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Characterization and Identification Compounds of Liquid Smoke from Used Tire Waste Pyrolysis

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Abstract

Research on compounds from liquid smoke resulting from the pyrolysis of used tire waste has been conducted. The results of tire pyrolysis produce liquid hydrocarbons and charcoal which can be utilized and developed into more useful materials or finished goods. This study aims to identify the compound components of liquid smoke resulting from the pyrolysis of used tire waste. The research begins with manufacturing a pyrolysis tool based on the principle of dry distillation. The pyrolysis process is carried out by heating at high temperatures. Liquid smoke analysis was carried out using GC-MS. In contrast, the components of the compound were identified and characterized by comparing the mass spectrum data obtained with the mass spectrum data in the GC-MS library. The results showed that the pyrolysis of 6 kg of used tires produced 1500 mL of liquid smoke. Analysis of liquid smoke by GC-MS produced 88 chromatogram peaks, with the most dominant peak height at peaks 3, 17, 37, 38, 72, and 73. The most dominant compound component identified by MS spectrum analysis is 2-propanone (peak 3), 4-methyl-2-pentanone (peak 17), ethyl-benzene (peak 37), 1,2-dimethyl-benzene (peak 38), 1-methyl-4-(1-methyl ethyl)-benzene (peak 72) and limonene (peak 73).

Keywords: Pyrolisis, liquid Smoke, used Tire, GC-MS, destillation

INTRODUCTION

Tire production in Indonesia is increasing in line with the growth of the automotive industry. Tires used for motor vehicles are manufactured from synthetic rubber of the polystyrene type. In developing countries, used tire waste is a very common problem and is a solid waste that is harmful to the environment. The accumulation of used tire waste can become a breeding ground for mosquitoes and a source of disease. Tires take a long time to degrade naturally because tires are made of rubber in the form of CxHy, which has a high carbon and volatile matter content. This condition creates severe environmental problems if used tires are unused (Arita, Assalami, & Naibaho, 2015). Syamsiro, Suliastiwati, Ridwan, & Dwicahyo (2016) stated that with an average tire life of 5 years, it could be estimated that millions of used tires are wasted and have the potential to disturb the environment. These tires will pollute the surrounding environment because used tires cannot decompose easily if left alone. Therefore, it is necessary to make an effort to convert used tires into something more useful. Rubber-based tires are a type of synthetic polystyrene polymer (polystyrene). Polystyrene cannot be easily recycled so polystyrene waste treatment must be done correctly so as not to harm the environment (Damayanthi & Martini, 2009).

Craftsmen of ropes, chairs, pots, mats, industrial fuel, and others use used tire waste. In addition, it can be converted by pyrolysis to help tackle the problem of used tire waste. Pyrolysis is to obtain liquid hydrocarbons (liquid smoke) and charcoal (carbon black), which can be utilized and developed into more useful materials or finished goods products to substitute raw materials. Based on petroleum or other chemicals (Alam, 2023). Liquid smoke from the pyrolysis of used tires can be a source of essential chemicals. In addition, judging from its composition, it also has the potential to be developed into fuel. The composition of the tire components varies according to the specifications set by the manufacturer. In general, the composition of tires is Carbon, Hydrogen, Oxygen, Nitrogen, Sulfur, and Ash (Falaah, Cifriadi, & Maspanger, 2013).

The used tires used in this study is expected to reduce waste by substituting finished goods for petroleum-based materials or other important chemicals. This innovation is a breakthrough in making materials derived from materials that have only been wasted so far (Falaah & Cifriadi, 2012).

Used tire degradation can be done through a cracking process. The cracking process can be done through pyrolysis with or without a catalyst at high temperatures. One alternative technology that has added value and has been developed to reduce the volume of waste, especially rubbish, includes recycling technology and thermal transformation technology, namely by thermal degradation through a pyrolysis process. The pyrolysis is an alternative waste treatment that is considered quite prospective to be developed because it has several advantages, including having a high conversion ratio and its products having a high energy content. They can be upgraded to become basic materials for other needs and have easier process control when compared to conventional waste processing others (Damanhuri, Nurtanto, Soffan, & Sulaeman, 2017). The parameters that affect the speed of the pyrolysis reaction have a very complex relationship. Pyrolysis products are affected by temperature, time, heating rate, and catalyst presence. Pyrolysis at various temperatures is useful for knowing the best product to produce in the pyrolysis process (Mukharoma, 2017). This study aims to identify and characterize the components of liquid smoke from used tire pyrolysis.

