# DEVELOPMENT OF GLASS COATING USING LIQUID EPOXIDISED NATURAL RUBBER

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**JUDUL : DEVELOPMENT OF GLASS COATING USING LIQUID EPOXIDISED NATURAL RUBBER**

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Signature:
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Date: 01 JANUARY 2012

# DEVELOPMENT OF GLASS COATING USING LIQUID EPOXIDISED NATURAL RUBBER

KARTHINI A/P KANESTION

A thesis submitted in fulfillment of the
requirements for the award of the degree of
Bachelor of Chemical Engineering

FACULTY OF CHEMICAL & NATURAL RESOURCES ENGINEERING
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JANUARY 2012

## STUDENT'S DECLARATION

I hereby declare that this thesis entitled **"Development of Glass Coating Using Liquid Epoxidised Natural Rubber"** is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree."

Signature
Name: KARTHINI A/P KANESTION
ID Number: KA08088
Date: 01 JANUARY 2012

*To my beloved family*
*especially my late mother, Paramesvari K.S Maniam. She taught me to preserve and prepared me to face challenges with faith and humanity. She was a constant source of inspiration to my life. Even though she is not here with me to give strength and support I always feel her presence, urging me to strive to achieve my goals in life. You are forever in my heart "Amma". God bless her soul.*

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## ABSTRACT

The purpose of this study on glass coating using ENR is to study the development of glass coating. The ENRs are selected as their moderate cost, environmentally friendly, great adhesion properties, and easily produced thru a simple and quite rapid route using peroxy acid onto NR (Natural Rubber). The experiment including determine drying time of the LENR (Liquid Epoxidized Natural Rubber) coating at various ratio 1:15, 2:47, 2.5:50, 3:54, 4:60, 5:72 and compare drying at ambient temperature, at $40^0$C and $50^0$C and find out the physical properties of 25 mol% epoxidation LENR (Liquid Epoxidized Natural Rubber) coating viscosity, pH and density at various ratio. The adhesion test was also performed to determine how well LENR coating is bonded to the glass. In this research, the experiment was completed by determining the pH, viscosity and density of samples using Gas Pycnometer, pH meter and Viscometer. For the adhesion test a peel test method was conducted revealing the quality of coating. The experimental running proved that the most convenient sample coating compare from the entire composition ratio, sample E, ratio 4:60 is the coating with average viscosity 631cP and pH 8.55 which act as alkaline coating that possibly protect the glass and have less environment impact. The density of sample E coating is 0.895g/cm3. Furthermore, the cure time of the coating is 360s at $30^0$C, 240s at $40^0$C and 180s $50^0$C that higher drying temperature result in short dry time. Also the hydrophobic property is an advantage for coating to protect glass. The result of this study is helpful to provide specific guidance in selection for developing high quality glass coating using LENR.

## ABSTRAK

Tujuan kajian ini pada lapisan kaca menggunakan ENR adalah untuk mengkaji penambahbaikan lapisan kaca. Getah ENR telah dipilih kerana kos sederhana, mesra alam, sifat lekatan besar, dan mudah dihasilkan melalui kaedah yang mudah dengan menggunakan asid peroksi pada NR (Getah Asli). Eksperimen yang dilaksanakan termasuk menentukan masa pengeringan lapisan LENR (Cecair Epoxidized Getah Asli) pada pelbagai nisbah 1:15, 2:47, 2.5:50, 3:54, 4:60, 5:72 dan perbandingkan pengeringan pada suhu bilik, pada $40^0$C dan $50^0$C dan mengetahui sifat-sifat fizikal 25 mol peratus pengepoksidaan LENR (Cecair Epoxidized Getah Asli) kelikatan salutan, pH dan ketumpatan pada nisbah berbeza. Ujian rekatan telah juga dilakukan untuk menentukan kekuatan salutan LENR terikat pada kaca. Dalam kajian ini, eksperimen tersebut telah siap dengan menentukan pH, kelikatan dan ketumpatan sampel yang menggunakan alat Piknometer Gas, meter pH dan Meter Kelikatan. Untuk ujian rekatan kaedah mengupas telah dijalankan untuk mendedahkan kualiti salutan. Perjalanan eksperimen yang membuktikan bahawa sampel lapisan yang paling mudah bandingkan dari nisbah komposisi keseluruhan, sampel E, nisbah 4:60 lapisan dengan 631cP kelikatan purata dan pH 8,55 yang bertindak sebagai salutan alkali yang mungkin melindungi kaca dan mempunyai persekitaran kesan yang kurang. Yang ketumpatan salutan E sampel adalah 0.895g/cm3. Tambahan pula, masa pengeringan salutan 360-an pada 30 $^0$ C, 240s pada 40 $^0$ C dan 180s 50 $^0$ C yang lebih tinggi suhu pengeringan hasil dalam masa yang pendek. Sifat hidrofobik juga adalah satu kelebihan bagi salutan untuk melindungi kaca dari pembiakan bakteria dan lain-lain lagi. Hasil kajian ini adalah membantu untuk menyediakan panduan khas dalam pemilihan bagi salutan lapisan kaca yang berkualiti tinggi menggunakan LENR.

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| ENR | Epoxidized Natural Rubber |
| LENR | Liquid Epoxidized Natural Rubber |
| NR | Natural Rubber |
| MRB | Malaysian Rubber Board |
| ABR | Acrylonitrile Butadiene Rubber |
| ASTM | American Society for Testing and Materials |

# CHAPTER 1

# INTRODUCTION

## 1.1 BACKGROUND OF STUDY

In glass industry, many challenges were faced due to producing a quality glass according to the customers preferance. According to the glass container industry, since the 1980s the glass container market has suffered a steady loss of market share to alternate plastic and can packaging. Imports of glass containers in 2001 total 27.9 million gross, compared with 30.6 million gross in 2000; and glass container exports total 11 million gross, compared with 8.8 million gross in 2000 (Highbeam Bussiness,2011). This prove that almost all kind of glass surface without coating actually experience breakage normally that is both a mess and a safety hazard. For instance, glass containers that bang into each other during filling, shipment, or in retail stores have lesser toughness without coatings. Low-maintenance glass coatings have been around for decades. Protective coatings have become more popular in the residential and commercial glass market during the last several years. The coatings are only now reaching the point of economic and technical growth.

### 1.1.2 Background of Epoxidized Natural Rubber

Currently Malaysian Rubber Board produces ENR which also called as Liquid Epoxidized Natural Rubber with the trade name Epoxyprene. There are two grades available, that is ENR-25 and ENR-50, with 25, and 50 mol % epoxidation, respectively. However the market and applications for ENR found to be limited. Thus efforts are being made to broaden the horizons of the usage and application of this rubber, especially in advanced engineering field. Coating with ENR is the easiest and the cheapest way to modify the strength of glass and all together reduces the material cost. Furthermore, the presence of oxirane group in ENR was found to be effective in causing specific interaction with a second polymer (Kallitsis and Kalfoglou, 1989).

The purpose of this study on glass coating using ENR is to study the development of glass in other words to improve the strength of glass. These ENRs are chosen as their moderate cost, environmentally friendly, great adhesion properties, and easily gained by means of the production line via a simple and quite rapid route using peroxy acid onto NR (Natural Rubber). A slightly changes in chemical structure in the presence of oxiranes making possible for ENR to possess other properties such as heat resistance, air permeability resistance, and stability due to chain re-arrangement.

## 1.2 PROBLEM STATEMENT

One of the marketing advantages the glass industry uses to sell its product is the perception by consumers that glass is a higher quality package than plastic. With glass's perceived quality and beauty, however, there is several characteristics that are viewed as negatives by bottlers, retailers, and the public especially the breakability of glass. Therefore to overcome the problems this research is conduct enhance the strength of glass. Besides glass experience breakage normally that is both a mess and a safety hazard. Thus handling the glass will reduce the unwanted accident to be occurred.

Recently, the Malaysian Rubber Board (MRB) officially annouced the price of the rubber rising, therefore the price of ENR also increasing. ENR is easy to be obtain yet it has disadvantage in its property toward the glass. It is not suitablenfor certain application since it is not transparent once ENR turn out to be thin film on glass. Hence,it is not suitable for optical glass, window glass and etc.

## 1.3 OBJECTIVES

The main objective of this research is to improve the glass coating using liquid epoxidized natural rubber.

## 1.4 SCOPE OF RESEARCH

In the current study, several significant parameters have been investigated. The detail of the scope is as below:

i. To investigate drying time of the LENR (Liquid Epoxidized Natural Rubber) coating at various ratio 1:15, 2:47, 2.5:50, 3:54, 4:60, 5:72 and compare drying at ambient temperature, at $40^{0}$C and 50 $^{0}$C.
ii. To find out physical properties of 25 mol% epoxidation LENR (Liquid Epoxidized Natural Rubber) coating viscosity, pH and density at various ratio.
iii. To determine the adhesion of a LENR coating on glass surface, a peel off test is performed.

