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Research Article

Pyrolytic Conversion of Waste High-Density Polyethylene to Wax: Temperature Optimization and Characterization of Wax

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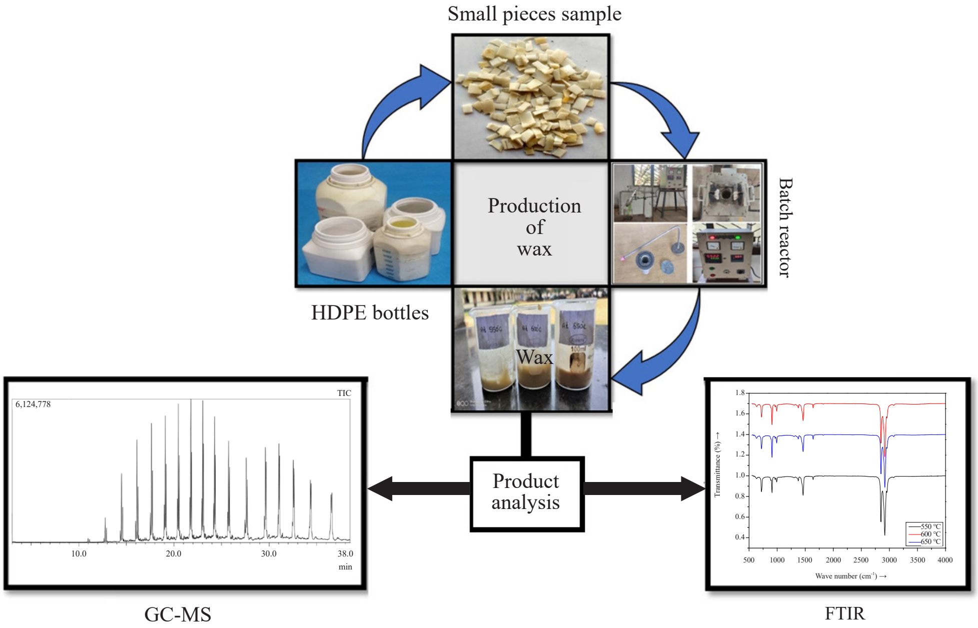
Abstract: The formation of wax from waste high-density polyethylene by thermal decomposition through pyrolysis is examined in this work. To get wax from waste high-density polyethylene, the thermal cracking reaction is run in a semi-batch pyrolysis reactor at 550 °C, 600 °C, and 650 °C. The yield percentage, melting point, specific gravity, and penetration degree of wax varied depending on the temperature. At 600 °C, waste high-density polyethylene yielded the highest wax output (87.25%) with a 0.7768 specific gravity, a 59 °C melting point, and an 81.8 mm penetration degree. The fourier transform infrared spectroscopy (FTIR) analysis concluded the presence of aliphatic hydrocarbon compounds (alkane, alkene, alcohol, and cycloalkane), which is also confirmed by gas chromatography-mass spectrometry (GC-MS) analysis. The innovation in this study lies in the systematic exploration and optimization of temperature conditions for wax production from waste high-density polyethylene (HDPE) bottles.

Graphical abstract



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***Keywords*:** waste HDPE, pyrolysis, wax

1. Introduction

The growth of plastics and their products as wastes in the environment increases day to day and hence accelerates pollution. People are being urged to act instantly to ensure efficient plastic recycling and processing as the amount of waste plastic pollution in the world keeps rising. So, modern research focuses primarily on the growth of new technologies and methods, or the upgrading of existing technologies and methods, for converting plastic waste into useful products. Modern industries place a high priority on advancing sustainable technologies and assisting in creating value-added products from waste. Among the valuable products, wax is also considered an important and useful product. As per the global polyethylene (PE) wax market, the projected global sales of wax in 2022 are US$ 980.2 million, representing a 5.5% year-over-year increase. By the end of 2032, the market is expected to grow at a compound annual growth rate (CAGR) of 5.2%, with a market value of US$ 1.63 billion.[1]

