CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Anodizing, or anodising, is an electrolytic passivation process used to increase the thickness of the natural oxide layer on the surface of metal parts. Anodizing increases corrosion resistance and wears resistance, and provides better adhesion for paint primers and glues than bare metal. Anodic films are most commonly applied to protect aluminium alloys, although processes also exist for titanium, zinc, magnesium, and niobium.

The history of electrochemical oxidation of aluminum dates back to the beginning of the last century. Anodic treatment of aluminum was intensively investigated to obtain protective and decorative films on its surface [7].More recently; applications of porous alumina with a huge surface area and a relatively narrow pore size distribution have been exploitedAnodizing was first used on an industrial scale in 1923 to protect Duralumin seaplane parts from corrosion. This early chromic acid process was called the Bengough-Stuart process and was documented in British defence specification DEF STAN 03-24/3. It is still used today despite its legacy requirements for a complicated voltage cycle now known to be unnecessary. Variations of this process soon evolved, and the first sulphuric acid anodizing process was patented by Gower and O'Brien in 1927. Sulphuric acid soon became and remains the most common anodizing electrolyte [7].

Oxalic acid anodizing was first patented in Japan in 1923 and later widely used in Germany, particularly for architectural applications. Anodized aluminum extrusion was a popular architectural material in the 1960s and 1970s, but has since been displaced by cheaper plastics and powder coating [8]. The phosphoric acid processes are the most recent major development, so far only used as pretreatments for adhesives or organic paints [7]. A wide variety of proprietary and increasingly complex variations of all these anodizing processes continue to be developed by industry, so the growing trend in military and industrial standards is to classify by coating properties rather than by process chemistry

1.2 PROBLEM STATEMENT

Many metals are structurally weakened by the oxidation process, but not aluminum. Aluminum can actually be made stronger and more durable through a process called 'anodizing' [1].

Anodized aluminum can be nearly as hard as diamond under the right anodizing process. Many modern buildings use anodized aluminum in places where the metal framework is exposed to the elements. Anodized aluminum is also a popular material for making high-end cookware such as frying pans and pots. Heat is distributed evenly across anodized aluminum, and the process of anodizing provides a naturally protective finish. It is possible to use another electroplating process to make anodized aluminum look like copper or brass or other metals. Special dyes can also be used to color the anodized aluminum for decorative uses.

Because of its strength and durability, anodized aluminum is also used in a number of other applications. Many of the satellites circling the Earth are protected from space debris by layers of anodized aluminum. The automobile industry relies heavily on anodized aluminum for trims and protective housings for exposed parts. Furniture designers often use anodized aluminum as the framework for outdoor pieces as well as the base metal for lamps and other decorative items. Modern home appliances and computer systems may utilize anodized aluminum as protective housing [1].

Anodized aluminum may not be appropriate for all applications because of its non-conductive nature. Unlike other metals such as iron, the oxidation process doesn't seem to weaken aluminum. The layer of 'aluminum rust' is still part of the original aluminum and will not transfer to food or easily flake off under stress. This makes it especially popular for food-service applications and industrial applications where durability is crucial.

This analysis and research is to analyze the effect of different electrical parameter (voltage) during the anodization process towards the anodized aluminum plates. Since there are a lot of applications of anodized aluminum in daily life, it is important to study on the improvement of this material. Anodized aluminum can be found on mp3 players, flashlights, cookware, cameras, sporting goods, window frames, roofs, in electrolytic capacitors, and on many other products both for corrosion resistance and the ability to retain dye. Although anodizing only has moderate wear resistance, the deeper pores can better retain a lubricating film than a smooth surface would. Most aluminum aircraft parts, architectural materials, and consumer products are anodized.

Figure 1.1: Hospital hollowware using anodized aluminum

1.3 OBJECTIVE

The main objective is to study the relationship between the voltage applied (electrical parameter) with effect on the structure and properties of anodized aluminium plate in the anodization process. Throughout the analysis and experiment, the comparison between the non anodized and anodized aluminium plate will be observe with different voltage applied in different time.

1.4 SCOPE OF STUDY

The scope of this study would be focus on:

- Compares the experiment result to state the different effects when different voltage applied on anodized aluminum plate.
- Conducting exact material testing on the specimen to examine the effect of electrical parameter towards structure and properties of anodized aluminium plates.

CHAPTER 2
LITERATURE REVIEW

2.1. ANODIZING ALUMINIUM

Anodizing is an electrochemical conversion process, not an applied coating. The surface of the aluminum metal is converted to aluminum oxide as a result of reactions occurring at the anode in an acidic solution. Aluminum has a very high chemical affinity for oxygen. Therefore, aluminum oxide can be formed easily. If you merely expose aluminum to air, a very thin oxide film of a few angstroms (1 angstrom is 10^{-8} cm) will be formed on the aluminum surface. This oxide film is called "air-formed oxide film" or "natural oxide film." The thickness of the air-formed oxide film is very small. Therefore, it cannot be used as a protective film [6].

The thickness and properties of the anodic coating will vary with alloy, anodizing process employed and cycle time (ampere-hours). Oxide formation proceeds inward, toward the source of fresh metal. The first formed oxide remains in contact with the anodizing solution throughout the process cycle; the last formed oxide is at the metal interface. The coating is 30-50 pct thicker than the metal it replaces, since the volume of oxide produced is greater than that of the metal replaced. Structure of most common anodic coatings is predominantly cellular/ porous. There is a very thin non-porous barrier layer at the interface.

The quality of the film is very sensitive to electrolyte temperature, concentration and the alloy used. The thickness is dependent on electrolyzing time and current density. If the temperature of the electrolytic bath is low, the growth of the oxide film is good and a hard oxide film can be formed. An oxide film formed in a sulfuric acid bath at approximately 0°C by anodizing of aluminum is called "hard anodizing film." This type of film is poor for dying and is usually naturally dark drown to black. At high electrolytic bath temperatures of 60 to 75°C the oxide film formed is thin and soft, and the surface will easily scratch and may even rub off [6].

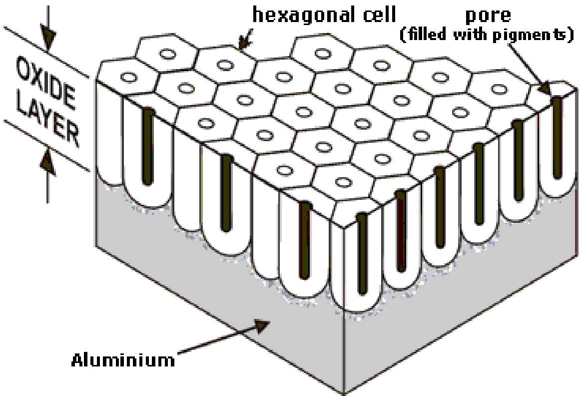


Figure 2.1: Aluminum oxide layer

2.2 ANODIZING PROCESS

If aluminum is used as the anode and electrolysis carried out using an electrolyte, an oxide film is formed on the surface of the aluminum. This electrolytic process is called "anodizing of aluminum." The process of anodizing is based on electrolysis method. When the passage of an electric current through a substance is accompanied by definite chemical changes which are independent of the heating effects of the current, the process is known as electrolysis, and the substance is called an electrolyte. As an example we may take the case of a solution of a salt such as copper sulphate in water, through which an electric current is passed between copper plates. We shall then observe the following phenomena [5].

1. The bulk of the solution is unaltered, except that its temperature may be raised owing to the usual heating effect which is proportional to the square of the strength of the current.
2. The copper plate by which the current is said to enter the solution, *i.e.* the plate attached to the so-called positive terminal of the battery or other source of current, dissolves away, the copper going into solution as copper sulphate.
3. Copper is deposited on the surface of the other plate, being obtained from the solution.
4. Changes in concentration are produced in the neighborhood of the two plates or electrodes.

In an anodizing cell, the aluminum work piece is made the anode by connecting it to the positive terminal of a dc power supply. The cathode is connected to the negative terminal of the supply. The cathode is an aluminum foil (electronic conductor) that is unreactive (inert) in the anodizing bath. When the circuit is closed, electrons are withdrawn from the metal at the positive terminal, allowing ions at the metal surface to react with water to form an oxide layer on the metal. The electrons return to the bath at the cathode where they react with hydrogen ions to make hydrogen gas.

Bath electrolytes are selected in which the oxide is insoluble, or dissolves at a slower rate than it deposits, and then an adherent oxide layer grows. The bath composition is the primary determinant of whether the film will be barrier or porous. Barrier oxide grows in near neutral solutions in which aluminum oxide is hardly soluble, most commonly ammonium borate, phosphate, or tartrate compositions. Porous oxide grows in acid electrolytes in which oxide can not only be deposited but also dissolves. The most widely used bath is dilute sulfuric acid, typically about 1 molar or 10 weight percent concentration. Other baths used for particular applications are made with oxalic acid or phosphoric acid [2].

Metals that can be anodized also react readily (oxidize) with oxygen in air, so that under ambient conditions the surface is always covered with a thin oxide film. The details of film structure and composition depend on the history of exposure to the ambient atmosphere but, on aluminum, there is always a barrier oxide layer next to the metal that is 2-3 nm thick. The barrier oxide stabilizes the surface against further reactions with its environment and is an excellent electrical insulator [2]. Figure 2.2 shows the oxide layer formed after the anodization process applied on the aluminum substrate.

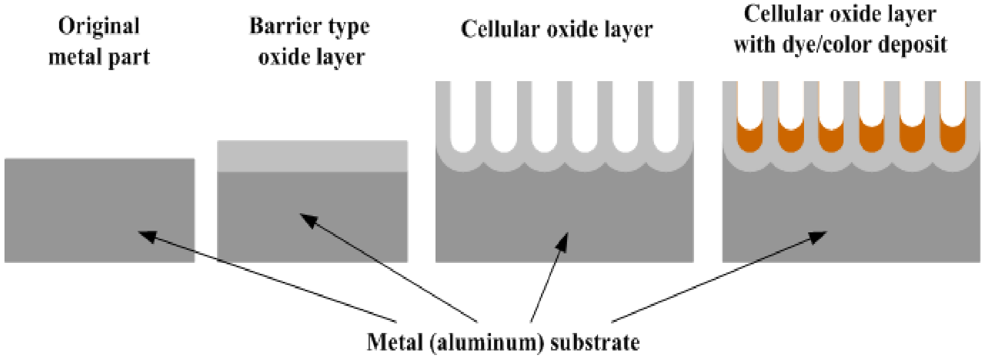


Figure 2.2: Oxide layer formed after the anodizing process [2]

When an aluminum piece covered with this oxide is made the anode in an electrolytic cell, containing, say, a acid electrolyte, no significant current flows until the voltage is raised to between 1 and 2 volt. This oxide supports an electric field (volts/thickness) of order 1 V/nm, a very high field indeed. If this were electronic current then water would be oxidized to evolve oxygen. But oxygen evolution is not seen – it does not occur because the oxide blocks electrons moving in the direction from electrolyte to metal. The voltage across the oxide can be increased, without initiating current flow, until the field in the oxide is large enough to drive aluminum and oxygen ions through the oxide. The current through the oxide is an ionic current and these ions react to form the oxide layer. The process of high-field ionic conduction is central to anodization [2].

Oxide anions move inward to react with aluminum at the metal/oxide interface to form oxide. Aluminum cations move outward from the metal to react with water at the oxide/electrolyte interface to form oxide at that surface. At the cathode, the circuit is completed by the reduction of hydrogen ions to hydrogen gas. New oxide deposits at both oxide interfaces, as illustrated in Figure 2.3.

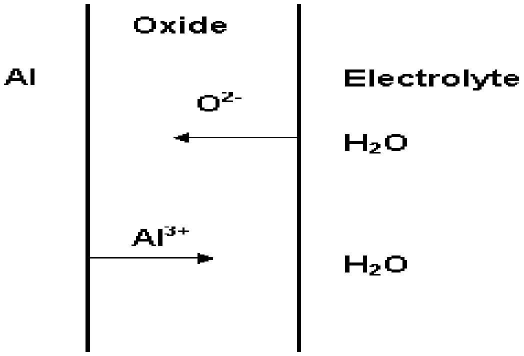


Figure 2.3: Sketch illustrating ion transport through the oxide film.

The rate at which the oxide thickens is proportional to the current density (A/cm^2). The field in the oxide does not change with oxide thickness, and has only a small dependence on current density and temperature. As the oxide thickens the voltage across the oxide increases proportionally, and at room temperature the thickness/voltage ratio is close to1.2 nm/V. Thickness is very uniform across the surface because everywhere the voltage drop must be the same [6].

2.2.1 Summary of anodizing reactions

The overall reaction that takes place during anodization is:

$$\mathbf{2Al + 3H_2O \rightarrow Al_2O_3 + 3H_2}$$

This is the sum of the separate reactions at each electrode. The reactions at the anode occur at the metal/oxide and oxide/ electrolyte interfaces. The ions that make up the oxide are mobile under the high field conditions. At the metal/oxide interface the inward moving oxygen anions react with the metal:

$$\mathbf{2Al + 3O^{2-} \rightarrow Al_2O_3 + 6e^-}$$

At the oxide/electrolyte interface outward moving aluminum cations react with water:

$$\mathbf{2Al^{3+} + 3H_2O \rightarrow Al_2O_3 + 6H^+}$$

(In case of aluminum dissolution into the electrolyte during porous film formation, the anodic reaction is:

$$\mathbf{2Al \rightarrow 2Al^{3+} + 6e-}$$

The reaction at the cathode is hydrogen gas evolution:

$$\mathbf{6H^+ + 6e- \rightarrow 3H_2}$$

The sealing reaction can be written as:

$$\mathbf{Al_2O_3 + 3H_2O \rightarrow 2AlOOH.H_2O}$$

2.3 BARRIER-TYPE AND POROUS-TYPE ALUMINIUM OXIDE

Depending on several factors, in particular the electrolyte, two types of anodic films can be produced. Barrier type films can be formed in completely insoluble electrolytes (5 < pH <7), e.g., neutral boric acid, ammonium borate, tartrate, and ammonium tetraborate in ethylene glycol. Porous type films can be created in slightly soluble electrolytes such as sulfuric, phosphoric, chromic and oxalic acid [2].

As shown in Fig. 2.4, both the barrier-type and the porous-type aluminum oxide films consist of an inner oxide of high purity alumina and an outer oxide layer comprised of alumina which has incorporated anions. In fact, the nomenclature of the inner and the outer oxide are determined in terms of the interfaces. The inner oxide is adjacent to the oxide/metal interface, while the outer oxide is adjacent to the electrolyte/oxide interface.

