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Preparation of the Antioxidant Compounds from the Styrene and P-Benzoquinone Compounds to the Lubricant Oils

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Abstract

In this study, new antioxidants were synthesized using (thiol-ene) click reaction where, octane and dodecane thiol were added to styrene which produced from plastic waste as (1:1) mole ratio at rate temperature to obtain the following antioxidant compounds: Octyl(2-phenylethyl) sulfane and Dodecyl(2-phenylethyl) sulfane respectively. Octane and dodecane thiol were also added to p-benzoquinone compound to obtain of the following antioxidant compounds, 2-(Octylthio) benzene-1,4-diol and 2-(Dodecylthio) benzene-1,4-diol respectively. These synthesized antioxidants were added to the lubricating oils such as, engine and hydraulic oils with base oil as a solvent, and then mixed well. Then the characterization of these synthesized antioxidant compounds were specified by using techniques such as, (FT-IR), (NMR) and mass spectroscopy. Then study their efficacy as an antioxidant compounds by using of (FT-IR) and (cyclic voltammetry) techniques.

Keywords: (1-Octanethiol), (1-Dodecanethiol), Styrene, P-Benzoquinone, Michael addition, Lubricants oils.

تحظير مركبات مضاد الاكسدة من مركبات الستايرين والبارابنزوكوينون الى زيوت التزييت.

فائز سمير صالح رائد كاظم زيدان خنساء عبد الرزاق علي ٣
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المستخلص

في هذه الدراسة، تم تحظير مضادات اكسدة باستخدام تفاعل (ثايول-اين)، حيث تم اضافة اوكتان و دوديكان الثايول الى الستايرين والذي تم انتاجه من المخلفات البلاستيكية بنسبة (١:١) مول بدرجة حرارة الغرفة للحصول مركبات

مضاد الاكسدة التالية : اوكتايل - ٢ – فينايل ايثايل سلفين و دوديكايل - ٢ - فينايل ايثايل سلفين على التوالي . اوكتان ودوديكان الثايول يتم اضافتهم ايضا الى مركب بارا بنزو كوينون للحصول على مركبات مضاد الاكسدة التالية : ٢ - اوكتايل ثايو بنزين - ١,٤ - دايول و ٢ - دوديكايل ثايو بنزين - ١,٤ - دايول على التوالي . مركبات مضادا الاكسدة التي تم تحضيرها يتم اضافتهم الى زيوت التتزييت مثل ، زيوت المكائن والهيدروليك مع الزيت الاساس الذي يعمل كمذيب ثم يتم مزجهم جيدا. يتم تشخيص مركبات مضاد الاكسدة التي تم تحضيرها باستخدام تقنيات الاشعة تحت الحمراء و مطيافية الرنين النووي المغناطيسي و مطيافية الكتلة ويتم دراسة كفائتها باستخدام تقنية الاشعة تحت الحمراء وتقنية الفولتامتري الحلقي.

الكلمات المفتاحية :- وكتان ثايول ، دوديكان ثايول ، ستايرين ، بارابنزوكوينون ، اضافة مايكل ، زيوت التزييت .

1. Introduction

The lubrication process decreases the friction that happens to the engines during their operation, as the work of lubricating oils is to form an insulating layer between two surfaces to prevent the process of their friction.

Lubricating oils additives are materials or mixes of materials combined with lubricant fluid. They improve the properties of the lubricating oils and help to increase their efficiency in the applications [1]. Oxidation is a process that gets to lubricating oils when exposed to external influences like, moisture and heat [2]. Antioxidants are compounds that are able to inhibit the oxidative processes for lubricating oils and prevent the peroxidation of lubricants [3]. The oxidation process is the main reason for increasing the thickness of oil (viscosity), the (pour point), sediment and machine corrosion [4]. The degradation process which obtains to the lubricating oils extremely hasten if the machine operating conditions are under harsh conditions like, highly temperatures degrees, operating the machine under cold or extremely height pressure [5].

The oxidation process leads to an increase in the viscosity to the lubricant oil; it depends on the rate of the oxygen which is utilized as a result of this process [6]. The oxidation impedance of the lubricating oils can be defined as a chemical function of the efficiency of the additive compound used to counter the different ways of the oxidizing deterioration of the lubricating oils when exposed to oxygen and water at sufficient rising temperatures and the impact of the metals that are found in the lubricant oils [7].

When the oxidation process occurs to the lubricating oils leads to the formation of the oxides to the carbonyl compounds. This can be explained by the (FT-IR) technique [8]. There are many ways for determining the characteristics of the antioxidant compounds in lubricant oils. In accordance with various antioxidant compound characteristics of lubricant oil, various ways may be utilized, like: measurement of the variations of the antioxidant compound that is used in the lubricant oil, like the cyclic voltammetry, measurement of the degeneration of the chemical characteristics, like the rate of total acid and total base, limitation of variations in the content of chemical function series of the lubricant oil, like the infrared spectroscopy technique [8,9]. Antioxidants work to block dissociation processes by inhibiting the propagation of free radicals. For this purpose, thermal stabilizers are used, which are phenolic compound (substituted phenolic), secondary aromatic amines, or sterically amines that work by giving them the effective hydrogen of free radicals, especially peroxide radicals, according the equation below, (AH) represent as antioxidant.

$$ROO^{\cdot} + AH \rightarrow ROOH + A^{\cdot}$$

($A^{\cdot}$) must be stable to prevent propagation of new free radicals through the delocalization of electron or by the resonance [8,9,36].

2. Experimental Materials:

The materials that were used in this study were obtained from (Sigma-Aldrich) and the solvents were obtained from (Fluka, AG).

3. The characteristics of the (base oil) that is utilized in this study

The table below shows the physical properties of the base oil according to (American Society for Testing and Materials, ASTM).

Table -1- indicate the characterization of the base oil

Test	Base Oil	Method (ASTM)
Viscosity @ ($40C^0$)	62.0	D-445
Viscosity @ ($100C^0$)	8.4	D-445
Viscosity Index (VI)	99	D-2270
Specific Gravity (Sp.Gr) @($15.6C^0$)	0.8830	D-4052
Pour Point (p.p) (C^O)	-5	D-97
Flash Point (F.P) (C^0)	249	D-92
Color	2.5	D-1500

4. Instruments

Melting point was measured using meter (BuCi510) made in (Switzerland). (FT-IR) spectroscopy to indicate the functional groups in the compounds (84005-SHIEMADZU- made in Japan), through the use of a desk made of KBr compound (college of education, chemistry department, Basra University). The 1H-NMR spectra are recorded using (Burker, 400MHz), solvent that is utilized is (DMSO, d^6 at $25C^0$), university of Tehran. Mass spectra were measured, (SHIMADZU, Teheran University).

5. Testing methods

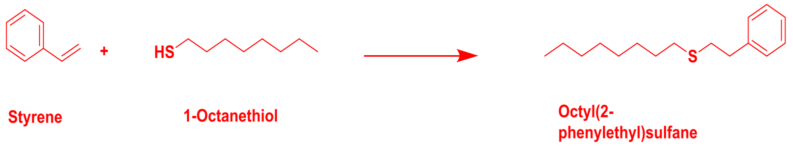
Thin – layer chromatography (TLC), was used to monitor the reaction and to check the purity of the synthesized compounds. (TLC) is performing through the utilizing of sheets from the aluminum that is pre-coating together with (silica gel, 60), which is supplies by (Merck), the solvent in (TLC) (ethanol and n-hexane) (2:7) respectively [32].

5.1. Synthesis of (P-Benzoquinone)

Take 1g of hydroquinone and 10ml of distilled water in a beaker 25ml. Heat the mixture to obtain a clear solution. Take 2g of potassium dichromate in a conical flask and dissolve in 20ml of distilled water and add (2ml) of concentration (H_2SO_4). Shake and cool the conical flask in ice water. To this ice cold solution add hydroquinone solution drop wise over a period of 30 minutes with constant shaking, until showed (yellow crystals) of p-benzoquinone, filtered the mixture and drying the crystals, then recrystallization of the p-benzoquinone by using of the ethanol.

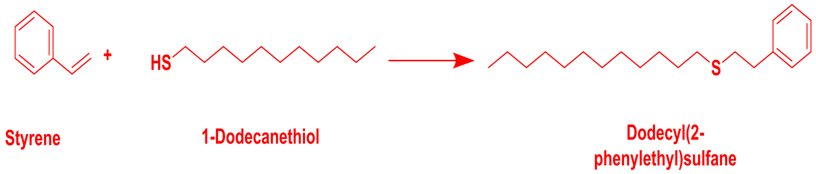
5.2. Synthesis of Octyl (2-phenylethyl) sulfane

Styrene (1.04g, 10mmol) was mixed with (1-octanethiol) (2.19g, 15mmol), for (4.5 hrs) at (80 C^{o}), the yield of the product is (86%).



