Preparation and Characterization of Transesterified Waste Cooking Oil as Processing Oil for Sustainable Rubber Compound

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

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfillment of the requirement for Bachelor Degree of Manufacturing Engineering (Hons).
The member of the supervisory committee is as follow:

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(TS. DR. JEEFFERIE BIN ABD RAZAK)

ABSTRAK

Bahan getah banyak digunakan dalam kehidupan seharian manusia. Penggunaan bahan getah yang paling biasa berlaku pada industri tayar, pembinaan dan automotif. Komponen utama sebatian getah ialah getah, pengisi penguat dan minyak pemprosesan. Salah satu tujuan utama memproses minyak adalah untuk mengurangkan kelikatan dan meningkatkan kualiti sebatian getah. Minyak aromatik yang mengandungi hidrokarbon aromatik poliklik digunakan secara meluas dalam menghasilkan banyak jenis sebatian getah kerana kesesuaiannya dengan bahan tak jenuh. Walau bagaimanapun, minyak aromatik mempunyai potensi karsinogenik dan cenderung diganti dengan pilihan lain yang mesra alam. Oleh itu, kajian ini memaparkan potensi minyak masak sisa yang dirawat secara kimia sebagai minyak pemprosesan untuk pengeluaran sebatian getah. Isu pembuangan sisa menggoreng atau minyak masak sisa telah banyak menyumbang kepada masalah persekitaran yang serius. Penggunaan minyak masak semakin meningkat dan bekalan yang konsisten menjadikan minyak masak sebagai bahan makanan komersial. Kira-kira, terdapat kira-kira 3 bilion liter minyak masak yang digunakan di Malaysia setiap tahun yang mana sekitar 30% minyak masak sisa tersedia untuk pengeluaran biodiesel atau pelupusan alam sekitar. Ciri penyembuhan bagi sebatian dan sifat mekanik pemvulkanan akan diukur dan dibandingkan dengan getah berasaskan minyak pemprosesan rujukan. Penyediaan dan ciri minyak masak sisa transesterifikasi akan diukur dengan menggunakan ujian mekanikal dan fizikal iaitu ujian tegangan, pemerhatian fraktur mikroskop SEM, ujian kekerasan, ujian lelasan, ujian termal (TGA, DSC), dan ujian penyerapan.

ABSTRACT

Rubber materials are widely used in human daily life. Most common usage of rubber material is mostly in tires, construction and automotive industry. The main components of rubber compounds are rubber, reinforcing fillers and processing oils. One of the main purposes of processing oils is to decrease the viscosity and to raise the quality of rubber compounds. The aromatic oils which containing polycyclic aromatic hydrocarbon is widely used in producing many type of rubber compound due to their compatibility to unsaturated materials. However, aromatic oils have carcinogenic potential and tend to be replaced with other environmentally friendly option. Therefore, this study presents the potential of chemically treated waste cooking oil (WCO) as processing oil for rubber compound production. Waste management issues of frying or waste cooking oil have led greatly to severe environmental problems. Moreover, around three billion liters of cooking oil consumed in Malaysia yearly which about 30% of WCO accessible for biodiesel production or disposal to environment. In this study the waste cooking oil was modified through the transesterification process to be used as the processing oil for rubber compound. The cure characteristic of the rubber compound and the mechanical properties rubber vulcanization were measured and compared than commercial processing oil based rubber. The resulted NR compounds processed by using the WCO as PO at varying collect (0, 10, 20, 30 grams) has shown promising and comparable cure characteristic, physical and mechanical performances than commercial parafinic as processing oil added to industrial NR compound. This study was significant as WCO used in this study, able to replace parafinic oil as sustainable candidate of processing oil for manufacturing of rubber compound.

DEDICATION

Dedicated to

my beloved country Yemen

My beloved father, Mohammed Alkahlani, my beloved mother, Nadia Alsrori

my beloved family,

my beloved friend, Abdulsalam Khaled

my beloved cousin. Dr. Sahker Alshibani

For giving me honest support, encouragement, cooperation, money, and also understanding.