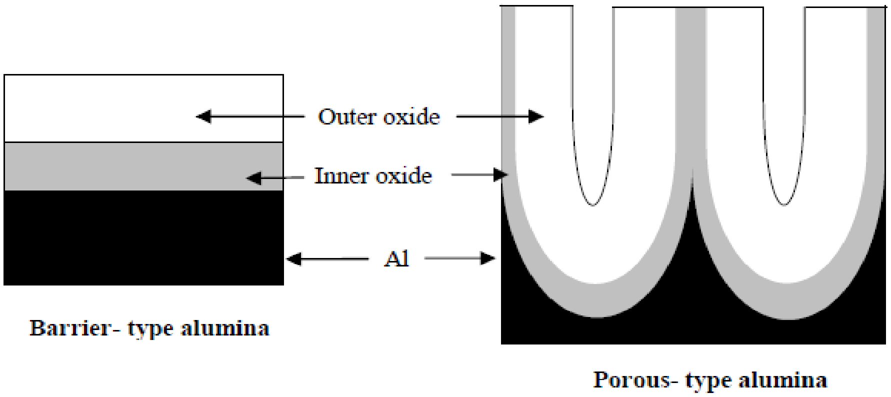


Figure 2.4: Schematic diagram for barrier type and porous type aluminum oxide [6].

The aluminum metal, an inner oxide consisting of pure alumina and an outer oxide consisting of an anion-contaminated alumina are indicated. Porous aluminum oxides are most commonly grown in dilute sulfuric acid, typically 10 weight percent concentration, but there also are commercial processes using phosphoric acid, chromic acid, oxalic acid, and mixtures of inorganic and organic acids. A feature common to these anodizing baths is the ability to retain a relatively high concentration of aluminum in solution. This is essential, because a large fraction of the aluminum that is oxidized is not retained in the film, but passes into solution [2].

For example, for anodization in sulfuric acid, about 60% of the oxidized aluminum is in the film and the remainder is found in solution. Porous films 100 µm thick can easily be made – this is 100 times thicker than the thickest barrier film. Unlike barrier films, a high voltage is not needed to make a thick porous film because of the unique structure of these films [6].

An idealized sketch of film structure is shown in Figure 2.5. The oxide has a cellular structure with a central pore in each cell. The sketch shows uniform hexagonal cells, but most anodization conditions produce films with more disorder, with a distribution of cell size and pores diameter. Cell and pore dimensions depend on bath composition, temperature, and voltage, but the end result always is an extremely high density of fine pores. The cell diameter is in the range 50-300 nm and the pore diameter is typically 1/3 to 1/2 of the cell diameter. The cell population density is from approximately 10 to more than 100 per μm^2. The aspect ratio is even more startling – commonly of the order of 1000:1. For example, film thickness of 20 to 50 µm with 20 nm pores is typical for coatings grown in sulfuric acid [6].

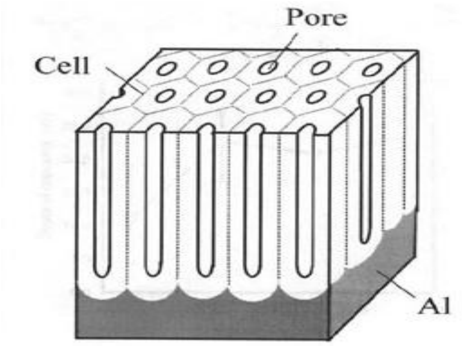


Figure 2.5: Idealized structure of anodic porous aluminum oxide [6].

A thin barrier oxide is at the base of each pore. Figure 2.6-top shows a film in the vicinity of the metal/oxide interface that was grown in phosphoric acid. This is a transmission electron micrograph of a carbon replica of the fracture surface. Four pores are in the micrograph, and each pore is centered over a scallop-shaped depression in the metal. At the base of each pore is anodic oxide 0.2 µm thick and pores are separated by oxide comprising the cell walls.

As illustrated in Figure 2.6-center, the cell geometry concentrates current through the oxide at the pore base, the dark grey zones in the figure. The shortest path between metal and electrolyte is within this zone, and the field is uniform and at its highest value. This is a critical feature for development of the porous structure. Ions move by high field conduction: aluminum cations reach the pore surface and pass into solution, and oxide deposition is confined to the metal/oxide interface at the pore base. As aluminum metal is oxidized, the metal/oxide interface moves into the metal [6].

This is illustrated in Figure 2.6-bottom where we try to show this inward motion by superposing the pore positions at an earlier and later time of anodization. What had been barrier oxide at the periphery of the pore base is no longer penetrated by the field and becomes part of the cell wall. The cell wall and pore increase in height, that is, the film thickens, while cell and pore diameters remain fixed. Because the barrier oxide thickness remains constant, the cell voltage and current remain nearly constant as the film thickens.

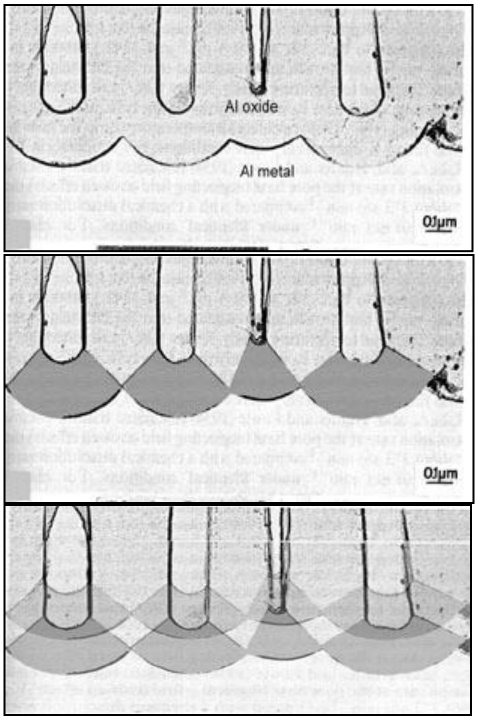


Figure 2.6: Top: Cross-section of porous oxide in the vicinity of metal/oxide interface. Center: Ion transport occurs only within the dark grey zones. Bottom: Snapshots near the metal surface at short and long anodization times [6].

2.4 FARADAY LAW

Rather than electrolysis process, this study also includes the theories of Faraday Law. The first exact quantitative study of electrolytic phenomena was made about 1830 by Michael Faraday (Experimental Researches, 1833). When an electric current flows round a circuit, there is no accumulation of electricity anywhere in the circuit, hence the current strength is everywhere the same, and we may picture the current as analogous to the flow of an incompressible fluid. This theory is to examine the relation between the flow of electricity round the circuit and the amount of chemical decomposition [3].

The current driven passed by a voltaic battery ZnPt (Fig. 2.6) through two branches containing the two electrolytic cells A and B. The reunited current was then led through another cell C, in which the strength of the current must be the sum of those in the arms A and B. It found that the mass of substance liberated at the electrodes in the cell C was equal to the sum of the masses liberated in the cells A and B. For the same current, the amount of chemical action was independent of the size of the electrodes and proportional to the time that the current flowed. Regarding the current as the passage of a certain amount of electricity per second, it will be seen that the results of all these experiments may be summed up in the statement that the amount of chemical action is proportional to the quantity of electricity which passes through the cell.

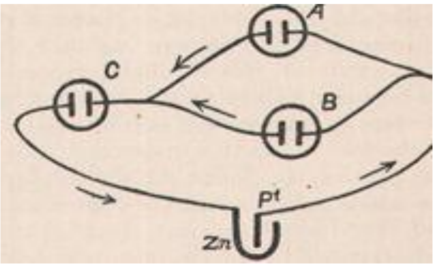


Figure 2.7: Electric circuit using by Faraday [3].

The next step was to pass the same current through different electrolytes in series. The result found that the amounts of the substances liberated in each cell were proportional to the chemical equivalent weights of those substances. Thus, if the current be passed through dilute sulphuric acid between hydrogen electrodes, and through a solution of copper sulphate, it will be found that the mass of hydrogen evolved in the first cell is to the mass of copper deposited in the second as is to 31.8. Now this ratio is the same as that which gives the relative chemical equivalents of hydrogen and copper, for gramme of hydrogen and 31.8 grammes of copper unite chemically with the same weight of any acid radical such as chlorine or the sulphuric group, SO 4. This theory examined also the electrolysis of certain fused salts such as lead chloride and silver chloride. Similar relations were found to hold and the amounts of chemical change to be the same for the same electric transfer as in the case of solutions [3].

CHAPTER 3

METHODOLOGY

3.1 PROJECT FLOW

The flow chart of the methodology is shown in Figure 3.1.

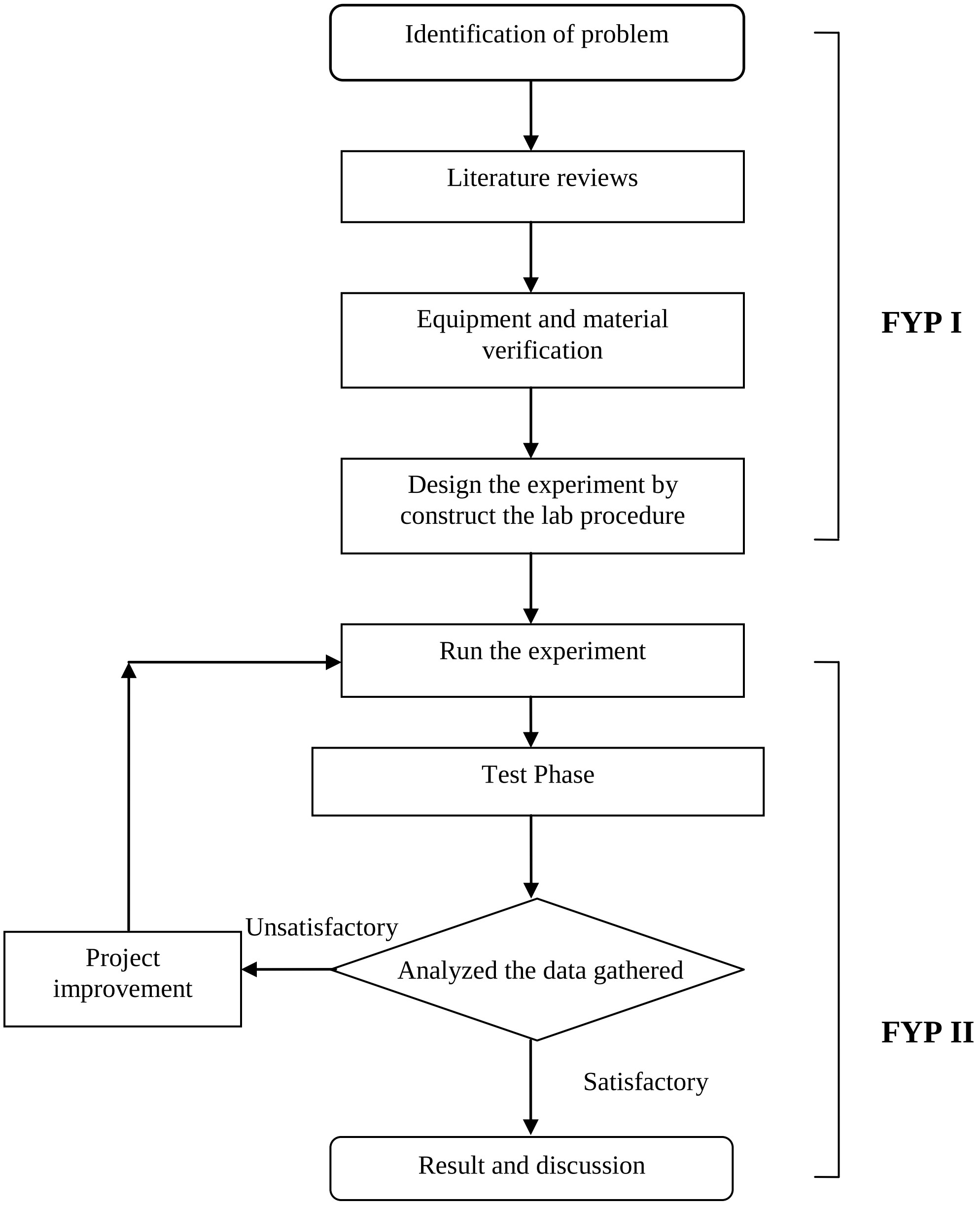


Figure 3.1: Flow Chart of Project Methodology

Since this is experimental and analysis project, therefore the project should be done step-by-step so that all tasks done are clearly understood and a good result can be obtained. The methodology is as follow.

3.1.1 Identification of problem

First of all, introduction of the study need to be identify to know the relevancy to conduct the project. Introduction must include the background of the project, the problem statement, the objective and scope of the study. Problem statement needs to focus on the situation of the problem and research question which lead to the objective of the study. Clarification the boundary project work needs to be done to ensure the feasibility within the given time frame.

3.1.2 Literature review and theory

Basically, at this stage all information related to anodising such as passivation process, electrolysis and Faraday Law are clearly analyzed in order to get a strong background of the subject matter. The medium of the research can be through the reading of the related reference books, journals as well as from surfing the internet. Most of the information were discussing in different sub topic and need to be correlated for each part and this need to be done as soon as possible. It also including revising all lab related lab work that need to be perform in the future finding.

3.1.3 Equipment and material verification

After all necessary information has been collected; it comes to part of identifying and selecting type of method to be used. Certain chemical solutions, tools and equipment need to be identified before start to do the experiment. This process need the student to consult with lab coordinators, lecturers and technician to ensure all the material needed are ready before doing the experiment. HSE matter is the critical part since the experiment needs the student to handle some hazardous solution.

3.1.4 Design the experiment

Then, construct the procedure of the experiment to with supervision by the supervisor to ensure that the experiment run systematically. The method that has been used is the electrolysis method using the some specifically combination of circuit system. Refer to appendix A for the specific laboratory lab procedure.

3.1.5 Run the experiment

The method that has been used is the electrolysis method using the some specifically combination of circuit system. Certain chemical solutions, tools and equipment need to be identified before start to do the experiment.

3.1.6 Material Testing

Flow chart also shows that this project has reached the critical part of the milestone which is to conduct the specimen testing and identification. Ten different specimens that have undergone the anodizing process will be analyzed by several materials testing technique. This process includes SEM and FTIR. The testing method has been covered in Chapter 2 above.