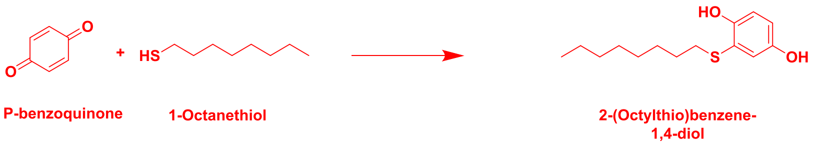
5.3. Synthesis of Dodecyl (2-phenylethyl) sulfane

(1.04g, 10mmol) of styrene was mixed with (1-dodecanethiol) (3.03g, 15mmol) for (4.5 hrs) at (80C^{0}), the yield of the product is (86%).



5.4. Synthesis of 2-(Octylthio) benzene-1,4-diol

Weight (0.21g, 2mmol) of P-Benzoquinone was mixed with (1-octanethiol compound) (0.30g, 2mmol) and (5ml of water), the mixture is being extracted twice through the utilizing of (10ml) ethyl acetate; the yield of the product is (94%).



5.5. Synthesis of 2-(Dodecylthio) benzene-1,4- diol

Weight (0.21g, 2mmol) of p-benzoquinone was mixed with the (1-dodecanethiol compound) (0.40g, 2mmol) and (5ml) of water, the mixture is being extracted twice through the utilizing of (10ml) of ethyl acetate. The yield of the product is (93%).

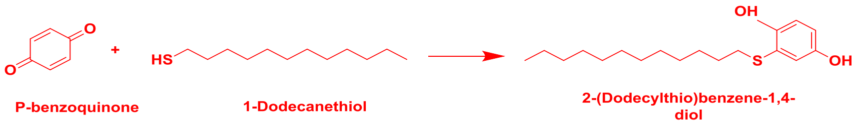


Table -2- indicate the molecular structure of the synthesis antioxidant compounds

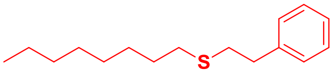
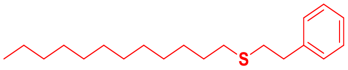
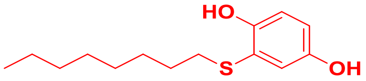
Com.No	Formula	Structure
1	$C_{16}H_{26}S$	
2	$C_{20}H_{34}S$	
3	$C_{14}H_{22}O_{2}S$	
4	$C_{18}H_{30}O_{2}S$	

Table-3-indicate the physical characteristics of the synthesis antioxidant Compounds

Com.No	M.Wt (g/mole)	Color	M.P (C^{O})	Yield (%)
1	250.4	Pale Yellow	viscous	86
2	306.6	Pale Yellow	viscous	86
3	254.4	Dark Red	256-258	94
4	310.5	Dark Red	301-303	93

6. Synthesis of the lubricant oil blends

(0.02g) of prepared compounds (antioxidants) were mixed with (base oil: lubricating oil) in a ratio (1:1), mixed them well through the utilization of a magnetic stirrer at a temperature ($60C^{0}$, 30 minutes).

7. Results and discussion

7.1. FT-IR spectroscopy of compounds (1,2)

The spectrum of the compound (1): Octyl (2-phenylethyl) sulfane was interpreted by using the infrared spectroscopy (FT-IR) technique in the figure (1)[10-15]:

IR(cm^{-1}) : 1452 (C=C, Ar), 3026 (CH, Ar), 2870 (CH,CH_3, Al), 698 (C-S).

Ar = Aromatic compound.

Al = Aliphatic compound.

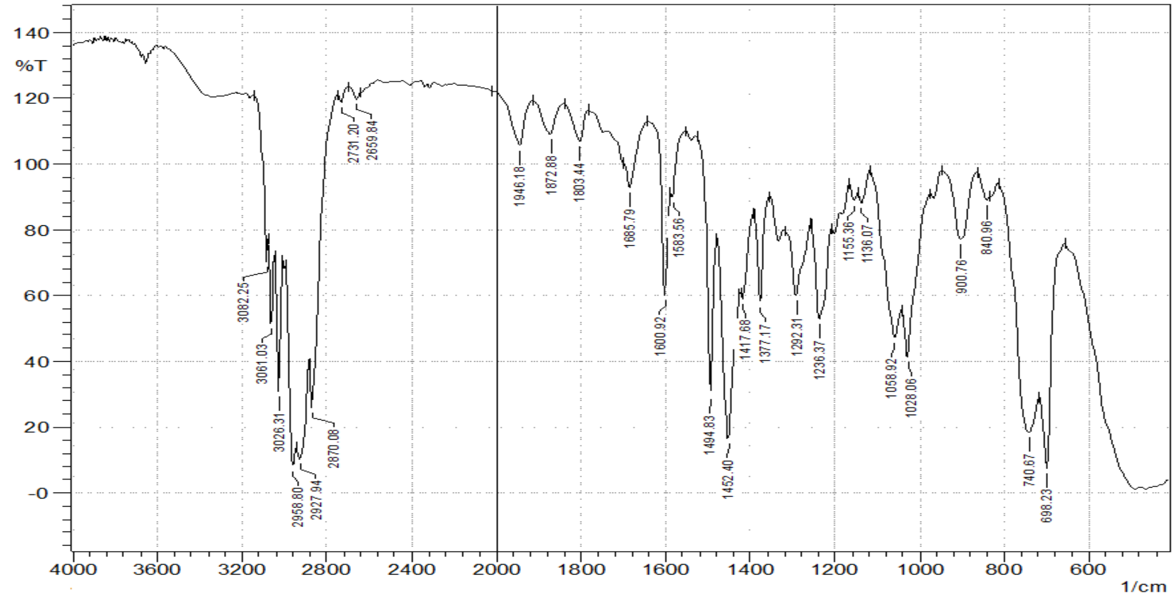


Figure – 1- (FT-IR) spectrum of compound (1).

The (FT-IR) spectrum of the compound (2): Dodecyl (2-phenylethyl) sulfane, as interpreted in the figure (2)[10-15] :

IR(cm^{-1}) : 1450 (C=C, Ar), 3025 (CH, Ar), 2871 (CH,CH_3,Al), 699 (C-S).

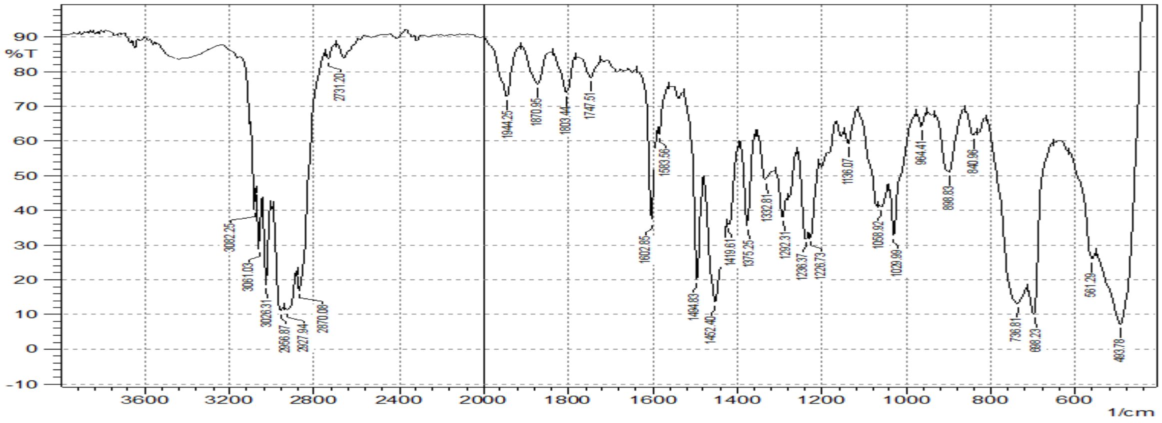
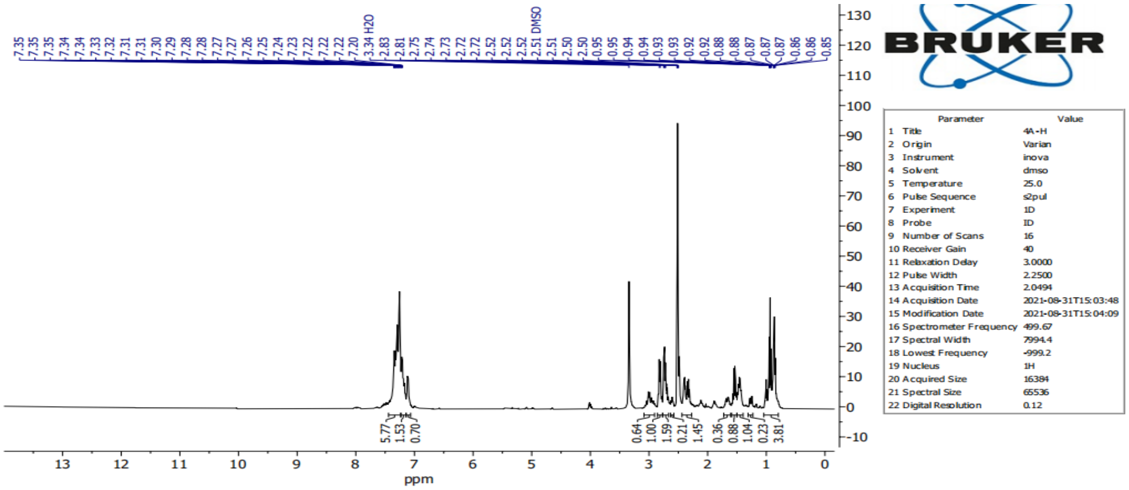


Figure - 2- (FT-IR) spectrum of compound (2).

