SYNTHESIZE OF CRUMB RUBBER USING PULSED LASER ABLATION IN LIQUID

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To my beloved husband, Safwan Aziz, mama, abah and family

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ABSTRACT

A novel crumb rubber production technique incorporating laser technology is introduced as an alternative to mechanical grinding and cryogenic processing techniques. This technique offers solution to major drawbacks of the existing techniques in terms of the size of the crumb rubber particles. The effectiveness of utilizing the produced crumb rubber as oil absorbent applied in oil spilled recovery application has been explored. A nanosecond Nd:YAG pulse laser with 1064 nm wavelength was used as a light source in the pulse laser ablation in liquid (PLAL) technique. The rubber target used in this work is a piece of scrap tyre immersed in the liquid medium comprising acidic, neutral and base solutions. Two chosen acidic solutions were the D-limonene and acetic acid, while sodium hydroxide (NaOH) and sodium chloride (NaCl) were selected for base solutions. A high-speed photography system with femtosecond laser and CCD camera was used to capture the mechanism involving the crumb rubber production. This technique was successfully conducted in producing finer crumb rubber particles. The shock wave and water jet from the cavitation bubble were identified as the main mechanisms responsible for the fragmentation of rubber surface after interacting with laser. The highest concentration of crumb rubber (1.97 g ml^{-1}) was achieved in acetic acid solution generated with 300 mJ laser pulse at repetition rate of 3 Hz. Ablation of rubber sample in NaOH solutions produced crumb rubber particles with average diameter about 12000 mesh (1.44 µm) which is smaller than the average size of crumb rubber produced from conventional techniques. The energy-dispersive X-ray spectroscopy analysis indicated that crumb rubber consists of carbon, oxygen, zinc, sulphur and silicon which is similar to the element composition of the target sample. Results from field-emission scanning electron microscope showed that the generated crumb rubber particles have irregular shape. The crumb rubber was then immersed into cooking oil and illuminated by a diode pumped solid state laser to measure the oil absorption efficiency. The absorption was found to be dependent on two main parameters namely the crumb rubber size and the time interaction. Crumb rubber particles with average diameter 10, 60 and 3650 mesh were tested, and the measured absorption coefficients were 485 m^{-1}, 769 m^{-1} and 2906 m^{-1} respectively. This result shows that smaller crumb rubber particles have higher absorption coefficients. In conclusion, controllable, safe and environmental friendly technique of crumb rubber production based on PLAL is a promising technique to be used in oil spill recovery.

ABSTRAK

Teknik penghasilan getah serbuk novel yang merangkumi teknologi laser diperkenalkan sebagai alternatif kepada teknik pemprosesan mekanikal pengisaran dan kriogenik. Teknik ini menawarkan penyelesaian kepada kelemahan utama teknik sedia ada daripada segi saiz zarah getah serbuk. Keberkesanan penggunaan getah serbuk yang dihasilkan sebagai penyerap minyak digunakan dalam aplikasi pemulihan tumpahan minyak telah diterokai. Laser denyut Nd:YAG nano-saat dengan panjang gelombang 1064 nm digunakan sebagai sumber cahaya dalam ablasi laser denyut dalam teknik cecair (PLAL). Sasaran getah yang digunakan dalam kajian ini ialah sekeping tayar skrap yang direndam dalam medium cecair yang terdiri daripada larutan berasid, neutral dan alkali. Dua larutan asid yang dipilih ialah asid D-limonena dan asid asetik, manakala natrium hidroksida (NaOH) dan natrium klorida (NaCl) dipilih untuk larutan alkali. Sistem fotografi berkelajuan tinggi dengan laser femto-saat dan kamera CCD digunakan untuk merakam mekanisme yang terlibat dalam penghasilan getah serbuk. Teknik ini telah berjaya dijalankan bagi menghasilkan zarah getah serbuk halus. Gelombang kejutan dan jet air daripada gelembung kavitasi dikenalpasti sebagai mekanisme utama yang bertanggungjawab dalam pemecahan permukaan getah selepas berinteraksi dengan laser. Kepekatan tertinggi getah serbuk (1.97 g ml^{-1}) dicapai dalam larutan asid asetik yang dijanakan dengan laser denyut 300 mJ pada kadar pengulangan 3 Hz. Ablasi sampel getah dalam larutan NaOH menghasilkan zarah getah serbuk dengan diameter purata sekitar 12000 mesh (1.44 μm) iaitu lebih kecil daripada saiz purata getah serbuk yang dihasilkan dengan teknik konvensional. Analisis spektroskopi sinar-X tenaga terserak menunjukkan bahawa getah serbuk terdiri daripada karbon, oksigen, zink, sulfur dan silikon yang sama dengan komposisi elemen sampel sasaran. Hasil daripada mikroskop elektron imbasan medan menunjukkan bahawa zarah getah serbuk yang dijana adalah dalam bentuk tak sekata. Getah serbuk itu kemudiannya direndam ke dalam minyak masak dan disinari dengan laser keadaan pepejal berpam diod untuk mengukur kecekapan penyerapan minyak. Penyerapan itu didapati bergantung kepada dua parameter utama iaitu saiz getah serbuk dan masa interaksi. Zarah getah serbuk dengan diameter purata 10, 60 dan 3650 mesh diuji, dan pekali penyerapan yang diukur masing-masing adalah 485 m^{-1}, 769 m^{-1} dan 2906 m^{-1}. Keputusan ini menunjukkan bahawa zarah getah serbuk yang lebih kecil mempunyai pekali penyerapan yang lebih tinggi. Sebagai kesimpulan, teknik pengeluaran getah serbuk yang terkawal, selamat dan mesra alam berdasarkan PLAL adalah satu teknik yang berpotensi untuk digunakan dalam pemulihan tumpahan minyak.