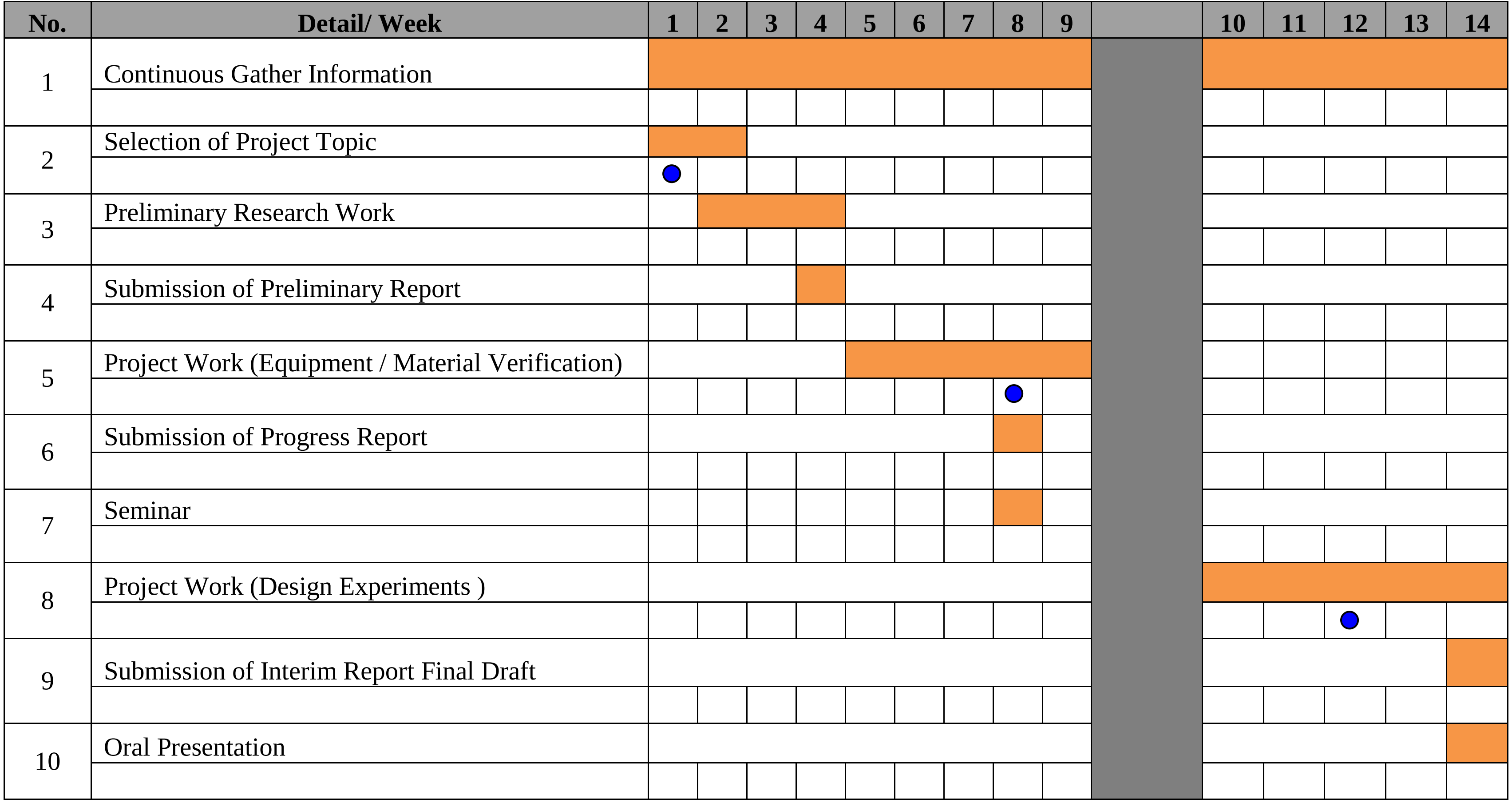
3.1.7 Analyzed the data for result and discussion

Lastly, all the data gathered should be analyzed to achieve the objective of this project. Further improvement will be plan and implement to the project to ensure this project achieve the objective.

3.2 MILESTONE FOR FINAL YEAR PROJECT

See Table 1 below for the Gantt chart that shows the project flow and milestone for the Final Year Project 1:

Table 1: Gantt chart for the Final Year Project 1



No.	Detail/ Week	1	2	3	4	5	6	7	8	9		10	11	12	13	14
1	Continuous Gather Information	🟧	🟧	🟧	🟧	🟧	🟧	🟧	🟧	🟧		🟧	🟧	🟧	🟧	🟧
2	Selection of Project Topic	🟧	🟧													
		●														
3	Preliminary Research Work		🟧	🟧	🟧											
4	Submission of Preliminary Report				🟧											
5	Project Work (Equipment / Material Verification)					🟧	🟧	🟧	🟧	🟧						
									●							
6	Submission of Progress Report								🟧							
7	Seminar								🟧							
8	Project Work (Design Experiments)											🟧	🟧	🟧	🟧	🟧
														●		
9	Submission of Interim Report Final Draft															🟧
10	Oral Presentation															🟧

● Suggested milestone

🟧 Process

See Table 1 below for the Gantt chart that shows the project flow and milestone for the Final Year Project 2:

Table 2: Gantt chart for the Final Year Project 2

No.	**Detail/ Week**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**		**10**	**11**	**12**	**13**	**14**
1	Continuous Gathering Information	🟧	🟧	🟧	🟧	🟧	🟧	🟧	🟧	🟧		🟧	🟧	🟧		
2	Project Work (Conduct Project Experiment)	🟧	🟧	🟧	🟧											
				●												
3	Submission of Progress Report 1				🟧											
4	Project Work (Conduct Material Testing)					🟧	🟧	🟧	🟧	🟧						
									●							
5	Submission of Progress Report 2								🟧							
6	Seminar (compulsory)								🟧							
7	Project work (Data Analysis & Evaluate Results)											🟧	🟧	🟧		
													●			
8	Poster Exhibition											🟧				
9	Submission of Dissertation (soft bound)														🟧	
10	Oral Presentation															🟧
11	Submission of Project Dissertation (hard bound)											7 days after oral presentation				
																●

● Suggested milestone

🟧 Process

3.3 TOOLS AND EQUIPMENT

To construct the electrolysis circuit, some apparatus are needed which are:

1. Aluminium foil
2. Aluminium strip
3. DC power supply
4. Crocodile clips
5. Beaker

Chemical solutions required during the process as follow:

1. Acid Sulphuric
2. Acid Nitric
3. Sodium Hydroxide
4. Acetone
5. Organic Dye (Carmoisine)

Figure 3.2 shows the illustration of laboratory design of electrolysis circuit used during the anodizing process.

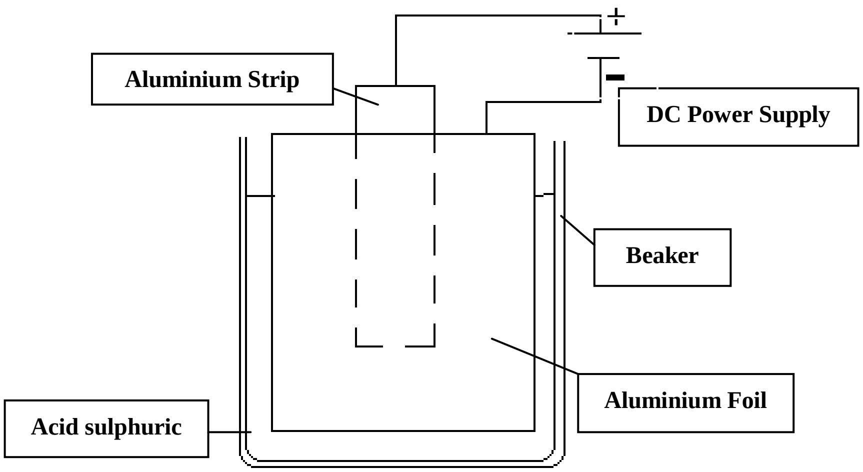


Figure 3.2: Laboratory design of electrolysis circuit

3.4 LABORATORY PROCEDURE

Appropriate laboratory analysis should be done to prove that the early assumption that increasing in current density and time taken during the anodizing process can provide better anodizing effect on aluminum. From the data obtained, each specimen will be conducting same material testing technique to identify the effect of its properties and structure after the experiment. The result of this testing will determined the new characteristic of the aluminum surface after different electrical parameter applied on the specimens. A specific procedure of anodization is being used by using all specified equipment and chemical solutions to analyze the effect on aluminum substrate. Figures 3.3 - 3.7 below shows the steps taken in order to conduct the anodizing process:

Figure 3.3: Labeled chemical solution.

1. All the chemicals and material needed are being identified. All chemical solutions are being labeled safely to avoid any accidents occur during the experiment.

Figure 3.4: Removed oil & greases on specimens.

2. Oils and greases are removed in weak alkali solutions and the surface is etched to remove heavy oxides. After rinsing, the aluminum is dipped into a desmut solution to remove the insoluble components of the aluminum which remain on the surface after etching, rinsed and presented for anodizing.

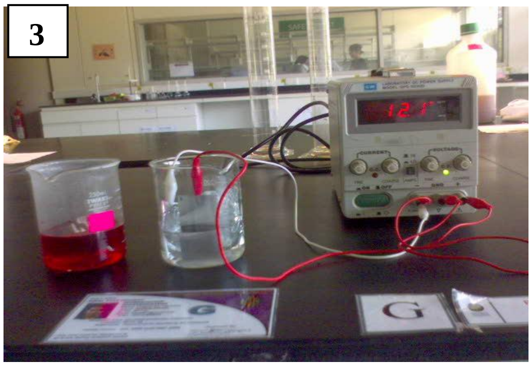


Figure 3.5: A DC current passed between aluminium strip and aluminium foil.

3. During anodizing the part is made the anode (positive electrode) in an electrolytic cell. The aluminium is immersed in an electrolyte consisting of an acid/water solution.

Figure 3.6: Specimen dipped into dye solution

4. The clear anodized aluminium with its unsealed porous film is immersed in a bath containing organic dyes. The colorant is absorbed into the pores of the film and subsequently sealed in.

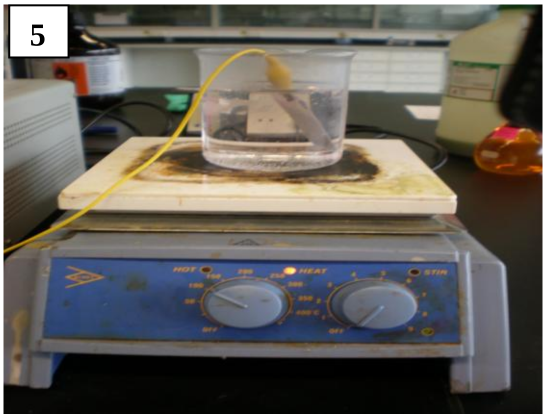


Figure 3.7: Specimen dipped into boiling water

5. The anodic film thus produced is quite porous and will accept or trap any material into its pores. To prevent this occurring these pores are closed or the coating is "sealed". This is done by adding water to the oxide (hydrolyzing).The sealing process may be carried out in boiling water.

CHAPTER 4

RESULT AND DISCUSSION

4.1 CURRENT DENSITY AND MASS GAIN ANALYSIS

From the anodizing process discussed on Chapter 3, a set of data were obtained. These data were analyzed to obtain the relationship between the current density and mass gain of anodized aluminum plate. The data shown in the tables 3-6 below:

Table 3: Anodizing process by using 12 Volt

12V	Time (m)	Mass before (g)	Mass after (g)	Mass different (g)	Current (A)	Current Density (A/cm^2)
Specimen 1	15	1.511	1.555	0.004	0.01 – 0.05	0.0017
Specimen 2	30	1.521	1.563	0.042	0.01 – 0.07	0.0022
Specimen 3	60	1.475	1.526	0.051	0.13 – 0.15	0.0078

Table 4: Anodizing process by using 20 Volt

20V	Time (m)	Mass before (g)	Mass after (g)	Mass different (g)	Current (A)	Current Density (A/cm^2)
Specimen 4	15	1.510	1.522	0.012	0.07 - 0.13	0.0056
Specimen 5	30	1.533	1.625	0.092	0.08 - 0.17	0.0069
Specimen 6	60	1.498	1.625	0.099	0.13 – 0.29	0.0116

Table 5: Anodizing process by using 21 Volt

21V	Time (m)	Mass before (g)	Mass after (g)	Mass different (g)	Current (A)	Current Density (A/cm^2)
Specimen 13	15	1.437	1.452	0.015	0.09-0.49	0.0161
Specimen 14	30	1.433	1.45	0.017	0.15-0.48	0.0175
Specimen 15	60	1.338	1.356	0.018	0.16-0.47	0.0175

Table 6: Anodizing process by using 24 Volt

24V	Time (m)	Mass before (g)	Mass after (g)	Mass different (g)	Current (A)	Current Density (A/cm^2)
Specimen 7	15	1.497	1.431	-0.066	0.13 – 2.00	0.059
Specimen 8	30	1.539	1.441	-0.098	0.06 – 1.59	0.0458
Specimen 9	60	1.451	1.147	-0.304	0.38 – 0.51	0.0247

From the data obtained, current density applied on the specimens calculated by using:

Current density (A/ cm$^{2)}$ = Current (A) / Aluminium surface area (cm^2)

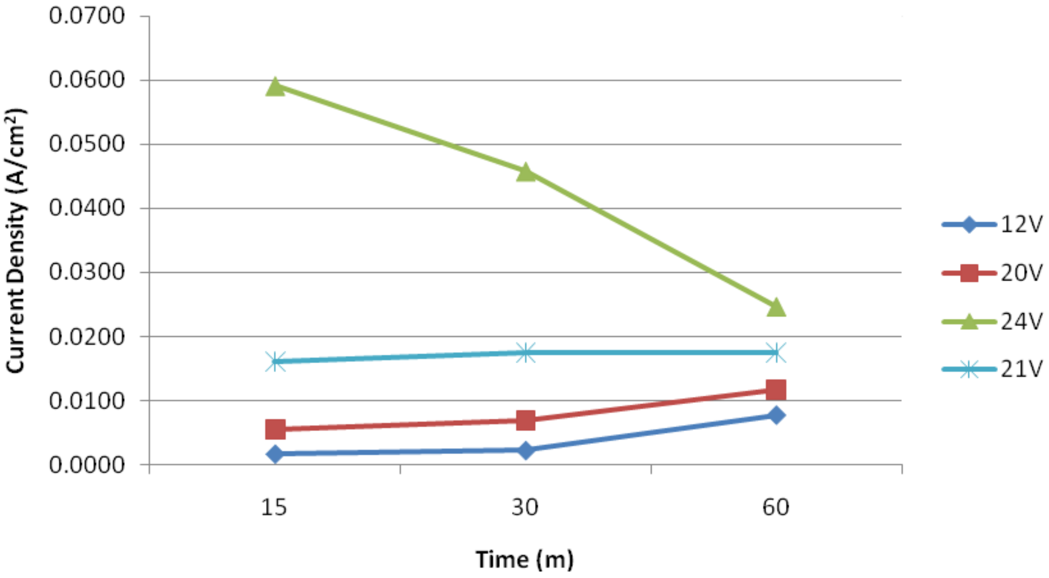


Figure 4.1: Current density (g) VS time (m) graph

From the Figure 4.1 above, there are four different trends occur after applying different electrical parameter (voltage) to the specimen. Current density is a measure of the density of flow of a conserved charge. It is defined as a vector whose magnitude is the current per cross-sectional area. On this analysis, the cross-sectional area of the aluminium that have been anodized has been specified to 18 cm^2.It is well known that current density increase with time when specified voltage is applied on the specimens. In other words, it means that the mass gain and current density are directly proportional to the time taken. However, this hypothesis is only valid for group of specimen for 20V and 21V.