Thank You So Much & Love You All Forever.

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LIST OF ABBREVIATIONS

AO	-	Aromatic oil
PO	-	Processing oil
PCA	-	Polycyclic aromatic compounding
VO	-	Vegetable oil
VVO	-	Vulcanized vegetable oil
IIR	-	Isobutylene rubber
Mc	-	Cross-Links
NR	-	Natural rubber
SR	-	Synthetic rubber
PAHs	-	Polycyclic aromatic hydrocarbons
WCO	-	Waste cooking oil
CB	-	Carbon black
FFA	-	Free Fatty Acide
FAME	-	Fatty acid methyl ester
HA	-	Highly aromatic
BR	-	Butadiene rubber
ENR	-	Epoxidized natural rubber
SBR	-	Styrene butadiene rubber
TDAE	-	Treated distillate aromatic oil
ODR	-	Oscillating disk rheometer
DSC	-	Differential scanning calorimeter
SEM	-	Scanning selection microscope
SMR	-	Standard Malaysia rubber

CHAPTER 1

INTRODUCTION

This chapter has emphasized a background of study, problem statement, objectives, scope, and significance of study, report organization and summary of study. Moreover, the research scope also encompasses the scope and depth of research, although the real requirements of performing this analysis were specifically explained in problem statement part.

1.1 Research Background

Highly aromatic oil derives from petrochemical industry derivatives, historically been commonly used as rubber compounding extender oils. Their success is attributed to strong ability with most of the natural elastomers. In the manufacturing process of rubber based products, extender oils also referred as softening oils and are applied to rubber compounds in order to ensure acceptable process ability (Chandrasekara *et al.*, 2011).

The main aim of this research is to maneuver the treatment of waste resource to balance the ecosystem and develop alternative processing oil for producing the sustainable rubber compound. There are several choices of bio-based resources with higher possibility to substitute petrochemical resources such as vegetable oil, castor oil, palm oil, and many others. However, waste cooking oil is chosen as processing oil for producing the sustainable rubber compound due to their abundant accessibility. As well as, this initiative also will help to eliminate massive disposal issue of WCO and to protect the environment.

Lately, enormous quantities of waste cooking oil are created and discharged. This could seriously added to serious environmental problems (Ibrehim *et al.*, 2014). In 2013, in Petaling District of Selangor State, Malaysia, a study was conducted for 352 households to examine the awareness, attitudes and best practices into WCO recycling. In this research, it was recorded that the average waste cooking oil generated per household is about 2.34 kg/month (Ibrehim *et al.*, 2014). Thus, this quantity of waste could make a huge disposal problem which later may led into serious pollution issues.

In this research, waste cooking oil was filtered to minimize the highest number of contaminates and particles. Then, the filtered WCO was characterized by using FTIR spectroscopy to evaluate the presence of organic functional groups as well as the chemical substances. By having the understanding from waste cooking oil results, the WCO was modified into processing oil for producing the sustainable rubber compound through transesteification process. The transesterified WCO was then utilized as green processing oil for NR based rubber compound. The resulted compounds were characterized for their cure characteristic behavior, physical and mechanical properties. This research provides obvious of sustainability element by converting waste into wealth and provides the platform into resolving never ending story of waste cooking oil disposal issues.

1.2 Problem statement

The rising attention in environmental issues had maximized the needs for developing new applicant of superior materials that are environmental friendly. Literally, demand of rubber compound resources are enhanced dramatically. Most of existing processing oil to produce the rubber compound is the aromatic types processing oils.

However, most of the aromatic oils have higher content of potential carcinogens which containing polycyclic aromatic hydrocarbons (PAH). On 13-Febrauary 2004, the council of the European communities (2005) has adopted a resolution to prohibit the use and selling of PAH-rich extender oils. Moreover, since viable substitutes are not present in the market, the restriction has not been strictly applied to date (Chandrasekara *et al.*, 2011).

