# ODOR REDUCTION AND MECHANICAL PROPERTIES OF NATURAL RUBBER LATEX/SEPIOLITE FILMS

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## DEDICATION

This thesis is dedicated to my supervisor, co-supervisor, family members, friends, and staff in UTM who have supported me throughout the process.

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## ABSTRACT

The bacterial action on non-rubber components and thermal degradation lead to offensive odor in natural rubber. This study aims to investigate the odor reduction, mechanical and morphological properties of the natural rubber latex (NRL)/sepiolite films with sepiolite from Tolsa (sepiolite-T), and sepiolite from China (sepiolite-C). The NRL/sepiolite films were prepared by heating of the NRL and curatives with various sepiolite content, from 0 to 2.0 parts per hundred rubber (phr), in a water bath at 70 ± 1 °C. Then, casting the prevulcanized latex onto the glass mold. The results of nitrogen adsorption-desorption isotherms and pore size distribution show that sepiolite-T is a mesoporous material with high specific surface area ($S_{BET}$) and total pore volume, while sepiolite-C is a macroporous material with low $S_{BET}$ and total pore volume. The presence of volatile organic compounds (VOCs) included volatile fatty acids, alcohol, amide, hydrocarbon, terpene, and piperidines in neat NRL films and NRL/sepiolite films has been confirmed by using gas chromatography-mass spectrometry. The results show that the mesoporous sepiolite-T adsorbed more odorous VOCs compared to macroporous sepiolite-C in the neat NRL films. The result of dynamic olfactometry analysis also shows a negative correlation between odor concentration and the sepiolite content. Besides that, the odor concentration of NRL/sepiolite-T samples was much lower than NRL/sepiolite-C samples and the lowest odor concentration was achieved by adding 2.0 phr sepiolite-T ($T_{2.0}$). The addition of 2.0 phr sepiolite in NRL/sepiolite-T and NRL/sepiolite-C reduced the odor concentration of NRL films by 69.1% and 59.4%, respectively. The morphological studies of NRL/sepiolite films reveal that the defects including sepiolite agglomerate, free volume and sepiolite pull-out were evident in NRL/sepiolite films. It was found that the tendency of defects formation increased with sepiolite content and consequently led to a decrease in tensile strength of NRL/sepiolite films. The rubber/sepiolite interaction in NRL/sepiolite-C films was proven by the ratio of the volume fraction of rubber in unfilled rubber sample to the volume fraction of rubber in a filled rubber sample ($V_r/V_{rf}$). Therefore, NRL/sepiolite-C films demonstrated a higher tensile strength than NRL/sepiolite-T films. The increase in modulus at 300% elongation (M300) and decrease in elongation at break of NRL/sepiolite films show that the sepiolite provided stiffness to NRL/sepiolite films. The tear strength of NRL/sepiolite-T films was found to increase with sepiolite-T content due to the improvement of crack propagation resistance conferred by sepiolite-T. However, no significant changes of tear strength have been noticed in NRL/sepiolite-C films. Thus it can be concluded that sepiolite-T is a promising deodorizer for NRL films due to its higher odorous VOCs adsorption potentials. However, the high stiffness of the NRL/sepiolite films may not be appropriate for the thin rubber products except $T_{0.5}$ and $T_{1.0}$, which has the high elongation at break and low M300.

