# OPTIMIZATION OF COMPRESSION MOLDING PROCESS FOR NR/EPDM ELASTOMERIC MATERIAL

# ROSLAN BIN HASSAN B051410087

# UNIVERSITI TEKNIKAL MALAYSIA MELAKA 2017



### OPTIMIZATION OF COMPRESSION MOLDING PROCESS FOR NR/EPDM ELASTOMERIC MATERIAL

This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Manufacturing Process) (Hons.)

by

ROSLAN BIN HASSAN B051410087 931123-01-5399

FACULTY OF MANUFACTURING ENGINEERING 2017





# UNIVERSITI TEKNIKAL MALAYSIA MELAKA

#### BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

Tajuk: OPTIMIZATION OF COMPRESSION MOLDING PROCESS FOR NR/EPDM ELASTOMERIC

Sesi Pengajian: 2016/2017 Semester 2

perlu dikelaskan sebagai SULIT atau TERHAD.

#### Saya ROSLAN BIN HASSAN (931123-01-5399)

mengaku membenarkan Laporan Projek Sarjana Muda (PSM) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

- 1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
- 2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
- 3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
- 4. \*Sila tandakan ( $\sqrt{}$ )

SULIT		g berdarjah keselamatan atau kepentingan rmaktub dalam AKTA RAHSIA RASMI 1972)
TERHAD	(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)	
TIDAK TI	ERHAD	Disahkan oleh:
Alamat Tetap: No 79, Jalan Jati Felda Inas,	3,	Cop Rasmi:
81000 Kulai, Joh Tarikh:	or.	Tarikh:

\*Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini

### **DECLARATION**

I hereby, declared this report entitled "Optimization of Compression Molding Process for NR/EPDM Elastomeric Material" is the results of my own research except as cited in references.

Signature :....

Author's Name : ROSLAN BIN HASSAN

Date : 19<sup>TH</sup> JUNE 2017

### **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of University

Teknikal Malaysia Melaka as a partial fulfilment of the requirements for the degree of

Bachelor of Manufacturing Engineering (Manufacturing Process) (Hons.). The members of

the supervisory committee are as follow:

(ASSOCIATE PROFESSOR DR RAJA IZAMSHAH BIN RAJA ABDULLAH)

### **ABSTRAK**

NR/EPDM elastomer semakin popular dalam industri automotif yang semakin meningkat. Dengan pertambahan dan kepelbagaian aplikasi serta kajian untuk kemajuan masa akan datang, keperluan untuk mempunyai panduan yang tepat dalam pemprosessan elastomer ini telah meningkat dengan mendadak. Objektif utama dalam proses ini adalah untuk mencapai sifat mekanikal yang baik. Kajian ini telah dilakukan dengan mengkaji parameter pemprosessan (iaitu, suhu, tekanan, kadar masa pemanasan dan kadar masa tekanan) dan kesannya terhadap sifat mekanikal (iaitu kekuatan tegangan dan ketumpatan sambung) untuk menghasilkan kombinasi parameter yang optima dalam mencapai prestasi pemprosessan yang terbaik berdasarkan pelbagai objektif. Melalui Response Surface Methodology (RSM), reka bentuk eksperimen dipilih berasaskan pendekatan Box-Behnken dan seterusnya 29 susunan spesimen telah dirancang. Seterusnya, model matematik bagi setiap tindak balas dibangunkan. Kecukupan model dianalisis secara statistic menggunakan ANOVA dalam menentukan input pembolehubah (faktor) yang penting dan kemungkinan adanya interaksi di antara pembolehubah. Pelbagai faktor diagnostik dinilai untuk memeriksa keberkesanan model. Pengoptimuman dengan pelbagai objektif dilakukan melalui pengoptimum berangka dan keputusan yang dijangka akan disahkan. Pemerhatian dan analisis melalui eksperimen mencadangkan bahawa suhu dan kadar masa pemanasan merupakan faktor utama yang mempengaruhi kekuatan tegangan spesimen, manakala bagi tindak balas ketumpatan sambungan pula hanya dipengaruhi oleh satu parameter sahaja iaitu suhu. Kekuatan tegangan dan ketumpatan sambungan akan menurun sekiranya suhu dan kadar masa pemanasan meningkat disebabkan degradasi (suhu terlalu tinggi untuk suhu kerja NR/EPDM). Oleh itu, proses dibawah suhu titik degradasi NR/EPDM amat digalakkan bagi mengelakkan berlakunya pemotongan rantaian di dalam getah tersebut dan seterusnya bagi meningkatkan keupayaan sifat mekanikal NR/EPDM.

