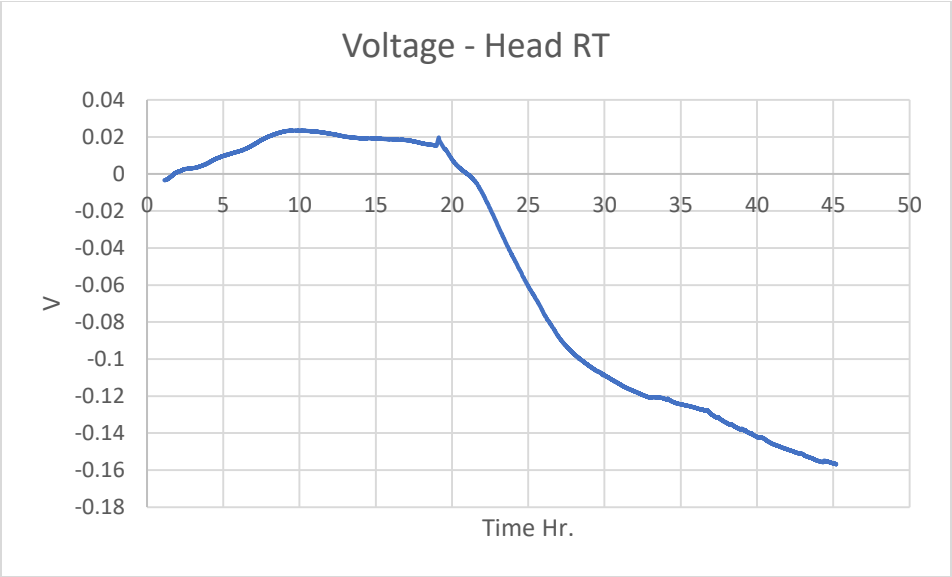

Figure 9: Graph indicating voltage recorded by sensor from Head kept at room temperature

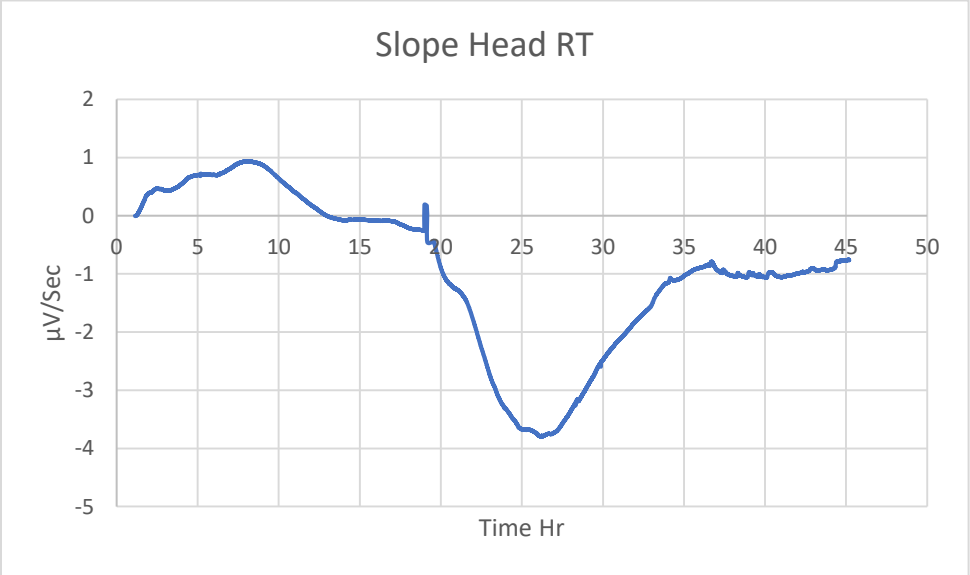

Figure 10: Graph showing slope of Voltage-Head RT Curve

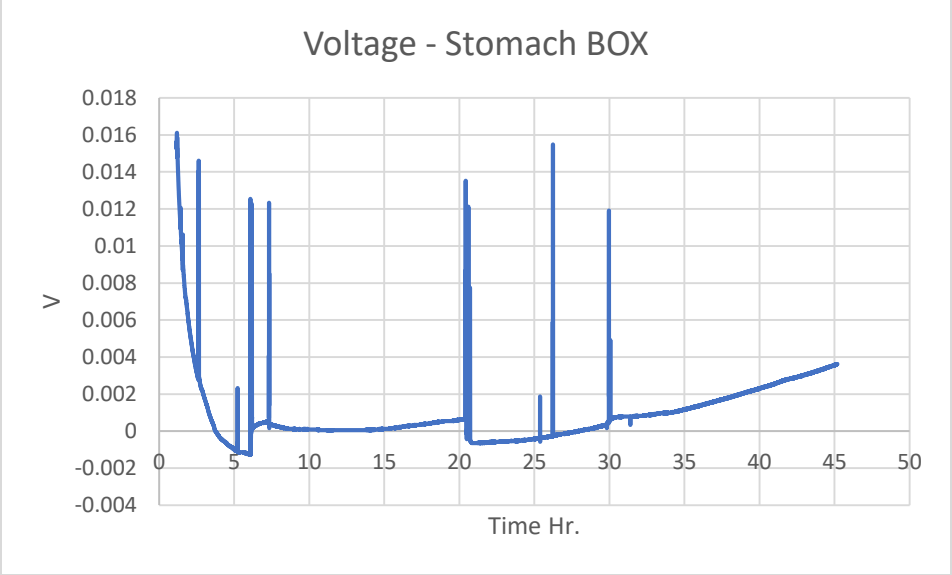

Figure 11: Graph indicating voltage recorded by sensor from Stomach kept at Refrigeration Temperature (0-4ºC)

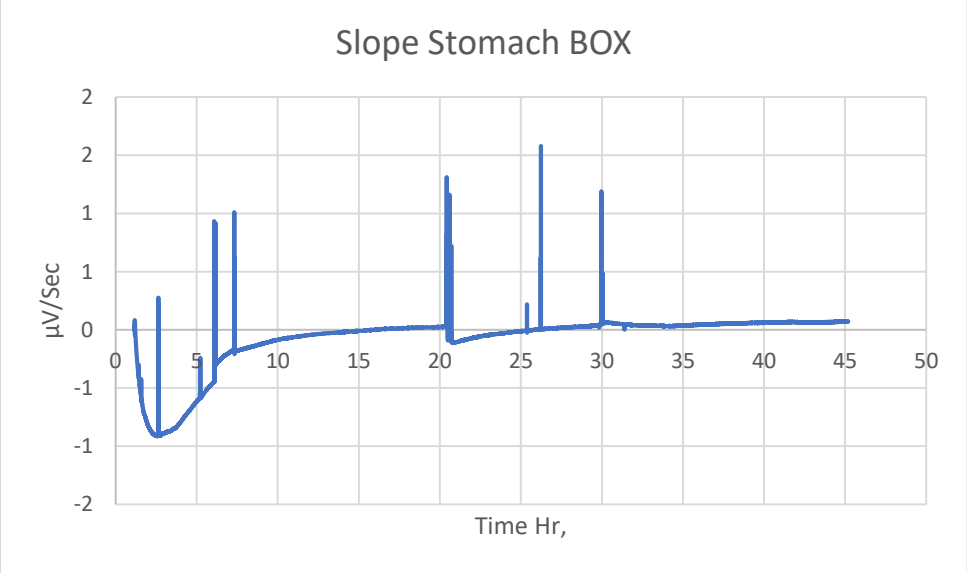

Figure 12: Graph showing slope of Voltage-Stomach BOX Curve

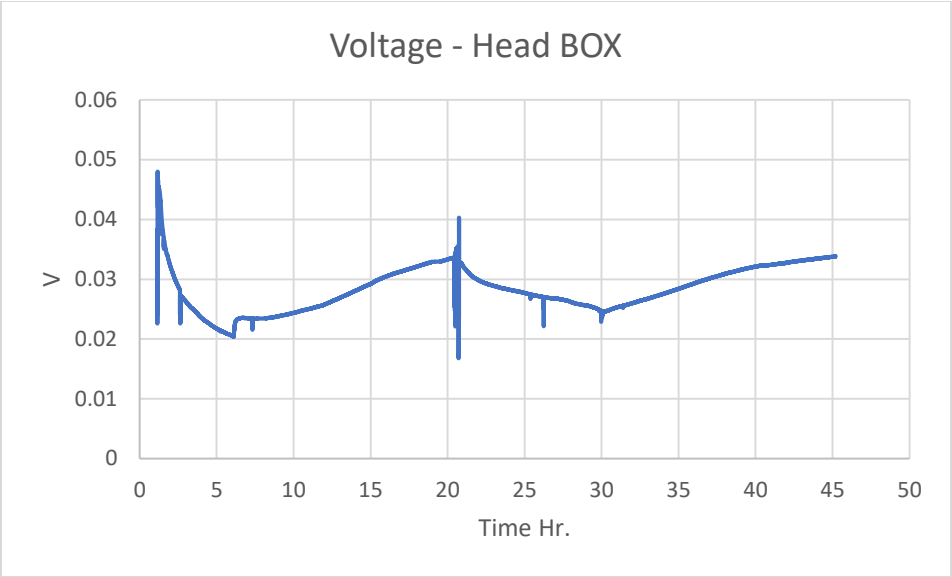

Figure 13: Graph indicating voltage recorded by sensor from Head kept at Room Temperature

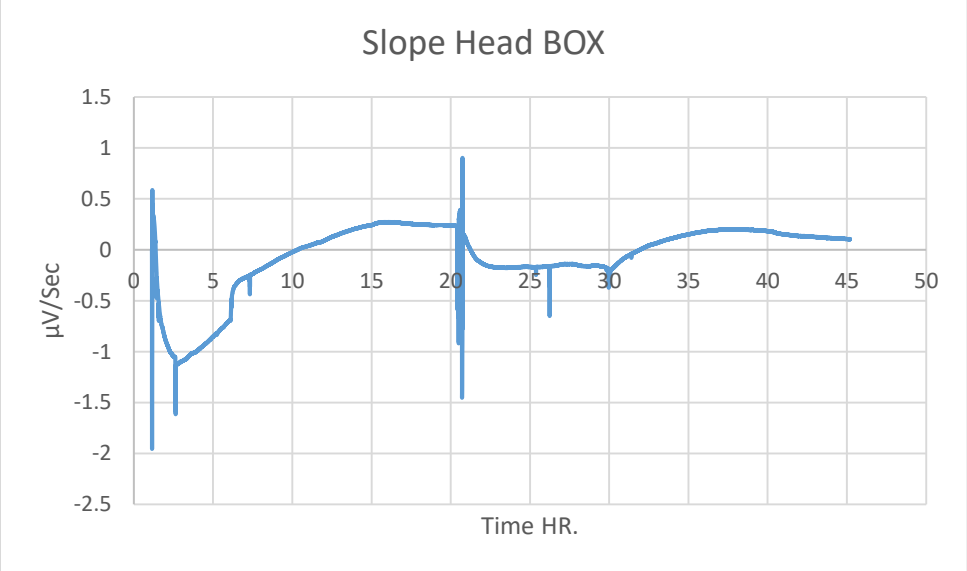

Figure 14: Graph showing slope of Voltage-Head BOX Curve

4.3 Experiment 2

In this study, two Tilapia fishes are used for the experimentation. Here one fish is cut into two symmetrical sections of the stomach, and another fish is cut into two symmetrical fillet sections (Figure 15). All of the fish sections used in this study were wrapped using plastic wrap in a similar manner as compared to the previous experiment (Figure 16) and maintained at room temperature. Here all fishes are kept at room temperature (18ºC - 22ºC) to study the effect of the bacterial activity on the spoilage of the fish. Since bacterial activity happens at a higher rate when temperature close to room temperature.

In this part of the experiment, the guts of the fish are cleaned. If the guts of the fish were kept inside, then that will increase the core body temperature of the fish. The increase in body temperature of the fish will indirectly promote the bacterial activity inside the fish. And the increase in bacterial activity will spoil the fish more rapidly.