METHODOLOGY

Materials and Instrumentals

The materials are waste tires. The Laboratory tools used are a pyrolysis kit, funnel, analytical balance, filter paper, beaker, aluminum foil, glassware, Erlenmejer flask, tire grinder/cutter, Gas Cromathography-Mass Spectrophotometer (GC-MS) SHIMADZU QP2010S.

Methods

Pyrolisis Process

The series of pyrolysis equipment used is shown in Figure 1. Pyrolysis equipment consists of (1). Pyrolysis reactor equipped with cover, thermometer, and barometer (2). The pyrolysis furnace is equipped with a gas stove (3). Cover (4). Barometer (5). Thermometers (6). Smoke distribution pipe (7). Tar container (8). Water container for cooling (9). Spiral pipe/conductor and smoke cooler (10). Container support (11). Liquid smoke container.

Used tires were cut into 5 cm x 5 cm pieces, weighed as much as 6 kg, and put into the pyrolysis reactor. The pyrolysis reactor and other pyrolysis equipment are assembled as shown in Figure 1, then heated at a high temperature (room temperature up to 450 °C) using a gas stove for 5 hours (until the liquid smoke stops dripping) in the liquid smoke distribution pipe.

Figure 1. Pyrolisis Equipment

The pyrolysis process is carried out in a pyrolysis reactor. The pyrolysis process ends when no more liquid smoke drips down the liquid smoke distribution pipe. Pyrolysis products in liquid smoke and charcoal are determined by the conversion percentage and product distribution (Arita et al., 2015); (Lombok, Setiaji, Trisunaryanti, & Wijaya, 2014).

Characterization and Identification of Liquid Smoke

GC-MS analyzed the liquid smoke obtained. Characterization and identification of compound components are carried out by analyzing spectrum data, namely by interpreting the resulting liquid smoke spectrum data and comparing it with spectrum data in the literature and spectrum data in the GC-MS library (Lombok et al., 2014; Lombok, Setiaji, Trisunaryanti, & Wijaya, 2012).

RESULTS AND DISCUSSION

Results of Used Tire Waste Pyrolysis

The used tires used in this study before being pyrolyzed were cut into pieces with a size of approximately 5x5 cm so that they had a large surface area and could be easily put into the pyrolysis reactor/furnace. The results are shown in Figure 2a. After being cut into pieces, samples were weighed and put into the pyrolysis reactor (Figure 2b). After then, the other pyrolysis equipment was carefully

assembled and arranged so that the smoke/volatile substances were not released outside the pyrolysis reactor system and cooling when the heating process was carried out until liquid smoke was obtained as desired.

a b

Figure 2. (a). Used tires that have been cut into pieces, (b). Used tires in a pyrolysis reactor

Pyrolysis of 6 kg of used tires produces 1500 mL of liquid smoke equivalent to 25% of the initial sample weight used, as shown in Figure 3. The pyrolysis lasted for 5 hours, and the pyrolysis temperature reached 450 ºC. The characteristics that appear during the pyrolysis of used tires are: during the pyrolysis process, droplets of liquid smoke occur when the temperature of the thermometer shows 200 ºC. The liquid smoke drips continuously without stopping, accompanied by an increase in pyrolysis temperature, as indicated by the increase in the thermometer scale placed on the lid of the pyrolisator reactor.

In addition, it also appears that in the pyrolysis process, liquid smoke dripping below the thermometer scale of 270 ºC appears clear (yellowish) in color, followed by a gradual change in the color of the liquid to blue-green at temperatures above 270 ºC. The pyrolysis process lasted for 5 hours, and the heating time until the initial dripping (temperature 200 ºC) was around 1 hour, while the absence of dripping liquid smoke indicated the end of the pyrolysis process. Liquid smoke dripping through the pipeline lasts 4 hours, while the temperature at the end of the pyrolysis process reaches 450 ºC. In this study, besides producing liquid smoke, 2.7 kg of charcoal, or about 45% of the total used tires, was also produced, and 240 grams of tar, or 4% of the total used tires, were produced.

