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Characterization of Agar Hydrogel Electrolyte of Zinc-air Battery

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DECLARATION

I declare that this thesis entitled "Characterization of Agar Hydrogel Electrolyte of Zinc Air Batteries." is the result of my research except as cited in references.

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Characterization of Agar Hydrogel Electrolyte of Zinc Air Batteries.

ABSTRACT

In this work, the characterization of Agar hydrogel electrolytes of zinc-air batteries was studied. Issues with synthetic polymer hydrogels include a lack of inherent bioactivity while having acceptable mechanical and other properties. Hydrogel electrolytes are ecologically safe, biocompatible, degradable, and have inherent flexibility, which makes them ideal for real-world energy storage applications. Agar powder was first prepared by dissolving it with 100 mL distilled water and heated until 80 – 100℃ to form hydrogels. The ideal Agar hydrogel composition was 1:10, and it was dissolved in various KOH concentrations to create an electrolyte. This study's characterization was Open circuit potential (OCP), FTIR, morphological, and XRD. A 4M KOH was recorded for the best conductivity in the agar hydrogel electrolyte.

Keywords: Hydrogel electrolyte, Agar, Hydrogel, Zinc air batteries, Potassium hydroxide.

Pencirian Agar Hidrogel Elektrolit Bateri Udara Zink.

ABSTRAK

Dalam kerja ini, pencirian elektrolit Agar hidrogel bagi bateri udara zink telah dikaji. Isu dengan hidrogel polimer sintetik termasuk kekurangan bioaktiviti yang wujud sementara mempunyai sifat mekanikal dan lain-lain yang boleh diterima. Elektrolit hidrogel adalah selamat dari segi ekologi, biokompatibel, boleh terurai, dan mempunyai fleksibiliti yang wujud, yang menjadikannya sesuai untuk aplikasi penyimpanan tenaga dunia sebenar. Serbuk agar mula-mula disediakan dengan melarutkannya dengan 100mL air suling dan dipanaskan sehingga 80 – 100℃ untuk membentuk hidrogel. Komposisi hidrogel Agar yang ideal ialah 1:10, dan ia telah dilarutkan dalam pelbagai kepekatan KOH untuk menghasilkan elektrolit. Pencirian kajian ini ialah potensi litar terbuka (OCP), FTIR, morfologi dan XRD 4M KOH direkodkan untuk kekonduksian terbaik dalam elektrolit.

Kata kunci: Elektrolit hidrogel, Agar, Hidrogel, Bateri air zink, Kalium hidroksida.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

1.1 Background of study

Gel electrolytes are showing promise as practical materials for technological uses. Due to their expected conductivity being very close to that of the corresponding liquid electrolyte, which is typically greater than 10−3 S cm−1, gel electrolytes are used in many electrochemical solid state ionic devices, including fuel cells, sensors, high-energy density batteries, and electrochemical display devices (Mohamad, 2006).

Very few studies on polymer-based electrolyte zinc-air batteries have been published, despite the fact that the original zinc-air (Zn-air) battery was first commercialised in the 1920s. Examples of hydroponic gels used as the electrolyte component in a zinc-air battery are epichlorohydrin-ethylene oxide [P(ECH-co-EO)] and polyvinyl alcohol (PVA)–polyethylene oxide (PEO)–glass-fibre-mat doped with potassium hydroxide (KOH) (Chen et al., 2020).

The zinc–air batteries attract more attention due to their high energy density, safety, environmental protection, and low cost. However, the traditional aqueous electrolyte has the disadvantages of leakage and water evaporation, which cannot meet the application demand of zinc–air batteries.

Hydrogels possessing good conductivity and mechanical properties become a candidate as the electrolytes of flexible zinc–air batteries. However, the problems of electrolyte leakage and water evaporation in traditional zinc-air batteries not only have great safety risks but also directly affect the lifetime and performance of the batteries, which cannot meet the demands of flexible electronic devices.

Hydrogels are three-dimensional macromolecular polymeric substances that can absorb water and swell, which can remain gelatinous in water without dissolution. Hydrogel electrolytes have been widely used in flexible zinc-air batteries due to their good operability and application, certain ionic conductivity, mechanical strength, and good interface contact with electrodes.

However, an insulating zinc oxide layer will be formed between the SSE and zinc electrode surface in the discharge process. The increase in the thickness of the zinc oxide layer will gradually lead to battery failure. To alleviate the passivation of the zinc electrode and the formation of the insulating zinc oxide layer (P. Zhang et al., 2021).

Hydrogels are polymeric polymers with three-dimensional networks that can contain significant volumes of water. Natural hydrogels are widely utilized in industrial and environmental applications, but synthetic hydrogels are replacing them due to their increased water absorption capacity, long service life, and diverse variety of basic chemical resources (Ahmed, 2015).

Hydrogels are cross-linked polymeric structures that swell with water. Since the development of poly (2-hydroxyethyl methacrylate), or PHEMA, the usage of hydrogels in medical applications has grown in popularity. Hydrogels may be tailored to satisfy particular requirements such as swelling and mechanical properties, making them ideal for a wide range of biomedical applications ranging from contact lenses to controlled-release medication delivery and tissue engineering. This will touch on hydrogel characterisation, biocompatibility, biomedical applications, and mathematical modelling of hydrogels.

Agar is a natural polymeric polysaccharide with a high number of hydroxyl and ether groups that can form hydrogen bonds with polyacrylamide. To improve the water retention and ionic conductivity of polyacrylamide gels, and maintain their shape without leakage under external compression, providing a long-term stable operating environment for Zinc-Air Batteries. (Weiying et al., 2022).

Agar is a polymer found in the cell walls of agarophyte algae. It is protected in a system of crystallised cellulose fibres, which serves as its polysaccharide reserve (Armisén & Galatas, 2009).

Agar hydrogel is a form of hydrogel made from agar. A natural polysaccharide taken from seaweed. Because of their unique features and wide range of uses, agar hydrogels have received substantial interest in a variety of scientific and technical areas (Jiang et al., 2023).

Agar hydrogel electrolytes are a solid-state alternative to liquid electrolytes that overcome leakage and flammability problems. The hydrogel generates a three-dimensional network structure with a high-water content, which facilitates the flow of ions required for the battery's electrochemical processes (Liu et al., 2021).

1.2 Problem Statement

The problem statement is conventional electrolytes, which can leak, have limited mechanical flexibility, and are incompatible with electrode materials, restrict the performance of zinc-air batteries. Agar hydrogels have showed promise as alternative electrolytes, but further research into their performance and appropriateness for zinc-air batteries is required. The goal is to study and characterise the features of agar hydrogel electrolytes, such as their ionic conductivity, mechanical stability, compatibility with electrode materials, and overall performance in zinc-air batteries.

Conventional electrolytes, which are utilised in a variety of technical applications such as batteries and electrochemical devices, frequently confront problems with leakage, mechanical flexibility, and compatibility with electrode materials. Hydrogels have emerged as a viable technical respond to these issues. However, in order to overcome these technical tissue difficulties, it is required to investigate the feasibility and efficacy of hydrogenizing common electrolytes. The challenge is to determine whether hydrogels can successfully address the technical tissue issues associated with conventional electrolytes by improving mechanical flexibility, reducing leakage, improving compatibility with electrode materials, and overall improving performance in technical applications.

1.3 Objective

The objectives of this research are:

I. To investigate morphology and functional group of agar hydrogel containing different KOH concentrations

II. To evaluate circuit potential of Agar-KOH hydrogel as potential electrolyte in zinc-air battery

1.4 Scope of Study

The scope of the study score is to provide the best composition of Agar to obtain a texture like a thick sample and to investigate the zinc-air batteries produced from agar hydrogel electrolytes. The agar hydrogel electrolyte was characterized using Fourier Transform Infrared Spectroscopy (FTIR) to study the functional groups of the agar hydrogel electrolyte and the voltage performance of a zinc-air battery system was investigated using open circuit potential (OCP).

1.5 Significant of Study

The significance of this study is to see if this Agar is a natural polymer that can be used as a hydrogel. Agar is a well-known polysaccharide that has long been used in hydrogels. Agar hydrogels have been shown in studies to be biocompatible and work great. These hydrogels' mechanical properties, biocompatibility, and adaptability were also found to be quite good, including self-healing ability, viscosity, and toughness. The benefit of electrolytes and medications is that they are much safer and better for the environment because they are natural and inexpensive ingredients. As a result, Agar is one of the best options for energy storage.

CHAPTER 2

LITERATURE REVIEW

2.1 Zinc-Air Batteries

Metal-air batteries are gaining attention due to their high specific energy density when compared to other energy storage technologies, particularly Li-ion systems, from both basic and industrial perspectives (Li & Lu, 2017).

Among metal-air batteries, zinc-air batteries provide a safe, environmentally beneficial, potentially cheap, and simple means to store and supply electrical energy for portable and stationary equipment as well as electric vehicles.

Zinc-air batteries are categorised as primary (including mechanically rechargeable), secondary (electrically rechargeable), and fuel cells. Since many years, research on primary zinc-air batteries has been effectively consolidated.

On the other hand, research on electrically rechargeable batteries still requires more efforts to tackle materials science and electrochemical difficulties associated with charge and discharge processes. Furthermore, zinc-air fuel cells have a high potential for smart grid energy storage and another. (Caramia & Bozzini, 2014).

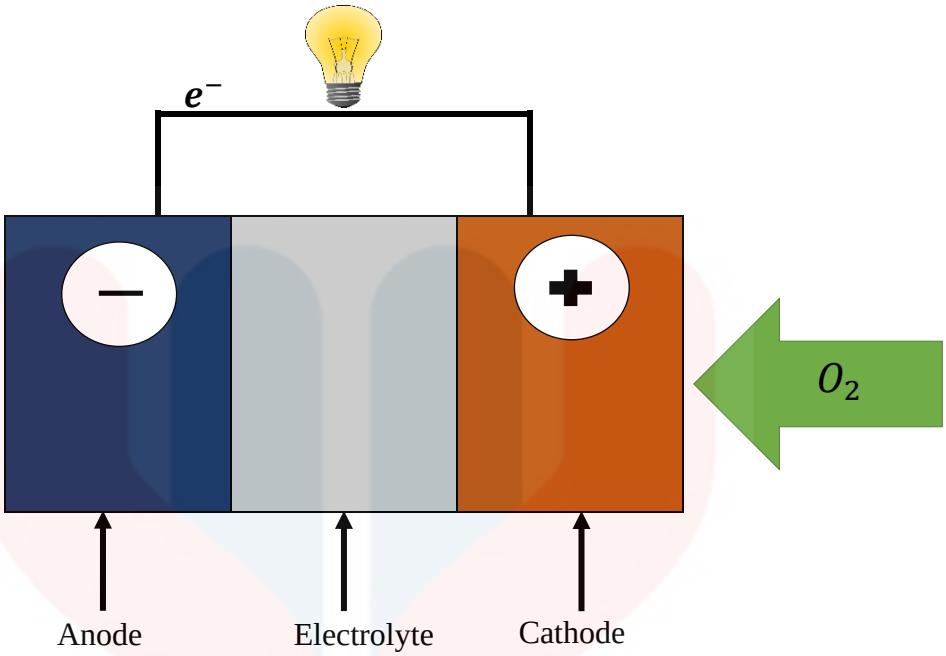


Figure 2.1: Schematic illustration of zinc–air battery

2.1.1 Structure of Zinc-Air Batteries

A flexible battery is a battery having mechanical flexibility and the ability to tolerate repeated folding, stretching, and curling. It has a wide range of applications in the realm of flexible electronics. The notion of flexible batteries is in contrast to solid or liquid batteries that cannot deform, and the use of hydrogel electrolytes allows the creation and popularization of flexible batteries a reality(Mo et al., 2022).

Flexible zinc-air batteries are made up of three components: a metal electrode, a hydrogel electrolyte, and an air electrode. A linear metal electrode is covered with a layer of hydrogel electrolyte and wrapped with an air electrode to produce a cable-style structure in one common battery(Qu et al., 2020).

Another common construction is a sandwich structure composed of an air electrode with the catalytic layer, a hydrogel electrolyte, and a metal electrode placed one on top of the other. There are also foldable constructions of among other things(Lorca et al., 2020).

2.1.2 Working Principle of Batteries

The flexible zinc-air battery's discharge process is identical to that of the classic one, which is realised by the redox reaction between the anode and cathode in the electrolyte. During the discharge process, the metal zinc combines with the basic ions in the hydrogel electrolyte, electrons are released and sent to the air cathode through the external circuit, and oxygen from the air diffuses and adsorbs into the air cathode, where it is reduced to hydroxyl ions(Wang et al., 2021).

These produced hydroxyl ions are transported to the zinc electrode via the hydrogel electrolyte's ion transport channels to form zincate ions. When the concentration of zincate ions in the hydrogel electrolyte becomes supersaturated, the process of breakdown into insoluble zinc oxide (ZnO) begins (Iqbal et al., 2023).

The ion transport in the hydrogel electrolyte and the electron transport in the external circuit complete the discharge process of the flexible zinc-air battery.

Air Cathode:

$$O_2 + 4e^- + 2H_2 = 40H^- \quad (1)$$

Zinc Anode:

$$Zn + 40H^- = Zn\,(OH)_4^{2-} + 2e^- \quad (2)$$

$$Zn\,(OH)_4^{2-} = ZnO + H_2O + 2OH^- \quad (3)$$

Overall reaction:

$$2Zn + O_2 = 2Zn \quad (4)$$

An oxygen reduction reaction occurs during the discharge procedure of alkaline hydrogel type zinc-air batteries. The oxygen in the air from the surroundings diffuses to and adsorbs on the catalyst layer surface of the air cathode(Iqbal et al., 2023).

The oxygen reacts with the electrons produced by the metal zinc to generate hydroxide ions. Ions are released from the catalyst's surface into the hydrogel electrolyte and subsequently transported to the zinc anode via the ion channels. During discharge, this is how the flexible inc-air battery works (P. Zhang et al., 2021).