### **ABSTRACT**

Natural Rubber/Ethylene Propylene Diene Monomer (NR/EPDM) elastomeric is gaining popularity in the growing automotive industry owing to the fact in terms of sustainability. With extensive studies and increasing number of applications for future advancement, the need for accurate and reliable guide in processing this type of elastomer has increased enormously. Good mechanical properties are always the ultimate objectives. The present work, deals with the study of compression molding parameters (i.e. temperature, pressure, heating time and pressure time) and its effects against NR/EPDM elastomeric mechanical properties (i.e. ultimate tensile strength (UTS) and crosslink density) aim on establishing optimized setup of processing parameters. The optimization are achieve through Response Surface Methodology (RSM), Box-Behnken approach as the design of experiment. A mathematical model for each response is developed to access the relationship between the parameters. Adequacy of models is analyzed statistically using analysis of variance (ANOVA) in determination of significant input variables and possible interactions. Various diagnostic plots are evaluated to check the model effectiveness. Multi objectives optimization is performed through numerical optimization and predicted results are validated. The agreement between experimental and selected solution are found to be strong in between 93% and 96%, thus validating the solution as optimal run condition. The findings suggest that temperature and heating time is the main factor affecting ultimate tensile strength, whereas for crosslink density there is only one significant parameter which is temperature. UTS and crosslink density decrease with the increases of the temperature and heating time due to the degradation (temperature too high for NR/EPDM working temperature). Therefore, it is recommended to start the process below the NR/EPDM degradation point to avoid from the scissoring rubber take place and subsequently improving the mechanical properties.

### **DEDICATION**

### Only

my beloved father, Hassan Bin Yusuf my appreciated mother, Turiah Binti Kasmani my adored sister and brother, Hamidah, Fazilah, Azizah, Suriati, Farhana, Jamal and Syah Irwan

my friends, Bazlaa, Zainal, Izzul, Rouh, Fiza and Amilza for giving me moral support, financial support, encouragement and understandings Thank You So Much & Love You All Forever

### **ACKNOWLEDGEMENT**

First and foremost, in the name of Allah, the Almighty, the most merciful, the most gracious, with the highest praise to Allah that I manage to complete this final year project successfully.

Second, my sincere and deepest appreciation goes to my respected supervisor, Associate Professor Dr Raja Izamshah Bin Raja Abdullah for the great mentoring and guidance that were given to me throughout the project. Besides that, I also would like to express my gratitude to all assistance engineers for guidance and advise in handling the machine and equipment that I used throughout the project.

Last but not least, I would like to give special thanks to my parents for the continuous advice and motivation given to me to complete this project.

Finally, I would like to thank who was important to this report, as well as expressing my apology that I could not mention personally each one of you.

# **TABLE OF CONTENTS**

Abst	strak	i
Abst	stract	ii
Dedi	lication	iii
Ackı	rnowledgement	iv
Tabl	le of Contents	V
List	of Tables	Vii
List	of Figures	ix
List	of Abbreviations	xi
List	of Symbols	xii
CHA	APTER 1: INTRODUCTION	
1.1	Background	1
1.2	Problem Statement	2
1.3	Objectives	3
1.4	Scope of the Research	3
1.5	Organization of Final Year Project	4
CHA	APTER 2: LITERATURE REVIEW	
2.1	Compression Molding	5
	2.1.1 Technique	6
	2.1.2 Mold	7
2.2	Parameters of the Compression Molding	8
2.3	Polymer	9
	2.3.1 Thermoplastic	10
	2.3.2 Thermoset	11
	2.3.3 Elastomer	11
	2.3.3.1 Classification	12

