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Waste Plastics: Pyrolysis of Powdered and Powder-free Laboratory Examination Waste Gloves

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Authors' contributions

This work was carried out in collaboration between all authors. Author NH designed and setup the experiment, selected the materials, performed the experiment, operated GC-MS and performed GC-MS analysis, wrote the first draft and finalized the manuscript. Author RM provided GC-MS expertise; and assisted with the revision of the manuscript. Author SS participated in performing the experiments, collecting data, and participated in writing the first draft. Author LL participated in performing the experiments, collecting the data, and performed the statistical analysis of GC-MS data. Author TP participated in performing the experiments, collecting the data and performed the statistical analysis of GC-MS data. Author CO was a part-time participant student during 5 weeks of summer. Author LW had the original idea and supported the work in all stages. All authors read and approved the final manuscript.

Original Research Article

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ABSTRACT

Aims: The aim of this investigation was to develop a methodology that provides insights to clean up environmental pollution caused by the ever increasing amount of waste plastic materials. The procedure, however, would be practical whenever it is economically advantageous. Thermal cracking of laboratory safety glove (LSG), without catalyst, to

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useful chemicals is an important part of this research which is reported here.

Study Design: To design the experimental procedure, we primarily concentrated on the thermal stability of the LSG by bearing in mind the results of thermo gravimetric analysis (TGA). Based on such results the appropriate set-up for the decomposition of the LSG was designed.

Place and Duration of Study: The study was done in the Department of Biological and Physical Sciences at South Carolina State University (SCSU), Orangeburg, SC, USA, during the summer of 2013.

Methodology: The thermal cracking process without catalyst was used to convert LSGs into useful liquid and solid chemicals. The LSGs were pushed into the reactor one by one without cutting. Prior to pyrolysis, the thermal stability of materials were determined by thermo gravimetric analysis (from 50°C to 800°C) with a heating rate of 10°C/min while the samples were purged with 10 mL/min argon. The condensed liquids were analyzed by a Shimadzu GC-MS model GCMS-QP 2010s using helium as the mobile phase.

Results: The thermal stability of the LSG both powdered and powder-free was very similar as was expected since it depended on the nature of constituent polymers. The highest decomposition rates were observed at temperatures around 410°C. The 15% leftover of the powder-free LSG were less than leftover of powered LSG (30%) at 500°C, in the same way the 18% leftover ashes of powdered LSG was higher than 7% ashes of powdered free LSG at 800°C. The GC-MS chromatogram of pyrolysis liquids indicated over 350 chemicals. The most abundant compound of pyrolysis was HCl, as was expected from the chemical constituents of chlorinated plastics, followed with eight member carbon isomers. Also, a variety of phthalic acid derivatives with high concentration were recognized in all samples.

Conclusion: The liquids obtained from pyrolysis of LSGs were a complex mixture of hydrocarbons and cyclic compounds. Also, the liquids were saturated with inorganic and organic acids, esters and anhydrides. Therefore, the refinement of liquids resulting from pyrolysis is of necessity to obtain a quality fuel. Also, the condensed liquids contained highly reactive chemicals such as acids, alcohols and alkene, which made them unsuitable fuel for internal combustion engines prior to refinement.

Keywords: Environment; pollution; lab examination gloves; latex gloves; pyrolysis of gloves; neoprene; pyrolysis of waste gloves; chemicals from pyrolysis of gloves; phthalic acid derivatives.

DEFINITIONS, ACRONYMS, ABBREVIATIONS

LEG: laboratory examination glove; LSG: laboratory safety glove; PVC: poly(vinyl chloride); PMMA: poly(methyl methacrylate); PS: poly(styrene); PE: poly(ethylene); PET: poly (ethylene terephthalate); RT: retention time; TIC: total ion chromatogram; GC-MS: gas chromatography-mass spectrometer; MS: mass spectrometer; WHO: world health organization.

1. INTRODUCTION

Recycling plastics waste by thermal cracking or pyrolysis is one of the most used methods [1-22]. Pyrolysis has been employed to convert waste plastic into useful products such as fine chemicals, transportation fuels and lubricant oils. The pyrolysis process uses elevated temperatures to crack down long chains of hydrocarbons and polymers into shorter chain molecules. The waste laboratory safety gloves (LSGs) in this process decomposed randomly

into three phases of matter: gaseous materials such as HCl and C1 to C4 hydrocarbons, a complex mixture of liquid and solids. By pyrolysis, the solid wastes were converted to liquids with potentialities of transportation fuel and the chemical energies stored within LSGs waste were recycled. Also, this process provided environmental advantage by minimizing plastic pollution [1,2].

The sources of waste pollution originating with LSGs are safety precautions in professional work. The safety protection required in scientific laboratories, technological industries and health industries has an increased demand for the production of latex gloves. As a consequence, the demand for natural rubber latex has risen about four times from 159 thousand tons in 1984 to 597 thousand tons in 2003 [1,3]. Since then in 2005, Malaysia, one of the world's leading rubber glove manufacturers, alone consumed 226.2 thousand tons of natural rubber for glove production [3]. Additionally, the production of natural rubber is expected to increase worldwide. Reports from Indonesia and Malaysia show effort to increase their output to 2900 thousand tons and 970 thousand tons, respectively. The private sector in Vietnam is also expected to increase their natural demand to 780 thousand tons, while Cambodia has announced that they expect an increase in their plantation area (45 thousand tons) [4].

Examination gloves or disposable gloves also are used during medical examinations and procedures and as safety protection in work to prevent contaminations [5,6]. They are available in the form of un-powdered, or powdered with cornstarch to lubricate the gloves, making them easier to use [7]. Cornstarch replaced tissue-irritating lycopodium powder and talc. The powders can impede healing if they get into tissues (as during surgery) and can contaminate experiments in laboratory procedures. Therefore, un-powdered gloves are being used more often. Surgical gloves have more precise sizing with better precision and sensitivity and are made to a higher standard than examination gloves. The gloves that are used in chemistry laboratories as safety protection media are examination gloves (Fig. 1a). The wastes LEGs, reported here were generated in our laboratory as safety protection ware.

Examination gloves are made of diverse polymers including latex, nitrile rubber, vinyl and neoprene. Neoprene or polychloroprene ($-CH_2-CCI=CH-CH_2-$)_n is a family of synthetic rubbers that are produced by free-radical polymerization of 2-chlorobutadiene ($CH_2=CCI-CH=CH_2$) [8]. It exhibits good chemical stability, and maintains flexibility over a wide temperature range. It is used in a wide variety of applications, such as laptop sleeves, orthopedic braces (wrist, knee, etc.), electrical insulation, liquid and sheet applied elastomeric membranes or flashings, and automotive fan belts [9]

The decomposition of polymeric materials has been of scientists' interest from both points of view: stability of goods produced [10,11] and reeducation of generated wastes [12-14]. Thus, decomposition kinetics of polymeric materials [15,16] and the mechanism of decomposition have been studied by many researchers [17,18]. Researchers have discovered that some widely used polymeric materials such as poly (methyl methacrylate) (PMMA), upon heating, decomposed to original monomers [19] likewise polystyrene (PS) to styrene [20-23] with very high yields at micro-scales. Thus, a relatively inexpensive source to the basic chemicals for manufacturing industries was considered. However, the most-used polymeric materials such as poly (vinyl chloride) (PVC), decomposed to smaller stable toxic corrosive chemical that were part of the original constituents constituent units of the polymers. For example, PVC, upon heating, first decomposed to hydrogen chloride and unsaturated polymers, these newly produced unsaturated polymers upon heating decomposed later to other chemicals [24-26]. Pyrolysis process of neoprene is very similar to that of PVC. It first produces HCI [27] and

unsaturated polymers. It is noteworthy that most of the well documented investigations were done on pure polymers with known molar mass distribution or clean materials provided directly by manufacturers. Therefore, the knowledge of decomposition of waste LSGs gained relevance whenever we deal with waste processing and recovery which is the subject of this work.

The pyrolysis of LSGs got relevance since it is not as easy as recycling of styrene based materials. Especial design requirements of this process have attracted the attention of engineers for designing pilot plants [28] at the kg level. On the other hand, the wide use of LSGs resulted in the accumulation of untraditional wastes not native to the mother earth life cycle [29]. The disposal of latex gloves in medical wastes, in particular, presents several problems as they are hazardous biologically, non-biodegradable and do generate HCl and chlorinated organic compounds upon incineration. Thus, the World Health Organization (WHO) is opposed to land filling or incineration of this waste. Therefore, a new technology that is both environmentally friendly and biologically safe would be required to process these wastes. There is still lack of data concerning the characteristics and the kind of chemicals produced from pyrolysis of LSGs. More research and relevant data are required for better understanding of the process [28]. A study on kinetic properties of pyrolysis of selected medicals wastes such as absorbent cotton, medical respirator, bamboo sticks [30] and cotton gauges, packaging boxes, capsule plates and transfusion tubes [25] were conducted by a number of researchers [25,30].

To describe a current picture of the situation, it is safe to say, wastes of modern materials are accumulated without effective decomposition and recycling routes in the landfills. The increase of petroleum and petrochemical prices opened the way for industries to invest in recycling of plastic wastes to petrochemicals [31-33] nowadays; plastic landfills are as valuable as petroleum mines. Models for reaction's kinetics for optimal pyrolysis conditions of plastic waste mixtures have been proposed by researchers [29-33]. Literature abounds on the recycling of these non-traditional wastes to petrochemicals [34-36] and many industries are sustained and developed based on decomposition of natural and synthetic polymers [37,38]. From a scientific-engineering point of view, non-degradability of plastics is no longer an environmental issue in landfills since the plastics can be recycled [39]. However, run-away plastic wastes are continuing to be an enormous hazard on the surface and surface water such as waterways, seas and oceans, endangering safe life for both animals and humans [40].

A real waste container in the waste collection site contains many kinds of waste. Therefore, co-pyrolysis of waste plastic with other natural wastes is relevant for researchers. For example, the co-pyrolysis of pine cone with synthetic polymers [41] and characterization of products from the pyrolysis of municipal solid waste [42] and isothermal co-pyrolysis of hazelnut shell and ultra-high molecular weight polyethylene [43] are indicative of the fact that wastes are also useful materials. To avoid the landfill problem and plastic wastes hazards, various techniques for the treatment of waste plastics have been investigated to complement existing landfill and mechanical recycling technologies. The objectives of these investigations were to convert the waste into valuable products such as fuel, synthetic lubricants and tar for asphalt pavement before the waste headed to a landfill.

This study reports the results of non-catalytic conversion of LSGs which are neoprene based material. LSGs are disposed regularly, in teaching and research laboratories and in hospitals and medical offices, as mentioned above. Therefore, the conversion of LSGs to useful chemicals has both economic and environmental benefits. Thus the material used here were

waste LSGs (not freshly prepared polymers or clean products supplied by manufacturers) to simulate real conditions at collection centers and the results were close to those expected from waste processing pyrolysis.

2. MATERIALS, INSTRUMENTATION AND METHODS

2.1 Materials

The material used (Fig. 1a) was a mixture of un-powdered and powdered waste latex examination gloves (LEGs); which were collected during 2012-2013 in our lab. LEGs were pushed, one by one, into a 5 L three-necked round bottom flask for pyrolysis process.

2.2 Instrumentation

2.2.1 The thermo gravimetric analyzer

A Perkin-Elmer thermo gravimetric analyzer (TGA-7) was used to study the thermal stability of the LEG from 50°C to 800°C with a heating rate of 10°C/min while the sample was purged with 10 mL/min argon. The TGA was calibrated properly before use.

2.2.2 Gas chromatographer-mass spectrometer (GC-MS)

A Shimadzu GC-MS model GCMS-QP 2010s was used to analyze the liquid samples using helium as the mobile phase. The oven program was set on 6 min at 40°C, followed by a 10°C/min temperature increase to 220°C and then an isothermal on the final temperature for 15 min.

One-tenth to one microliter of each sample was injected into GC-MS by AOC-20i autosampler. The auto sampler was set for three rinses before and after injection with a mixture of methanol/water, 90/10, v:v and two rinses with the sample before injection. The plunger speed and syringe speed were set at high.