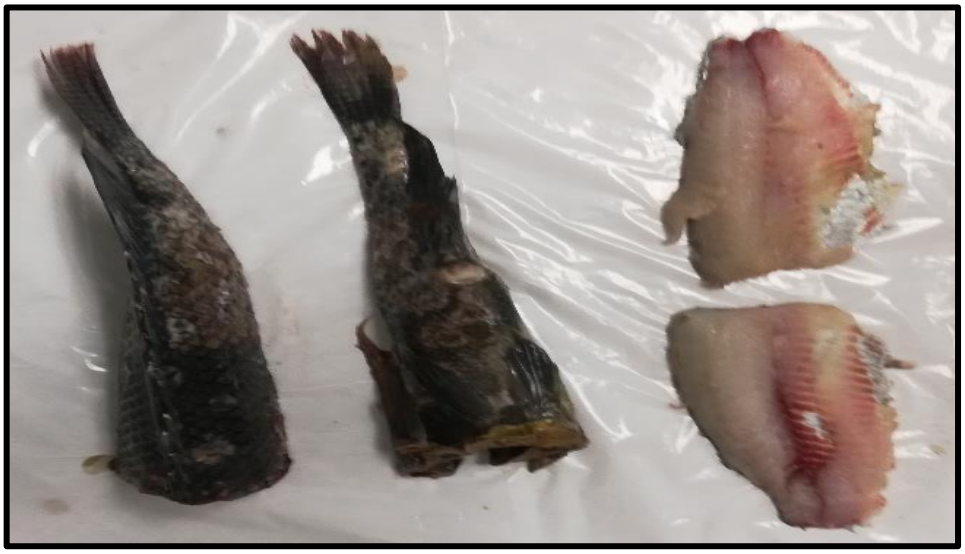

Figure 15: Image showing stomach and fillets sections of the tilapia fish

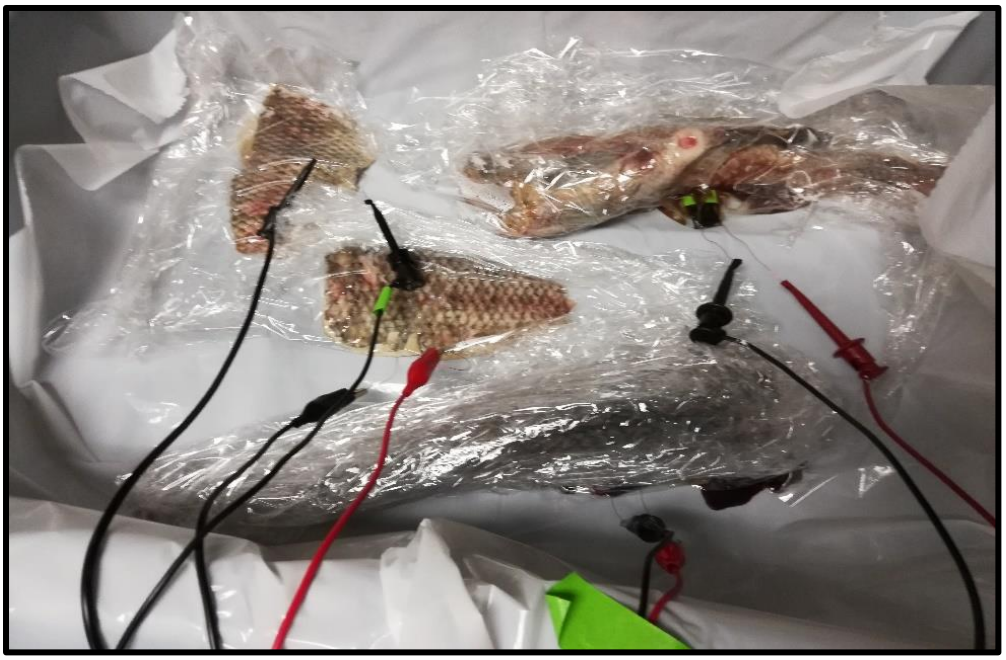

Figure 16: Showing wire connection to each section of the fish

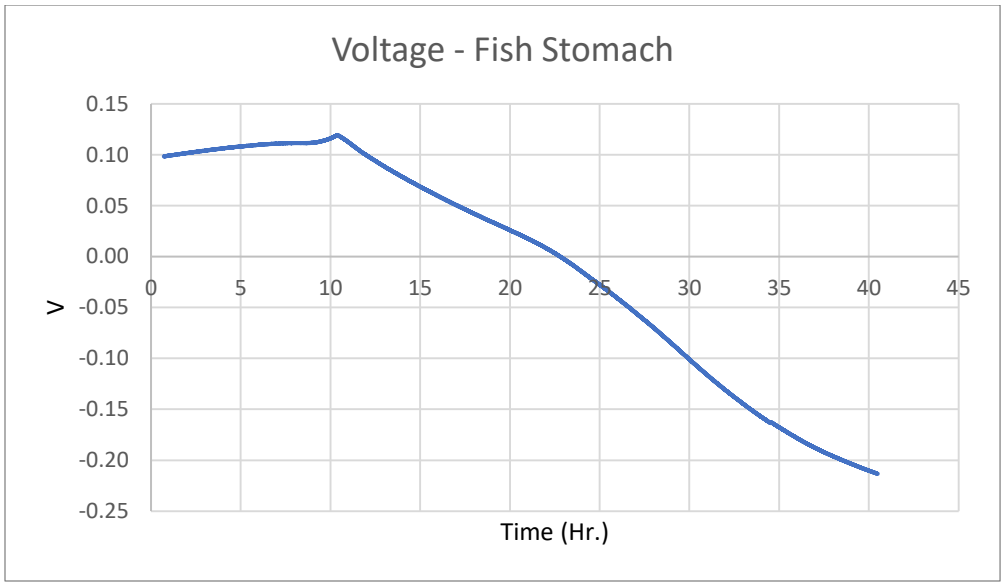

Figure 17: Graph indicating voltage recorded by sensor from Stomach non-gut side

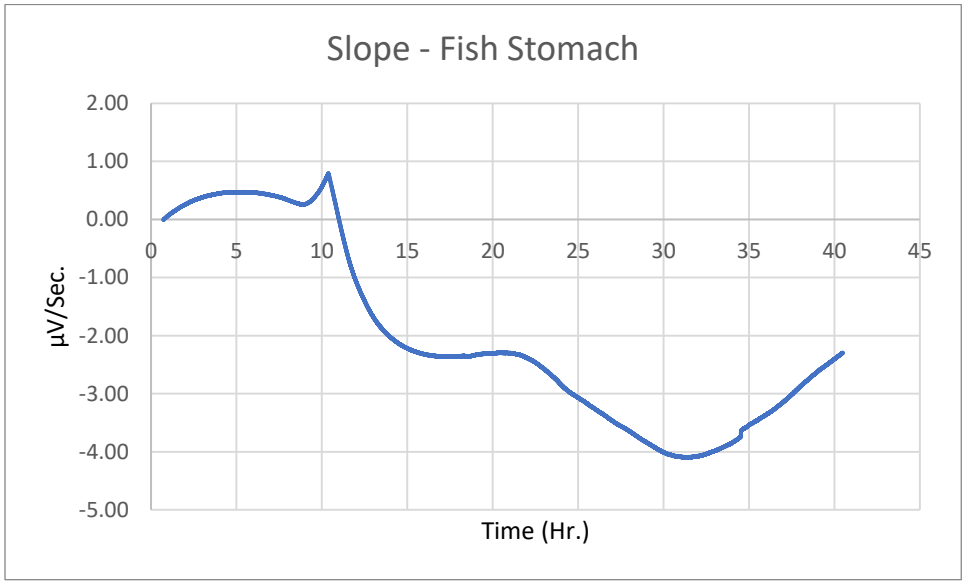

Figure 18: Graph showing slope of Voltage-Fish Stomach Curve

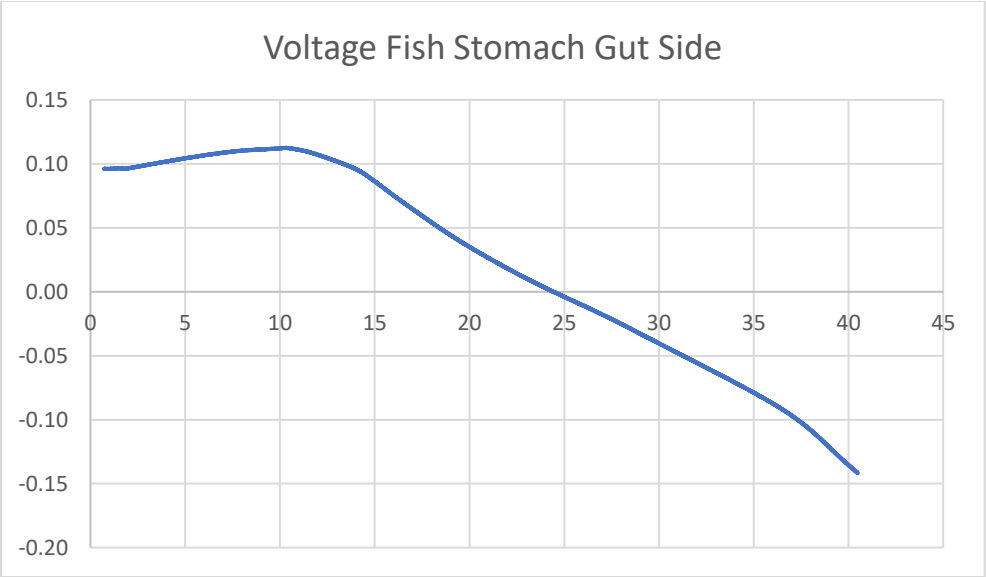

Figure 19: Graph indicating voltage recorded by sensor from Stomach on gut side

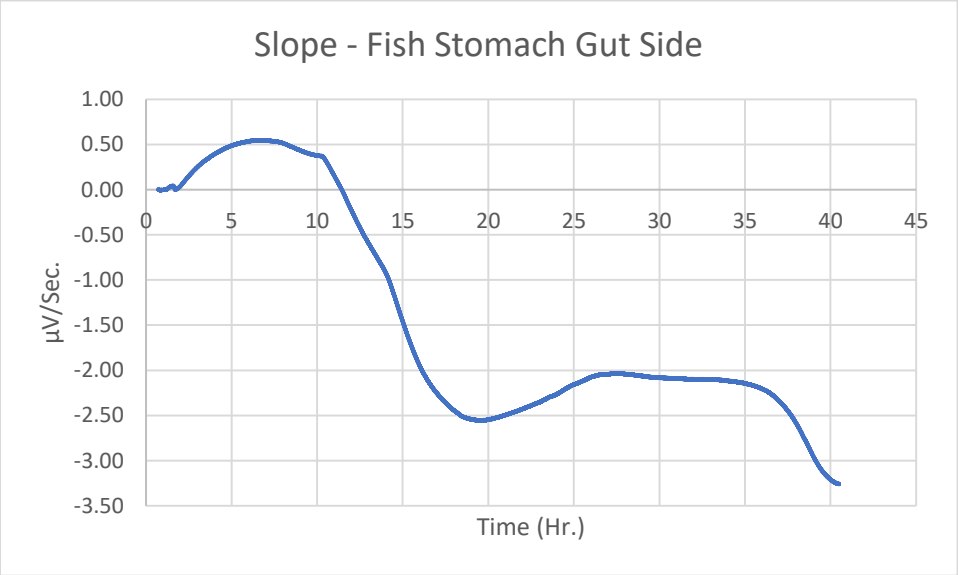

Figure 20: Graph showing slope of Voltage-Fish Stomach Gut Side Curve

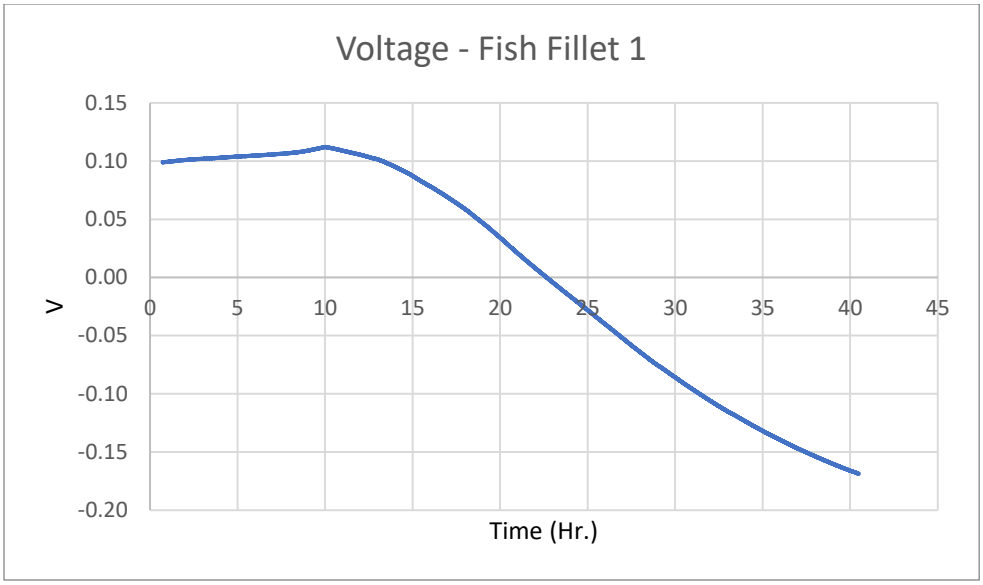

Figure 21: Graph indicating voltage recorded by sensor from Fish Fillet-1

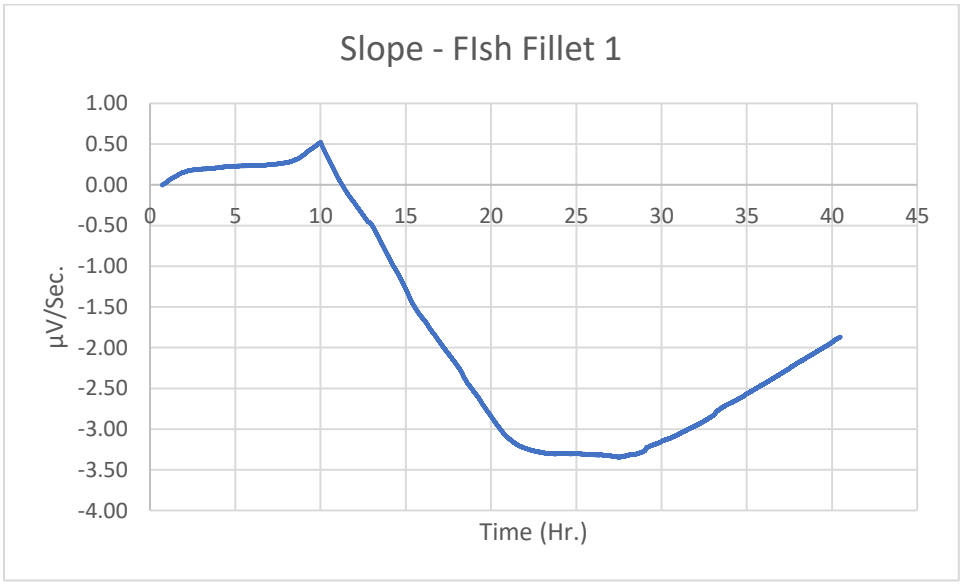

Figure 22: Graph showing slope of Voltage-Fish Fillet-1 Curve

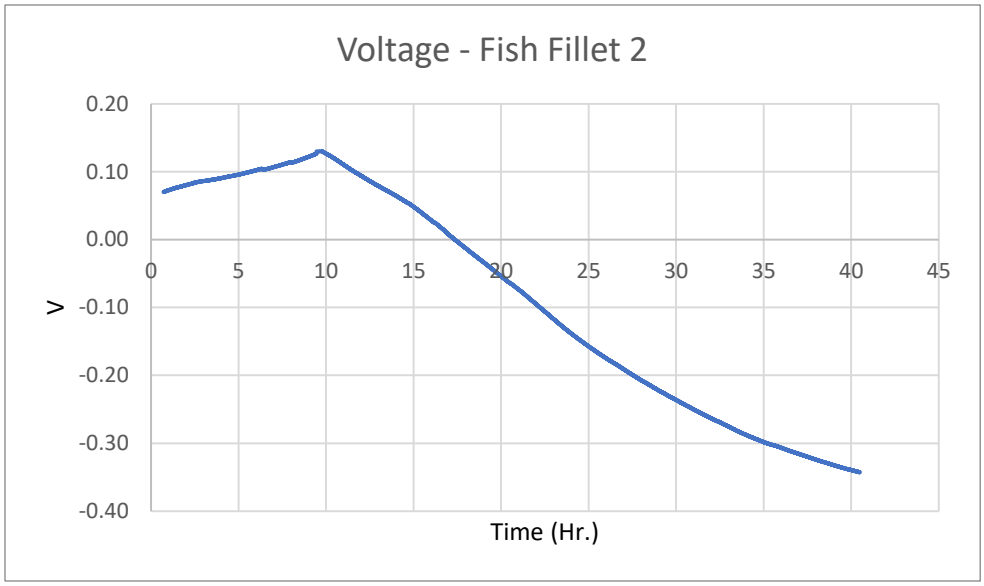

Figure 23: Graph indicating voltage recorded by sensor from Fish Fillet-2

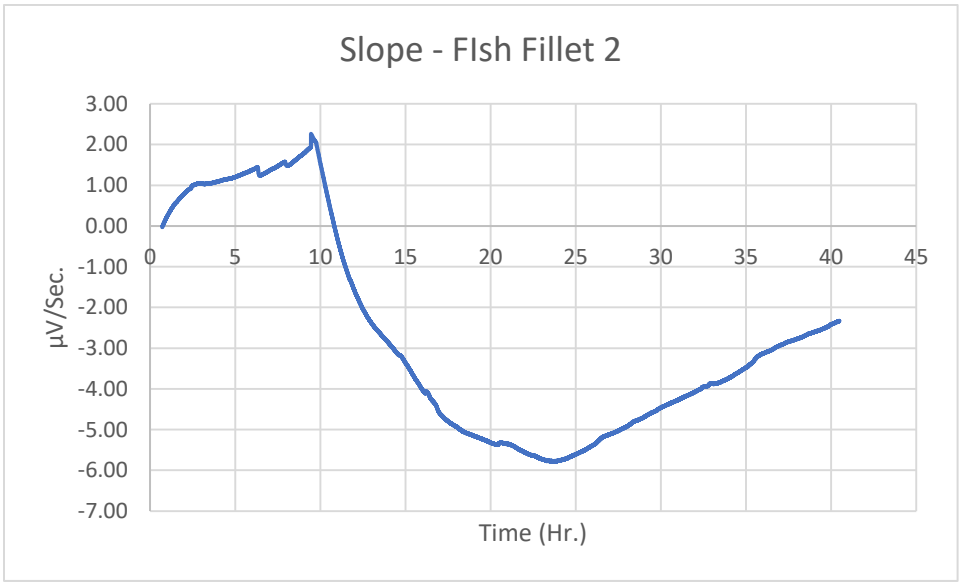

Figure 24: Graph showing slope of Voltage-Fish Fillet-2 Curve

4.4 Experiment 3

In this study, Catfish is used for experimentation purpose. Here fish is divided into 2 symmetrical sections of stomach and guts of fish were removed. Both of section was wrapped in a plastic film (Figure 26) and placed on top of the sensors for testing purpose. Head of fish is not used for the experimentation because the head of Catfish has a very strong bone which makes it difficult to cut it into the section. Experimentation time here was 41 Hrs.