2.4	Rubb	er	12
	2.4.1	Natural Rubber (NR)	13
	2.4.2	Synthetic Rubber	15
		2.4.2.1 Ethylene Propylene Diene Monomer (EPDM)	17
	2.4.3	Natural Rubber/ Ethylene Propylene Diene Monomer (NR/EPDM)	18
2.5	Desig	n of Experiment (DoE)	19
	2.5.1	Response Surface Methodology (RSM)	20
CHA	PTER 3	3: METHODOLOGY	
3.1	Introd	luction	22
3.2	Flowe	chart	22
3.3	Exper	imental Apparatus and Procedure	24
	3.3.1	Mold	24
	3.3.2	Raw Materials	24
		3.3.2.1 NR/EPDM	24
	3.3.3	Machine and Equipment	25
		3.3.3.1 Lever-Controlled Sample Cutter	25
		3.3.3.2 Laser Jet Cutting Machine	26
		3.3.3.3 Compression Molding Machine	27
		3.3.3.4 Analytical Balances	27
3.4	Data A	Analysis	28
	3.4.1	Tensile Test	28
	3.4.2	Swelling Test	29
		3.4.2.1 Swelling Measurement	29
3.5	Exper	imental Parameters	30
3.6	Desig	n of Experiment (DOE)	30
	3.6.1	Response Surface Methodology (Box Behnken Approach)	31
СНА	PTER 4	4: RESULT AND DISCUSSION	
4.1	Exper	imental Results	33
4.2	Ultim	ate Tensile Strength (UTS)	35

	4.2.1	ANOVA Analysis of Ultimate Tensile Strength	37
	4.2.2	Parametric Influence of UTS	38
		4.2.2.1 Mathematical Model of UTS	42
		4.2.2.2 3D Interaction Effects of UTS	43
4.3	Cross	link Density	43
	4.3.1	ANOVA Analysis of Crosslink Density	45
	4.3.2	Parametric Influence of Crosslink Density	46
		4.3.2.1 Mathematical Model of Crosslink Density	50
		4.3.2.2 3D Interaction Effects of Crosslink Density	51
4.4	Optin	nization	52
4.5	Mode	l Validation	54
4.6	Sumn	nary	54
CHA	APTER S	5: CONCLUSION AND RECOMMENDATION	
5.1	Concl	usion	56
5.2	Recor	nmendation for Future Work	57
5.3	Recor	mmendation for Future Work	58
REF	ERENC	CES	59
APP	ENDIC	ES	
A	Mold	of Compression Molding	65
В	Gantt	Chart of FYP I	66
C	Gantt	Chart of FYP II	67

# LIST OF TABLES

2.1	The effect of parameters on elastomer	8
2.2	Summary of the parameters compression molding for NR/EPDM	9
2.3	Classification of elastomers according ASTM D 2000	12
2.4	Properties of Natural Rubber	14
2.5	Types and applications of synthetics rubbers	16
2.6	The properties of ethylene propylene diene monomer	18
2.7	Environmental resistance of NR and EPDM	18
2.8	Characteristics of natural, synthetic and combination elastomer	19
2.9	List of published studies on polymer that using RSM	21
3.1	Processing parameters for NR/EPDM elastomeric	30
3.2	Processing parameters in RSM for NR/EPDM using Box-behnken approach	31
4.1	Experimental result of ultimate tensile strength	35
4.2	R-square analysis of ultimate tensile strength	37
4.3	ANOVA of ultimate tensile strength	38
1.4	Experimental result of crosslink density	44
4.5	R-square analysis of crosslink density	46
4.6	ANOVA of crosslink density	46
1.7	Optical combination of input factors for single and multi-response optimization	53
4.8	Result for confirmation research for NR/EPDM elastomeric responses	54
4.9	Summarized results of the research	55
5.1	Present range of the temperature and recommendation for future study	58