## 1.5 RATIONALE & SIGNIFICANCE

The rationale of this research project is to provide empirical evidence to find out the finest quality LENR coating material on glass surface at various ratios 1:15, 2:47, 2.5:50, 3:54, 4:60, 5:72 at levels of 25 mol% epoxidation. The results of this research would signify the identification of an alternative coating between the various ratios to perform a safe and protective layer on the glass.

One of the important products of chemical modification of natural rubber is the epoxidized natural rubber, ENR. Through chemical modification, natural rubber properties would be enhanced. Hence, ENR has the potential to be further exploited for its usage as advanced materials in glass industry.

## 1.6 THESIS OUTLINE

The outline of this thesis is presented as a schematic form as displayed in Figure 1.1, and the brief description of each individual chapters is showed at the remainder of this chapter.

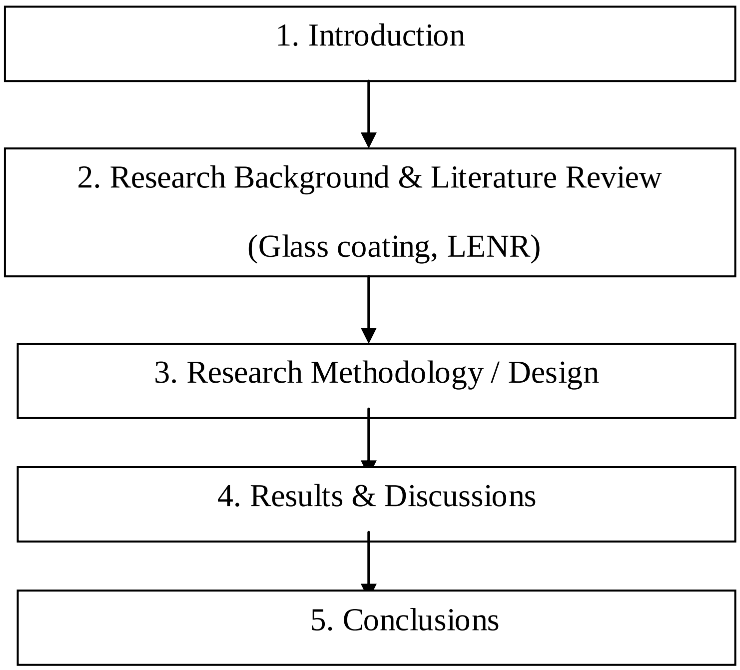


**Figure 1-1**: Road map for the thesis

**Chapter 2** describes literature review and research background of ENR including the related fields of glass coating. It also involves LENR's properties along with technological challenges involved in the glass coating industry.

**Chapter 3** is a detailed description of research methodology or design for this research that revealing about the various materials used and the methods of characterizing the samples and standards. Furthermore, it includes LENR quality analysis techniques which are the pH, viscosity and density including the drying time and adhesion property of several ratios at 25 mol% epoxidation.

**Chapter 4** is about the results and discussions of this research. The detail reports on experimental result along with the discussion of the quality coating material liquid epoxidized natural rubber on the glass surface.

**Chapter 5** delivers the conclusion of this research where the contribution of main findings and also the recommendations for future work.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 EPOXIDATION OF NATURAL RUBBER

Epoxidised natural rubber (ENR) is a derivative of natural rubber produced by chemical modification. It was not until the mid 1980s that pure samples of ENRs were prepared and their properties fully recorded (Gelling, 1999).For the most general natural rubber originated from the plant Hevea brasillensis. The main natural rubber producers in the world are Malaysia, Thailand and Indonesia. The major uses of a natural rubber are for production of tires, molded goods and mechanical parts while the lesser uses are the chemical derivatives of rubber. Enhanced properties of natural rubber for certain applications can be made via chemical modifications. This is possible because of the existence of double bonds (C=C) in the natural rubber polymer chain that perform as simple olefin. One of the important products of chemical modification of natural rubber is the epoxidized natural rubber, ENR. Reacting natural rubber with peroxy formic acid can produce ENR 46 (Figure 2.1). It was stated that 1-90% epoxidation of the natural rubber is possible, however only 3 types of ENR were considered as commercial standard. These are ENR-10, ENR-25 and ENR-50 where the integers designate 10, 25 and 50-mole % of epoxide included into the natural rubber chain respectively (C.H. Teoh, 2006). Through chemical alteration, natural rubber properties would be improved. Therefore ENR also display properties that are the same as a specialty elastomer such as decrease in air permeability, that is similar to a butyl rubber and increment in oil resistance, which is comparable to Acrylonitrile Butadiene Rubber (ABR) (C.H. Teoh, 2006) including Figure 2.2 shows the structure of Epoxidised Natural Rubber (ENR).

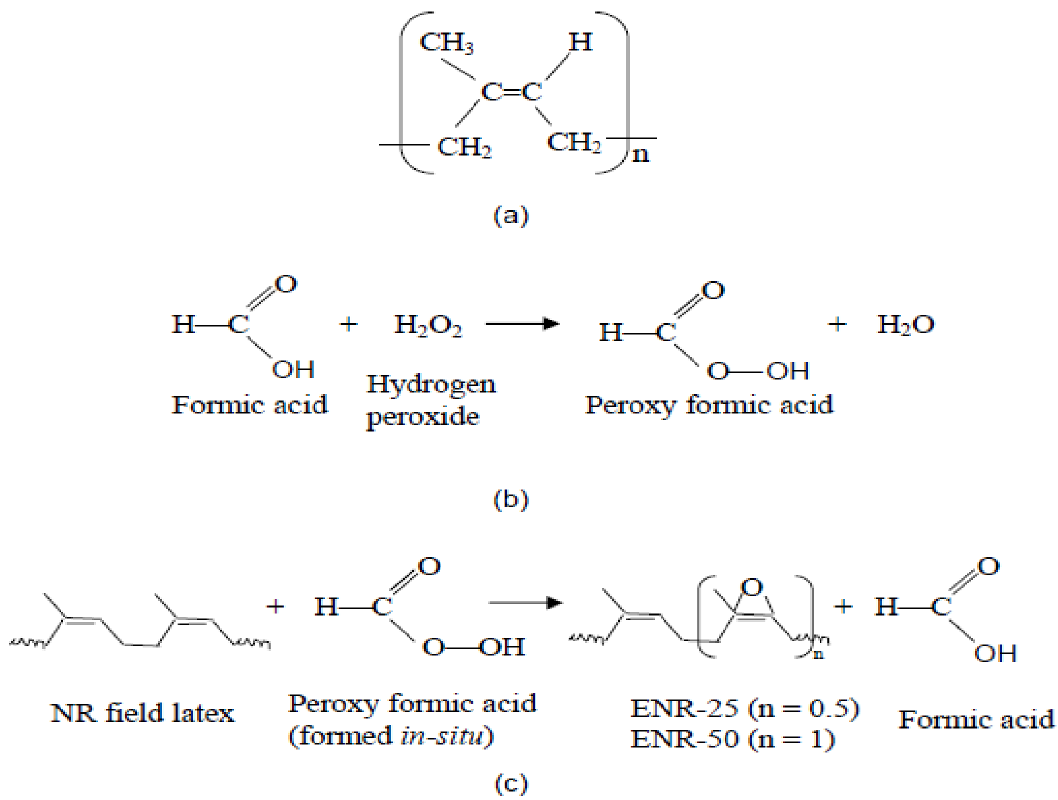


**Figure 2.1:** (a) Structure of natural rubber, cis-1, 4-polyisoprene, (b) formation of peroxy formic acid and (c) the production of ENR

Source: C.H. Teoh (2006)

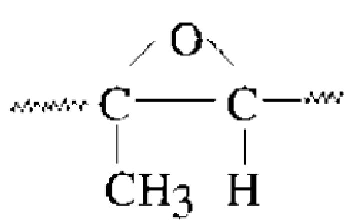

**Figure 2.2:** Structure of Epoxidised Natural Rubber (ENR)

Source: Ratnam et al.,(2001)

The mechanical properties of natural rubber (NR) are generally superior to those of synthetic rubbers. Moreover, NR cannot compete with the specialty synthetic elastomers with regards to such properties as gas permeability and oil resistance like mention above. The epoxidation reactions recognized for the chemical modification of NR, that guide to the development of epoxidised natural rubber (ENR) .These modified polymer have enhanced oil resistance and decrease gas permeability, at the same time as maintaining most of the properties of NR (M.Zurina,2007)

**Table 2.1**: Typical Properties of ENR (Ismail, 2004).

| Properties | ENR 10 | ENR 25 | ENR 50 |
|---|---|---|---|
| Glass transition temperature, $T_g$ (ºC) | -60 | -45 | -25 |
| Specific gravity | 0.94 | 0.97 | 1.03 |
| Mooney viscosity,$M_{L,1+4}$ (100ºC) | 90 | 110 | 140 |

## 2.2 APPLICATION OF EPOXIDIZED NATURAL RUBBER

Epoxidized Natural Rubber, ENR shows both rubber and special elastomer characteristics that can be modified for various usages. Some of the potential commercial uses of ENR with respect to its special characteristics are use for Tires, non-slip flooring, sports shoe soles, Hoses, seals, blow-out preventors, milking inflation, connector and tubing, as for silica and pigments reinforcements characteristic is used in cosmetics, color coding, adhesives, cover for PVC conveyor belt and finally as for the characteristic of gas permeability the bladders, inner tubes, and tire liners is used (C.H. Teoh, 2006). Besides the proposed commercial uses shown, ENR has the potential to be further exploited for its usage as advanced materials such as in blends, additives and fuel cells applications.