2.2 Agar

Agarose is a linear polymer made from agar, a seaweed extract. It is often used as a matrix for electrophoresis, a method used to separate and analyse DNA, RNA, and proteins depending on their size and charge, in molecular biology and biochemistry. Agar is a highly purified and neutral version of agar. Dissolving in water and heating dissolved in water and heated. It creates a gel-like material that solidifies upon cooling.

Agarose's gel structure comprises holes of varying diameters, allowing biomolecules to be separated based on their molecular weight. Smaller molecules can move easily through the gel matrix, but bigger molecules have difficulty and move more slowly (Arote et al., 2011).

The agarose concentration may be changed to modify the pore size and separation range. Lower concentrations are appropriate for separating bigger molecules, whilst higher concentrations are appropriate for resolving smaller molecules. Agarose gels are often employed in procedures such as gel filtration chromatography and agarose gel electrophoresis.

Agarose is biocompatible and inert, making it appropriate for a wide range of biological applications. It is simple to use, non-toxic, and has a high resolution for distinguishing biomolecules. Given these qualities, agarose is commonly employed in molecular biology research and genetic analysis (Jiang et al., 2023).

Agar is a hydrophilic polysaccharide generated from red algae that is mostly composed of 1,3-linked -D-galactopyranose and 1,4-linked 3,6-anhydro--L-galactopyranose or agarose (Qiao et al., 2019).

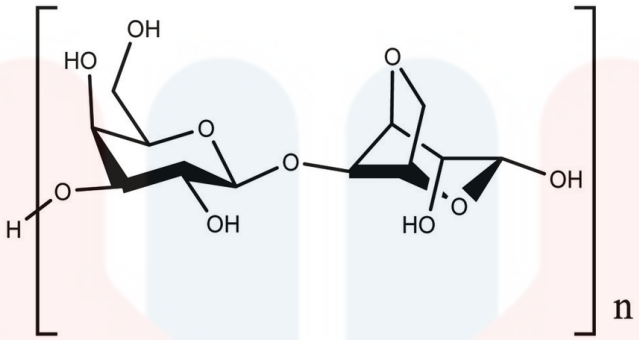


Figure 2.2 Chemical Structure of Agar (Shahidi & Rahman, 2018).

2.3 Agar Hydrogel

Agar hydrogel is a gelatinous substance made from agar, a naturally occurring polysaccharide taken from seaweed. Agar is a gelling agent and thickening that is extensively. When agar is dissolved in water and then chilled, it creates an agar hydrogel, which is a gel (Hull, 2014).

Crosslinked polymeric networks of two or more polymers form co-hydrogels. For their biocompatibility and low cost, gelatin-agar co-hydrogels have gained a lot of interest. Agar is a polysaccharide biopolymer, whereas gelatin is a protein biopolymer because of the thermodynamic instability of gelatin-polysaccharide mixtures at large concentrations of gelatin and polysaccharides, gelatin-polysaccharide-based phase-separated hydrogels have been described (Zhao et al., 2023).

The goal of this research was to create gelatin-agar-based phase-separated hydrogels that could be comprehensively characterized utilizing FTIR and electrical inspections. There are no studies comparing the characteristics of phase-separated hydrogels, which were discovered.

The advantages of using agar hydrogel electrolytes over other forms of electrolytes is strong conductivity. The strong ion conductivity of agar hydrogels allows for effective charge transfer and quicker electrode responses.

The agar is a biocompatible natural substance that is commonly utilised in biomedical applications. Although agar hydrogels are non-toxic, non-irritating, and biodegradable, they are well suited for use in implanted devices and medicinal applications.

Agar hydrogels may be made from agar powder utilizing basic techniques including gelation and cross-linking. This makes them inexpensive and simple to create in big quantities(Wakhet et al., 2015).

Agar hydrogel is used as an electrolyte in battery systems. Because of its unique features, agar hydrogel, a biocompatible and ecologically harmless substance, has received interest as a potential replacement to traditional electrolytes. (Zheng et al., 2023)

2.4 Hydrogel Electrolyte

Hydrogel electrolytes are a kind of electrolyte composed of hydrogel matrices. Hydrogels are three-dimensional networks of cross-linked polymers capable of absorbing and retaining huge volumes of water or other aqueous solutions. Hydrogel electrolytes combine hydrogel's unique features with the capacity to conduct ions, making them appropriate for a wide range of electrochemical applications such as batteries, fuel cells, supercapacitors, and sensors(Lu et al., 2020).

The electrolyte plays a role in electrochemical energy storage and conversion devices because it allows ionic transport between the two electrodes. Polymer hydrogel electrolytes are made up of polymer networks, a solvent, and conductive salt that has been dissolved in the solvent.

Hydrogel electrolytes solve the disadvantages of a small contact surface between the electrode material and the solid-state electrolyte while providing the same benefits of high ionic conductivity and ion mobility as liquid electrolytes.

However, because current hydrogel electrolytes are derived from fossil fuels and are non-biodegradable, and their wastes pollute the environment, there is an urgent need to create sustainable biomass-based materials and matching energy storage/conversion applications.

Given their low cost, environmental friendliness, and degradability, gel-state electrolytes generated from biomass-derived biopolymer materials. Biopolymers like as cellulose, chitosan, chitin, alginate, and lignin have been investigated for use in the production of functional hydrogel electrolytes.

The presence of hydrophilic (-OH, -COOH, -NH2, and -CONH2) groups in biopolymer structures leads in high wettability to polar solvents and the ability to preferentially interact with salt anions, improving salinity solubility and cation transport characteristics.

Although biopolymer-based hydrogel electrolytes are non-toxic and ecologically harmless, they do have drawbacks such as weak mechanical strength and probable loss of electrochemical effectiveness under mechanical pressures.

To meet the high mechanical qualities of electrolytes, biopolymer-based hydrogels must be changed by grafting, inorganic filler doping, and mixing. According to ion transport processes, hydrogel electrolytes have better ionic conductivity than solid electrolytes(Xu et al., 2022).

Chemically crosslinked hydrogel electrolytes are combined with conducting copolymer electrodes to create all-in-one flexible supercapacitors. The hydrogel electrolyte is made by chemically crosslinking polyvinyl alcohol (PVA) and polyethylene glycol (PEG) with glutaraldehyde (GA).

In which the hydroxyl groups (-OH) of PVA and PEG react with the aldehyde groups (-CHO) of GA to generate acetal or hemiacetal in sulphuric acid solution. The produced hydrogel electrolyte outperforms pure PVA hydrogel in terms of mechanical characteristics and ionic conductivity (L. Guo et al., 2020).

A hydrogel-based lithium-ion battery is another example of a battery that uses a hydrogel electrolyte. Traditional lithium-ion batteries employ liquid or solid electrolytes, which can cause leakage, fire hazards, and mechanical limits. In order to fix these issues, considerable research has been conducted to substitute traditional electrolytes with hydrogels(Cao et al., 2020).

Lithium-ion battery electrolyte hydrogels can be built of a variety of materials, including polymers that incorporate water in a three-dimensional network. This hydrogel can outperform traditional electrolytes in terms of mechanical stability, ion transport, and safety (Y. Guo et al., 2019).

2.5 Characterization of agar hydrogel electrolyte

The electrolyte potential of agar hydrogels can be determined using standard characterization methods. This chapter provides an overview of the characterization of hydrogel electrolytes for conductivity in structural as discovered in previous studies.

2.5.1 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier-transform infrared spectroscopy (FTIR) is used to identify the presence of functional group and chemical bonds in different wavelength. Figure 2.3 showed the FTIR patterns of the Agar powder with 400-4000 cm^{-1} of wavenumber range used.

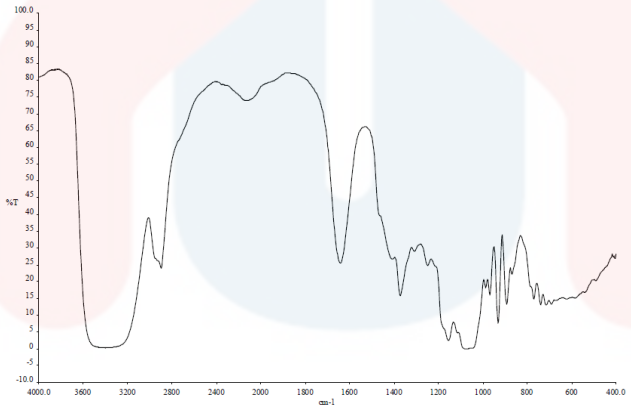


Figure 2.3 FTIR pattern of agar (El-Hefian et al., 2012).

The FTIR pattern confirmed that Agar has abundant of functional groups. At 3300 and 2930 cm-1 wavenumber, the stretching vibrations of O-H and C-H bonds caused broad and strong peaks. At 1030 cm-1 the peak is related to the C-C stretching vibrations within the sugar rings that make up agar's backbone.

The FTIR spectrum displayed the main component in Agar. At 1640 and 1100 cm−1, the peak is from the symmetric or asymmetric vibrations of C-O bonds. At low frequency range (around 600-850 cm-1), weak absorption peaks appeared caused by the asymmetric vibrations of C-O bonds.

2.5.2 Optical Microscope

Optical microscope is used to identify the morphology surface of agar hydrogel. Figure 2.4 showed the morphology surface of the Agar hydrogel.

Figure 2.4 Optical microscope in agar hydrogel (Giordano et al., 2022)

Micro-photographing agar hydrogel on a microscope slide at 240× magnification using a Dino-lite digital microscope under normal lighting showed agar beads with areas ranging from 0.013 to 0.308 mm.

2.5.3 X-Ray diffraction analysis (XRD)

X-ray diffraction is used to identify the amorphous phases of agar hydrogel. Figure 2.5 showed the xrd graph of the Agar hydrogel.

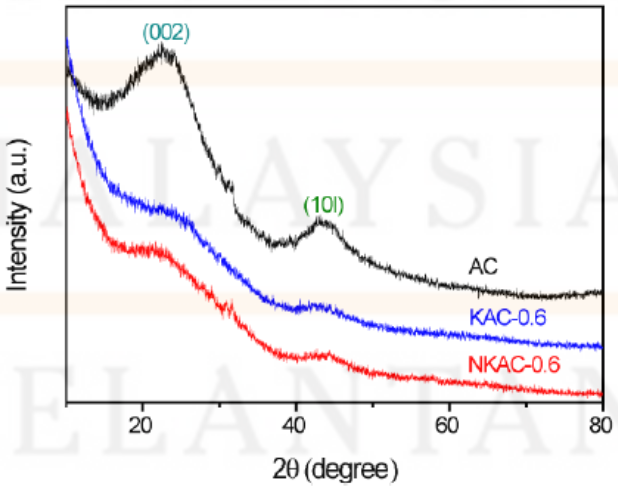


Figure 2.5: Xrd graph of agar and potassium hydroxide(F. Zhang et al., n.d.).

AC has poor crystallinity, as seen by its wide diffraction peaks (0 0 2) and (1 0 l). The peaks of KAC and NKAC are less visible than those of AC due to their amorphous form after KOH and NH3 activation.

2.6 Characterization of Zinc-air batteries

In order to confirm the compatibility of agar hydrogel electrolyte, It is the electrolyte in zinc-air battery, the characterization of battery should be taken. This subchapter discussed the overview of the electrochemical properties of hydrogel electrolyte by using open circuit potential (OCP) that have been applied in zinc-air batteries.

Figure 2.6 presents the OCP profile of gel electrolyte with 6 M KOH for 24 h.The zinc-air battery system achieved plateau region at 1.48 ± 0.03 V through 24 h of storage. Commonly, the practical cells have range of OCP between 1.42 and 1.52 V.

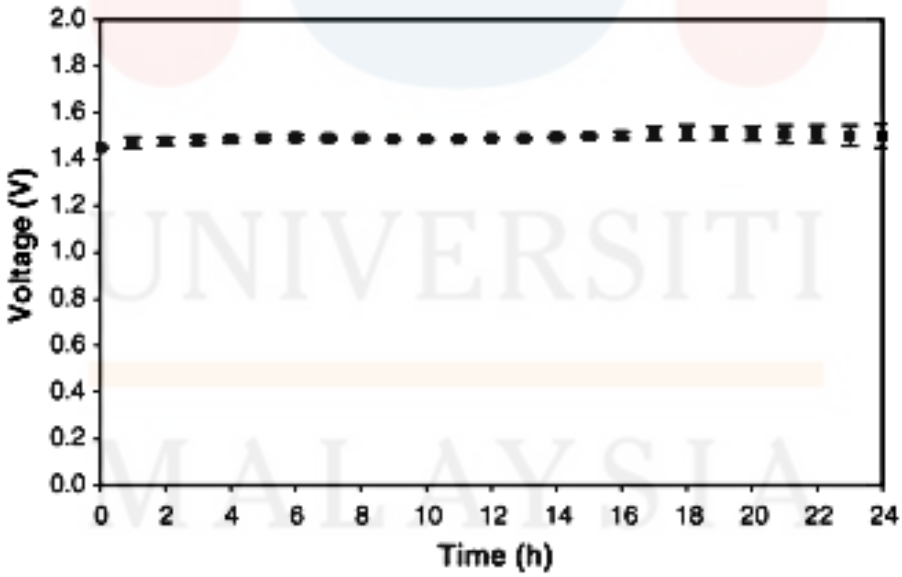


Figure 2.6: The measurement open circuit potential of Zn/gelled 6M of KOH (Mohamad, 2006)

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

Materials that were going to use in this research to produce the agar hydrogel electrolyte were Agar powder purchased from Shaanxi, China, distilled water and alkali metal hydroxides (potassium hydroxide (KOH)) from quality reagent chemical.

3.2 Preparation of Agar Hydrogel

First, 100 mL of distilled water was mixed with 2 g of Agar powder. To ensure that the powder was completely dissolved, the mixture was heated with a hot plate at roughly 70 - 80 °C while being stirred with a magnetic stirrer.

The mixture was constantly heated until hydrogel formed and then cooled to 25 °C room temperature. As indicated in Table 3.1, the same process was used to make different hydrogels using 4, 6, 8, and 10 g of Agar powder.

