

Improvement of the thermo Oxidation properties for low-density polyethylene using curcumin analogues

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DOI /2022 10.54633/2333-021-042-037

Abstract:

In this study, three analogs of curcumin were prepared and used as antioxidant for LDPE. The thermal oxidation inhibition was investigated using carbonyl index, TGA, and DSC as well as the antioxidant inhibition was evaluated using DPPH. The results showed that the curcumin analogs derived from 4-hydroxyl benzaldehyde, compound had high antioxidant efficiency for LDPE.

Keywords: low-density polyethylene (LDPE), curcumin analogs, thermal antioxidant, Carbonyl index, TGA, DSC.

الكلمات المفتاحية: - البولي ايثيلين واطى الكثافة ، نظائر الكركمين ، مضادات الاكسدة الحرارية ، معامل الكربونيل ، التحليل الوزني الحراري ، المسح التفاضلي المسعري.

1. Introduction

The last few decades have seen a large usage for the low-density polyethylene, particularly in the agriculture and packaging operation and multiple

applications, because of the increasing consumption and production of plastic in the world. In 2019 alone, about 360 million tons (Mt) of plastics were produced globally¹. So, plastics need to more of studies to protect the environment from the negative effects. The question is how long the properties of plastic materials can be maintained in outdoor environments². The thermal degradation of polymers refers to the case where polymers at elevated temperatures start to undergo from a chemical change without the simultaneous involvement of another compound³. Thermal degradation of polymers is of paramount importance in developing a rational technology for polymer processing. By using the polymers at a higher temperature, it leads to understanding thermal decomposition

mechanisms for the synthesis of safe polymeric materials. Degradation of polymers includes all the changes in the chemical structure and physical properties of the polymers due to external chemical or physical stresses that caused by chemical reactions⁴. Polyethylene may be exposed to several stresses such as oxidation, therefore polymers can react with molecular oxygen in a process called spontaneous oxidation. This process occurs due to many of reasons, such as heat, ultraviolet light, mechanical stress, catalyst residue, or reaction with impurities, to form alkyl radicals⁵. Free radicals can be reacting with others to cause a polymer decomposition, then they resulting in embrittlement, instability of melt flow, loss of tensile properties and discoloration⁶. Oxidation can be slowed by chain-breaking antioxidants, to reduce the rate of diffusion, or protective antioxidants, which prevent the initial formation of free radicals⁶. LDPE is available as plastic bags and packaging films⁷, it is widely used in packaging due to its easy feedstock availability and processability, versatility, low cost and

recyclability⁸. Thermo oxidation degradation of polyethylene (PE) has an essential economic impact. It impacts polymers that exposure to heat and light, which is related to the lifetime of the manufactured product⁹. Several studies on the PE thermo oxidative have shown that hydroperoxides are formed and then decompose to form carboxyl and alcohol compounds, so the polymer chains are breakdown and decrease in molecular weight¹⁰⁻¹¹. Antioxidants are molecules that can react and inhibit the initiation and propagation of oxidizing chain reaction produced by reactive free radical before polymers degradable¹². Antioxidants can inhibit free radical reactivity through sundry mechanisms including the donation of hydrogen, radical scavenging, and singlet oxygen quenching¹³. Many techniques were used to evaluate of antioxidant efficiency such as IR spectrophotometer and thermal analysis (TGA, and DSC)¹⁴ and assessed by DPPH-scavenging¹⁵.

2. Experimental

2.1 Materials

All starting material were purchased from Sigma-Aldrich and used without any further purification. The NMR spectra were measured in deuterated dimethyl sulfoxide DMSO-*d*₆, Food grade LDPE 463 pellets were supplied from the state Company for Petrochemical of Basrah, Iraq (density 0.922 g/cm³ and melt flow index 0.32 g/10min).

2.2 Preparation of curcumin analogues

A basic condition was used for synthesizing curcumin analogues¹⁴. A solution of para hydroxybenzaldehyde (0.02 mol) in ethanol (2.5 ml) was placed in one-neck round bottom flask under constant stirring at room temperature. Then appropriate ketone (cyclohexanone, cyclopentanone or acetone) (0.01 mol) was added to the solution and stirred vigorously, followed by dropwise addition of 20% NaOH (50 ml) with keeping the temperature below 40 °C. The stirring of the red mixture was kept for 24 hours. Then, distilled water (15 ml) was added to the mixture and then neutralized at acidic conditions (pH = 5.5) by adding (6N HCl)

where a yellow-colored precipitate was observed. The product then was filtered and dried. Scheme 2 shows the preparation reaction, and table (1) shows some of the physical properties of the prepared compounds.

2.3 Purification of LDPE

Commercial pellets of LDPE were purified using xylene solvent and refluxed under nitrogen atmosphere for 1 hour to remove any antioxidant traces. Then precipitate using cold methanol then filtered and dried at 70 °C for 1 hour¹⁵.

2.4. Fabrication of the films

Different wt % of curcumin analogue was mixed with LDPE using extrusion and carried out through a three-heating zone, the rotation speed was fixed at 64 rpm throughout the extrusion process. During the process, the temperature of the three heating zones was set at 145 °C, for 8 min¹⁶. The extruded filaments were subsequently pelletized, and the formed pellets were used for the fabrication of the films. After that, pure LDPE and (antioxidant/LDPE) were exposed to thermal oxidation at 200 °C for 1 hour¹⁷ by using air oven after that, pure LDPE and (antioxidant/LDPE) composite films were produced by compression molding at 140 °C for 5 mins to get films with on

average thickness of 1 mm by using press Hydraulic.

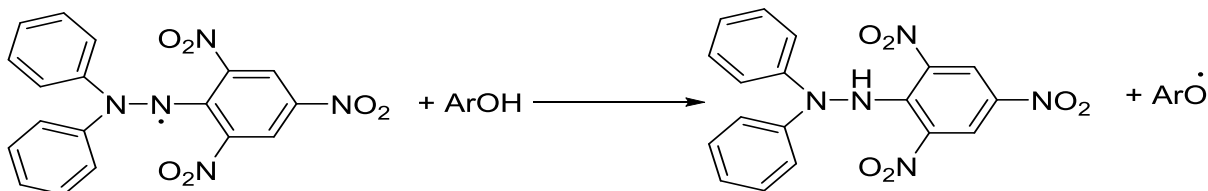
2.5 Carbonyl index study

The IR spectra of the films were recorded before and after the thermal oxidation using FT-IR Shimadzu 8400s. After that the carbonyl index¹⁸ (CI) was obtained using equation (1)¹⁹ We notice a significant change in the infrared spectrum with the increase in the absorption of the carbonyl group²⁰.

$$CI = \frac{Abs_{1726\text{ cm}^{-1}}}{Abs_{1472\text{ cm}^{-1}}} \times 100\% \quad \dots (1)$$