## 2.3 GLASS COATING

According to Oxford dictionary the definition of glass coating is a layer or film spread or paint over a surface for protection or decoration purpose. Coatings applied to glass surfaces are an essential part of manufacturing in all segments of the glass industry. Without coatings, not only would many glass products not have the properties that make them so widely used, they would be impossible to make.( Mark D. Allendorf,2001).

Coating is an essential method to enhance the property of the material especially in glass. The important role for coatings in strength is to improve mechanical barrier in term of modulus and abrasion resistance and water barrier compressive residual stress, flaw healing especially in cut edges. Glass is an essentially a strong material. Nevertheless, due to the presence of defects, practical strength of glass articles is significantly lower than the theoretic strength.

An effective method of strengthening glass is highly desirable for making stronger, safer, lighter, or cheaper glass products. Strengthening of glass is traditionally achieved using techniques like thermal tempering and ion exchange. Yet, both approaches create compressive stress on the glass surface for strengthening.( Mei Wen and S.W. Carson,2008, "A Study on the Strengthening Glass by Polymeric Coatings")

### 2.3.1 Significant of Glass Coating

This report also points to the significance of coatings in the glass industry. Without coatings, countless glass products would not have the properties that which no longer valuable, useful and many would also be impossible to make. Examples can be found throughout the industry. For instance, Because of its abrasiveness, glass fiber could not be formed into products such as fiberglass insulation and composites for automobiles without protective and lubricating coatings.

The studied increases in energy efficiency achieved by low-E and solar-control glass (a nearly twofold increase in the R value of a dual-pane window over uncoated glass) are due entirely too sophisticated application of multiple coatings. The high throughputs of today's container lines would not be possible without lubricious coatings; coatings also increase the burst strength of glass containers threefold. New glass products at the forefront of the industry, such as "smart windows" and flat-panel displays, depend on coatings to accomplish their functionality. The glass industry aspiration document, Glass: A Clear Vision for a Bright Future (Jan. 1996), identifies that the "development of innovative uses of glass is a target of the industry's future." This report also points to the importance of coatings in the glass industry, Examples of some of the many innovative glass products that use coatings are given in Table 2-3. The Glass Technology Roadmap (Sept. 1997), includes a partial list of industry-wide product categories essential to broadening the market for glass products. (Coatings on Glass Technology Roadmap Workshop,2000)

**Table 2.2:** Current products apply coatings and functions of the product. (Source: R. Gordon, Harvard University)

| **Current Products** | **Function** |
|---|---|
| Low-E window glass | Energy-conserving windows |
| Solar control + low-E glass | Windows in large buildings, hot climates |
| Photovoltaics | Solar electricity |
| Flat-panel displays | TV, computers |
| Electrochromic mirrors | Automatic rear-view mirrors in cars |
| Touch-panel controls | Appliances |
| Anti-reflection TV | picture framing |
| Anti-static | Copiers |
| Defogging Supermarket freezers | windows in vehicles |
| Anti-abrasion | Bar-code readers |
| UV protection | Reduced fading of fabrics and art work |

## 2.4 EFFECT OF COATING

There are `many factors lead to the production of perfect coating material such as the pH, thickness, temperature, viscosity of coating, and organic coating formulation itself and moisture of the coating. These effects are crucial in many industries concern on these qualities and durable of coating with the requirement of customer.

### 2.4.1 Formulation of Organic Coating

The preparation of the organic coating has effect upon on the chemistry of polymer chain formation and molecular weight. The crucial form of the polymer chain, its length, shape, and configuration determines the properties and physical characteristics of the coating, such as durability, hardness, and adhesion.

### 2.4.2 Coating Viscosity

Apart from that, the viscosity of the coating affects the coating especially during the transition from the wet to the cured state (dried) is particularly important. As the coating cures, its viscosity fluctuates and increases, and its mobility or flow decreases. If the epoxy is not properly formulated, flow of the coating into the substrate microstructure may be hampered, adversely affecting adhesion and producing a number of voids and holidays in the film. The low viscosity coating flows better is easier to roll or brush, saturates surface of glass quickly, and penetrates more evenly in fact drying period taken is short compare to more viscous coating. There are two methods of temporarily thinning epoxy. One is to heat the mixture and the other is to add solvent to the mix. The aim of both methods is to reduce the coating viscosity. (Gaynes and Norman, 1977)

### 2.4.3 Temperature Coating

Usually glass is exposed to ambient temperature. Thus, the capability of heat resistance of glass coating is crucial to protect the glass especially country that have weather changes need coating that resistance at neither extremely high nor low temperature; the coating will become soft and fluid or become harden and tear into piece, and even will be susceptible to deterioration. Therefore, coating material should be able to withstand the maximum temperature of glass, steel or plastic otherwise the coating will be failed.

### 2.4.4 Moisture of coating

Moisture is directly significant as the temperature of coating. Unless moisture is present, there is possible chance to have permanent loss of adhesion. The existence of high temperature only causes temporary loss of adhesion, but immediately after the organic coating cools, adhesion can be regained. Therefore, high temperatures will generally produce loss of adhesion when moisture is present. Equally, moisture alone produces loss of adhesion over time, but high temperatures help to accelerate the disbondment process ( Enrique Vaca-Cortés., Miguel A. Lorenzo., et al., 1998).

## 2.5 ADHESION

According to online oxford dictionary adhesion means the action or process of adhering to a surface or an object like the adhesion of the epoxy or other organic coating to the steel to avoid corrosion of steel. Adhesion strength between the coating and the substrate is a crucial factor in successful attaching and long-lasting stability of any coated implant. A varies range of methods is used to evaluate to adhesion of the coating (K.L. Mittal, 1976).

### 2.5.1 Tape Test

The most usually applied methods employed to assess adhesion strength of coating on substrate include tensile test (pull-out test) (D.M. Liu, Q. Yang and T. Troczynski, 2002) and scratch test (M.F. Hshieh, L.H. Pwerng and T.S. Chin, 2002), scratch test and tape test. A variation of peel adhesion test was carried out by McDonald et al. Known that the porous microstructure and the high surface roughness of these coatings peel test deems a better method the reason is the strongly influences such as glue infiltration which are expected in peel off test (P.A. Steinman, Y. Tardy and H.E. Hintermann, 1987).

These methods include the peel-off method by using adhesive tape for instance 3M Scotch brand tape, pressure sensitive tape and etc. are performed based on ASTM standard-American Society for Testing and Materials. This standard test method set up a procedure for determining whether the adhesion of a polymer coating to a glass, steel and plastic material is an adequate level.

In this study the tape used was 3M emulsion water based acrylic adhesive tape, the direct pull-off method have to do with ASTM D 3359 Standard Test Methods for Measuring Adhesion by Tape Test. Even though the lack of a fully satisfactory analytical model of its mechanics (J. Williams, 1996), the tape test is normally used to quantitatively assess the quality of adhesion of coatings to substrates.

## 2.6 DRYING TEST

The drying process of coatings and paints plays a vital role in substrate or in other word the coating material is crucial in protection of object (for instance; steel, plastic or glass) since many cases the object to protect is immobilized. The understanding of the drying processes provides the development of suitable drying systems process for each different application.

Based on the researchers named J.I. Amalvy, and C.A. Lasquibar a previous work has been reported on the application of drying period of coating in industry identified to be mixed with the petrol or any interferometry to the drying of paints with relatively long drying times. ( P.A. Facciaa, O.R. Pardinia, J.I. Amalvya,b, N. Capc, E.E. Grumelc, R. Arizagac, M. Trivic,2009).