# LIST OF FIGURES

2.1	Part of ompression molding	6
2.2	Side view of flash mold and positive mold	7
2.3	Critical process parameters of compression molding method	8
2.4	Polymer chains in amorphous and crystalline regions	11
2.5	Natural rubber structure 2-methyl-1,3- butadiene	13
2.6	Rubber polymeric chain	13
2.7	Ozone cracking that occurs on the side wall of tire	14
2.8	Some monomers used to produce synthetic rubbers	15
2.9	Structure of ethylene propylene monomer	17
2.10	Chemical structure of ethylene propylene diene monomer	17
2.11	Summary of RSM analysis flow	20
3.1	Process flow of the research from the start until the end	23
3.2	Drawing of the flash mold using SolidWorks2013	24
3.3	Several pieces of NR/EPDM elastomeric before being process	25
3.4	Lever-controlled sample cutter	25
3.5	Mold of types L tension-impact specimens (ASTM D1822)	26
3.6	Mold dimensions of types L tension-impact specimens (ASTM D1822)	26
3.7	Laser jet cutting machine	26
3.8	Flash mold that had been manufacture using laser jet cutting machine	27
3.9	GT 7014 compression molding machine	27
3.10	Analytical balance	28
3.11	Universal Testing Machine (AG-1)	28
4.1	A sample from run no. 17 which has a minimum average UTS (at temperature	36
	180°C, pressure 12.35 MPa, heating time 8 min and 4 min pressure time)	

4.2	A sample from run no. 8 which has a maximum average UTS (at temperature	36
	140°C, pressure 12.35 MPa, heating time 4 min and 4.5 min pressure time)	
4.3	Diagnostic plots for UTS: (a) Normal plot, (b) Residuals vs Predicted, (c)	40
	Residual vs Run and (d) Predicted vs Actual	
4.4	Perturbation graph of parameters towards UTS	41
4.5	3D surface graph of UTS vs temperature (A) vs heating time (B)	43
4.6	A sample from run no. 1 which has a minimum density crosslink (at temperature	45
	160°C, pressure 12.35 MPa, heating time 12 min and 5 min pressure time)	
4.7	A sample from run no. 7 which has a maximum density crosslink (at temperature	45
	160°C, pressure 12.35 MPa, heating time 4 min and 5 min pressure time)	
4.8	Diagnostic plots for crosslink density: (a) Normal plot, (b) Residuals vs	48
	Predicted, (c) Residual vs Run and (d) Predicted vs Actual	
4.9	Perturbation of parameters towards crosslink density	49
4.10	3D surface graph of crosslink density vs temperature (A) vs heating time (B)	51

### LIST OF ABBREVIATIONS

ANOVA - Analysis of Variance

ASTM - American Society for Testing and Materials

CM - Compression Molding

DOE - Design of Experiment

DRC - Dry Rubber Content

EPDM - Ethylene Propylene Diene Monomer

EPM - Ethylene Propylene Monomer

HT - Heating Time

IR - Isoprene Rubber

ISO - International Standard Organization

Max - Maximum

Mc - Crosslink Molecular Weight

NR - Natural Rubber

NR/EPDM - Natural Rubber/Ethylene Propylene Diene Monomer

Press - Pressure

PT - Pressure Time

RSM - Response Surface Methodology

SBR - Styrene Butadiene Rubber

Temp - Temperature

UTS - Ultimate Tensile Strength

Vc - Crosslink density

### LIST OF SYMBOLS

°F - Fahrenheit

% - Percentage

 ${\mathbb C}$  - Degree Celcius

Cal/cc - Calories/cubic centimeter

cm - centimeter

g - Gram

m - Meter

min - Minute

mm - millimeter

MPa - Mega Pascal

N - Newton

psi - Pounds per square inch

sec - Seconds

 $\beta$  - Beta

ρ - Density

#### **CHAPTER 1**

#### INTRODUCTION

This section describes about background, objective, problem statement, scope of the final year project and ends up with the organization of this final year project. Background discusses about the material, compression molding and the software used. While the objective mentions about the mission that needed to be achieved for this project and the scope covers everything what is supposed to perform in this project.