Several literatures report the preparation and characterization of wax from plastics like high-density polyethylene (HDPE), including low-density polyethylene (LDPE), and polypropylene (PP), which are summarized as follows: Berrueco et al. investigated the effect of temperature on the product distribution (liquid and solid) in bubbling fluidized bed pyrolysis of HDPE. The wax obtained contains an olefinic compound (up to sixty carbon atoms) that has a high molecular weight. When the temperature rises, heavier olefinic fractions crack, yielding lighter fractions with higher aromatic concentrations in the liquid fraction. Gas and solid waxes are the primary fractions obtained at temperatures below 730 °C. These primary fractions can be combined with naphthas and processed in a steam cracker. At 730-780 °C, the oil produced is around 15-20% of the feed. At temperatures above 730 °C, the yield of aromatic compounds increases. Polyaromatic production increases significantly at 780 °C. Waxes and oils are the predominant fraction recovered at low temperatures (650 °C), yielding 75.7% of the feed. The production of wax and oils reduces as the temperature rises. Wax production is 18.2% at 730 °C and 13% at 780 °C. The output of oils improves somewhat at 850 °C, reaching 13.8% of the input. Gas and solid waxes are the primary fractions formed at temperatures below 730 °C. At temperatures above 600 °C, aromatic compounds form, and with increasing temperatures, the aromatic content of oils and waxes increases.[2] Elordi et al. pyrolyze HDPE continuously at 500-700 °C in a pilot plant unit by using a conical spouted bed reactor to produce products like gas (C_{4-}), gasoline (C_5-C_{11}), diesel (C_{12}-C_{20}), and waxes (C_{21+}). At 500 °C, the yield of wax from pyrolyzed HDPE is roughly 67 weight percent. However, as the temperature rises to 700 °C, the yield of wax decreases to 12 weight percent. The yield of gases (C_{4-}), and gasoline fractions (C_5-C_{11}) increase, whereas the yield of diesel (C_{12}-C_{20}) decreases. At 700 °C, the yield of gases (C_{4-}), gasoline fraction (C_5-C_{11}) and diesel (C_{12}-C_{20}) are 39, 33, and 16 weight percent, respectively.[3] The conversion of HDPE to PE wax was investigated by Luo et al. in a 1 L continuous stirred-tank reactor operating at 673 K for 40-60 minutes. The less-branching PE wax, which was obtained at 673 K, has an average molecular weight of 3,000 and a melting point between 104 and 120 °C.[4] According to Undri et al., HDPE degrades thermally at temperatures above 380 °C and mostly transforms into wax and liquid products at temperatures below 730 °C. The amount of wax that transforms into liquid and gas products reduces as the temperature increases. Wax completely vanishes at 730 °C, leaving only liquid and gaseous products left. At temperatures above 850 °C, the polymer is almost converted into a gas (91.2%) that is composed of hydrogen, methane, ethane, ethene, propane, and propene. The composition of the liquid and wax is alkanes (C_{11} to C_{57}) and their equivalents, 1-alkene and 1,3-dialkene.[5] In a simple pyrolysis reactor system, Kumar and Singh polysized virgin HDPE between 400 °C and 550 °C. They reported that the pyrolytic oil produced during pyrolysis contains alkanes, alkenes, alcohols, ethers, carboxylic acids, esters, and derivatives of phenyl rings. The major compounds present in the pyrolytic oil are alkane (n-octadecane, n-heptadecane, and pentadecane) and alkene (1-pentadecene and 1-nonadecene). The pyrolytic oil that is produced has physical characteristics that are more similar to those of a blend of petroleum products. At low temperatures, extremely volatile compounds can be produced, whereas viscous liquids and wax can be produced at 500 °C and 550 °C. At 450 °C, the liquid yield is highest. The reaction time decreases with an increase in temperature.[6]