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LIST OF ABBREVIATIONS

CR	-	Crumb Rubber
PLA	-	Pulsed Laser Ablation
PLAL	-	Pulsed Laser Ablation in Liquid
FESEM	-	Field Emission Scanning Electron Miscroscope
EDX	-	Energy Dispersive X-ray Spectroscopy
NR	-	natural rubber
IR	-	synthetic polyisoprene
BR	-	polybutadiene
EPDM	-	Ethylene-propylene-diene monomer rubber
SBR	-	styrene-butadiene rubber
NBR	-	acrylonitrile butadiene rubber
UV	-	ultraviolet
PBN	-	N-phenyl-b-naphthylamine
CO2	-	carbon dioxide
SO2	-	sulphur dioxide
TALC	-	tire-added latex concrete
SEM	-	scanning electron microscope
PCRC	-	portland cement rubber concrete
MOCRC	-	magnesium oxychloride cement rubber concrete
TRA	-	tire-rubber ash
MPa	-	Mega Pascal
TDF	-	tire-derived fuel
GTR	-	ground tire rubber
RR	-	pre-devulcanized tire rubber
DD	-	Diphenyl Disulfide
FTIR	-	Fourier Transform Infrared Spectroscopy

PTFE	-	polytetrafluoroethylene
LLDPE	-	Linear Low-Density Polyethylene
IR	-	infrared lasers
Nd:YAG	-	Neodymium doped yttrium aluminium garnet
LCD	-	liquid crystal display
CCD	-	charge couple device
NaOH	-	sodium hydroxide
NaCl	-	sodium chloride
RPM	-	rounds per minute

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CHAPTER 1

INTRODUCTION

1.1 Overview

The volume of scrap tire wastes is increasing at a fast rate. An estimated 1000 million tires reach the end of their useful lives every year and 5000 million more are expected to be discarded in a regular basis by the year 2030. In America, more than a quarter of a billion tires is thrown out annually [1,2]. By the year 2030 the number of tires from motor vehicles is expected to reach 1200 million representing almost 5000 million tires to be discarded in a regular basis.

One of the biggest obstacle in waste management is the safety of scrap tires disposal system. End of life tires is a global issue which raise severe environmental problems and must be disposed of or recycled. Due to lack of effective disposal way, scrap tires prolong to be a main source of waste. Due to their cross-linked structure, they do not melt or dissolve. It is impossible to expulse the rubber tires in the environment because they require long time to start decaying and increase the amount of pollution. So, it is required to have an appropriate use of these waste scrap tires.