### 1.1 Background

Natural Rubber/Ethylene Propylene Diene Monomer (NR/EPDM) consists of two types of rubber which are Natural Rubber (NR) and Ethylene Propylene Diene Monomer (EPDM) where it was blended together in order to enhance certain properties. NR have a high strength and good dynamic properties (Bottros, 2002) while EPDM has a better chemical resistance and weathering oxidation (Sahakaro *et al.*, 2009). The blending between Natural Rubber and Ethylene Propylene Diene Monomer has given upward push to their heat and ozone resistance (Sahakaro *et al.*, 2009).

In general, rubber material product can be formed by using various processes such as compression molding, injection molding and extrusion. Each process has their own advantages. Compression molding process offers high production rates, accurate repeatability and produced an end product (Collyer, 2016). Generally, compression molding process already been introduced for molding rubber compound starting from the twentieth century and it has been a surprising development in automotive and appliance applications until now. It is a forming

process where the material is placed in the mold that had been heated at certain temperature and then is pressed to form the product that looks exactly like the shape of the mold. Just like making waffles.

In the compression molding process, the parameter plays an important role in production of rubber product, subsequently the control of process parameters is fundamental. From literatures it shows that there are only four parameters that influence the mechanical properties in compression molding which are temperature, pressure, heating time and pressure time. Therefore, this study is concentrate on optimization of compression parameter for NR/EPDM in order to have a longer shelf life in term of strength and good hydrogel netwok in term of crosslink density.

In order to enhance the feedback that might be altered by several input variables, design of experiment (DOE) method is applied. This is carried out according box behnkan design tools of Response Surface Methodology (RSM) using Design-Expert 8 P (DX8P) software for selected factors. With RSM method, the cost for this project can be reduced and the experiment becomes faster and effective (Kandar and Akil, 2016).

#### 1.2 Problem Statement

Nowadays, there are various process for manufacturing rubber product and this has attract many industries around the world to test and determine the best parameter that should be applied in order to get a good product in term of mechanical properties and physical properties while improving service life and reduced cost. One of the famous process is a compression molding which forming the rubber by means of heat and stress in the mold.

Every process has its disadvantages as well as compression molding. The compression molding process is slower and expensive (De and White, 1996). Compression molding process for NR/EPDM can be disturbed by mold curing and cause shrinkage, poor strength and crosslink. There are three factors related to mold curing which are temperature, time and pressure (Campbell, 2004). The different between the thermal linear growth of rubber and mold will affect the shrinkage of the mold cured rubber product while the poor vulcanization process

will result of porosity. In addition, product that manufacture by NR/EPDM usually causes ozone cracking on the surface of the product and directly reduce the shelf life of that product.

### 1.3 Objectives

Based on the challenges faced in compression molding process for NR/EPDM, this project intends to look on the following objectives:

- 1. To establish the effects of compression parameters (temperature, pressure, heating time, pressure time) on the mechanical properties (UTS and crosslink density).
- 2. To optimize the compression molding parameters for achieving high performance of NR/EPDM elastomeric
- 3. To validate the effectiveness of the proposed optimization compression molding parameters for NR/EPDM elastomeric material.

### 1.4 Scope of the Research

In order to achieve research objectives and to plan the research procedure framework, the scopes of this study have to be predetermined. The compression parameters to be studied as aforementioned in the objective will be obtain from the specimen (NR/EPDM) that through the compression molding process using the following tests:

- Response Surface Methodology (RSM)
- Tensile test (ultimate tensile strength)
- Swelling test (crosslink density)

### 1.5 Organization of Final Year Project

- i. Chapter 1 is an introduction chapter that explains about the background of this project, the problem and objective that must be achieved by follow the scope that has been identified.
- ii. Chapter 2 is a literature review that explains on related to this project that has been done by various previous researchers.
- iii. Chapter 3 is a methodology is an overview of study which explain on how the project is done by discussing the process and method that to be used.
- iv. Chapter 4 is a result and discussion that explains on the result of data that has been getting from tensile test and swelling test.
- v. Chapter 5 is a conclusion and recommendation is an overview of the overall of a project that has been done.