The resulting pyrolysis products show that the total yield of liquid smoke and tar is 29%. In comparison, the solid product in the form of charcoal is 45%, which indicates that in used tires, there is a volatile component of 29% which turns into pyrolysate. The total yield of liquid smoke, tar, and charcoal is 74% (45% charcoal + 29% liquid smoke and tar), so around 26% of the gas is lost or not condensed. In this pyrolysis process, the percentage of gas that is not condensed is still relatively high, so it is necessary to design a pyrolysis reactor that can accommodate very light and non-condensable gases so that no product is wasted.

Figure 3 Liquid smoke from pyrolysis of used tire waste

The release of used tire smoke that had escaped through the pyrolysis furnace and the smoke distribution pipe may have occurred due to defects in the pyrolysis furnace which were not perfectly isolated, resulting in not optimal pyrolysis products being produced. In addition, this may also be caused by very volatile substances (which easily evaporate) so that they do not have time to condense during the cooling process. The chemical components lost in the pyrolysis process are volatile compounds that cannot be condensed with water as a coolant in the form of gas. These gases include CO_2, CO, H_2, CH_4, and several other low molecular-weight hydrocarbons (Ridhuan, Irawan, & Inthifawzi, 2019).

The products of the pyrolysis process are solid, liquid, and gas phases. The solid phase is in the form of charcoal (char), the liquid phase is in the form of oil (oil), and the gas phase is in the form of non-condensable compounds (pyro-gas). The pyrolysis process is conventionally carried out at atmospheric pressure. Still, several developments have resulted in pyrolysis technology in the form of Vacuum Pyrolysis Technology, Atmospheric Inert Gas Pyrolysis, Molten Salt Pyrolysis Technology, Flash Pyrolysis, and Thermal Plasma Pyrolysis. The reactor is expected to be able to produce synthetic gas to replace fuel oil4.

Falaah & Cifriadi, (2012) stated that pyrolysis is a destructive process in a material using heat (thermal). In the pyrolysis process, a material will experience a breakdown of the chemical structure into gas, liquid, and solid phases. The fundamental difference between the pyrolysis and combustion processes is that the process occurs without oxygen or minimal oxygen. In contrast, the combustion process requires oxygen so that the material can burn

completely. In other words, pyrolysis is a thermal degradation with little or no oxygen.

The results of the pyrolysis process are hydrocarbon compounds, while the complete combustion process will produce carbon dioxide (CO_2) and water (H_2O). Falaah et al. (2013) stated the pyrolysis process to obtain liquid products from used tire waste could be carried out in several pyrolysis reactors such as Fixed Bed Reactors, Fluidized-bed Pyrolysis Units, Vacuum Pyrolysis Units, Spouted Bed Reactors. The main component of the reactor feed material in the form of used tire waste consists of natural rubber, synthetic rubber, fillers such as carbon black, sulfur, zinc oxide, processing oil, accelerators, and others. Generally, unused, used tires are recycled by renewing the treads or the vulcanization process. In retreading used tires, the first thing that is done is the process of buffing (smoothing process) on the tire tread using a tire shredder. The result of the buffing process is in the form of tire powder, which is no longer of value and becomes solid waste. The tire powder waste is then processed by pyrolysis. The mechanism and kinetics of pyrolysis are fundamental in determining the design of the reactor and the resulting product.