The current density is decreasing tremendously by time happen on 24V specimen. This due to the increasing of the oxide layer on the surface of aluminium, the current flow is inadequate. This phenomena occur when the area of surface contact of aluminium decreasing after the formation and deposition of the oxide layer on aluminium. Increasing current density will result to the increment of the deposition of oxide layer.

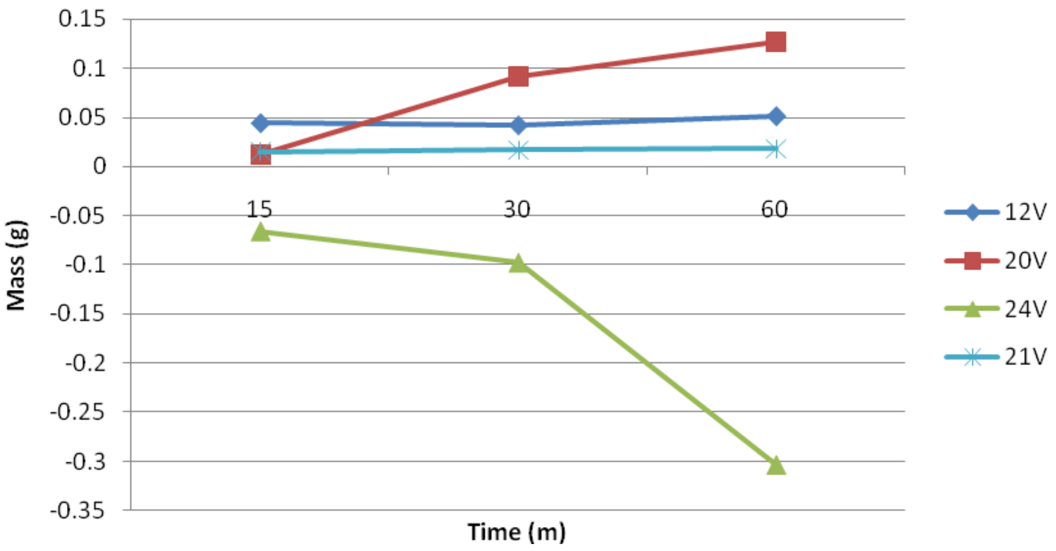


Figure 4.2: Mass gain (g) VS time (m) graph

Same phenomena take place on the mass gain versus time for 12V, 20V, 21V and 24V.For 12 V graph trend, there is a slightly change in the mass gain. it happen because deposition of oxide aluminium not taking place reactively since the reaction of anodizing is minima on 12 V. Nevertheless, a number of amount in mass gain occur on the 20V due to the increment of voltage on the anodizing process.

The trends of the graphs for 24V specimens seem different compared to the previous specimen (12V and 22V). This is because when 24V is applied on the specimens, the mass gain will decrease with increasing in time. That means that the mass gain is inversely proportional by time. The mass of the aluminium plate decrease because the aluminium on the plate is dissolving into the acid solution. The oxidation rate due to the higher voltage applied exceeding the deposition ensuing to the competition between Al^{3+} ion from the cathode and anode. Therefore, the aluminium plate tends to erode to Al^{3+} ion and dissolve in the solution. This occurrence supports the previous fact that the chemical reaction dominated the process of anodizing rather than electrical process. 24V is considered as overvoltage since it damages the structure of aluminium.

For current density and mass gain versus time graph above, there are slightly no change of current density and mass gain by time. There is constant current flow for 21 V specimens due to the stabilization of reaction between ions in the process. There is also no mass gain since the process reach the optimum voltage applied for anodizing process. To conclude that 21V is the optimum voltage should been applied in anodizing process.

4.2 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

Scanning Electron Microscopy (SEM) scan the surface of a specimen with an electron beam and the reflected (back scattered) beam of electrons is collected and displayed at the same scanning rate on a cathode-ray tube. The image on the screen contains the surface features (with good depth of field) of the specimen, which may be photographed. The major requirement for the specimen is that it be electrically conductive. Accessory equipment allows analysis of the chemical composition of much localized areas of the sample.

4.2.1 Objective

- To analyse the surface features and compare between pure aluminium, anodized aluminium without and with addition of dye.
- Define the thickness and interface between aluminium and oxide layer on the specimens.

4.2.2 Sample preparation

All samples must also be of an appropriate size to fit in the specimen chamber and are generally mounted rigidly on a specimen holder called a specimen stub

For conventional imaging in the SEM, specimens must be electrically conductive, at least at the surface, and electrically grounded to prevent the accumulation of electrostatic charge at the surface. Metal objects require little special preparation for SEM except for cleaning and mounting on a specimen stub. Nonconductive specimens tend to charge when scanned by the electron beam, and especially in secondary electron imaging mode, this causes scanning faults and other image artefacts. They are therefore usually coated with an ultrathin coating of electrically-conducting material, commonly gold, deposited on the sample either by low vacuum sputter coating or by high vacuum evaporation. Coating prevents the accumulation of static electric charge on the specimen during electron irradiation.

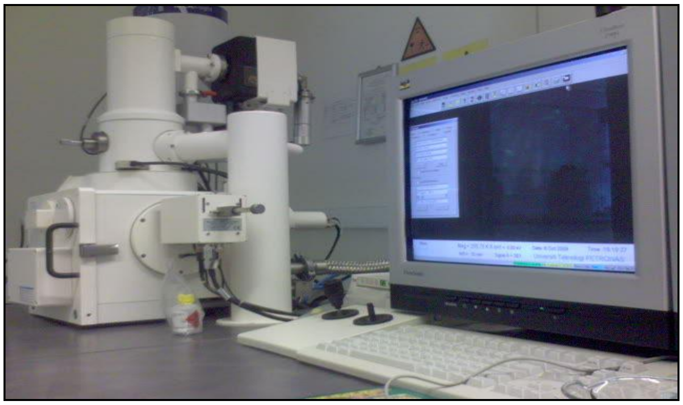
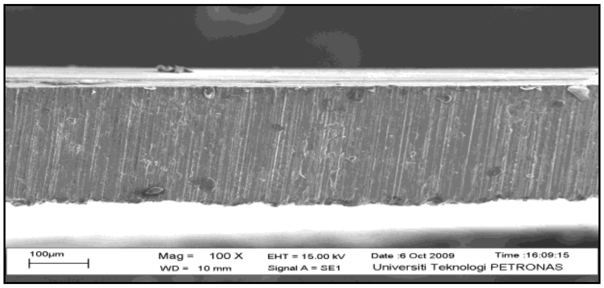
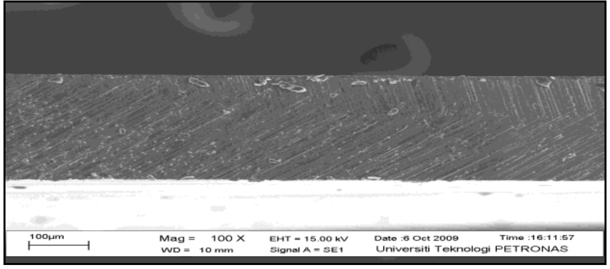
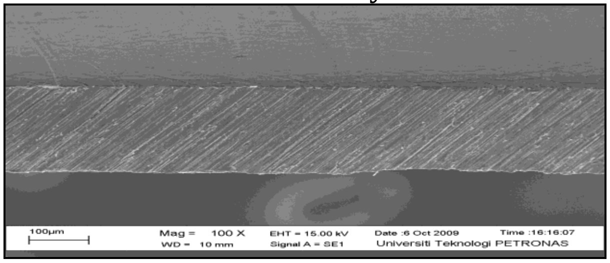
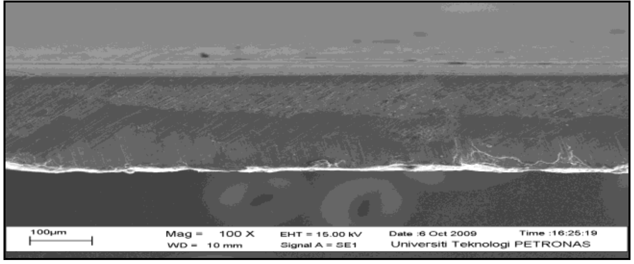


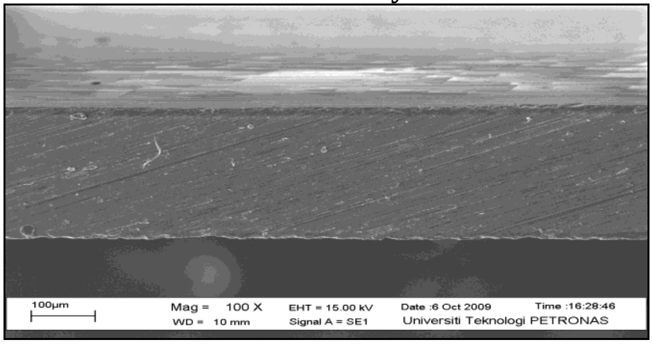
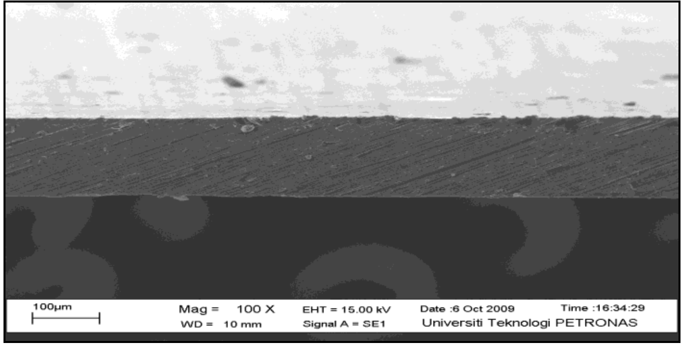
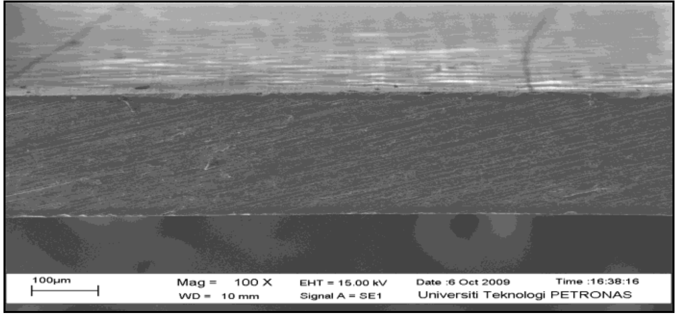
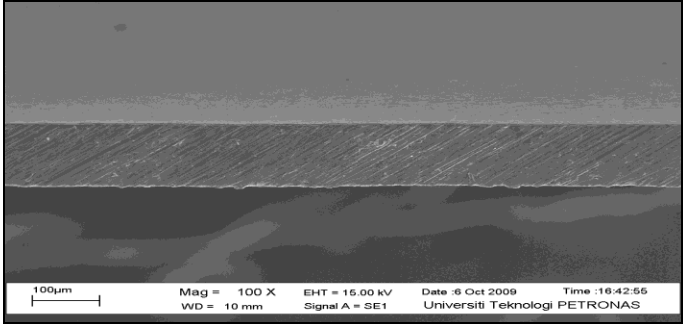
Figure 4.3: Scanning Electron Microscopy (SEM) machine

4.2.3 Data gathering and analysis

After running the SEM analysis, a bunch of data is obtained. Table below shows the differentiation between the thicknesses of the aluminium plate.

Table 7: Thickness of anodized aluminium wit and without dye

No.	Sample	Thickness (µm)
1.	**Pure aluminium**  	320.4
2.	**12V Non-dye**  	304.8
3.	**12V With dye**  	255
4.	**20V Non-dye**  	271.6

No.	Sample	Thickness (µm)
5.	**20V With dye** 	296
6.	**22V Non-dye** 	175.8
7.	**22 V With dye** 	295
8.	**24 V With dye** 	145.6

Based on Table 7 above, study has been made to examine the differences in thickness when applying different electrical parameter (voltage).