Figure 25: Image of Catfish used for experimentation

Figure 26: Image showing catfish cut into stomach sections and wrapped using plastic film

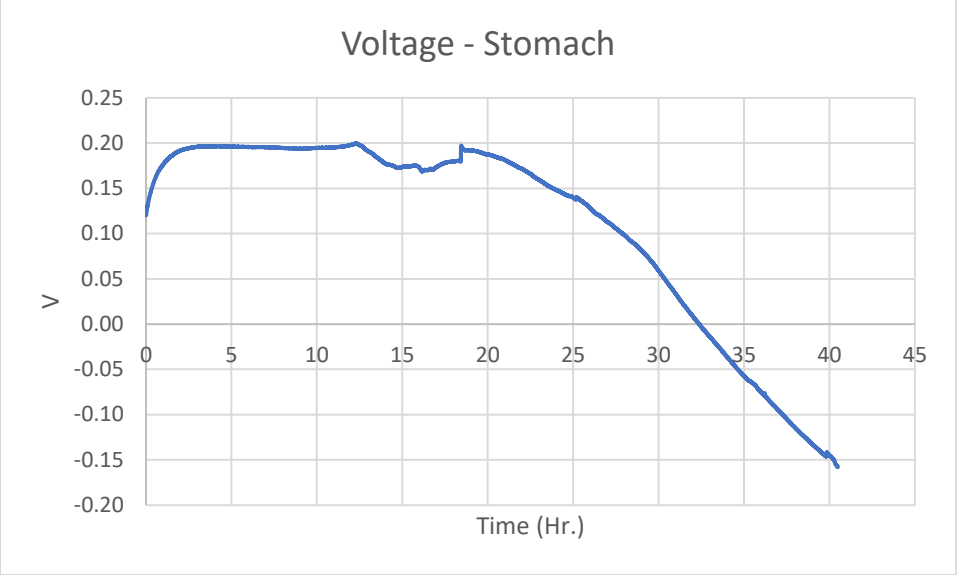

Figure 27: Graph indicating voltage recorded by sensor from Stomach

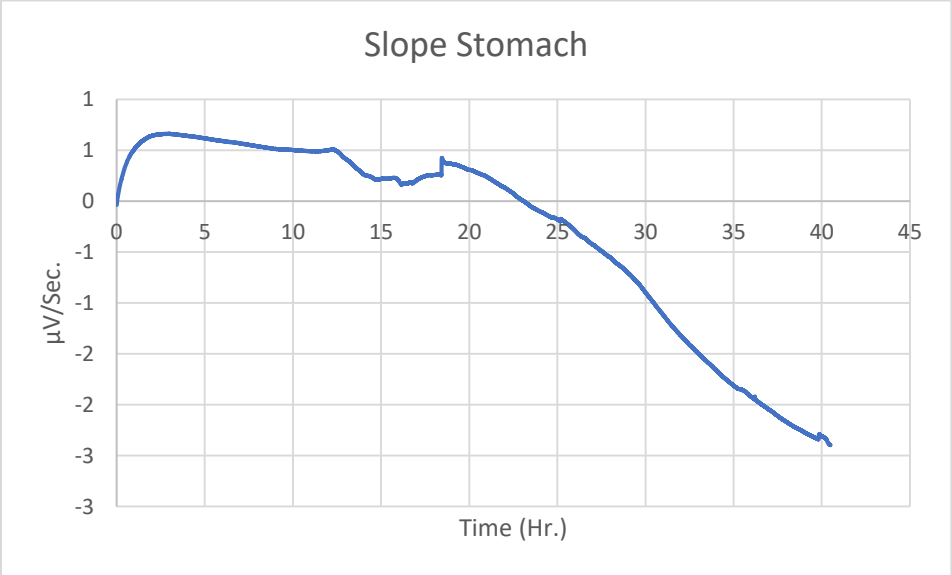

Figure 28: Graph showing slope of Voltage-Stomach Curve

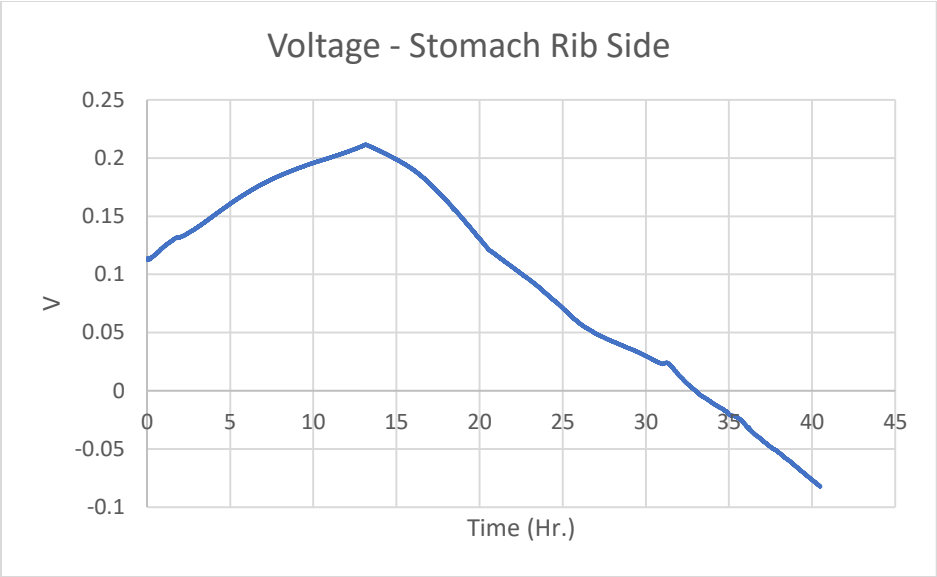

Figure 29: Graph indicating voltage recorded by sensor from Stomach on Rib Side

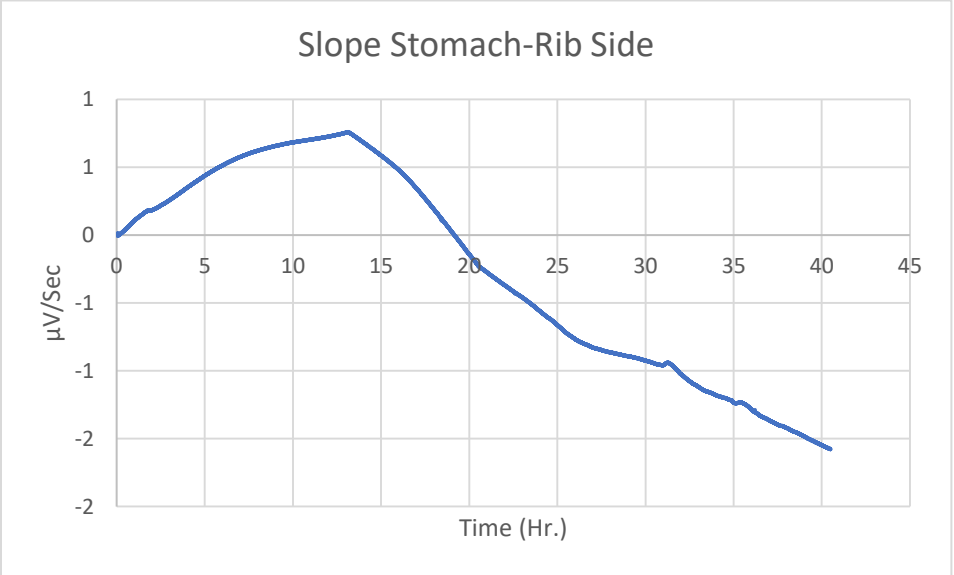

Figure 30: Graph showing slope of Voltage-Stomach Rib Side Curve

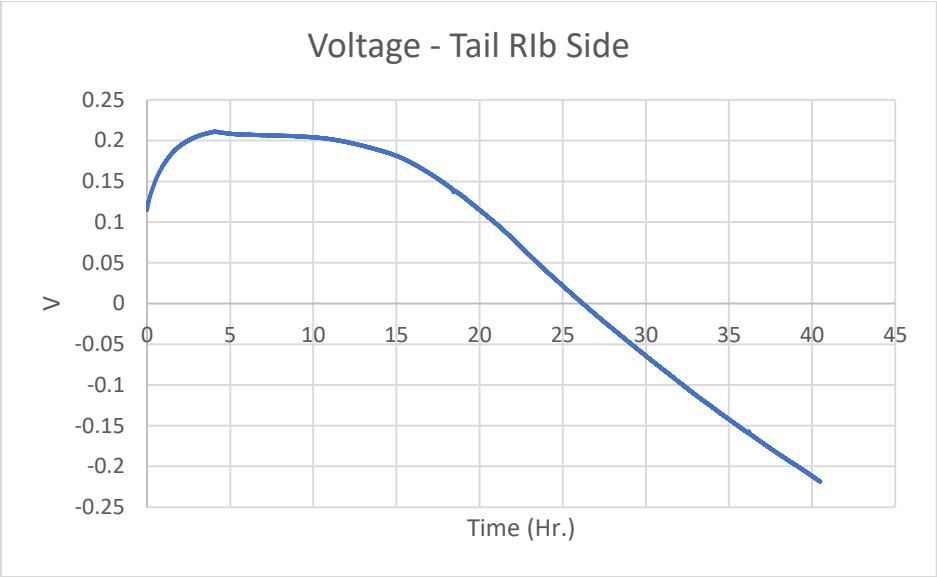

Figure 31: Graph indicating voltage recorded by sensor from Tail on Rib Side

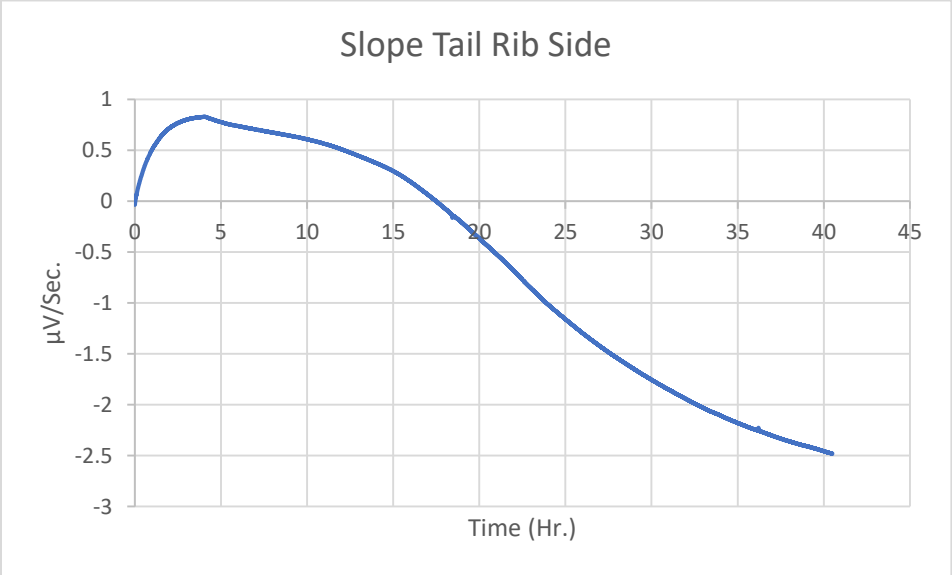

Figure 32: Graph showing slope of Voltage-Tail Rib Side Curve

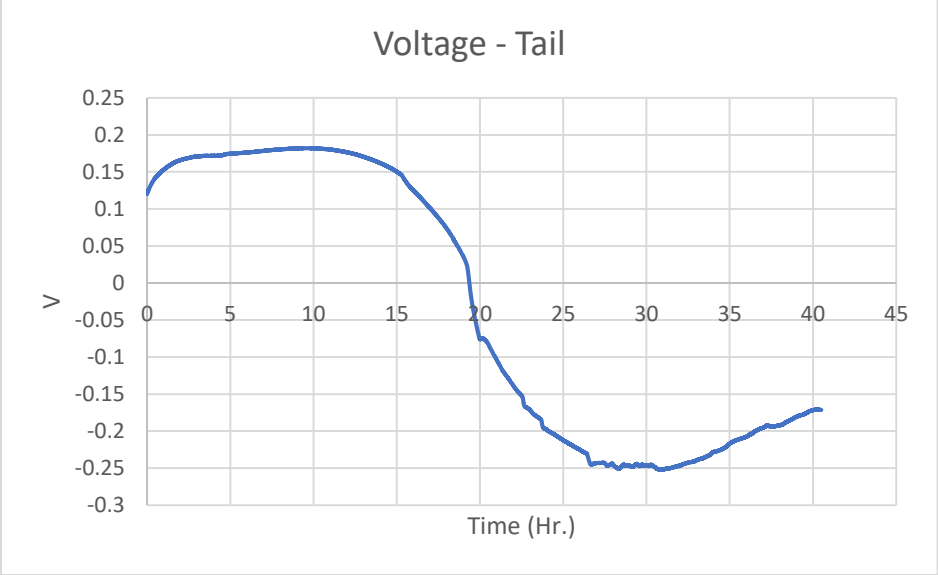

Figure 33: Graph indicating voltage recorded by sensor from Tail non-rib side

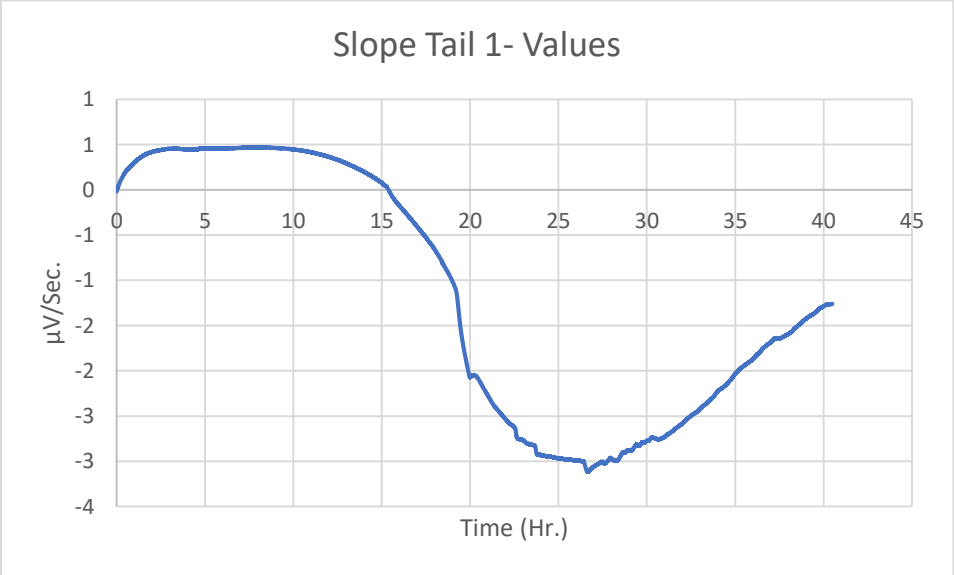

Figure 34: Graph showing slope of Voltage-Tail Curve

4.5 Experiment 4

Similar to the previous experiment, in this study also Catfish was used for experimentation purpose. Also, Fish was cut into similar sections like in case of the previous experiment. The only difference in this experiment is one of the sensors was inserted into the Rib side of the stomach. Other sensors were kept below the fish like previous experiments. All rest parameters were kept constant. Experimentation time for this experiment was 44 Hrs.