Where $Abs_{1726\text{ cm}^{-1}}$ and $Abs_{1472\text{ cm}^{-1}}$ are the absorption intensity of C=O and CH₂ peaks, respectively. The efficiency of antioxidant has been calculated according to equation²¹ (2):

$$\%E = \frac{CI \text{ Before addition} - CI \text{ After addition}}{CI \text{ Before addition}} \times 100 \quad \dots (2)$$



2.6 Radical scavenging activity

The 1,1-diphenyl-2-picrylhydrazyl (DPPH) is one of the few stable and commercially organic nitrogen radicals²². It is widely testified method for determination of antioxidant activity, the (DPPH•) radical (Figure 4) is characterized as a stable free radical because π electrons of the aromatic systems that present in the molecule which can compensate for the lack of an electron. DPPH• has a deep violet color led to absorption in solution at around 517 nm²³. When a solution of DPPH• is in contact with a substance that can donate a hydrogen atom or with another radical (R•), the reduced form DPPH-H or DPPH-R is produced with the consequent loss of color and therefore the decrease or loss of absorbance (Figure 8). Consequently, the reduction of DPPH• provides an index to estimate the ability of the test compound to trap radicals.

Figure 4 DPPH• reduction by an antioxidant.

ArOH is an antioxidant that acts by donating hydrogen atoms, to obtain radicals with stable molecular structures that will stop the chain reaction²⁴. The assay was carried out according to the modified method of Blois (1958)²⁵. 1 mL of 0.1 mM solution of DPPH (Sigma Aldrich) in methanol was mixed with 2 mL of the aqueous extracts at different concentrations (50, 100, 150, 200, 250, 300, 350 and 400 µg/mL). The mixture was incubated at room temperature for (30-60) min in the dark. The control was prepared by mixing 1 mL of DPPH solution with 2 ml Methanol. The

absorbance was measured against a blank at 517 nm by using spectrophotometer. Lower absorbance of the reaction mixture indicates higher DPPH free radical scavenging activity. BHT was used as a standard, samples were prepared and measured in triplicates. The percentage of scavenging activity of each extract on DPPH radical was calculated as %inhibition of DPPH and a curve was drawn between the inhibition efficiency and concentration. The IC₅₀ was calculated by plotting the percentage of inhibition against different concentrations of the antioxidant sample. The IC₅₀ value indicates the concentration required to eliminate²⁶ 50% of the free radicals, low IC₅₀ values indicates a high radical uptake activity^{27, 28}.

$$\text{DPPH inhibition (\%)} = \frac{A_{\text{Control}} - A_{\text{Sample}}}{A_{\text{Control}}} \times 100$$

Where A_{control} is the absorption of control and A_{sample} is the absorption of the tested sample solution

2.7- Thermal Analysis

The TGA and DSC of Pure LDPE and LDPE mixed with Curcumin analogues

were investigated using (TGA technique SDT Q600 V20.9 Build 20 (and DSC SDT Q600 V20.9 Build 20) under oxygen condition at heating rate 10C/min

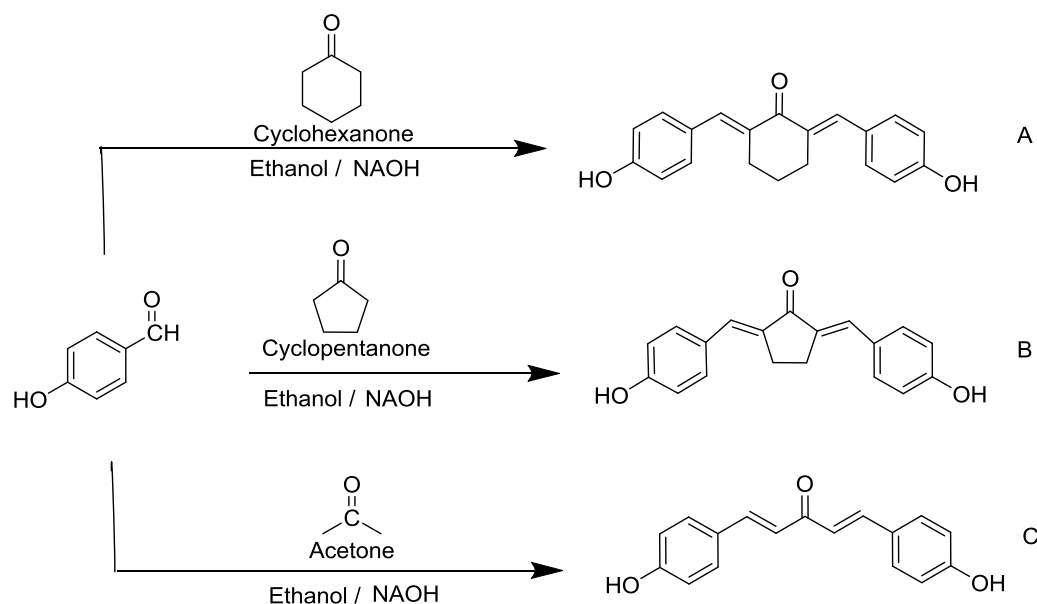
3. Results and discussion

3.1 Synthesis of Curcumin analogues

Curcumin analogs (A-C) were synthesized according to literature¹⁵ protocols through condensation of appropriate ketone (cyclohexanone, cyclopentanone, or acetone) with a variety of aromatic aldehydes in basic conditions,

in good to excellent yields. The structures of these analogs are

shown in scheme 1. The prepared compounds were characterized using FTIR figures(1-3) and H1NMR figures (4-6).



Scheme 1: Synthesis of curcumin analogues

**2,6-bis(4-Hydroxybenzylidene)
cyclohexanone A.**

The product was recrystallized from ethanol, yellow crystals, Yield 60% m.p. > 300 °C. IR (cm⁻¹): 3321, 3259, 2943, 1647, 1593, 1508, 1435, 1215, 1161, 968, 840. ¹H NMR (500 MHz, DMSO-*d*₆) δ 9.93 (s, 2H, -OH), 7.54 (s, 2H, -CH=), 7.40 (d, *J* = 8.65 Hz, 4H, ArH), 6.84 (d, *J* = 8.65 Hz, 4H, ArH), 2.85 (t, *J* = 5.2 Hz,

4H, CH₂), 1.70 (p, *J* = 6.4 Hz, 2H, -C-CH₂-C-)

**2,5-bis(4-Hydroxybenzylidene)
cyclopentanone B.**

The product was recrystallized from ethanol, yellow crystals, Yield 85 %, m.p. > 300 °C. IR (cm⁻¹): 3321, 3171, 2960, 1666, 1597, 1512, 1431, 1257, 1165, 987, 837. ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.05 (s, 2H, -OH), 7.54 (d, *J* = 8.6 Hz,

4H, ArH), 7.33 (s, 2H, -CH=), 6.87 (d, J = 8.6 Hz, 4H, ArH), 3.01 (s, 2H).