**CHAPTER 2** 

LITERATURE REVIEW

The literature review was carried out with the attention to attain the objectives of this

project. It consists of the facts associated with natural rubber materials, compression molding

machine, overall performance observations and modelling by response surface methodology

(RSM). All the information and facts in this section served as a reference and guidelines for this

project.

2.1 Compression Molding (CM)

Compression molding is a process where pressure and heat are subjected to a mold that

is placed with a resin. It is a stable and quick and has been being used to process thermosets,

thermoplastics and elastomer for a very long while. Compression molding condition usually

falls within the following ranges (Allen et al., 2009):

Temperature : 300 - 400°F

Pressure : 2000 - 10000 psi on part

Cure time : 30 - 300 seconds

According to De Focatiis (2012), CM is a process that focus on produce homogeneous,

isotropic test specimens that cannot be manufactured by other process. CM is depending on

accomplishing controlled and repeatable thermal transition of the polymer being shaped. There

are square measure elementary variations within the method that a solid state is achieved

5

C Universiti Teknikal Malaysia Melaka

that depend upon whether or not the polymer being molded is in a position to crystallize. Figure 2.1 shows the compression molding part.

Although CM is widely used in the industry now, but it also has disadvantages. Some disadvantages of CM are the complexity within the process of the metal mold surfaces and the difficulty in reducing damage to the specimens when removing them from the molds (De Focatiis, 2012). In general, the surface area of polymer that contact with the mold give a high impact when removing the specimen.

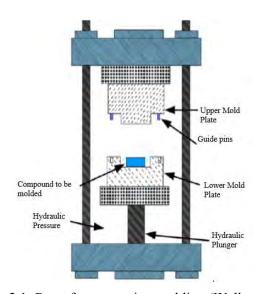


Figure 2.1: Part of compression molding (Walker, 2012)

### 2.1.1 Technique

The process of compression molding is start by heating the mold and followed by loading applicable amount of molding material into the lower half of the mold. The mold then is preheated for a certain temperature and time before the pressure is applied. As the mold under pressure, the material is compressed having the shape of the mold. To avoid the distortion when mold is opened, the material must be fully hardened first. Hardening for thermosetting is affected by further heating and under pressure in the mold while for thermoplastic if effected by chilling and under pressure in the mold (Allen *et al.*, 2009).

#### 2.1.2 Mold

In CM, the material to be loaded into the mold must be thicker than final part, but smaller in length and width and is located at the geometric center. The cavity will be fill out by the preform that spreads during the closing of the mold. According to Bickerton and Abdullah (2003), there are one critical parameter that must be highlighted for implementation of CM which is the mold platen separation at the start of resin injection. Initial separation defines the resistance a mold cavity offers by filling and also the nature of filling that predominates.

There are two types of mold classes which are flash molds and positive molds as shown in Figure 2.2 (Institution, 2005). Flash mold is designed to not exert pressure on the molded polymer in order to enable excess polymer to squeeze out of the mold cavity in the course of molding. Flash molds are normally easy to assemble, as they can be manufactured out of sheet metal of suitable thickness (De Focatiis, 2012). Unlike flash mold, positive molds are completely different. Positive mold are designed to exert direct pressure on the polymer being molded. Positive mold are more complicated to construct and need compress of cavities in solid metal component. The removal of molded specimens is difficult except sufficient shrinkage happens to attract the specimen away from the mold upon cooling.

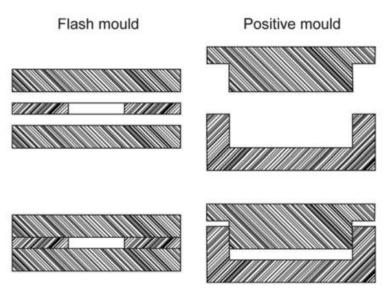


Figure 2.2: Side view of flash mold and positive mold (Institution, 2005)