Figure 35: Image showing sensor inserted inside the meat of fish (Rib-Side)

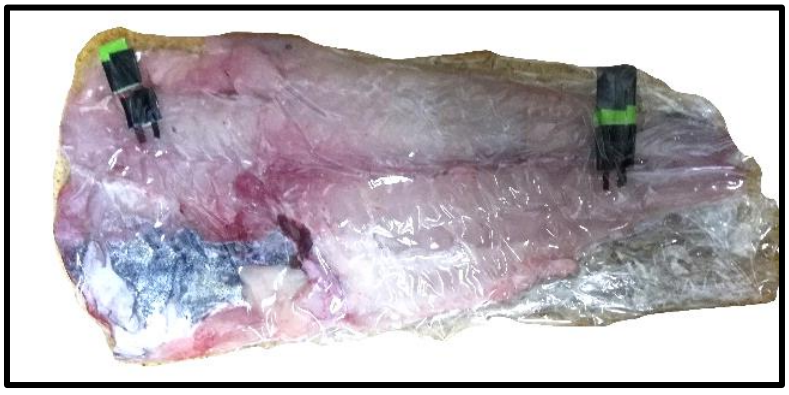

Figure 36: Image showing placement of sensors below the fish Stomach and Tail

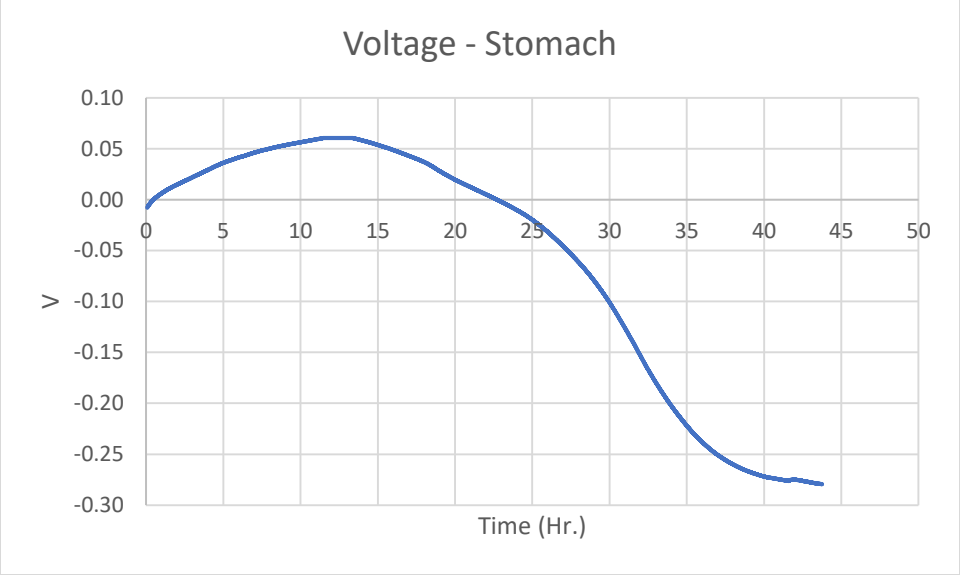

Figure 37: Graph indicating voltage recorded by sensor kept below Stomach

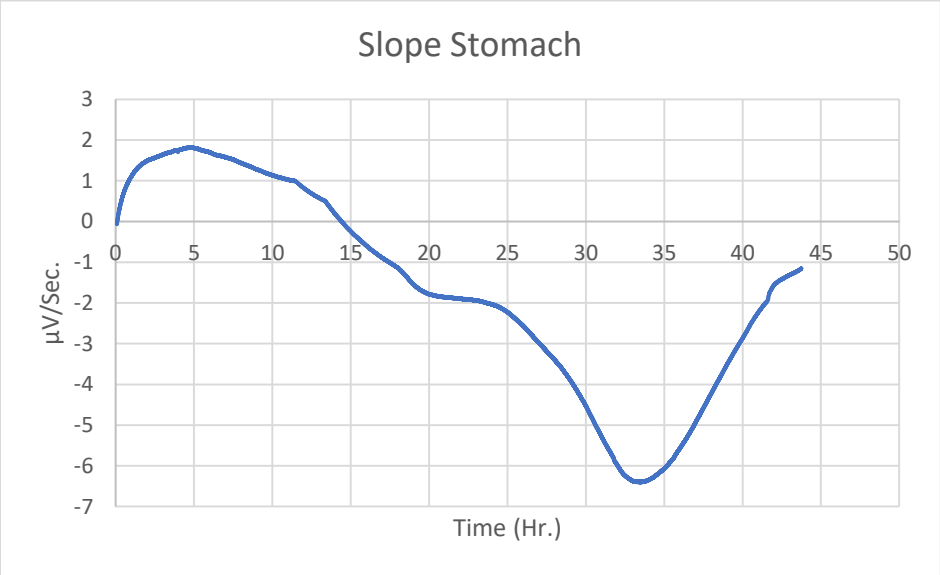

Figure 38: Graph showing slope of Voltage-Stomach Curve

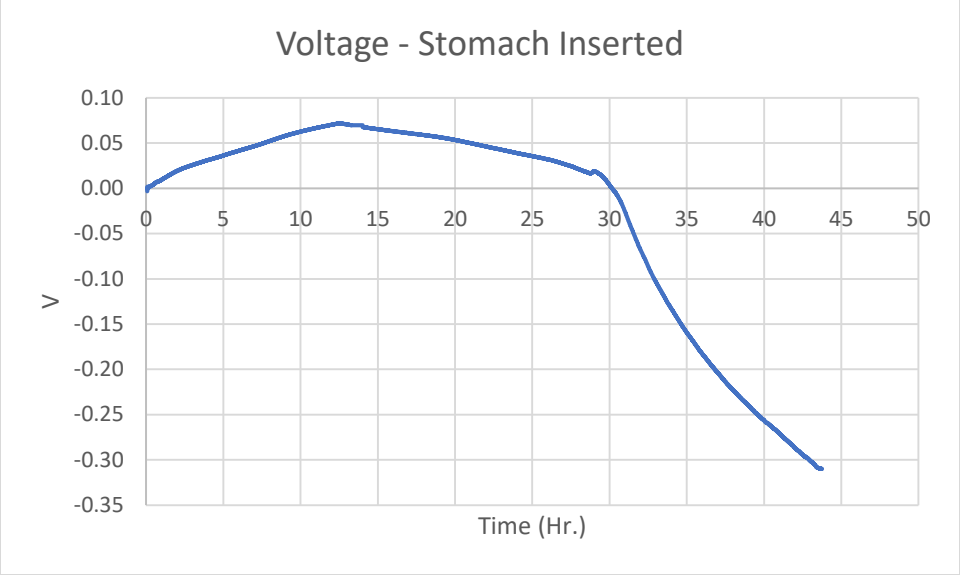

Figure 39: Graph indicating voltage recorded by sensor inserted into Stomach

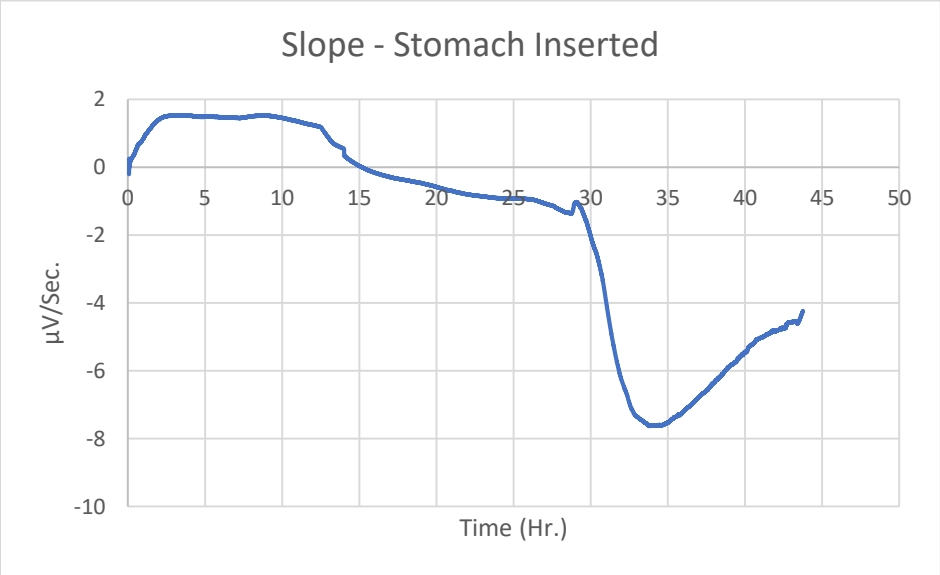

Figure 40: Graph showing slope of Voltage-Stomach Inserted Curve

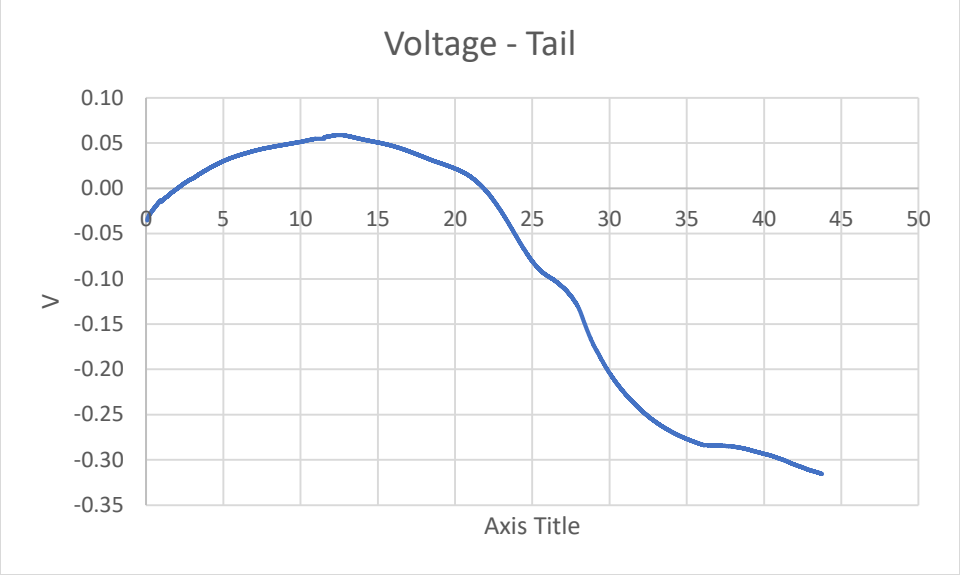

Figure 41: Graph indicating voltage recorded by sensor kept below Tail

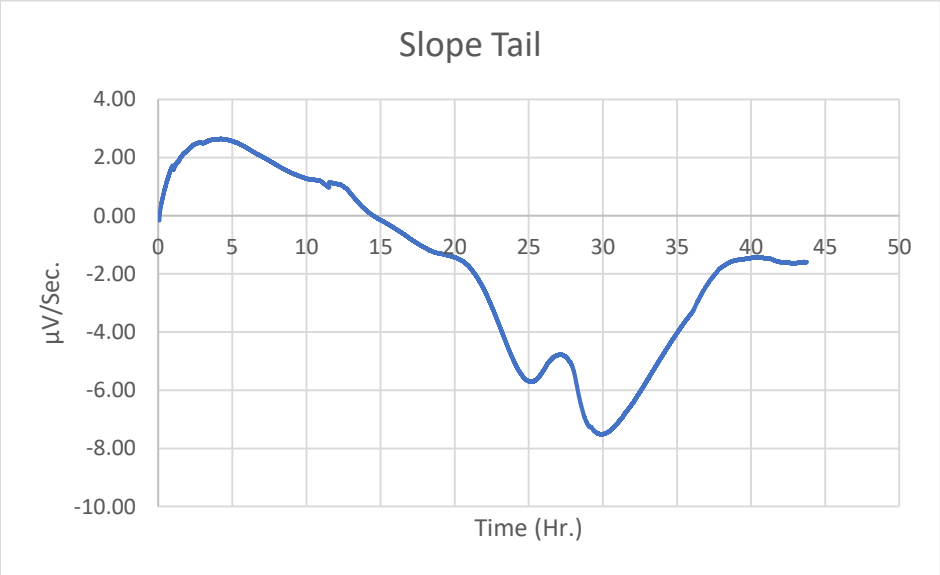

Figure 42: Graph showing slope of Voltage-Tail Curve

4.6 Experiment 5

The aim of this study is to measure the pH of fish meat using a commercial pH sensor for reference purpose. Here Catfish is used for demonstration. Total experimentation time is 45 Hrs. All experimental conditions were kept similar to the previous experiments as a commercial sensor APERA's pH60F flat pH sensor is used. Since the sensor is big in size due to which sensor was difficult to hold steady at one position to measure reading. Also, the fish flesh was getting stuck in between the small protrusion at the bottom of the sensor, thus cleaning was required.

Figure 43: Sensor with electrode tip

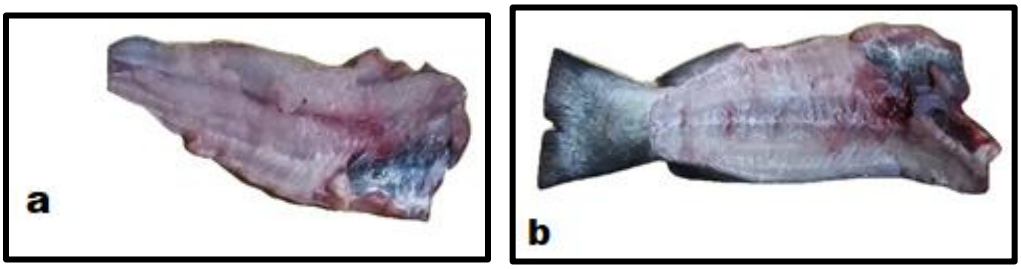

Figure 44: (a) Stomach section without rib, (b)Stomach section with rib

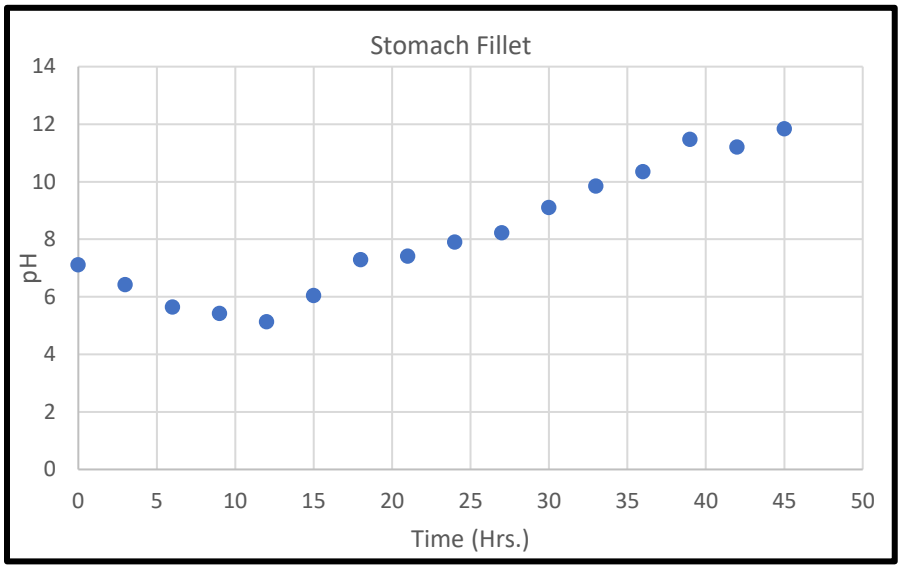

Figure 45: Image showing results obtained using commercial sensors on Stomach Fillet

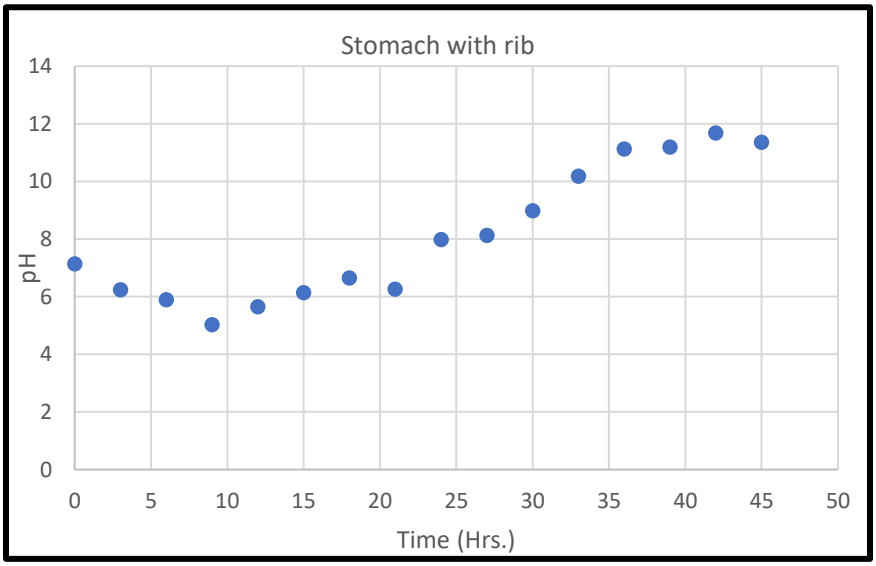

Figure 46: Image showing results obtained using commercial sensors on Stomach Fillet (Rib-Side)

CHAPTER 5

RESULTS AND DISCUSSION

5.1 Experiment 1

Figure 47 and 48 represent the pH of the Stomach at Room temperature and refrigeration temperature (BOX) respectively. Figure 49 and 50 represent the pH of the head at Room temperature (18ºC - 22ºC) and refrigeration temperature (0ºC - 22ºC). After comparison of figure 47 and 49 with figure 48 and 50 we can see that the pH in case of BOX remains constant throughout the experimentation and in case of fish sections kept at room temperature goes through variation. The sample stored at room temperature shows the above-mentioned spoilage steps.