1,5-bis(4-Hydroxyphenyl)-1,4-pentadiene-3-one C.

The product was recrystallized from ethanol, orange crystals, yield 86 %, m.p. 232–234 °C. IR (cm⁻¹): 3506, 3120, 2827, 1643, 1589, 1516, 1442, 1172, 1114, 983, 829. ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.04 (s, 2H, -OH), 7.66 (d, J = 15.95 Hz, 2H, -CH=), 7.62 (d, J = 8.7 Hz, 4H, ArH), 7.10 (d, J = 15.95 Hz, 2H), 6.83 (d, J = 8.65 Hz, 4H, ArH).

3.2- The antioxidant activity of curcumin analogs

In table 1 showed that the prepare curcumin analogs have a good antioxidant activity. The inhibition of the thermo oxidation for low density poly ethylene increased as the wt% increased from 0.5 to 6% . The best results were recorded for curcumin analogs derived from 4-Hydroxy benzaldehyde (A,B and C).

Among these three compounds, compound B has the highest inhibition of thermo oxidation which reached zero, and the lowest inhibition result was recorded for compound A. For a more convenient comparison, the antioxidant efficiency (%E) of the curcumin analog was calculated according to equation (2) and the results are summarized in Table 2. Again, compounds derived from 4-Hydroxy benzaldehyde namely (A, B, and C) as can be seen from the results in table 2, The high antioxidant efficiency of compounds (A, B, and C) as shown in Table 2, might be due to the extra two Hydroxy donor groups added to the benzene rings at meta position which make the free radical, generated through the oxidation process, more stable. The antioxidant efficiency of curcumin analogs toward LDPE oxidation appeared to increase with raise active functional groups.

Table 1: Effect of concentration of curcumin analogs on %CI of LDPE

Compound (w/w)%	CI					
	0	0.50%	1%	2%	4%	6%
LDPE+A	100	29.4	27.3	4.3	0	0
LDPE+B	100	20	5.2	3.3	0	0
LDPE+C	100	14.2	13.3	6.5	3.1	0

Table 2: Antioxidant efficiency(%E) of curcumin analogs toward LDPE oxidation

Compound (w/w)%	%E					
	0	0.5%	1%	2%	4%	6%
LDPE+A	0	70.6	72.7	95.7	100	100
LDPE+B	0	80	94.8	96.7	100	100
LDPE+C	0	85.8	86.7	93.5	96.9	100

3.3 DPPH Free Radical Scavenging Activity A

In the DPPH assay, the antioxidant activity analysis is based on the inhibition of the dpph radical by the antioxidant. The optimal initial concentration of dpph was evaluated first to determine the assay sensitivity the violet color intensity increased rapidly with the dpph concentration from 1mM, but after adding antioxidants and comparing them with a

standard compound such as BHT²⁹, we noticed the disappearance of the violet color, which indicates that the activity of antioxidants through absorption. A table was made that includes different concentrations, as shown in table (3). Compound B was more active than the rest in scavenging free radicals by giving a hydrogen atom through the value of IC₅₀. It can be seen from the results in this table that compound B needs only (46,96

$\mu\text{g/ml}$) to scavenge half of the free radicals, while the compound A needs (57.78 $\mu\text{g/ml}$) and compound C needs

(63.24 $\mu\text{g/ml}$), the lower of the IC_{50} value more active and stable the antioxidants.

Table 3: Assay radical scavenging activity towards DPPH for antioxidant.

[conc] $\mu\text{g/ml}$	<i>Inhibition Free Radical Scavenging Activity%</i>			
	BHT	A	B	C
0	0	0	0	0
50	70	51.5	55.4	49.9
100	80.5	57.2	60.6	55.4
150	86.6	63.6	67.2	61.2
200	87.3	71.7	78.4	68.1
300	90.1	79.9	80.5	77.6
350	94	82.3	85.3	80
400	94.5	86.9	90.1	83.4
IC_{50} $\mu\text{g/ml}$	20.13	57.78	46.96	63.24

3-4 Thermal Analysis

TA can be used in laboratories as quality control method for the development of new polymeric materials³⁰, that explain the connection between temperature and specific physical properties of materials³¹.

(TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are being observed as a result of increase in temperature³². The thermo oxidation temperatures of LDPE mixed with the prepared compounds Figures (7-11) were summarized in Table

3-4-1 Thermo gravimetric analysis

TGA

Table 4 : The values of LDPE Decomposition temperatures in the presence and absence of compounds prepared under an atmosphere of oxygen.

Compounds	1 st stage Decomposition temp. $^{\circ}\text{C}$	2 nd stage Decomposition temp. $^{\circ}\text{C}$
LDPE	383	430

LDPE/A	416	452
LDPE/B	413	482
LDPE/C	410	435

It showed that the decomposition temperatures of two stages increased for LDPE/ Curcumin analogs (A,B and C) compared with decomposition temperatures of pure LDPE.

3-4-2 Differential scanning calorimetric (DSC)

The DSC of LDPE was investigated under oxygen atmosphere figure (12-15) and show that an endothermic peak³³ appears at 126 °C with a heat absorption capacity of 70.95 J/g attributed to melt of LDPE. The method involves introducing

heat energy into a sample cell and a reference cell simultaneously while identically increasing the temperature of both cells over time^{34,35}. After that, a number of exothermal peaks appears that correspond to the decomposition of LDPE which corresponding with TGA, and the total energy of decomposition 4229 J / g to ldpe but after antioxidants (compounds prepare) mixer with ldpe , the Energy decomposition reached 8445 J/g. On the other hand, the decomposition energy of (LDPE/curcumin analogs) higher than LDPE alone because have hydroxyl group³⁶ were summarized in Table 5.

Table: 5 Dissociation energy of LDPE and LDPE/curcumin analogs.

Compounds	LDPE	LDPEL/A	LDPE/B	LDPE/C
Dissociation Energy J/g	4229	4644	8445	5578

4. Conclusion

This study has shown that the antioxidant efficiency of (A, B and C) compounds for LDPE falls between very

good to excellent. Compounds derived from para-hydroxybenzaldehyde have the highest efficiency which might be due to the extra donor hydroxyl group attached in

meta position which stabilizes the free radicals produced during the oxidation process. Compound (B) had the highest antioxidant activity, while compound C had the lowest.