7.2. 1H-NMR spectroscopy of compounds (1,2)

The (1H-NMR) spectrum of the compound (1): Octyl (2-phenylethyl) sulfane**,** indicate the chemical shifts, as interpreted in the figure (3)[15-17,37]:

(Ar-H) appeared as (multiple signals) at (7.02-7.94) (ppm), 2.69 (q, S-CH_2), 1.06-1.65 (m, CH_2), 0.88 (t, CH_3).

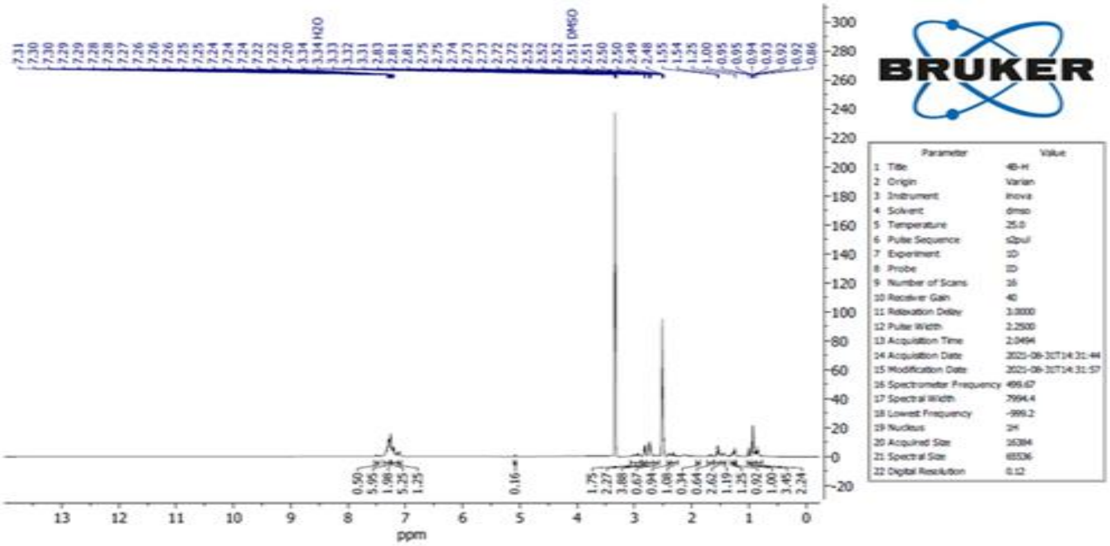


Parameter	Value
1 Title	4A-H
2 Origin	Varian
3 Instrument	inova
4 Solvent	dmso
5 Temperature	25.0
6 Pulse Sequence	s2pul
7 Experiment	1D
8 Probe	ID
9 Number of Scans	16
10 Receiver Gain	40
11 Relaxation Delay	3.0000
12 Pulse Width	2.2500
13 Acquisition Time	2.0494
14 Acquisition Date	2021-08-31T15:03:48
15 Modification Date	2021-08-31T15:04:09
16 Spectrometer Frequency	499.67
17 Spectral Width	7994.4
18 Lowest Frequency	-999.2
19 Nucleus	1H
20 Acquired Size	16384
21 Spectral Size	65536
22 Digital Resolution	0.12

Figure -3- (1H-NMR) spectrum of compound (1).

The (1H-NMR) spectrum of the compound (2): Dodecyl (2-phenylethyl) sulfane, indicate the chemical shifts, as interpreted in the figure (4)[15-17,38]:

(Ar-H) appeared as (multiple signals) at (7.11-7.51) (ppm), 2.61 (q, S-CH_2), 1.00-1.86 (m, CH_2), 0.85 (t, CH_3).



Parameter	Value
1 Title	4B-H
2 Origin	Varian
3 Instrument	inova
4 Solvent	dmso
5 Temperature	25.0
6 Pulse Sequence	s2pul
7 Experiment	1D
8 Probe	ID
9 Number of Scans	16
10 Receiver Gain	40
11 Relaxation Delay	3.0000
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17 Spectral Width	7994.4
18 Lowest Frequency	-999.2
19 Nucleus	1H
20 Acquired Size	16384
21 Spectral Size	65536
22 Digital Resolution	0.12

Figure -4- (1H-NMR) spectrum of compound (2).

7.3. MASS spectroscopy of compounds (1,2)

Mass spectrum to the compound (1)[18-24]: Octyl (2-phenylethyl) sulfane, show the (molecular ion peak M^+) at (m/e=250.4), as interpreted in the figure (5).

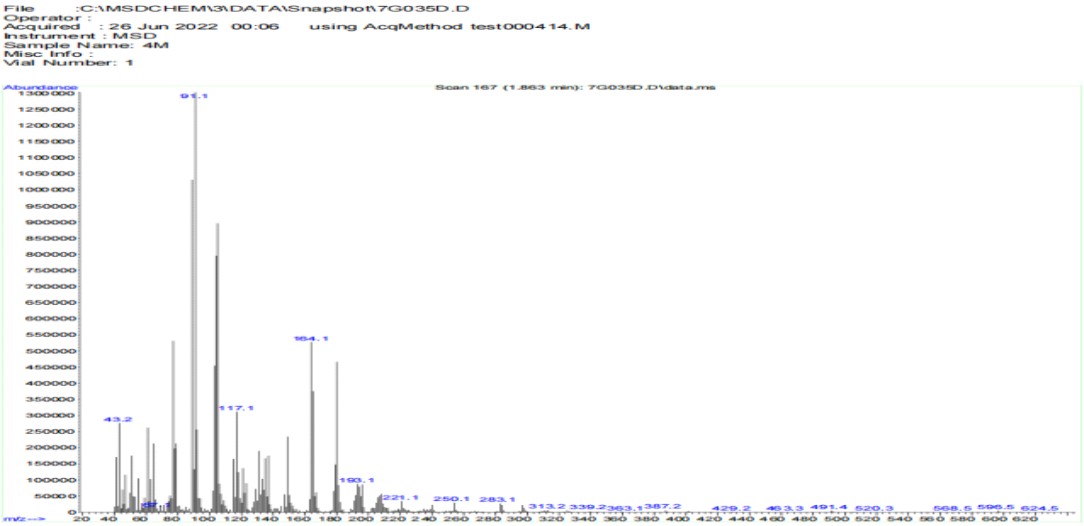


Figure -5- mass spectrum of compound (1).

Mass spectrum to the compound (2)[18-24]: Dodecyl (2-phenylethyl) sulfane, show the (molecular ion peak M$^+$) at (m/e=306.6), as interpreted in the figure (6).