# ABSTRAK

Tindak balas bakteria pada komponen bukan getah dan perosotan terma menyebabkan getah asli berbau. Kajian ini bertujuan untuk mengkaji pengurangan bau, sifat-sifat mekanik dan sifat-sifat morfologi bagi filem lateks getah asli (NRL)/sepiolit bersama sepiolit daripada Tolsa (sepiolit-T) dan sepiolit daripada China (sepiolit-C). Filem NRL/sepiolit telah disediakan dengan memanaskan NRL bersama bahan penvulkan dengan kandungan sepiolit yang berbeza dari 0 hingga 2.0 bahagian per seratus getah (phr), dalam air pada 70 ± 1 °C. Kemudian, lateks dituangkan ke dalam acuan kaca. Hasil isoterma penjerapan-nyah penjerapan nitrogen dan taburan saiz liang menunjukkan bahawa sepiolit-T adalah bahan mesopori yang mempunyai luas permukaan spesifik ($S_{BET}$) dan jumlah isipadu liang yang tinggi, manakala sepiolit-C adalah bahan makropori yang mempunyai $S_{BET}$ dan jumlah isipadu liang yang rendah. Kewujudan sebatian organik meruap (VOCs) termasuk asid lemak, alkohol, amida, hidrokarbon, terpen dan piperidin dalam filem NRL tanpa sepiolit dan filem NRL/sepiolit telah dipastikan dengan menggunakan gas kromatografi-spektrometri jisim. Hasil analisis menunjukkan bahawa mesopori sepiolit-T dapat menyerap lebih banyak VOCs berbau berbanding makropori sepiolit-C dalam filem NRL. Keputusan analisis olfaktometri dinamik juga menunjukkan hubung kait yang negatif antara kepekatan bau dan kandungan sepiolit. Di samping itu, kepekatan bau sampel NRL/sepiolit-T juga jauh lebih rendah berbanding sampel NRL/sepiolit-C dan kepekatan bau yang paling rendah dapat dicapai oleh filem NRL dengan kandungan sepiolit-T sebanyak 2.0 phr ($T_{2.0}$). Penambahan 2.0 phr sepiolit dalam filem NRL/sepiolit-T dan NRL/sepiolit-C dapat mengurangkan kepekatan bau masing-masing sebanyak 69.1% dan 59.4%. Kajian morfologi filem NRL/sepiolit telah membuktikan terdapat gumpalan sepiolit, rongga udara, dan penarikan-keluar sepiolit dalam filem NRL/sepiolit. Ianya didapati bahawa kecenderungan untuk menghasilkan kecacatan tersebut meningkat apabila kandungan sepiolit bertambah dan seterusnya menyebabkan kekuatan tensil bagi filem NRL/sepiolit menurun. Interaksi antara getah/sepiolit dalam filem NRL/sepiolit-C telah dibukti menggunakan nisbah jumlah pecahan getah dalam sampel getah kosong kepada jumlah pecahan getah dalam sampel getah yang berisi ($V_r/V_{rf}$). Oleh itu, filem NRL/sepiolit-C mempunyai kekuatan tensil yang tinggi berbanding filem NRL/sepiolit-T. Peningkatan dalam modulus pada pemanjangan 300% (M300) dan pengurangan dalam pemanjangan pada takat putus bagi filem NRL/sepiolit menunjukkan bahawa sepiolit memberikan kekakuan terhadap filem NRL/sepiolit. Kekuatan koyak bagi filem NRL/sepiolit-T didapati meningkat apabila jumlah sepiolit-T bertambah disebabkan oleh pernambahan rintangan penyebaran rekahan yang diberikan oleh sepiolit-T. Walau bagaimanapun, tiada perubahan ketara pada kekuatan koyak bagi filem NRL/sepiolit-C. Oleh itu, dapat disimpulkan bahawa sepiolit-T berpotensi untuk menghilangkan bau pada filem NRL disebabkan oleh kadar penjerapan VOCs berbau yang lebih tinggi. Walau bagaimanapun, kekakuan yang tinggi pada filem NRL/sepiolit menjadikan ia tidak sesuai digunakan untuk produk getah yang nipis kecuali $T_{0.5}$ dan $T_{1.0}$, dimana ia mengandungi pemanjangan pada takat putus yang tinggi dan M300 yang rendah.

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## LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| 3-APTMS | - | 3-Aminopropyltrimethoxysilane |
| Al | - | Aluminium |
| BET | - | Brunauer–Emmett–Teller |
| Ca | - | Cacium |
| CEC | - | Cation Exchange Capacity |
| Cu | | Copper |
| DT | - | Detection Threshold |
| EDTA | - | Ethylenediaminetetraacetic Acid |
| EDX | - | Energy-Dispersive X-Ray |
| EPA | - | Environmental Protection Agency |
| EVA | - | Ethylene-Vinyl Acetate |
| Fe | - | Ferum |
| FEP | - | Tetrafluoroethylene Hexafluoropropylene Copolymer |
| FESEM | - | Field-Emission Scanning Electron Microscope |
| FTIR | - | Fourier Transformer Infrared |
| GC-MS | - | Gas Chromatography-Mass Spectrometry |
| GP | - | Gas Permeability |
| $H^+$ | - | Hydrogen Ion |
| HA | - | High Ammonia |
| HDTMA | - | Hexadecyltrimethylammonium |
| $HNO_3$ | - | Nitric Acid |
| HS | - | Headspace |
| LA | - | Low Ammonia |
| LRP | - | Large Rubber Particle |
| MB | - | Methylene Blue |
| Mg | - | Magnesium |
| MMT | - | Montmorillonite |
| Mn | - | Manganese |
| $N_2$ | - | Nitrogen |
| NaOH | - | Sodium Hydroxide |

