Communication

Hydrotreatment over the Pt/Al$_2$O$_3$ Catalyst of Polyethylene-Derived Pyrolysis Oil and Wax

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ABSTRACT: Upgrading fuels is an essential requirement to reach environmental specifications, namely, in removing sour components deemed hazardous nowadays and that cause fouling and disturb operations in downstream industry too. A lack of studies that report fundamental data also exist in the literature, as to the impact of hydrotreatment on thermochemical conversion products of plastics. We hereby report for the first time in this communication the effect of hydrotreatment on the products of plastic pyrolysis. Two types of pyrolysis products, oils and wax, were extracted from fluidized bed reactor pilot-plant operations at two operating temperatures (600 and 700 °C). These were subjected to hydrotreatment in a batch reactor over a Pt/Al$_2$O$_3$ catalyst for 6−8 h. Dependent upon the sample type, the hydrotreatment temperature reached 350 °C and was initialized at 140 °C. The hydrotreatment reactor handles a 180 barg pressure, and the operating pressure was kept steady in operation during measurements. The pressure rates (0.29−0.62 bar h^{-1}), which decreased with the temperature (140−200 °C), were also recorded. The olefinic compounds were saturated from wax post-hydrotreatment and detected by alteration of carbon singular and double bond existence. Naphthalene and paraffins were also eliminated from oils treated, as confirmed by infrared spectroscopy and chromatography. The conversion during the hydrotreatment reduced the reactivity of the pyrolysis oil and promoted the production of diesel and kerosene production. It is therefore recommended to extend these types of studies using different catalysts that can provide environmental specifications to fuels with minimal costs in the near future as an essential route for integrated operations in industry.
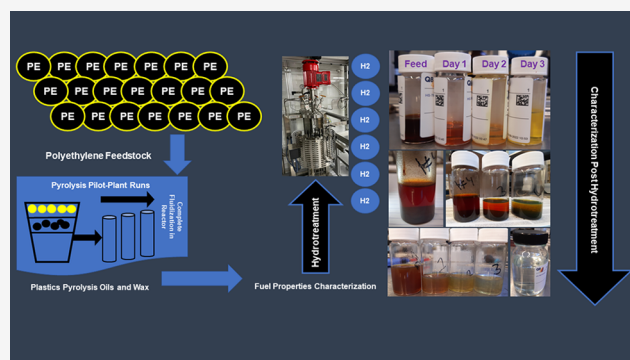
1. INTRODUCTION

Banning or obliteration of plastics from the manufacturing cycle could result in more harm to the environment. This is due to promoting industrial manufacturing of other alternatives (e.g., metals and glass), which would double energy consumption and increase greenhouse gas emissions by 3-fold.[1] Synthetic plastic production has increased 20 times over the past 50 years. The year 2020 had a total production rate of 367 million tonnes,[2] with a projected production reaching 500 million tonnes by 2050, resulting in a 40% crude consumption.[3] A mere 9% of the plastic waste (PW) is recycled annually,[4] and the majority of PW generated annually (≈464 million tonnes) is still being landfilled.[5] On the other hand, chemical recycling of PW offers a plethora of options to manage this particular type of feedstock, with several advantages that include producing valuable chemicals, fuels, and energy.[6] Chemical recycling refers to a range of technologies encompassing solvolysis, gasification, and pyrolysis.[7] Plastic pyrolysis involves the treatment of PW in an oxygen-free environment to generate various products, such as crude oil equivalent products, liquid fuels, and char residue.[6]

The specific composition of each product is influenced by several factors, with the most crucial being the temperature and the type of feedstock subjected to pyrolysis.[8] If PW was efficiently recycled through chemical recycling, it could result in significant savings of some 3.5 billion barrels of oil, equating to a monetary saving of $176 billion annually.[7]

Hydrotreatment is a process that involves hydrogenating olefins and aromatics, eliminating sulfur, nitrogen, and oxygen, and removing metals, using a reactive hydrogen (H$_2$) atmosphere. It is an upgrading process of oils to the hydrocarbon (HC) fuel level for demonstration use.[9] There are two types of hydrotreatment: One is conducted at mild temperatures, where molecules can be saturated without significantly affecting the boiling temperature of the

components in the oil, because there is no cracking involved. The other type is known as destructive hydrotreatment or hydrocracking, which combines cracking and saturation in a single step. This method is primarily employed in petroleum refining.[10] In this study, hydrotreatment of pyrolysis oils and wax derived from pilot-plant studies on plastics was conducted in a batch reactor using a 1 wt % Pt/Al$_2$O$_3$ catalyst. To date, numerous works have been published on hydrotreatment (upgrading) of bio-oils derived from various biomass feedstocks or the actual pyrolysis process of various materials. However and to best of knowledge, this is the first study that reports fundamental results on upgrading plastic pyrolysis products in the literature using the methods and catalyst described hereafter.