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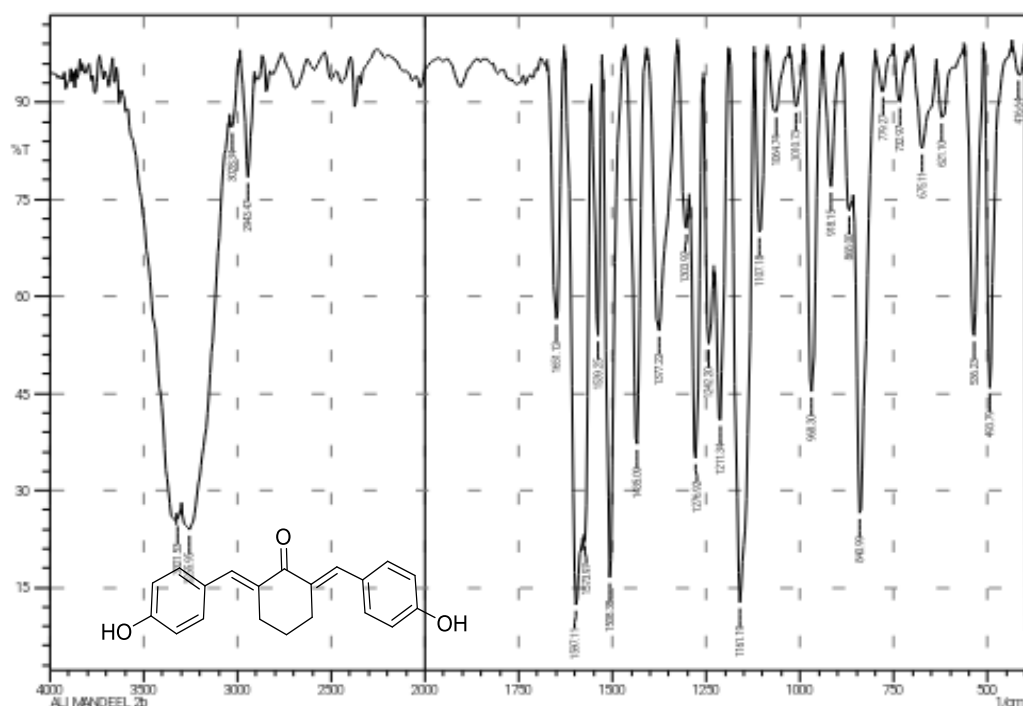