After determining the proper composition of Agar hydrogel, the synthesis of Agar hydrogel for the Agar hydrogel electrolyte section was repeated with different KOH concentrations.

For the creation of KOH solutions, various concentrations of KOH (Table 3.1) were dissolved in a constant amount of distilled water. The hydrogel electrolyte solution was thoroughly mixed with the KOH solution.

Table 3.1: Different molarity and gram for KOH that will be mixed in Agar and distilled water mixture to create agar hydrogel.

SAMPLE	Agar Powder (g)	KOH (M)	Distilled Water (ml)	KOH (g)
AGKOH1	10	0	100	0
AGKOH2	10	2	100	11.22
AGKOH3	10	4	100	22.44
AGKOH4	10	6	100	33.67
AGKOH5	10	8	100	44.89

3.3 Characterization Hydrogel Electrolyte

This discussed the characterization of Agar properties as a hydrogel electrolyte. The evaluations were based on open circuit potential, optical microscope (OM), Fourier transform infrared (FTIR) and X-Ray diffraction analysis (XRD).

3.3.1 Fourier transform infrared (FTIR)

FTIR-ATR (Thermo Scientific Nicolet iZ10, Nexus Analytics) was executed to investigate the absorbance and functional group of raw material, agar hydrogel and agar hydrogel electrolyte. Attenuated Total Reflectance (ATR) technique was used to generate the peaks. The samples were analysed with 4000 – 500 cm-1.

.3.2.2 Optical microscope (OM)

Optical Microscope (OM) was used for observing morphological surface of Agar hydrogel and hydrogel electrolyte.

3.3.3 X-Ray diffraction analysis (XRD)

A bench-top diffractometer branded Bruker model D2 Phaser machine to perform X-ray diffraction (XRD) on the hydrogel electrolyte at an angle of 10-90 degrees^0 can provide important insight into the phase composition of the material and crystal structure. The sample to observing the amorphous for agar and agar hydrogel electrolyte.

3.4 Preparation of Zinc-Air Batteries

The preparation of Zinc-Air Battery, collect all the needed components, including the zinc anode, air cathode, separator, electrolyte, and battery housing or container. Make the zinc anode: Remove any impurities or oxides from the zinc anode's surface. This may be accomplished by cleaning it down with a cloth or brush. Figure 3.4 shows the image of Zinc-Air Battery.

Prepare the air cathode is usually constructed of porous carbon that has been coated with a catalyst such as platinum or carbon-supported catalysts. Ascertain that the air cathode is clean and devoid of pollutants.

Assemble the battery by inserting a separator between the zinc anode and the air cathode to prevent direct contact. The separator allows ions to pass while preventing solid zinc particles from migrating. Check that the electrodes and separator are correctly aligned.

The electrolyte is needed for ion conduction between the anode and cathode. The electrolyte of a zinc-air battery can be a hydrogel. Pour or inject the electrolyte into the battery casing, ensuring that it comes into touch with both electrodes without leaking.

Make sure the battery housing or container is strongly sealed to prevent electrolytes or air from escape. This helps to keep the battery's integrity and protects it from drying out. The battery is ready to use once it has been constructed and sealed. Connect the battery terminals to a suitable device or circuit and monitor its opera.

3.5 Characterization zinc-air batteries

The measurement of open-circuit voltage (OCV) helps determine a battery's charge level of a battery when no load is present. It estimates how much capacity is still left in the battery.

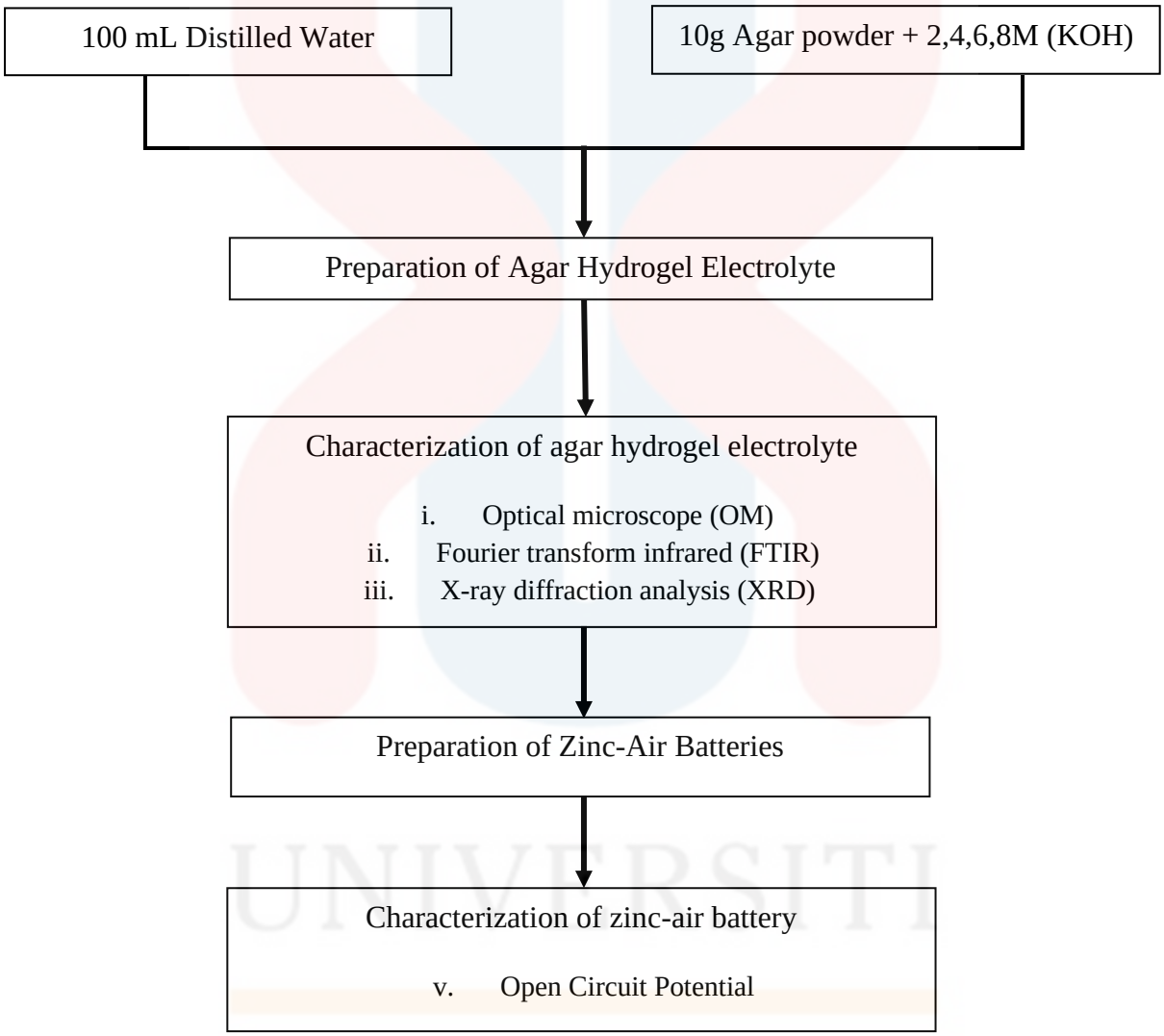
When the battery is idle or resting, the OCV is the voltage across the terminals. This voltage is controlled by the battery's composition and charge state. The OCV steadily drops as a battery drains, representing the decrease in available energy. In contrast, when a battery charges, the OCV rises.

The OCV is the voltage across the battery terminals while the battery is idle or resting. The chemistry and charge state of the battery has an impact on this voltage. As a battery drains, the OCV steadily falls, representing the decrease in available energy. In contrast, when a battery charges, its OCV increases.

3.6 Research flow chart

This research flow chart showed process of characterisation of Agar hydrogel

Figure 3.6.1 Flow chart for characterisation of Agar hydrogel electrolyte



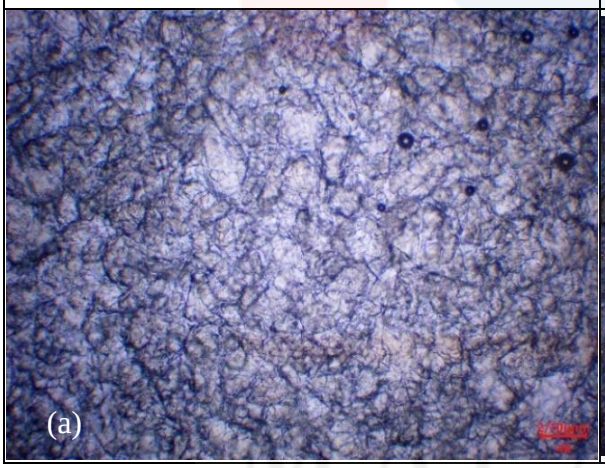
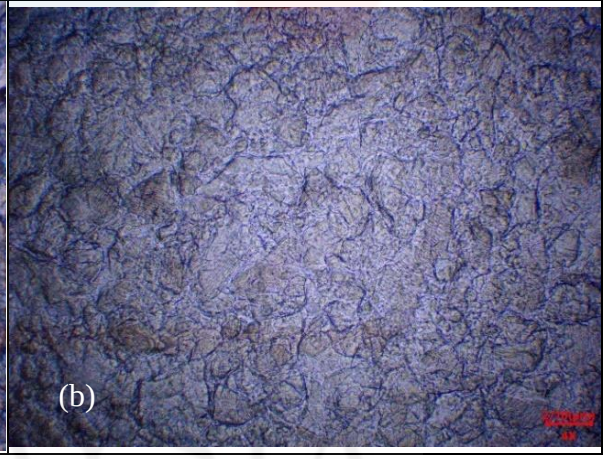
CHAPTER 4

RESULTS AND DISCUSS

4.1 Optical Microscope

The properties under an optical microscope were observed to see the surface morphology of the Agar hydrogel and Agar hydrogel electrolyte. An optical microscope (OM) has four magnifications. For this study, two magnifiers were used, namely 4× and 10× magnification.

4× Magnification

Agar hydrogel	Agar hydrogel electrolyte
 (a)	 (b)

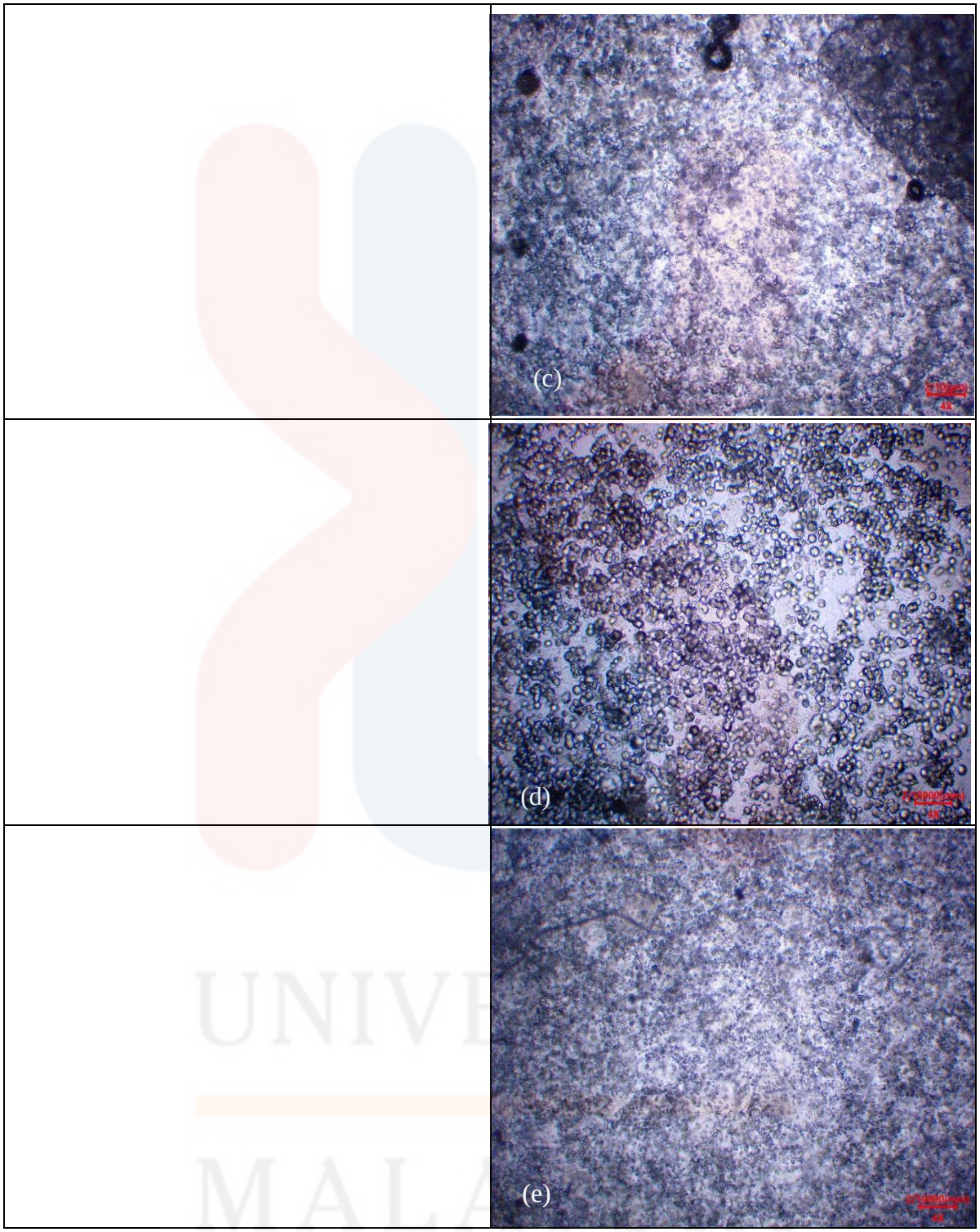


Figure 4.1 Morphology of agar hydrogel and hydrogel electrolyte on 4x magnification.

Figure 4.1 shows 4x magnifications in agar hydrogel and electrolyte hydrogel. The (a) is labeled for 10 g of agar powder mixed with 100 mL of distilled water. In this observation at (a), the surface is less foamy but rough. It is possible because it contains the water for 10g of agar only.