In figure 47 and 49, pH goes on decreasing for 8 h-11 h which represent the release of lactic acid, that indicate Rigor Mortis period. After that, it starts gradually increasing till 18 h that shows autolysis during which muscles release amino groups. In the final stage, pH increases rapidly indicating spoilage inside the fish due to the growth and release of ammonia-based chemicals. However, the fish kept at refrigeration temperature shows no variation in the pH value of the fish.

After comparing figure 47 and 49, it is clearly visible that fish goes bad early at 8 h in case of Stomach at room temperature as compared to the head at room temperature. This happens due to presence of gut contains in the stomach.

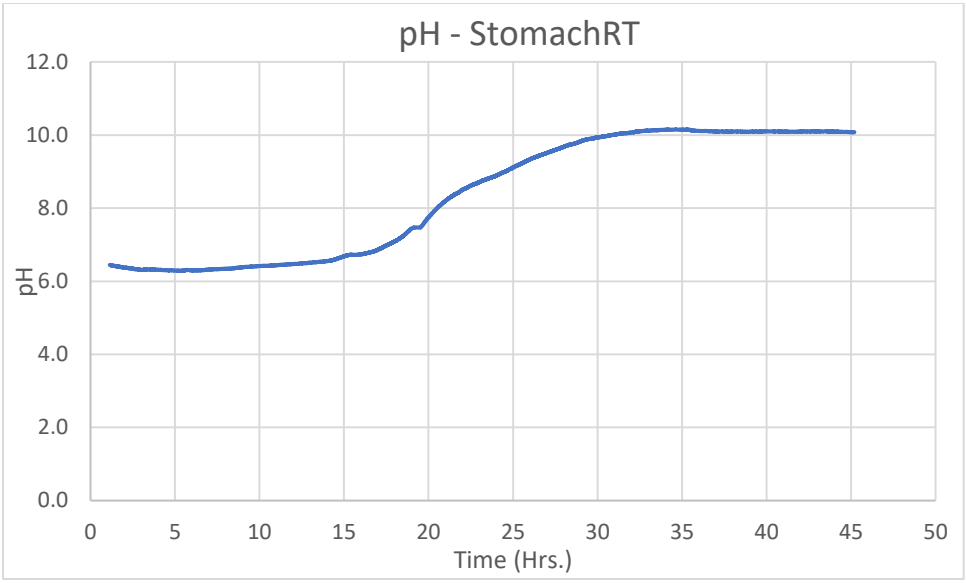

Figure 47: Plot of pH of Stomach at room temperature from Experiment 1

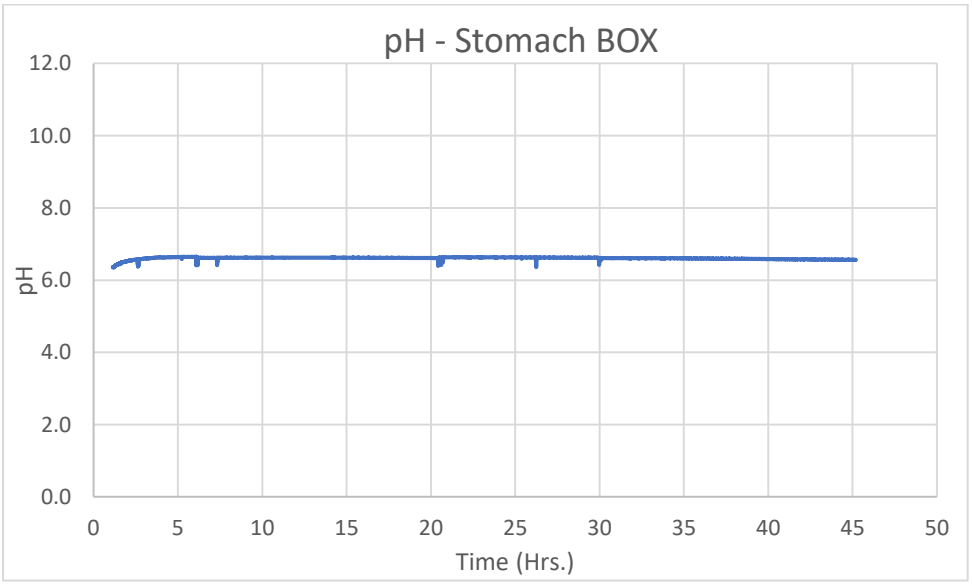

Figure 48: Plot of pH of Stomach at refrigeration temperature from Experiment 1

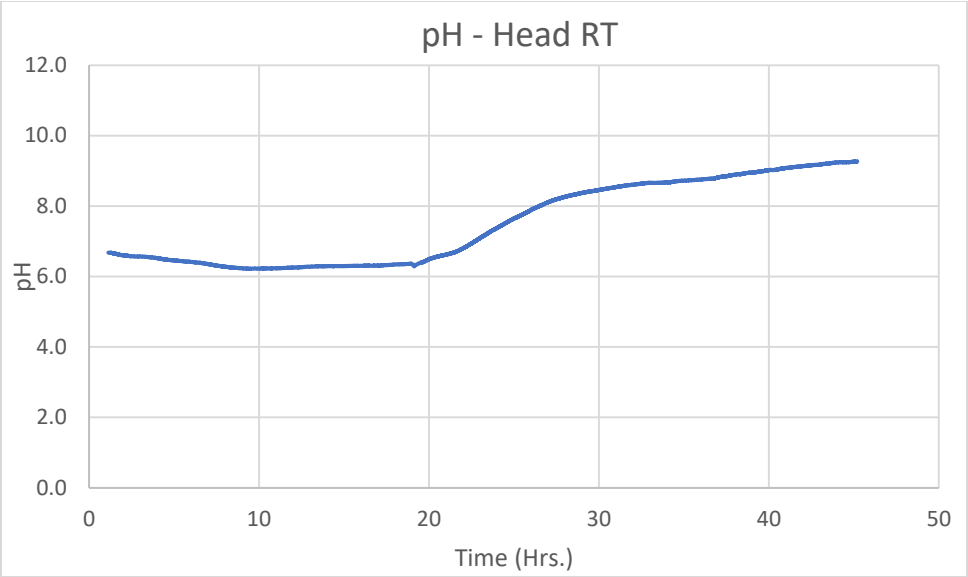

Figure 49: Plot of pH of Head at room temperature from Experiment 1

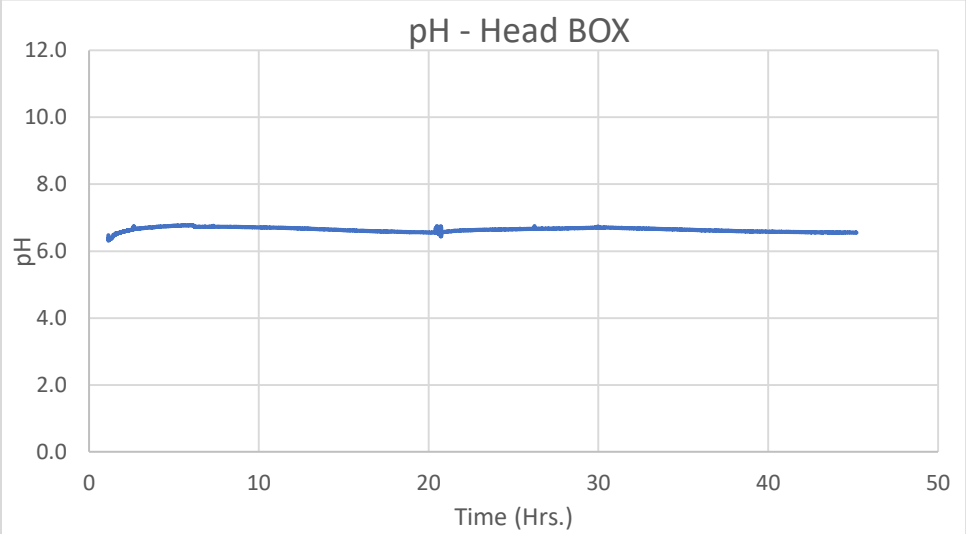

Figure 50: Plot of pH of Head at refrigeration temperature from Experiment 1

5.2 Experiment 2

In this experiment, two, Tilapia fishes were used for demonstration of the spoilage process in fish meats. One Tilapia fish is cut into two symmetrical sections of the stomach which other is cut into two sections of the fillet. Since in this case gut, contains are not present in stomach there will be no early spoilage seen in fish samples results. Here in this experiment, all the samples which were used for demonstration show similar trends in the curve indicating each spoilage stage clearly.

Figure 51, 52, 53, and 54 shows a decrease in pH for 10-11 h indicating rigor mortis, followed by a slight increase in pH indicating autolysis, and finally, there will be a sharp increase in pH indicating spoilage.

In this experiment, all fish samples are maintained at room temperature (18ºC -22ºC).

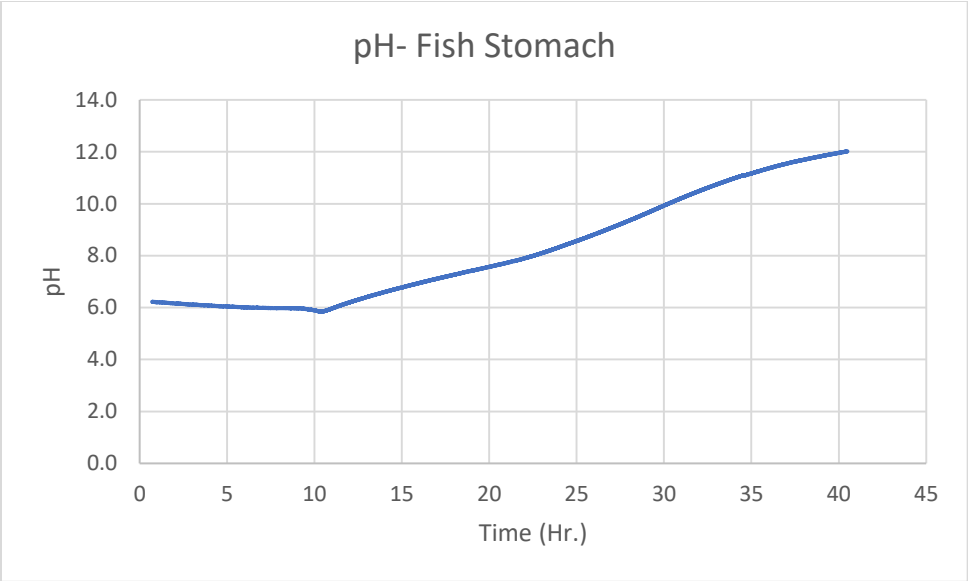

Figure 51: Plot of pH of Stomach at room temperature from Experiment 2

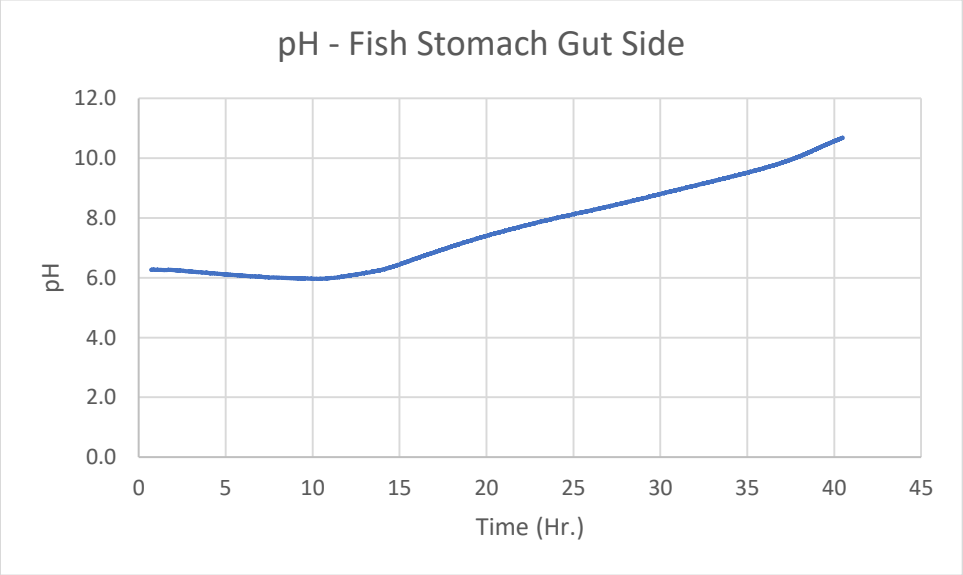

Figure 52: Plot of pH of Stomach Gut Side at room temperature from Experiment 2

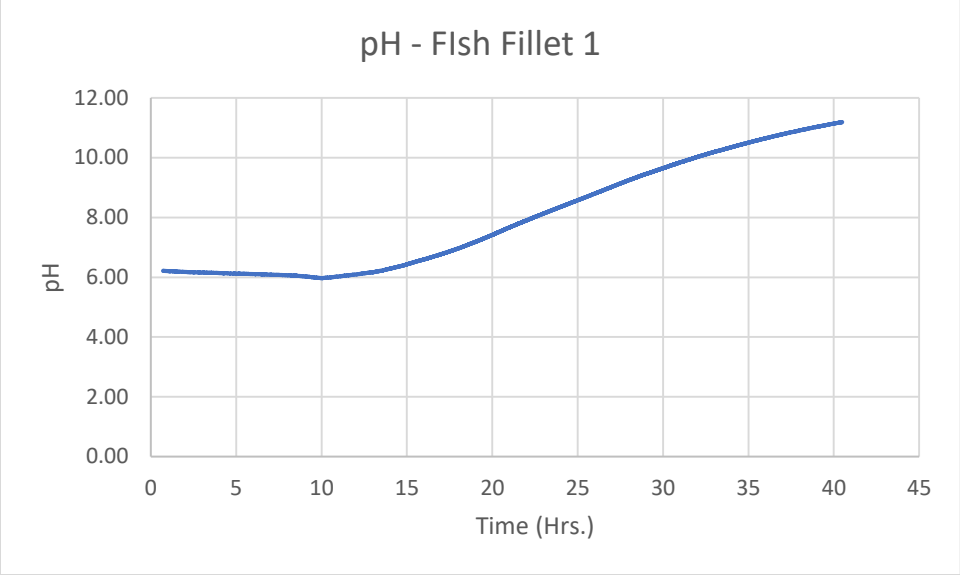

Figure 53: Plot of pH of Fish Fillet at room temperature from Experiment 2

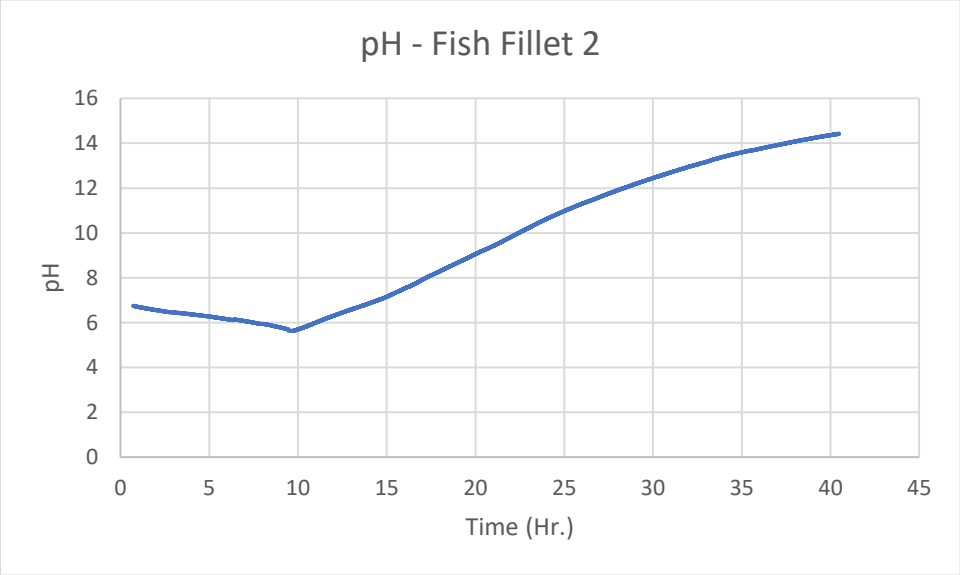

Figure 54: Plot of pH of Fish Fillet at room temperature from Experiment 2

5.3 Experiment 3

In this experiment, Catfish was used for demonstration and results from following graphs indicates spoilage inside the fish also the stages involved in spoilage of the fish. Further, the results of this experiment are compared with the results from the commercial sensor.