Conventionally, some of the tires are recycled and the others are just stockpiled (whole tire), landfilled (shredded tyre) or buried. Mainly, waste tires disposal areas lead to the decrease of biodiversity and increasing the amount of toxicity towards the environment [2]. Buildups of waste tires are dangerous because they can lead to a potential environmental pollution, fire hazards and potential breeding grounds for deadly mosquitoes that transmit disease. Conventional way disposing scrap tires are usually done by burning them in a large fires, however this method release pollutant which may endanger humans, wildlife and the environment [3]. Tire pile fires have been bigger environmental problem, that can be burn for long time up to months, distribute up a black plume that can be seen for away. That plume contains toxic chemicals and air pollutants, thus producing an oil that will contaminate soil and water. Still millions of tyres are just being buried all over the world.

A large percentage of these tires are sent to landfills where they can contribute to the spread of diseases by becoming breeding grounds for rodents and mosquitos. Tire landfilling is in control for a serious environmental risk. This phenomenon will contribute directly to pollutant, while wasting tremendous amount of rubber that could have been recycled. More importantly, waste rubber from tire will take longer time to naturally degrade because of the sulphur cross-link that presence in the compounds [4].

Recycling tire is an innovative idea to avoid the environmental problem from rising. It is a process of reusing automobiles tires that are no longer appropriate for use on vehicles due to wear or permanent damage such as punctures. To reduce the amount of scrap tires waste, waste rubber is incorporated into products that have extensive demand and applications.

A practical solution to this problem is to recycle the crumb rubber (CR). Crumb rubber is a granulated material derived from the scrap tires and then is used to develop other valuable products. The three mains component contains in scrap tires are fibers, steel belts and tread rubber. During scrap tires recycling process, rubbers are being separated from fibers and steel belts by mechanical separation method. The rubber later is being shredded into smaller pieces by motor blades. Cryogenic processing will further reduce the size of the rubber into fine-size particles. The cracker mill process tears apart or reduces the size of tire rubber by passing the material between rotating corrugated steel drums. By this process an irregularly shaped torn particle having large surface area are produced that known as crumb rubber.

Improvements of mechanical and dynamical properties can be achieved with addition of rubber in the concrete. Additional energy absorption, better crack resistance and better ductility are some of the concrete properties that being improved. By using the crumb rubber, one can decrease the destructive effect towards environment as well as providing a sustainable concrete. Due to alarming environmental issues, utilization of waste from industrial product in construction sector has gained much attention globally.

Based on literature, it is found that tire rubber wastes are being utilized for paving purposes [5]. Another usage of tire rubber wastes is in the area of artificial reef, however some research and investigations have questioned the validity and impact of it in many aspects [6]. Other than that, it has been reported that tire rubber can also be utilized to produce carbon black through pyrolysis technique [7]. In the absence of oxygen, thermal decomposition of the tire will produce numbers insignificance by-product. Some researchers have already been carried out on the utilization of tire rubber waste as alternative aggregate in many types of concretes. This type of aggregate will enhance the concrete toughness and improvise the sound insulation properties of the materials. Technically, rubber aggregates are produced from waste tire by using two different technology namely mechanical and cryogenic grinding. In the first method, waste tire will undergone controlled grinding at ambient temperature to produce chipped rubber that is used to replace coarse aggregates. As for the second method, the grinding of waste tire took place below the glass transition temperature, which further yields smaller crumb rubber to be used as aggregates.

Fattuhi *et al.*, [8] in his investigation on rubber concrete report that the concrete made by low quality rubber will suffer poor comprehensive strength in comparison with the higher grade of rubber concrete. Another similar observation is also reported by Topcu [9] which conclude that this phenomenon is actually originated from the weak interfacial bonds between the tire rubber and the cement paste. Tarun *et al.*, [10] in his work has reported that compressive strength of any rubberized concrete could be further improved by replacing aggregates with finer crumb rubber particles. Crumb rubber that undergone pre-treatment will significantly improvised the bonding between the particles and the surrounding matrix, which results in better compressive strength. In another work, Piti *et al.*, [11] report that the utilization of crumb rubber in concrete will further improve the toughness and flexibility of the concrete samples, as well as having higher fracture energy and longer post-peak load response.

In general, waste tire disposal poses great challenge throughout the world. It is hardly surprising that the responsible bodies prefer stockpiling and open burning as disposal option, which pose alarming threat to environmental and health. Thus, it is urged to make more use of the scrap tire waste. Numbers of researches have been carried out in this area, with majority of the works focussing on the emerging technology to process the recycled rubber for many purposes.