When used tire powder is heated in a pyrolysis reactor, the temperature will increase on the surface of the particles. There are two stages of pyrolysis, namely Primary Pyrolysis and Secondary Cracking. The steam first produced from used tire dust consisted of several hydrocarbon compounds that could react in the second-stage reaction. Anom (2021) and Kauwo, et. al. (2021) stated that the kinetics of the tire pyrolysis process is an exothermic reaction and endothermic evaporation of pyrolysis products. Since pyrolysis is the thermal decomposition of organic polymers, the four general mechanisms identified are a) random chain breaks, b) chain end breaks, c) chain stripping, and d) cross-linking. In general, there are several pyrolysis parameters, namely temperature, the residence time of the vapor in the reaction zone, and atmospheric gas pressure. Bond breaking occurs at high temperatures, and the main product can be converted into components with high selling value.

Gas Chromatography (GC) Analysis Result

GC-MS analysis of liquid smoke from pyrolysis of used tires yielded 88 GC chromatogram peaks, as shown in Figure 4. The number of peaks indicates that the liquid smoke of used tires contains at least 88 types of compounds. The GC chromatogram indicates that the most dominant compounds are at the peaks: 3, 17, 37, 38, 70, 72, and 73 with % area each: 4.66; 3.44; 3.68; 8.22; 3.84; 5.32 and 7.57.

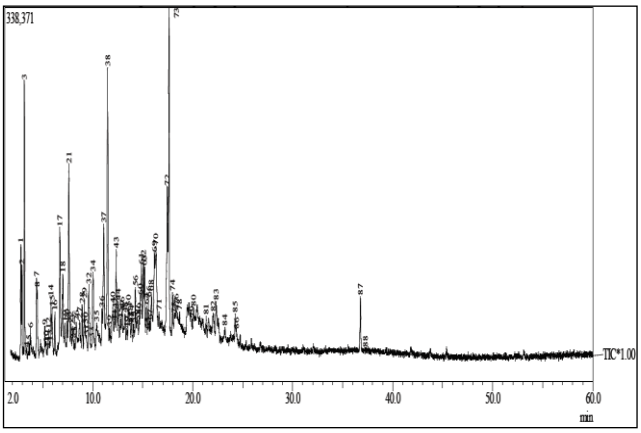

Figure 4. GC Chromatogram of liquid smoke from used tires (GC-MS QP2010S Shimadzu, column Rtx 5 M, carrier gas: helium, EI 70 Ev)

***Mass Spectrophotometry* (MS) Analysis Result**

A compound in Peak Three (3)

Compound identification in GC Chromatography peak 3, with a retention time of 3.181 minutes with the abundance is 4.66%, shows the similarity of the fragmentation pattern of the MS spectrum with the existing 2-propanone compound according to the MS spectra of reference at the library Wiley229.LIB data. A similarity comparison of the MS spectra of the compound in peak three and 2-propanone is shown in Figures 5a and 5b.

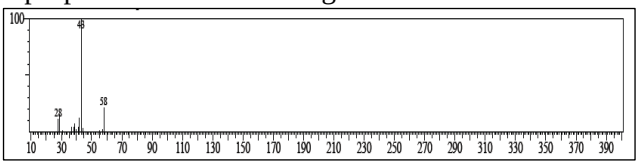

Figure 5a. Mass Spectra of the compound in peak 3

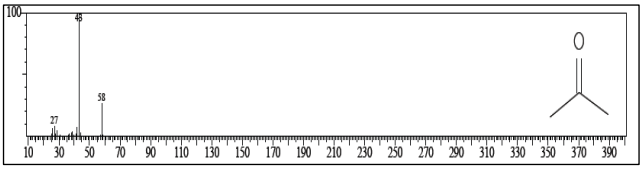

Figure 5b. Mass Spectra of 2-propanone

The compound at peak 3 of the GC chromatogram of used tire liquid smoke, when analyzed with MS produces a spectrum with the same basic peak as the 2-propanone compound shown by the library of Wiley229.LIB data, namely at m/z=43. Both spectra show a molecular ion peak (M^+) the same, namely at m/z=58, which states the molecular weight of these compounds (Anom & Lombok, 2016). The release of electrons in the

molecule m/z=58 produces fragments (molecular ions) at m/z=58. The release of $–CH_3$ radicals (m/z=15) from ions with m/z=58 results in ionic fragmentation at m/z = 43. The release of the $–CH_3$ radical (m/z=15) to the ion with m/z=43 resulted in ionic fragmentation at m/z=28. Based on the similarity of the base peak at m/z=43 and the molecular ion M^+=58, it can be concluded that the compound at peak 3 of the GC chromatogram of liquid smoke from used tires is identical to acetone or 2-propanone compound with a molecular weight of 58 (have similarity 96%) and has the molecular formula C_3H_6O.