2. EXPERIMENTAL SECTION

2.1. Sample Acquirement and Hydrotreatment. Details regarding the pyrolysis in the fluidized bed reactor (FBR) pilot plant and polyethylene (PE) feedstock used could be found in our previous study,[11] including the experimental setup and details regarding the design and configuration.[12] The samples considered in the current work were extracted from the pyrolysis of linear low-density PE after catalytically pyrolyzing it, as shown in Figures S1 and S2 of the Supporting Information. The pyrolysis temperature was chosen on the basis of the thermogravimetric analysis (TGA) of the feedstock, which revealed that the measured melting point (T_m) was 124 °C and the onset temperature of the polymer was 462 °C,[22] and the maximum detected temperature of the thermogram based on varying heating rates between 5 and 25 °C min^{-1} was 490−625 °C.[23] This provides a range for the operating temperature between 500 and 700 °C, upon which our work is based.[11,12] The detailed procedure and process data analysis could be found elsewhere,[12,13] and a summary of sample coding of the pyrolysis products is shown in Table S1 of the Supporting Information. Basic properties of the samples were depicted in our previous study.[11] Hydrotreatment was conducted using a 500 mL Premex system high-pressure batch reactor equipped with a gas-induced impeller for mixing gases into the liquid phase, following the protocol and configuration shown in Figure S3 of the Supporting Information. The reactor was filled with the prepared catalyst in the amount of 1.75 g of Pt/Al$_2$O$_3$ (Table S2 of the Supporting Information), with 172.54 g of pyrolysis oil/heptane mixture, which corresponds to 43.4 g of pyrolysis oil and 129.14 g of heptane. Heptane is added to aid in precipitating the products (in hydrotreatment) and characterizing it further analytically, which was eliminated in the analysis program. The details of each conducted hydrotreatment experimental run are shown in Figures S4−S6 of the Supporting Information, including process parameters, reduction of the catalyst, product recovery, sample collection, and conditions. Samples were extracted for analytical studies after each 24 h span, as long as a pressure change occurred, resulting in four withdrawn samples for each of the plastic pyrolysis oil samples (600 and 700 °C) hydrotreated and three samples of the pyrolysis wax recovered at 600 °C.

2.2. Characterization. A vast range of experimental work was conducted on the pyrolysis oil and wax samples studied, which were first subjected to simulated distillation performed on a PerkinElmer Clarus 680 gas chromatograph with an autosampler, on-column injection (100 °C), and using a flame ionization detector (FID) (Table S3 of the Supporting Information). Nitrogen was used as the carrier gas (5 mL/min, with 10 min holding time). About 0.05 g was separated on a Restek MXT 5 m/0.53 mm column into components at the boiling point using gas chromatography. Gas chromatography/mass spectrometry (GC/MS) was conducted using Thermo Trace Ultra ISQ GC/MS. The analysis started with the gas chromatograph, where the sample was vaporized into the gas phase and separated into its various components using a capillary column. A quadrupole mass analyzer was used to differentiate mass-to-charge (m/z) ratios. A Bruker Tensor 27 Fourier transform infrared (FTIR) spectrometer

was used to collect the spectra of the samples with a 2 cm^{-1} resolution and an absorbance band range from 400 to 4000 cm^{-1} as per the experimental protocol described in Table S4 of the Supporting Information. The obtained spectra were processed using OPUS Bruker's software. Density was measured using an Anton Paar density meter (DMATM 4100 M) at 15 °C following the method described in ASTM D4052 (Figure S7 of the Supporting Information). Inductively coupled plasma atomic emission spectroscopy (ICP−AES) was conducted using a Thermo Scientific ICAP 6000 series ICP spectrometer as per Table S5 of the Supporting Information. A 10 mg sample was also subjected to thermogravimetry using Mettler Toledo TGA/DSC 3+; data processing was conducted using StarE software; and carbon was also confirmed using Tanaka micro carbon residue analyzer ACR-M2 (Table S5 of the Supporting Information). The sulfur and nitrogen contents were also analyzed in the samples using Trace Elemental Instruments (Xplorer-NS) after dilution and detection using ultraviolet (UV) fluorescence and chemiluminescence detectors (Table S5 of the Supporting Information). Table S6 of the Supporting Information summarizes the experimental protocols followed for the analytical tests.