Figure (1) IR SPECTRA for compound A

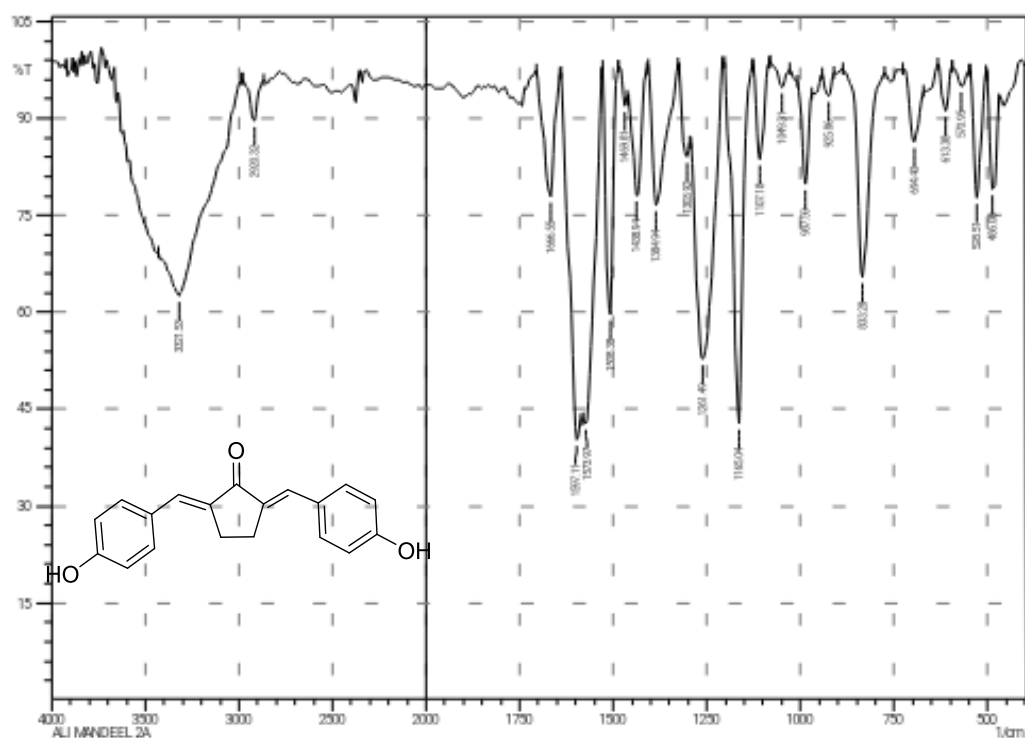


Figure (2) IR SPECTRA for compound B

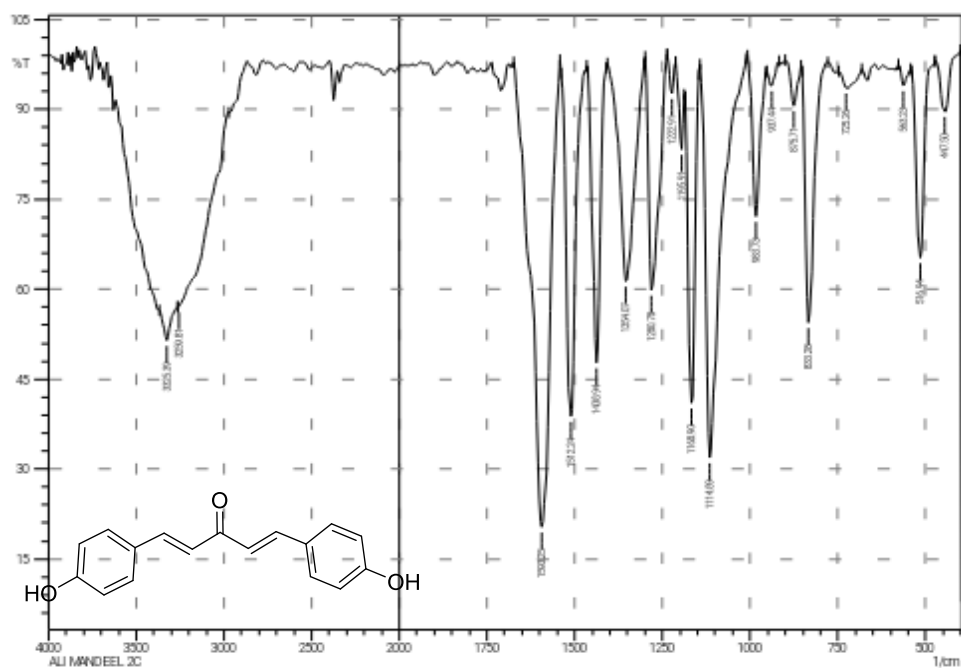
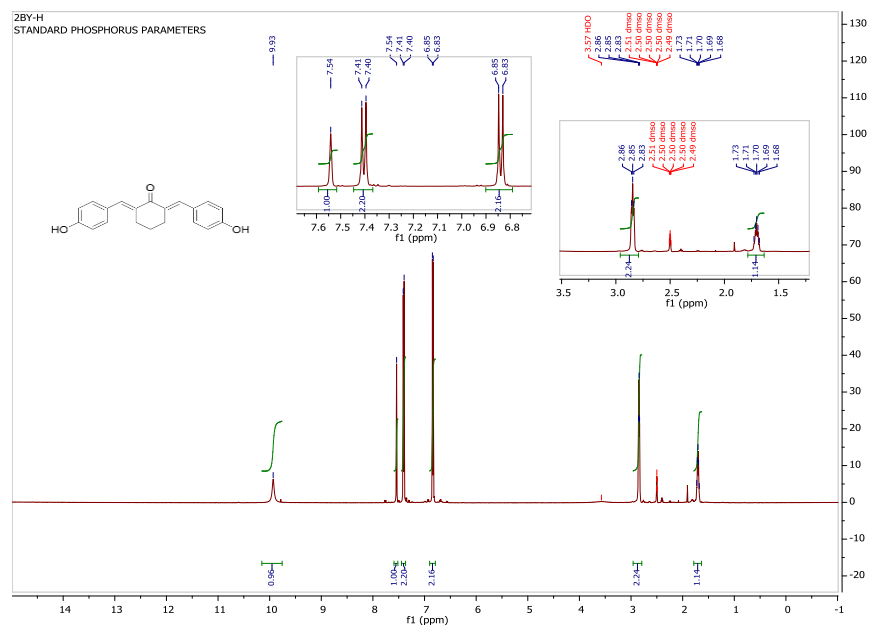
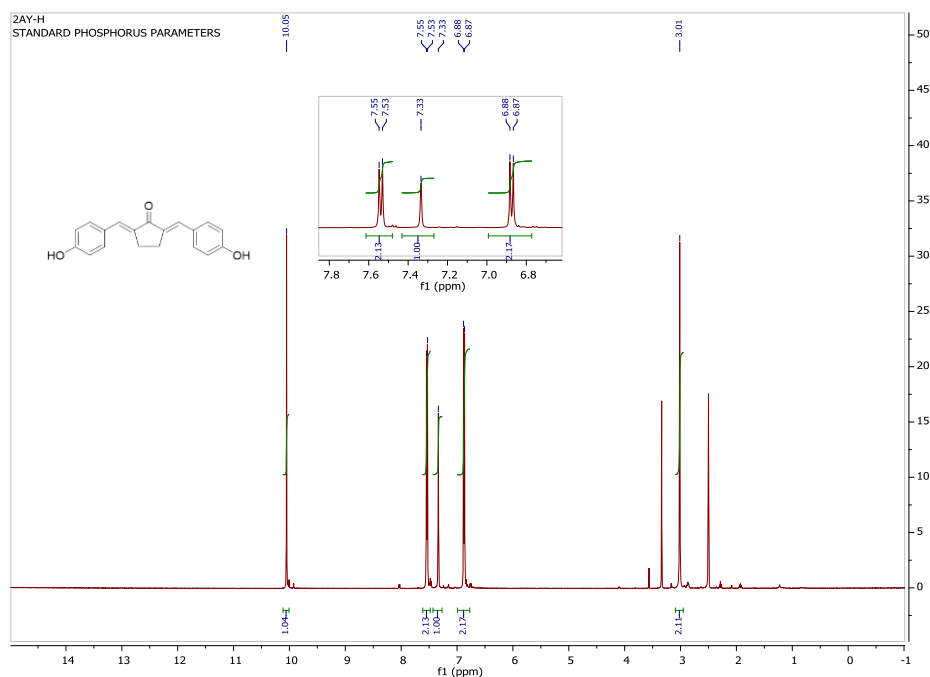