# CHAPTER 3

# METHODOLOGY

## 3.1 INTRODUCTION

In this particular chapter, a detail of experiment framework will be presented, also included materials to be used, description of experimental adhesion test, drying test, density test, pH and viscosity analysis and also sequence of experimental procedures. The main purpose of this study is to determine the adhesion and physical properties of the LENR as a glass coating material on the glass surface. As well, the part of studies is also to identify the drying time of LENR at various ratio. Adhesion test is performed by conducting the tape test.to determines how well a coating is bonded to the glass. Commonly used measuring techniques are performed with tape test adhesion tester.

## 3.2 MATERIALS AND EQUIPMENT

The following chemicals will be engaged for the preparation of coater (LENR) and painting process;

i) Epoxidized Natural Rubber (ENR-25,25mol% epoxidation)
ii) Toluene ($C_7H_8$)
iii) Isopropyl alcohol, IPA ($C_3H_8O$)

Below table is the function of each component in the experiment setup

**Table 3.1**: Function of each component in the experiment setup.

| No. | Apparatus / Equipment | Function |
|---|---|---|
| 1 | Beaker | To hold and mix liquids. |
| 2 | Cylinder | To measure the volume of liquids. |
| 3 | Stirrer | To stir the solution constantly. |
| 6 | Rod glass | To mix the liquid and used for spreading liquids onto a surface |
| 9 | Timer | To control the drying time. |
| 10 | Glass plate | Act as a substrate to apply coating on its surface |
| 11 | Filter paper | Act as medium to inspect the drying time of a liquid |
| 13 | 3M emulsion water based acrylic adhesive tape | To peel-off the coater (adhesive tape) |
| 14 | Gas Pycnometer | To measure the density of a material |
| 15 | Viscometer | To measure the viscosity of liquid |
| 16 | pH meter | For measuring pH of liquid |
| 17 | Weighing scale | To determining the weight of an object |
| 18 | Paint brush | To paint the coating on glass surface |

### 3.2.1 Preparation of Liquid Epoxidized Natural Rubber (LENR)

In this study ENR 25 -25 mol% epoxidation were supplied by the Lembaga Getah Malaysia at Selangor. The liquid of ENR were produced by diluting with the toluene according to desired ratio and stir using the stirrer. In research, Liquid Epoxidized Natural Rubber (LENR) used to coat on the glass surface.

### 3.2.2 Preparation of Glass Plate

A plate glass size 10 x 15cm was purchased about 10 pieces at glass workshop Changloon, Kedah. The glass with a chemical composition typical for commercially available are made ($SiO_2$; CaO and MgO as main components). (C.Noemi, W.Katrinand R.Hannelore, Consolidation of paint on stained glass windows: Comparative study and new approaches, 2008).

## 3.3 METHOD OF EXPERIMENT PROCESS

First, samples of two commercially available LENR-25 glass plate topcoats were prepared with various ratios; at various ratios 1:15, 2:47, 2.5:50, 3:54, 4:60, 5:72. Then followed by, cleaning the glass thoroughly using Isopropyl alcohol, IPA before paint LENR on the glass. Isopropyl alcohol, IPA function to wipe out any debris on the glass, so make sure the glass surface completely smooth and clean before start to paint. Next, lay the glass down so the side to paint glass surface faces upward by using LENR as a coating material like shown in figure 2.1. After done with cleaning, avoid touching the paint side/ surface of the glass once it is clean (http://www.ehow.com/how_8006310_layer-glass-paint.html, 10 March 2011)

Samples were prepared with from 1 to 6 coats of this layer to give a range of final coating thicknesses (R. I. Trezona and I. M. Hutchings, Resistance of paint coatings to multiple solid particle impact: effect of previous coating thickness and substrate material) like shown in figure 2.2. Finally, the analysis part was conducted which briefly explained in the following topic, testing.

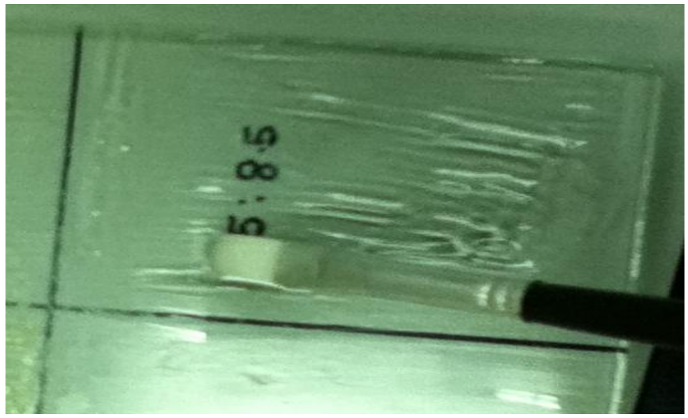

**Figure 3.1**: Glass painting

### 3.3.1 Process Flow of the Experiment

ENR-25 was diluted and stirred with toluene at various ratios; 1:15, 2:47, 2.5:50, 3:54, 4:60, 5:72 (**Figure 3.2 and Figure 3.3**)

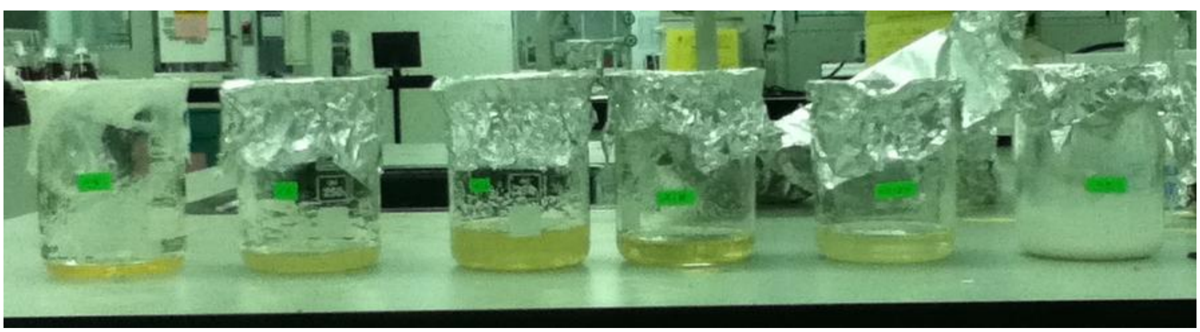

**Figure 3.2**: Samples

**Figure 3.3:** Stirring the sample

First analysis was completed by determining the pH, viscosity and density of samples using Gas Pycnometer, pH meter and Viscometer. Refer **Figure 3.4 and Figure 3.5**

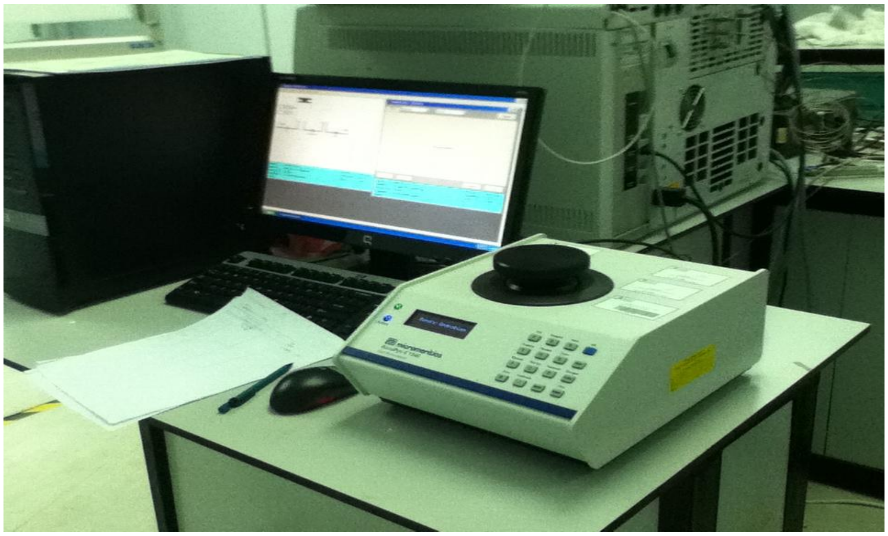

**Figure 3.4:** Gas Pycnometer

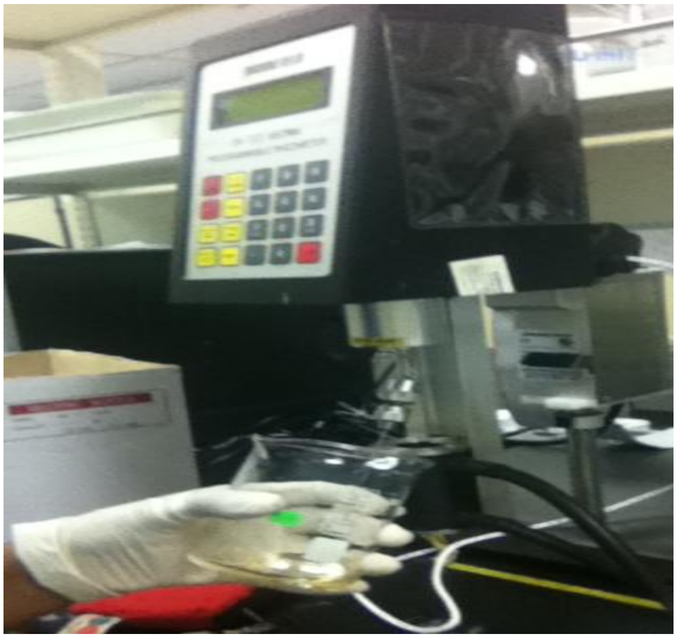

**Figure 3.5:** Viscometer

Clean the glass using IPA followed by coating the sample on the glass plate. Refer **Figure 3.6**