Salem et al. reported that during bench-scale continuous auger pyrolysis of plastic wastes at 500 °C, 5.5% pyro-oil, 23.8% light wax, 69.4% heavy wax, and 1.3% gases are formed. The wax products are used in lubrication, coating, and covering. A less pyro-oil and more wax-like fraction (94 wt%) with high conversion is formed during the slow pyrolysis. However, more pyro-oil and less wax content are formed during fast pyrolysis.[7] In a patented fixed-bed reactor, by pyrolyzing virgin HDPE, LDPE, and plastic solid waste (PSW), Salem and Dutta can obtain the wax. The highest 64.5% of wax is collected from LDPE by pyrolysis at 500 °C. Due to less branching, the yield of wax from HDPE (32%) at 500 °C is less as compared to LDPE. The highest yield of wax from PSW (9.25%) occurred at 700 °C. The waxes extracted from PSW, HDPE, and LDPE have average densities of 851.7 ± 1.0, 849.4 ± 5.3, and 879.5 ± 2.2 kgm^{-3}, respectively. Several alkane and alkene aliphatic hydrocarbons are present in the wax. The resulting waxes, however, have flash points that exceed diesel fuel specifications. So, the wax obtained is within the acceptable flammability range.[8] Arabiourrutia et al. pyrolyzed polymers like HDPE, LDPE, and PP at 450, 500, and 600 °C in a conical spouted bed reactor. They concluded that the production of the wax is highly selective. Low residence times and high heating rates, which lessen secondary reactions and boost the yield of primary pyrolysis products (waxes), are the reasons for this high selectivity. The waxes produced at 600 °C have heating values of 44-45 MJ/kg, which is comparable to standard fuels. They also reported that the waxes produced from HDPE, LDPE, and PP at 500 °C have a low melting point.[9] The pyrolysis of polyolefins (PP, HDPE, and LDPE) at 450-600 °C was studied by Aguado et al. in a conical spouted bed reactor. They reported that the activation energies of LDPE, HDPE, and PE are 214, 225, and 176 kJ mol^{-1}, respectively. The wax yields at 450 °C from PP, LDPE, and HDPE are 92 wt%, 80 wt%, and 80 wt%, respectively. When compared to commercial waxes, the resulting wax has a higher olefinic content and is less branched. The resulting waxes are mostly paraffinic with a melting point of 70 °C, but they are also more olefinic than marketable waxes.[10]

Zhang et al. conducted the catalytic pyrolysis in a two-stage fixed bed reactor with Zn-P/HZSM-5 catalysts for selective production of monocyclic aromatic hydrocarbons (MAHs) from polyolefin plastics. The pyrolysis of HDPE produces 54.6% liquid, 43.9% gas, and 1.5% coke with 86.43% MAHs and BTEX (9.25% benzene, 25.96% toluene, 4.94% ethylbenzene, and, 37.16% xylene). However, the catalytic co-pyrolysis of LDPE, HDPE, and PP (1:1:1) showed synergistic effects, achieving 55.7 wt% liquid yield with 87.66% MAHs, and 79.89% BTEX (10.99% benzene, 27.57% toluene, 4.49% ethylbenzene, and 36.84% xylene).[11] Qian et al. studied the effects of pretreatment and the structure of zeolite on the aromatization of HDPE over gallium-promoted zeolites under different gallium loading amounts and different pretreatments. The hydrogen reduction of gallium-promoted HZSM-5 resulted in the formation of highly active gallium species, leading to increased alkene yield but no significant improvement in total aromatic hydrocarbons from HDPE. However, the reduction and oxidation of Ga/Z5 produced stable GaO^+ species, which showed high dehydrogenation activity, achieving a remarkable 65% yield of aromatics with a 90% BTEX selectivity in the liquid product. A 6% gallium loading in the zeolite caused a decrease in aromatic hydrocarbon formation due to pore blockage and a reduced Brønsted acid amount.[12] However, conducting a comprehensive comparative analysis of the waxes about yield, composition, and physical properties is imperative to maximize economic gains and ensure the environmentally sustainable transformation of waste HDPE into wax. Therefore, in this work, waste HDPE bottles used as chemical containers are transformed into wax at various temperatures (550 °C, 600 °C, and 650 °C) in a batch pyrolysis reactor, and the resulting waxes are analyzed. The novelty of this work is that it improves on previous research by optimizing the conversion process, conducting comprehensive characterization, evaluating economic and environmental considerations, and using a batch reactor for more controlled experiments. These advancements contribute to a deeper understanding of converting waste HDPE into valuable products like wax and pave the way for more sustainable solutions to plastic waste management.