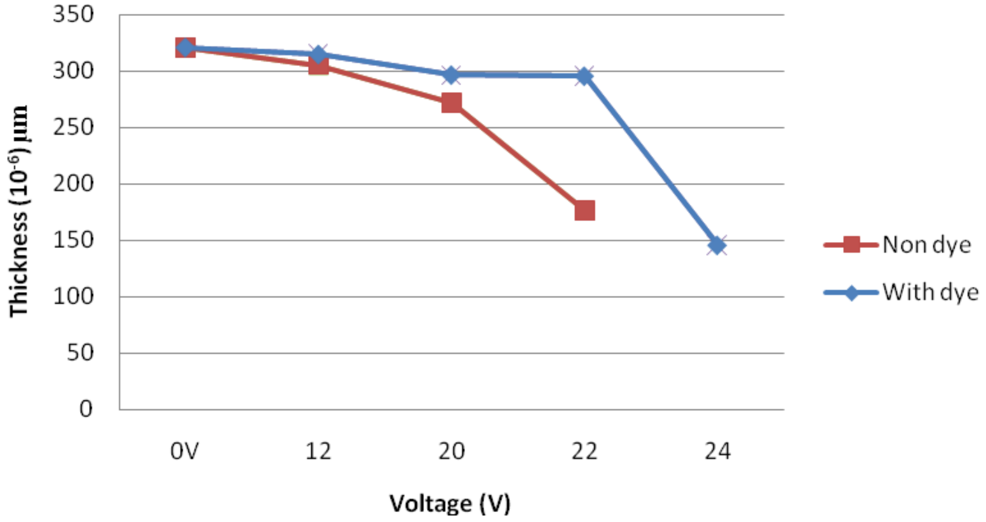


Figure 4.4: Thickness (µm) VS voltage (V) graph

Figure 4.4 above shown that the thickness of the aluminum strip is decreasing when the voltage applied is rising. The thicknesses of anodized aluminum with dye thicken compared to the non anodized aluminum. The degree of incorporation of electrolyte species in the outer oxide layer of barrier-type alumina strongly depends on the type of electrolyte, the concentration of adsorbed anions, and the faradic efficiency of film growth. This group found that the electrolytes can be classified into three categories in terms of ionic mobility in the oxide; immobile, outwardly mobile, and inwardly mobile ions. Immobility of electrolyte species can be attributed to effectively no charge in the film (compensated or low mobility). Therefore, the thickness of the outer oxide film is strongly influenced by the directionality of the electrolyte species.

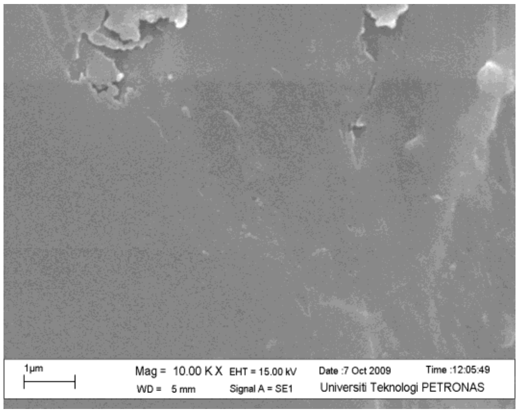
On the other hand, since the thickness in the porous aluminum oxide film is time-dependent, much thicker films than those in barrier-type films can be obtained. Anodizing time, current density and electrolytes are important parameters in determining the film thickness of porous aluminum oxide. For instance, thick, compact, and hard porous films are formed at low temperatures

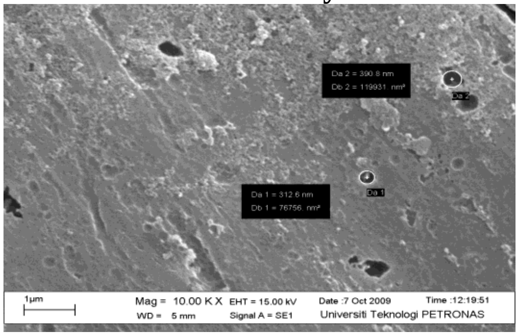
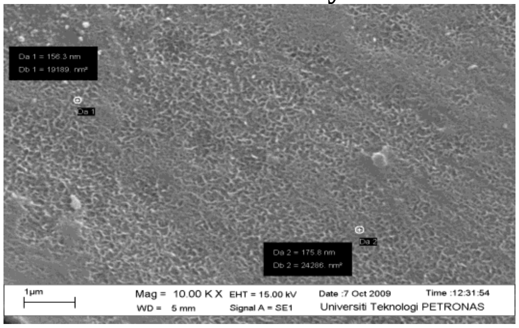
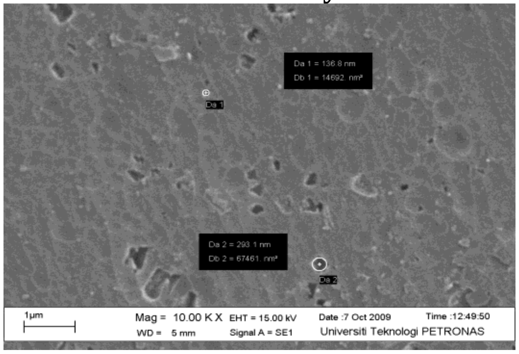
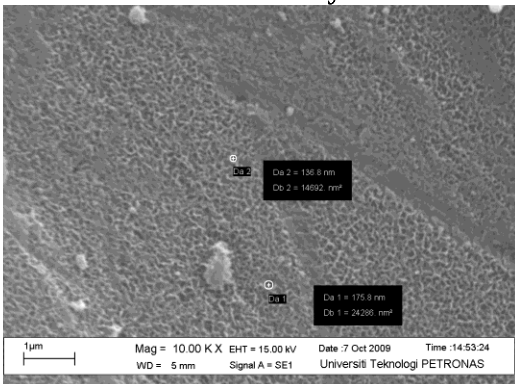
(0 < T < 5, so-called hard anodizing conditions), whereas thin, soft, and non-protective films are produced at high temperature and (60 < T < 75, so-called soft anodizing conditions). As the temperature increases, the corresponding current density also increases. In this analysis, room temperature has been applied which is 27^{o}C.

In typical commercial aluminum anodization processes, the aluminum oxide is grown down into the surface and out from the surface by equal amounts. So anodizing will increase the part dimensions on each surface by half of the oxide thickness. For example a coating that is (2 μm) thick, will increase the part dimensions by (1 μm) per surface. If the part is anodized on all sides, then all linear dimensions will increase by the oxide thickness.

This does not mean that a higher current density increases the film thickness since the rate of complex dissolution at the electrolyte/oxide interface increases, too. If the temperature is too high so that the rate of dissolution is faster than that of oxide formation, the film even vanishes; resulting in electropolishing of aluminum .The thickness of the thin layer at the bottom of the porous structure is only dependent on the anodizing voltage, regardless of anodizing time. Additive composition of solution need to be include in the chemical to prevent the aluminum for being corroded.

Table 8: Surface features of anodized aluminium

No.	Sample	Pores diameter (nm)	Pores Area (nm^2)
1.	**Pure aluminium**  	0	0

No.	Sample	Pores diameter (nm)	Pores Area (nm^2)
2.	**12V Non-dye**  	351.7	98343.5
3.	**12V With dye**  	166.05	21737.5
4.	**20V Non-dye**  	214.95	41076.5
5.	**20V With dye**  	156.3	19489

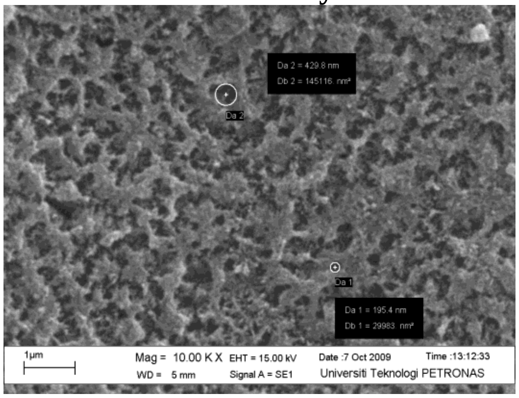
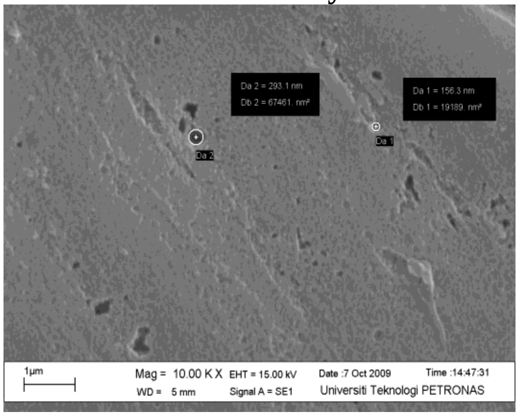
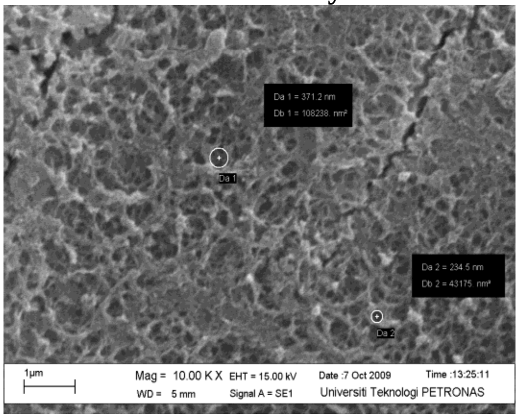
No.	Sample	Pores diameter (nm)	Pores Area (nm^2)
6.	**22V Non-dye** 	312.6	87549.5
7.	**22V With dye** 	224.7	43 325
8.	**24V With dye** 	302.85	26 9706.5

Figure 4.5 shows the effect of electrical parameter applied towards the dimension of pore space formed:

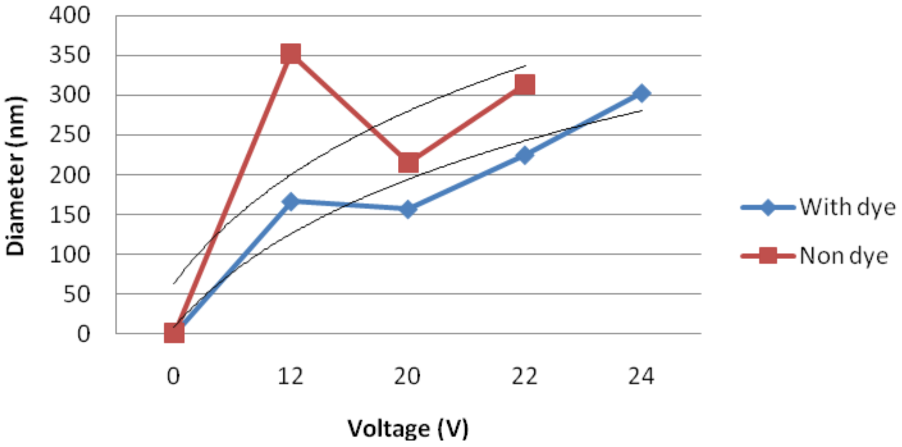


Figure 4.5: Pore diameter (nm) VS voltage (V) graph

Figure 4.5 shows that the increment of diameter of pore space after increasing the amount of voltage on the process. There is a little decrement of pore space diameter when the voltage reached 20V and continues to raise back. Since some pores begin to stop growing due to competition among the pores, the current decreases again. In this stage, pores grow in a stable manner. However, it is very often observed that during the stable pore growth, the current density continues to decrease slightly. This is due to diffusion limits in the long pore channels.

There are also other factors that contribute to the decrement of the diameter of pore space when 20V applied. The measurement of the diameter was taken randomly in not specified space. Therefore, some error may occur when author misleading to read the wrong position of pore space.

Furthermore, cell and pore dimensions depend on bath composition, temperature, and voltage, but the end result always is an extremely high density of fine pores. The cell diameter is in the range 50-350 nm and the pore diameter is typically 1/3 to 1/2 of the cell diameter. The cell population density is from approximately 10 to more than 100 per µm2. The rates of penetration of the dye are suitable for 20V-22V anodization process because there are no cracks or defects on the surface of aluminum.

On the other hand, too high voltage applied will result on the roughness of the specimen's surface. Table 8 above shows that when the 24V of voltage was applied to the specimen, the pore spaces dimension increase while there are disadvantages on the surface finish. Crack happen if the thickness of oxide are too much and getting higher. The differences of the thermal expansion between oxide layer and the aluminum also a factor of crack occurs. Anodized coatings have a much lower thermal conductivity and coefficient of linear expansion than aluminum.

From the result obtained above, author proposed a mechanical stress model to explain the formation of pore arrays by considering the following factors;

1. The oxidation takes place at the entire metal/oxide interface mainly by the migration of oxygen containing ions (O^{2-} or OH^{-}) from the electrolyte.
2. The dissolution and thinning of the oxide layer is mainly due to the hydration reaction of the formed oxide layer.
3. Pores grow perpendicular to the surface when the field-enhanced dissolution at the electrolyte/ oxide interface is equilibrated with oxide growth at the oxide/metal interface.
4. The formed alumina is assumed to be Al_2O_3. The volume of the anodized alumina expands greater than the original volume of metal.
5. This volume expansion leads to compressive stress during the oxide formation in the oxide/metal interface. The expansion in the vertical direction pushes the pore walls upwards.

According to their studies, the degree of the volume expansion of aluminum varies with the applied potential and determines either the formation of self-ordered pores or the formation of disordered pores.

4.3 FOURIER TRANSFORMED INFRARED (FTIR) SPECTROSCOPY

FTIR Analysis is a failure analysis technique that provides information about the chemical bonding or molecular structure of materials, whether organic or inorganic. It is used in failure analysis to identify unknown materials present in a specimen, and is usually conducted to complement EDX analysis.

The technique works on the fact that bonds and groups of bonds vibrate at characteristic frequencies. A molecule that is exposed to infrared rays absorbs infrared energy at frequencies which are characteristic to that molecule. During FTIR analysis, a spot on the specimen is subjected to a modulated IR beam. The specimen's transmittance and reflectance of the infrared rays at different frequencies is translated into an IR absorption plot consisting of reverse peaks. The resulting FTIR spectral pattern is then analyzed and matched with known signatures of identified materials in the FTIR library.

Unlike SEM inspection or EDX analysis, FTIR spectroscopy does not require a vacuum, since neither oxygen nor nitrogen absorbs infrared rays. When the library of FTIR spectral patterns does not provide an acceptable match, individual peaks in the FTIR plot may be used to yield partial information about the specimen. [5]

Figure 4.6: SHIMADZU FTIR8400s spectrometer machine

4.3.1 Objective

- To study the effect of different electrical parameter applied to the sample with association of dye and how it includes into the aluminium oxide.

4.3.2 Sample preparation

Infrared spectra may be obtained for gases, liquids or solids. Solids are usually examined as a mull, as a pressed disk or as a deposited glassy film. The pellet technique depends on the fact that dry, powdered potassium bromide can be compacted under pressure to form transparent disks. The sample (0.5-0.1mg) is intimately mixed with approximately 100mg of dry, powdered KBr. Mixing can be affected by through grinding in a smooth agate mortar or more efficiently with a small vibrating ball mill, or bye lyophilization. The mixture is pressed with special dies under a pressure of 10000 – 15000 psi into a transparent disk.