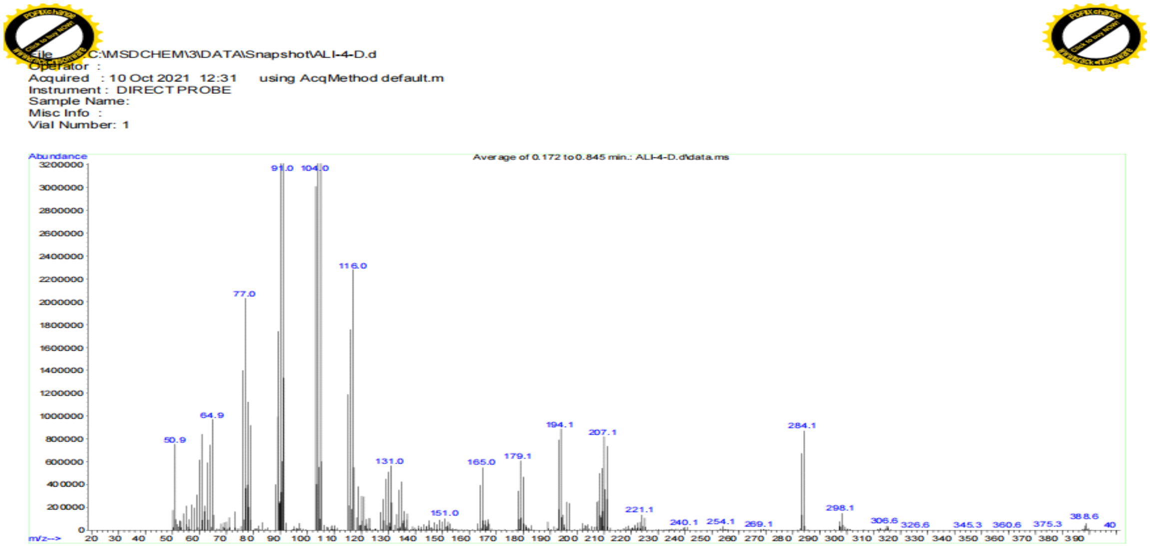


Figure -6- mass spectrum of compound (2).

7.4. FT-IR spectroscopy of compounds (3,4)

The spectrum of the compound (3): 2-(Octylthio) benzene-1,4-diol, was interpreted by using the infrared spectroscopy (FT-IR) technique in the figure(7)[10-15,40]:

IR(cm^{-1}) : 1460 (C=C, Ar), 3045 (CH, Ar), 2964 (CH,CH_3, Al), 3365 (OH, Ar), 624 (C-S, Al).

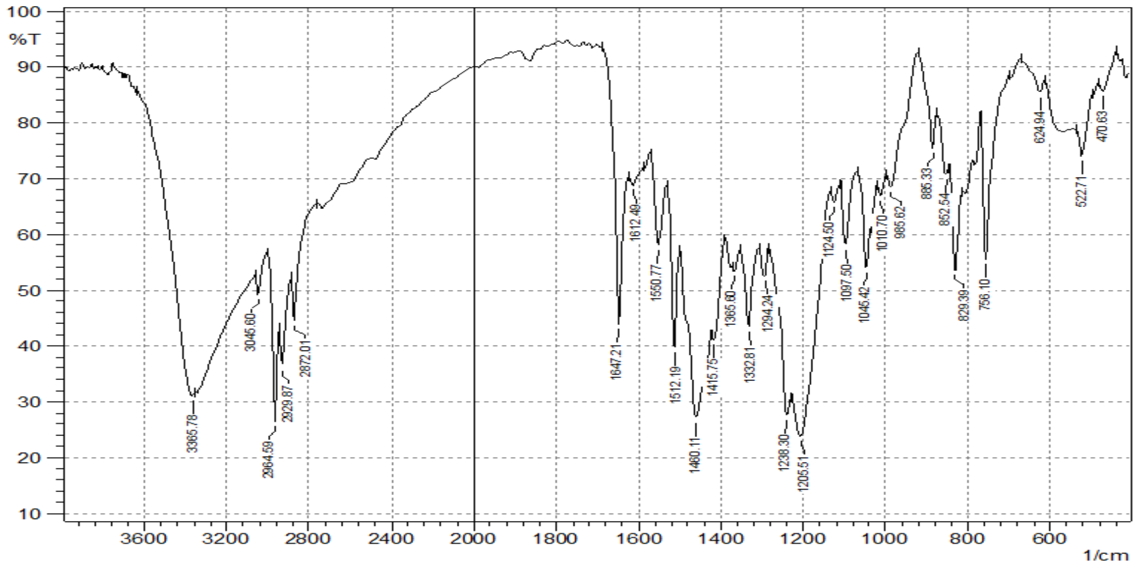


Figure -7- (FT-IR) spectrum of compound (3).

The spectrum of the compound (4): 2-(Dodecylthio) benzene-1,4-diol was interpreted by using the infrared spectroscopy (FT-IR) technique in the figure (8)[10-15,40]:

IR(cm^{-1}) : 1460 (C=C, Ar), 3043 (CH, Ar), 2962 (CH,CH_3, Al), 3342 (OH, Ar), 578 (C-S, Al).

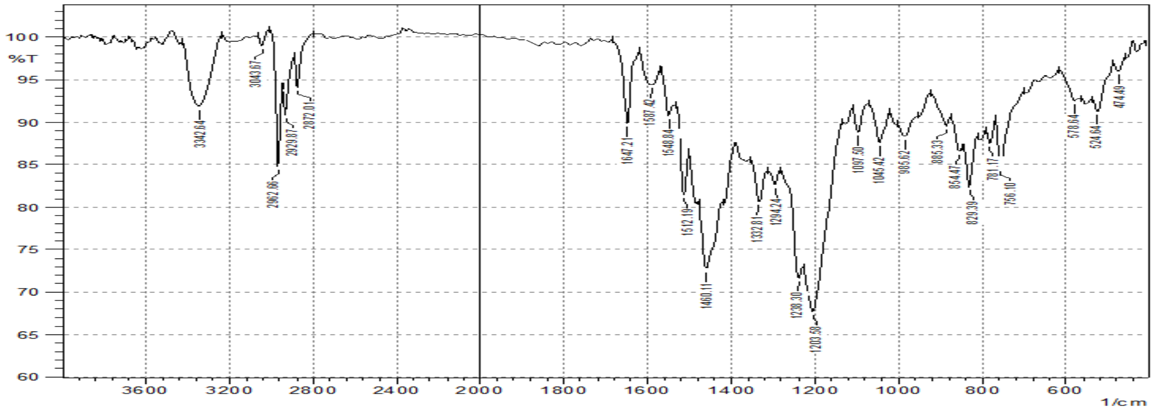
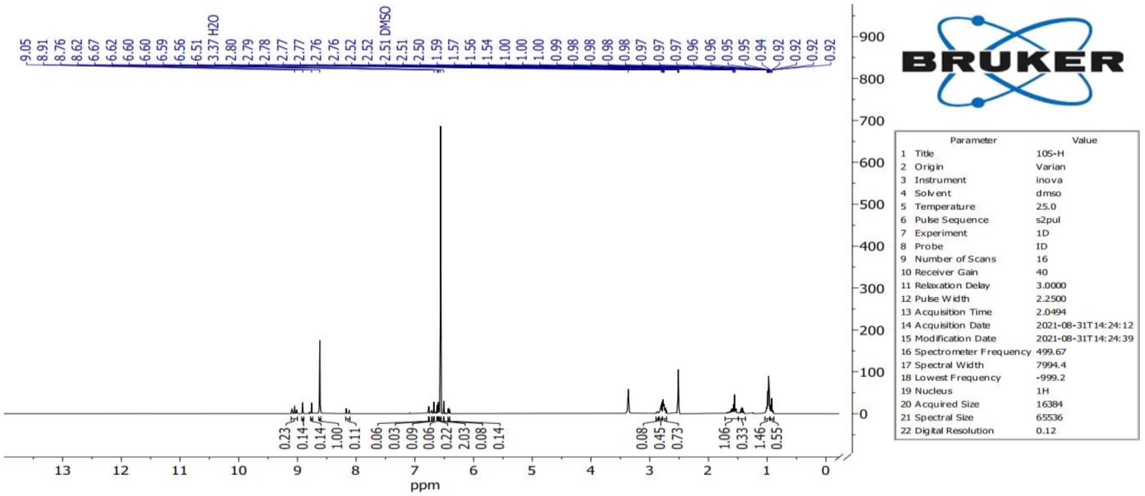


Figure -8- (FT-IR) spectrum of compound (4).

7.5. 1H-NMR spectroscopy of compounds (3,4)

The (1H-NMR) spectrum of the compound (3): 2-(Octylthio) benzene-1,4-diol, indicate the chemical shifts, as interpreted in the figure (9)[15-17]:

(Ar-H) appeared as (multiple signals) at (6.52-6.73) (ppm), (Ar, OH) (1) appeared as singlet at (8.62), (Ar, OH) (2) appeared as singlet at (8.91), (S-CH_2) appeared as (quartet signals) at (2.77), (CH_2) appeared as (multiple signals) at (1.31-1.63), (CH_3) appeared as triplet signals at (0.88).