Figure 55 shows the variation of pH monitored on non-rib side stomach and comparison with the commercial sensor. Figure 56 indicates the variation of pH on rib side stomach and comparison with the commercial sensor. Figure 57 and 58 represent the pH value variation on the tail section on a rib and non-rib side respectively of the fish. Since in this experiment tail section was not separately cut, here for consideration purpose the rear side of the is considered as a tail section.

In this experiment, all fish samples are maintained at room temperature (18ºC -22ºC).

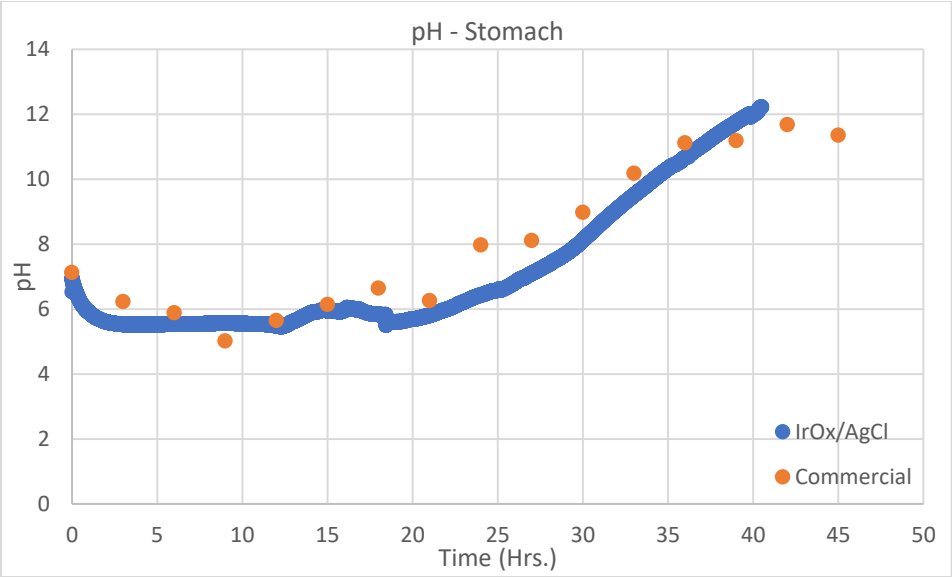

Figure 55: Comparison of pH of Stomach at room temperature from Experiment 3 with commercial sensor

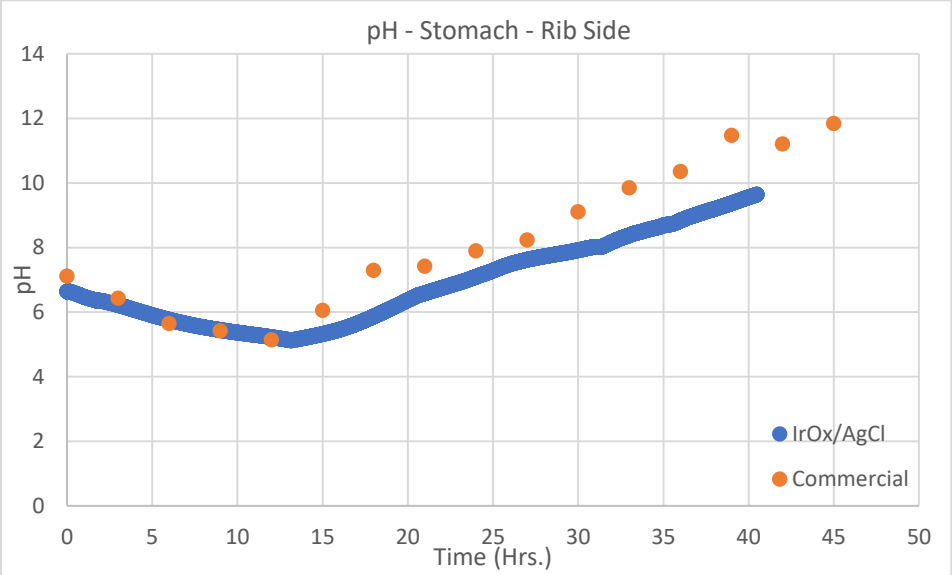

Figure 56: Comparison of pH of Stomach rib -side at room temperature from Experiment 3 with commercial sensor

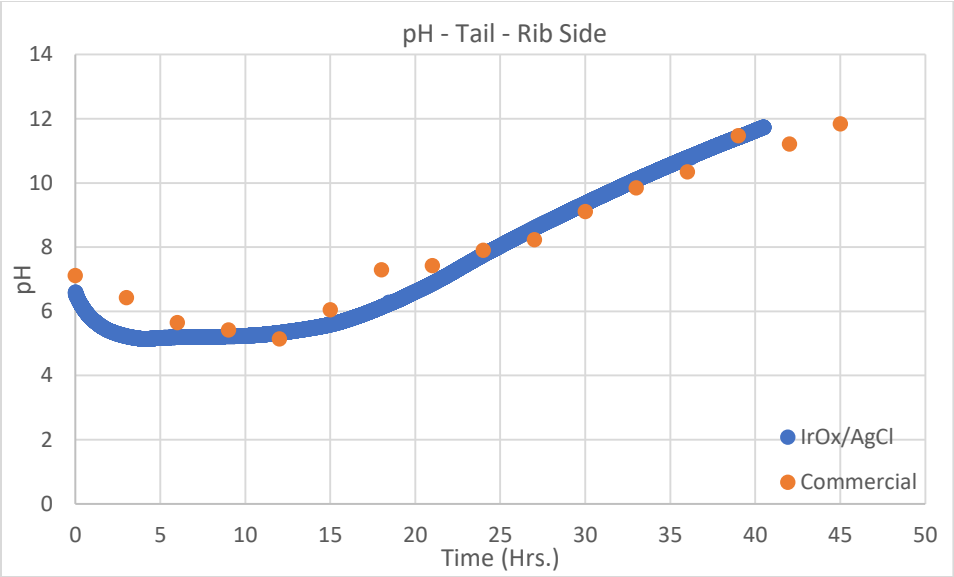

Figure 57: Comparison of pH of Tail rib -side at room temperature from Experiment 3 with commercial sensor

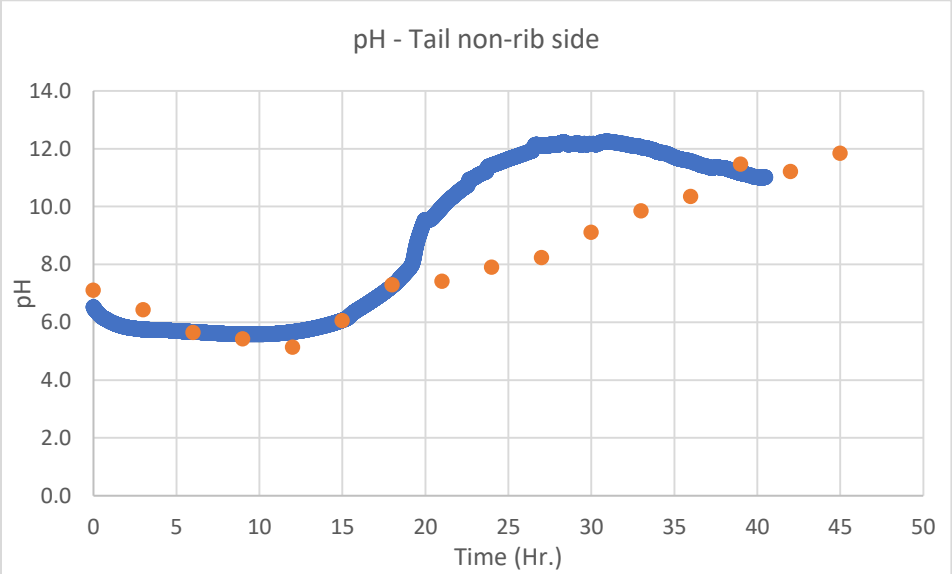

Figure 58: Comparison of pH of Tail non- rib -side at room temperature from Experiment 3 with commercial sensor

5.4 Experiment 4

In this experiment, similar to the previous experiment the Catfish is used for the demonstration purpose. The only difference in this experiment is one of the flexible IrOx/ AgCl sensors was inserted into the flesh of the fish, the other two sensors placed below the fish stomach.

In figure 59 and figure 60 difference in the pH values are visible, figure 60 represents pH values recorded by the sensor after insertion of the sensor into the flesh and figure 59 represent the pH values recorded by the sensor place below the fish.

In this experiment, all fish samples are maintained at room temperature (18ºC -22ºC).

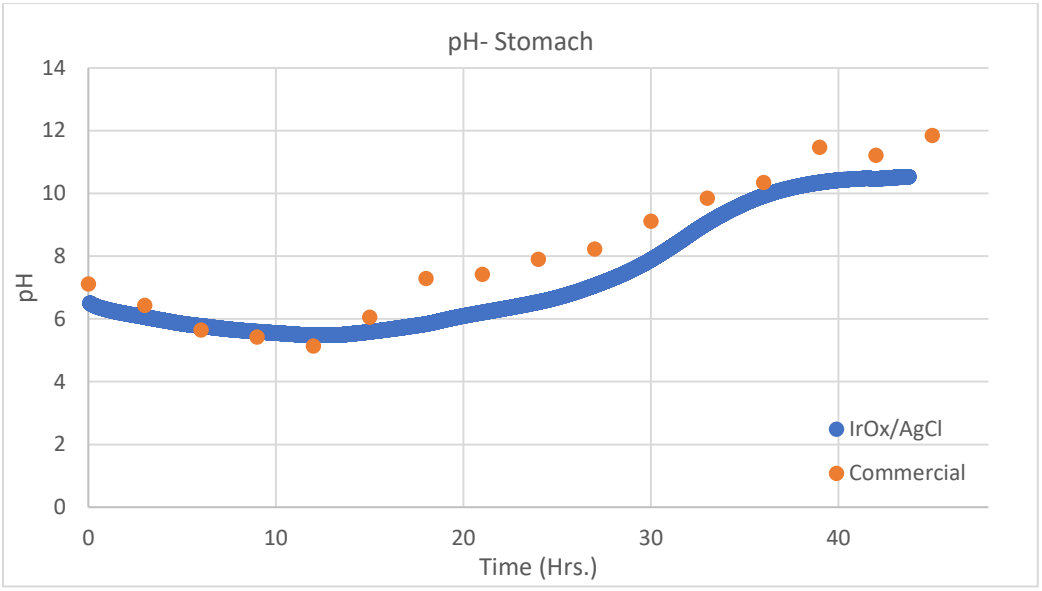

Figure 59: Comparison of pH of Stomach at room temperature from Experiment 4 with commercial sensor

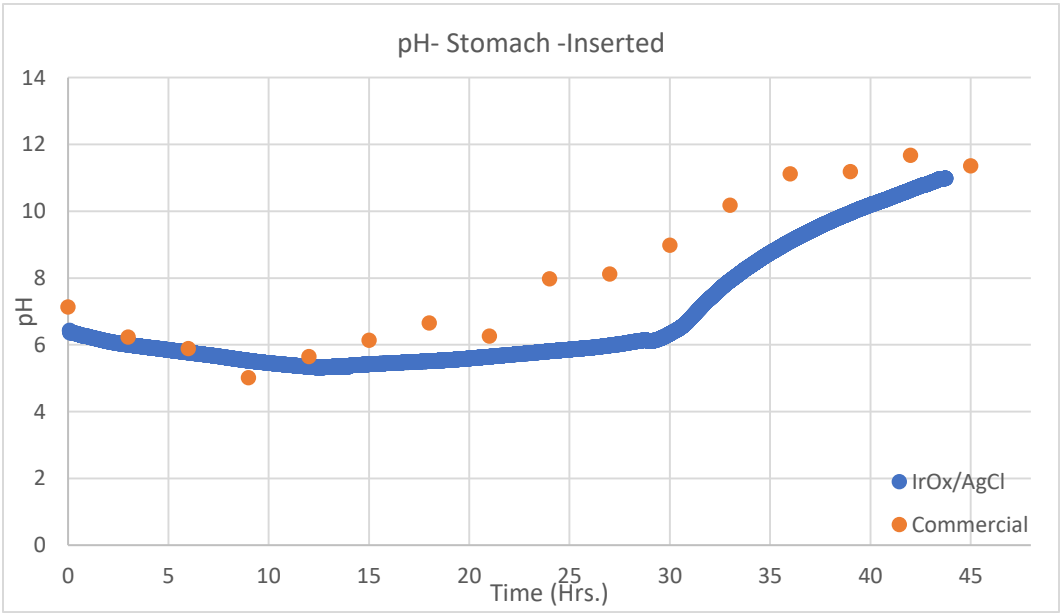

Figure 60: Comparison of pH measured by inserting sensor into Stomach at room temperature from Experiment 4 with commercial sensor

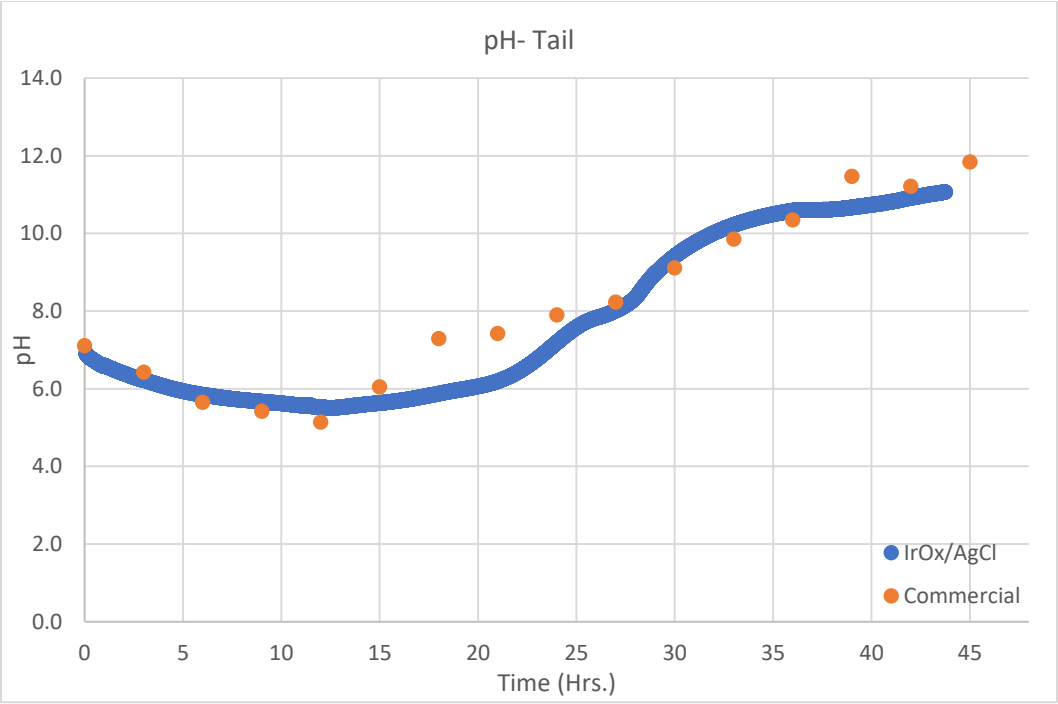

Figure 61: Comparison of pH of Tail at room temperature from Experiment 4 with commercial sensor

5.5 Results Comparison

5.5.1 Experiment 1 vs Experiment 2

Figure 62 represents the comparison of pH values from Stomach of Tilapia fish for Experiment 1 and Experiment 2. Since there was gut contains were present in Stomach used in case of experiment 1, there is the quick increase in pH and early end of rigor period, that is because gut will increase body temperature leading to rapid growth in the bacterial generation. However, in the case of Experiment 2 gut, contains were removed therefore the growth of bacteria reduces.