In this work, another successful technique for producing crumb rubber is carried out by pulsed laser ablation (PLA) as an alternative method. PLA also can be produced in liquid surrounding (PLAL). Briefly, this method focuses a pulsed laser beam onto the target material surface inside liquid. This will create an ablation plume from the interaction of the laser pulse with both the target and the liquid environment. The experimental setup used for obtaining crumb rubber and the monitoring process is presented in this thesis.

The ablation plume consists of small amounts of evaporated liquid forming micro-bubbles and some melted target material. The micro-bubbles will expand until certain critical combination of temperature and pressure is reached, resulting in the collapse of the bubble structure. During the destruction of the bubble, the material particles inside it experience an extreme temperature and pressure, leading to the creation of a new material. This method provide certain advantages, such as the possibility large quantity of target to evaporate and the final product usually having a form of finer particles remaining suspended within the host liquid. In comparison with mechanical crumb rubber production, the PLAL method is controllable, safer and environmental friendly.

1.2 Problem Statement

The crumb rubber industry is considered to be in its adolescent evolutionary stage, struggling with significant challenge to reach its maturity. There are numbers of different manufacturing process; two of the most common techniques are ambient grinding and cryogenic processing which suffers notable drawback in terms of maintenance and size of particle production. No doubt that mechanical technique can product in a big scale however there are still limitation that cannot be solved by grinding machine. Currently by using mechanical technique through grinding can be achieved up to 40 mesh only as reported by Revocomm Technologies Sdn Bhd. There is still indeed need another technique that can solve for producing a finer crumb runner. To throw some light on this matter, pulse laser ablation in liquid is proposed. Therefore in this project two type of laser that are a Q-switched Nd:YAG and a femtosecond laser are used to produce finer crumb rubber. Although it might have limitation in producing in a large scale at least it can solve the problem in size of crumb rubber.

1.3 Objectives of the Study

The main objective of this study is to synthesize finer crumb rubber by using pulse laser ablation in liquid technique. In order to achieve this goal, the following tasks are established:

i. To synthesize crumb rubber by pulse laser ablation technique incorporating Q-switched Nd:YAG laser and Femtosecond Laser.
ii. To set-up high-speed photography technique to determine the mechanisms responsible to induce finer crumb rubber.
iii. To characterize and optimize the crumb rubber generated in different liquid mediums by EDX, FESEM and FTIR.
iv. To determine the optical absorbance of different sizes of crumb rubbers in oil suspension.

1.4 Scope of Study

In this study, the scrap tyre was chosen as the main material to produce crumb rubber through pulse laser ablation which obtained from Revocomm Technologies Sdn Bhd. Others preferred material is the chemical solution like D-limonene, Acetic acid, De-ionized water, Sodium Hydroxide and Sodium Chloride were used as medium for synthesize. Two sources of ablation were used that are a Q-switched Nd:YAG laser with 1064 nm wavelength and 10 ns pulse duration and a femtosecond laser with 1064 nm wavelength and 340 fs pulse duaration. In order to understand the mechanism responsible for inducing crumb rubber, high speed photography system using shadowgraph technique was developed to capture the shockwave, cavitation bubble and liquid jet formation that occur during the phenomena. The crumb rubber obtained from the laser ablation technique was analysed using FESEM and EDX. Finally, the crumb rubber particles obtained were used to determine the oil absorbent efficiency.

1.5 Significances of this Study

A finer crumb rubber is produced by using laser technology. The size can be achieved down to 3000 mesh. This is a huge contribution for industrial application. This type of finer crumb rubber will enhance the toughness and improvise the sound insulation properties of building materials like concrete. Beside finer crumb rubber also have big contribution as an absorbance material because of large surface area especially in spill oil industry.

1.6 Thesis Outline

This thesis documented the complete work of this research. It consists of five main chapters whereby, in every chapters are divided into several subchapters. Chapter 1 present the overview of the thesis, problem statement, research objectives, scope of study as well as significance of this work. As in Chapter 2, it provides extensive literature review of crumb rubber production by previous researchers including current problems on the technique. While Chapter 3 describes detail information regarding the instruments and description of sample properties which has been used in this work. Furthermore, it also discusses the calibration procedure and research methodology of system development. All the results are presented in Chapter 4. The initial work comprised of system calibration. Then followed by the mechanism involve in producing crumb rubber, maximizing the crumb rubber production based on laser key parameter, under effect of different pH solution and under effect of the high repetition rate laser. Finally, Chapter 5 concluded the finding of this study. It also contained the recommendation for future study.

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