Parameter	Value
1 Title	10S-H
2 Origin	Varian
3 Instrument	inova
4 Solvent	dmso
5 Temperature	25.0
6 Pulse Sequence	s2pul
7 Experiment	1D
8 Probe	ID
9 Number of Scans	16
10 Receiver Gain	40
11 Relaxation Delay	3.0000
12 Pulse Width	2.2500
13 Acquisition Time	2.0494
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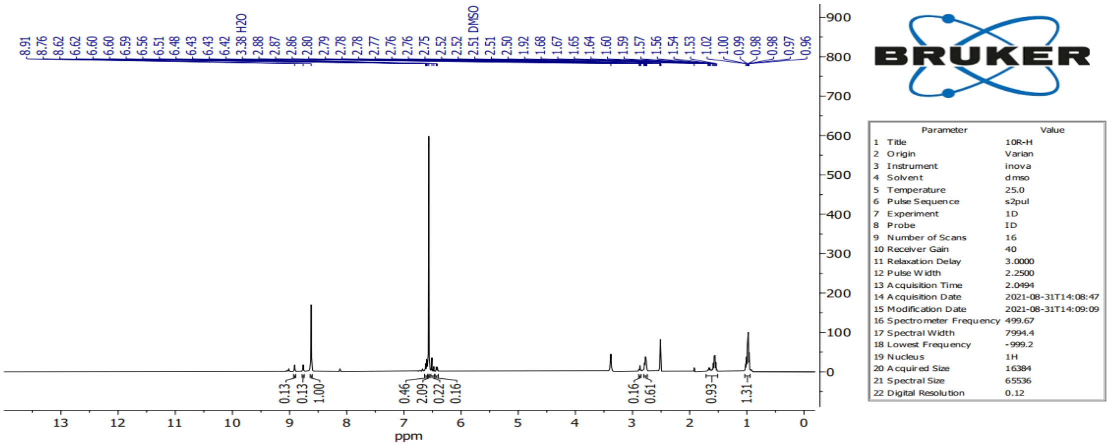
17 Spectral Width	7994.4
18 Lowest Frequency	-999.2
19 Nucleus	1H
20 Acquired Size	16384
21 Spectral Size	65536
22 Digital Resolution	0.12

Figure -9- (1H-NMR) spectrum of compound (3).

The (1H-NMR) spectrum of the compound (4): 2-(Dodecylthio) benzene-1,4-diol, indicate the chemical shifts, as interpreted in the figure (10)[15-17]:

(Ar-H) appeared as (multiple signals) at (6.51-6.71) (ppm),), (Ar, OH) (1) appeared as singlet at (8.62), (Ar, OH) (2) appeared as singlet at (8.91), (S-CH_2) appeared as (quartet signals) at (2.77), (CH_2) appeared as (multiple signals) at (1.13-1.94), (CH_3) appeared as triplet signals at (0.94).



Parameter	Value
1 Title	10R-H
2 Origin	Varian
3 Instrument	inova
4 Solvent	dmso
5 Temperature	25.0
6 Pulse Sequence	s2pul
7 Experiment	1D
8 Probe	ID
9 Number of Scans	16
10 Receiver Gain	40
11 Relaxation Delay	3.0000
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17 Spectral Width	7994.4
18 Lowest Frequency	-999.2
19 Nucleus	1H
20 Acquired Size	16384
21 Spectral Size	65536
22 Digital Resolution	0.12

Figure -10- (1H-NMR) spectrum of compound (4).

7.6. MASS spectroscopy of compounds (3,4)

Mass spectrum to the compound (3)[18-24]: 2-(Octylthio) benzene-1,4-diol, show the (molecular ion peak M^+) at (m/e=254.4), as interpreted in the figure (11).

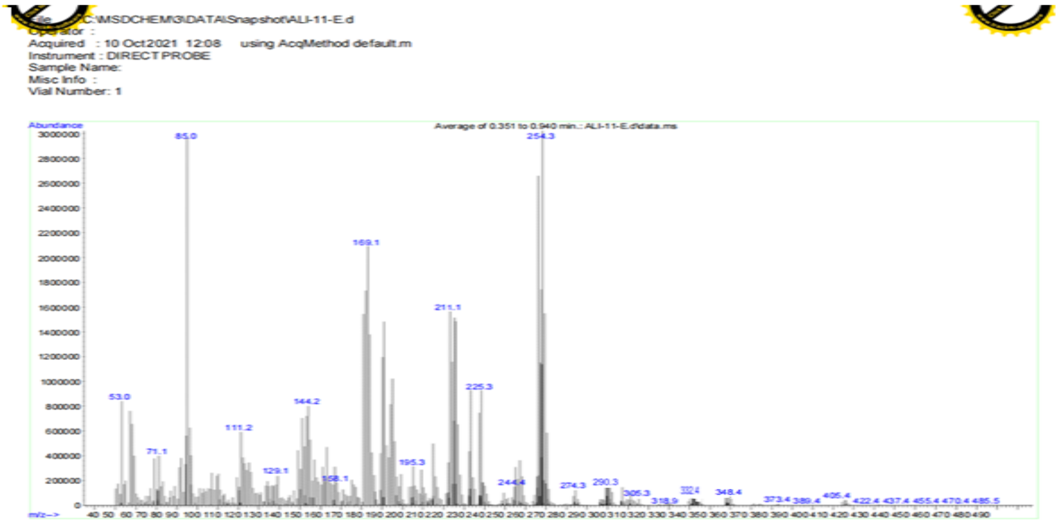

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Figure -11- mass spectrum of compound (3).

Mass spectrum to the compound (4)[18-24]: 2-(Dodecylthio) benzene-1,4-diol, show the (molecular ion peak M^+) at (m/e=310.5), as interpreted in the figure (12).

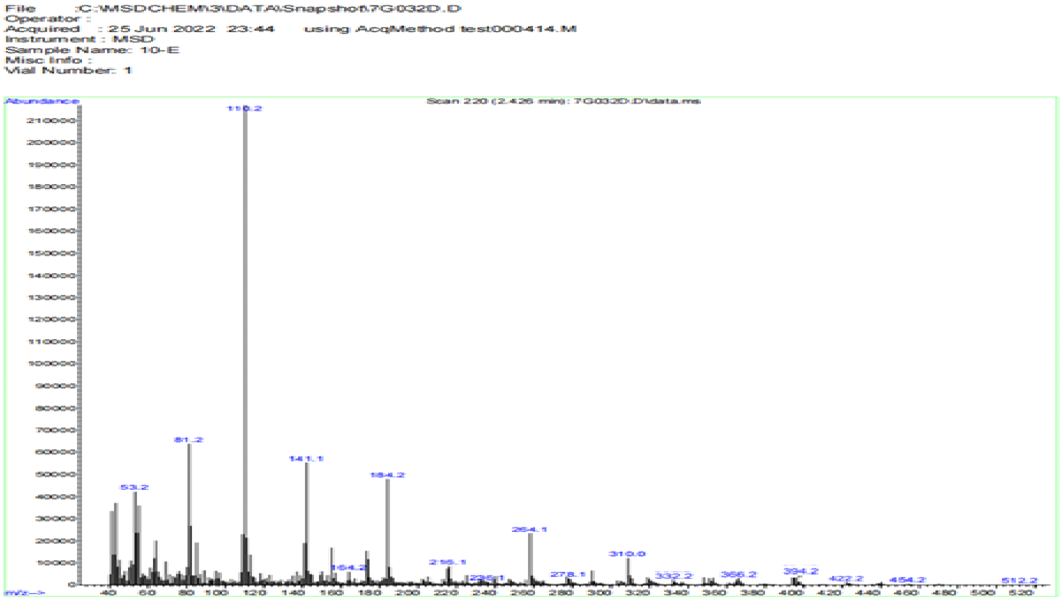

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Figure -12- mass spectrum of compound (4).

8. Discussion

To explain the efficacy of the produced antioxidant compounds, they were compared to Butylated Hydroxy Toluene (BHT), one of the most important commercial antioxidants. As some advantages emerged for the prepared antioxidant compounds over the commercial antioxidants such as, the synthesized antioxidants having a higher molecular weight and being more compatible than BHT because of the long alky chains on the aromatic ring of the prepared antioxidants, BHT may pass through the polymer membrane especially when mixed with water and oil (emulsion) because the low molecular weight of BHT compared to the synthesized antioxidants which have a higher molecular weight. The antioxidants that were synthesized proved to be more effective to prevent the degradation process (oxidation) compared to commercial antioxidant (BHT) [33].