| | | |
|---|---|---|
| ND | - | Not Detected |
| $NH_3$ | - | Ammonia |
| NR | - | Natural Rubber |
| NRL | - | Natural Rubber Latex |
| O | - | Oxygen |
| ODT | - | Odor Detection Threshold |
| $OH^-$ | - | Hydroxide Ion |
| PET | - | Polyethyleneterephthalate |
| phr | - | Parts Per Hundred Rubber |
| PLA | - | Polylactic Acid |
| ppm | - | Parts Per Million |
| PTFE | - | Polytetrafluoroethylene |
| PVF | - | Polyvinylfluoride |
| RT | - | Retention Time |
| SEM | - | Scanning Electron Microscope |
| Si | - | Silicon |
| SPME | - | Solid Phase Micro-Extraction |
| SRP | - | Small Rubber Particle |
| TEM | - | Transmission Electron Microscopy |
| Ti | - | Titanium |
| TMTD | - | Tetramethylthiuramdisulfide |
| VFAs | - | Volatile Fatty Acids |
| VOCs | - | Volatile Organic Compounds |
| WVP | - | Water Vapor Permeability |
| Zn | - | Zinc |

## LIST OF SYMBOLS

| | | |
|---|---|---|
| $P/P_o$ | - | Relative Pressure |
| $ou/m^3$ | - | Odor Unit Per Cubic Meter |
| $V_r/V_{rf}$ | - | Ratio of the Volume Fraction of Rubber in Unfilled Rubber Sample to the Volume Fraction of Rubber in filled Rubber Sample |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Since 18th century, the discovery of natural rubber (NR) brings convenience to our daily life. Nowadays, we can find goods made from NR everywhere and cannot avoid its influence in modern civilization. Generally, NR is collected mainly in the form of the latex. This natural rubber latex (NRL) has gained significant importance in various common applications such as balloons, condom, gloves, adhesives and piping (Utara and Klinkaewnarong, 2015). Over the past few decades, the interest in attempting to enhance NRL qualities has never ceased. The NR industry still forged ahead to discover new technologies to overcome deficiency of NRL derived from *Hevea brasiliensis* tree.

The utilization of NRL in the manufacture of some thin rubber products is due to its unique physical properties that encourage its use in a wide range of end products. These interesting properties that NRL are known for such as outstanding flexibility, great elasticity, low modulus, and excellent surface abrasion (Simchareon et al., 2012; Mohamad et al., 2013). However, it also has a profound impact on our environment. The malodor from NR products has long been raised by the public since it affects both outdoor and indoor air quality. However, specific and stricter regulations as well as environmental quality act to control odor emissions and develop an odor discharge limits for all odor emitting factories are yet to be dictated and enforced by Department of Environment of Malaysia.

The obnoxious odor is mainly due to the obnoxious volatile organic compounds (VOCs) such as volatile fatty acids (VFAs), nitrogen-containing compounds, aldehydes, ketones, terpenes, sulphur-containing compounds, aromatic compounds, hydrocarbons and preservatives, for instance, ammonia and formaldehyde

(Idris et al., 2012; Hoven et al., 2004; Sakdapipanich and Insom, 2006; Juntarachat et al., 2013). The growth of microbes, including bacteria and fungi in the latex may be caused by direct contact with a contaminated object, such as a collecting instrument, equipment or an environmental surface. To make it clear, obnoxious VOCs are produced from the bacterial action on non-rubber organic compounds or thermal degradation (Hoven et al., 2004; Sakdapipanich and Rojruthai, 2012; Nornanysya et al., 2013).