The MS program consisted of the scanning masses 15 < M/z < 350; scanning time began from 0.4 min and ended after 35 minutes. The identities of chemicals were established by the aid of the automatic NIST library search. Among the suggested structures, the one that was better matched to the fragmentations pattern, consistent with boiling point and retention time of the compound was selected.

2.3 Pyrolysis Procedures

156 pairs of used LEGs weighing 879.8 g (Fig. 1b) were placed into the reactor (Fig. 1c). The end of a stainless steel type K thermistor was placed into the bottom of the threenecked 5-L flask (T1) to measure the decomposition temperature, another thermistor placed into the entrance of the side-arm distilling head (T2) to measure the temperature of the gases that left the reactor and the third thermistor was placed at the exit of the Claisen adaptor to record the temperature of volatiles that left the collection funnel and condensed into the next receiver. Table 1 shows the stages of heating and decomposition process of LEGs. At 253°C vapors resulting from decomposing LEGs filled up the flask, air condenser, and the other flasks (Fig. 1d). The first drop of condensate (97°C) was collected into the recipient graduated funnel when the melt temperature reached 488°C. The drops at the time of condensation were clear liquid but when they reached to the bottom of the container the color was changed from bright yellow to dark green. As time passed the color of the liquid became darker.



Fig. 1. Step-by-step illustration of decomposition process of LEGs: (a) LEGs before use, (b) waste LEGs collected during one year (2012), (c) LEGs loaded in the reactor and (d) the reactor at 393°C

Temperature (°C)			Observation, Remarks and Comments
T1	T2	Т3	
253	22.3	22	Smokey vapor started appearing, gloves started melting.
475.2	34.5	25.2	Gloves began to melt vigorously and rapidly into gases.
488.0	91.6	48.1	1st drop of liquid was observed at the collection point.
374.5	79.8	43.8	The contents of reaction flask were smoldering black.
382.9	89.5	60.6	The temperature increased steadily.
393.7	93.5	67.8	Sample 1 was collected.
436.8	935	33.2	Sample 2 was collected.

Table 1. The thermal histor	v of the reactor:	First attempt to	pyrolysis of LEGs.
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At this moment, the pressure of gases opened the joints, the liquids and uncondensed vapors were leaking out from the joints of the Claisen adaptor; therefore, the reaction was stopped. Days later, needle-like crystals were observed inside the reaction flask (Fig. 2a). Some of the gloves were not decomposed yet but covered by the black residuals of the decomposed ones. Some of the materials leaked out of the joints were solidified around the joints with a pale color (Fig. 2b and 2c). The solid was slightly soluble in acetone, soluble in a mixture of methanol: water (90:10 V:V) and boiling water. The aqueous solution of the solid was strongly acidic. The pyrolysis was resumed after the joints were cleaned (Fig. 2d). Table 2 summarizes the results of the second attempt to pyrolysis LEGs.

The reaction was stopped since unusually high vapor pressure opened the joints and the solids were deposited again inside the path of condenser (Fig. 2e) and outsides of the joints.



Fig. 2. Step-by-step illustration of the decomposition process of LEGs: (a) the white needle shaped crystals grown in tope of the cold residues, (b) accumulation of solids out of the joints, (c) a sample of the solids, (d) the setup and (e) the condenser with solids

Time	Temperature (°C)			Comments
	T 1	T 2	Т 3	_
11:00	152	25	25	The vapor and liquid equilibrium established inside of main flask.
11:10	355	48	25	Vapor raising out of the reactor.
11:13	355	74	25.7	The first drop of liquid was condensed near to the thermometer 1.
11:18	355	93	25.8	Vapors were condensing, flowing toward the gravity direction in air condenser. Their color was pail-yellow; however, the accumulated liquid in the funnel was dark black.

Table 2. Thermal history	of the reactor: Second	d attempt to pyrolysis LEGs
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2.3.1 Continuation of reaction (third time)

The collection parts of the system were cleaned and dried prior to resuming the pyrolysis. The pyrolysis lasted \sim 5 hours. Pyrolysis was discontinued when the cloud of vapors was solidified around the neck of the reaction vessel. Table 3 (Appendix) shows the steps of the pyrolysis.

At the beginning the thermometer (T1, T2 and T3) readings were 23.0°C, as was the room temperature. After six minutes of heating, a dark steam filled the flask then the argon gas valve was turned on (T1's reading was at 25.6°C; this indicated that the thermistor was not touching the bottom of the flask). After a period of 14 minutes the temperature of T1 increased to 89°C, while T2's temperature was recorded at 51°C. During this period, clouds of vapor travelled through the air-condenser and the first drops of liquid appeared around the road of the steam in the air condenser. Simultaneously, solid depositions in the air-condenser nearly blocked it; however, the incoming distillate dissolved most of the solids and moved them into samples (Fig. 3a to 3d). Next, a greenish solid filled the condenser but most of it was washed away by distillates (Fig. 3e). After that, a reddish solid filled the path then it was washed away by incoming distillates (Fig. 3f). At the end a dark yellow solid remained in the condenser and the path of liquid.



Fig. 3. Step-by-step illustration of the decomposition process of LEGs (a) solids deposited in the condenser, (b) and (c) magnification of selected sections of (a), (d) crystallization of solids in the collection funnel, (e) deposit of greenish solids and (f) deposit of reddish solids

After a period of fifteen minutes the first drop of liquid was observed in the funnel (Fig. 4b), and T2 readings were constant, recorded 51°C. The initial sample collected was clear yellow in color and part of the liquid was solidified around the glass funnel. After a few hours, the collected liquid divided into three phases, organic, aqueous and solid crystals (Fig. 4d and 4e).

As soon as a sample was collected, it was taken for GC-MS analysis. If GC-MS was busy in the previous sample and then the sample was queued into auto-sampler to be processed as soon as possible. The first sample (sample 3) was collected at the condensing temperature of 57°C. The second sample (sample 4) was collected when the temperature of T1 and T2 increased to 186°C and 86°C, respectively; the T3's temperature was 23.3°C. Table 3 (Appendix) shows the time and amount of sample collected. Noticeable changes in the color of the solids were observed: changed from almond (Fig. 4a-4d) to a darkish green (Fig. 4e).

Some of the volatiles, materials in conjunction with non-volatiles were condensed in the second container (Fig. 4a). A close examination of the liquid indicated that it had two phases: aqueous and organic. Later on when we examined the samples again, it was composed of three phase of organic, aqueous and solid (Fig. 4e).



Fig. 4. Results of pyrolysis of LEGs: (a) liquid condensed in the second flask, (b) The apparent of the first sample collected in the graduated funnel, (c) eleven samples collected from pyrolysis of LEGs, (d) magnification of the appearance of samples 3, 4 and 5, (e) sample 1 after 3 hours; it divided into three phases: organic, aqueous and solid

2.3.2 The extraction with boiling water

The solid deposited in the condenser during pyrolysis was soluble in boiling water. Therefore, extraction of the remains with boiling water may have resulted in removing the solids. For this, 100 mL of distilled water added to the reaction flask boiled for 10 min then was filtered; 75 mL of hot filtrate was recovered. The rest of the water was absorbed into remnants of the pyrolysis. The next day, a small amount of colorless crystals were visible on the walls and on top of the filtrate. By filtration, some greenish (~100 mg) fiber-like materials were recovered. Therefore, the amounts of boiling-water soluble materials were very low which indicated those solids were trapped in the polymer network; they were liberated after the pyrolysis of the polymeric network. Thus those organic acids and ester were not free and extractable prior to pyrolysis, the process that removed them from the polymer-network.

2.3.3 Continuation of the pyrolysis

The condensers and receivers were washed well with methanol/water, 90/10, (v:v) then reassembled. The heater was set on 6 (scale of 1 to 10). After recovering 50 mL of water (with a few mL of organic layers on top of it (sample A, not shown here) the receiver for Sample B was placed under the funnel. Six more samples were collected (Fig. 5B-5G). The sample B showed an aqueous phase with crystals suspended in it and an organic phase on top. The amount of organic phase increased in time as the water contents of the reaction flask consumed (Fig. 5B and 5C). By advancing the pyrolysis, the amount of organic layer increased and its color become darker. The last samples (Fig. 5F and 5G samples) had no water and its color was dark-red (Fig. 5E to 5G samples). A detailed description of the results is tabulated in Table 4 (Appendix).

During sample E collection, the condenser was filled with reddish solids (Fig. 5H). Most of these solids were dissolved in the distillates during the collection of samples E to G. At the end of pyrolysis at T1 = 620° C, little solids remained in the condenser.



Fig. 5. The samples collected after aqueous extractions and addition of 100 mL of water to the reaction flask

3. RESULTS AND DISCUSSION

3.1 Thermo Gravimetric Analysis

About 12 mg of a LEG was cut into small pieces that could fit into the TGA pan (Fig. 6a). The initial attempt was a heating scan from 50°C to 600°C under nitrogen. The end product was a grey color and contained dark chunks (Fig. 6b) which was indicative of incomplete decomposition. The next heating ramp was from 50°C to 800°C (under nitrogen), which resulted in white solid end products (Fig. 6c). This solid was soluble in water; the pH of its solution was very basic (Fig. 6d).



Fig. 6. Thermo gravimetric analysis of LEGs: (a) pan loaded with 12 mg of LEG, (b) the remains after heated to 600°C, (c) the ashes at 800°C, (d) strongly alkaline pH of the aqueous solution of the ashes

Fig. 7 shows the results of thermo gravimetric studies of LEGs, both powdered and unpowdered. The thermal stability of both types of LEGs is very similar; the decomposition began around 350°C reaching the highest rate at 410°C. The decomposition reaction was complete at 500°C; the residues of powdered LEG (~30%) were much more than that of powder-free sample LEG (~12%) as was expected. A second stage of decomposition was observed around 625°C for powder free LEG and 650°C for powdered LEG. The resulting ashes were white in color (Fig. 6c) and soluble in water, producing a strongly basic solution (Fig. 6d) which is a property of active metal oxides. The thermo gravimetric data agreed with the observations in the reactor (Tables 1 to 4).

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Fig. 7. Thermo gravimetric results of powdered (red line) and un-powdered (blue line) gloves from 50°C of 800°C under 10 mL/min N₂ (g) with heating rate of 10°C/min

3.2 Analysis of Liquid Samples Obtained from Pyrolysis of LEGs

The pyrolysis of the LEGs resulted in 25 samples. All samples were analyzed by GC-MS. Most of the chromatograms were similar to each other; however, nine of them were slightly different (Fig. 8). Below, these nine TICs will be annualized.





Fig. 8. TIC of the representatives samples as described on the TICs and in the Tables 1, 3, and 4

3.2.1 The most volatile sample

The most volatile sample consisted of the chemical that escaped the first collection container and accumulated in the second container (Fig. 4a) equipped with a water condenser, was kept cold in an ice-water bath. As the TIC (Fig. 8 Volatiles) showed, the first peak at the chromatogram (1.15-1.36 min) was hydrogen chloride as expected; the next peak (1.36-1.56) represents a mixture of HCl and C₅ compounds, C₅H₁₀, then, at the end of the peak (1.58-2.27 min) various isomers of C₆ compounds, C₆H₁₂, both cyclic and acyclic, with one saturated element, the broad peak located at 2.27-2.74 min was benzene, C₆H₆, at 2.78-4.30 min C₇ compounds were visible such as 2,2-dimethylpentane (2.742 min) then heptene isomers (2.78-3.59 min), octene isomers appeared at 3.68 – 6.79 min while toluene, C₇H₈, was co-eluted with octenes (4.11-4.81 min). Then C₈ compounds with two unsaturated units were at 6.82-7.48 min. From 7.48 – 9.00 min the dimethyl benzene derivatives (C₈) with C₉ linear isomers were visible. The peak at 11.45-11.83 was hard to identify; the NIST [44] library identifies it as 3-chlor-3-ethyl heptane with 88% similarity, however, the M/z = 91 may be indicative of a benezylic cation or tropylium ion resulting from cleavage at benzylic carbon in the compound.