3. RESULTS AND DISCUSSION

Table 1 shows the densities of the PE pyrolysis products studied. The density increased proportionally as the pyrolysis

Table 1. Density (g/mL) and Carbon Residue (wt %) Estimated for the Pyrolysis Products Studied in This Work

sample	pyrolysis temperature (°C)	density (g/mL)	carbon residue (wt %)
pyrolysis oil	600	0.8049 ± 0.006	0.595 ± 0.1
pyrolysis oil	700	0.947 ± 0.007	5.665 ± 0.8
pyrolysis wax	600	0.813 ± 0.003	0.27 ± 0.04

temperature increased from 0.8049 ± 0.006 g mL^{-1} (600 °C) to 0.947 ± 0.007 g mL^{-1} (700 °C). The density of hydrocarbon products is a clear indication, along with boiling point measurements, of the composition and properties of the hydrocarbon mixture.[12] Henceforth, it was essential to estimate these properties at the initiation stages of this work to obtain a clear vision of the type of samples provided. The molar weight has a proportional relationship with the density; hence, an increase was noted with the increase in the pyrolysis temperature, indicating a higher mixture of heavy hydrocarbons (raw data are also presented in Table S7 of the Supporting Information, along with typical densities of hydrocarbon mixtures). A higher density in the pyrolysis oil can be attributed to more absorbance of oil on catalysts used in the pyrolysis process, which also influences (decreases) the production of oils as the operating temperature increases.[9,11] Aromatic are within the density range of 0.88−0.95 g mL^{-1} associated with a high aromatic content increasing with respect to the pyrolysis temperature (Table S7 of the Supporting Information).[14] The carbon residue (potential coke) was lower with samples extracted at 600 °C (Table 1 and Figure S8 of the Supporting Information), which was expected because lower operating temperatures are associated with a longer residence time that provides higher cracking of products.[15] This is also in line with the previous observation that higher temperatures of pyrolysis result in more aromatic content samples, because it is the main precursor of coke formation. Higher carbon residues are associated with unstable aromatic components that can cause fouling (and are avoided) when fuels are processed with catalysts and equipment alike.

On the basis of the measured boiling points (molar distribution) (Figure S9 of the Supporting Information), it was noted that samples were not in the light fraction range (>100 °C) and had a high hydrocarbon content consisting mainly of diesel (290−350 °C), kerosene/jet fuel (150−290 °C), and naphtha (C_5−150 °C), which are typical refinery fuel ranges also consistent with our previous findings.[11] The pyrolysis wax samples were higher (>30%) in the diesel range (Figure S9 of the Supporting Information). The sulfur content in the pyrolysis samples reached elevated levels of 17 ppm in samples extracted at 700 °C (Table S8 of the Supporting Information), indicating that further cracking is required to reach the required environmental fuel specifications. For the pyrolysis oil samples studied, the sulfur content was noted to increase proportionally in relation to the operating temperature. The content of the sulfur in the pyrolysis wax was also noted to be higher in comparative terms. This could be due to sulfur vacancies on catalyst metal edges and C−O cleavage activity with the temperature increase as a result of the Brønsted acid sites (S−H groups) situated on the sulfur edges.[16] The pyrolysis of the feedstock was conducted originally using a Magnofil BT catalyst in the FBR unit, which resulted in the depiction of the data described prior.[11,12] The feedstock material contained some 2.6% S content, which was noted to change in the resulting pyrolysis oil and wax (Table S8 of the Supporting Information). This could be attributed to a cracking S removal reaction that was initiated and triggered, accompanied by a S cleavage occurring for nitrogen- and carbon-based species.[11] Furthermore, silicon levels were high (1−7 ppm), indicating that a precious earth metal catalyst will be required to avoid quick deactivation (Table S8 of the Supporting Information). Pertaining to the hydrotreatment impact on pyrolysis products, each sample studied is discussed individually hereafter. The first sample studied was the pyrolysis oil extracted at 600 °C. Four samples were extracted as hydrotreatment was conducted with respect to the experimental protocol described in Figure S4 of the Supporting Information, which also includes the overall mass balance of the process. Depressurizing after the operation on the first day showed almost no effect on catalyst reduction/recovery (panels a and b of Figure S4 of the Supporting Information). Hydrogen consumption reduced during the hydrogenation process on the next day (Figure S4c of the Supporting Information). The last sample was withdrawn by the fourth day, where there was no change in the pressure, reaching the operating temperature of 360 °C (Figure S4g of the Supporting Information). The GC/MS analysis of the feed (untreated sample) shows it to be very paraffinic in nature (Figure S10 of the Supporting Information). The carbon number of the molecules is divided into di-unsaturated, mono-unsaturated, and saturated forms, as shown in Figures S11 and S12 of the Supporting Information. All di-unsaturated and mono-saturated paraffins were eliminated post-hydrotreatment (Table S9 of the Supporting Information).