**Figure 3.6:** Clean the glass plate

Paint sample on the glass surface exactly in **Figure 3.1**

Testing performed after coating process; drying test and adhesion test (tape test) **Figure 3.7**

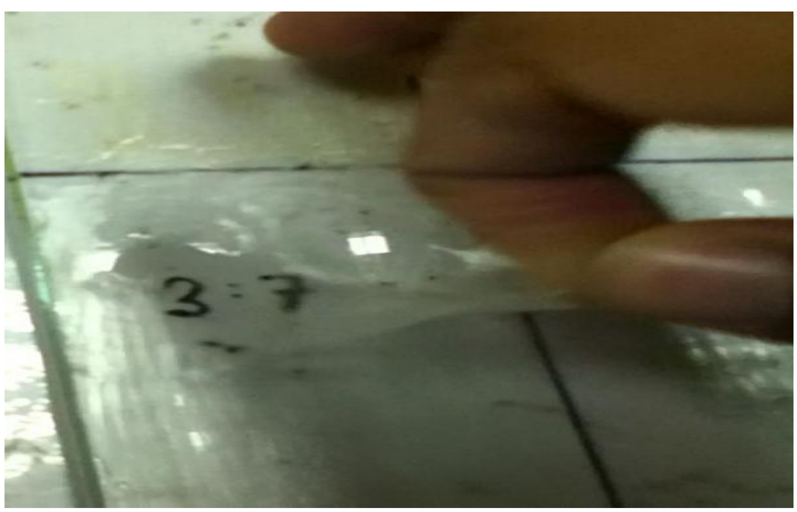


**Figure 3.7:** Peel test

Drying test conducted at ambient temperature, $40^0C$ and $50^0C$. Refer **Figure 3.8 and Figure 3.9**

**Figure 3.8:** Drying sample at ambient temperature


**Figure 3.9:** Drying sample in the oven

## 3.4 TESTING

In order to determine the sample can be applied as a coating material several testing was conducted; drying test and adhesion test which is the tape test. Below is the brief explanation of the testing conducted earlier in this research.

### 3.4.1 Tape Test

The adhesive tape used in this study is 3M emulsion water based acrylic adhesive tape. The method starts by preparing 2 x 8 cm tape. Make for 6 pieces of tape. Apply tape to the surface of the coating then followed with rubbing the tape. Next, peel off the tape slowly (require small force). Observe any coating stick on the tape. Record the observation.

### 3.4.2 Drying Test

This test method relies on a visual assessment of the extent of drying; very light colors and clear varnishes may present difficulties in quantifying the extent of drying. This test method also covers the procedure for determining the drying time of drying painting sample after coating, immediately, start the timer. Few minutes later, test the coating by touch the coating surface with the filter paper.

The test is applicable to all coating either on glass, steel and plastic that dry primarily by depending on the temperature and moisture of sample. Since the comparison of drying time taken between three different temperatures, thus oven is used to dry the coating at 40 and 50 degree Celsius. Record the time taken for the coating to dry.

# CHAPTER 4

# RESULT AND DISCUSSION

## 4.1 INTRODUCTION

The property of the liquid epoxidized natural rubber such have the role of degree of epoxidation and composition of epoxidized natural rubber on the property improvement of coating material was investigated in this study. Tensile strengths and modulus values decreased by the addition of LENR and the study suggests that LENR-25 act as elastic material (P. K. Biju, M. N. Radhakrishnan Nair et.al, 2007). Apply simply, a coating is a film forming substance which protects a substrate (i.e. a surface or material) against possibly damaging elements in its environment, as well as improving its appearance. (Akzo Nobel's Global Coatings Report, 2006). The coatings provided significant increase of the glass strength at the low end and the coatings were found to cover the flaw zone and partially fill in the cracks (Mei Wen and Stephen W. Carson, 2008). Thus, it's expected that the study of applying LENR on glass when viscosity, density, pH and adhesion is influenced by the coating solution itself. The selection of raw material is essential to produce the quality coating product and environmental friendly where the problem with non-degradable product can be prevented.

## 4.2 VISCOSITY COATING

Based on the graph below (**Figure 4.1**), the less viscous coating solution is sample A with ratio 1:15. The more high viscous coating solution is the sample F, with ratio 5:72. A high viscosity means that the liquid will not flow easily. A low viscosity means that the coating liquid flows very easily once applied on the glass surface. Coating needs to be neither thick not thin enough to withstand the environment. (Hans K. Pulker, H. Schmidt and M. A. Aegerter, 1998)

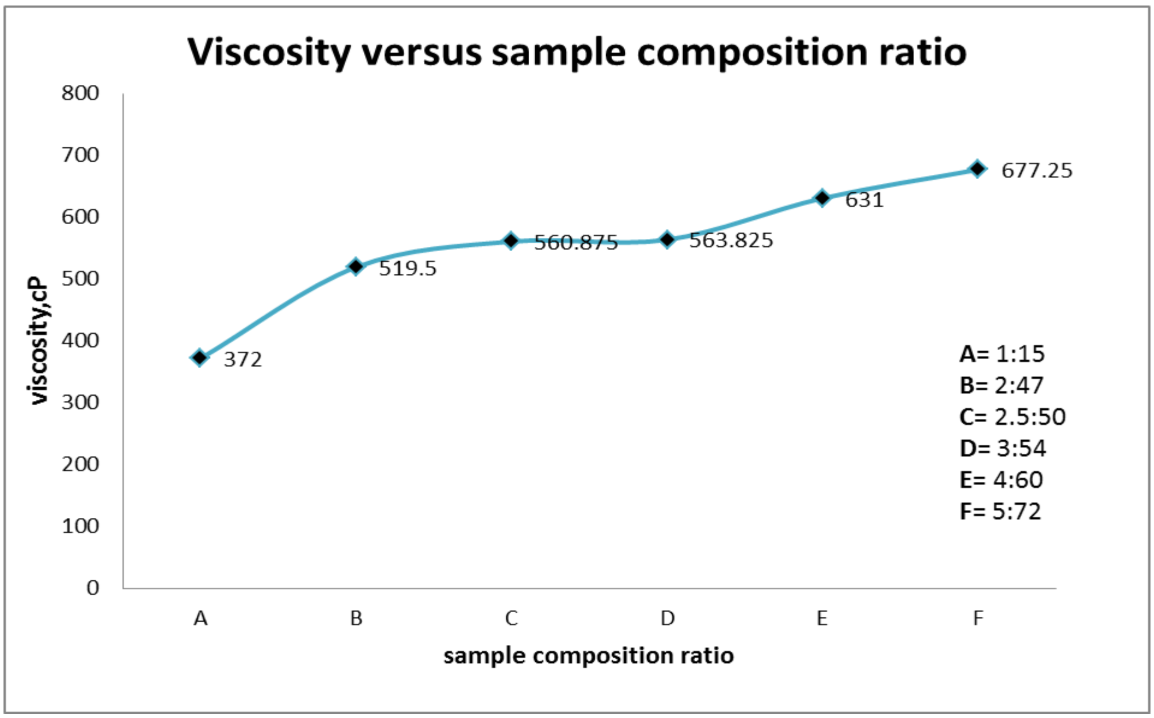


**Figure 4.1**: Relationship between viscosities of sample at different ratios

Viscosity measure of the flow behavior of a liquid plays a vital role in initial receiving inspection of coating and in its following application. Viscosity is a measure of the internal friction of the liquid resulting from intermolecular forces of attraction and other interactions among constituents. Measuring the viscosity of liquid or paste coating and comparing it with a specified value is an inexpensive method and the result are a good criterion for accepting or rejecting an incoming lot of material. The viscosity of a coating should be tested periodically during processing since variation occur during spraying or dip immersion of a part and adversely affect the quality, uniformity and thickness of the coating, Increase in viscosity occur from evaporation of solvent (for solvent- based coatings) and from premature polymerization. Viscosity can be controlled by adjusting the solvent. Controlling the temperature, or discarding the coating if its pot life has expired. (Hans K. Pulker,H. Schmidt and M. A. Aegerter, 1998).