Another series of hydrocarbons detected between 10-15 min covering 3 major peaks 11.45-11.96, 12.56-12.68 min and 12.93-13.17 min, identified as various isomers of straight chains $C_{11}H_{22}$ (192°C), $C_{11}H_{20}$ (bp 196°C) and $C_{11}H_{24}$ (bp 196°C) [45]. Table 5 (Appendix) provides

the list of major chemicals identified from the most volatile sample. Also, a comparison between the mass-spectrum (MS) of the compounds in the sample of volatiles and the MS in the NIST library is available (Fig. 9. Appendix).

3.2.2 Sample 1

Sample 1 was the first drops (head) collected, from the beginning of the pyrolysis reaction (383°C-393°C) and condensed below 94°C. The main component of sample 1 was hydrogen chloride, HCl and moisture (Fig. 9 Sample 1) which appeared on the chromatogram from 1.15 to 2.85 min.

As the TIC of sample 1 (Fig. 8 sample 1) showed there were over 170 chemicals identified by the auto-research of GC-MS with very low concentration relative to HCI. Fig. 10a shows the background or the baseline of the chromatogram at the initial time, these M/z are the ion of gasses that were dissolved in the sample: CO_2 , (M/z = 44 Da), oxygen (M/z = 32 Da), NO (M/z=30 Da) and N₂ (M/z=28 Da). These low molar mass compounds produced from decomposition of gloves; dissolved in the sample, as was also reported by Williams, et al. [1]. The chromatogram of sample 1 showed two main compounds: one appears from 1.25 min to 2.45 min, with M/z 36 and 37 (Fig. 10b) which has been identified as HCI, hydrogen chloride (Fig. 10c). HCl, at room temperature, is a colorless gas, which forms white fumes of hydrochloric acid upon contact with atmospheric humidity as was observed at the beginning of the pyrolysis reaction (Fig. 1d). HCl melts at -114.9°C and boils at -85.06°C. It dissolves well in polar protic solvents such as water and alcohols and aprotic organics containing oxygen and nitrogen. Further analysis of TIC indicated that the pyrolysis liquids are reached in oxygenated compounds. The pH of sample 1 was below 2 measured by a pH-paper which confirmed strong acidity of the sample. HCI produced by decomposition of chlorinated polymers that were utilized to fabricate the gloves. One of such polymers is neoprene or polychloroprene, a family of synthetic rubbers that are produced by polymerization of chloroprene [4]. The chloroprene decomposed first to HCl and then to other chemicals as:

 $(-CH_2-CCI=CH-CH_2-)_n(s) \rightarrow HCI(g) + other chemicals$

The other components of sample 1 were at 11.26 min 2-propyl-1-pentanol or 2-ethyl-1hexanol or 6-methyl-3-undecane, phthalic anhydride (Fig. 10d) which appeared at 16.4 min (compared with NIST library (Fig. 10e)) and at 18.5 min; phthalimide also appeared besides saturated and unsaturated hydrocarbons (Table 6 Appendix).

Other chemicals from the decomposition of neoprene are oligomers $H-(CH)_n-H$, such as methane, ethane, ethyne, propene, butadiene and cyclic aromatic compounds resulting from secondary reaction of the decomposition of products such as benzene and its derivatives and other polycyclic compounds such as naphthalene and its derivatives as reported by other researchers [27]. Visual observation of the reactor indicated that the liquefied vapors were very reactive. At the beginning of the air-condenser they were colorless, when they reached to the end of condenser, the color changed and within the liquid, solid crystals appeared. Table 6 (Appendix) shows 50 major chemicals identified by the NIST library in sample 1.



Fig. 10. (a) A background scan at the beginning of TIC, (b) a scan from the peak appeared from 1.25 -1.75 min, (c) MS of HCI from NIST library, (d) a scan of compound appeared at 16.45 min and (e) a MS of phthalic anhydride from the NIST library

3.2.3 Sample 2

Table 7 (Appendix) shows 50 major chemicals identified from sample 2. This sample contained a smaller amount of HCl compared to sample 1 (Fig. 8 Sample 2). This can be interpreted by the mechanism of pyrolysis of chlorinated polymers which occurs in various stages; the first step is emission of HCl and production of unsaturated polymers [25]. Then these unsaturated polymers decompose to other hydrocarbons (Reaction 1). Therefore, most of HCI were volatilized and left the reactor prior and during the collection of first the sample. The major chemicals of sample 2 were: at 2.27 min (Fig. 11a) identified as benzene (Fig. 11b). From 4.75 min to 5.89 min the isomers of octane were identified: at 5.27 min 3ethyl-2-hexene at 5.65 min 3-methyl-2-heptene and at 5.79 min 2-octene. The next major chemical at 7.40 min is another isomer of C₈, xylene. At 8.25 min noname was identified. At 8.70 min the C_{10} isomers began to appear. Also, some chlorinated compounds were visible; the peak at 10.05 min on the chromatogram was identified as 3-chloro-3-methyl-heptane. The MS of 3-chloro-heptane and 2-ethyl-hexanol are very similar to each other. At 11.0 min (Fig. 11c) was a chlorinated compound, 1-chloro-2-ethyl-heptane (Fig. 11d) and at 11.49 min (Fig. 11e) 2-ethyl-hexanol either (Fig. 11f) or 6-methyl-3-undecene was identified. At 11.90 min it was also a C₁₀ isomer which was identified as 3-carene. The reaction below (Reaction 1) shows a possible decomposition and composition path for the chemicals produced from pyrolysis of neoprene and detected by GC-MS.



Reaction 1. Schematic representation of some chemicals resulting from decomposition of neoprene



Fig. 11. MS of the major compounds in sample 2 compared with the MS of the most similar compound from NIST library

3.2.4 Sample 3

This sample was the first to be collected during the third attempt to pyrolyze LEGs. Table 8 (Appendix) shows 50 of the major chemicals in sample 3. Chromatogram of sample 3 (Fig. 8 Sample 3) shows nine major compounds, though the total number of chemicals in the sample exceed 250. The background at the initial time (Fig. 12a) shows moisture (M/z = 18 in excess), nitrogen (M/z = 28), oxygen (M/z = 32) and argon (M/z = 40); these are gases that were dissolved in the sample. The first peak (1.15-2.92 min) is a mixture of moisture (M/z = 18Da) and HCI (M/z = 36Da and 38Da) or $(H_3O^{+})CI^{-}(H_2O)_n$, as indicated in MS (Fig. 12b). The major compound appeared at 5,433 min (Fig. 12c) and was identified as 4-octene (Fig. 12d). At 12.52 min (Fig. 12e) 5-methyl-2-decane on the chromatograph (Fig. 12f), then 12.76 min (Fig. 12g) showed 2-ethyl-1,3-dimethylbenzene (Fig. 12h). The next compound was at 12.98 min (Fig. 12i) 2-propylpent-1-ol or 2-ethyl-hex-1-ol or 6-methyl-3-undecene. The compound at 17.81 min was identified as phthalic anhydride (Fig. 12j) at 30.8 min diisooctyl phthalate. The last compound was identified as 1,2-benzenedicarboxylic acid ester (30.94 min), mono (2-ethylhexyl) phthalate (Fig. 12k). Fig. 12 shows the MS of the major chemicals in the chromatogram compared with the most similar MS of chemicals from NIST library. The peak's RT also indicated on the corresponding MS.



Fig. 12. Chromatograph of major compounds in Sample 3. The retention time and the structure of compound are indicated in MS

3.2.5 Sample 4

The chromatogram (Fig. 9 Sample 4) shows at 1.16 min a mixture of water, HCl, and C5 isomers. At 1.59 min the concentration of moisture and HCI decreased very much, however, the C5 compounds continued to elute, 1-pentene (mp 30°C) was visible, the mixture of pentene and HCI was recognized by the NIST library as 2-chlorpentane (bp 98°C) which must have come at a later retention time. At 2.0 min, hexene (bp 63°) has the highest concentrations (C₆ compounds) and 2.28, benzene (80°C) appeared, at 2.87 min C₇ isomers are visible. From 3.84 to 8.00 min C_8 isomers were identified (4-octene). After 8 min also the C_8 isomers were eluting; these isomers possessed more than one double bond, for example, the scan at 8.33 min identified it as 3,5-octadiene. From 8.50 min we began to observe C₉ isomers for example the scan at the top of peak at 8.57 min was identified as 3,5,5trimethylcyclohexene. Xylenes (benzylic C_8) appeared from 8.63 to 9.10 min. At 9.13 min C_9 isomers began to appear, the scan on top of the peak at 9.24 min was identified as 4nonene. The peak at 9.50 min indicated the presence of 1-ethenyl-3-methylenecyclopentene. At 9.90 min 7-methyl-3-octene and 2,4-dimethyl-2,4-heptadiene appeared at 10.45-10.57min. From 10.88 to 10.58 min 2,5-diethyltetrahydrofuran, a cyclic ether and after it came 2,2'-bioxepane. C₁₀ isomers were visible after 11.3 min, for example, the scan at 11.30 min identified 2,6,6-trimethyl-bicyclo (3.1.1) heptane, a C₁₀ isomer. The peak centered at 12.72 min was 2,6,6-trimethylbicyclo (3.1.1) heptane $C_{10}H_{18}$. Brominated compounds also were identified in the products of pyrolysis, for example, the peak at 13.00 min was 4bromoheptane C₇H₁₅Br. The peak at 13.21 to 13.66 min was 2-propyl-1-pentanol or 2-ethyl-1-hexanol $C_8H_{18}O$ since their MS is very near to each other judgment is difficult. The lower section of the peak at 13.80 min identified as 6,8-nonadien-2-one, 6-methyl-5-(1methylethylidene), C₁₃H₂₀O. The compound at 26.97 min was isobutyl laurate dodecanoic acid $C_{16}H_{32}O_2$, the compound at 27.74 min was 1-propylpentyl laurate $C_{20}H_{40}O_2$ and at 27.73 min mono (2-ethylhexyl) phthalate C16H22O4, at 27.98 min eicosanoic acid, C20H40O2, at 28.08 to 28.15 min identified as 2,6-ditert-butyl-4-methylphenyl-1-(1-hydroxy-2-methylpropyl) cyclopropanecarboxylate C₂₃H₃₆O₃. The compound at 28.28 min was identified as silane, dimethyl (2-propylphenoxy) butoxy- $C_{15}H_{26}O_2Si$, the compound at 28.27 to 28.46 min was identified as succinic acid, 2-ethylhexyl undecyl ester C₂₃H₄₄O₄, the compound at 29.15 min was identified as (3-beta)- 14-methylcholest-7-en-3-ol C₂₈H₄₈O, the compound at 29.68 min was identified as heptyl ester, toluic acid $C_{15}H_{22}O_2$, the compound at 30.492 was identified as n-tetracosane C₂₄H₅₀, the compound at 30.62 min was identified as 2-ethylhexyl undecyl ester, fumaric acid C₂₃H₄₂O₄, the compound at 30.87 min was identified as 2-chloroethyl decyl ester phthalic acid C₂₀H₂₉ClO₄.

3.2.6 Sample 15

The appearances of the chromatograms of samples 5 to 14 were similar. Therefore, an extended analysis of every sample was omitted. As the chromatogram (Fig. 9 sample 15) showed the sample 15 has higher concentrations of compounds eluted after 15 min RT.

Decomposition of plasticizers derivative of phthalic acid such as di-(2-ethylhexyl) phthalate (Reaction 2,(1)) and polyesters may lead to compounds such as mono-(2-ethylhexyl) phthalate (Reaction 2, (2)), 2-ethylhexanol (Reaction 2, (3)), 2-ethyl-hexanol upon heating and acid catalysis converts to 2-octenes and 3-octenes, some of the chemicals that were observed in the chromatograms.



Reaction 2. A possible route for decomposition of 1,2-benzenedicarboxylic acid, di(2ethylhexyl) ester to 1,2-benzenedicarboxylic acid, mono(2-ethylhexyl) ester, 2-ethylhexanol, 2-octene and 3-octene

Sample 15 was the last sample collected before the air-condenser was clogged with precipitations of the solids. Further studies showed these solids are mostly derivatives of phthalic and benzoic acids (identification of solids at section 3.3).