When the lubricating oils are exposed to high temperatures, they will get oxidized. This leads to appearance of a band in the region (1716) cm^{-1} because the degradation process to the lubricating oils. This band can be identified by the measurements of (FT-IR). But when adding the antioxidants that have been synthesized, the band in this area (1716) cm^{-1} will start to decrease until it disappears, which indicates the efficiency of the prepared antioxidants that are added to the lubricant oils and their good resistance to the oxidation processes. It was also noted that the synthesized antioxidant compounds were more efficient in the degradation process (oxidation) than the commercial antioxidant compound (BHT). This can be seen through the intensity of the band in this region by the measurements of (FT-IR), as shown in the figures (13, 14, and 15) below. It can be also compared the activity of the prepared antioxidant compounds with the commercial antioxidant (Butylated Hydroxy Toluene, BHT), it was observed that the efficiency of the prepared antioxidants compounds (A1- A4) are more efficient than the (BHT) in terms of their activity as antioxidants towards the process of the oxidation to the lubricating oils, as shown in the figure (16) [25-28]:

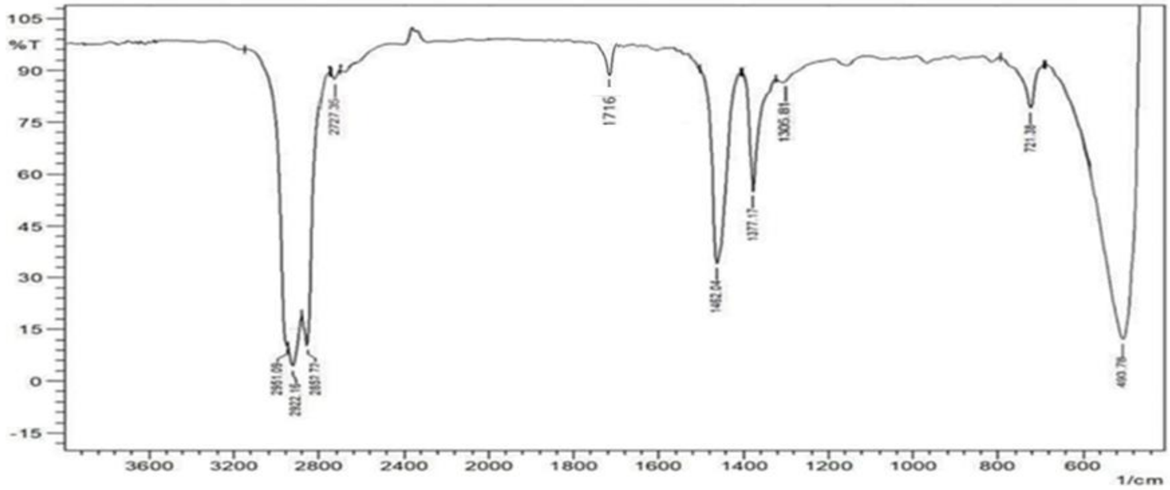

Figure (13) show the degradation process to the lubricant oils.

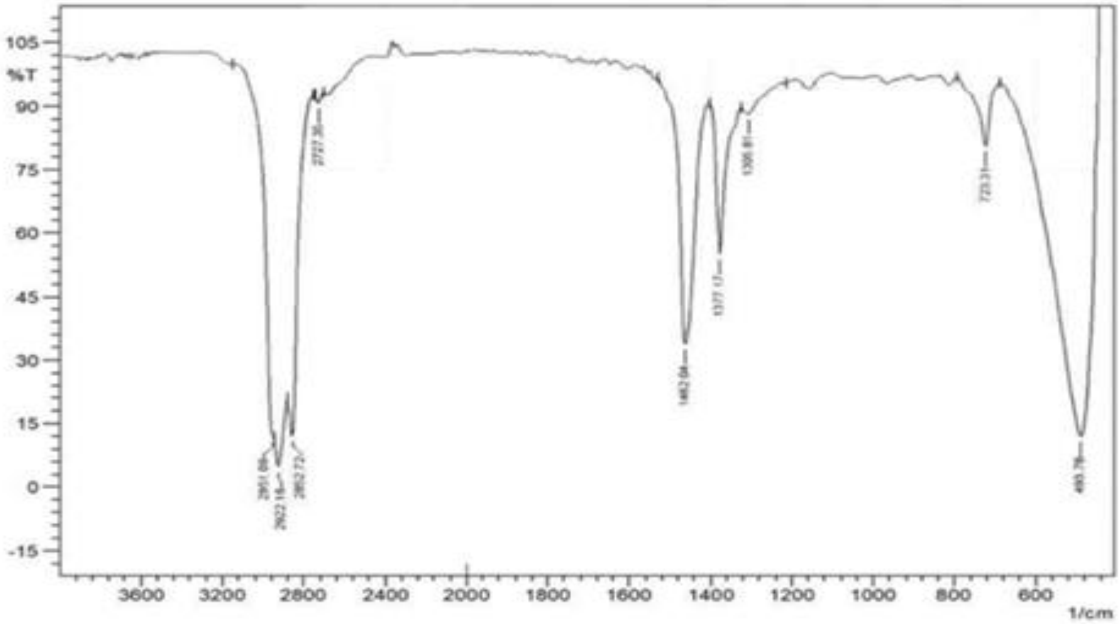

Figure (14) show the efficiency of the antioxidant compounds (A1, A2).

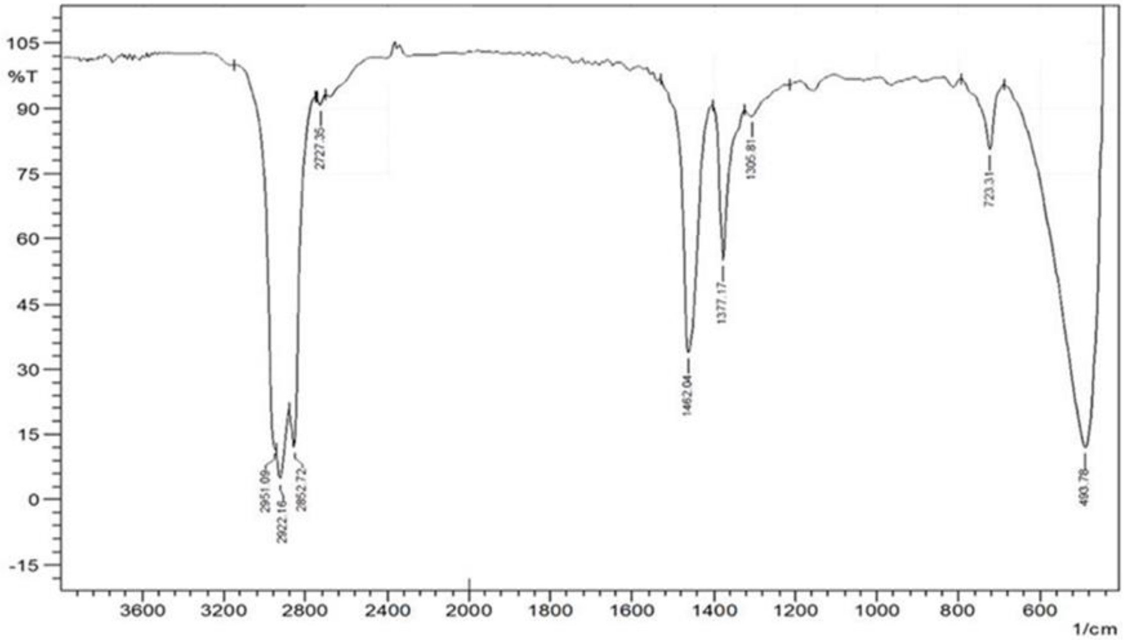

Figure (15) show the efficiency of the antioxidant compounds (A3, A4).

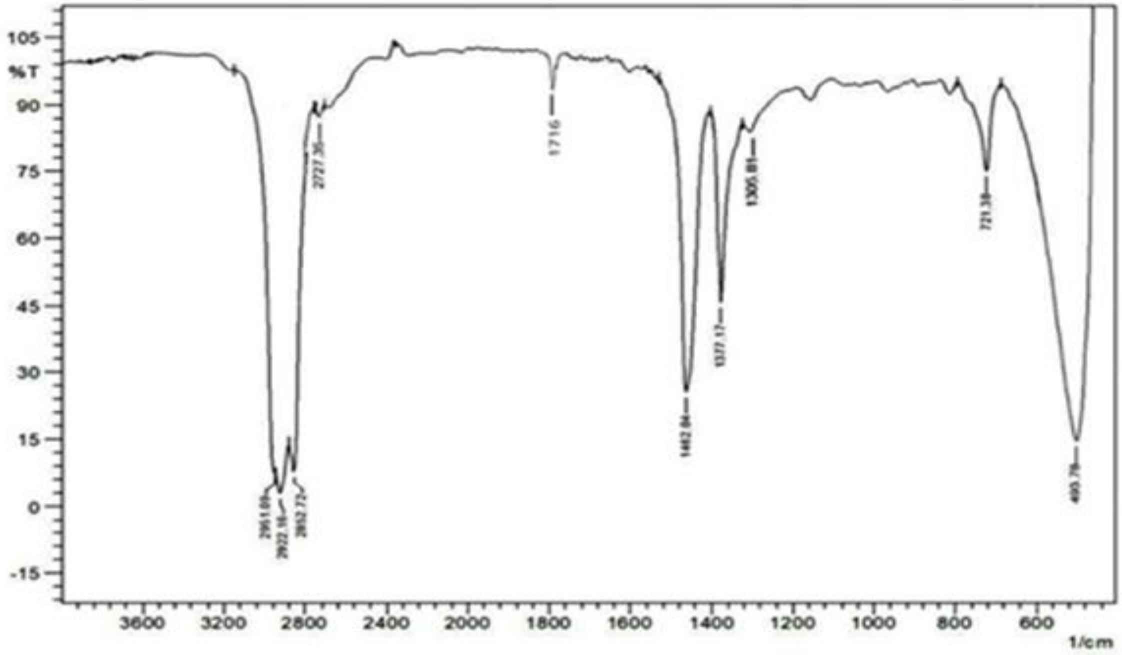

Figure (16) show the efficiency of the antioxidant compound (BHT).