This composition is known as the optimal composition for making hydrogels because it is not too thick which makes it gel-like. It's not too runny either. Under the microscope, the surface of ag01 is the most obvious because of the quantity of agar and water.

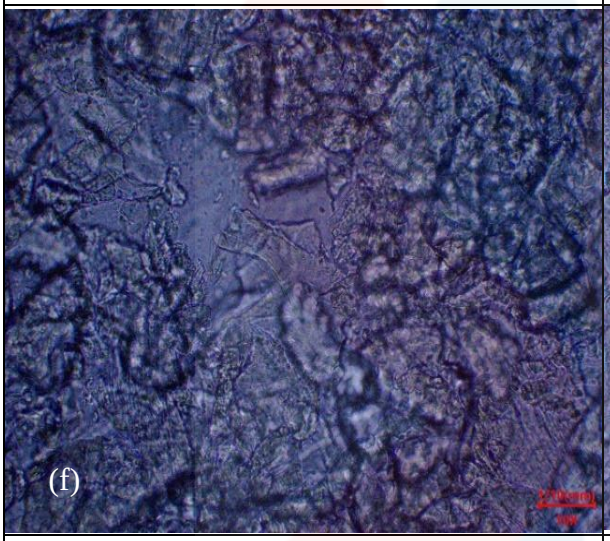
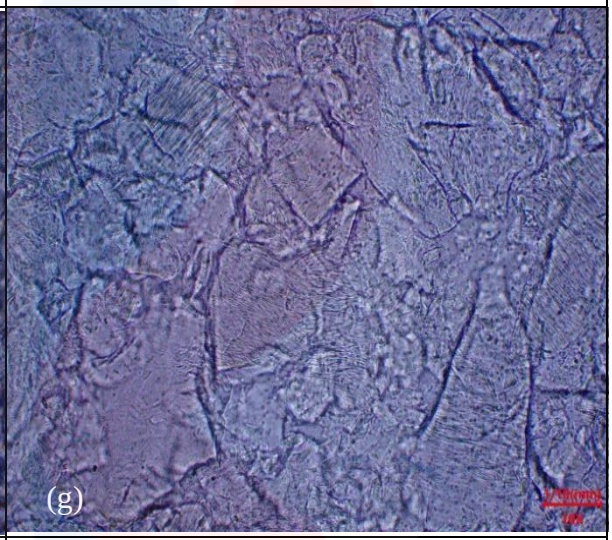
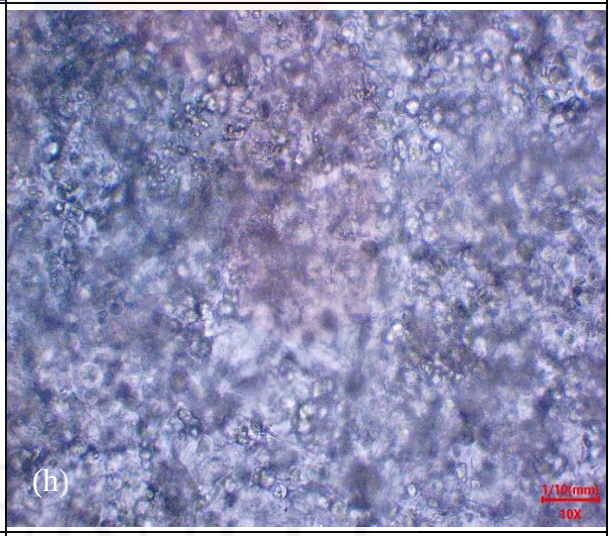
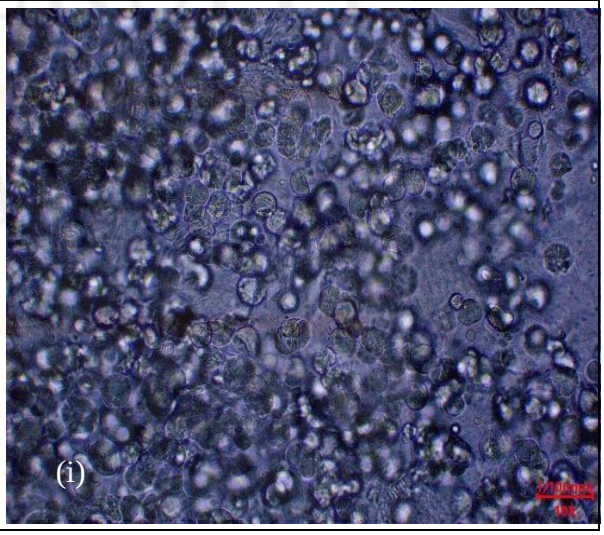
For the Agar hydrogel electrolyte on the (b) label which is at 4× magnification, 2 M KOH concentration is used. It can be observed that there are not many bubbles on the hydrogel compared to 4, 6, and 8 M which will be described further.

In 4× magnification, (c) represents 4M Koh concentration. Below the optical microscope shows that there are a few large bubbles and many small bubbles compared to 6 and 8 M which will be further elaborated on later.

This happens probably because the molarity of potassium hydroxide 2M of agkoh2 is the lowest molarity of others. For (d) representing 6 M Koh concentration, it shows more small bubbles than agkoh3 but not as many small bubbles at (e) representing 8 M Koh concentration.

It is because higher molarity in koh at the agar hydrogel electrolyte leads to smaller bubble production, indicating improved electrolysis reaction and increased ion. This is due to increased conductivity and release of gasses, such as oxygen and hydrogen at the electrodes, promoting faster electrolysis.

10× Magnifications

Agar hydrogel	Agar hydrogel electrolyte
	
	
	

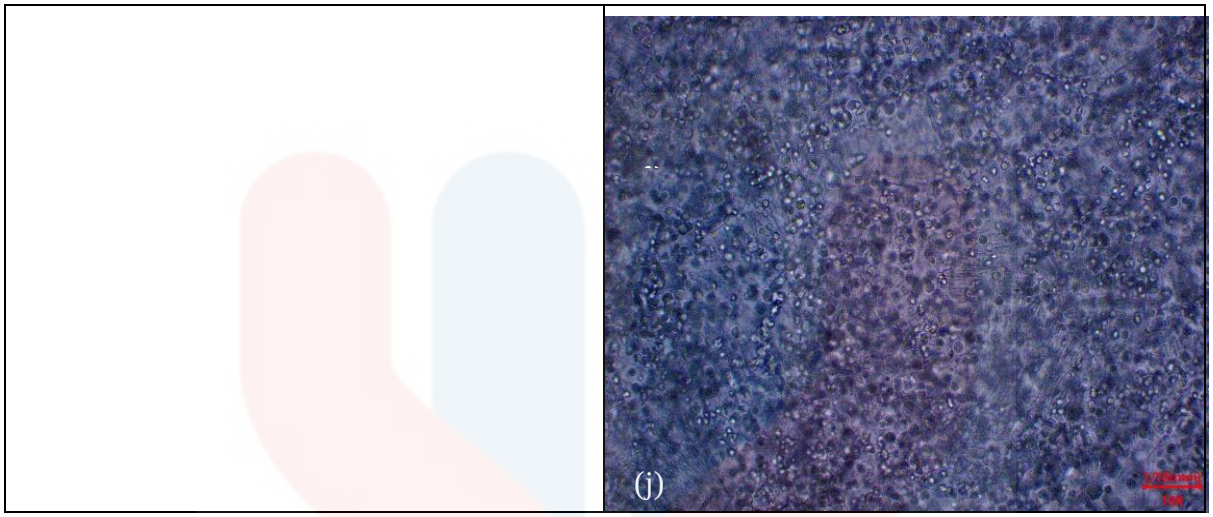


Figure 4.2: Morphology of agar hydrogel and hydrogel electrolyte on 10x magnification

From Figure 4.2, 10x magnification is shown. A f is labeled for 10 g of agar powder mixed with 100 mL of distilled water. In this observation at F, it can be seen that the surface is rough at the same time. It is possible because it contains water for 10 g agar to dissolve. For g, it begins to form a structure that is denser than F. It still shows a rougher surface due to the water content and the presence of Koh in the agar.

For g, h, i, and j, it starts to become more gel-like. Making it have a more structured surface in contrast to f. For h, it is 10g to be mixed with 2 M Koh dissolved in distilled water. This composition is known as the optimal composition for making hydrogels because it is not too thick which makes it gel-like, but it is also not too liquid. It has a stronger structure than the others.

In 10× magnification, h (2 M) has some bubbles and an uneven surface. They reduce electrolyte conductivity and increase ionic resistance. There is a correlation between bubbles, viscosity, and ionic conductivity. Higher conductivity occurs when it has a lower viscosity. Lower viscosity can be achieved if there is low foam.

From OM, the concentration at 6 M and 8 M is more than 4M because it's likely that the higher concentrations, like 6 M and 8 M, produce more foam or have a more noticeable effect than the 4 M solution because of the larger ion concentration.

Solutions with higher molarity have more solute particles per volume, which improves conductivity and speeds up electrolysis. During electrolysis, this leads to more vigorous gas generation (bubbles) than at lower concentrations like 4 M.

4.2 FTIR Analysis Properties

Using Fourier-transform infrared spectroscopy (FTIR), the absorbance, functional group, and chemical chain present were examined. In absorbance mode, FTIR spectra have been collected between 4000 and 500 cm 1.

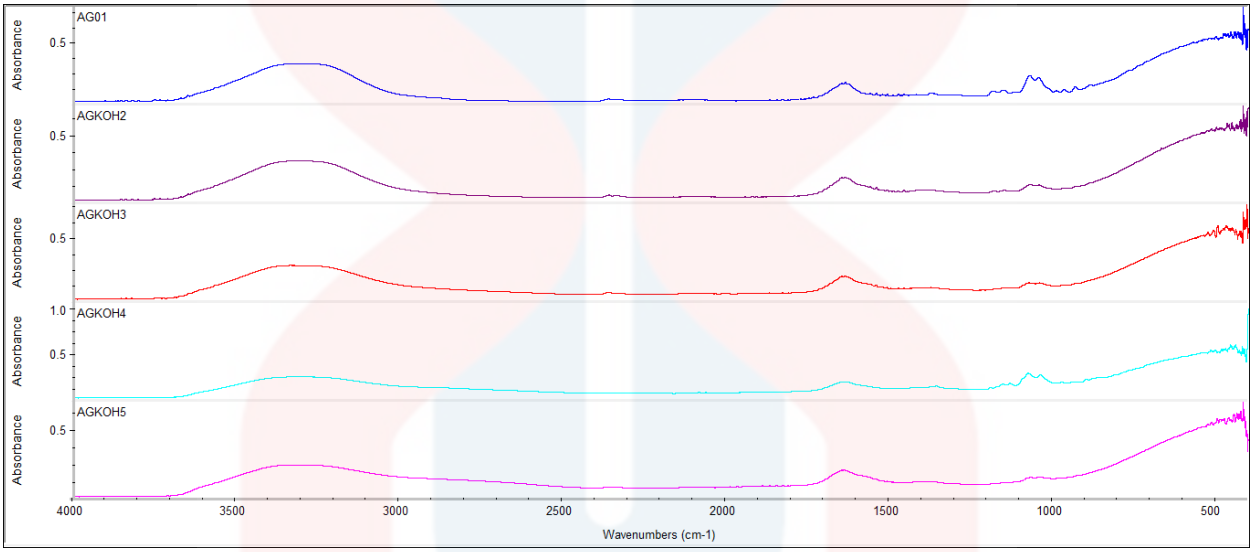
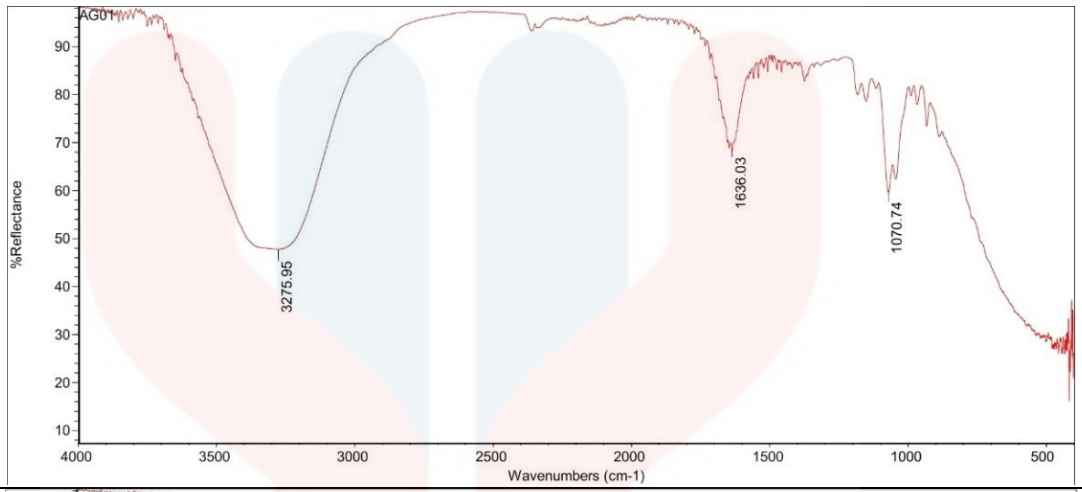
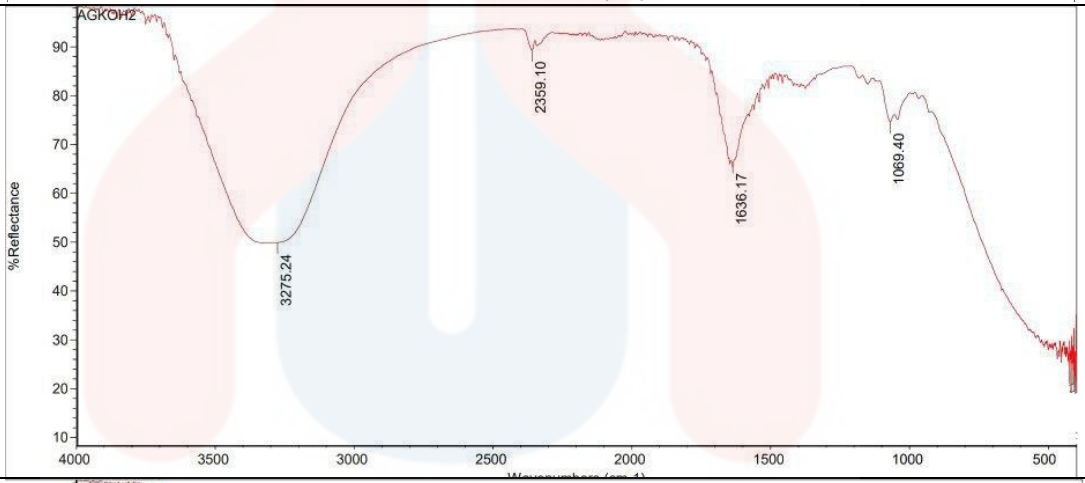
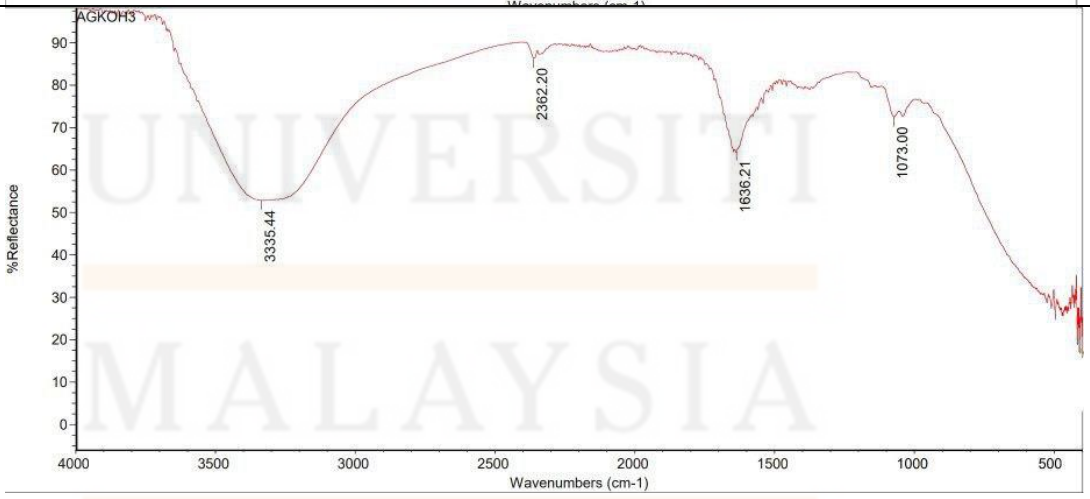