The content of the main peak at 1.34 min (Fig. 8 Sample 15) is HCI and moisture (Fig. 13, 1.34 min). The TIC at 1.48 also contains moisture and HCI plus other chemicals. When the background was subtracted from the TIC, 2-methyl-butane a C₅ saturated hydrocarbon was identified. At 1.61 min 2,3-dimethylbutane C_6H_{14} , from 1.70-2.27 min a mixture of hexanes was viable. The high abundant materials at 2.41 min identified as benzene, C_6H_6 . At 4.23 min toluene and at 5.06 - 5.84 min 2-ethyl-1-hexene C_8H_{16} , at 5.85 min 3-dimethyl-1-hexene C_8H_{16} were identified. At 8.06 min ethyl-benzene C_8H_{10} , at 8.43 p-xylene C_8H_{10} , at 9.30 min m-xylene C_8H_{10} , at 11.42 min cumene C_9H_{12} , at 11.47 min to 11.62 min 4-chloro-2,4-dimethylhexane C_8H_{17} CI. At 12.11 min benzonitrile C_7H_5N , at 12.21 min hemimellitene, 1,2,3-trimethyl benzene, C_9H_{12} , at 12.28 min undecane, $C_{11}H_{24}$ at 12.62 min 3,3-diethylpentane $C_{10}H_{22}$. It is hard to analyze after RT = 15 min because of super saturation of the column. Table 9 (Appendix) shows the detailed list of the 50 major compounds in the sample 15.

3.2.7 Sample E

Sample E was selected for GC-MS analysis as a representative sample of the fourth stage of pyrolysis. The aqueous samples A to C were strongly acidic. However, TIC of sample E did not show any presence of HCI. The highest abundance of chemicals were: at 1.29 min allyl vinyl ether, at 2.18 min 1-pentene, at 2.50 min benzene, at 4.70 min toluene, 5.00 min

octene, 7.90-9.40 xylene isomers, 9.60 min 2,4-dimethylhexane, 11.34 min ethyltoluene, 11.87 min benzonitrile, 12.08 min hemimellitene, 12.26 min undecane, 17.72 min, 1,2,4-trimethyl-benzene, at 12.94 min 2-ethyl-1-hexanol, at 13.86 min 1-ethyl-2,3-dimethyl-benzene, at 13.99 2-ethyl-1,4-dimethyl-benzene, at 14.08 4-dodecene, at 14.24 tridecane, 14.95 ethyl-styrene, at 15.17 min o-allyl-toluene, 15.25 min 2-methylindene, 15.84 naphthalene 15.79 min 1,3-dimethylindan 15.90 1-Isopropylvinyl)benzene. Table 10 (Appendix) shows the 50 major chemicals in the sample E.

3.3 The Solid Samples

The solid sample (Fig. 2c) and a sample of the precipitants in the condenser were dissolved in methanol: water (90:10) for GC-MS analysis. Fig. 14 contains the TICs used to analyzed the solid samples (a to f).



Fig. 14. TIC of methanol soluble solids: (a) solids crystallized out of joint (Fig. 2c), (b) Hexanes soluble parts of the solid collected from inside of condenser (Fig. 3b), (c) crude solid precipitated in condenser; (d) Purified crystals (Fig. 16e), (e) The solid precipitated in water (Fig. 16 a), (f) solids soluble in hexanes (Fig. 16 c) and its expansion (RT=9-25 min)

3.3.1 The sample crystallized outside of the joints

The solid samples resulted from the leak out of the distillates from the distillation head joints, crystalized outside of receiver joints (Fig. 2c) was soluble in the mixture methanol : water (90:20 v:v) and boiling water. Its aqueous solution was acidic. The sample was recrystallized from water (Fig. 16c). The GC-MS of a few crude crystals dissolved in methanol (Fig. 14a) showed three main picks. The pick at 12.82 min (Fig. 15a) was identified as 2-propylpentanol (Fig. 15b) or 2-ethyl-hexanol (Fig. 15c). The TIC pick centered at 17.80 min (Fig. 15d) was identified with phthalic anhydride (Fig. 15e) or phthalic acid (Fig. 15f) and the final peak located at 21.20 min (Fig. 15g) was identified with di-(1-hexen-5-yl) ester $C_{20}H_{26}O_4$ (Fig. 15 h).



Fig. 15. Mass spectrum of some picks of boiling-water-soluble solid (identified with their retention time (min)) and the most similar MS from NIST library for comparison

The result of GC-MS (Fig. 15a-h) indicated that the crystal was di-(1-hexen-5-yl) ester of phthalic acid. The ester is stable; however, it partially decomposed in GC chamber at high temperature (250°C) to alcohol, and acid (Reaction 3, step 1), the alcohols by losing water converted to alkenes (Reaction 3, step 2). The acid is not stable over 210°C temperature then it converted to anhydride and other products (Reaction 3, step 3).



Reaction 3. A schematic degradation of di-(1-hexen-5-yl) phthalate to phthalic anhydride and alcohol, then the alcohol to di-ene

3.3.2 The solid sample within the distillation head

The solid deposited in the distillation head during the pyrolysis reaction was contaminated with the distillate's liquids. This sample in the boiling water was separated into two phases; water soluble and water insoluble materials (Fig. 16a). The same sample in hexanes was also separated into two phases; part of this sample was hexanes soluble, red in color (Fig. 16b), and most of the solid was insoluble in hexanes then precipitated (pale color solids). Both solutions were strongly acidic. After the water solution cooled down, amorphous and crystalline materials were visible in the aqueous layer and organic materials on the top layer (Fig. 16c). However, there was no change in the hexanes solution (Fig. 16d). Recrystallization of precipitated solid in agues solution produced crystals and amorphous materials (Fig. 16e).



Fig. 16. Solubility of solid products: (a) in hot water, (b) in hexanes, (c) precipitation and crystallization of the solids in water, (d) no change in hexanes solution, (e) shape of pure crystals in water

3.3.3 Analysis of crude solids

The TIC of crude solid precipitate in in the condenser dissolved in methanol/water mixture (90:10) showed several chemicals (Fig. 14b). As Fig. 16a and b show, these solids are contaminated with liquid distillates. The first wide and high peak in TIC (Fig. 14b) relates to solvent, methanol and water mixture (90/10); the end shoulder at 2.28 min was hydrogen chloride. The first major peak (11.25 min) was cumene or Isopropyl benzene C₉H₁₂, and the one at (11.41 min) was 3-chloro-3-methylheptane, C₈H₁₇Cl, or C₁₁H₂₂. The sharp peak at 11.86 min was benzonitrile C₇H₅N, and next small one (12.66 min) was 1-methyl-3-ethylbenzene C₉H₁₂, and the next to it (12.89 min) was 2-propyl-1-pentanol C₈H₁₈O. Fig. 17 (Appendix) shows the MS of the major compound in the sample and the MS of most similar compounds to it searched from NIST library (Fig. 17 second column).

The major compound of this chromatogram located between 15-24 min consisted of phthalic esters derivatives. At 15.30 min benzoic acid $C_7H_6O_2$, at 17.03 min 2-methylbenzoic acid $C_8H_8O_2$, at 18.10 min to 18.53 min phthalic anhydride $C_8H_4O_3$, at 19.32 min 2,6-dimethylnaphthalene $C_{12}H_{12}$, at 19.34 min diphenyl methane, at 19.91 min N-ethylphthalimide $C_{10}H_9NO_2$, at 20.28 min phthalimide or phthalimidomethyl 3-

methoxybenzoate, $C_{17}H_{13}NO_5$, at 20.38 - 20.43 min o-cyanobenzoic acid, $C_8H_5NO_2$, at 21.075 min to 21.43 min phthalic acid, di-(1-hexen-5-yl) ester $C_{20}H_{26}O_4$, at 22.63 min ethylhexylbenzoate $C_{15}H_{22}O_2$, at 23.42 min o-toluic acid, 2-ethylhexyl ester $C_{16}H_{24}O_2$, were identified. Reaction 4 shows the origin of some chemicals found in the chromatogram.



Reaction 4. A schematic degradation of 2-ethylhexyl-2-methylbenzoate to 2methylbenzoic acid and 2-ethylhexanol, then the alcohol to alkenes

3.3.4 The clear crystals

A portion of the solid precipitated in water was dissolved in boiling water then recrystallized in water (Fig. 16e). The TIC of these crystals dissolved in a methanol/water mixture (Fig. 14d) showed three compounds at RT of 13.04, 14.17-17.1, 17.5-18.4, 19.2-21.3 min, identified as 2-ethyl-hexanol, benzoic acid (96% similarity), phthalic anhydride (96% similarity), phthalic acid, di (1-hexen-5-yl) ester (similarity 82%). The assumption that these crystals could be di (1-hexene 5-yl) phthalate that under high temperature in GC injector are decomposed to the indicated compounds is also a valid assumption (Reaction 2).

3.3.5 The precipitated in water

The TIC of amorphous sample precipitated in water (Fig. 14e) is very similar to the purified crystals (Section 3.3.4). MS shows the following chemicals: Benzoic acid (15.1 -17.1 min), 2-methyl benzoic acid (17.59 min), phthalic anhydride (17.69-19.93 min), 3-formyl-benzoic acid (19.40-19.81 min), phthalimide (19.93 min), phthalic acid, and di-(1-hexen-5-yl) ester (20.80-21.50). The results indicated that the amorphous material is a mixture composed of phthalic acid, benzoic acid, and their derivatives.

3.3.6 The hexane soluble

The TIC of hexane soluble portion of the precipitates in the condensers (Fig. 14f) showed chemical content similar to the liquid fractions in addition to be saturated with slightly soluble components which were detected in section 3.3.5. The following is a list of the major chemicals in sample: 3-chloro-3-methylheptane (11.87 min), benzonitrile (12.03 min), benzoic acid (15.50-17.00), phthalic acid (17.74), dimethylnaphthalene (18.74), di-(1-hexen-5-yl) ester of phthalic acid (21.65), ethylhexyl benzoate (22.62). Table 11 (Appendix) contains a more extended list of the chemicals which were absorbed to the solids precipitated in the condenser.

4. CONCLUSION AND REMARKS

The results showed that parts of the pyrolysis products of LSGs are rich in many varieties of chlorinated compounds which are potentially hazardous to the environment and human health. Comparing TICs (Fig. 9) showed that by increasing pyrolysis time the relative amount of HCl and water in the samples decreased. This is consistent with the reported mechanism for thermal degradation of chlorinated polymers and alcohols. The amount of benzene produced increased with time as observed in sample 15; the peak height and area of benzene was higher than combined HCl and moisture. All samples had three kinds of materials, volatile organic hydrocarbons that are suitable as gasoline, visible in the chromatogram from 1.58 to 10 min mainly C_6 to C_{11} hydrocarbons; semi-volatile materials were visible after 11 min with carbons numbers higher than 12 and solid organic acids and esters that appeared after 18 min such as phthalic acids and benzoic acid and their esters used as plasticizers in the processing of the LSG.

The liquid produced from pyrolysis of LEGs was saturated with hydrochloric acid, phthalic acid, benzoic acid and their derivatives. Before using it as clean fuel, particularly, fuel for internal combustion engines, further refinements -- separating fuel worthy chemicals from acids and chlorinated compounds -- is required. The compositions of the remaining materials at the end of the process also depended on the heating limit of the reactor. Below 500°C the residues contained carbon compounds and the ashes obtained above 800°C were white which we suppose they were carbon free. Therefore, the nature, yields and the chemical compositions of the liquids produced by pyrolysis of the waste gloves depended on the engineering of the process and the heating limitation of the reactor. Last but not least, the products of pyrolysis contained highly reactive chemicals; these materials were not chemically stable making the storage life of these liquids rather short. Therefore, the products must be stabilized either by chemical stabilizers or hydrogenation for long-time storage promptly after collection.

In this experiment, 23% of the materials remained in the bottom of the reactor as hard dark solid residue, 36% was collected as liquid saturated in solids, and 4% of the total mass was collected as solid deposited in the condenser. Therefore, 36% of the mass was converted to HCI, and carbon derivative gases (C, C_2 , C_3 , C_4 and C_5). Neoprene has 40% by mass chlorine and potentially, 41.2% of it will convert to HCI, a theoretical yield of 58.8% was expected for combined carbon derivative gases, liquid and solid products from pure neoprene pyrolysis. The higher yield of combined solids and liquids (63%) was due to production of some chlorinated hydrocarbons. In addition a part of gloves mass is chlorine-free fillers and plasticizers which increased the yield of pyrolysis.