2. Materials and methods

2.1 *Materials*

The chemical storage bottles are used as a source of HDPE in this work. The chemical bottles are collected from the chemistry laboratory at Veer Surendra Sai University of Technology (VSSUT), Burla. After complete use of the chemicals present in the plastic bottle, the chemical bottles are properly washed with tap water. The washed bottles are manually broken into tiny pieces.

2.2 *Methods*

2.2.1 *Characterization of sample*

The standard procedures like ASTM D 4442 (moisture content), ASTM D 3172 (volatile matter content), ASTM D 3175 (ash content), and ASTM D 3177 (fixed carbon content) are used for the proximate analysis. The ultimate analysis is performed on an air-dried basis using the Vario El III CHNS elemental analyzer Germany, which is operated at 1,200 °C using a thermal conductivity detector (TCD) with He as a carrier gas. The sample is instantly oxidized through flash combustion, and the combustion products are separated using a chromatographic column and identified using TCD. A bomb calorimeter is used to determine the calorific value of HDPE according to ISO 1716 and ASTM D240 standard procedures.

2.2.2 *Batch pyrolysis study*

The batch pyrolysis of HDPE is conducted by the batch reactor. The stainless-steel tube is the main part of the reactor, which is attached to an outlet tube at one end to release the pyrolyzed products. The stainless-steel tube is heated by an electric furnace. The temperature of the reactor is regulated with an accuracy of ± 3.6 °C by a proportional-integral-derivative controller. The Cr-Al: K-type thermocouple is attached to the batch reactor to detect the temperature. The pyrolysis experimental temperature to yield the maximum condensable fraction is optimized by experimenting with different temperatures from 550 to 650 °C. 30 g of HDPE pellet sample is placed into the reactor setup, which is kept at the desired temperature (550 °C, 600 °C, and 650 °C) for the pyrolysis experiment. The condenser is used to collect the condensable wax and then weigh it. To ensure that the results are reproducible, the experiment is repeated three times. The water-cooled condenser accumulates the wax. These waxes are collected and subjected to weight. The batch pyrolysis experiment is repeated three times to confirm that the results are reproducible.

2.2.3 *Characterization of products*

The GC-MS of the wax sample is analyzed by using Shimadzu Gas Chromatography-Mass Spectrometry equipment (GC-MS QP 2020 NX), which has an SH-Rxi-5ms capillary standard non-polar column (dimension: 30 m, ID: 0.25 mm, and film thickness: 0.25 µm). The wax sample is run over a range of 35-650 m/z under the following experimental conditions: flow rate of He = 1.0 ml/min; oven temperature = 35 °C-280 °C at 5 °C/min; and injection volume = 1 µl. The gas chromatography-mass spectrometry (GC-MS) results of the pyrolytic wax are compared using the National Institute of Standards and Technology (NIST) 20 (2020) mass spectral library. The Digital Melting Point Apparatus can calculate the melting point of wax. The capillary tube was filled with wax, and after adding the appropriate amount of castor oil to the beaker, the base of the beaker was placed over a magnetic stirrer. This magnetic stirrer aids in the stirring of castor oil. The magnetic stirrer and heater are set to 40% speed. The temperature at which the wax melts is recorded on the digital display. The specific gravity is measured by a dried and cleaned pycnometer by weighing the weight of the dried and cleaned pycnometer and the weight of wax filled pycnometer. The American Society for Testing and Materials (ASTM) D1321 standard method is used to determine the degree of penetration. A standard needle (3.2 mm, 2.5 gm, hardened stainless steel) is dropped freely into the sample for 5 seconds at room temperature after being released from a penetrometer (which comes with a 100-gram weight). Tenths of a millimeter is the unit used by the penetrometer to measure the needle's depth of penetration into the sample. The characterization of the product is repeated three times to confirm that the results are reproducible.