Figure 4.3: FTIR absorbance of Agar hydrogel and electrolyte, (a) Ag1, (b) Agkoh2, (c) Agkoh3, (d) Agkoh4, (e) Agkoh5

Figure 4.3 shows FTIR spectroscopy of agar hydrogel and agar hydrogel electrolyte under absorption mode. The figure shows that the peaks are most identical to each other although the difference is the different amount of potassium hydroxide (KOH) used in sample. Below, the peaks are shown one by one for easier interpretation.

Bil	Peaks
(a)	
(b)	
(c)	

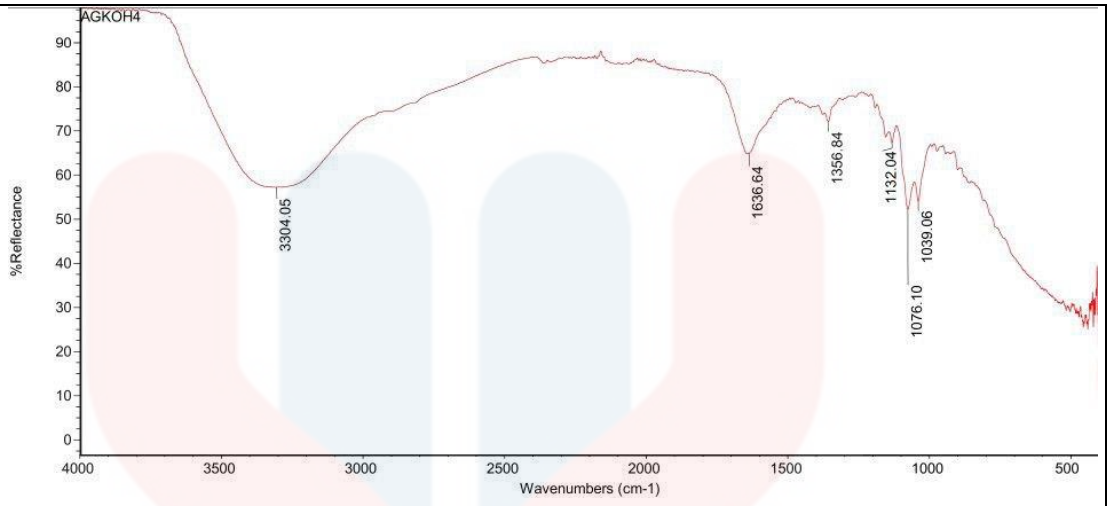
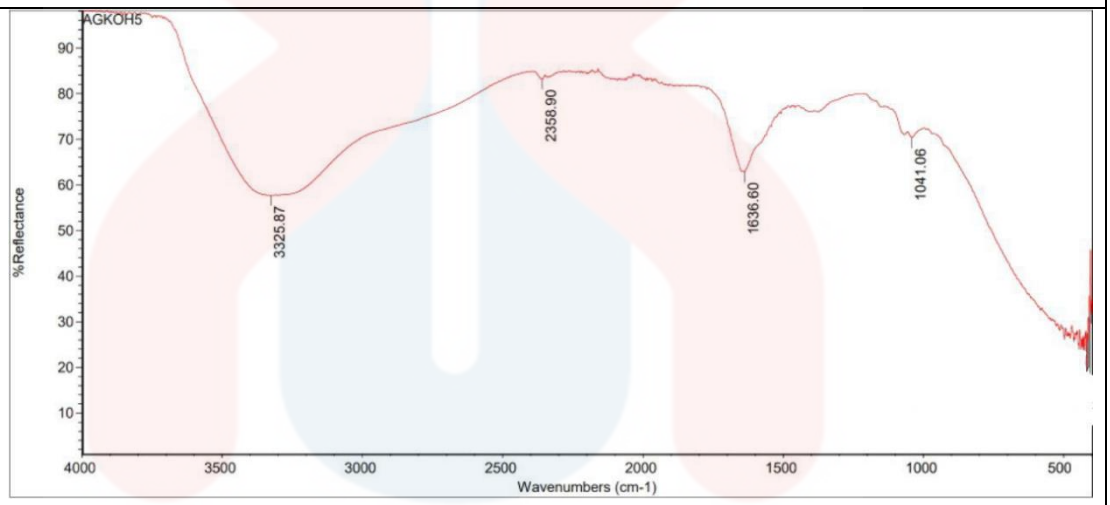
(d)	
(e)	

Figure 4.4: Peaks for Agar hydrogels and electrolyte, (a) Ag1, (b) Agkoh2, (c) Agkoh3, (d) Agkoh4, (e) Agkoh5

From figure 4.4, it is shown broad absorption, (a) broad absorption for agar hydrogel which is 3275.95 cm-1. Alcohol shows broad absorption in the range between 3600 - 3200 cm-1. This occurs due to the presence of O-H bonds.

This is common for samples containing water or wet. Next, the strong absorption is shown in the graph. The range is 1676.73 - 1603.52 cm-1. From Table 4.3, the strong absorption shown from (a) is 1636.03 cm-1.

This indicates that the second peak is from the class of amide compounds. This is due to the presence of C=O stretching. Amide compounds are in the range of 1690 – 1630 cm-1.

The last absorption shows a strong absorption. The range is 1166.38 - 1070.74 cm-1 of figure 4.4 shows the peak with 1070.74 cm-1. The third peak results from the presence of the S=O stretch which makes it belong to the class of sulfoxide compounds. The sulfoxide of the compound is between 1070 – 1030 cm–1.

Figure 4.4, it is shows that moderate absorption. Table 4.3 is agar hydrogel electrolyte at agkoh 2 (b), agkoh 3(c), agkoh 4 (d) and agkoh 5 (e), The (a) absorption which is 3275.24 cm-1, (b) 3335.44, (c) 3304.05 and (d) 3325.87. This shows that the first peak is primary aliphatic alcohol has a range between 3400 – 3100 cm-1.

The peak occurs due to the stretching of the O-H bond. The next peak suggests that strong absorption occurs. (a) shows its absorption peak is 1636.17 cm-1, (b) shows 1636.21 cm-1, (c) 1636.64 cm-1 and finally, (d) 1636.60 cm-1. Amide can be found between the range of 1637cm-1. This causes the second peak to enter the class of amide compounds. This happens because of C=O stretching.

The third peak has a peak at (a) 1069.40 cm-1, then (b) 1073.00 cm-1, (c) which is 1076.10 cm-1 and (d) 1041.06 cm-1 respectively. The third peak reveals that it has strong absorption. The third peak is at primary alcohol compounds. It has strong absorption because it has a C-O stretch.

Therefore, from FTIR spectroscopy it was observed that both Agar hydrogel and agar hydrogel electrolytes have similar functional groups which are hydroxyl compounds (-OH), amide compounds (-NH), sulfoxides, and primary alcohol compounds.

4.3 X-ray diffraction (XRD)

The graph displays the amounts of X-rays scattered by two different graphs. The various atomic or molecular spacings in the sample are represented by the peaks on the graph.

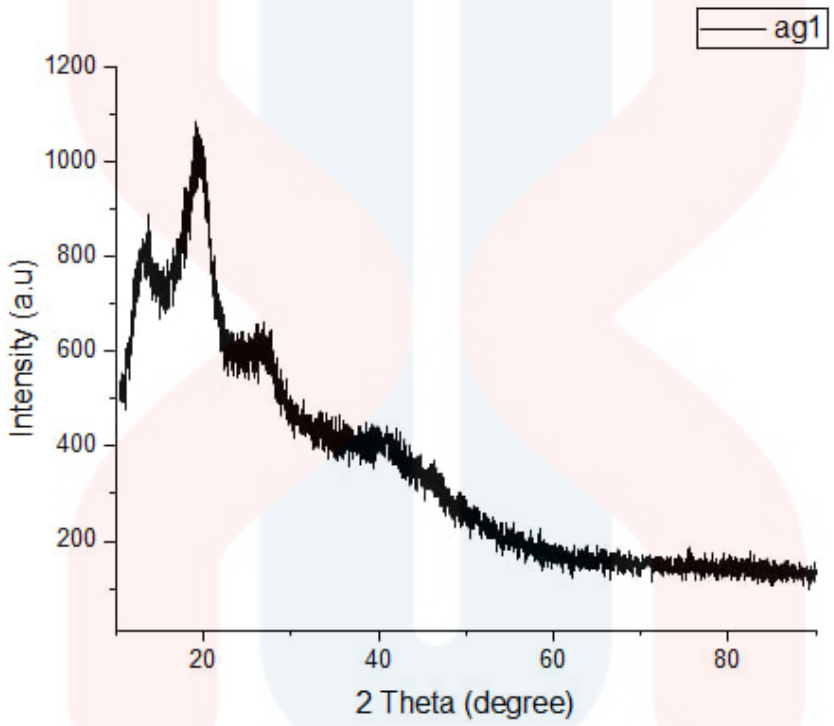


Figure 4.5 X-ray diffraction of Agar hydrogel

Agar performs a significant role in hydrogel electrolytes utilised in XRD. The molecules in amorphous materials are not arranged in a certain, crystalline structure because they lack long-range order. This characteristic makes it easier for X-rays to penetrate materials and produce diffraction patterns, which is crucial for XRD investigation.

The XRD pattern of the agar shows broad peaks, which indicate that the material is amorphous. As opposed to the accurate distinct peaks typical of crystalline materials, these broad peaks indicate that the X-rays appear to be randomly organised molecules, resulting in a diffuse scattered pattern.

The X-ray diffraction pattern of agar reveals two broad peaks, one at generally 20 degrees 2θ and another at around 40 degrees 2θ. These peaks show that the agar is amorphous, which means the molecules are not grouped in a predictable pattern. The broadness of the peaks also suggests that the agar molecules are actually small.

The intensity of the peaks is also important. The peak at 20 degrees 2θ is more obvious than the peak at 40 degrees 2θ. At 20 degrees 2θ, there are more patterns of atoms diffracting X-rays compared to 40 degrees 2θ. This might be because agar molecules get closer together in specific orientations.

The X-ray diffraction pattern of agar indicates that it is an amorphous substance composed of relatively small molecules. The molecules are not packed in a regular pattern, yet there is a chosen orientation.

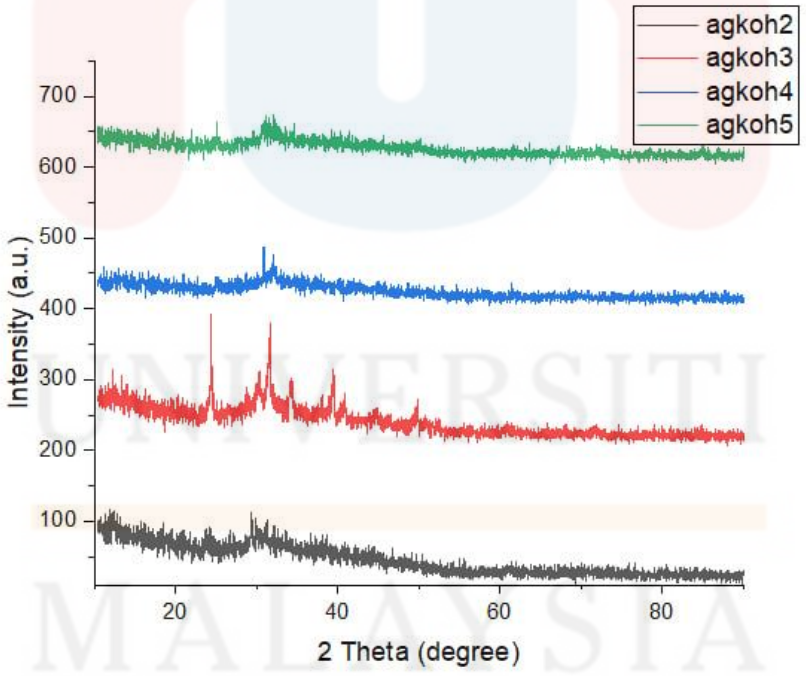


Figure 4.6 X-ray diffraction of Agar hydrogel with different concentration.

