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DETECTION OF HYDROCARBON LEVEL IN DISTILLED WATER USING

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A thesis submitted in fulfilment of the requirements for the award of the degree of Doctor of Philosophy (Physics)

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To:

My parents and my beloved family

تقدیم به پدر بزرگوار و مادر مهربانم

آن دو فرشته ای که از خواسته هایشان گذشتند، سختی ها را به جان خریدند و خود را سپر بلای مشکلات و ناملایمات کردند، تا من به جایگاهی که اکنون در آن ایستاده ام برسم.

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ABSTRACT

Oil spill occurs almost every day. Department of chemistry Malaysia (JKM) took almost a week or more days to analyze it. Therefore alternative technique should be considered. In this work, a new technique is introduced by using laser technology and piezoelectric transducer. The system is known as Laser Induced Acoustic (LIA). Lube oil was used as a hydrocarbon sample. Distilled water and hydrochloric acid were employed for solution preparation with different concentrations in the range of 0 - 1000 ppm. Hydrocarbon became impurities in the solution, which can be observed via CCD video camera after illumination by diode pumped solid-state laser (DPSS). Refractive index of hydrocarbon solution was measured by He-Ne laser following Snell's law. A Q-switched Nd:YAG laser was focused to induce optical breakdown and shock wave generation. This phenomenon was recorded via high-speed photography system. Dye laser pumped by nitrogen laser was employed as a source of flashlight. Digital delay generator was deployed to synchronize both lasers. CDD camera was interfaced with personnel computer with Matrox version 9 software, which was used to record shock wave. Silicone photodiodes were employed to detect both lasers. Optical delay between two lights represented the frozen time of shock wave generation. The time delay was manifested via digital oscilloscope. Shock wave propagation in hydrocarbon solution was also detected via piezoelectric transducer. The sound signal was also displayed on the same oscilloscope. The sound amplitude (volt) was calibrated via hydrometer to estimate shock wave pressure (atm). Shadowgraph image of shock wave was analyzed via ImageJ software. Shock wave radius was measured and divided by optical delay to determine sound speed in hydrocarbon solution at different concentration. Observation result showed that sound speed linearly increases with hydrocarbon concentrations. Similarly sound amplitude was found linearly increasing with hydrocarbon concentrations. This is due to a lot of mass transfer which gives rise to high impact to the transducer. Combination of high-speed photography and transducer detection validate the shock wave as the mechanism to determine the hydrocarbon concentration. Hence sound speed is the fingerprint for every hydrocarbon solution. Furthermore sound speed has linear relationship with hydrocarbon concentration. Similarly the sound amplitude has linear relationship with hydrocarbon concentration. This similarity indicates that the hydrocarbon concentration can be detected based on sound generation via laser induced acoustic technique.

ABSTRAK

Tumpahan minyak berlaku hampir setiap hari. Jabatan Kimia Malaysia (JKM) mengambil masa hampir seminggu atau lebih untuk menganalisisnya. Oleh itu, teknik alternatif perlu dipertimbangkan. Satu teknik baru diperkenalkan dengan menggunakan teknologi laser dan transduser piezoelektrik. Sistem ini dikenali sebagai akustik beraruh laser (LIA). Minyak pelincir telah digunakan sebagai sampel hidrokarbon. Air suling dan asid hidroklorik digunakan untuk menyediakan larutan dengan kepekatan dalam julat 0 ke 1000 ppm. Hidrokarbon menjadi benda-asing dalam larutan dan dapat diperhatikan melalui kamera video CCD yang disinari dengan laser keadaan pepejal berpamkan diod. Indeks biasan larutan hidrokarbon diukur dengan menggunakan Laser He-Ne dan mengikut hukum Snell. Laser Nd:YAG bersuis-Q difokuskan untuk membentuk runtuhan optik dan menjana rambatan gelombang kejutan. Fenomena ini dirakamkan dengan sistem fotografi kelajuan tinggi. Laser pencelup yang dipam oleh laser nitrogen digunakan sebagai lampu kilat. Penjana tundaan masa digital digunakan untuk menyerentakan keduadua laser tersebut. Kamera video CCD yang dihubungkan dengan komputer peribadi melalui perisian MATROX versi 9 telah digunakan untuk merakamkan gelombang kejutan. Fotodiode silikon digunakan untuk mengesan kedua-dua laser. Tundaan masa optik antara kedua-dua cahaya ini mewakili masa pembekuan rambatan gelombang kejutan. Masa tundaan ini dipaparkan pada osiloskop digital. Gelombang kejutan dalam larutan hidrokarbon juga dikesan melalui transduser piezoelektrik. Isyarat bunyi ini dipaparkan dalam osiloskop digital yang sama. Amplitud bunyi (volt) telah dikalibrasi menggunakan hidrometer untuk menganggarkan tekanan gelombang kejutan (atm). Imej geraf bayangan gelombang kejutan dianalisis menggunakan perisian ImageJ. Jejari gelombang kejutan diukur dan dibahagikan dengan masa tundaan optik untuk menentukan halaju bunyi dalam larutan hidrokarbon pada kepekatan yang berbeza. Hasil pemerhatian menunjukkan halaju bunyi bertambah dengan kepekatan hidrokarban. Begitu juga amplitud bunyi bertambah dengan kepekatan hidrokarbon. Ini disebabkan oleh pemindahan jisim yang banyak memberi impak yang tinggi pada transduser. Gabungan sistem fotografi kelajuan tinggi dan pengesanan transduser mengesahkan bahawa gelombang kejutan adalah mekanisma penentu kepekatan hidrokarbon. Oleh itu halaju bunyi menjadi cap jari bagi setiap larutan hidrokarbon. Tambahan pula halaju bunyi mempunyai hubungan linear dengan kepekatan hidrokarbon. Begitu juga amplitud bunyi mempunyai hubungan linear dengan kepekatan hidrokarbon. Penyamaan ini menunjukkan bahawa kepekatan hidrokarbon boleh dikesan berdasarkan bunyi yang dijanakan melalui teknik akustik aruhan laser (LIA).