A compound at Peak Seventeen (17)

GC chromatograms identification of compounds at peak 17 have a retention time of 6.711 minutes and an abundance of 3.44%. This data showed a similar MS spectral fragmentation pattern 4-methyl-2-pentanone from the library of Wiley229.LIB reference data. The comparison of the MS spectra of peak compound 17 GC chromatogram of liquid smoke from the used tires with that of 4-methyl-2-pentanone is shown in Figures 6a and 6b.

This compound has MS spectra with the same basic peak as the MS spectra of 4-methyl-2-pentanone, as shown by Wiley 229.LIB data library, namely at m/z=43. Both the spectra show the same M^+ peak at m/z=100, expressing the compound's molecular weight. This similarity strengthens the allegation that GC chromatogram of the compound at peak 17 of used tire liquid smoke is identical to the compound 4-methyl-2-pentanone with the molecular formula $C_6H_{12}O$.

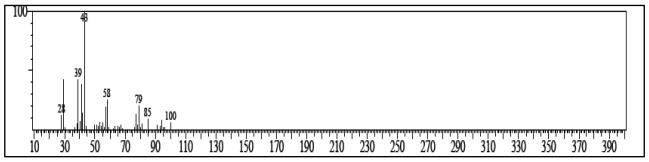

Figure 6a. Mass Spectra of a compound in peak 17

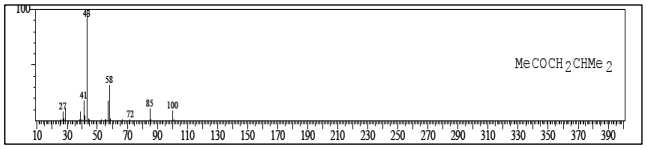

Figure 6b. Mass Spectra of 4-methyl-2-pentanone.

A compound at Peak Thirty-seven (37)

The GC chromatogram compound at peak 37 liquid smoke of used tires has retention time of 11.109 minutes and abundance of 3.68%. This data shows the similarity of the MS spectra with an ethyl-benzene compound according to the MS spectra from the Wiley 229.LIB data library. A comparison of the MS spectra of peak compound 37 and ethyl-benzene contained in the MS spectrophotometer library is shown in Figure 7a and 7b.

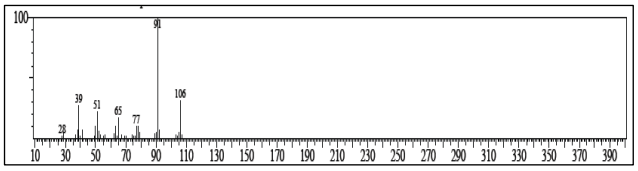

Figure 7a. Mass Spectra of a compound at peak 37

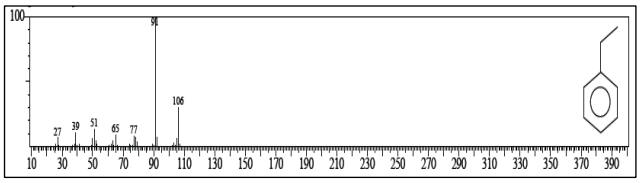

Figure 7b. Mass Spectra of ethyl-benzena.

The 37-peak compound has the same MS spectrum as the MS spectrum of the ethyl-benzene compound, according to Wiley 229.LIB data library has a molecular ion at m/z = 106, representing the compound's molecular weight and the basic peak at m/z = 91. The release of electrons in a molecular ion with m/z=106 produces an ion with m/z=106. The release of $–CH_3$ radical (m/z=15) from a molecular ion with m/z=106 produces ion with m/z=91. Ion with m/z=91 is the basic peak of the spectrum. The release of the $–CH_2$ radical (m/z=14) from ion with m/z=91 produces ion with m/z=77.