Figure (3) IR spectra for compound C

Figure (4) ^1H NMR for compound AFigure (5) ^1H NMR FOR Compound B

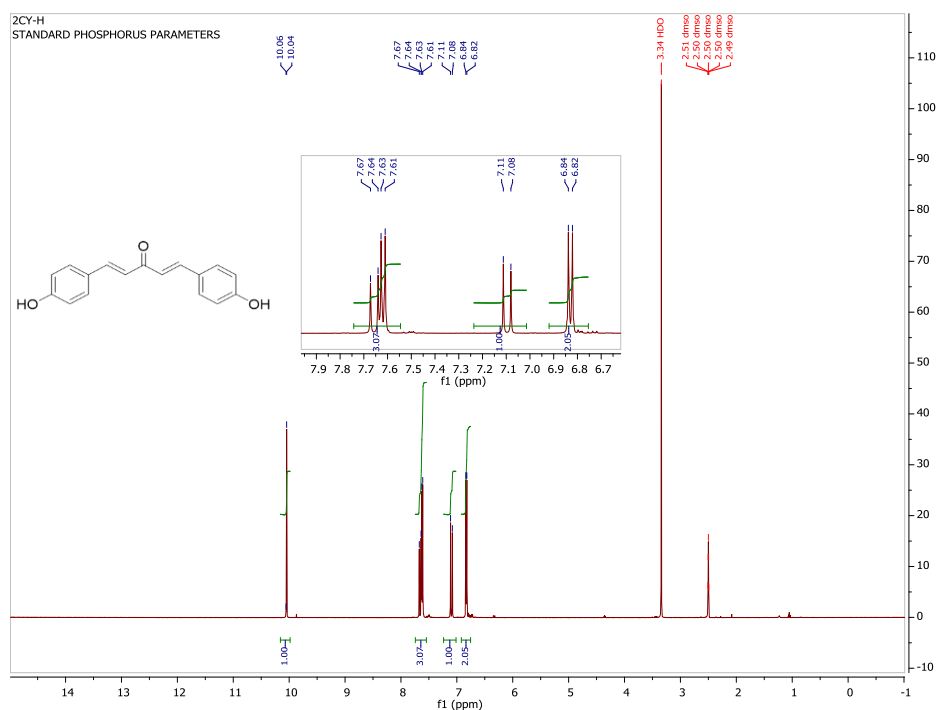
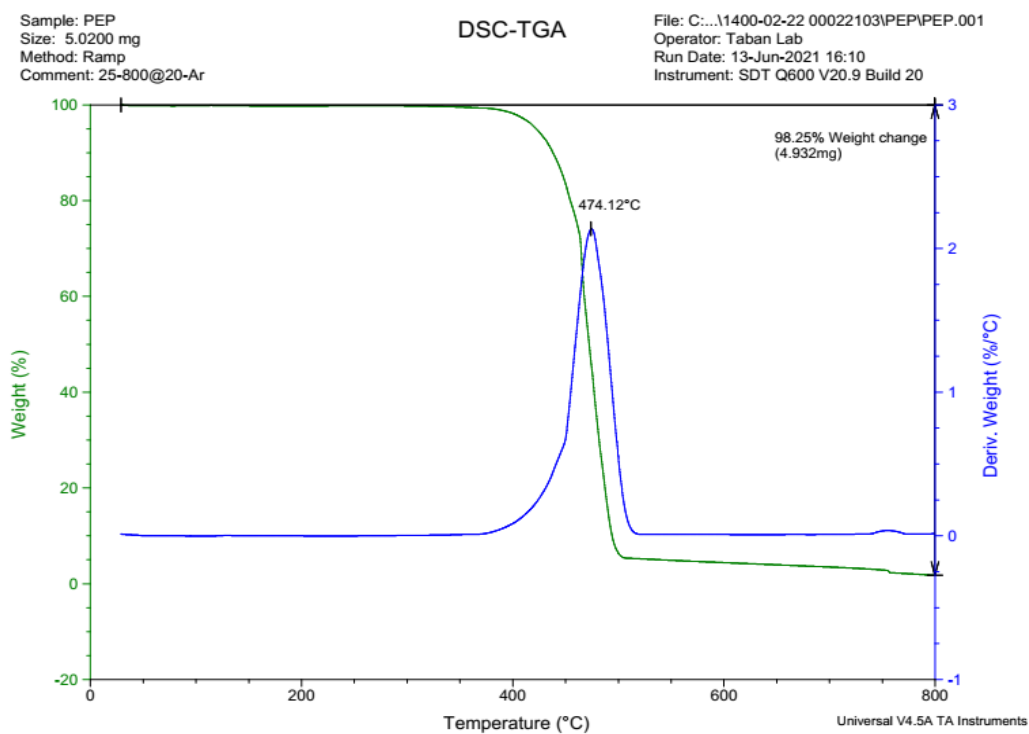
Figure (6) ^1H NMR FOR COMPOUND C