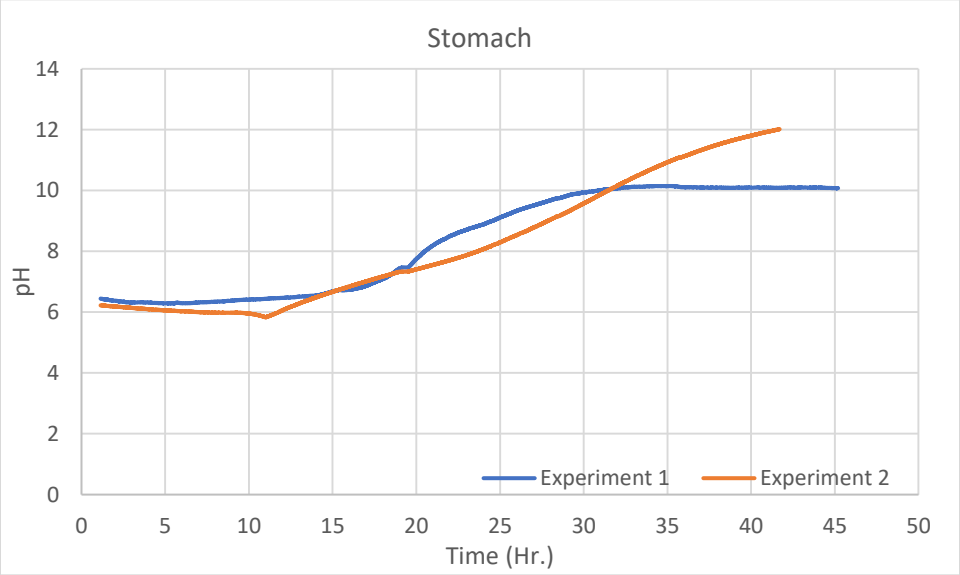

Figure 62: Comparison of pH of Stomach at room temperature from Experiment 1 with Experiment 2

5.5.2 Experiment 3 vs Experiment 4

Figure 63 and 64 represents the comparison of pH values from the Stomach and Tail of Catfish for Experiment 3 and Experiment 4 respectively. Here both the experiments were contained at room temperature (18ºC-22ºC) for 40-44 h. pH measurements from below curve show the initial decrease in pH due to rigor mortis, followed by the increase in pH due to biochemical and microbiological spoilage and generation of ammonia.

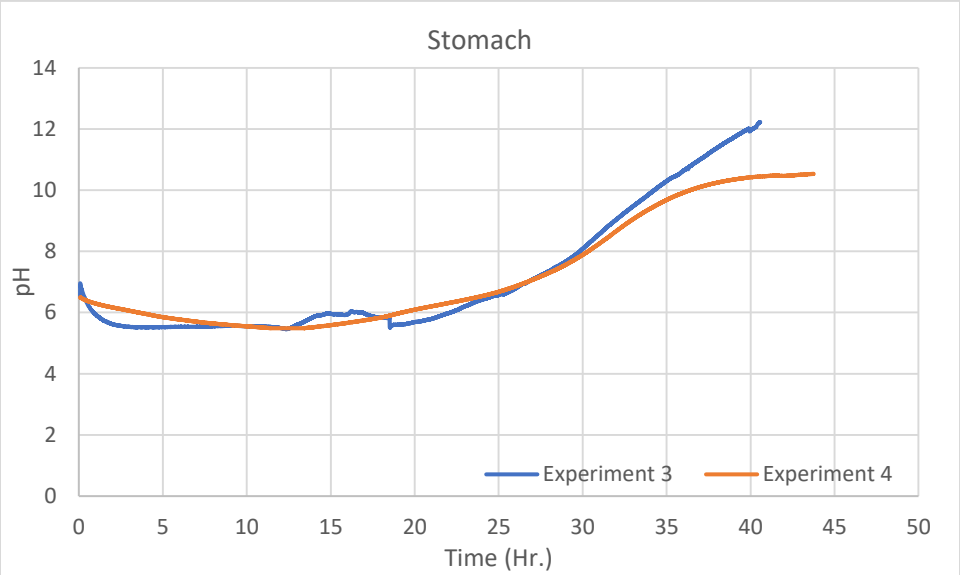

Figure 63: Comparison of pH of Stomach at room temperature from Experiment 3 with Experiment 4

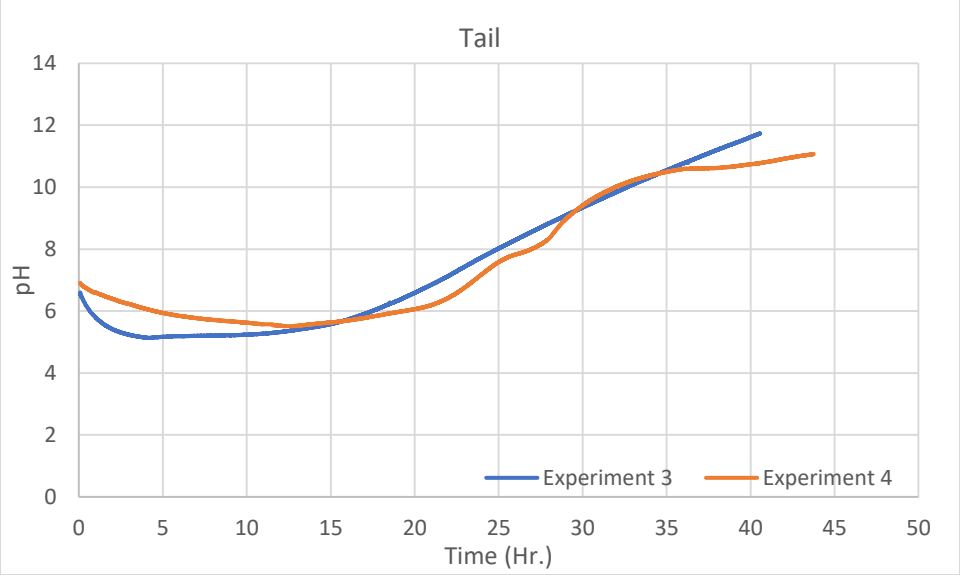

Figure 64: Comparison of pH of Tail at room temperature from Experiment 3 with Experiment 4

CHAPTER 6

CONCLUSION AND FUTURE WORK

The pH sensing system described in this work provides a convenient way to monitor the quality of food/produce. Since it can detect the bacterial activity inside any food product and can correlate with the pH value, that makes this sensing system more suitable for multiple food and agricultural applications where bacterial activity is required to monitor. Both of the IrOx working and AgCl reference electrodes were fabricated on the flexible substrate. Therefore, this sensor can be deformed on any food surface to measure food pH without puncturing or pressing into the food.

The results of the pH profile for the fish sections stored at room temperature shows the distinct difference. This indicates that the flexible IrOx/AgCl sensors are suitable for a wide temperature range. Also, the sensing system described here is suitable for real-time-on-demand continuous sensing requirement.

In order to make this sensing system more user-friendly the wireless sensing mechanism can be embedded into the existing system without changing the functional requirement. The next step in this study will be to design a small patch like device that is directly stick to any food sample and pH will be recorded wirelessly thus transforming existing system into user-friendly method for food/produce and agriculture spoilage detection.

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PART B: BIOMEDICAL APPLICATION

CHAPTER 7

INTRODUCTION

7.1 Motivation

Aspirational Pneumonia generally is known as Aspiration is one of the important causes of anesthesia related death [1] and also complicates the surgical procedure. Aspiration is the entry of material for example food or drink, or stomach juices from the gastrointestinal tract (esophagus) into the larynx (trachea) and lower respiratory tract to the lungs.

The major cause of aspiration is conditioned involving depression in the level of consciousness such as alcohol/drug overdose, anesthesia [2]. An anesthesia effect, full stomach (emergency surgical procedure), delaying in the gastric emptying, decreased gag reflex, as well as conditions such as obesity, stroke, and pregnancy can all increase the risk of aspiration in the semiconscious. A nasogastric feeding tube also increases the risk of aspiration and aspirational pneumonia [3].

The Anesthetic Incident Monitoring Study database consists of 5000 reports were studied and out of 5000 cases, 240 incidents of aspiration and vomiting cases were found [4]. Of these 240 incidents, 133 cases of aspiration were recorded. Out of these all cases, 5 cases of deaths were reported. Contributing factors for all of these cases were studied, these factors include the error in judgment, the fault in technique. Implementation of simple techniques may have prevented at least 60% of the all cases of aspiration [4]. One of the simple techniques to avoid aspiration involved securing the airway. Airway can be secured by using Supraglottic Airway Devices (SAD) such as Laryngeal Mask Airway (LMA)

during the surgical procedure. Different types of SAD and their way of application were discussed in the following chapter.

According to the World Health Organization (WHO), approximately 250 million patients undergo general anesthesia for major surgeries [5]. As per reports from Royal College of Anesthesia, almost 60% of the patients used SAD [5], so we can estimate that annually worldwide approximately 150 million such devices have been used.

Reports revealed that even after implementation of SAD during surgical procedure there are still 80% cases of aspiration were reported due mal-positioning of SAD [5]. Causes of a mal-positioning [5] of SAD involved the Improper fitting of SAD with the esophageal orifice creating additional space for gastric fluid entering, Blind insertion SAD into person mouth does not an exact judgment about fitting with the esophagus. Misalignment of SAD with the tracheal orifice, downward or double folding of epiglottis creating in error in alignment of SAD on the esophagus.

Therefore, there is very essential need of active and real-time monitoring gastric reflux episode in were esophageal canal during use of anesthesia and surgery.

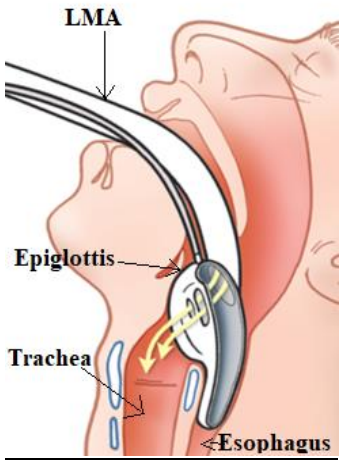


Figure 65: Image showing inner portion of oral cavity with LMA

7.2 Objective

The main objective of this part of the thesis is to actively track and real-time monitor the gastric reflux in esophageal channel coming from the stomach. Further to identify the nature of the gastric reflux like acidic or alkaline.

In this part, tracking and actively monitoring is demonstrated by using impedance sensor and identifying of gastric content is demonstrated by using pH sensor. The demonstration of the proof of concept of a dual sensing mechanism for gastric fluid monitoring is achieved by embedding impedance and pH sensors on LMA and further to simulate the gastric reflux episode for monitoring.

Dual sensing system involved the use of 3 pairs of impedance sensor and one pH sensor. For demonstration, here mimicking device (discussed in chapter 11) is designed to carry-out experiments involving gastric reflux.

CHAPTER 8

FABRICATION AND WORKING OF IMPEDANCE AND pH SENSOR

8.1 Fabrication and Working of Impedance sensors:

In this part, the main purpose of using an impedance sensor is mainly to detect real-time occurrence of gastric reflux episode and to gauge the level of rising of gastric fluid in the esophageal channel. The reason of using impedance sensor in this application is, Impedance changes instantly when any fluid touches the electrodes of the impedance sensors. Two copper (Cu) electrodes are used as an impedance sensor in this method. The dimensions of each of the two electrodes are 12mm x 4mm, with 2.5 mm gap in between the electrodes. Connecting wire are attached to these electrodes using silver epoxy.

The resistance of 450Ω is connected in parallel with the electrodes as shown in the figure below. The reason for using and attaching resistance is to avoid unwanted fluctuations caused before any fluid makes contact with the electrodes.

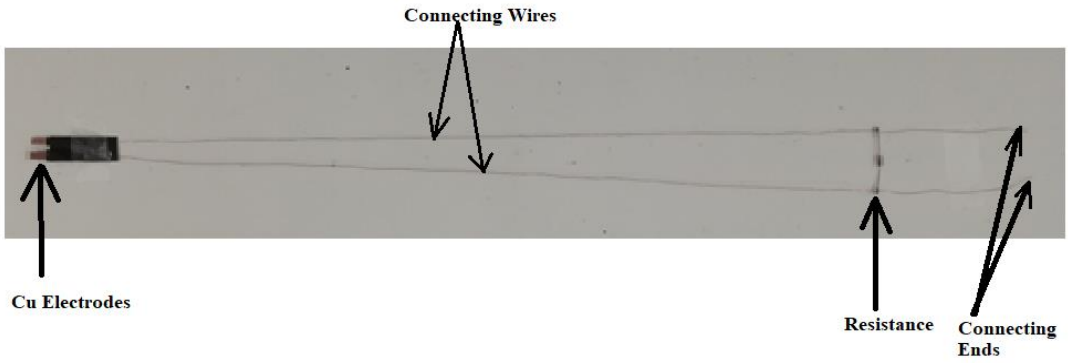


Figure 66: Image showing Impedance sensor along with resistance

8.2 Fabrication and Working of pH sensors:

Fabrication and working of pH sensor are explained in Chapter 2.

CHAPTER 9

SUPRAGLOTTIC AIRWAY DEVICES

LMA is supraglottic device (SAD) and, it is used since 1988. Since it is very difficult to intubate some patient especially in case of emergency, this device is developed as alternative traditional intubation. SAD has no risk as those are associated with intubation. The main advantage of SAD is it reduces the cause of gastric distention and are less likely to cause aspiration in the patient.

When inserted correctly SAD sits above epiglottis in the hypopharynx. The proximal end of the SAD can be attached to the Ambu bag. The distal end of an elliptical-shaped mask of SAD delivers air through the trachea and into lungs, at the same time it blocks the esophagus to prevent air from causing gastric reflux.

Although it considered being safe to use SAD during surgery, some complication has been noticed during surgery. As discussed in above chapter aspiration of gastric contents is major challenge in the use of SAD.

Types of SAD:

1st generation SAD (figure 67): They includes the cLMA, flexible LMA, laryngeal tube. The major disadvantage of this device they may or may not protect against aspiration.

The 2nd The generation of SAD (figure 68): They includes PLMA, i-gel, SLMA, etc. This generation of SAD are designed for safety, and, it has design features to reduce aspiration. But, in several cases the, effectiveness of this device is proven unsatisfactory.