Ion with m/z=77, the aromatic ring is broken, then the arrangement of compound ions occurs. The release of –C radical (m/z=12) to ion with m/z=77 gives ion with m/z=65. Release of $–CH_2$ radical (m/z=14) to ion with m/z=65 gives m ion m/z=51. In the ion with m/z=51, compound ions are arranged. Removing the –C radical (m/z=12) from an ion with m/z=51 gives an ion with m/z = 39. Based on the similar basic peak, ion with m/z = 91, molecular ion M^+ = 106, it can be concluded that the compound at the peak 37 of liquid smoke from used tires is identical to that of the ethyl-benzene compound with molecular weight 106 and has the molecular formula C_8H_{10}.

A Compound at Peak Thirty-eight (38)

The GC chromatogram compound at peak 38 liquid smoke from used tires has a retention time of 11.497 minutes and an abundance of 8.22%. This data shows the similarity of MS Spectrum fragmentation patterns the 1,2-dimethyl-benzene compound according to the MS spectra from reference data in the Wiley229.LIB library. The MS

spectra of peak compound 38 and the MS spectra of 1,2-dimethyl-benzene are shown in Figures 8a and 8b.

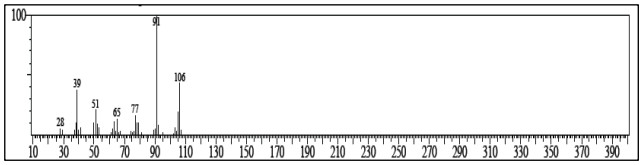

Figure 8a. Mass Spectra of a Compound at Peak 38

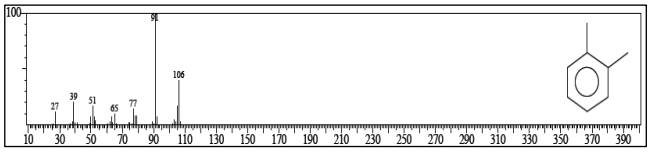

Figure 8b. Mass Spectra of 1,2-dimethyl-benzene

The compound at peak 38 of used tire liquid smoke has the same basic peak in the MS spectrum as that of the 1,2-dimethyl-benzene compound shown by the Wiley 229.LIB data library, namely at m/z=91. Both the spectra show the same molecular ion peak at m/z=106, its representing the compound's molecular weight. The release of electrons in the molecule m/z=106 produces ions m/z=106. The release $–CH_3$ radicals (m/z=15) from the molecular ion m/z=106 produces ions at m/z=91. Release $–CH_2$ radicals (m/z=14) from an ion with m/z=91 produce an ion with m/z=77.

In the ion with m/z=77, the aromatic ring is broken, then the arrangement of compound ions occurs. Release of C radical (m/z=12) from ion with m/z=77 gives ion with m/z=65. The release of $–CH_2$ radical (m/z=14) from ion with m/z = 65 gives ion with m /z=51. From ions with m/z=51, there is an arrangement of compound ions. Removing the –C radical (m/z=12) from an ion with m/z=51 produces an ion with m/z=39. Based on the similar basic peak (m/z=91), the molecular ion (m/z=106), and the compound fragmentation pattern, it can be concluded that the compound at the peak of 38 from liquid smoke is identical to the 1,2-dimethyl-benzene, with a molecular weight of 106 and a molecular formula C_8H_{10}.

A Compound at Peak Seventy-two (72)

The GC chromatogram identification of peak 72 compounds has a retention time of 17.478 minutes and a percent abundance of 5.32%. This data showed a similarity in the spectral fragmentation pattern with the existing 1-methyl-4-(1-methyl-ethyl) compound according to the MS spectra in the reference data library Wiley229. LIB. The MS spectra of the peak compounds 72 and 1-methyl-4-(1-methyl-ethyl)-benzene are shown in Figures 9a and 9b.