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COMPETING INTERESTS

Authors declare that there are no competing interests.

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APPENDIX

Table 3. Thermal history of the reactor: Continuation of pyrolysis and sampling (third time)

Time	T1	T2	Т3	Observations
(Min)	(°C)	(°C)	(°C)	
0	23	23	23	Initial temperature, room temperature
10	42	23	23	Dark steam filled the 5L flask
14	89	51	23	Steam condensation
15	120	59	23	First drop of liquid
16.5	153	73	23	Sample 3 (6.87g)
23	244	107	23.6	Sample 3 (g)
32	328	112	37	Sample 4 (12.4g)
35	350	117	34	Sample 5(19.82g)
39		123	34	Sample 6 (14g)
42	388	140	32	Sample 7(10.97g)
51	402	130	28	Sample 8(12.77g)
56	395	134	28	Sample 9(16.92g)
65	396	147	27	Sample 10 (11.64g)
72		143	28	Sample 11 (12.5g)
90	385	86	28	Liquids in third flask was collected, Volatile Sample(13.26 g)
107	385	54	24	Sample 13 (9.75g)
155	414	71	23.9	Sample 14 (13.5g)
260	410	66	23.1	Sample 15 (16.67g)
297	435	175	23.9	Sample 16 (5 g)

Time	T1	T2	Т3	Observations	
0:00	25.7	27.8	27.7	Initial temperatures	
0:14	82.7	31.3	28.1	Condensation began to appear around the neck of reaction flask	
0:18	105.1	51.8	28.1	H ₂ O droplets appeared in the condenser.	
0:22	117.9	86.3	28.2	First drops of H ₂ O began to condense in the graduated funnel	
0:28	121.3	97.1	28.9	H ₂ O droplets appeared by T3	
0:42	121.6	97.1	39.4	50 mL of H ₂ O was collected (Sample A)	
1:00	121.8	96.8	30.1	45ml of H_2O was collected and some organics were visible in the beaker as well. The heat intensity was increased from 6 to 8	
1:14	123.3	109.2	51.7	Over 75 mL of H ₂ O and organics were collected. Sample B was placed	
1:36	181.5	114.5	41.4	Sample B's container was collected (20.09 g) and Sample C was placed	
1:50	299.9	106.2	31.4	Yellow like crystalline solids were forming around the condenser	
1:56	353.9	97.8	30.8	Sample C's container was collected (16.54 g) and Sample D was	
2:00	388.3	87.9	29.7	Sample B and C's composition and color was noted to be similar. Both being a clouded light yellow solid like substance, with a light reddish brown liquid settling on the top. Sample C had slight solid formation around the container.	
2:18	459.8	60.8	29.0	No liquid appeared to be coming out, solid formations was	
2:26	466.3	71.6	28.9	The solid precipitated around condenser appeared to be bright	
2:36	482.7	69.7	28.9	Sample D's flask was filled with ¼ liquid similar to that of Sample	
			~~ -	B and C	
2:40 2:44	488.1 490.5	73.1 192.9	28.7 28.7	Heat intensity was increased from 8 to the maximum 10 Vigorous clouds of vapor began to rise from the 3 necked round bottom flask and travel down the condenser. Sample D's flask was collected (8.21 g) and Sample F was placed	
2:48	495.0	277.0	32.2	The solid formation began to change color from a yellow to a vibrant red orange as the vapors continued to emit from the reaction flask. The vapors continued to emit from the flask vigorously. Sample E's flask was collected (16.66 g) and Sample E was placed	
2:50	498.1	277.4	35.6	The heat intensity was decreased from the maximum to 9	
2:52	506.1	271.8	36.6	Solid condensate and vapors continued to form as described previously	
2:58	525.1	236.2	32.1	Sample F's flask was collected (17.93) and Sample G was placed	
3:06	548.4	221.6	30.5	Sample G's flask was collected (18.02 g) and Sample H was placed	
3:14	570.5	184.9	30.5	The formation of the crystalline solids were seen throughout the distillation system	
3:28	614.4	135.8	35.3	An aliquot of Sample E (Sample 44) was taken for GC-MS analyzes	
3:32	616.3	130.0	29.9	Sample H's flask was collected (14.52 g) and Sample I was	
3:38	618.2	115.0	30.0	Sample I was collected (1.50 g) and the system was turned off due to the observation that no more liquid was coming from the system	

Table 4. Thermal history of the reactor: Continuation of pyrolysis and sampling (fourth time)









Fig. 9. The MS of some picks of the most volatile sample (identified with their retention time, min) and the MS of most similar compounds to the MS from NIST library

Retention	Start	End	Area	Name
0.46	0.41	0.59	<u>%</u> 0.48	nentene
1 36	1 14	1 38	6 29	Hydrogen chloride
1.84	1.14	2.01	5.13	2 3-dimethyl-hutene
2 32	2.01	2.01	4 68	henzene
4.05	3.72	2.00 4 13	0.72	hentene
4 51	4 25	4 55	0.95	Toluene
4 62	4 55	4 75	0.68	Heptane 3-methyl-
5.33	4.75	5.37	8.42	4-Hexen-3-one, 4-methyl-
5.58	5.37	5.69	9.40	3-Heptene, 3-methyl-
6.09	6.03	6.23	0.90	2-Octene. (Z)-
8.43	8.23	8.55	0.57	o-Xylene
9.23	9.11	9.39	0.14	o-Xylene
9.61	9.42	9.71	0.15	3-Tridecene
9.80	9.71	9.93	0.07	1-Octene, 3-methyl-
10.11	9.95	10.21	0.24	2-Ethyl-4,6-dimethyltetrahydropyran
10.50	10.38	10.57	0.33	Furan, tetrahydro-2,2-dimethyl-5-(1-methylethyl)-
10.62	10.57	10.70	0.23	Furan, 2,5-diethyltetrahydro-
10.81	10.70	10.96	0.29	1,3-Hexadiene, 2,3,5-trimethyl-
11.46	11.38	11.66	1.22	Heptane, 3-chloro-3-methyl-
11.81	11.77	11.88	0.21	Bicyclo[4.1.0]heptane, 3,7,7-trimethyl-,
40.04	44.00	40.40	0.50	(1.alpha.,3.alpha.,6.alpha.)-
12.04	11.88	12.12	0.52	2-Cyclonexene-1-carboxaldenyde, 2,6,6-trimetnyl-
12.55	12.43	12.58	2.60	Pentane, 3,3-dietnyl-
12.70	12.05	12.82	1.07	Benzene, 1-methyl-3-(1-methylethyl)-
12.98	12.82	13.33	3.4Z	2-Propyi- i-pentanoi
13.07	13.75	13.92	0.30	Childroacelle acid, 2-elinyinexyl ester
14.04	14.00	14.15	0.20	methyl-1-propenyl)-, 2-methyl-4-oxo-3-(2,4- pentadienyl)-2-cyclopenten-1-yl ester
14.21	14.13	14.28	0.15	2,6,10-Trimethylundeca-1,3-diene
15.23	15.18	15.25	0.07	Benzene, pentyl-
15.59	15.54	15.62	0.09	3-Phenyl-4,5-dimethyl-2,1-oxaborolane
15.75	15.62	15.78	0.41	2-Benzoyl-4,4-dimethyl-5-phenyl-4H-1,3-diazol-1- ium-1-olate
15.83	15.78	15.87	0.14	1,7,7-Trimethyl-2-vinylbicyclo[2.2.1]hept-2-ene
15.89	15.87	15.95	0.19	1-lodo-2-methylnonane
17.11	17.08	17.15	0.28	n-Butyric acid 2-ethylhexyl ester
17.53	17.49	17.56	0.08	Naphthalene, 5-butyl-1,2,3,4-tetrahydro-

Table 5. The major chemical compositions of the most volatile sample of the pyrolysisprocess

17.76	17.56	17.85	0.88	1,2-Benzenedicarboxylic acid
17.88	17.85	17.92	0.11	Naphthalene, 1,2,3,4-tetrahydro-1,5-dimethyl-
19.50	19.40	19.61	0.39	4-Methylnonanoic acid
19.72	19.61	19.74	0.31	Dodecane, 1-chloro-
20.10	20.08	20.20	0.16	Hexadecane, 1,16-dichloro-
20.27	20.20	20.31	0.17	2-Bromotetradecane
20.38	20.31	20.43	0.10	1,3,2-Dioxaborinane, 2,4-diethyl-5-methyl-6-propyl-
20.51	20.43	20.57	0.09	Naphthalene, 2-butyl-3-hexyl-1,2,3,4-tetrahydro-
20.60	20.57	20.65	0.07	Azulene, 4,6,8-trimethyl-
21.30	21.19	21.38	0.19	2,2,4-Trimethyl-1,3-pentanediol diisobutyrate
22.01	21.88	22.05	0.09	Benzene, 1,1'-(1,3-propanediyl)bis-
22.33	22.23	22.38	0.05	2-Methyl-cis-7,8-epoxyoctadecane
22.41	22.38	22.43	0.03	Hexadecane
22.51	22.43	22.55	0.03	Octanoic acid, 2-ethylhexyl ester
26.83	26.78	26.93	0.16	Dodecanoic acid, isooctyl ester
30.60	30.28	30.73	1.04	1,2-Benzenedicarboxylic acid, diisooctyl ester

Retention	Start	End	Area %	Name
time	time	time		
1.14	1.11	2.22	52.00	Hydrogen chloride
2.27	2.23	2.56	1.23	2-Chloroethanol
2.68	2.65	2.79	0.48	1-Chloro-2-Propanol
2.86	2.79	2.95	0.61	1.4-Dioxane
2.98	2.95	3.07	0.24	2-Chloro-1-propanol
3.47	3.45	3.60	0.43	1.2-Ethanediol
3.69	3.60	3.78	0.10	2-ethoxy-ethanol
4.80	4.77	4.97	0.38	Cvclopentanone
6.03	5.97	6.13	0.10	Cyclotrisiloxane, hexamethyl-
6.69	6.63	6.81	0.23	Thiophene, tetrahydro-2-methyl-
8.41	8.38	8.45	0.08	2-Cvclopenten-1-one, 2-methyl-
8.61	8.45	8.68	0.27	3-Nitropropanoic acid
8 71	8 68	8 78	0.06	2-Ethyl-4 6-dimethyltetrahydropyran
9.05	9.02	9.12	0.15	Euran 2.5-diethyltetrahydro-
9 15	9.12	9.22	0.10	Furan 2.5-diethyltetrahydro-
9.56	0.12	9.68	0.06	Formamide N N-diethyl-
9.90	0.00 0.83	9.00	0.00	2-Nonanol 5-ethyl-
0.00 0 00	0.00 0.01	10 02	0.15	Eumaric acid 3-fluorophenyl hexadecyl ester
10.06	10.07	10.02	1 / 2	Ethanol 2-(2-chloroethoxy)-
10.00	10.02	10.10	0.38	Propanoic acid 3-chloro-
10.76	10.10	10.24	0.00	1 2-Enovyundecane
10.70	10.00	10.75	0.00	Ethanol 2-(2-ethoxyethoxy)-
11 26	11 21	11 30	2 11	2-Pronyl-1-nentanol
11.20	11.21	11.00	0.32	2(3H)-Euranone 5-ethyldihydro-
13.18	13.12	13.25	0.02	2 5-Pyrrolidingdiong
13.10	13.72	13.20	0.05	Benzoic acid
1/ 88	1/ 83	14.03	0.10	2-Hentanol 6-methyl-
15.00	1/ 08	15.05	0.00	Cyclobexane 1 methyl 2 propyl
15.05	15 10	15.05	0.05	3-Nitro-2-butanol
16.45	16.08	16.58	21 78	Dhthalic anhydride
16.80	16.00	16.83	0.13	1/3H) Isobenzofuranone
16.00	16.83	16.05	0.13	Hexanedioic acid
18.46	18.28	18.60	3.43	Phthalimide
10.40	10.20	10.00	0.45	Ethanol 2 [2 (2 chloroethoxy)ethoxy]
10.19	10.15	19.24	0.13	Bis/2 (2 chloroethovy)ethyl)ether
19.51	10.52	19.55	0.12	Dis(2-(2-cillolocilloxy)etilly)etiller Detellic acid, di (1 bayan 5 yl) astar
19.57	10.60	19.02	0.19	Philipalic aciu, ur-(1-nexen-5-yi) ester
19.79	20.26	19.04	0.41	Philiphic acid di (1 bayan 5 yl) astar
20.33	20.20	20.45	0.30	2 Apotulbonzoio poid
21.40 22.55	21.30	21.02	0.07	Z-AUGLYIDENZUIG dulu Ethanol 2 [2 (2 chloroethayy)athayy]
22.00	22.00	22.02	0.00	Pin(2, (2, chloroothovy)othyl)othor
22.03	22.10	22.00	0.00	
24.07 26.26	24.79	24.99	0.12	
20.30	20.24	20.48	0.24	Denzenemenanamine, N-Nydroxy-N-
06.07	26.62	07 4 4	1.46	(prenyimetry)- 1.2 Prepagadial 2 hangulayer 1.2 disected
20.07	20.03	27.11	1.40	I, 2-Propanediol, 3-Denzyloxy-1,2-diacetyl-
29.87	29.73	29.98	0.07	Heneicosane