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LIST OF ABBREVIATIONS

AP	-	Amplified Photodiode
API	-	American Petroleum Institute
CCl4	-	Carbon Tetrachloride
CCD	-	Charge-Coupled Device
CFC	-	Chlorofluorocarbon
DOP	-	Delayed-Output Pulse
DPSS	-	Diode Pumped Solid-State
DDG	-	Digital Delay Generator
EB	-	Expanded Beam
GCMS	-	Gas Chromatography Mass Spectrometer
He-Ne	-	Helium-Neon
HCL	-	Hydrochloric Acid
JKM	-	Jabatan Kimia Malaysia
LIA	-	Laser Induced Acoustic
LLPG	-	Liquid-Liquid Partition-Gravimetric
NIR	-	Near Infrared
Nd: YAG	-	Neodymium-Doped Yttrium Aluminum Garnet
NDT	-	Nondestructive Testing
ND	-	Nitro-Dye
ОТ		Opthothermal
Osc	-	Oscilloscope
ppm	-	Parts Per Million
PA	-	Photoacoustic
PD	-	Photo Detector

PZT	-	Piezoelectric Transducer
SNR	-	Signal To Noise Ratio
FTIR	-	Transform Infrared Spectrometry
UV	-	Ultra Violet

LIST OF SYMBOLS

(atm)	-	Atmosphere
a.u	-	arbitrary unit
cm	-	Centimeter
°C	-	Centigrade
gr	-	Gram
Hz	-	Hertz
J	-	Jules
MHz	-	Mega hertz
m	-	Meter
ml	-	Milliliter
μl	-	Microliter
ms	-	Millisecond
mm	-	Millimeter
ns	-	Nanosecond
μs	-	Microsecond
mJ	-	Milli jules
nm	-	Nanometer
Ра	-	Pascal
ppm	-	Part per million
S		Second
V	-	Voltage
W	-	Watt
λ	-	Wavelength

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CHAPTER 1

INTRODUCTION

1.1 Overview

Oil poses a range of environmental risks and causes wide public concern when released into the environment, whether as catastrophic spills or chronic discharges [1]. An oil spill is the release of liquid petroleum hydrocarbon into the environment, especially marine areas, due to human activity, and is a form of pollution. Oil spills may be due to releases of crude oil from tankers, offshore platforms, drilling rigs and wells, as well as spills of refined petroleum products (such as gasoline and diesel) and their by-products, heavier fuels used by large ships such as bunker fuel, or the spillage of any oil refuse or waste oil [2]. Cleanup and recovery from an oil spill is difficult and depends upon many factors, including the type of oil spilled, the temperature of the water (affecting evaporation and biodegradation), and the types of shorelines and beaches involved. Spills may take weeks, months or even years to clean up. Therefore, to unambiguously characterize, identify, categorize, and quantify all sources of hydrocarbons entering the environment is important. It is crucial for the environmental damage assessment; the evaluations of the relative risks to the ecosystem posed by each spill, and for selecting the appropriate spill response and taking effective cleanup measures.

The usual method uses the liquid-liquid separation technique to determine quantitatively the amount of oil dispersed at sea [3-7]; However the separation

technique has certain constraints, including being time consuming, only being performed in a laboratory, and using hazardous chemicals such as N-hexane and Freon, which are carcinogens. The recent Fourier transform technique too has the same constraints [8]. That is why scientists seek a fast and easy in situ technique to determine amounts of oil spilled at sea. The high power laser is one of the best candidates to be used to solve this problem. A Q-switched laser is able to induce an acoustic shock wave, which has high potential for transient detection techniques. Nanosecond- pulsed breakdown in a liquid phase has wide application in many other fields [9-14].

The Laser-Induced Acoustic (LIA) is a very sensitive technique to apply for a wide range of applications. Laser Induced Acoustic is part of a family of optothermal techniques (OT), which are based on the conversion of optical into mechanical energy. Especially for the solid and liquid media, the generating and detecting of acoustic waves accrued using a short pulse laser-based technique. This technique was demonstrated as an important tool and used for many applications such as in the medical areas and applied sciences. Furthermore, this technique can be used for material characterization [15, 16], surface cleaning from the contaminations [17], laser tissue ablation, corneal sculpting [18] and indirect gall stone fragmentation [19]. The rapid heating, thermoelastic expansion, and phase change occur during the interaction between the laser and surface of solid media, while the interaction between the laser and liquid media, emission of a strong ultrasonic or shock wave would occur [20, 21].