Since WCO is derived from a vegetable source, no hydroxyl group has been found (Fan *et al.*, 2011). In due to that, great effort to find the best replacement for aromatic oil as processing oils should be taken into consideration utilizing of green resources from the household waste that could be the best idea for replacing all those synthetic type of processing oils. Therefore, special step of structural adjustment must be taken in order to introduce the hydroxyl group at the structure of WCO. This can be actualized by performing the transesterification process.

Although the process of transesterification could introduce the hydroxyl group (OH^-) into the WCO structural unit, there are some challenges that must be addressed. Monomer heterogeneity, caused by the existence of double bonds in the triglyceride structure, is a key problem that often occurs in the transesterification process (Maisonneuve *et al.*, 2016).

In addition, until now, there are no specific study to prepare and characterized the NR rubber compounds which produced by using the WCO based processing oils as the extenders. Hence, up until now, there are no ideas about their compatibility, the cure characteristic and even their resulted physical and mechanical properties results that can be referred.

1.3 Objectives

To ensure the success of this research, there are three objectives which are:

1- To modify WCO into usable PO for rubber compound using transesterification method.

2- To evaluate the potential WCO, to be used as PO for NR based compound.

3- To test the mechanical and physical performance of NR based compound by performing related testing.

1.4 Scope of study

1- Prepare rubber compound by using transesterified WCO as extender to replace the aromatic toxic based synthetic oil.

2- Analysis the presence of hydroxyl group (OH^-) in transesterified WCO and compounded natural rubber using Fourier Transform Infrared (FTIR) spectroscopy method.

3- Evaluate the physical and mechanical properties of transesterified WCO based natural rubber compounds.

1.5 Significant of study

WCO is used in this study due to its capability to restore the petrochemical resources. Waste cooking oil contains required molecular structure and could be customized in order to produce the processing oil. This study is significant due to its own advantages.

The use of transesterified WCO in this study is one of the great efforts to minimize the massive quantity of WCO disposal in Malaysia, which decreases the use of non-renewable energy.

At the same time, it is also beneficial to evaluate the potential of WCO as new source of sustainable processing oil for NR based compound manufacturing using standard Malaysia rubber grade (SMR-L 20).

1.5 Organization of study

This report consists of five chapters which are introduction, literature review, methodology, result and discussion as well as conclusion and recommendation. First chapter is introduction; it provides an overview of history and background of this study, problem statement relating to this study, objectives and scope of study concerned as well as the significance of study.

Chapter two outlines on the literature review of previous related studies and articles on this topic. This chapter concerned primarily on the procedure to make processing oil for sustainable rubber compound. This chapter concerning on the waste cooking oils and its applications, collections, and its properties. Moreover, highlights the transesterification process with its classification and applications. Chapter three which is the methodology had modified the method concerned in order to prepare and to characterization waste cooking oil as processing oil for developing sustainable rubber compound and also the physical and mechanical testing performance as the resulted NR based compounds. All of the findings and results are described and evaluated in Chapter 4, which under the "Results and Discussion". Finally, chapter five provides summarization of findings and results, as well as suggestions for further study.

1.6 Summary

In conclusion, there is a need to modify waste cooking oil by inventing the suitable method to manufacture rubber compound by using transesterified waste cooking oil as processing oil for natural rubber applications. The importance of this research had been explained in subtopic of background. The problem statement of this study has fully justified. There are three objectives stated for this study. It was expected that the waste cooking oil could be utilized into as processing oil using for sustainable rubber compound development.

CHAPTER 2

LITERATURE REVIEW

This second chapter highlights the concept and application of processing oil (PO) as extender for rubber compound. Commonly in literature review, it discussed further on rubber compound, its applications, and its properties. Furthermore, this chapter has summarized the transesterified waste cooking oil as sustainable replacement for synthetic processing oil for rubber compound development.

2.1 PO for rubber compound

Processing oils are often utilize to minimize the rubber compound viscosity to improve the compound process ability (Ren et al., 2020). Also Dasgupta et al., (2009) had stated that, the processing additives were added to promote the interaction between rubber and dispersion of the added filler.