The idea of utilization of sepiolite as anti-odor agent in the manufacture of NRL is invented as a response to the new global trend towards a sustainable development in countries as well as consumer pressure. By October 2018, Malaysia was the sixth largest NR producer in the world, therefore, it can be concluded that rubber industry is one of the major source of malodor VOCs emissions in Malaysia as well (DOSM, 2019). The VOCs control remains in the forefront of the mind of planners, designers, and operators due to the regulation governing the ambient standard for indoor air contaminants by Malaysia government (Malaysia, 2014) as well as the emission standards for VOCs under Industry Code of Practice on Indoor Air Quality 2010 (DOSH, 2010). Nevertheless, it is next crucial step to urge government to implement and enact legislation in order to combat odor generation. In fact, atmospheric pollution resulting from malodor VOCs emissions can truly affect the public health.

Sepiolite, $Si_{12}O_{30}Mg_8(OH)_4(OH_2)_4 \cdot 8H_2O$ is a porous clay mineral which has a trioctahedral character. Sepiolite has an open structure exhibiting a microfibrous structure with a high specific surface area and a large micropore volume. Its high porosity and molecular-sieving properties are also responsible for the high sorption capacity and selectivity of sepiolite, respectively. Furthermore, this abundant clay is actually low-cost (Weir et al., 2002; Cobas et al., 2014). Some of the research signified that the sepiolite are being considered for adsorption purpose such as, carrier for chemicals (Ma et al., 1999), industrial adsorbent for liquid spills and leaks (Zadaka-Amir et al., 2013), and absorbent for toxic and hazardous wastes (Rodríguez et al., 2010; Ortega et al., 2011; Uğurlu and Karaoğlu, 2011).

Many studies confirmed porous materials (activated carbon, carbon black, silica, zeolite, alumina and sepiolite) with high specific surface area have a potential to reduce the malodor (Hoven et al., 2004; Eza et al., 2014; Köse et al., 2005; Wypych, 2013; Shuler, 1954; Güner et al., 2019). This is because the increase in number of pores provides more adsorption sites for odorous VOCs. As a matter of fact, sepiolite possesses both hydrophilic and hydrophobic properties could capture and retain the odor molecules in its interior and exterior as well (Benli et al., 2012; Yariv, 2001). The ability of porous materials to interact with the odor molecules is attributed to the physical forces or chemical reaction (Hoven et al., 2004). The presence of hydrophilic structural tunnels (particularly for small polar organic molecules), silanol groups (particularly for adsorption of polar organic compounds) and siloxane groups at the external surface (particularly for adsorption of hydrophobic and organophilic compounds) confirmed the sepiolite is a good candidate used for odor reduction (Yariv, 2001).

To assist in the possible improvement of malodor reduction in NRL, obtaining the lowest perceptible odor concentration of each sample by dynamic olfactometry is necessary. Dynamic olfactometry can provide accurate odor intensity for implementing effective adsorbent tailored for odorous VOCs adsorption.

Besides that, sepiolite has been used as it possesses many advantages, including provide improvement on processing, dimensional stability, mechanical strength and thermal resistance. This contributes a significant role on reinforcement for polymer and elastomer (Bokobza et al., 2004; Bidsorkhi et al., 2014; Manchanda et al., 2015). The presence of structural tunnel and silanol groups of sepiolite has a potential to enhance the interaction between sepiolite and the polymer chain (Manchanda et al., 2015). Also, the interaction between the hydrophobic polymer and hydrophobic surface is possible due to the presence of hydrophobic character on sepiolite external surfaces (Benli et al., 2012).

## 1.2 Problem Background

Malaysia as being one of the major NR producer in the world is well aware of the problem with malodour generation by the raw rubber processing factories. The safety issue of malodor from NR is mainly attributed to the obnoxious VOCs emitted (Nornanysya et al., 2013). Influence of odor can have adverse effects in employee health and annoyance, especially respiratory and pulmonary system (Dalton, 2003; Wolkoff and Nielsen, 2017; Jaén and Dalton, 2014; Gorguner and Akgun, 2010). Neighbors close to NR processing factories do not want to be impacted by odorous compounds emission and atmospheric pollution from the facilities. Besides that, impairment of olfactory function in humans has been associated with exposure to these obnoxious VOCs (Wypych, 2013).