3. Results and discussion

3.1 *Characterization of sample*

The proximate analysis belongs to the calculation of moisture content, volatile content, ash content, and fixed carbon content. The amount of energy used to dry the raw material is calculated by the quantity of moisture present. The amount of fuel gas to be produced is measured by knowing the volatile content. Volatile matter content and fixed carbon, on the other hand, suggest how easily a raw material can burn and oxidize, depending on how the raw material will be used as a raw material for energy. The ash content affects both the cost of handling and processing of a process. Lower energy density is found in samples with higher moisture and ash contents.[13]

The ultimate analysis includes the estimation of the elements like carbon, hydrogen, nitrogen, sulphur, and oxygen in the sample. To produce oil products, a sample with a higher carbon and hydrogen content will yield a higher percentage of aromatic compound yield and a higher heating value. The sulphur and nitrogen content specifies the emission of pollutants like SOx and NOx during pyrolysis. A higher oxygen content indicates a lower calorific value of the sample and the formation of oxygenated compounds during pyrolysis.[14] The temperature at which raw materials burn in devices is regulated by the calorific value, which also shows the effectiveness of combustion.[15] The sample having higher ash content, moisture content, and oxygen content with a low carbon content shows a low calorific value.[16] The proximate analysis, ultimate analysis, and calorific value of waste HDPE are displayed in Table 1.

Table 1. Proximate, ultimate and calorific value analysis of waste HDPE

Proximate analysis		Ultimate analysis	
Moisture content (%)	0	Carbon (%)	85.80
Volatile matter (%)	99.92	Hydrogen (%)	13.93
Ash content (%)	0.08	Nitrogen (%)	0.20
Fixed carbon (%)	0	Sulphur (%)	0.07
		Oxygen (%)	0
Calorific value (cal/gm)		10,935	

Table 1 shows that the nitrogen and sulfur content of HDPE samples is zero or negligible. As a result, a negligible amount of SOx and NOx pollutants are released. Since HDPE contains high carbon content, low oxygen content, and high ash content. So, the calorific value of HDPE is high. Since the volatile content is higher in HDPE, so more condensable products are formed leaving less residue which supports the formation of an adequate amount of wax.

Table 2. Yield, melting point, specific gravity, and penetration power of waxes

Pyrolysis temperature (°C)	Yield (%)	Melting point (°C)	Specific gravity	Penetration degree (mm)
550	84.38	58	0.7932	30.4
600	87.25	59	0.7768	81.8
650	86.74	62	0.7766	196

3.2 *Product analysis*

3.2.1 *Analysis of wax yield, melting point, specific gravity, and penetration power*

The formation of viscous and waxy products is due to improper cracking of plastic to high molecular mass hydrocarbon components.[17] The yield, melting point, specific gravity, and penetration degree of the wax sample produced at various temperatures are summarized in Table 2. With the increase in temperature from 550 °C to 600 °C, the yield of wax increased, then decreased as the temperature increased to 650 °C. As a result, the optimal temperature for producing wax from waste HDPE is 600 °C. The molecular weight range of wax and its quality are evaluated using the melting point. The specific gravity of the wax increases with increasing carbon number and vice versa.[18-19]