Table 6. Major chemicals identified in the sample 1 by GC-MS

Retention	Start	End	Area %	Name
time	time	time		
1.15	1.12	1.19	1.23	Hydrogen chloride
1.38	1.37	1.40	0.24	1,3-Butadiene, 2-methyl-
2.27	2.23	2.34	2.16	Benzene
2.71	2.65	2.75	0.51	Heptane
4.31	4.21	4.38	2.28	Heptane, 3-methyl-
4.94	4.84	5.07	3.69	3-Ethyl-3-hexene
5.36	5.13	5.44	11.54	4-Octene, (E)-
5.68	5.60	5.71	4.90	2-Heptene, 3-methyl-
5.81	5.71	5.83	2.91	2-Octene
6.97	6.86	7.03	1.31	Cyclohexane, 1,2-bis(methylene)-
7.41	7.33	7.45	2.69	p-Xylene
7.54	7.45	7.62	1.06	2,4-Hexadiene, 2,3-dimethyl-
7.90	7.85	7.94	0.85	Dichloroacetic acid, 1-cyclopentylethyl ester
8.04	7.94	8.10	1.58	p-Xylene
9.37	9.32	9.59	1.57	1,5,5-Trimethyl-6-methylene-cyclohexene
9.71	9.59	9.80	4.36	trans-2-Undecen-1-ol
10.01	9.80	10.16	15.89	Heptane, 3-chloro-3-methyl-
10.18	10.16	10.22	0.52	Bicyclo[2.2.1]heptane-2,3-dione, 1,7,7- trimethyl-, (1S)-
10.27	10.22	10.29	0.85	Benzene, 1-ethyl-3-methyl-
10.33	10.29	10.38	0.73	Bicyclo[4.1.0]heptane, 3,7,7-trimethyl-, (1.alpha.,3.alpha.,6.alpha.)-
10.49	10.48	10.52	0.50	1,1,4-Trimethylcyclohexane
10.54	10.52	10.61	1.05	Benzene, 1,3,5-trimethyl-
11.00	10.90	11.08	6.97	Heptane, 3-(chloromethyl)-
11.11	11.08	11.13	1.44	Bicyclo[4.1.0]hept-2-ene, 3,7,7-trimethyl-
11.21	11.14	11.23	1.86	Benzene, 1-ethyl-4-methyl-
11.26	11.23	11.28	1.37	Benzene, 1-methyl-2-(1-methylethyl)-
11.67	11.18	11.83	12.24	Unknown
11.76	11.73	11.81	0.43	Bicyclo[3.1.1]hept-2-ene-2-ethanol, 6,6- dimethyl-
11.93	11.89	12.01	1.49	1,4-Cyclohexadiene, 1-methyl-4-(1- methylethyl)-
12.13	12.10	12.20	0.68	Cyclohexene, 4-methyl-1-(1-methylethenyl)-
12.32	12.27	12.38	1.27	2-Bromopropionic acid, 2-ethylhexyl ester
12.45	12.38	12.47	1.93	1,3-Cyclohexadiene, 1-methyl-4-(1-ethylethyl)-
12.50	12.47	12.58	1.67	Benzene, 1-ethenyl-3,5-dimethyl-
12.64	12.60	12.68	0.60	Tridecane
13.39	13.37	13.43	0.44	Benzene, 1-methyl-2-(2-propenyl)-

 Table 7. 50 Major chemicals identified in the sample 2 by GC-MS

13.54	13.43	13.57	0.87	Acetic acid, 2-ethylhexyl ester
13.59	13.57	13.65	0.79	Benzene, 1-ethenyl-4-ethyl-
14.20	14.16	14.27	1.01	Azulene
14.34	14.27	14.38	0.65	Octane, 3,4,5,6-tetramethyl-
14.41	14.38	14.46	0.72	Benzene, 1-methyl-4-(1-methyl-2-propenyl)-
14.87	14.80	14.91	1.54	Bicyclo[3.1.1]heptane, 6,6-dimethyl-3 methylene-
15.62	15.58	15.65	0.49	n-Butyric acid 2-ethylhexyl ester
16.36	16.22	16.38	1.61	Phthalic anhydride
17.61	17.58	17.65	0.44	Pentanoic acid, 2-ethylhexyl ester
18.17	18.13	18.21	0.50	3-Chloropropionic acid, 2-ethylhexyl ester
18.32	18.28	18.35	1.16	3-Hexanol, 3,5-dimethyl-
18.98	18.97	19.01	0.35	3-Heptanone, 2-methyl-
21.45	21.40	21.48	0.86	Benzoic acid, 2-ethylhexyl ester
26.39	26.21	26.58	0.18	Dodecanoic acid, isooctyl ester
27.40	26.58	28.11	1.80	1,2-Benzenedicarboxylic acid, mono(2- ethylhexyl) ester

Retention	Start	End	Area %	Name
time	time	time		
1.18	1.14	2.80	40.00	Hydrogen chloride
4.02	3.67	4.19	0.63	3-Heptene, 3-methyl-
4.22	4.19	4.28	0.13	1,7-Dichloroheptane
4.50	4.28	4.78	1.05	4-octane
5.12	4.78	5.15	2.33	3-Heptene, 3-methyl-
5.43	5.15	5.69	10.03	3-Heptene, 3-methyl-
6.43	6.27	6.87	0.29	Cyclohexene, 3,5-dimethyl-
7.00	6.87	7.32	0.04	4,6-di-tert-Butylresorcinol
8.14	7.97	8.27	0.18	Ethylbenzene
8.42	8.27	8.79	0.50	o-Xylene
8.94	8.79	8.99	0.02	4-Nonene
9.62	9.42	9.74	0.10	3-Tridecene
10.13	9.99	10.42	0.18	2-Ethyl-4,6-dimethyltetrahydropyran
10.52	10.42	10.74	0.36	Furan, 2,5-diethyltetrahydro-
10.83	10.74	10.99	0.16	1,3-Hexadiene, 2,3,5-trimethyl-
11.47	10.99	11.69	0.79	Heptane, 3-chloro-3-methyl-
11.84	11.69	11.92	0.23	Bicyclo[4.1.0]heptane, 3,7,7-trimethyl-
12.19	12.14	12.37	0.29	Octane, 2-chloro-
12.54	12.37	12.59	1.89	Pentane, 3,3-diethyl- or 2,3,5-trinethlyheptane
12.97	12.84	13.24	2.56	2-Propyl-1-pentanol
13.63	13.54	13.79	0.09	Benzoic acid, 4-
10.00	40 -			isopropenylcyclohexenylmethyl ester
13.89	13.79	13.94	0.21	Chloroacetic acid, 2-ethylhexyl ester
14.24	14.14	14.29	0.08	Bicyclo[4.1.0]heptane-7-methanol, 7-bromo-
14.85	14.59	14.99	0.19	1-Decene, 10-bromo-
15.12	14.99	15.29	0.25	Acetic acid, 2-ethylhexyl ester
15.72	15.47	15.82	0.42	Benzoic acid
16.14	16.07	16.22	0.05	Bicyclo[2.2.1]heptane-1-carbonitrile, 4,7,7-
				trimethyl-3-oxo-
16.52	16.22	16.57	0.12	Trichloroacetic acid, 2-ethylhexyl ester
17.24	16.94	17.29	0.25	Benzene, (2-propynyloxy)-
17.43	17.29	17.52	0.11	Naphthalene, 2-methyl-
17.81	17.52	18.04	1.39	1,2-Benzenedicarboxylic acid
18.24	18.19	18.29	0.09	1(3H)-Isobenzofuranone
18.77	18.72	18.82	0.06	1-lodo-2-methylundecane
19.24	18.87	19.37	0.38	Naphthalene, 1,7-dimethyl-
19.85	19.62	19.99	1.12	Phthalimide

Table 8. 50 major chemicals identified in the liquid obtained by the pyrolysis of LEGssample 3

20.85	20.72	20.92	0.07	Bis(2-(2-chloroethoxy)ethyl)ether
21.52	21.47	21.92	0.12	Fluorene, 2,4a-dihydro-
22.04	21.92	22.09	0.05	Benzene, 1,1'-(1,3-propanediyl)bis-
22.64	22.39	22.77	0.26	Benzoic acid, 2-ethylhexyl ester
23.12	23.07	23.17	0.04	1-Adamantanecarboxylic acid, 3-tetradecyl ester
23.19	23.17	23.22	0.02	Benzene, (1-ethyldodecyl)-
23.72	23.64	23.84	0.08	Pentafluoropropionic acid, dodecyl ester
24.52	24.37	24.74	0.23	Hexanedioic acid, dioctyl ester
25.67	25.54	25.82	0.14	6,7-Dimethyl-3,4-dihydro-1H- [1,4]diazepino[3,2,1-hi]indol-2-one
26.32	26.19	26.49	0.13	Xanthophyll
26.87	26.49	26.97	0.28	Dodecanoic acid, isooctyl ester
31.01	30.27	31.82	5.51	1,2-Benzenedicarboxylic acid, mono(2- ethylhexyl) ester









Fig. 13. Mass spectrum of some picks of sample 15 identified with their retention time (min) (first column) and the most similar MS from NIST library (second column) corresponding to the MS of first column.