## 4.3 DENSITY OF SAMPLE COATING

According to the result (**Figure 4.3)** the coating solution with highest density is sample E, ratio 4:60. Sample F, ratio 5:72 is the coating solution with lowest density. The density of a liquid is the weight per unit volume at a specified temperature. The density of coating is often used as a quality control test to verify that changes have not been introduced in the coating formulation by the supplier (James J. Licar, 2003)

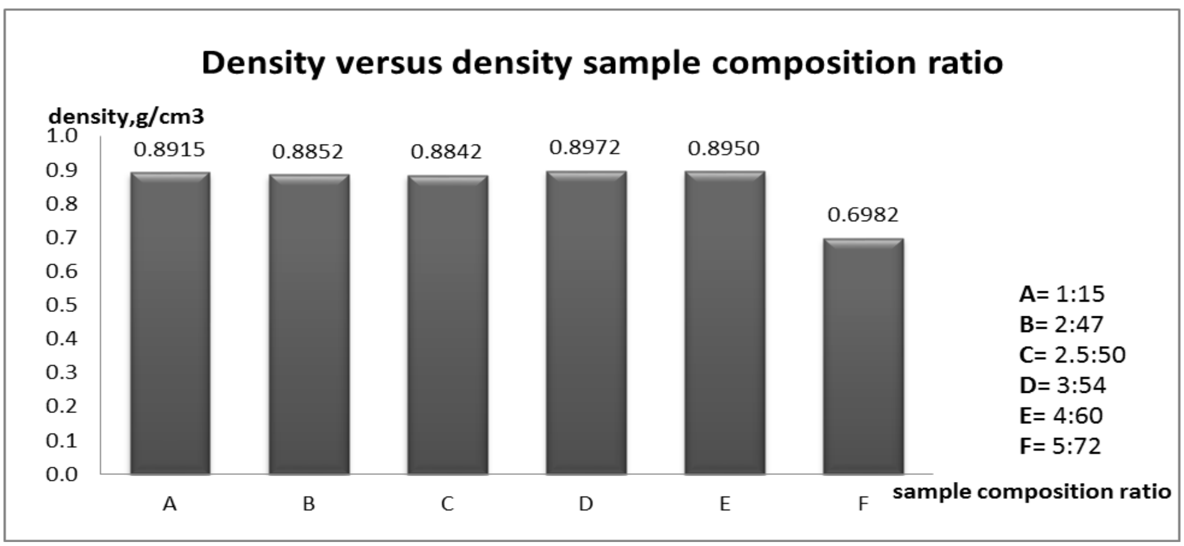


**Figure 4.2**: Density at various LENR composition ratios

## 4.4 pH COATING

A solution of this composition is suitable for property characterization of a surface before and after coating application. According to coating expertise, an extremely high or low pH atmosphere can also shorten the life of the coating life also. The ideal pH is 7, (neutral) but a pH of 6– 9 is acceptable for most coatings unless otherwise specified. Unless the pH is below 6 residual acids (from surrounding factors) on the glass surface and must be neutralized. According to Drisko R.W researcher, oil, grease and dirt can be removed by using an alkaline solution in the pH range of 11 to 12, but not greater than 13 as this will damage the coating. Based on the bar graph below **(figure 4.4)**, the highest sample coating E (4:60), pH 8.55 and the lowest pH coating sample is sample C (2.5:50) and F (5:72) which is pH 8.29. From result obtained all the samples false in optimum pH range. Thus, the all the sample are eligible for pH of coating which false in the finest coating.

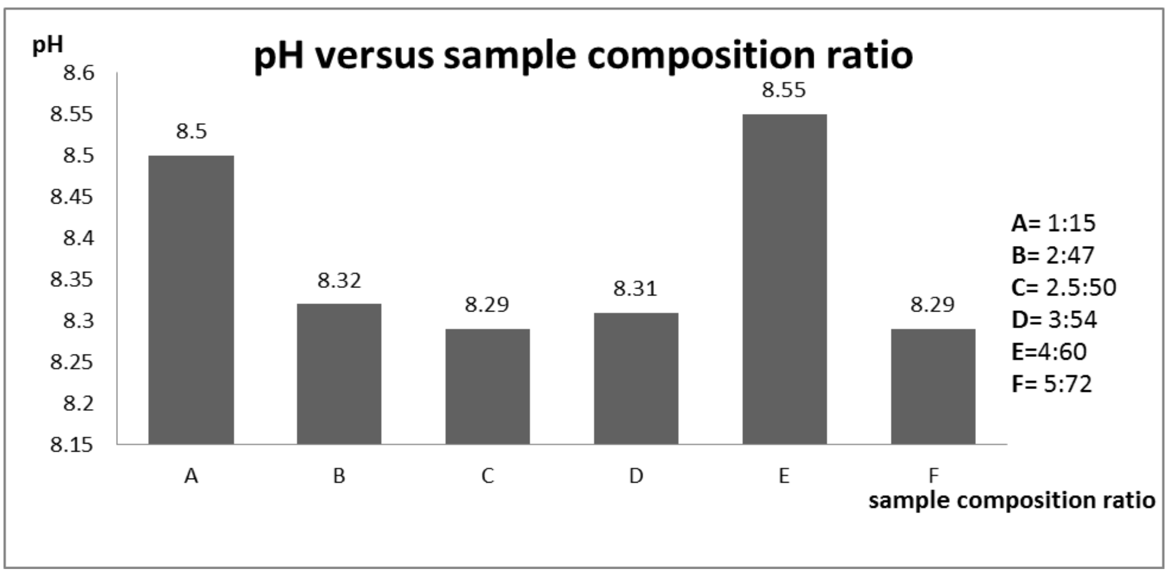


**Figure 4.3:** pH at various LENR composition ratios

## 4.5 DRYING TIME OF SAMPLES

The result (**Table 4.1)** indicates that the drying or curing time of a coating on the glass surface influenced by the temperature. At ambient temperature the drying time required from range (180-360)s and at temperature 40$^{o}$C the range is (120-240)s and at 50$^{o}$C curing time is around (60-180)s.

Cure speed can significantly reduce the drying time. Temperature is inversely independent to the curing time. Besides, the viscosity of sample is dependent to the period of drying time. Under cooler temperatures the viscosity or thickness of the coating increases. This reduces the spread rate and dramatically increases drying times sometimes doubling or tripling it. Due to the rate of coating evaporation, area /spread of coating on glass, viscosity, thickness and temperature of coating are the factors affect the curing time. The graph below (**Figure 4.5**) illustrates the effects of various sample ratios on dry time of the coating. Lower temperatures will increase viscosity and reduce coverage significantly. When high viscosity coat are applied the time taken to cure the coating are longer. Hence, the coating that takes time longer to dry is sample F, ratio 5:72 and the shorter time to dry is sample A, ratio 1:15.

| Sample | Ratio (ENR**:** Toluene) | Drying Time, s | | |
|---|---|---|---|---|
| | | 30$^{o}$C | 40$^{o}$C | 50$^{o}$C |
| A | 1:15 | 240 | 120 | 60 |
| B | 2:47 | 300 | 120 | 60 |
| C | 2.5:50 | 300 | 180 | 60 |
| D | 3:54 | 300 | 180 | 120 |
| E | 4:60 | 360 | 240 | 180 |
| F | 5:72 | 360 | 240 | 180 |