Figure 67: 1st generation LMA

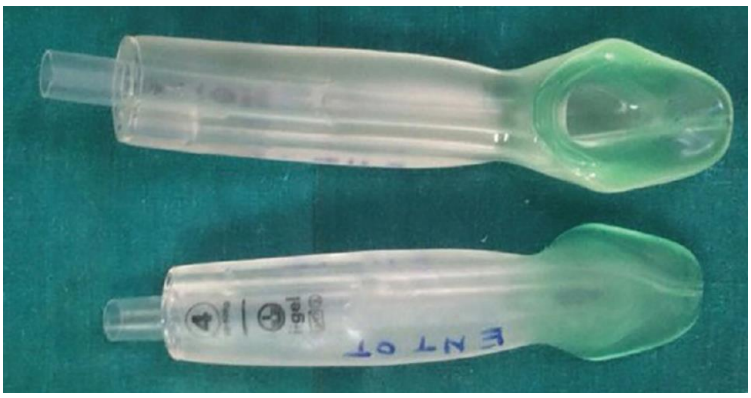


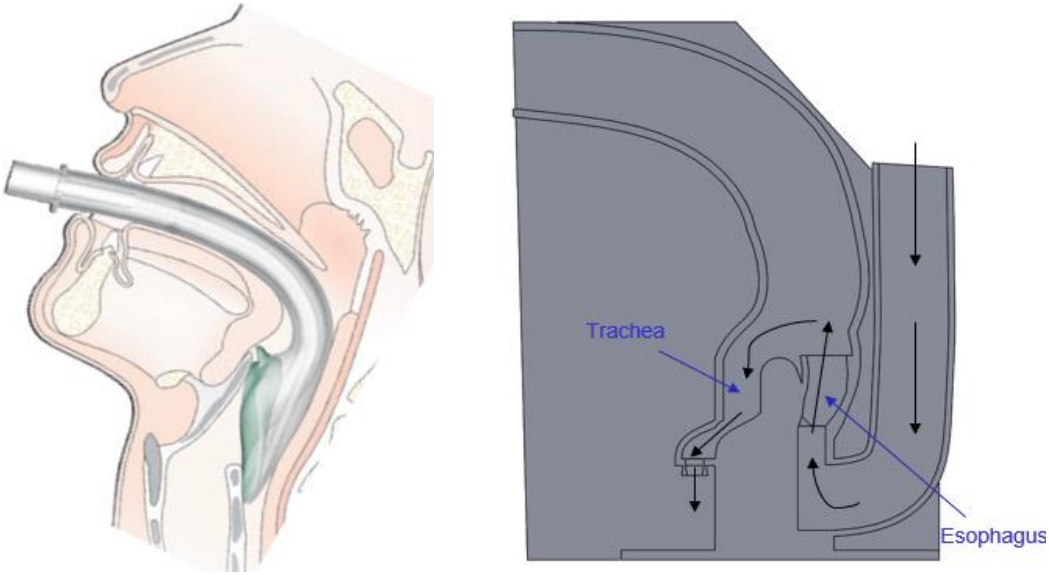
Figure 68: 2nd generation LMA

CHAPTER 10

EXPERIMENTAL SET-UP

10.1 Experimental Test Rig:

It is difficult to predict the gastric reflux episode in real-time it might involve complex and expensive technique. Also, it is tough to predict how the gastric reflux episode happen. To overcome this challenge and to ensure the continuous data acquisition, special model (Figure 69 (b)) is conceptualized, designed, and 3D printed using Solidworks. This device mimics the Pharynx, Larynx, Esophagus, Trachea, and mouth cavity. Also, it gives enough room for LMA to sit on top of the esophageal opening.



(a) (b)

Figure 69: (a) Image showing inner portion of oral cavity, (b) Image showing model designed for experimentation purpose

10.2 Mounting of Sensors on the LMA:

As discussed in the chapter (9), three impedance sensors and one pH sensor are mounted on top of the LMA. As shown in fig below. Impedance sensor 1 (IS 1) is the first sensor with whom the fluid will make the first contact. After that pH sensor is attached right on above of IS 1 to predict the type of the gastric fluid. After the impedance sensor 2 is attached and impedance sensor 3 is attached on the back side on LMA.

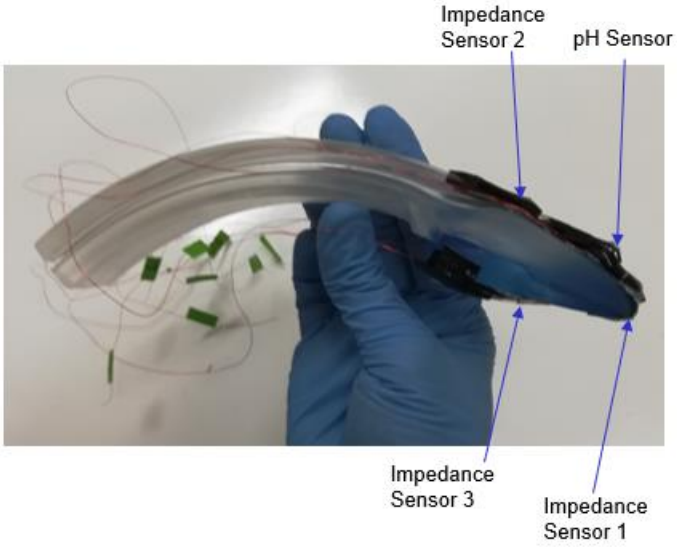


Figure 70: Side profile of LMA with sensors embedded

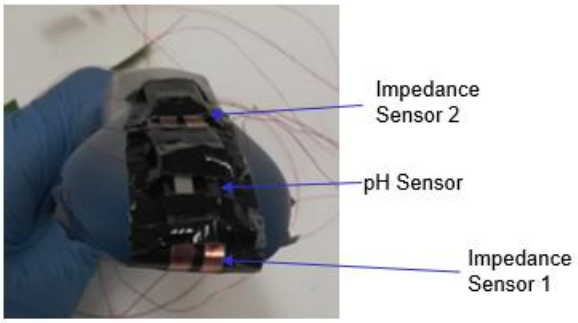


Figure 71: Front profile of LMA with sensors embedded

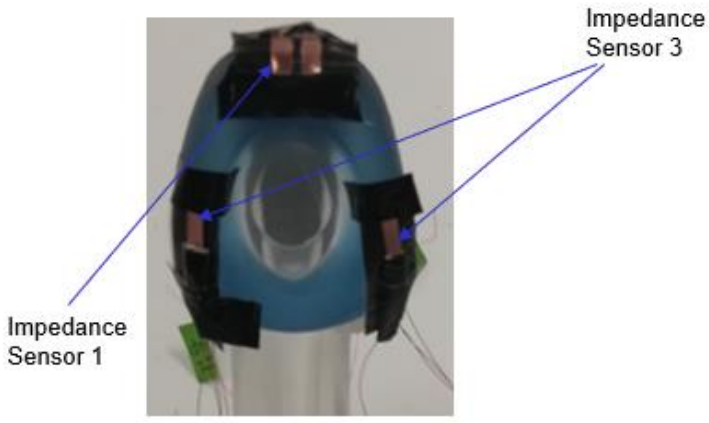


Figure 72: Back profile of LMA with sensors embedded

Figure 73: Image showing final experimental set-up with LMA

All sensor here is attached to the NI-DAQ and LabView based program is designed to monitor the gastric reflux. For simulation to mimic the gastric fluid, the KCl solution is used.

CHAPTER 11

RESULTS AND DISCUSSION

Here 70 seconds of reflux episode is created and during which the by using impedance sensors and pH sensor the reflux was tracked and monitored. From figure (74) we can predict that the fluid makes the first contact with impedance sensor at 38 seconds. After that, it touches the pH sensor at 40 seconds figure (75). After touching the pH sensor fluid moves upward and makes contact with impedance sensor 2 at 42 seconds figure (76). Since impedance sensor 3 is located at the back of the LMA fluid makes final contact with it at 47 seconds figure (77) before entering into the trachea.

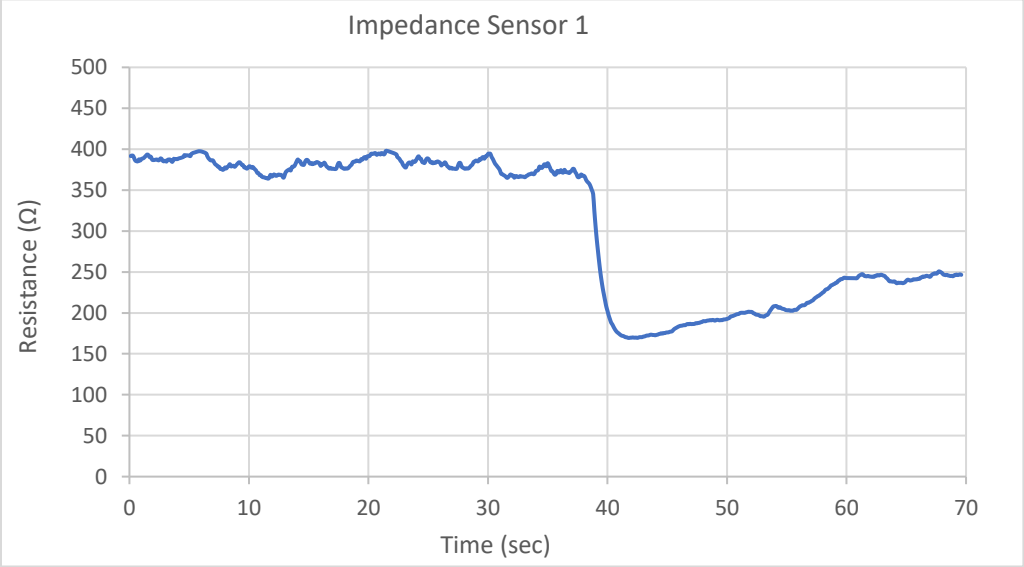


Figure 74: Plot showing results of Impedance Sensor 1

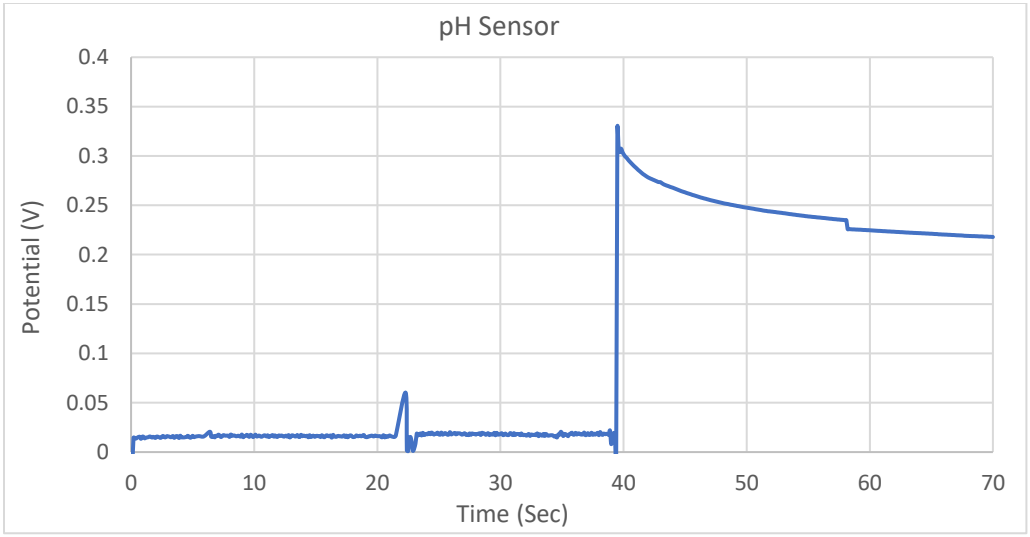


Figure 75: Plot showing results of pH sensor

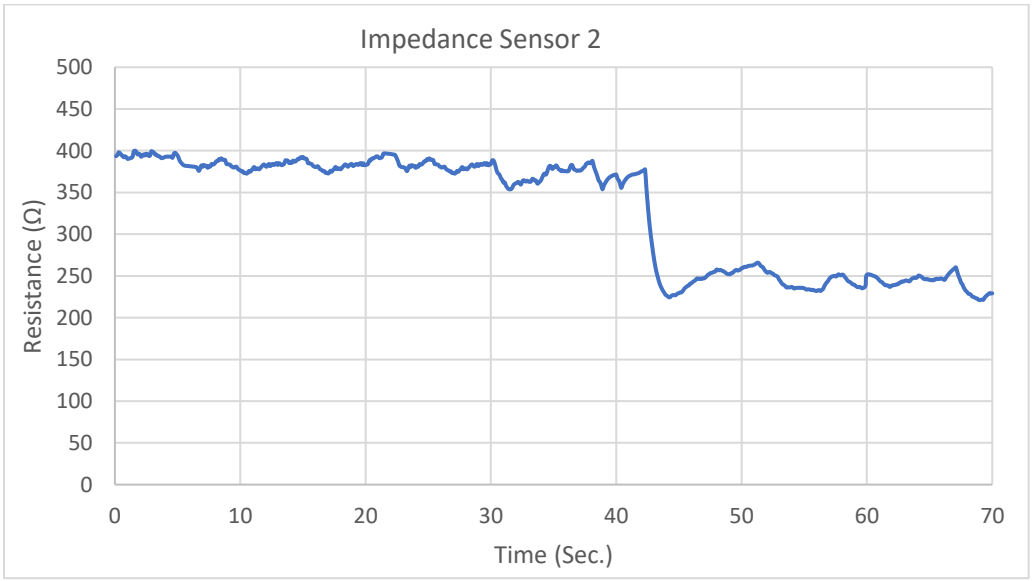


Figure 76: Plot showing results of Impedance Sensor 2

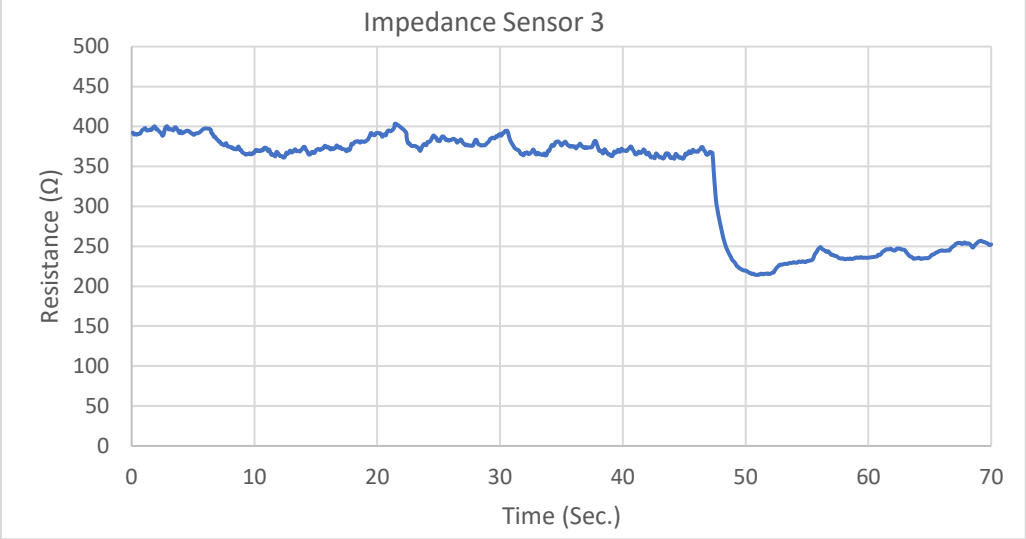


Figure 77: Plot showing results of Impedance Sensor 3

CHAPTER 12

CONCLUSION AND FUTURE WORK

The integration of dual sensors on LMA provides a better way for live tracking and real-time monitoring of gastric reflux. Impedance sensor used here to provides a better way of measuring the level of gastric reflux inside the esophageal channel. Also, the pH sensor used here to provides an ideal prediction of type the of gastric fluid. This dual sensing system will give the surgeon an advantage to take a decision based on the real-time data prediction. Once the surgeon noticed the reflux coming from the channel, surgeon can use gastric liquid suction pump to suck the fluid out before it enters the patient's lungs or before it creates/ increases complications. Both of the impedance and pH sensors used here does not have any adverse effect on human internal organ. Since both of these sensors are flexible, therefore, they can be easily integrated into the LMA without changing and dimensional requirement.

To avoid wiring hassle, this dual sensing system can be made wireless. That will give further ease to a surgeon if they can predict the reflux directly on screen without the requirement of the any wiring system.

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Biographical Information

Shardul S. Pawar received his Bachelor's degree in Mechanical Engineering from Maharashtra Institute of Technology, University of Pune, India in July 2015. He pursued his Master's in Mechanical Engineering at The University of Texas at Arlington from Fall 2016. He joined the iMEMS group and started working with Dr. Jung-Chih Chiao in 2017. Shardul served as a Technology and Management Intern at the Office of Technology Management at The University of Texas at Arlington for one and half year. His research interest includes Product Design, Prototype Development, MEMS sensor, aerodynamics, and thermal system management.