The quality of the spectrum is depends on the intimacy of mixing and reduction of suspended particles to 2µm or less. Microdisks, 0.5 -1.5 mm in diameter, can be used with a beam condenser. The microdisk technique permits examination of samples as small as 1µg.Bands near 3448 and 1639 cm^{-1} resulting from moisture, frequently appear in spectra obtained by the press disk technique.

The use of KBr disks or pellets has often been avoided because of the demanding task of making good pellets. Such KBr techniques can be less formidable through the Mini-Press, which affords a simple procedure; the KBr-sample mixture is placed in the nut portion of the assembly with one bolt in place. The second bolt is introduced and pressure is applied by tightening the bolts. Removal of the bolts leaves a pellet in the nut that now serves as a cell.

Figure 4.7: Sample mixed with KBr (left) and compacted to form the transparent disk (right)

4.3.3 Data gathering and analysis

There are no rigid rules for interpreting an IR spectrum. Certain requirements, however a must be met before an attempt is made to interpret a spectrum.

1. The spectrum must be adequately resolved and of adequate intensity.
2. The spectrum should be that of a reasonably pure compound.
3. The spectrophotometer should be calibrated so that the bands are observed at their proper frequencies or wavelengths. Proper calibration can be made with reliable standards such as polystyrene film.
4. The method of sample handling must be specified. If a solvent is employed, the solvent, concentration, and the cell thickness should be indicated.

A precise treatment of the vibrations of a complex molecule is not feasible; thus the IR spectrum must be interpreted from empirical comparison of spectra and extrapolation of studies of simpler molecules. Infrared absorption of organic molecules is summarized in the chart of characteristic group absorption. Many of the group absorptions vary over a wide range because the bands arise from complex interacting vibrations within the molecule.

Absorption bands may, however represent predominantly a single vibrational mode. Certain absorption bands, for example, those arising from the C-H, O-H, and C=O stretching modes, remain within fairly narrow regions of the spectrum. Important details of structure may be revealed by the exact position of an absorption band within these narrow regions. Shifts in absorption position and changes in molecular environment, may also suggest important structural details.

In this area of analysis, the main focus is to study the rate of penetration organic composition of dye use (carmoisine) onto the pores space formed after the anodizing process. Comparison have been made between sample with dye, sample without dye and dye only to get the better overview of the absorption rate of the dye on aluminum.

Figure 4.8 below show the three different spectrums linear in wavenumber in specified condition. Figure 4.8 (a) show the spectrum of sample when 20V and 22V voltage applied without dye and Figure 4.8 (b) is the spectrum after the dye has been applied. Then, these two spectrums will be compared with Figure 4.8 (c) which is the dye only spectrum. Result of this comparison will be utilize into the final step which is to know the free group of the dye that is not absorb into the pores space.

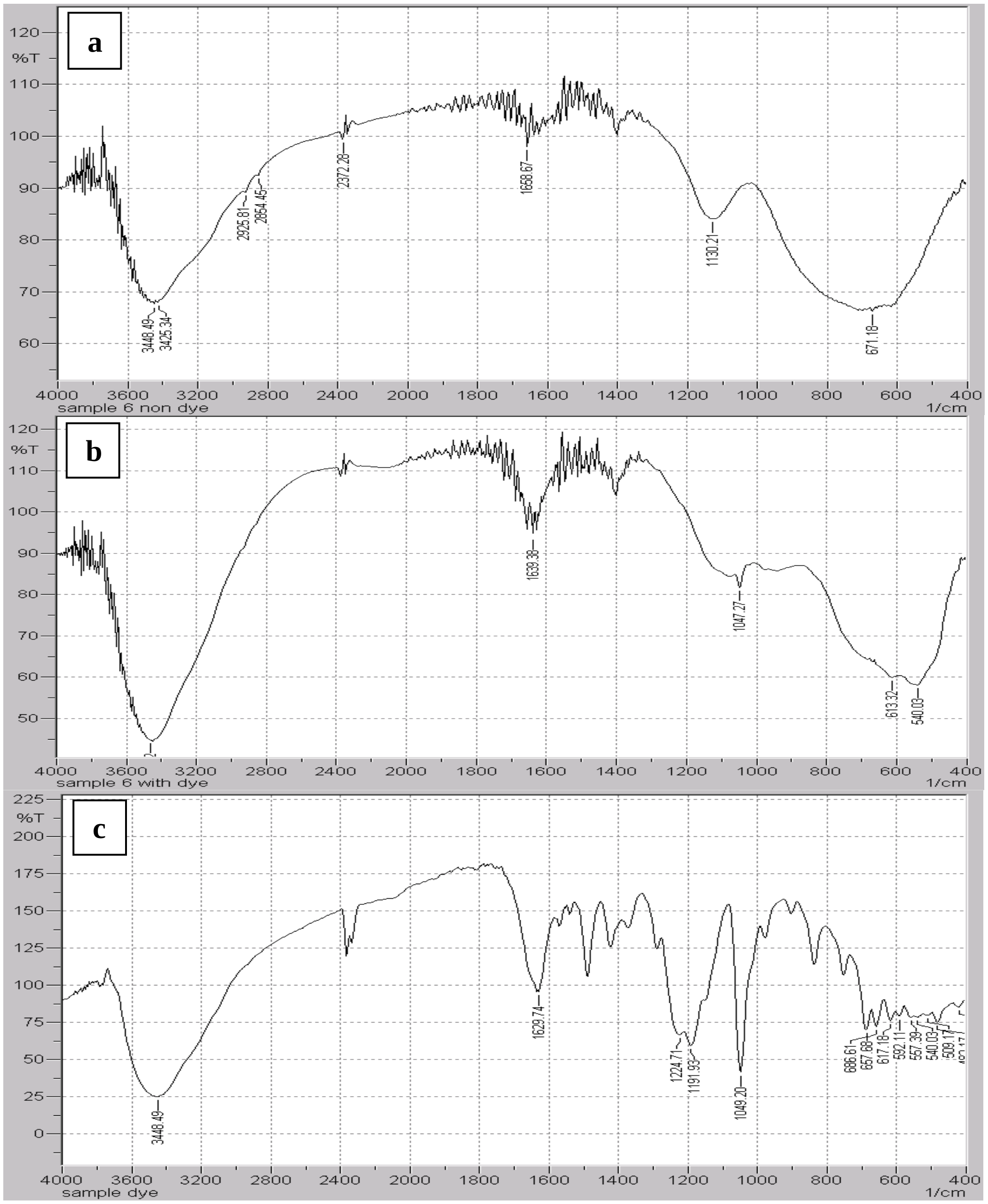


Figure 4.8: Spectrum for (a) anodized aluminium without dye (b) anodized aluminium with dye (c) sample of organic dye linear with wavenumber (cm^{-1}) for 20V

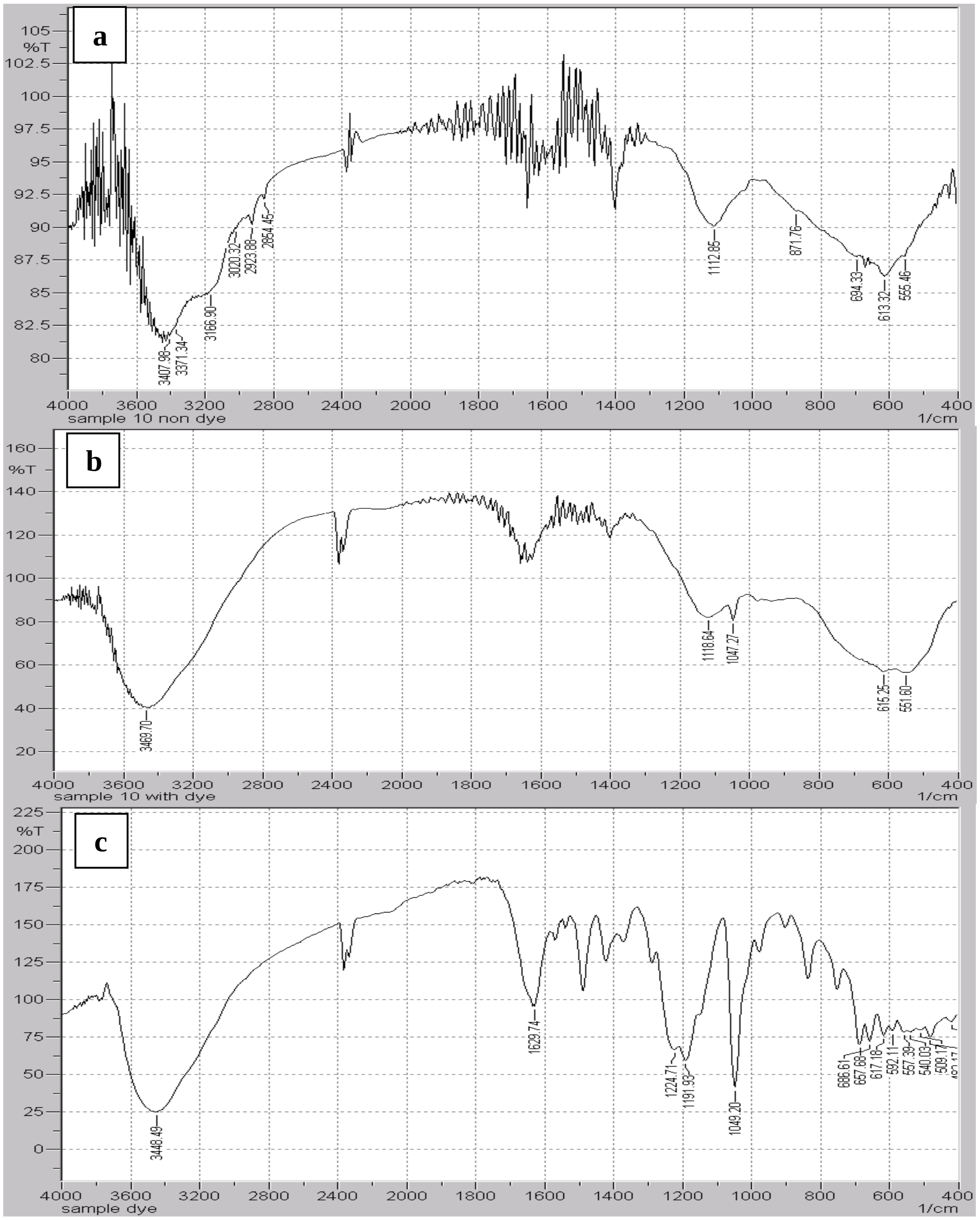


Figure 4.9: Spectrum for (a) anodized aluminium without dye (b) anodized aluminium with dye (c) sample of organic dye linear with wavenumber (cm^{-1}) for 22V

Figure 4.8 and 4.9 shows that the different electrical parameter applied will result to the different intensity of the peak in the spectrum. The more intense the peak meaning that there are many organic groups is free from bonded with the aluminum oxide. Increasing of intensity means interaction is shorter between dye and the aluminum oxide layer.

This FTIR method works on the fact that bonds and groups of vibrate at a characteristic frequencies. The FTIR analysis was found to confirm the interaction between the organic dye (carmoisine) chemicals groups with the internal pores surface of the aluminum oxide layer. Spectrum from Figure 4.8 and 4.9 shows that sample with dye alone has the highest frequency compared with the sample with and without the organic dye. When the frequency is reducing, the bonded between atoms in the dye of the group are weaken.

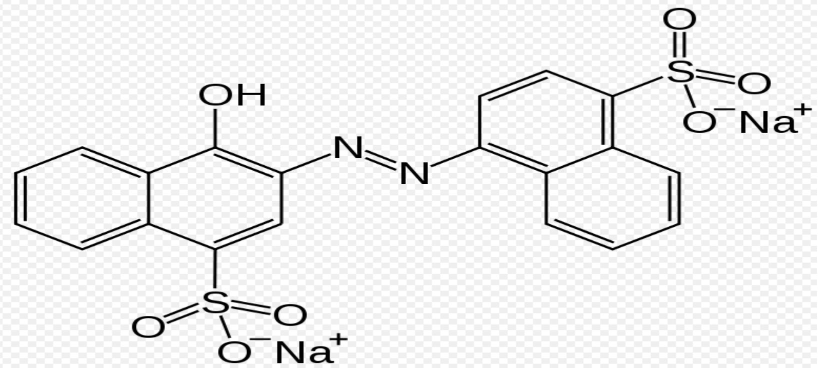


Figure 4.10: Azorubine or carmoisine is a synthetic red food dye from the azo dye group.

Table 9 below shows that the possible characteristic group for each wavenumbers based on the sample tested. The possible characteristic group is defined by referring the chemical structure of carmoisine (Figure 4.10). The defined possible characteristic groups is the group which is vibrate due to the freely in the aluminum oxide. Some of the groups are no longer vibrating because the groups need to attach into the aluminum oxide. Refer to appendix B for the table of characteristic of IR absorptions.

Table 9: Possible characteristic groups for specified wavenumbers.

No.	Sample	Wavenumbers	Possible Characteristic Groups
1.	20 V / 22V	1049.2	Alkenes, Mononuclear Aromatic
2.	20 V / 22V	1191.93	Mononuclear Aromatic, Lactones, Amines, Thiocarbonyl Group
3.	20 V / 22V	1419.2	Alkenes, Amines, Carbodimides, Thiocarbonyl Group
4.	20 V / 22V	1492.3	Mononuclear Aromatic, Carbodimides, Thiocarbonyl Group
5.	20 V / 22V	1629.74	Mononuclear Aromatic, Lactam, Amines
6.	20V	2311.1	Amines

7.	20V	3448.49	Alcohol And Phenol

The two important areas for a preliminary examination of a spectrum are the regions 4000-1300 and 900-650 cm^{-1}. The high frequency portion of the spectrum is called the functional group region. The characteristic stretching frequencies for important functional group such as OH, and C=O occur in this portion of the spectrum. The absence of absorption in the assigned ranges for the various functional groups can usually be used as evidence for the absence of absorption in the assigned ranges for the absence of such groups in the molecule.

Weak bands in the high frequency region, resulting from the fundamental absorption of functional groups, such as S-H and C=C, are extremely valuable in the determination of structure. Such weak bands would be of little value in the more complicated regions of the spectrum. Overtones and combination tones of lower frequency bands frequently appear in the high- frequency region of the spectrum. Strong skeletal bands for aromatics and heteroaromatics fall in the 1600-1300 cm^{-1} region of the spectrum.