**Table 4.1:** Drying time for coating to dry

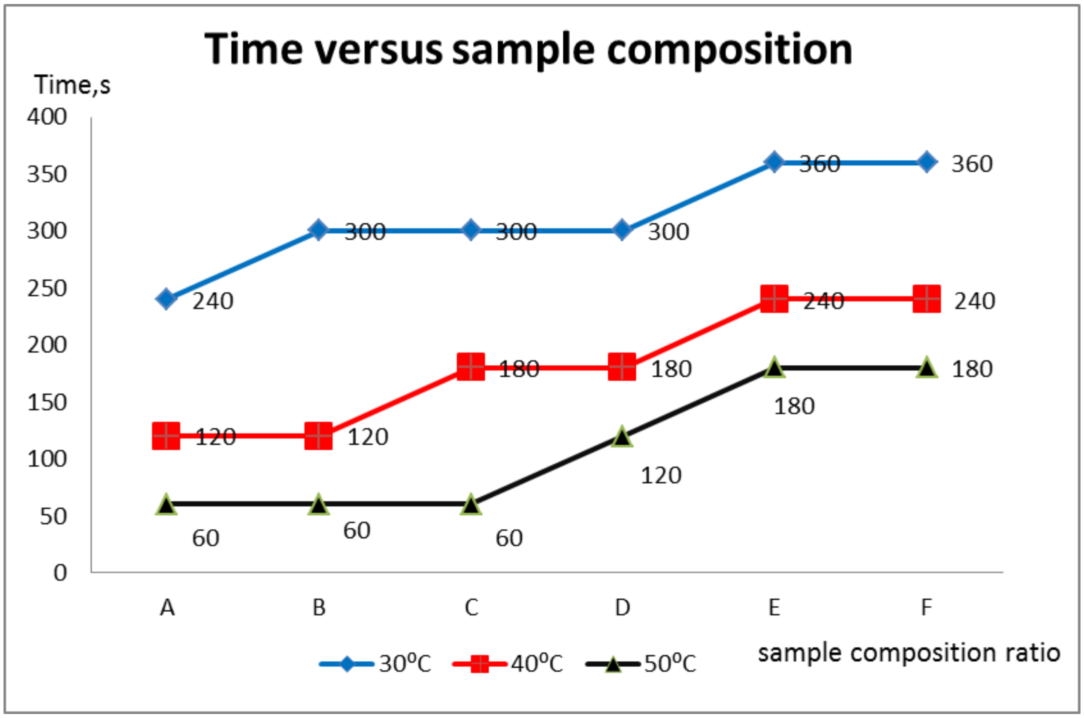


**Figure 4.5**: Cure time of various sample composition ratios

## 4.6 ADHESION TEST

This observation has important implications, since it is common to compare performance of coating materials based on adhesion test of the coating material (LENR) accordance to ASTM standard.

### 4.6.1 Peel Test

Results illustrated below in **Figure 4.6** and **Figure 4.7** for all LENR-based adhesives, F coated sample showed the highest peel strength whereas the lowest peel strength was exhibited by A coated sample, ratio 1:15. As inspected the coating pattern that stick on the tape sample F has small flakes of the coating are detached along the edges of the tape. The area affected is 5 to 15% of the tape. The sample B ratio 2:47 coating has flaked along the edges and on parts of the squares of the tape. The area affected is 15 percent to 35 percent of the tape. However, sample A coating ratio 1:15, the coating has flaked along the edges of the line patterns in long ribbons and whole squares have detached. Refer figure 4.6 and figure 4.7 in appendix.

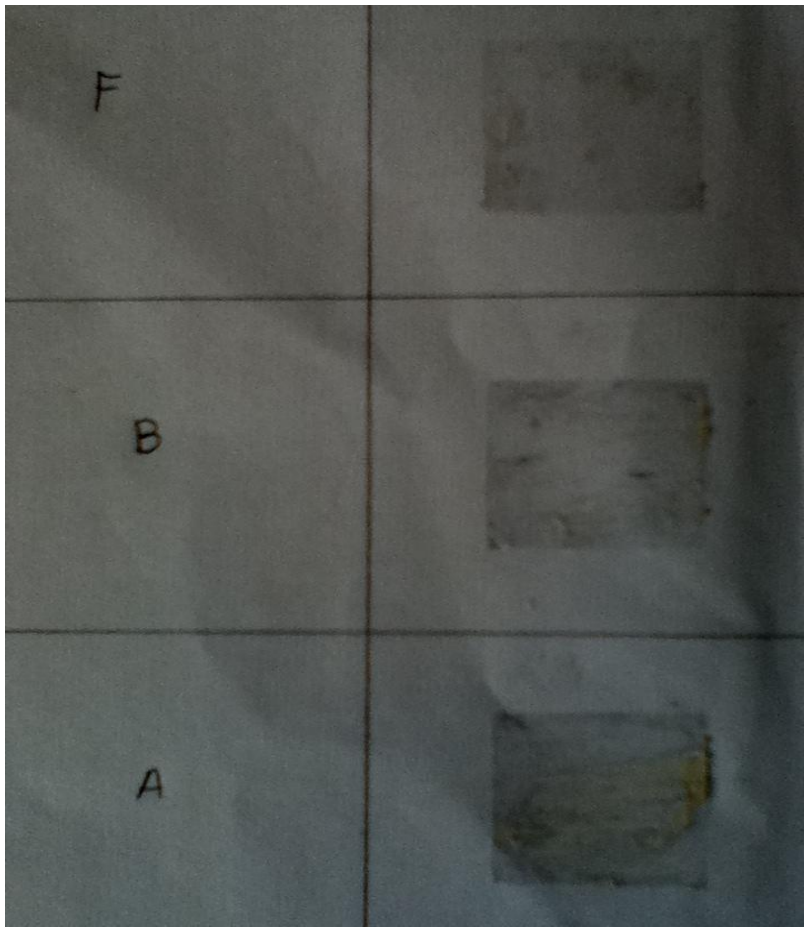


**Figure 4.6**: Peel test of coating samples

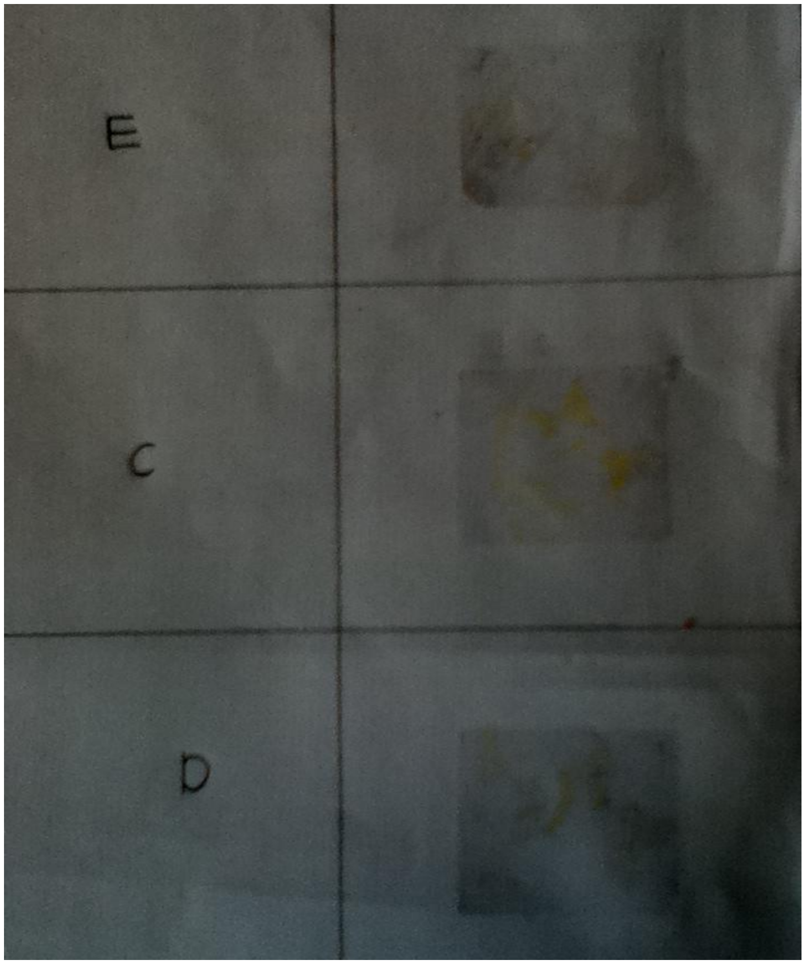


**Figure 4.7**: Peel test of coating samples

# CHAPTER 5

# CONCLUSION AND RECOMMENDATION

## 5.1 CONCLUSION

The experimental running shows that the most convenient sample coating compare from the entire composition ratio, sample E, ratio 4:60 is the coating with average viscosity 631cP and pH 8.55 which act as alkaline coating that possibly protect the glass and have less environment impact. The density of sample E coating is 0.895g/cm$^3$. Moreover, the cure time of the coating is 360s at 30$^0$C, 240s at 40$^0$C and 180s 50$^0$C that higher drying temperature result in short dry time. Also the hydrophobic property is an advantage for coating to protect glass.

From the analysis, the harder to peel hence small flakes of the coating are detached on the tape due to the high viscosity of the coating hence short time taken to dry. The thicker the paint coat is the less influence of glass on the paint scale. (Janina Zieba-Palus, 2006). Mostly natural rubber is used widely and the published literature on epoxidized rubber studies is very limited. Moreover, a study on LENR quality during glass coating is very much needed for developing high quality glass products.

## 5.2 RECOMMENDATION

Recommendations are very crucial in order to improve experimental results for better development of researches in this related field. Based on the present study, here are some recommendations to further improve future studies:

a) For industrial purpose ,it is suggested the peel study nowadays preferably use the equipment of peel test necessary to determine the adhesion of coating by using the Lloyd adhesion tester instrument using three different testing modes (T-Peel Test, $90^o$ Peel Test, $180^o$ Peel Test)
b) For future research recommendation, the formulation of coating solution is suggested to use other alternative solvent or improve the coating solution thru blending of polymer.
c) Further study on coating performance is proposed to perform by using other alternative mechanical testing like scratch resistance, abrasion, hardness test and scratch resistance.such as study on coating thickness on viscosity of coating solution, surface is recommended to test the ability and effectiveness of the removal under conditions of actual use which will have greater validity because of their greater specificity than laboratory tests.
d) A study more on ENR quality on glass coating is very much crucial for developing high quality coating product due to a very limited published literature on epoxidation studies.