1.45	1.11	1.59	0.60	HCI
1.62	1.59	1.65	0.09	Butane, 2,3-dimethyl-
1.77	1.65	1.80	0.25	Pentane, 2,3-dimethyl-
1.82	1.80	2.16	0.47	Cyclopropane, 1.2.3-trimethyl-
2.19	2.16	2.23	0.05	Cyclopentene, 1-methyl-
2.42	2.23	2.74	2.92	Benzene
2.81	2 74	3.09	0.56	Oxalic acid allyl nonvl ester
3 13	3.09	3.32	0.19	Cyclopentane 1-ethyl-2-methyl-
3 34	3 32	3 4 3	0.05	Cyclopentane, ethyl-
3.48	3 4 3	3 63	0.00	Cyclohexene 4-methyl-
3.40 3.00	3 63	4 03	0.00	Cyclopentane, 2-isopropyl-1 3-dimethyl-
1 30	1 03	4.00 1 70	3.61	
4.30 5.45	4.03	4.75	5.01	2 Hontono 2 mothyl
5.45	4.79	5.74	0.02	2.2 Dimethyl 1 hoveno
0.00	5.74	0.24	2.13	2,3-Dimethyl 1 evelopsychol
0.34	0.24	0.49	0.21	I,S-Dimetry- I-cyclonexene
0.54	0.49	0.77	0.10	Replane, 2,0-0imethyl-
0.93	6.77	7.32	0.22	Dichloroacetic acid, 2-ethylcyclonexyl ester
8.07	7.32	8.19	0.91	Etnylbenzene
8.44	8.19	8.94	1.74	p-xyiene
9.31	8.94	9.99	2.28	Benzene, 1,3-dimethyl-
10.29	9.99	11.04	1.40	cis-1,4-Dimethyl-2-methylenecyclohexane
11.48	11.04	11.74	3.67	Acetophenone
12.11	11.74	12.49	3.69	Benzonitrile
12.63	12.49	12.74	1.50	Pentane, 3,3-diethyl-
13.10	12.74	13.64	5.56	2-Propyl-1-pentanol
13.79	13.64	13.89	1.49	Benzhydrazide, N2-(2-methoxy-5-nitrobenzylideno)-
13.99	13.89	14.59	4.10	1,4-Cyclohexadiene, 3-ethenyl-1,2-dimethyl-
14.69	14.59	14.94	1.64	Benzene, 1,2,3,5-tetramethyl-
15.29	14.94	15.69	4.49	2,4-Dimethylstyrene
16.06	15.69	16.39	4.72	Naphthalene, 1,2,3,4-tetrahydro-1-methyl-
17.54	16.39	17.74	9.75	Benzoic acid
17.98	17.74	18.24	3.42	Phthalic anhydride
18.95	18.24	19.34	7.27	Tetradecane
19.55	19.34	19.79	2.90	2-Oxabicyclo[2.2.1]heptan-3-one-1-carboxylic acid,
				4,7,7-trimethyl-, 8-oxo-8a-methyldecalin-1-yl ester
19.84	19.79	19.94	0.93	Naphthalene, 2,3-dimethyl-
20.05	19.94	21.09	6.58	1H-Isoindole-1,3(2H)-dione, N-ethyl-
21.24	21.09	22.24	4.86	5-(4-Methylphenyl)cyclohexane-1,3-dione
22.30	22.24	22.59	1.21	Undecyl heptafluorobutyrate
22.73	22.59	23.14	1.68	Benzoic acid, 2-ethylhexyl ester
23.52	23.14	24.94	3.17	Hexadecane
24.99	24.94	27.04	1.46	2H-1-Benzopyran-3-ol. 3.4-dihydro-5.7-dimethoxy-2-
				(3.4.5-trimethoxyphenyl)-, (2R-cis)-
27.47	27.04	28.04	0.54	Phthalic acid, decyl 2-ethylbexyl ester
28 19	28.04	28.94	0.21	1 2-Benzenedicarboxylic acid diisooctyl ester
29.04	28.94	29.14	0.04	Dimethyl 1 2-diisopropenyltricyclo[3 1 0 0(2 4)] hexane-
20.01	20.01	_0.11	0.01	3.6-dicarboxylate
29.34	29 14	29 44	0.06	1 1-Dinhenvl-2-propanol
29.53	29 44	30.04	0.12	Indan-2-one 1-[2-chlorohenzylidene]-
30.76	30.04	31 84	0.12	1.2-Benzenedicarboxylic acid mono(2-ethylbeyyl) ester
00.70			0.00	1,2 Denzene diodrooxyne dolu, mono(2-ethymexyl) ester

Table 9. 50 major chemicals identified in the liquid obtained by the pyrolysis of LEG sample 15

R T	Start T	End	Area %	Name
(min)		Time		
1.12	1.09	1.13	0.03	Unknown mixture
1.15	1.13	1.17	0.06	Water
1.19	1.17	1.23	0.12	Methyl Alcohol
1.29	1.23	1.33	0.17	Ethanol
1.34	1.33	1.38	0.22	Acetone
1.40	1.38	1.53	0.18	Ethyl hydrogen oxalate
1.57	1.53	1.59	1.70	Pentane, 2-methyl-
1.64	1.59	1.68	4.86	Pentane, 3-methyl-
1.86	1.68	1.88	15.55	Hexane
1.92	1.88	1.97	14.46	Trichloromethane
2.01	1.97	2.14	15.14	Hexane, 2-chloro-
2.16	2.14	2.68	7.36	Cyclopentane, methyl-
2.63	2.56	2.66	0.01	1-Hexene
2.73	2.71	2.98	0.01	Heptane
3.00	2.98	3.07	0.04	n-Propyl acetate
4.45	4.03	4.60	6.25	Toluene
4.62	4.60	4.69	0.03	Pentane, 3-chloro-3-methyl-
4.93	4.88	5.04	0.04	Cvclopentane, 1-ethyl-3-methyl-, cis-
5.22	5.04	5.34	0.07	3-Octene. (Z)-
5.48	5.34	5.53	0.03	2-Octene. (E)-
5.59	5.53	5.68	0.03	1.6-Heptadiene, 2.3.6-trimethyl-
7.93	7.87	8.06	0.06	Ethylbenzene
8.27	8.20	8.41	0.15	o-Xvlene
8.45	8.41	8.54	0.03	Octane, 3-methyl-
9.14	9.08	9.33	0.14	o-Xvlene
9.49	9.43	9.58	0.04	Nonane
11.00	10.97	11.04	0.03	Benzene, propyl-
11.08	11.04	11.15	0.11	1-Pentanol, 4-methyl-2-propyl-
11.25	11.15	11.36	0.22	Benzene. 1-ethyl-3-methyl-
11.41	11.36	11.57	1.74	Heptane, 3-chloro-3-methyl-
11.67	11.57	11.71	0.08	Benzene, 1-ethyl-3-methyl-
11.78	11.71	11.88	0.91	Benzonitrile
11.93	11.88	11.96	0.03	Carbonic acid, phenyl undec-10-enyl ester
11.99	11.96	12.09	0.23	Benzene, 1,3,5-trimethyl-
12.18	12.09	12.28	0.11	Octane, 2,7-dimethyl-
12.45	12.28	12.49	0.16	Pentane, 3,3-diethyl-
12.52	12.49	12.57	0.05	Cyclopropane, 1,2-dimethyl-3-pentyl-
12.64	12.57	12.78	0.29	Benzene, 1,2,3-trimethyl-
12.86	12.78	12.98	0.30	2-Propyl-1-pentanol
13.02	12.98	13.07	0.02	2-Tridecen-1-ol, (E)-
13.10	13.07	13.17	0.06	Indene
13.26	13.17	13.30	0.10	Benzene, 1-methyl-3-propyl-
13.35	13.30	13.38	0.07	1,2,3,4,5,8-Hexahydronaphthalene
13.41	13.38	13.44	0.06	Benzene, 4-ethyl-1,2-dimethyl-
13.47	13.44	13.50	0.02	3a,6-Methano-3aH-indene, 2,3,4,5,6,7-hexahydro-
13.59	13.50	13.68	0.40	Benzeneacetonitrile, .alphaacetyl-

Table 10. 50 major chemicals identified in the liquid obtained by the pyrolysis of LEG sample E

13.81	13.68 13.80	13.89	0.18	Benzene, 1-ethyl-2,3-dimethyl-
15.95	15.05	13.90	0.11	methylene-
14.04	13.98	14.09	0.12	5-Chloropentanoic acid, 2-octyl ester
14.19	14.09	14.24	0.17	Undecane
14.32	14.24	14.35	0.08	Benzene, 4-ethyl-1,2-dimethyl-
14.38	14.35	14.41	0.03	Benzene, 1-methyl-4-(1-methylpropyl)-
14.49	14.41	14.53	0.05	Spiro[3.5]nona-5,7-dien-1-one, 5,9,9-trimethyl-
14.57	14.53	14.63	0.05	Benzene, 1,2,4,5-tetramethyl-
14.68	14.64	14.71	0.03	Decane, 3,7-dimethyl-
14.91	14.83	14.95	0.09	1H-Indene, 2,3-dihydro-5-methyl-
14.99	14.95	15.03	0.05	Benzene, 1,4-diethyl-2-methyl-
15.10	15.03	15.16	0.23	1-Phenyl-1-butene
15.18	15.16	15.24	0.11	2-Methylindene
15.30	15.24	15.33	0.06	Benzene, 2,4-diethyl-1-methyl-
15.35	15.33	15.42	0.08	1-Butanone, 1-phenyl-
15.45	15.42	15.48	0.26	Benzoic acid, ethyl ester
15.73	15.48	15.78	0.39	1H-Indene, 2,3-dihydro-1,6-dimethyl-
15.90	15.78	15.95	0.67	1H-Indene, 2,3-dihydro-1,6-dimethyl-
17.05	15.95	17.14	3.51	Benzoic acid
17.22	17.14	17.25	0.28	1H-Indene, 2,3-dihydro-1,3-dimethyl-
17.41	17.25	17.48	0.72	Naphthalene, 2-methyl-
17.52	17.48	17.55	0.30	Benzene, 4-(2-butenyl)-1,2-dimethyl-, (E)-
17.60	17.55	17.63	0.31	1H-Inden-1-one, 2,3-dihydro-2-methyl-
17.80	17.63	17.82	2.18	Phthalic anhydride
17.89	17.82	17.94	0.53	1H-Indene, 2,3-dihydro-1,1,5-trimethyl-
17.96	17.94	18.01	0.10	3-(4-Cyanomethyl-1H-pyrrol-3-yl)-propionitrile
18.03	18.01	18.08	0.13	3-Hydroxymethyl-4-(1-hydroxy-2-methylprop-2-
				enyl)toluene
18.13	18.08	18.18	0.38	Naphthalene, 1,2,3,4-tetrahydro-2,7-dimethyl-
18.21	18.18	18.26	0.21	1(3H)-Isobenzofuranone
18.31	18.26	18.34	0.26	Naphthalene, 1,2,3,4-tetrahydro-1,1,6-trimethyl-
18.42	18.34	18.49	0.28	2-Buten-1-one, 2-methyl-1-phenyl-
18.54	18.49	18.57	0.38	Benzene, 2-(2-butenyl)-1,3,5-trimethyl-
18.61	18.57	18.68	0.31	Biphenyl
18.76	18.68	18.78	0.46	Silane, trichlorooctadecyl-
18.82	18.78	18.84	0.42	Naphthalene, 2-ethyl-
18.87	18.84	18.92	0.27	Naphthalene, 1-ethyl-
18.97	18.92	19.05	0.48	Naphthalene, 2,6-dimethyl-
19.09	19.05	19.13	0.31	7-Isopropylidenebicyclo[2.2.1]hept-5-en-2-one p-
				toluenesulfonylhydrazone
19.23	19.13	19.26	2.04	Naphthalene, 2,6-dimethyl-
19.31	19.26	19.40	0.63	Diphenylmethane
19.45	19.40	19.58	0.49	Benzene, 2-chloro-1,3,5-trimethyl-
19.64	19.58	19.67	0.40	Naphthalene, 1,2,3,4-tetrahydro-1,1,6-trimethyl-
19.76	19.67	19.79	0.63	o-Cyanobenzoic acid
19.86	19.79	19.88	0.58	1H-Isoindole-1,3(2H)-dione, N-ethyl-
19.96	19.88	19.98	0.27	Hexadecane, 1,16-dichloro-
20.05	19.98	20.08	0.71	Pentadecane
20.11	20.08	20.19	0.39	1,2-Naphthalenediol, 2-ethyl-1,2,3,4-tetrahydro-1-
				methyl-, cis-