The efficiency of the prepared antioxidants can also be observed by their oxidation potentials through the cyclic voltammetry technique. The antioxidants that have a high oxidation potential (E_p^a) like the compounds (A1-A4) are considered as an excellent to prevent the degradation process (oxidation) that is occur to the lubricant oils[29,36].

From observing the values of the oxidation potentials of the antioxidant compounds in the table (4), we deduced that the compounds (A1-A4) have more activity than the compound (Butylated Hydroxyl Toluene, BHT) as antioxidant because they possess high oxidation potentials (E_p^a) than the compound (BHT), as shown in the figures (17,18,19,20), but the (BHT) possess low oxidation potential as shown in the figure (21), table (4).The range of the potential that is used in this method is from (-1.5) to (+1.5). The cell of the cyclic voltammetry is formed from three electrodes: the working electrode, the reference electrode, and the counter electrode. We must clean the electrode before utilize by using a cloth polishing pad in water-alumina slurry. The solvent that is used in the cyclic voltammetry must be, liquid, dissolving the (analyte) and settling towards the (oxidation) and (reduction) processes, not harmful to the reaction with the (analyte) or the (supporting electrolyte) and must be very pure. The supporting electrolyte which is used in this technique must be very soluble in the solvent, inactive (chemically) and (electrochemically) and purifiable. The (x-axis) in the cyclic voltammogram acts as the utilized (potential, E) and the (y-axis) acts as the current used (i) [29,30]. The voltage of the working electrode is controlled relative to the reference electrode (saturated calomel electrode) or the electrode (Ag/AgCl) in cyclic voltammetry by applying an electric potential to the respective electrode immersed in a solution containing electrically active groups and measuring the resulting current. An electrical excitation signal can be defined as the controlled voltage applied across these two electrodes [34].

This signal is a linear potentiometric scan in an equilateral triangle, or more precisely, it is the computation of the single electrode potential during the electron transition process at the electrode's surface, which corresponds to the flow of a net current through the cell [35,39].

According to the scanning in cyclic voltammetry technique, the working electrode potential (micro-static) electrode with time, starting from the potential at which electrode reactions do not occur and continuing to the potentials at which the sample under study's oxidation and reduction process occurs [31].

The working electrode potentials are wiped in the anodic direction with the maximum values resulting from the oxidation of the reactive substances. The voltage is then wiped in the cathodic direction with the maximum values resulting from the reduction of the intermediate compounds formed during scanning in the anodic direction [29,30].

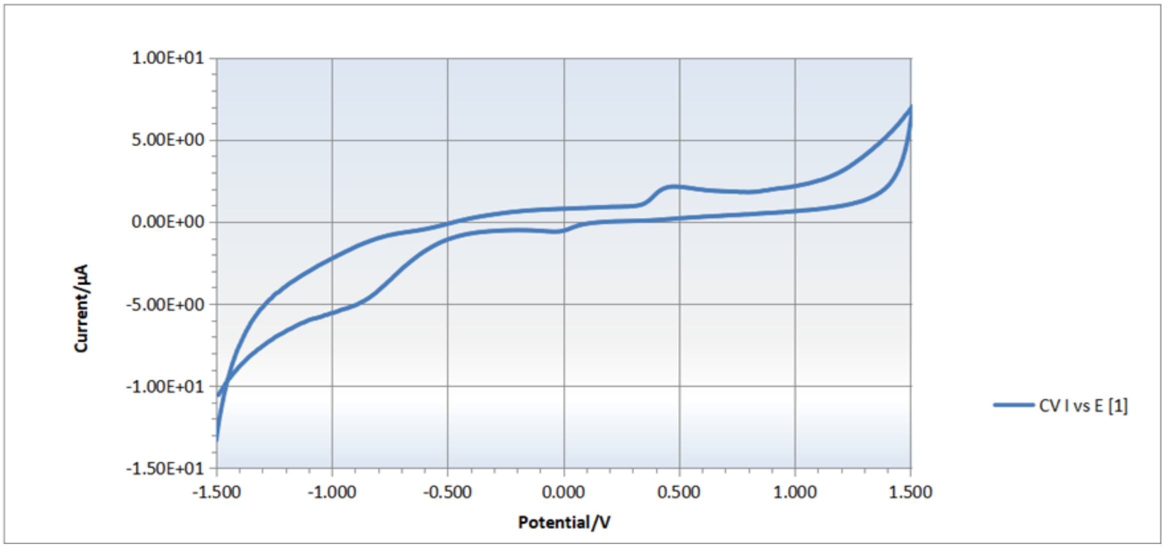

Figure (17) show the activity of the antioxidant compound (A1).

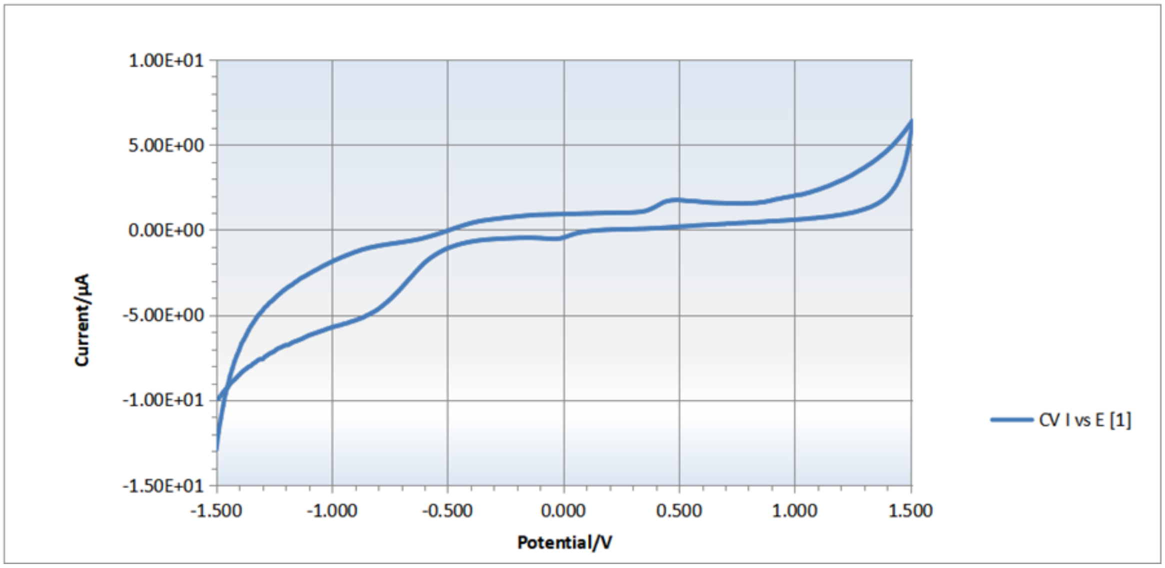

Figure (18) show the activity of the antioxidant compound (A2).