The lack of strong absorption bands in the 900-650cm^{-1} region generally indicates the nonaromatic structure. Aromatic and heteroaromatic compounds display strong out-of-plane C-H bending and ring bending absorption bands in this region that can frequently be correlated with the substitution pattern. Broad moderately, intense absorption in the low-frequency region suggest the presence of carboxylic acid dimers, amines, or amides, all of which show out of plane bending in this region. If he region is extended to 1000cm$^{-1,}$ absorption bands characteristic of alkene structures are included.

The intermediate portion of the spectrum, 1300-900 cm^{-1}, is usually referred to as the "finger-print" region The absorption pattern in this region is frequently complex, with the bands originating in interacting vibration modes. This portion of the spectrum is extremely valuable in examine reference to the other regions. Any conclusion reached after examination of a particular band should be confirmed where possible by examination where possible by examinations of other portions of the spectrum. Similar compounds will give virtually identical spectra under normal conditions but fingerprint difference can be detected with an expanded vertical scale or with a very large sample.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

The main purpose of this project is basically to observe the effect of electrical parameters towards structure and properties of anodized aluminum plates. Since there are a lot of applications of anodized aluminum in daily life, it is important to study on the improvement of this material. Based on the study and analysis that have been done, the improvement can be achieved by several recommendations.

Increasing the voltage applied on the specimens as it can give better quality on the structure and properties in anodized aluminum plate. However, it should be reminded that there is a limit to apply the voltage on the specimens. Exceeding the optimum voltage can cause crack to happen on the surface of anodized aluminum due to the different coefficient of linear expansion between aluminum and aluminum oxide. Therefore, based on the experiments done, it is found that the optimum voltage that needs to be implementing during anodization process 21V. There are also have been identified that the saturated or stopping time for anodization process is within 50 – 60 minutes since the current density flows is constant.

The voltage required by various solutions may range from 1 to 300 V DC, although most fall in the range of 12 to 21 V. Higher voltages are typically required for thicker coatings formed in sulfuric. The anodizing current varies with the area of aluminum being anodized, and typically ranges from 0.3 to 3 amperes of current per square decimeter. Harder, thicker films tend to be produced by more dilute solutions at lower temperatures with higher voltages and currents. Conditions such as electrolyte concentration, acidity, solution temperature, and current must be controlled to allow the formation of a consistent oxide layer. Harder, thicker films tend to be produced by more dilute solutions at lower temperatures with higher voltages and currents.

The FTIR analysis was found to confirm the interaction between the organic dye (carmoisine) chemicals groups with the internal pores surface of the aluminum oxide layer. From FTIR analysis result, the possible characteristic groups of organic dye (carmoisine) that are freely vibrated on the aluminum oxide are defined. Some of the group are no longer vibrate because the group need to attach into the aluminum oxide layer.

Some improvements are identified during this study. During the anodization, temperature should be kept lower than room temperature to prevent the formed oxide structure from being dissolved in acidic electrolytes. A second reason to keep the temperature as low as possible is to avoid a local heating at the bottom of the pores during the course of anodization (specially, in the case of anodization at a high potential). The local heat causes an inhomogeneous electric field distribution at the bottom, leading to local electrical breakdown of the oxide. In fact, cracks and bursts of the oxide film are generated if porous alumina is formed without temperature controlling. If the temperature is too low (just below zero degree) and diluted electrolytes are used, the electrolyte may freeze. In addition, the speed of the growth of porous alumina is affected by the temperature. The lower the temperature, the lower is the growth rate.

The type and the concentration of the electrolyte for a given potential has to be selected properly to obtain self-ordered pore growth. In other words, the choice of the type of electrolyte is restricted. Usually, the anodization of aluminum is carried out in sulfuric acid in low potential ranges (5 - 40 V), oxalic acid is used for medium potential ranges (30-_ 120 V) and phosphoric acid for high potential ranges (80-200 V). This restriction is due to the conductivity and pH value of the electrolyte. For example, if aluminum is anodized in sulfuric acid at a high potential (note that sulfuric acid has a very high conductivity), breakdown of the oxide layer takes place very often. In addition, the pH-value of the electrolyte determines the size of the pores. The lower the pH value, the lower the potential threshold for field-enhanced dissolution at the pore tip. This leads to a smaller size of the pores. Therefore, large pore diameters are formed by using the phosphoric acid and small pore diameter are obtained by using sulfuric acid.

From the conclusion and recommendation above, the study on the effect of electrical parameters towards structure and properties of anodized aluminum plates will achieve the better result in improving the effect of electrical parameter towards structure and properties of anodized aluminum plates.

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APPENDICES

APPENDIX A

Laboratory Procedure

Title: Effect of electrical parameters towards structure and properties of anodized aluminium plates

Materials:

1. Aluminium foil (thickest)
2. Aluminium Strip

Apparatus:

1. 12 V battery / DC Adapter
2. 100 Ω potentiometer
3. Ampere meter (1A)
4. Voltmeter (15 V)
5. Crocodile Clips
6. Beaker

Chemical Solutions:

1. Acid Sulphuric (H_2SO_4) 2M - 250cc
2. Acid Nitric (HNO_3) 1M - 250cc
3. Sodium Hydroxide (NaOH) 1.5 M - 250cc
4. Acetone - 250cc
5. Acetic Acid (HOAc) - 2cc
6. Fabric Dye (red colour)

Procedure:

Part 1(Anodizing Process)

1. Clean an aluminium strip (which is the object to be anodized) by mechanically scrubbing it with steel wool then rinsing it with acetone. Once it clean, do not handle the rod with bare hands.
2. Dip in acetone for 10 seconds and dry the aluminium strip.
3. Hold the top portion and dip half bottom in NaOH for 1 min and after that into HNO_3 for 5 seconds to remove heavy oxide s, oil and grease.
4. Rinse with deionised water and then weight the strip before the electrolysis process.
5. Take a 20cm x 25 cm piece of household aluminium foil, fold it to 10cm x 25 cm, roll it into a cylinder and insert into a 400 ml beaker so as to line the vertical part of the inside beaker.
6. Put the aluminium strip into the circuit.
7. Before adding acid, and before turning on the power supply in the next few steps, take a few minutes to arrange the foil in the beaker so that it is very near the outside and has an alligator clip connected to the negative (-) side of the power supply. The wire connecting this should be supported by a ring stand and clamp. Also attached the strip to positive (+) electrode on the power supply using a second alligator clip and wire supported by a ring stand and clamp.
8. Turning on the power supply and adjust the potentiometer with constant current density 10, 15, and 20 mA cm^{-2}
9. The process will be conducting for different time for every specimen of aluminium strip. 15, 30 and 60 minutes.

Area: 3cm x 3cm x 2 sided = 18 cm^2

Current = current density x area

$= 10 \text{ mA cm}^{-2} \times 18 \text{ cm}^2$

$= 180 \text{ mA}$

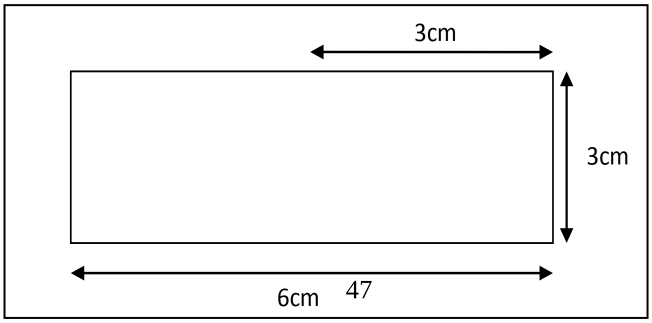


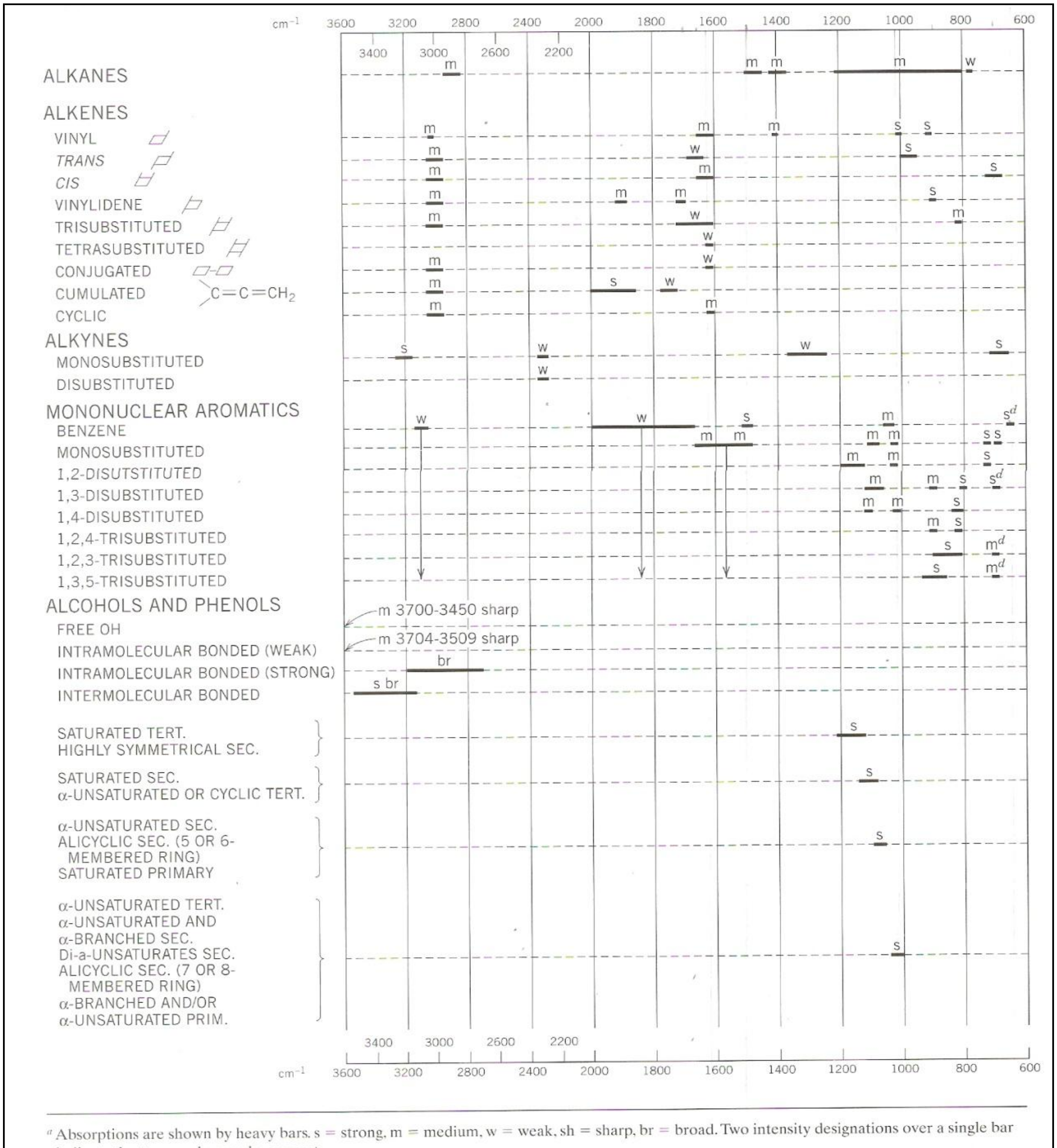
Figure A: Aluminium strip dimension

Part 2 (Sealing and Testing)

1. Remove the strip after all after all the part 1 procedure complete.
2. Rinse with deionised water and weight again the aluminium strip.
3. Dip into dye solution for 30 minutes.
4. Rinse the aluminium strip with water again.
5. Dip in boiling water for 2 minutes to seal the pores spaces.
6. Dry out the aluminium strip and read the weight it again
7. Apply several testing to identify the change of properties of anodized aluminium.

APPENDIX B

Characteristic Group Absorptions[14]



[a] Absorptions are shown by heavy bars. s = strong, m = medium, w = weak, sh = sharp, br = broad. Two intensity designations over a single bar indicate that two peaks may be present.

[b] May be absent.

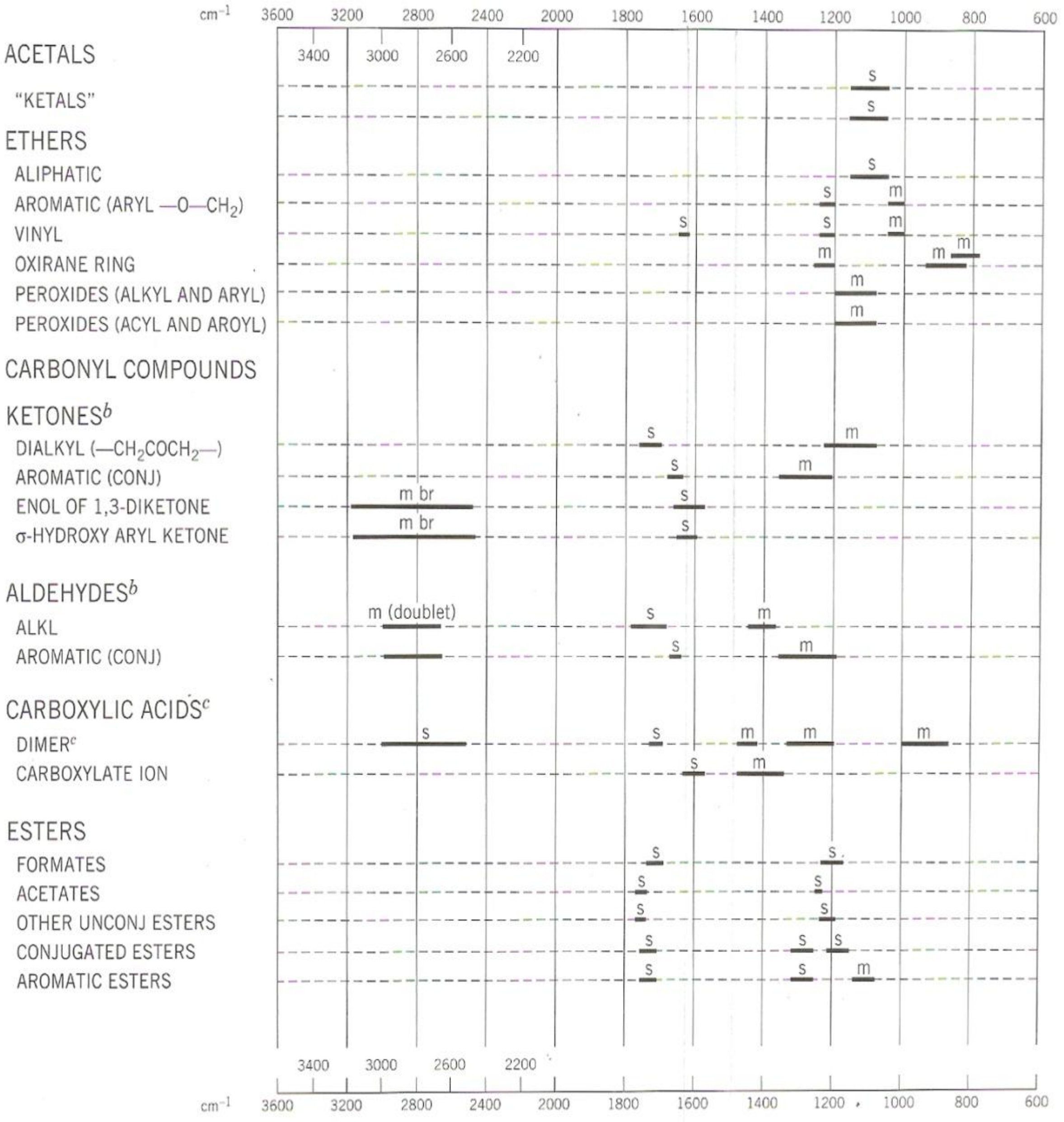
[c] Frequently a doublet.