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# APPENDIX A1

micromeritics®

| AccuPyc II 1340 V1.05 | Unit 1 | Serial #: 815 | Page 8 |
|---|---|---|---|

Sample: 1:9
Operator:
Submitter:
Bar Code:
File: C:\1340\DATA\000-247.SMP

| | | | |
|---|---|---|---|
| Analysis Gas: | Helium | Analysis Start: | 12/7/2011 4:46:57PM |
| Reported: | 12/7/2011 5:05:52PM | Analysis End: | |
| Sample Mass: | 2.1149 g | Equilib. Rate: | 0.005 psig/min |
| Temperature: | 25.25 °C | Expansion Volume: | 9.1895 $cm^3$ |
| Number of Purges: | 4 | Cell Volume: | 11.7170 $cm^3$ |

**Density and Volume Table**

| Cycle# | Volume ($cm^3$) | Volume Deviation ($cm^3$) | Density ($g/cm^3$) | Density Deviation ($g/cm^3$) | Elapsed Time (mm:ss) | Temperature (°C) |
|---|---|---|---|---|---|---|
| 1 | 2.3724 | 0.0000 | 0.8915 | 0.0000 | 14:50 | 25.25 |

| Summary Data | Average | Standard Deviation |
|---|---|---|
| Volume: | 2.3724 $cm^3$ | 0.0000 $cm^3$ |
| Density: | 0.8915 $g/cm^3$ | 0.0000 $g/cm^3$ |

Appendix A.1: Sample density for ratio 1:15

# APPENDIX A2

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AccuPyc II 1340 V1.05 Unit 1 Serial #: 815 Page 8

Sample: 2:8
Operator:
Submitter:
Bar Code:
File: C:\1340\DATA\000-246.SMP

Analysis Gas: Helium
Reported: 12/7/2011 4:39:31PM
Sample Mass: 1.6821 g
Temperature: 25.21 °C
Number of Purges: 4

Analysis Start: 12/7/2011 4:27:00PM
Analysis End:
Equilib. Rate: 0.005 psig/min
Expansion Volume: 9.1895 $cm^3$
Cell Volume: 11.7170 $cm^3$

**Density and Volume Table**

| Cycle# | Volume ($cm^3$) | Volume Deviation ($cm^3$) | Density ($g/cm^3$) | Density Deviation ($g/cm^3$) | Elapsed Time (mm:ss) | Temperature (°C) |
|---|---|---|---|---|---|---|
| 1 | 1.9003 | 0.0000 | 0.8852 | 0.0000 | 12:02 | 25.21 |

| Summary Data | Average | Standard Deviation |
|---|---|---|
| Volume: | 1.9003 $cm^3$ | 0.0000 $cm^3$ |
| Density: | 0.8852 $g/cm^3$ | 0.0000 $g/cm^3$ |

Figure A.2: Sample density with ratio 2:47

## APPENDIX A3

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AccuPyc II 1340 V1.05 Unit 1 Serial #: 815 Page 8

Sample: 2.5:8.5
Operator:
Submitter:
Bar Code:
File: C:\1340\DATA\000-245.SMP

| | | | |
|---|---|---|---|
| Analysis Gas: | Helium | Analysis Start: | 12/7/2011 4:03:18PM |
| Reported: | 12/7/2011 4:19:47PM | Analysis End: | |
| Sample Mass: | 2.6639 g | Equilib. Rate: | 0.005 psig/min |
| Temperature: | 25.18 °C | Expansion Volume: | 9.1895 $cm^3$ |
| Number of Purges: | 4 | Cell Volume: | 11.7170 $cm^3$ |

**Density and Volume Table**

| Cycle# | Volume ($cm^3$) | Volume Deviation ($cm^3$) | Density ($g/cm^3$) | Density Deviation ($g/cm^3$) | Elapsed Time (mm:ss) | Temperature (°C) |
|---|---|---|---|---|---|---|
| 1 | 3.0127 | 0.0000 | 0.8842 | 0.0000 | 15:04 | 25.18 |

| Summary Data | Average | Standard Deviation |
|---|---|---|
| Volume: | 3.0127 $cm^3$ | 0.0000 $cm^3$ |
| Density: | 0.8842 $g/cm^3$ | 0.0000 $g/cm^3$ |

Figure A.3: Sample density with ratio 2.5:50

# APPENDIX A4

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AccuPyc II 1340 V1.05 Unit 1 Serial #: 815 Page 8

Sample: 3:7
Operator:
Submitter:
Bar Code:
File: C:\1340\DATA\000-248.SMP

Analysis Gas: Helium
Reported: 12/7/2011 5:25:19PM
Sample Mass: 2.5749 g
Temperature: 25.19 °C
Number of Purges: 4

Analysis Start: 12/7/2011 5:18:08PM
Analysis End:
Equilib. Rate: 0.005 psig/min
Expansion Volume: 9.1895 cm³
Cell Volume: 11.7170 cm³

**Density and Volume Table**

| Cycle# | Volume ($cm^3$) | Volume Deviation ($cm^3$) | Density ($g/cm^3$) | Density Deviation ($g/cm^3$) | Elapsed Time (mm:ss) | Temperature (°C) |
|---|---|---|---|---|---|---|
| 1 | 2.8700 | 0.0000 | 0.8972 | 0.0000 | 6:55 | 25.19 |

| Summary Data | Average | Standard Deviation |
|---|---|---|
| Volume: | 2.8700 cm³ | 0.0000 cm³ |
| Density: | 0.8972 g/cm³ | 0.0000 g/cm³ |

Figure A.4: Sample density with ratio 3:54

## APPENDIX A5

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AccuPyc II 1340 V1.05 Unit 1 Serial #: 815 Page 8

Sample: 4:6
Operator:
Submitter:
Bar Code:
File: C:\1340\DATA\000-249.SMP

Analysis Gas: Helium
Reported: 12/7/2011 6:07:54PM
Sample Mass: 2.2498 g
Temperature: 25.21 °C
Number of Purges: 4

Analysis Start: 12/7/2011 5:50:00PM
Analysis End:
Equilib. Rate: 0.005 psig/min
Expansion Volume: 9.1895 cm³
Cell Volume: 11.7170 cm³

**Density and Volume Table**

| Cycle# | Volume (cm³) | Volume Deviation (cm³) | Density (g/cm³) | Density Deviation (g/cm³) | Elapsed Time (mm:ss) | Temperature (°C) |
|---|---|---|---|---|---|---|
| 1 | 2.5137 | 0.0000 | 0.8950 | 0.0000 | 17:25 | 25.21 |

| Summary Data | Average | Standard Deviation |
|---|---|---|
| Volume: | 2.5137 cm³ | 0.0000 cm³ |
| Density: | 0.8950 g/cm³ | 0.0000 g/cm³ |

Figure A.5: Sample density with ratio 4:60

# APPENDIX A6

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AccuPyc II 1340 V1.05 Unit 1 Serial #: 815 Page 8

Sample: 5:5
Operator:
Submitter:
Bar Code:
File: C:\1340\DATA\000-250.SMP

Analysis Gas: Helium
Reported: 12/7/2011 6:31:55PM
Sample Mass: 2.6359 g
Temperature: 25.20 °C
Number of Purges: 4

Analysis Start: 12/7/2011 6:15:43PM
Analysis End:
Equilib. Rate: 0.005 psig/min
Expansion Volume: 9.1895 cm³
Cell Volume: 11.7170 cm³

**Density and Volume Table**

| Cycle# | Volume (cm³) | Volume Deviation (cm³) | Density (g/cm³) | Density Deviation (g/cm³) | Elapsed Time (mm:ss) | Temperature (°C) |
|---|---|---|---|---|---|---|
| 1 | 3.7753 | 0.0002 | 0.6982 | 0.0000 | 9:39 | 25.18 |
| 2 | 3.7752 | 0.0001 | 0.6982 | 0.0000 | 12:11 | 25.23 |
| 3 | 3.7747 | -0.0003 | 0.6983 | 0.0001 | 14:46 | 25.19 |

| Summary Data | Average | Standard Deviation |
|---|---|---|
| Volume: | 3.7751 cm³ | 0.0002 cm³ |
| Density: | 0.6982 g/cm³ | 0.0000 g/cm³ |

Figure A.6: Sample density with ratio 5:72