Figure (7) curve TGA to LDPE Under argon atmosphere

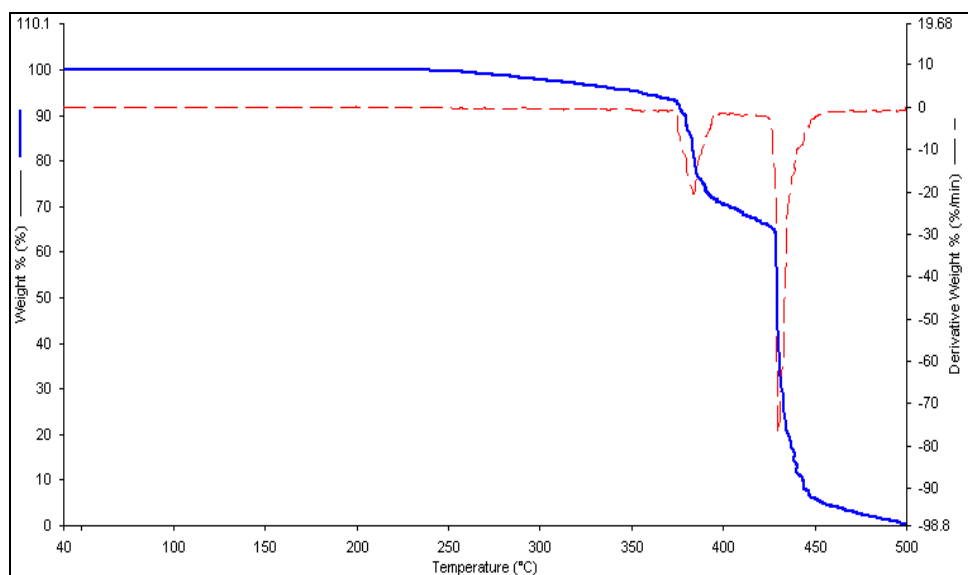


Figure (8) curve TGA to LDPE Under oxygen atmosphere

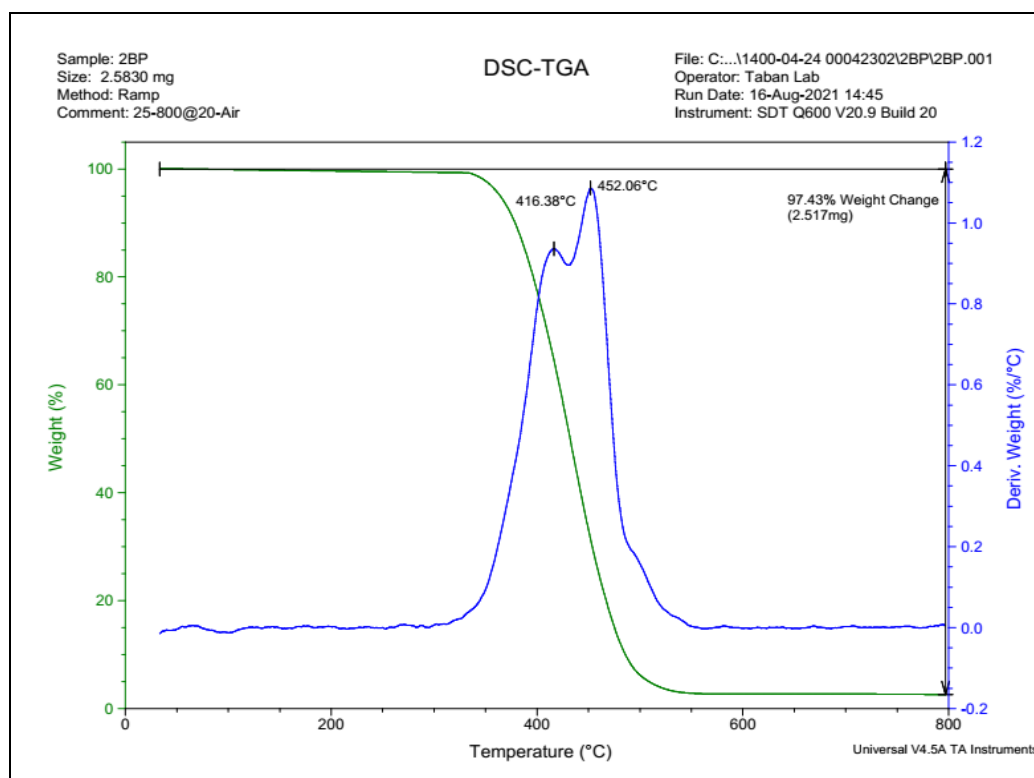


Figure (9) curve TGA of compound (LDPE/ A 6%)under oxygen

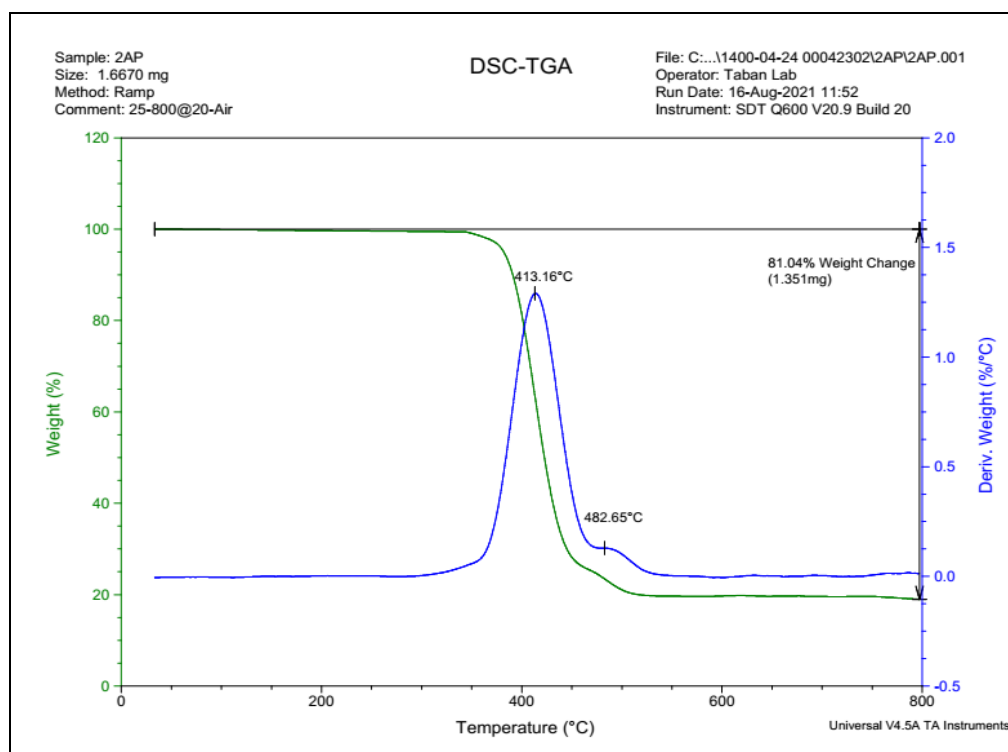


Figure (10) curve TGA to compound (LDPE/ B 6%)under oxygen

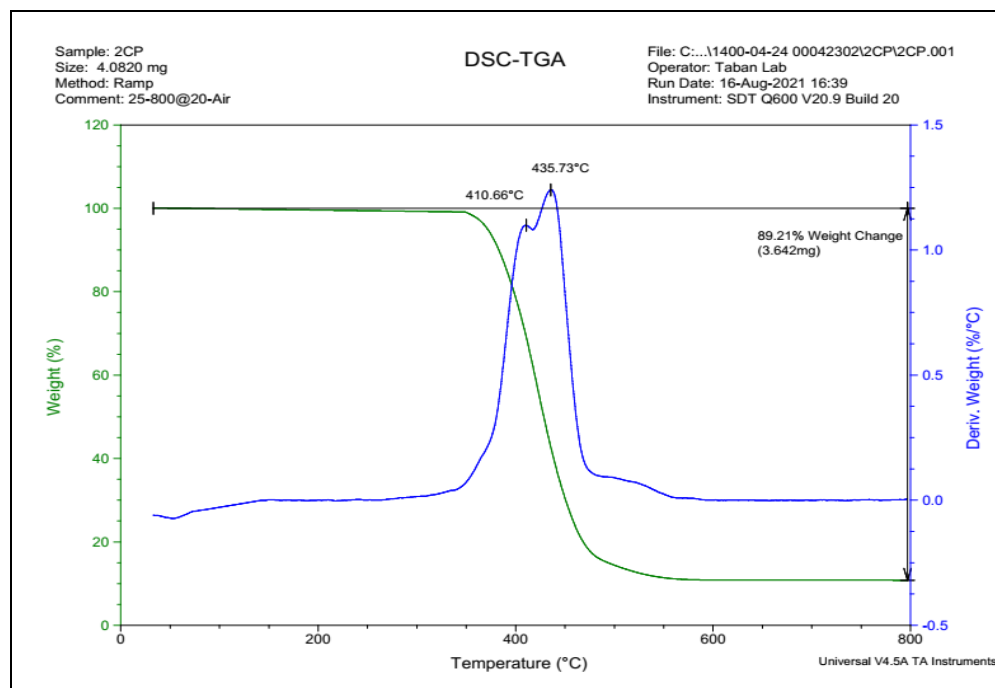