The graph displays several peaks, each representing all of the sample's amorphous phases. The strongest peak happens at around 20 degrees, with lower peaks at 40, 60, and 80 degrees. The positions and intensities of the peaks can be utilised to determine and measure the various amorphous phases in the sample.

For example, the sharp peak at 20 degrees is characteristic of KOH, showing that KOH is the primary amorphous phase in the sample. The lower peaks at 40, 60, and 80 degrees might be attributed to additional small amorphous phases, such as agar or contaminants.

The XRD graph may also be used to calculate the size of amorphous phases in a sample. The width of the peaks is inversely related to their size, thus larger peaks relate to smaller amorphous. The narrow peaks in the figure indicate that the amorphous in the sample are quite big.

4.4 Open circuit potential

The open circuit potential (OCP) characteristics of the zinc-air battery is with no current applied as a function of time. Moreover, the self-discharge of the battery was characterized by measuring the voltage stability of zinc air batteries where the battery stored at an open circuit condition for 24 h.

Without any load applied to the zinc air batteries, the voltage stability recorded every 2 hours for 24 h at room temperature of 25 ℃. Zinc air batteries employed with different KOH concentration of agar hydrogel electrolyte concentration were evaluated.

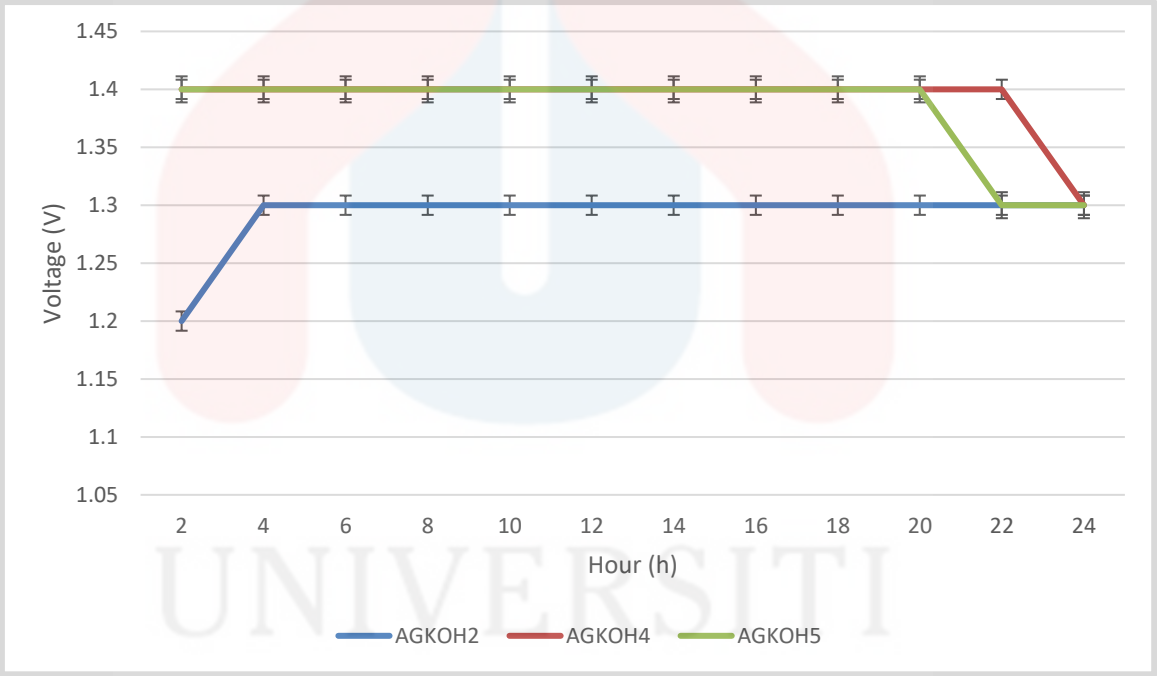


Figure 4.7 Open circuit potential of Agar hydrogel electrolyte.

For the Agar hydrogel electrolyte with KOH2, the voltage increased slightly from 1.20 V to 1.30 V for the first 2 hours. Then, the voltage was continuously reduced to 1.30 V for up to 12 hours and remained constant during that time. The cell dropped from 1.30 v to 0.9 V in the last 8 hours.

For AGKOH4, the voltage plateau area is about 1.40V. In the initial stage, a voltage drop occurs where it constantly decreases from 1.40 V to 1.30 V up to 14 hours. There was a voltage plateau at 6 hours which was 1.40 V then decreased in the last 2 hours from 1.40V to 1.30 V.

For AGKOH5, the voltage increased continuously from 1.30 V to 1.40 V until 18 hours and decreased in the last 2 hours after that. At there aren't enough charge carriers, it's also not recommended for using KOH concentrations that are too high.

Thus, in order to achieve optimal battery power density, solution optimization is needed. Evaluating the electrochemical stability of electrolytes is crucial in order to have a better understanding for battery applications(Laheäär et al., 2009).

Lower KOH concentration was utilized, which affected the kinetic reaction rate and resulted in reactant depletion (Ken & Nandi, 2018).

Table 4.1 presents the OCP profile of Agar hydrogel electrolyte. Therefore, the OCP profile and ionic conductivity of the hydrogel electrolyte is the result of an interplay between the concentration of charge carriers and ionic mobility, two properties determined by the polymer host to KOH concentration ratio.

The high ionic conductivity and OCP profile of the electrolytes contained KOH probably reflects the higher charge carrier concentration.

Table 4.1: OCP profile of Agar hydrogel electrolyte with different KOH concentration

Sample	Open circuit potential (V)
AGKOH 2	12.32 V
AGKOH 4	13.77 V
AGKOH 5	13.37 V

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In conclusion, the investigation into the morphology and functional group of agar hydrogel containing varying KOH concentrations provided valuable insights into its potential application as an electrolyte in zinc-air batteries. Through testing with optical microscopy, FTIR Fourier transform infrared, X-Ray diffraction analysis (XRD) analysis and evaluation of open circuit potential, the characteristics and performance of Agar-KOH hydrogel were elucidated. These findings lay a foundation for further research and development aimed at optimizing agar hydrogel electrolytes for enhanced efficiency and stability in zinc-air battery applications. This could summarize as testing followed:

(i) The open circuit potential

Open circuit potential increased in response to an increase in KOH concentration until 6 M KOH was gained. Following this, a decreasing pattern was observed at the highest KOH concentration of 8 M. Optical Microscope

(ii) Optical Microscope

An optical microscope was a useful technique to analyze the Agar hydrogel electrolyte's surface morphology. Our knowledge of the material's qualities has been improved by the important insights into its structural characteristics that the detailed images obtained provided.

(iii) Fourier transform infrared

Fourier Transform Infrared Spectroscopy, has shown to be a very useful technique for analyzing agar hydrogel electrolytes. FTIR has made it possible to gain a thorough understanding of the structure and function of agar-based electrolytes by providing significant details about their molecular structures and interactions. The observed spectra have provided helpful details about the hydrogel's chemical bonds, functional groups, and general stability.

(iv) X-Ray diffraction analysis (XRD)

Amorphous structure of agar hydrogel solution and agar hydrogel with potassium hydroxide at concentrations of 2M, 4M, 6M and 8M. All were shown to exhibit different diffraction patterns by X-ray diffraction (XRD) examination.The study contributes to a better understanding of these materials' features and their uses through providing knowledge about their structural properties.

The optimal composition can be observed with a ratio of Agar powder to distilled water of 1:10. By dissolving 10 g of Agar powder in 100 mL of distilled water is the best composition that forms agar hydrogel. After that, the optimal hydrogel was dissolved with different concentrations of potassium hydroxide to prepare agar electrolyte hydrogel. Therefore, AGKOH4 is the best composition to use as hydrogel electrolyte in zinc-air batteries throughout this study.

5.2 Recommendations

Some improvements should be made to be better in future research. The hydrogel Agar solution must be thoroughly stirred to ensure homogeneity of the solution to avoid excessive bubble formation. Next, it is better to use other materials than Agar because Agar does not have enough studies to do any backup research.

It is best to use Carrageenan Gum. Moreover, because Agar is very difficult to control its temperature. It should not be too high in temperature but also not too low. Agar also needs to be constantly monitored because it has the possibility of burning. Agar also has a tendency to gel easily even if it is still not at room temperature.

Agar hydrogel electrolytes show great potential as environmentally friendly and high-performance materials for energy storage applications. Future research using techniques like as open circuit potential measurements, FTIR spectroscopy, optical microscopy, and XRD analysis might help them reach their full potential by providing important information about their stability, composition, structure, and crystallinity. This insight will serve as a way for targeted improvements and improved performance in future product.

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APPENDIX A

CALCULATION

A.1 Calculations of molarity

For calculations of molarity, the equation presented as:

$$\text{Molarity} = \frac{mole}{volume\ (\ distilled\ water\ )}$$

$$2 = \frac{mole}{0.1}$$

$$= 2 \times 0.1$$

$$\text{Mole} = 0.2$$

A.2 Calculation of potassium hydroxide (KOH)

For calculations of molarity potassium hydroxide (KOH), the equation presented as:

Formula:

$$\text{Mole} = \frac{\text{Mass}}{\text{KOH (M)}}$$

Table A.2: Potassium hydroxide of agar hydrogel electrolyte.

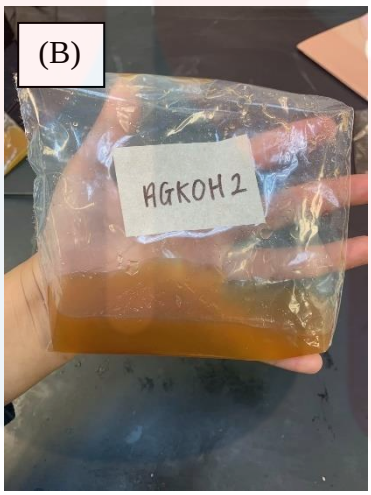
KOH (M)	Weight needed for 100 mL solution (g)
2	$0.2 = \frac{\text{mass}}{56.11}$ Mass = 0.2 x 56.11 = 11.22
4	$0.2 = \frac{\text{mass}}{56.11}$ Mass = 0.4 x 56.11 = 22.44
6	$0.2 = \frac{\text{mass}}{56.11}$ Mass = 0.6 x 56.11 = 33.67
8	$0.2 = \frac{\text{mass}}{56.11}$ Mass = 0.8 x 56.11 = 44.89

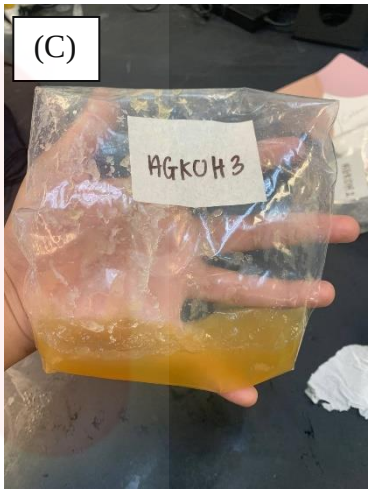
APPENDIX B

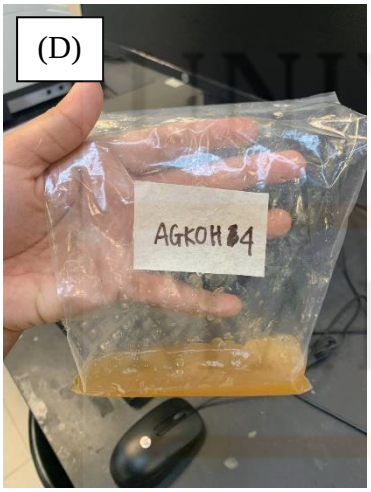
IMAGE

Agar hydrogel and agar hydrogel electrolyte

(A) 10g Agar (B) 2mole + 10g agar (C) 4mole + 10g agar (D) 6mole + 10g Agar (E) 8mole + 10g Agar







APPENDIX C

DATA AND GRAPH

C.1 Data of 15 sample with different concentrations low (2m of KOH), High (6m of KOH) and higher (8m of KOH)

Table C.1.1: The 5 sample of agar hydrogel electrolyte (2 moles)

Time	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
2 hours	1.20 v	1.20 v	1.30 v	1.30 v	1.30 v
4 hours	1.30 v	1.30 v	1.30 v	1.30 v	1.30 v
6 hours	1.30 v	1.30 v	1.30 v	1.30 v	1.30 v
8 hours	1.30 v	1.30 v	1.30 v	1.30 v	1.30 v
10 hours	1.30 v	1.30 v	1.30 v	1.20 v	1.30 v
12 hours	1.30 v	1.30 v	1.30 v	1.30 v	1.30 v
14 hours	1.30 v	1.30 v	1.30 v	1.30 v	1.20 v
16 hours	1.30 v	1.30 v	1.30 v	1.30 v	1.30 v
18 hours	1.30 v	1.30 v	1.30 v	0.9 v	1.30 v
20 hours	1.30 v	1.30 v	0.9 v	0.9 v	0.9 v
22 hours	1.30 v	1.30 v	0.9 v	0.9 v	1.30 v
24 hours	1.30 v	1.30 v	0.9 v	0.9 v	1.30 v

Table C.1.2: The 5 sample of agar hydrogel electrolyte (6 moles)