Processing aids are organic materials that when applied to polymers, developed their flexibility and enhance their process ability. They are primarily used to reduce or lessen the viscosity and elasticity of pre-vulcanized materials, while improving the ability to blend with other additives in the elastomeric matrix and their fluidity during molding (Zanchet *et al.*, 2016).

Plasticizers has important role in rubber factory due to their ability to minimize the viscosity of rubber, improve the processing ability, improve the elasticity, and minimize processing energy utilization. In addition, the best plasticizers for different rubbers are plant oils. For example, soybean oil, cashew oil, and castor oil (Xu et al., 2020).

Rubber materials are widely used in human daily life especially in tire, construction, textile, and biomedical applications. Most common ingredients of rubber compound are processing oils (PO), oil softeners. The roles of processing oil are to decrease the viscosity and increase the process ability of rubber compound (Öter et al., 2011).

Highly aromatic (HA) oils are usually used in rubber compound processing. In order to enhance the process ability and efficiency of the rubber compound, such as encouraging wet grip and wear resistance, this kind of processing oil was applied to the rubber compound beside other additives (Mohamed *et al.*, 2017).

According to Dasgupta et al., (2007), the Swedish National and Chemicals Inspectorate in 1994, KEMI, has declared that the aromatic oil with higher content of polycyclic aromatic compounds (PCA) as poisonous or carcinogenic. It was also supported by Li *et al.*, (2015) had stated that, the polycyclic aromatic hydrocarbon (PAH), which is the main elements of aromatic oil for tire rubbers, having the potential to cause cancer. Also, it can cause harmful to the community or human health and significant pollution to the environment. The following Table 2.1 demonstrates the commercial PO for rubber that are commonly used for compounding process.

In addition, Flanigan *et al.* (2011) had stated that, the European Union (EU) had prohibited a highly aromatic oils (DAE) which contain more than 10 mg/kg of the sum of eight listed polycyclic aromatic hydrocarbons (PAH) or more than 1 mg/kg. However, the prospective replacement oils for (DAE) has included the naphthenic oils, residual aromatic extracts (RAE), and treated distillate aromatic extracts (TDAE).

Table 2.1: The commercial PO for rubber which used for compounding process (Öter et al., 2011)

Type of Oil	Commercial name	Code
DAE	Aromatic Oil	DAE
MES	Vivatec 200 Nytex 832	MES-1 MES-2
TDAE	Vivatec 500 Nytex 840	TDAE-1 TDAE-2
NAP	Nytex 4700 Octopus N317	NAP-1 NAP-2

2.1.1 Types of PO

Dasgupta et al., (2007) had stated that, the most importance PO for rubber processing purposes is linseed oil, soya bean oil and castor oil which could afford fast drying and good color confinement properties. Soybean oil is known as a rubber plasticizer because the double bond in it will interact in the cross-linking stage along the dual bond of the rubber molecular chains.

All in all, soybean oil is regarded as one of the important vegetable oils that mostly utilized while; soybean oil consumption in 2018 only was over than 56 million tons (Xu et al., 2020).

The bio-based oil was soy oil, beaver oil, olive oil and vulcanized vegetable oils, as reported by Flanigan et al. (2011). Previously, the petroleum based oils used as processing oils for rubber compounds as plasticizers to minimize the compound viscosity and maximize the flexibility at lower temperature. The PO for rubber formulations is summarized as in Table 2.1.1.

For automotive application, due to higher output and lower emissions compared to other preferred methyl esters, linseed oil based methyl ester is the best performing engine fuel. Compared to diesel at rating load, brake specific fuel consumption for linseed oil is increased and is the result of delays in the ignition process (Antony *et al.*, 2017).