With growing concerns over personal health and safety, water scrubber treatment system has been adopted by most raw rubber processing factories in Malaysia to address the malodor issue (Kamarulzaman et al., 2012). However, this complaint is addressed to raw rubber processing factories, instead of rubber products manufacturing. As for NR products manufacturing, masking the unpleasant latex scent with aromatic, fragrances or flavor is a conventional method. In fact, masking does not get to the root of the odor problem, and there may be an instance wherein people exhibit fragrance sensitivity or allergy (Fukumoto et al., 1997). Not long ago Nornanysya et al. (2013) used antimicrobial agents such as sodium hypochlorite and formaldehyde to eliminate rubber odor. Hoven et al. (2004) addressed the issue of malodor emission from vulcanizates by using carbon black, zeolite 13x and chitosan. Also, Rattanaplome et al. (2015) reduced the malodour from rubber by using perlite, which is a naturally occurring siliceous rock. However, because of its relatively high cost, the attempt to use low cost, high specific surface area and naturally occurring adsorbents for VOCs removal from NR products has become popular. In more recent times, sepiolite has received great attention due to its low cost and highly porous structure with large surface area. Very recently, sepiolite has been employed as adsorbent to remove off-odor from fish oil (Güner et al., 2019). However, the study evaluated the odor reduction ability of sepiolite with the total concentrations (µg/kg) of all volatiles detected and compared with the control sample to get the % reduction

values. This comparison does not take the contribution of each compounds to the off-odor into consideration. There was also a preliminary study showed a positive result on the application of sepiolite in wound odor control (Köse et al., 2005). The wound odor study has been focused on only one type of odor molecule, which is diethylamine. Yet, there is no emphasis on a mixture of odor-causing molecules present in wound.

Porous clay minerals including montmorillonite, halloysite, kaolinite, bentonite and diatomite for benzene and dimethyl-benzene adsorption were conducted by Deng et al. (2017) and Zaitan et al. (2013). It showed that clay has potential to reduce odorous VOCs.

In this research work, NRL/sepiolite films are prepared to see the effect of sepiolite on the odor reducing. The odor reduction ability of sepiolite for total VOCs is evaluated by classifying the VOCs according to their respective literature odor detection threshold (ODT). Dynamic olfactometry is used to deal with the interpretation of total contribution of VOCs to the odor emission in NRL films.

## 1.3 Problem Statement

The objectives of this research are:

(a) To identify the odorous compounds of natural rubber latex films by GC-MS.

(b) To quantify the odor emitted from natural rubber latex films as a function of the different sepiolite contents by GC-MS and dynamic olfactometry.

(c) To evaluate the influence of sepiolite content on mechanical and morphological properties of the natural rubber latex films.

## 1.4 Scope of Study

The introduction highlights the odor problem arises from NR products, solutions used to combat the odor in NR, and the recent research progress in odor control by using sepiolite. There are many instances, achievements and early works mentioned in the introduction will be well elaborated on the literature review. This study will focus on reducing odor of NRL films with sepiolite. The important aspects relate to the odor reduction ability of sepiolite are the porosity and BET surface area, pore filling processes, as well as surface functional group of sepiolite. In order to achieve the objectives, the scope of research are:

(a) Preparation of NRL/sepiolite films with different compositions of 0, 0.5, 1.0, 1.5, and 2.0 parts per hundred rubber (phr) of sepiolite contents.

(b) $N_2$-BET method, Field Emission Scanning Electron Microscope (FESEM), and Fourier Transformer Infrared (FTIR) were used in sepiolite characterization.

(c) Headspace (HS) Solid-Phase Microextraction (SPME) and Gas Chromatography-Mass Spectroscopy (GC-MS) was used to identify the volatile organic compounds that lead to unpleasant odor of NRL/sepiolite films.

(d) Attenuated Total Reflection Fourier Transform Infrared was used to ascertain the interaction between sepiolite and VOCs in NRL films.

(e) Dynamic olfactometry was used to determine the odor concentration of NRL films and conducted in accordance with the MS 1963:2007.

(f) Mechanical and morphological test were performed according to ASTM standard.

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