1.2 Problem Statement

Pollution and its control are very important and effecting issues on human life. One of the crucial materials to be controlled from being polluted in human life is water. Thus one of the factors among many pollutant factors is hydrocarbon in water.

In Jabatan Kimia Malaysia (JKM), at Bahagian Alam Sekitar, a large number of samples from various organization either government or private sectors received daily for analysis. JKM provides the chemical analysis services (Appendix A) from liquid to solid form in different conditions. The analysis is divided in two parts; control analysis and enforcement analysis.

For the controlling part, they analyze the specific parameters in order to confirm that the samples are obliging with acts and regulations as stated in Act 127 "Environmental Quality Acts and Regulations". They perform this analysis monthly for the monitoring purposes. While for the enforcement analysis, the samples undergo specific tests in order to determine specific parameters depending on the needs. The samples can be sewage, discharged water from factory, and fire debris. The samples of seawater received by JKM, is to determine the level of Hydrocarbon. The chemical techniques known as liquid-liquid Partition-Gravimetric (LLPG) techniques, Gas Chromatography Mass Spectrometer (GCMS), and Fourier Transform Infrared Spectrometry (FTIR) are used.

In LLPG technique, liquid samples such as seawater and river water undergo the digestions process. Usually, the purpose of this technique is to separate the hydrocarbons from water. The digestion process takes approximately 24-36 hours. Finally, the concentration can be measured by using mass technique as follow:

Concentration = (Final mass - initial mass) / volume(1.1)

In this technique, the used solvent such as, carbon tetrachloride (CCl4), and

chlorofluorocarbon (CFC) are harmful to the environment according to the welldocumented ozone layer depletion. Then, the GCMS is used to determine the type of hydrocarbons by separating the chemical bonding in accordance to the mass percentage; however, GCMS requires the sample in gas form. Then, the technique namely, FTIR spectrometer is being used to determine the type of sample either water or oil. The disadvantages of the aforementioned technique in addition to the harmfully for the environment; it is also a time consuming technique and labor intensive. Therefore, other alternative technique needs to be considered to reduce the processing time as well as the chemical application. Therefore LIA was proposed in order to reduce the limitations of chemical method.

1.3 Research Objectives

This main objective of this research is to develop a new method namely Laser Induced Acoustic (LIA) technique to determine the hydrocarbon level in water. In attempts to achieve this goal the following tasks are established:

- 1. To observe and characterize the shock wave properties in different hydrocarbon concentration
- 2. To detect the acoustic signal in different hydrocarbon concentration
- 3. To correlate between the acoustic properties and the concentration of the hydrocarbon.

1.4 Scope of the study

In this study, Lube oil was used as hydrocarbon sample. Distilled water was used as solution instead of seawater, because hydrocarbon is assumed to be as contamination in water. The hydrocarbon concentration was studied in the range of 0-1000 ppm. A Q-switched Nd:YAG laser was deployed to induce optical breakdown associated with acoustic shock wave generation. Dye laser pumped by nitrogen laser was used as a flash of light to illuminate the shock wave propagation. A digital delay generator was used as a synchronizer unit. High-speed photography technique was used to grab high-speed phenomena. CCD video camera was used to record the acoustic shock wave via Matrox version 9 software. He-Ne laser was used to measure refractive index following the Snell's law. Diode pumped solid-state laser (DPSS) were utilized to characterize the hydrocarbon solution properties. Piezoelectric transducer was used to display the acoustic signal as well as the optical delay. Hydrometer was used to calibrate the transducer.

1.5 Significant of the study

A new technique that is laser induced acoustic shock wave was introduced to detect the level of hydrocarbon in water. The level of hydrocarbon is expected to be determined based on the knowledge either the speed of sound of the pressure of the acoustic signal. The proposed technique has a potential to be used for detecting the hydrocarbon level in water based on a green technology. It is faster, clean and environmentally friendly.

1.6 Thesis outline

This thesis is consisting of five chapters. Chapter 1 provides an overview on the hydrocarbon detection using current conventional methods such as physical and chemical methods and the necessity of new method for hydrocarbon detection in water. The research problem statement, objectives, scope and significance of this study are described in this chapter. The literature review of previous research of oil detection and techniques of detections are described in details in Chapter 2. This chapter tries to highlight a relationship between the hydrocarbon detection and laser induced acoustic. Chapter 3 presents in detail the methodology of sample preparation, system characterizations, calibration and data analysis. The method used for signal detection, high-speed photography system is explained in this chapter. Chapter 4 shows the results and discussions. This is also includes the shockwave captured by high-speed photography system and acoustic signal detection, hydrocarbon impurities in water and optical properties of media. Chapter 5 concludes the significant findings and further recommendations on this diversified research possibility. The limitations related with our investigation are highlighted.

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