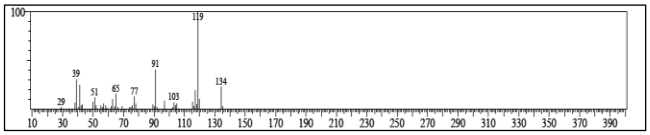

Figure 9a. Mass Spectra of Compound at Peak 72

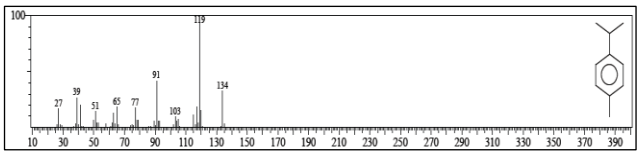

Figure 9b. Mass Spectra of 1-methyl-4-isopropyl-benzene

The compound at peak 72 GC chromatogram of used tire liquid smoke has the same basic peak as that of the 1-methyl-4-isopropyl-benzene compound as found in the library of Wiley 229.LIB data, namely at m/z=119. Both spectra show the same molecular ion peak at m/z=134, which states the compound's molecular weight. The release of electrons in the molecule m/z=134 results in the fragmentation of the ion m/z=134. The release of the radical $-CH_3$ (m/z=15) from the ion-molecule m/z=134 produces an ion with m/z=119 as its base peak. Releasing $-CH_3$ radicals (m/z=15) and H radicals (m/z=1) from ions with m/z=119 produces ions at m/z=103.

In m/z=103 molecules, an aromatic ring is broken, Then the arrangement of ionic compounds occurs. Based on the similarity of the basic peak (m/z=119), the molecular ion (M^+=134), and the compound fragmentation pattern, it can be concluded that the peak of 72 is identical to the 1-methyl-4-isopropyl-benzene. The 1-methyl-4-isopropyl-benzene molecular weight of 134 and has molecular formula $C_{10}H_{14}$.

A Compound at Peak Seventy-three (73)

The GC chromatogram compound at peak 73 with a retention time of 17.627 minutes and an abundance of 7.57% shows the similarity of the fragmentation pattern of the MS spectra of limonene compounds following the MS spectra in the library of Wiley229.LIB. The MS spectra of the compound at 73 GC chromatogram and the limonene compound are shown in Figures 10a and 10b.

The GC chromatogram compound at peak 73 of used tire liquid smoke has a basic peak in the MS spectrum that is the same as the basic peak of the MS spectrum of the existing limonene compound namely at m/z=68. In addition, both spectra also show the

same molecular ion peak at m/z=136, representing the compound's molecular weight.

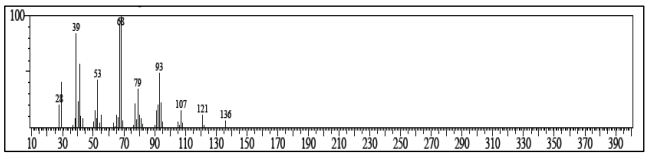

Figure 10 a. Mass Spectra of a Compound at Peak 73

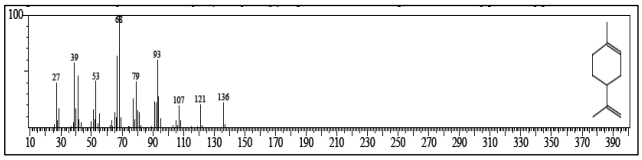

Figure 10 b. Mass Spectra of limonene

Based on the similarity of the base peak at m/z=68, the molecular ion M^+=136, it can be concluded that the compound at peak at 73 is identically to the limonene compound with a molecular weight of 136 and has the molecular formula $C_{10}H_{16}$. This reason as well as the similarity of the compound fragmentation pattern.

CONCLUSION

Liquid smoke has 88 peaks on GC chromatogram which indicates at least 88 types of compounds. The six most dominant chromatograms appear at peaks 3, 17, 37, 38, 72, and 73, with different retention times. The six most dominant compounds from GC chromatogram were identified using MS spectrum analysis. MS spectrum showed these compounds were: 2-propanone (peak 3), 4-methyl-2-pentanone (peak 17), ethyl benzene (peak 37), 1,2-dimethyl-benzene (peak 38), 1-methyl-4-(1-methyl ethyl) (peak 72) and limonene (peak 73).

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