Time	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
2 hours	1.30 v	1.30 v	1.30 v	1.40 v	1.40 v
4 hours	1.40 v	1.40 v	1.30 v	1.40 v	1.40 v
6 hours	1.40 v	1.30 v	1.30 v	1.40 v	1.40 v
8 hours	1.40 v	1.30 v	1.30 v	1.40 v	1.40 v
10 hours	1.40 v	1.30 v	1.40 v	1.40 v	1.40 v
12 hours	1.40 v	1.30 v	1.40 v	1.40 v	1.40 v
14 hours	1.40 v	1.30 v	1.40 v	1.40 v	1.40 v
16 hours	1.40 v	1.40 v	1.40 v	1.40 v	1.40 v
18 hours	1.40 v	1.40 v	1.40 v	1.40 v	1.40 v
20 hours	1.40 v	1.40 v	1.40 v	1.40 v	1.40 v
22 hours	1.40 v	1.40 v	1.40 v	1.40 v	1.30 v
24 hours	1.40 v	1.40 v	1.40 v	1.40 v	1.30 v

Table C.1.3: The 5 sample of agar hydrogel electrolyte (8 moles)

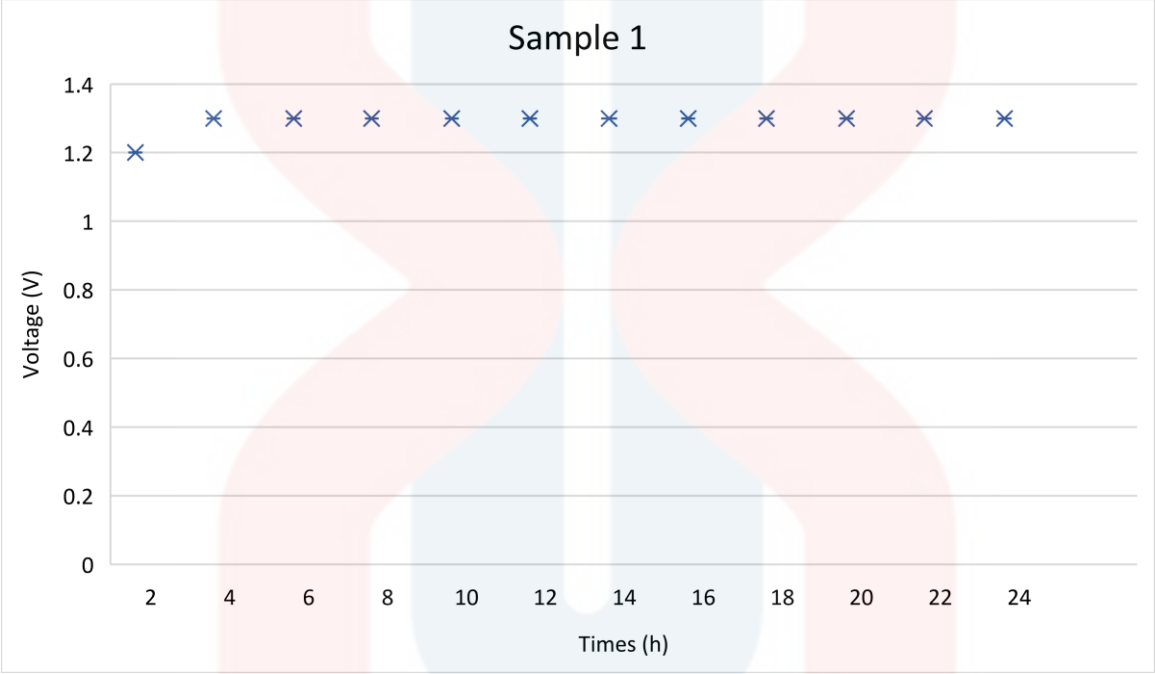
Time	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
2 hours	1.30 v	1.30 v	1.30 v	1.30 v	1.30 v
4 hours	1.40 v	1.30 v	1.30 v	1.30 v	1.30 v
6 hours	1.40 v	1.30 v	1.30 v	1.30 v	1.40 v
8 hours	1.40 v	1.30 v	1.30 v	1.30 v	1.40 v
10 hours	1.40 v	1.40 v	1.30 v	1.30 v	1.40 v
12 hours	1.40 v	1.40 v	1.30 v	1.30 v	1.40 v
14 hours	1.40 v	1.40 v	1.30 v	1.30 v	1.40 v
16 hours	1.40 v	1.40 v	1.40 v	1.30 v	1.40 v
18 hours	1.40 v	1.40 v	1.40 v	1.30 v	1.30 v
20 hours	1.40 v	1.40 v	1.30 v	1.30 v	1.40 v
22 hours	1.30 v	1.40 v	1.30 v	1.20 v	1.20 v
24 hours	1.30 v	1.40 v	1.30 v	1.20 v	1.20 v

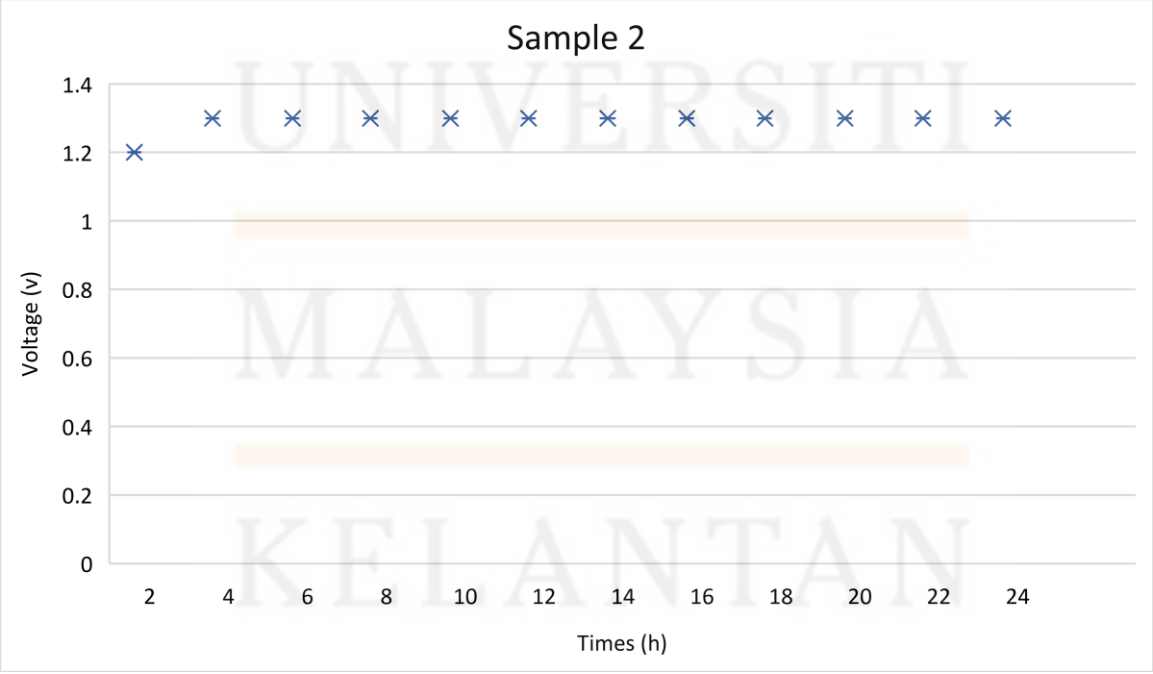
Table C.1.4: Average of 5 sample of Agkoh 2,4,5

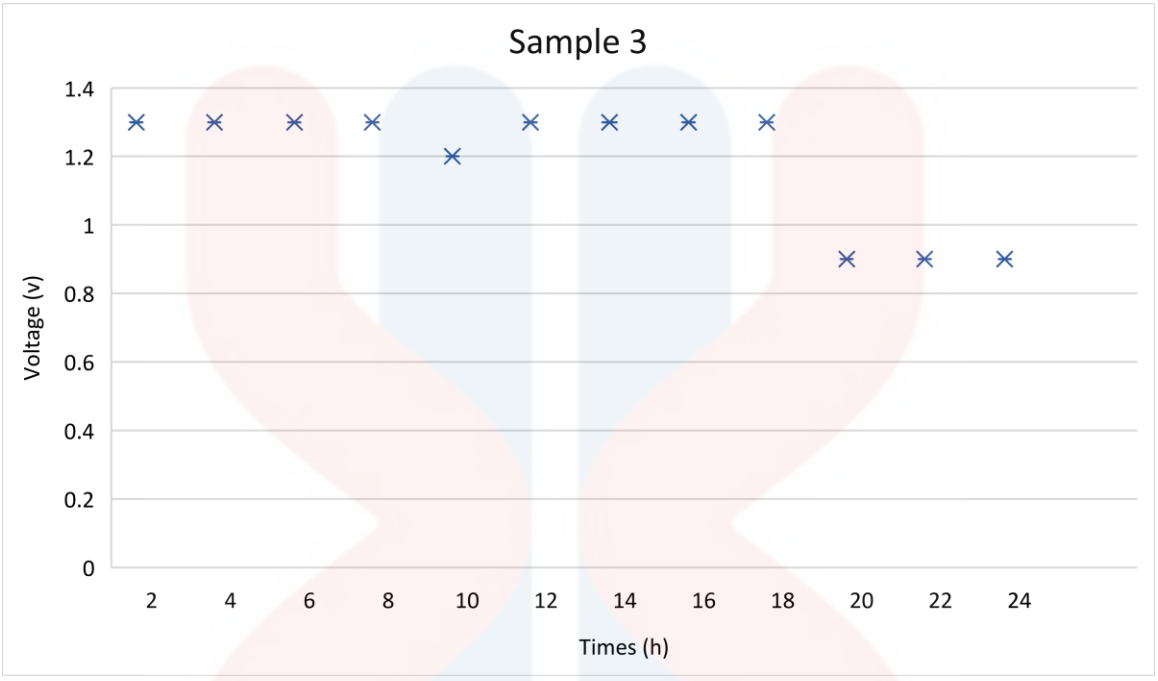
Types of samples	V_1 (Agkoh 2)	V_2 (Agkoh 4)	V_3 (Agkoh 5)
Sample 1	12.92 v	13.83 v	13.75 v
Sample 2	12.92 v	13.50 v	13.67 v
Sample 3	12.67 v	13.67 v	13.17 v
Sample 4	10.5 v	14.00 v	12.83 v
Sample 5	12.58 v	13.83 v	13.42 v
Average	**12.32 v**	**13.77 v**	**13.37 v**

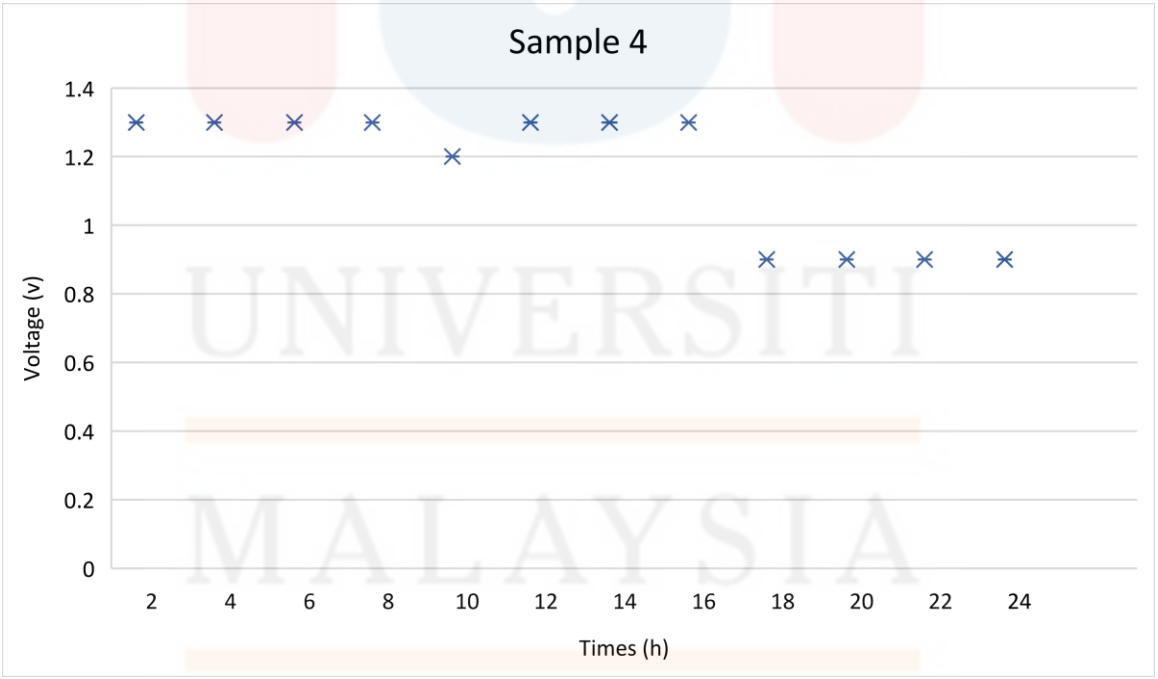
C.2 Graph of 15 sample agar hydrogel electrolyte with different concentrations low (2m of KOH), High (6m of KOH) and higher (8m of KOH)

Figure C.2.1: The graph of 5 sample of agar hydrogel electrolyte (2 Moles)