The melting point rises from 550 °C to 650 °C as the temperature rises, but the specific gravity decreases slightly. The wax penetration is unaffected by the low pyrolytic temperature. The hardness of the wax decreases and the degree of penetration increases as temperature and reaction time increase. However, at high temperatures and reaction times, it abruptly decreases.[20]

3.2.2 *FTIR analysis*

Figure 1 depicts the fourier transform infrared spectroscopy (FTIR) analysis of wax obtained by pyrolysis of waste HDPE. It can be concluded from Figure 1 that the nature of the compound produced at different temperatures is the same. The transmittance peaks at 636, 721, 911, 965, 994, 1,374, 1,644, 2,851, 2,919, and 3,080 cm^{-1} correspond to the OH-out of plane bending (alcohol), $-(CH_2)_n-$ rocking ($n \geq 3$, $-CH_2-$ group), C-H out of plane bending (Vinyl group), trans C-H out of plane bending (alkene), C-C stretching (alkane), C-H symmetry bending ($-CH_3$ group), C=C stretching (alkenyl group), C-H symmetry stretching ($-CH_2-$ group), C-H asymmetry stretching ($-CH_2-$ group), C-H stretching (terminal vinyl), respectively. The transmittance peak at 1,463 cm^{-1} shows both the C-H asymmetry bending ($-CH_3$ group) and the C-H bending ($-CH_2-$ group). Both the transmittance peaks at 2,919 and 3,080 cm^{-1} also indicate C-H stretching for cyclo alkane depending upon the angle strain.