Figure (11) curve TGA to compound (LDPE/C 6%) under oxygen

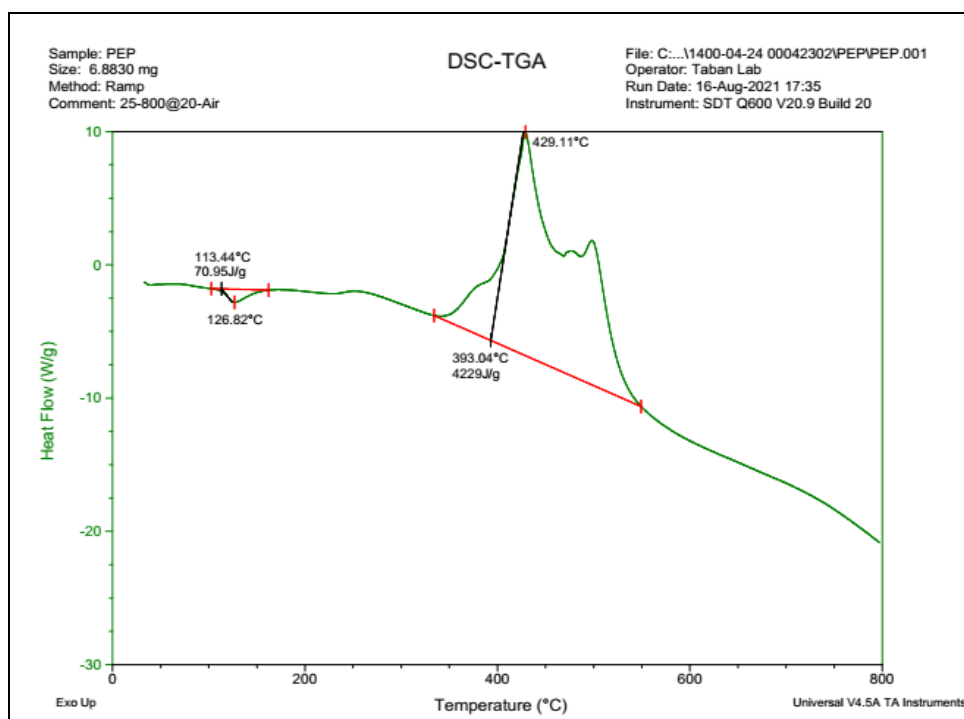


Figure (12) curve DSC to LDPE under oxygen

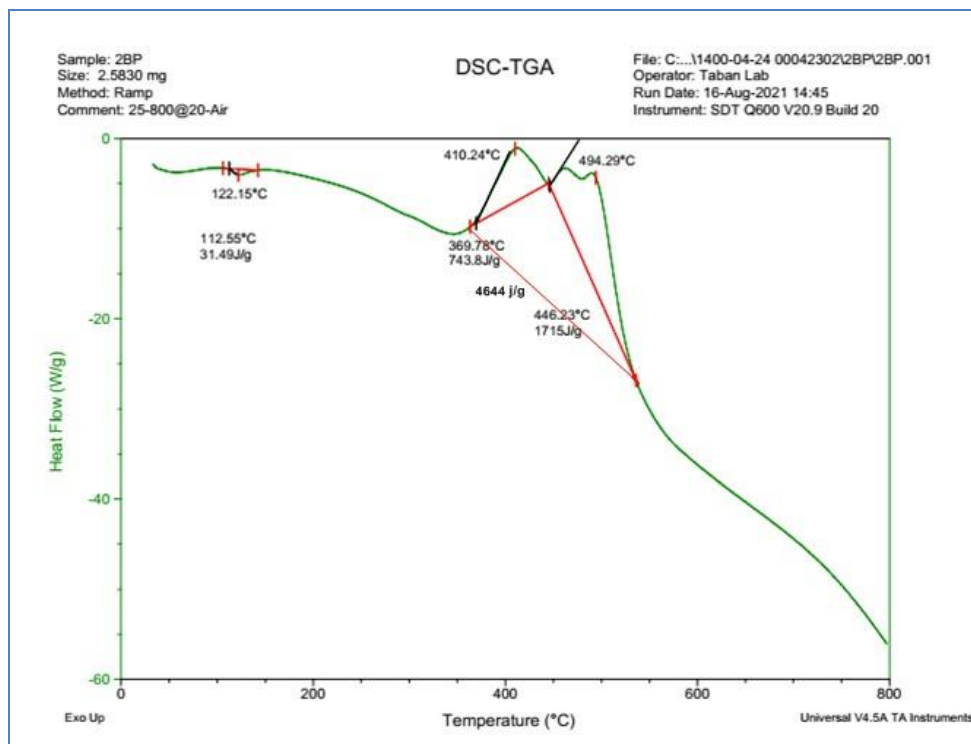


Figure (13) curve DSC to compound (LDPE/ A 6%)under oxygen

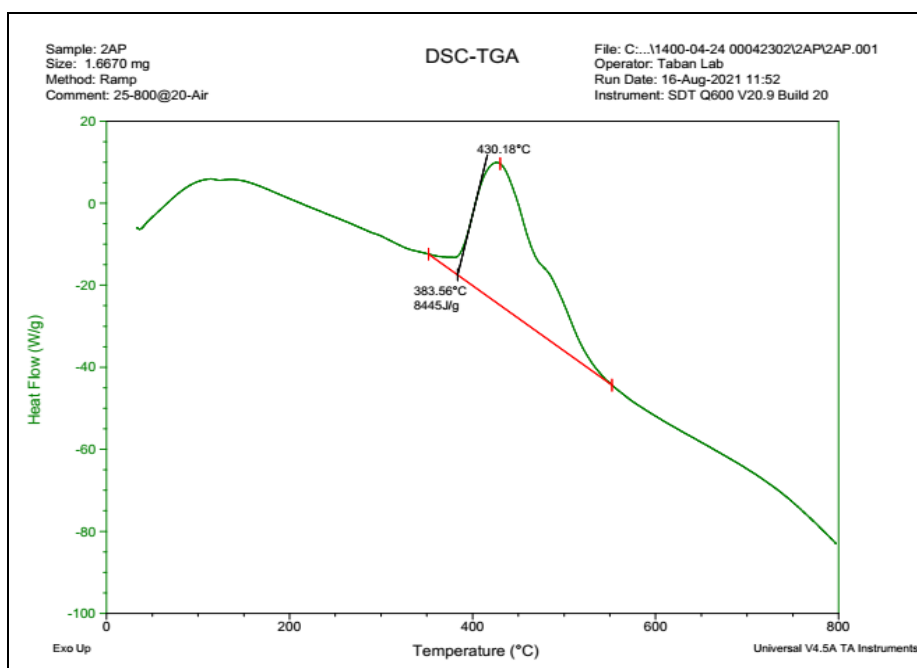


Figure 14 curve DSC to compound (LDPE/ B 6%)under oxygen

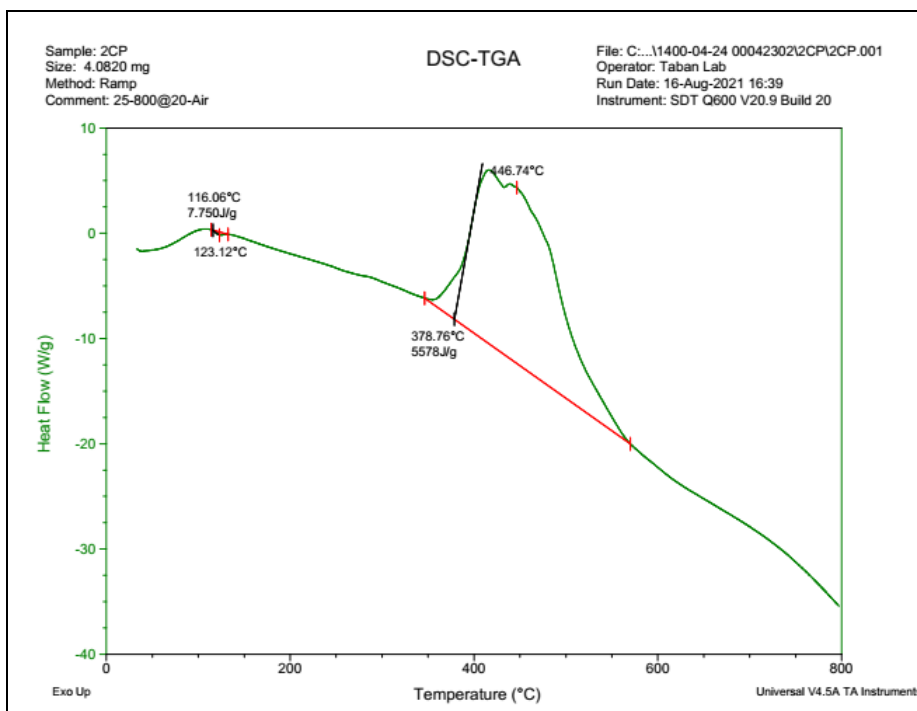


Figure 15 curve DSC to compound (LDPE/ C 6%)under oxygen