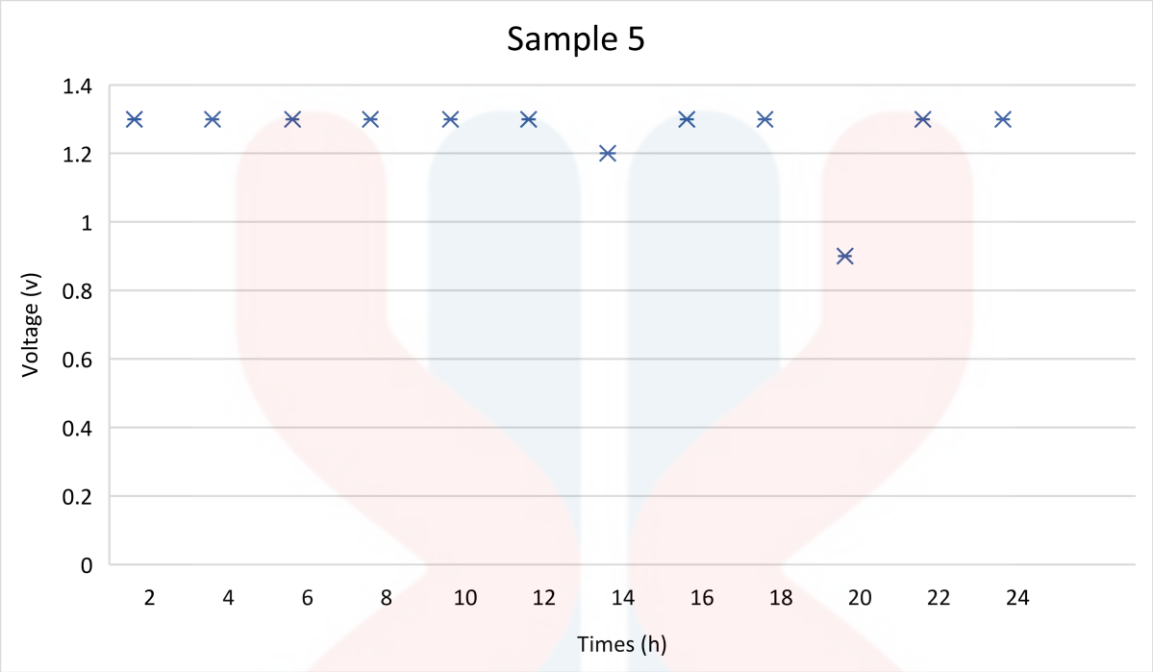
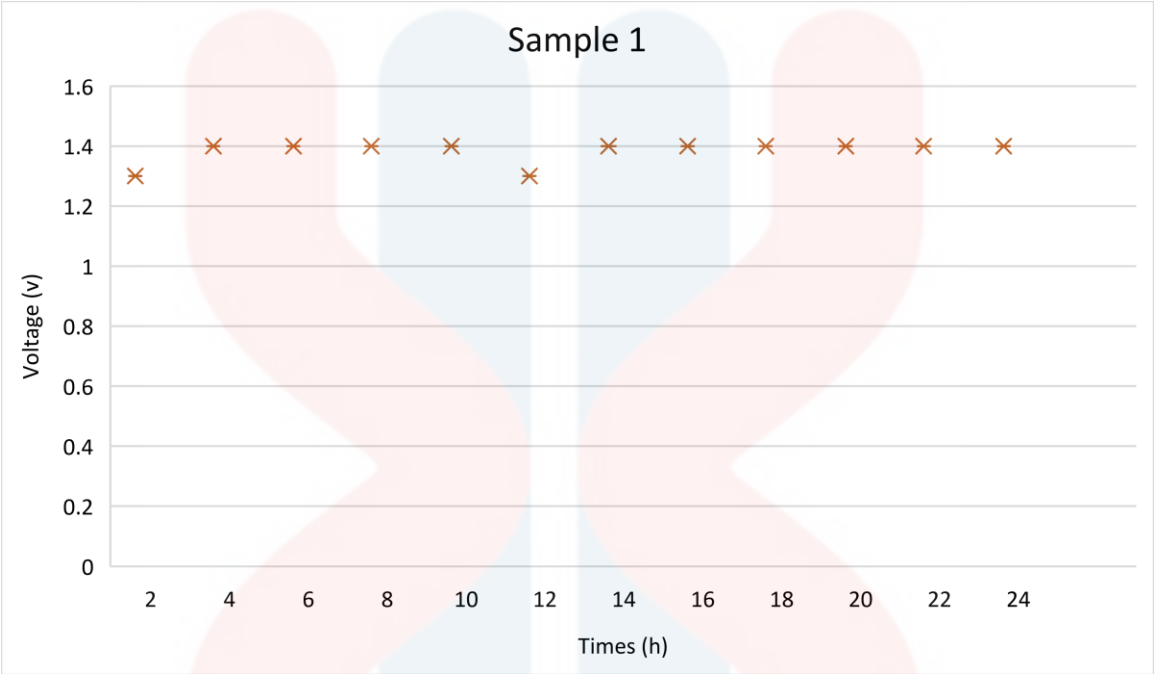
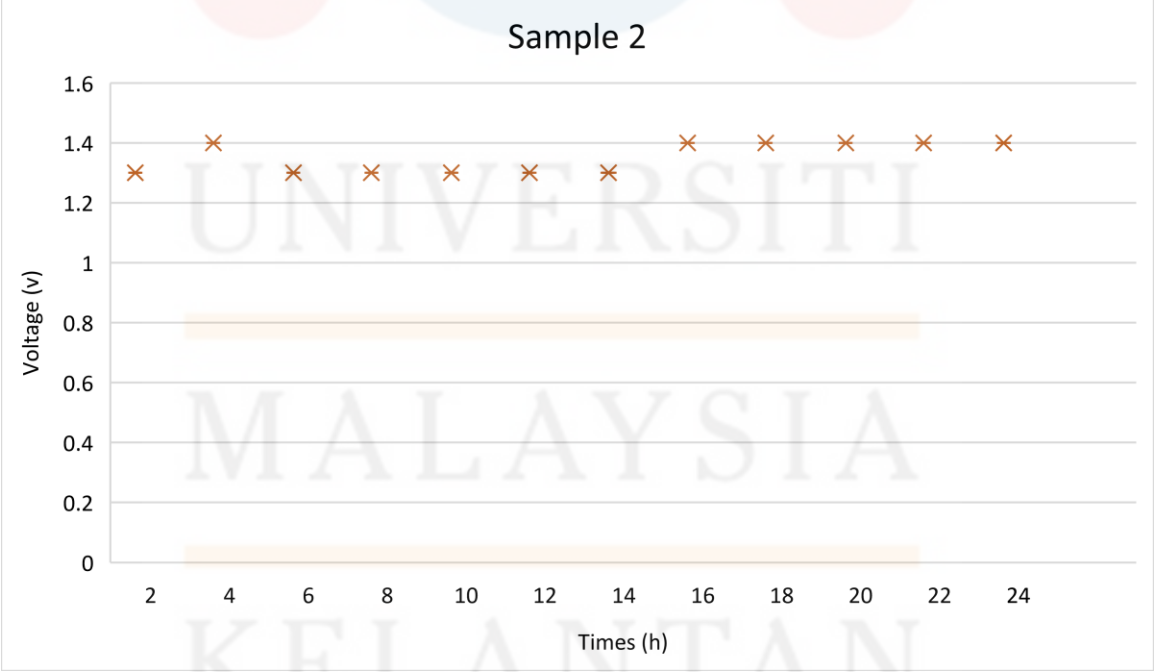
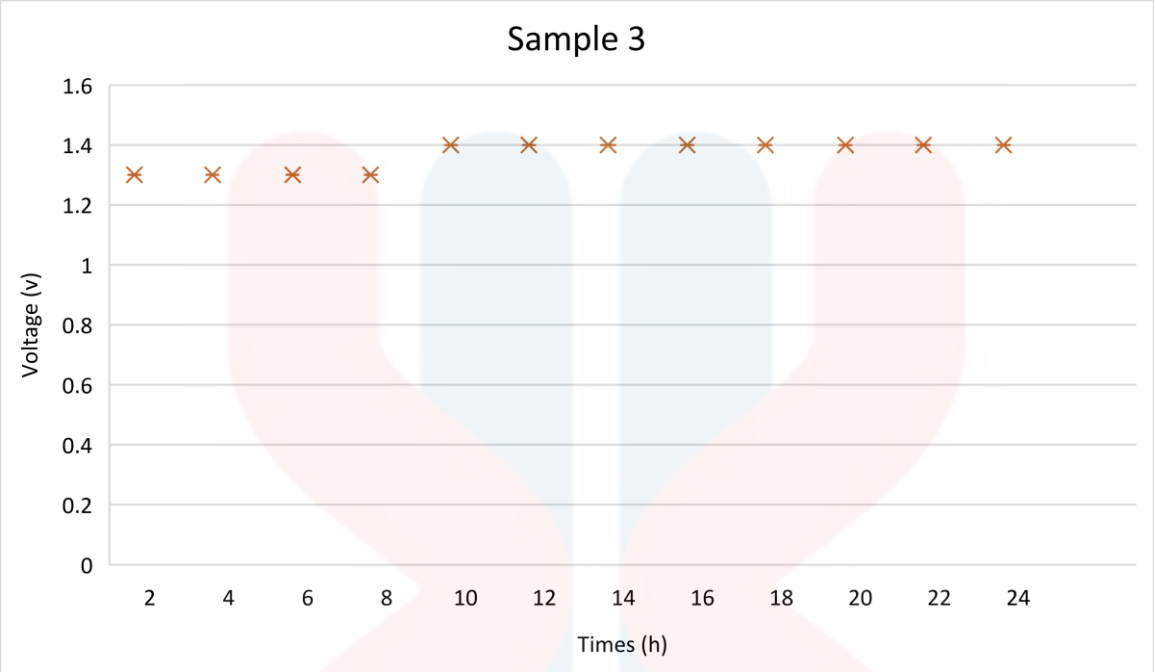
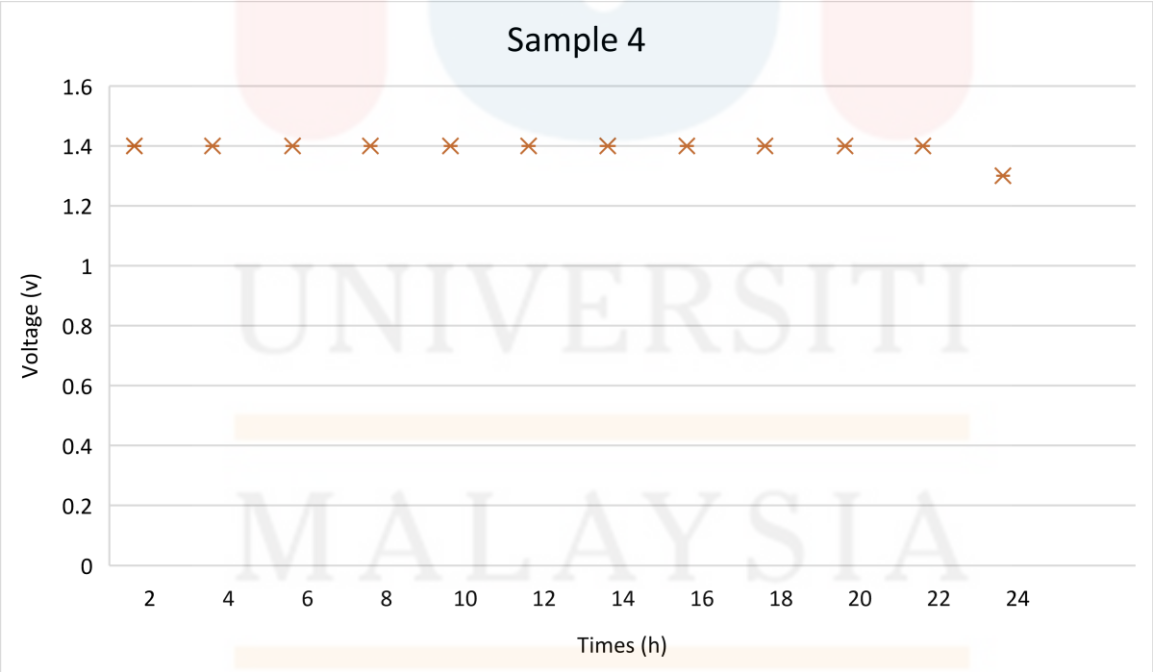


Figure C.2.2: The graph of 5 sample of agar hydrogel electrolyte (4 Moles)









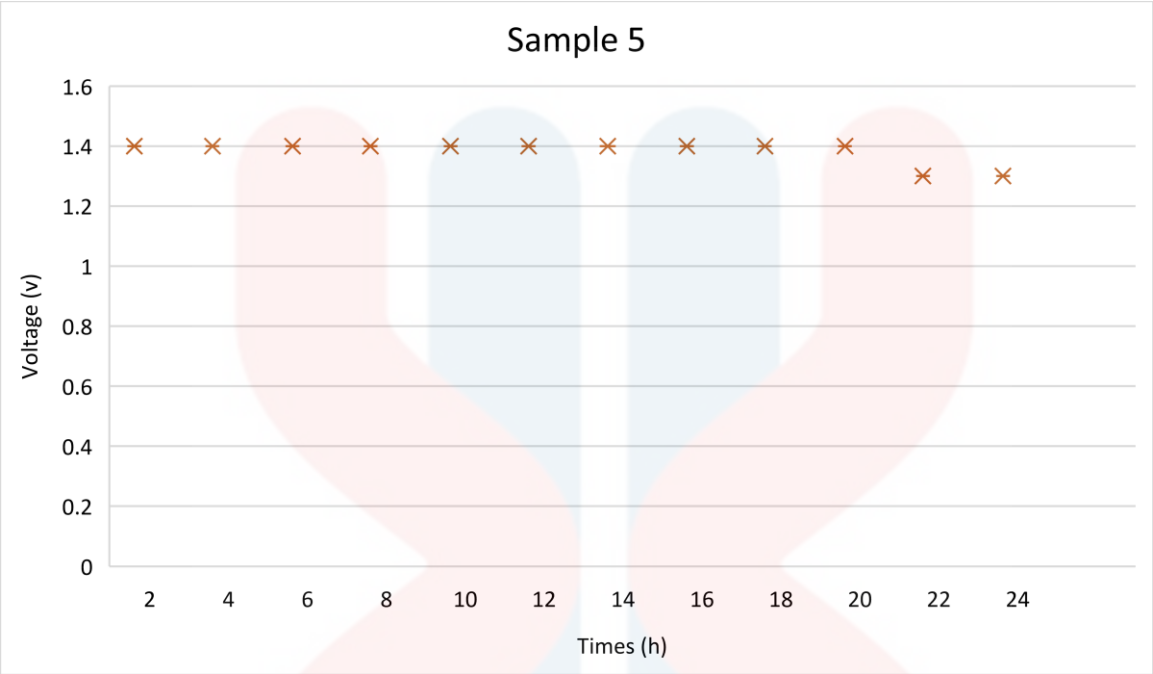


Figure C.2.3: The graph of 5 sample of agar hydrogel electrolyte (8 Moles)