[d] Ring bending bands.

Source: Robert M. Silverstein, Francis X. Webster, David J. Kiemle. 2005. *Spectometric Identification of Organic Compounds. Seventh Edition.* John Wiley & Sons, Inc.

APPENDIX B (Continued)

Characteristic Group Absorptions[14]



[a] Three bands, sometimes a fourth for ketals, and a fifth band for acetals.

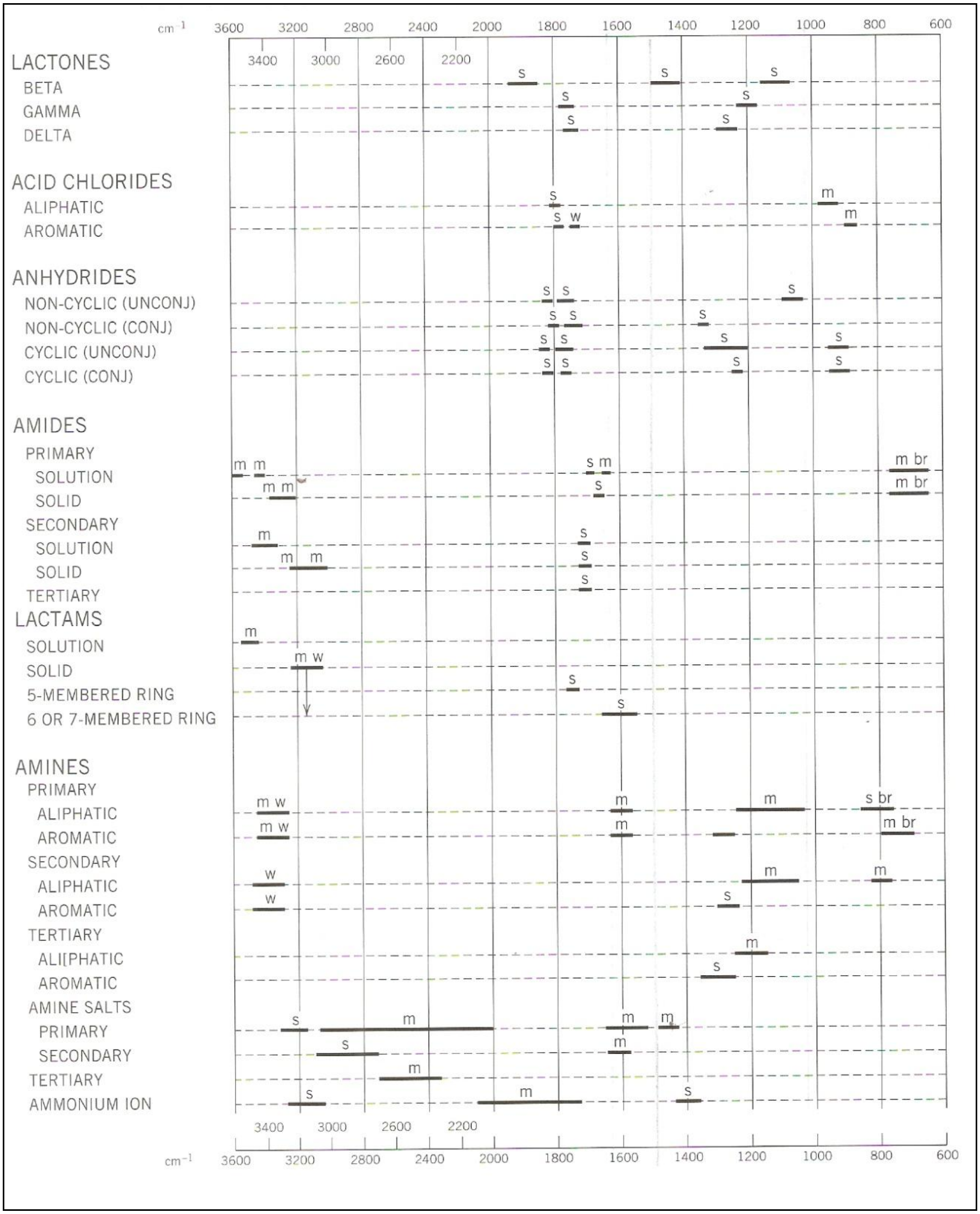
[b] Conjugated aliphatic examples show C=O stretch at virtually the same position as aromatic structures.

[c] Conjugated examples show C=O stretch at lower wavenumbers (1710–1680 cm⁻¹). The O—H stretch (3300–2600 cm⁻¹) is very broad.

Source: Robert M. Silverstein, Francis X. Webster, David J. Kiemle. 2005. *Spectometric Identification of Organic Compounds. Seventh Edition.* John Wiley & Sons, Inc.

APPENDIX B (Continued)

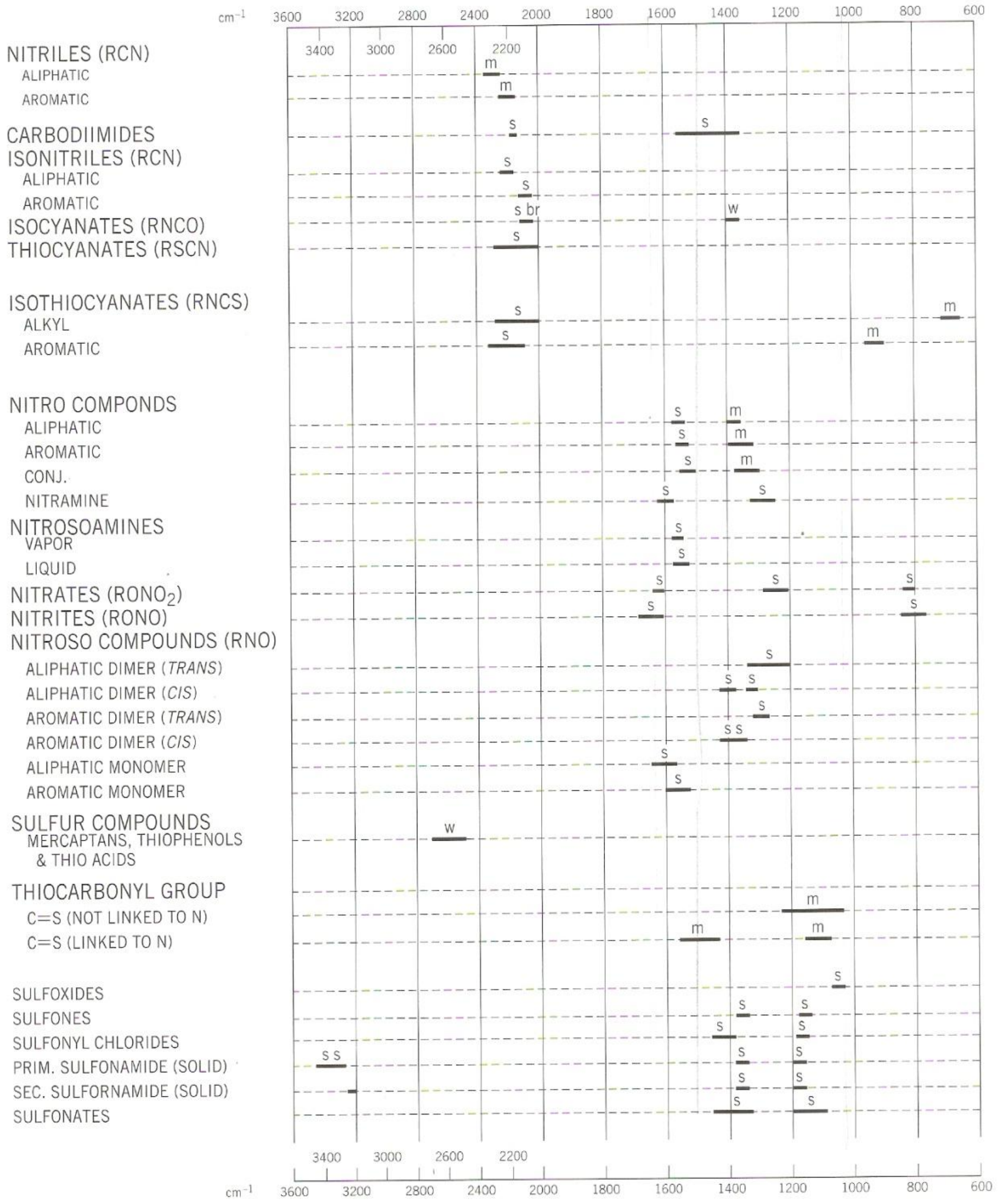
Characteristic Group Absorptions[14]



Source: Robert M. Silverstein, Francis X. Webster, David J. Kiemle. 2005. *Spectometric Identification of Organic Compounds. Seventh Edition.* John Wiley & Sons, Inc.

APPENDIX B (Continued)

Characteristic Group Absorptions[14]



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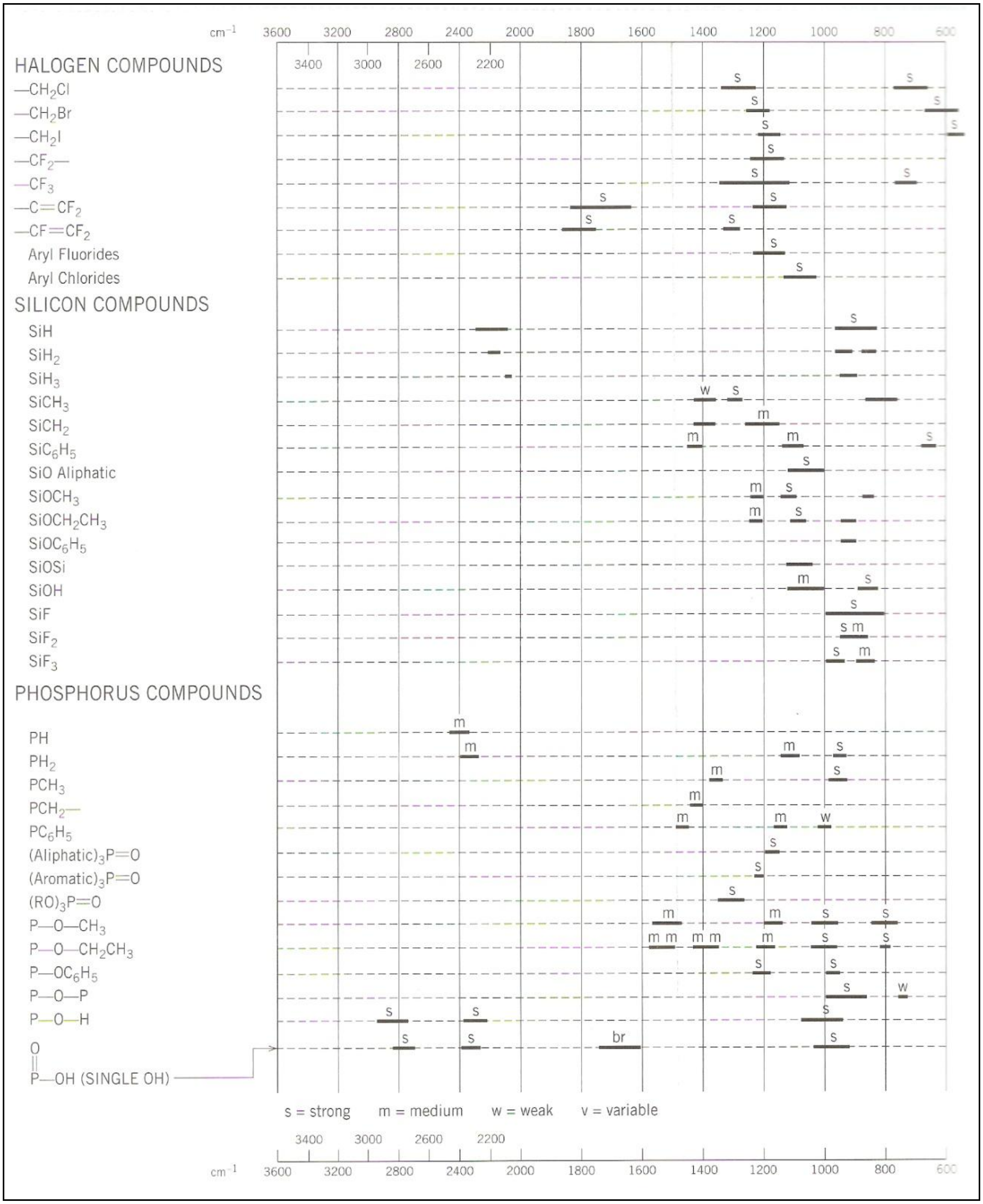
APPENDIX B (Continued)

Characteristic Group Absorptions[14]



Source: Robert M. Silverstein, Francis X. Webster, David J. Kiemle. 2005. *Spectometric Identification of Organic Compounds. Seventh Edition.* John Wiley & Sons, Inc.