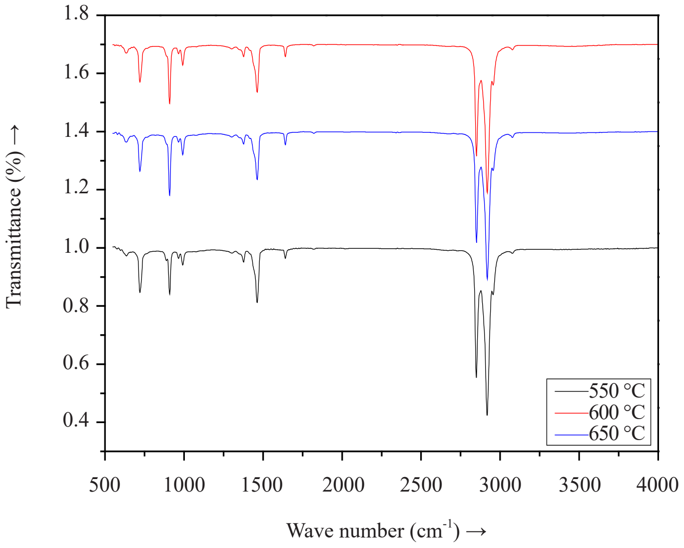

Figure 1. FTIR analysis of wax

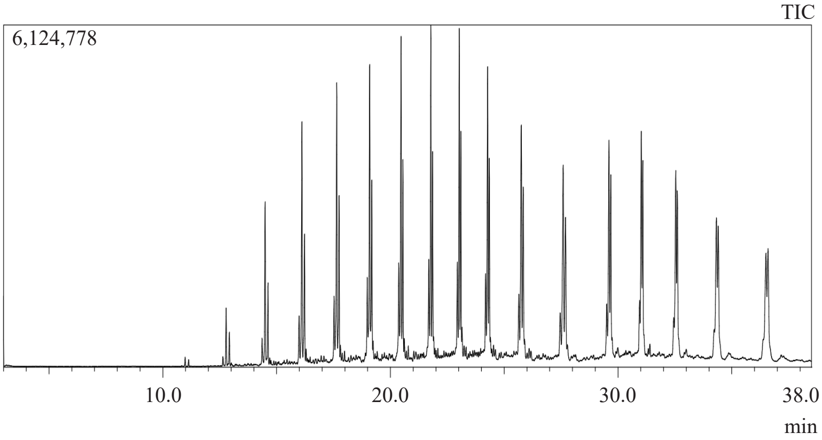

Figure 2. GC-MS plot of wax collected at 600 °C

Table 3. GC-MS analysis of wax obtained at 600 °C

Compound	Molecular formula	%
(1) Aliphatic alkane		
Triacontane	$C_{30}H_{62}$	7.82
Tetracosane	$C_{24}H_{50}$	4.9
Pentadecane	$C_{15}H_{32}$	4.61
Heneicosane	$C_{21}H_{44}$	4.49
Docosane	$C_{22}H_{46}$	3.2
Tetratriacontane	$C_{34}H_{70}$	2.55
Eicosane	$C_{20}H_{42}$	2.45
Heptadecane	$C_{17}H_{36}$	2.12
Hexadecane	$C_{16}H_{34}$	2.02
Tetradecane	$C_{14}H_{30}$	0.94
Tridecane	$C_{13}H_{28}$	0.37
(2) Aliphatic alkene		
1-Nonadecene	$C_{19}H_{38}$	23.7
1,19-Eicosadiene	$C_{20}H_{38}$	4.11
1-Tetra decene	$C_{14}H_{28}$	1.88
1,13-Tetradecadiene	$C_{14}H_{26}$	1.65
1,15-Hexadecadiene	$C_{16}H_{30}$	0.97
1,21-Docosadiene	$C_{22}H_{44}$	0.72
1-Tridecene	$C_{13}H_{26}$	0.68
(Z)-3-Heptadecene	$C_{17}H_{34}$	0.18
Cetene	$C_{16}H_{32}$	0.09
(3) Aliphatic alcohol		
1-Hexacosanol	$C_{26}H_{54}O$	25.79
(Z)-13-Docosen-1-ol	$C_{22}H_{44}O$	3.21
cis-9-Eicosen-1-ol	$C_{20}H_{40}O$	0.53
Behenic alcohol	$C_{22}H_{46}O$	0.17
(4) Aliphatic cyclo alkane		
Cyclo hexadecane	$C_{16}H_{32}$	0.53
Cyclo pentadecane	$C_{15}H_{30}$	0.12
[6-cyclopentyl-3-(3-cyclopentylpropyl) hexyl] cyclohexane	$C_{25}H_{46}$	0.20

3.2.3 *GC-MS analysis*

The GC-MS plot for the wax obtained at 600 °C during pyrolysis of waste HDPE under isothermal conditions is drawn in Figure 2.

From the GC-MS analysis, it is confirmed that aliphatic compounds like alkane, alkene, alcohol, and cyclo alkane are present in the wax obtained at 600 °C from waste HDPE, and these products are listed in Table 3.

The wax obtained at 600 °C contains a maximum of 1-Hexacosanol (25.79%) and a minimum of cetene (0.09%). Among different classes of compounds, wax contains a greater number of different types of aliphatic alkane fractions as compared to others. The GC-MS analysis is also supported by the FTIR analysis.

4. Conclusion

The results indicate a notable peak in wax production, reaching up to 87.25%, under the optimized condition of 600 °C. Additionally, the wax derived at this temperature exhibits specific characteristics, including a melting point of 59 °C, a specific gravity of 0.7768, and a penetration degree of 81.8 mm, as determined through rigorous experimentation. Furthermore, the identification of various aliphatic compounds such as alkane, cycloalkane, alkene, and alcohol through FTIR and GC-MS analysis adds depth to our understanding of the chemical composition of the produced wax. By establishing 600 °C as the optimal temperature for maximizing wax yield from waste HDPE, the findings of this study provide valuable insights into the temperature-dependent behavior of the pyrolysis process, shedding light on the optimal conditions for maximizing wax production efficiency from waste HDPE. The work has increased our understanding of the link between temperature and wax quality by characterizing wax generated at different temperatures, which has significant implications for industrial uses and waste management techniques.

Conflict of interest

The author declares no competing financial interest.

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