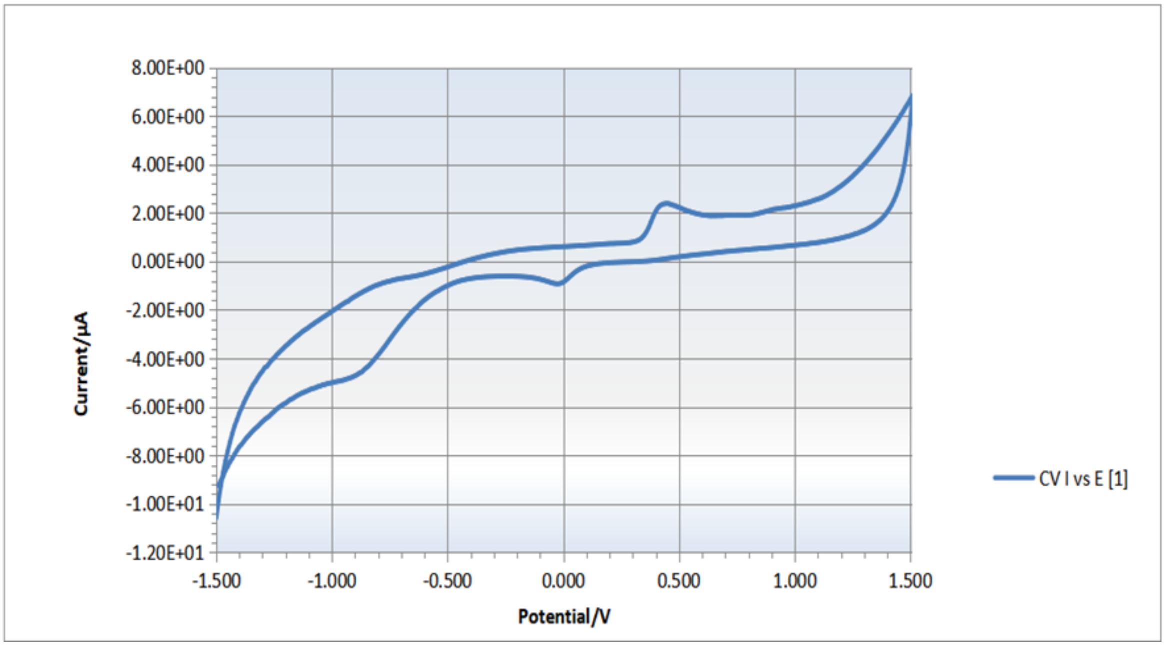

Figure (19) show the activity of the antioxidant compound (A3).

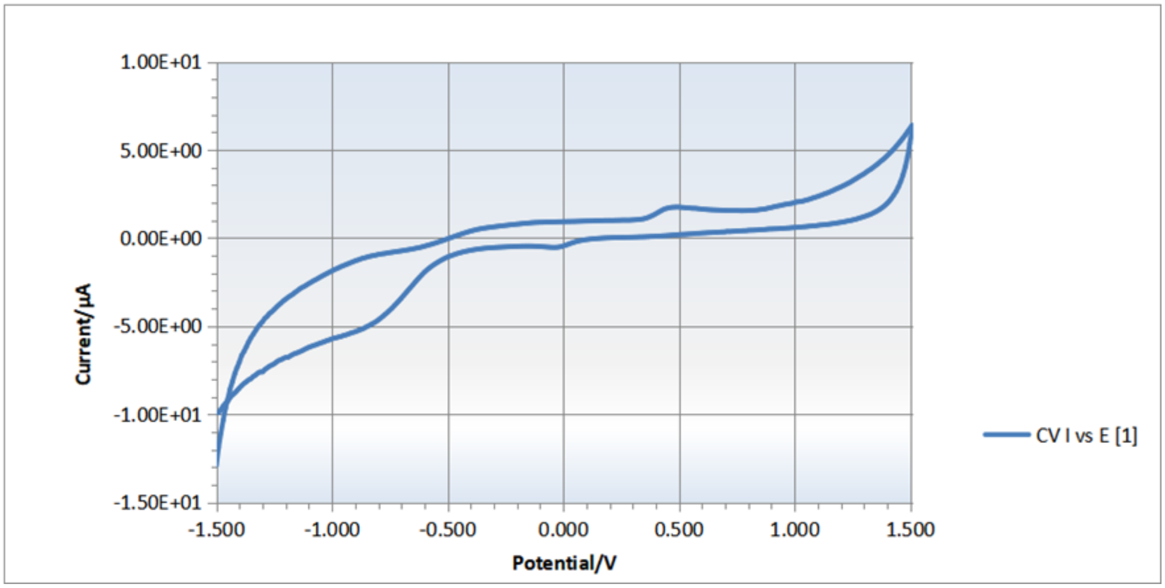

Figure (20) show the activity of the antioxidant compound (A4).

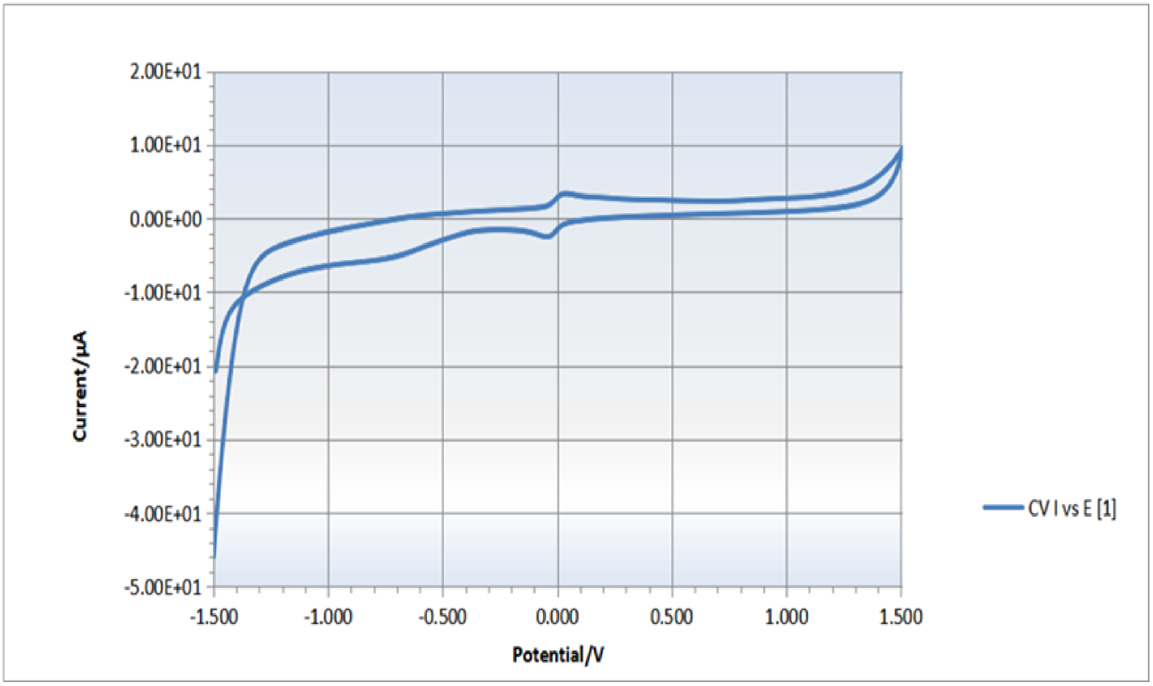

Figure (21) show the activity of the antioxidant compound (BHT).

Table (4) show the values of (E_p^a),(E_p^c $_{(1,2)}$), and ($\Delta E_{p(1,2)}$).

Comp.NO	E_p^a (v)	E_p^c $_{(1,2)}$ (v)	$\Delta E_{p\ (1,2)}$(v)=$\lvert E_P^C$ - $E_p^a \rvert$
A1	0.45	-0.03 -0.9	0.48 1.35
A2	0.45	-0.03 -0.9	0.48 1.35
A3	0.41	-0.05 -0.85	0.46 1.26
A4	0.45	-0.05 -0.85	0.5 1.3
BHT	0.05	-0.05 -0.75	0.1 0.8

9. Conclusion

The analysis of the (FT-IR) and the (cyclic voltammetry) appeared to be good methods for evaluating the activity of the prepared antioxidant compounds when added to the lubricating oils. The effectiveness of the prepared antioxidant compounds has been compared with each other and also with the effectiveness of the commercial antioxidant compound (BHT). It was observed that the compounds (A1- A4) were more efficient as antioxidants towards the degradation process (oxidation) of the lubricants oil than the (BHT).

As well as the values of the oxidation potentials (E_p^a) of the prepared antioxidant compounds by using the cyclic voltammetry technique, it was observed that the prepared antioxidant compounds possess high oxidation potential (E_p^a) than the (BHT), this lead to be more efficient to prevent the oxidation process in the lubricating oils . The quantitative (electrochemical) analysis considered as a good technique for detecting the reactions of the (electron transfer) and the substantial mechanistic of the (inorganic chemistry), the method of the cyclic voltammetry was characterized by simply, easy to use and rapidity. The (FT-IR) spectroscopy also indicated that the synthesized antioxidant compounds are more efficient to prevent the oxidation process in the lubricants, through observing the disappearance of the band in the region (1716) cm^{-1} compared to (BHT).

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