Table 2.1.1: PO used for rubber (Flanigan et al., 2011)

Oil Type	Raw Source
Aromatic Oil	Crude Oil
Naphthenic Oil	Crude Oil
Soy Oil	Soybeans
Tall Oil	Pine Trees
Linseed Oil	Flax Seeds
Castor Oil	Castor Plant
Orange Oil	Orange Peel
Vulcanized Vegetable Oil	Soybeans

2.1.2 Properties of PO

Boontawee *et al.* (2013) had stated that; the vegetable oils are plentiful natural products that used for greater performance in rubber compounds processing. Types of vegetable oils (VO) as the following, coconut, palms, and soybean oils can be relevant for rubber compound. By this way, the carcinogenic risk of processing oil, specifically petroleum based aromatic oils may be excluded.

Processing oils possessed several properties such as lower viscosity and good compatibility with rubber. It had deemed that the processing oils have a good compatibility with rubber and able to improve their process ability (Grosser, 2015) .

Processing oil as plasticizers are chosen on the basis of their chemical compatibility with the elastomer matrix and glass transition temperature, which influences the complex mechanical properties of the compounded rubber (Flanigan et al., 2013). Figure 2.1.2: present the properties of NR compound with types of PO.

Table 2.1.2: Properties of NR compound with types of PO (Flanigan et al., 2013)

Types of PO in the NR compound	Mixing energy [kJ]	Mooney viscosity [ML(1+4),100°C]	Scorch time [min]	Cure time [min]	MH-ML [dNm]
Without oil	775	41.5	2.59	10.4	14.76
Coconut oil	626	34.4	3.31	11.36	9.85
Palm oil	574	30.0	3.32	10.09	8.95
Soybean oil	557	25.2	3.37	10.02	8.71

2.1.3 Production of rubber compound

Dario *et al.* (2018) has claimed that, the disposal of waste cooking oil was considered as problem for the humankind. Thus, the feasible solution is to produce raw material for biodiesel production. Nevertheless, to analyse a process for waste cooking oils (WCO), via filtration with a cellulose filter, drying with silica gel, and esterification with methanol, for removing particles as standard procedure for WCO refining by employing a filter paper.

Higher aromatic oils are commonly used as extender oils for rubber compounding. Their success is because of strong compatibility with the most modern natural and synthetic elastomers. In addition, extending oils are also defined as processing oils which are added to the rubber compounding in the manufacturing process of rubber products to ensure reasonable process ability. They are also important to ensure the mechanical consistency of the tires, in particular their conformity to the road surface (Chandrasekara *et al.*, 2011).

Fillers are known as chemicals that are not functioning as a curative agent. The primary aim of their addition is to increase or maximize the mechanical properties of the resulted rubber vulcanizes, such as the tensile strength, tearing, abrasion and flexing resistance. Carbon black (CB) is considered as the most common filler for the manufacturing of different kinds of rubber products, and CB is usually used to strengthen the compound of rubber (Surya *et al.*, 2017).

Usually, fillers, additives, and other strengthening materials are applied to reinforce the rubber compound properties. The processing oils, is one of the compounds that are commonly applied during the manufacturing process of rubber copounds process, which serves as compatibility agent that increasing the process ability of them (Mohamed *et al.*, 2017).

2.2 Rubber compound

Compound refers to the science of selection and combination of elastomers and other additives to achieve the constant mixture and improving the importance physical and chemical properties of finish the products. The aim of rubber compound is to satisfy the end of properties, better processing, and cost reduction and economic processing (Sisanth *et al.*, 2017).

The growth of industry had made the use of rubber materials becoming widespread, and also brings out the higher standards for rubber properties as well as manufacturing techniques (He *et al.*, 2019).

The drawbacks which restrict the application of natural rubber are includes of being instable at higher temperature; being difficult to process into oil-based and organic solvents because of its unsaturated hydrocarbon chain structure and non-polar mechanical properties of cross-linked rubber that must changed via addition of fillers to confirm their final applications. Petroleum-based fillers are used in the preparation of natural rubber (Bao *et al.*, 2016).

Rubber and elastomers is considering as are of the most common materials that are substantially known for their versatility and capability to contain particles such as CB, silica and clay in order to enhance and achieve the required properties for particular applications wherever impact tolerance or hardness is required (Mohamed *et al.*, 2017).