20.25	20.19	20.36	0.37	Hexanoic acid, 2-ethylhexyl ester
20.38	20.36	20.42	0.21	11-Dodecen-1-ol, 2,4,6-trimethyl-, (R,R,R)-
20.49	20.42	20.55	0.26	Naphthalene, 1,4,6-trimethyl-
20.60	20.55	20.65	0.56	Azulene, 4,6,8-trimethyl-
20.67	20.65	20.68	0.16	Benzenebutanoic acid, 2,5-dimethyl-
20.70	20.68	20.75	0.16	Benzyl alcohol, .alphaisobutyl-2,4,5-trimethyl-
20.80	20.75	20.90	0.28	3-(2-Methyl-propenyl)-1H-indene
20.98	20.90	20.99	0.20	Benzene, 1,1'-[3-(3-cyclopentylpropyl)-1,5-
				pentanediyl]bis-
21.01	20.99	21.07	0.21	Azulene, 4,6,8-trimethyl-
21.11	21.07	21.13	0.28	2,3,4-Trifluorobenzoic acid, 3-phenylpropyl ester
21.17	21.13	21.19	0.20	Cycloeicosane
21.26	21.19	21.28	0.49	Pentadecane
21.32	21.28	21.37	0.38	3-(2-Methyl-propenyl)-1H-indene
21.42	21.37	21.53	0.16	1,1'-Biphenyl, 2,4'-dimethyl-
21.68	21.53	21.72	0.70	Phthalic acid, di-(1-hexen-5-yl) ester
21.78	21.72	21.81	0.22	.gammaChlorobutyrophenone
21.83	21.81	21.88	0.13	Heptacosane, 1-chloro-
21.93	21.88	21.98	0.11	Naphthalene, 1-methyl-7-(1-methylethyl)-
22.01	21.98	22.09	0.23	Benzene, 1,1'-(1,3-propanediyl)bis-
22.18	22.09	22.21	0.16	N-n-Butylphthalimide
22.25	22.21	22.29	0.10	7-Heptadecene, 1-chloro-
22.32	22.29	22.38	0.10	Naphthalene, 1,6-dimethyl-4-(1-methylethyl)-
22.40	22.38	22.43	0.34	Hexadecane
22.50	22.43	22.56	0.08	Hexane, 2-phenyl-3-propyl-
22.62	22.56	22.66	0.81	Benzoic acid, 2-ethylhexyl ester
22.69	22.66	22.73	0.06	1,1'-Biphenyl, 2,4'-dimethyl-
22.77	22.73	22.83	0.12	1,2-Diphenyl-1-isocyanoethane
22.92	22.83	22.96	0.10	1,2-Diphenylcyclopropane
22.99	22.96	23.05	0.04	Azulene, 7-ethyl-1,4-dimethyl-
23.15	23.05	23.19	0.02	Benzene, 1,1'-(1-methyl-1,3-propanediyl)bis-
23.41	23.38	23.46	0.08	o-Toluic acid, 2-ethylhexyl ester
23.49	23.46	23.59	0.14	Pentadecane
23.82	23.79	23.90	0.05	Benzene, 1,1'-(1,3-butadienylidene)bis-

RT	Start	End	%	Name
(min)			Area	
4.93	4.88	5.04	0.02	Cyclopentane, 1-ethyl-3-methyl-, cis-
5.22	5.04	5.34	0.06	3-Octene, (Z)-
5.48	5.34	5.53	0.02	2-Octene, (E)-
5.59	5.53	5.68	0.01	1,6-Heptadiene, 2,3,6-trimethyl-
7.93	7.87	8.06	0.03	Ethylbenzene
8.27	8.20	8.41	0.09	o-Xylene
8.45	8.41	8.54	0.01	Octane, 3-methyl-
9.14	9.08	9.33	0.08	o-Xylene
9.49	9.43	9.58	0.02	Nonane
11.00	10.97	11.04	0.01	Benzene, propyl-
11.08	11.04	11.15	0.04	1-Pentanol, 4-methyl-2-propyl-
11.25	11.15	11.36	0.15	Benzene, 1-ethyl-3-methyl-
11.41	11.36	11.57	0.55	Heptane, 3-chloro-3-methyl-
11.67	11.57	11.71	0.02	Benzene, 1-ethyl-3-methyl-
11.78	11.71	11.88	0.29	Benzonitrile
11.93	11.88	11.96	0.01	Carbonic acid, phenyl undec-10-enyl ester
11.99	11.96	12.09	0.08	Benzene, 1,3,5-trimethyl-
12.18	12.09	12.28	0.04	Octane, 2,7-dimethyl-
12.45	12.28	12.49	0.05	Pentane, 3,3-diethyl-
12.52	12.49	12.57	0.01	Cyclopropane, 1,2-dimethyl-3-pentyl-
12.64	12.57	12.78	0.11	Benzene, 1,2,3-trimethyl-
12.86	12.78	12.98	0.13	2-Propyl-1-pentanol
13.02	12.98	13.07	0.01	2-Tridecen-1-ol, (E)-
13.10	13.07	13.17	0.02	Indene
13.26	13.17	13.30	0.03	Benzene, 1-methyl-3-propyl-
13.35	13.30	13.38	0.03	1,2,3,4,5,8-Hexahydronaphthalene
13.41	13.38	13.44	0.02	Benzene, 4-ethyl-1,2-dimethyl-
13.47	13.44	13.50	0.01	3a,6-Methano-3aH-indene, 2,3,4,5,6,7-hexahydro-
13.59	13.50	13.68	0.14	Benzeneacetonitrile, .alphaacetyl-
13.81	13.68	13.89	0.08	Benzene, 1-ethyl-2,3-dimethyl-
13.93	13.89	13.98	0.04	1,3-Cyclopentadiene, 1,2,3,4-tetramethyl-5-
				methylene-
14.04	13.98	14.09	0.04	5-Chloropentanoic acid, 2-octyl ester
14.19	14.09	14.24	0.05	Undecane
14.32	14.24	14.35	0.03	Benzene, 4-ethyl-1,2-dimethyl-
14.38	14.35	14.41	0.01	Benzene, 1-methyl-4-(1-methylpropyl)-
14.49	14.41	14.53	0.01	Spiro[3.5]nona-5,7-dien-1-one, 5,9,9-trimethyl-
14.57	14.53	14.63	0.02	Benzene, 1,2,4,5-tetramethyl-
14.68	14.64	14.71	0.01	Decane, 3,7-dimethyl-
14.91	14.83	14.95	0.03	1H-Indene, 2,3-dihydro-5-methyl-
14.99	14.95	15.03	0.02	Benzene, 1,4-diethyl-2-methyl-
15.10	15.03	15.16	0.10	1-Phenyl-1-butene
15.18	15.16	15.24	0.05	2-Methylindene
15.30	15.24	15.33	0.03	Benzene, 2,4-diethyl-1-methyl-
15.35	15.33	15.42	0.04	1-Butanone, 1-phenyl-
15.45	15.42	15.48	0.06	Benzoic acid, ethyl ester
15.73	15.48	15.78	0.42	1H-Indene, 2,3-dihydro-1,6-dimethyl-

Table 11. Major chemicals in the solids precipitated inside of the condenser.

15.90	15.78	15.95	0.58	1H-Indene, 2,3-dihydro-1,6-dimethyl-
17.05	15.95	17.14	14.49	Benzoic acio
17.44	17.14	17.20 17.40	0.12	Nanhthalana, 2, mathul
17.41	17.20	17.40	0.49	Raphulaiche, z-meuryi- Ronzono 4 (2 butonyi) 1 2 dimothyi (E)
17.52	17.40	17.55	0.10	1H Indon 1 ono 23 dibudro 2 mothyl
17.00	17.55	17.03	1.07	Dhthalic anhydride
17.00	17.00	17.02	0.21	1H Indene 2.3 dibydro 1.1.5 trimethyl
17.09	17.02	18.01	0.21	3 (1 Cyanomethyl 1H pyrrol 3 yl) propionitrile
18.03	18.01	18.08	0.04	$3-Hvdroxymethyl_{-1}-(1-bydroxy-2-methylpron-2-$
10.05	10.01	10.00	0.00	envi)toluene
18 13	18 08	18 18	0 15	Naphthalene 1234-tetrahydro-27-dimethyl-
18 21	18.00	18.26	0.09	1(3H)-Isobenzofuranone
18.31	18.26	18.34	0.00	Nanhthalene 1234-tetrahvdro-116-trimethyl-
18.42	18.34	18.49	0.20	2-Buten-1-one 2-methyl-1-phenyl-
18.54	18.49	18.57	0.13	Benzene, 2-(2-butenvl)-1.3.5-trimethvl-
18.61	18.57	18.68	0.21	Biphenyl
18.76	18.68	18.78	0.21	Silane, trichlorooctadecyl-
18.82	18.78	18.84	0.13	Naphthalene, 2-ethyl-
18.87	18.84	18.92	0.13	Naphthalene, 1-ethyl-
18.97	18.92	19.05	0.26	Naphthalene, 2.6-dimethyl-
19.09	19.05	19.13	0.13	7-Isopropylidenebicyclo[2.2.1]hept-5-en-2-one p-
				toluenesulfonylhydrazone
19.23	19.13	19.26	0.67	Naphthalene, 2,6-dimethyl-
19.31	19.26	19.40	0.31	Diphenylmethane
19.45	19.40	19.58	0.39	Benzene, 2-chloro-1,3,5-trimethyl-
19.64	19.58	19.67	0.16	Naphthalene, 1,2,3,4-tetrahydro-1,1,6-trimethyl-
19.76	19.67	19.79	0.29	o-Cyanobenzoic acid
19.86	19.79	19.88	0.27	1H-Isoindole-1,3(2H)-dione, N-ethyl-
19.96	19.88	19.98	0.17	Hexadecane, 1,16-dichloro-
20.05	19.98	20.08	0.25	Pentadecane
20.11	20.08	20.19	0.22	1,2-Naphthalenediol, 2-ethyl-1,2,3,4-tetrahydro-1-
				methyl-, cis-
20.25	20.19	20.36	0.31	Hexanoic acid, 2-ethylhexyl ester
20.38	20.36	20.42	0.08	11-Dodecen-1-ol, 2,4,6-trimethyl-, (R,R,R)-
20.49	20.42	20.55	0.20	Naphthalene, 1,4,6-trimethyl-
20.60	20.55	20.65	0.21	Azulene, 4,6,8-trimethyl-
20.67	20.65	20.68	0.04	Benzenebutanoic acid, 2,5-dimethyl-
20.70	20.68	20.75	0.08	Benzyl alcohol, .alphaisobutyl-2,4,5-trimethyl-
20.80	20.75	20.90	0.21	3-(2-Methyl-propenyl)-1H-indene
20.98	20.90	20.99	0.10	Benzene, 1,1'-[3-(3-cyclopentylpropyl)-1,5-
	~~ ~~	04.07	0.40	pentanediyljbis-
21.01	20.99	21.07	0.10	Azulene, 4,6,8-trimethyl-
21.11	21.07	21.13	0.10	2,3,4-1 rifluorobenzoic acid, 3-phenylpropyl ester
21.17	21.13	21.19	0.07	
21.26	21.19	21.28	0.17	
21.32	21.28	21.37	0.13	3-(2-ivietnyi-propenyi)-1H-indene
21.42	21.37	21.53	0.15	I, I -DIPRENYI, 2,4 -OIME(NYI- Deteolio opid, di (1, hover, 5, vi) opter
∠1.00 21.70	∠1.03 01 70	Z1./Z	0.40 0.40	rinnanc aciu, ui-(i-nexen-o-yi) ester
21./Ö	21.72	∠1.ŏ1 04.00	0.10	.yanimaChiorobutyrophenone
∠1.ŏJ	21.01	∠1.ŏŏ	0.05	neptacosane, i-chioro-

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21.93	21.88	21.98	0.07	Naphthalene, 1-methyl-7-(1-methylethyl)-
22.01	21.98	22.09	0.11	Benzene, 1,1'-(1,3-propanediyl)bis-
22.18	22.09	22.21	0.07	N-n-Butylphthalimide
22.25	22.21	22.29	0.05	7-Heptadecene, 1-chloro-
22.32	22.29	22.38	0.05	Naphthalene, 1,6-dimethyl-4-(1-methylethyl)-
22.40	22.38	22.43	0.07	Hexadecane
22.50	22.43	22.56	0.05	Hexane, 2-phenyl-3-propyl-
22.62	22.56	22.66	0.17	Benzoic acid, 2-ethylhexyl ester
22.69	22.66	22.73	0.03	1,1'-Biphenyl, 2,4'-dimethyl-
22.77	22.73	22.83	0.05	1,2-Diphenyl-1-isocyanoethane
22.92	22.83	22.96	0.04	1,2-Diphenylcyclopropane
22.99	22.96	23.05	0.02	Azulene, 7-ethyl-1,4-dimethyl-
23.15	23.05	23.19	0.01	Benzene, 1,1'-(1-methyl-1,3-propanediyl)bis-
23.41	23.38	23.46	0.02	o-Toluic acid, 2-ethylhexyl ester
23.49	23.46	23.59	0.03	Pentadecane
23.82	23.79	23.90	0.01	Benzene, 1,1'-(1,3-butadienylidene)bis-









Fig. 17. Mass spectrum of some picks of crude solids deposited in the condenser identified with their retention time (min) (first column) and the most similar MS from NIST library (second column) corresponding to the MS of first column

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