The hydrotreatment of the pyrolysis oil extracted at 700 °C showed a similar pressure drop behavior and catalyst reduction (Figure S5 of the Supporting Information). A larger mixture of hydrocarbons was present in the sample in comparison to the that extracted at 600 °C (Figure S13 and S14 of the Supporting Information), which is attributed to a lesser cracking extent related to the residence time in the reactor.[11] Both pyrolysis oil samples showed conversion of di-unsaturated and mono-saturated paraffins after hydrotreatment

(Table S10 of the Supporting Information). However, the sample at 700 °C contained large amounts of naphthalene and methylnaphthalene, which were also converted, alongside the aromatics, to a vast degree with hydrotreatment following the schematic representation in Figure S15 of the Supporting Information. Naphthalene will be hydrolyzed to decalin (*cis/trans* decahydronaphthalene), with tetralin (tetrahydronaphthalene) as an intermediate. The naphthalene balance is further shown and confirmed in Figure 1 to depict the total conversion during hydrotreatment.
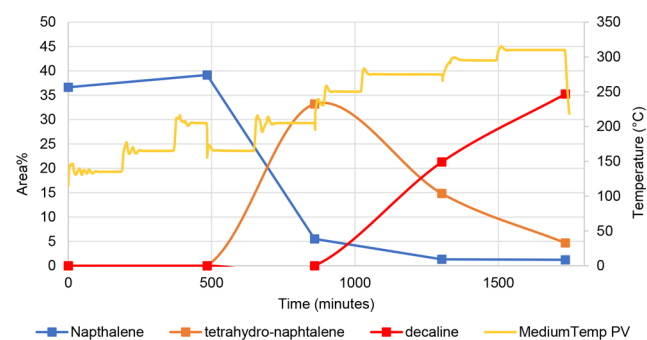

Figure 1. Total naphthalene balance for the pyrolysis oil (700 °C) during hydrotreatment.

The pyrolysis wax sample presented a different case, whereby the majority of its constituents were of olefinic nature (Figure S16 and Table S11 of the Supporting Information). These were all converted in the final hydrotreated sample (as depicted in Figure S16 of the Supporting Information and confirmed by infrared (IR) spectra (Figure 2).
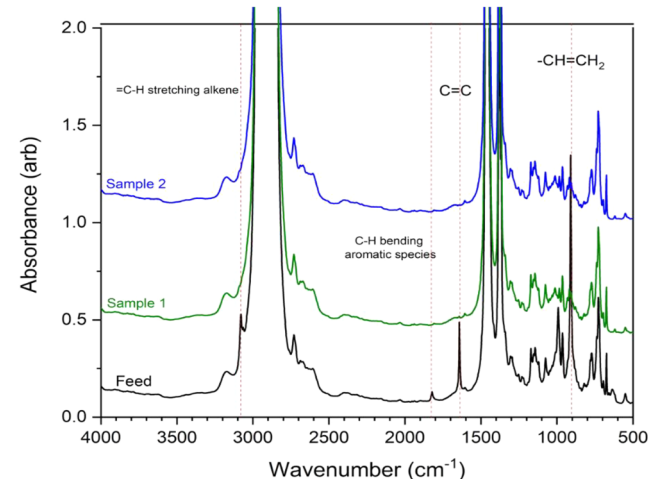

Figure 2. Combined IR spectra of the pyrolysis wax sample before (feed) and after hydrotreatment.

The IR spectra show prominent peaks in the wax samples, namely, =C−H, C=C, and −CH=CH$_2$ (Figure 2). These show that, with hydrotreatment, the saturation of olefins and aromatics (peak C−H bending aromatics) occur. Pyrolysis has been previously noted to produce a high aromatic content product,[15] with a high coke deposition precursor as the operating temperature increases. Pt as a choice of precious metal with a support of alumina is the optimal choice that can have good stability and anti-toxin properties as previous

reports show.[17] It is also highly compatible for hydrotreatment and fuel cell applications as a result of its nature.[18] This was the reason that conversion of paraffins was evident and was attributed to the use of hydrotreatment for pyrolysis oil (Table S10 of the Supporting Information). Hydrotreating is a process that requires a running cost of the hydrogen supply. It is important to understand that the goal of hydrotreating is not to completely saturate all double bonds. Typically, the aim is to strike a balance between the amount of hydrogen used and the composition of the resulting distillates in industry. Efforts that will combine various aspects of chemical recycling, including thermochemical conversion (TCC), biodegradation (e.g., use of bioplastics), and solvent use, could be combined in integrated efforts to support the downstream industry.[6,19−21] This will have both an operation and an environmental impact, with future industrial plans to reduce pollutants and the carbon footprint of processing energy distillates. The work conducted herein shows that hydrogen consumption was minimal for hydrogenating olefins and aromatics, removing sulfur and nitrogen via hydrodesulfurization and hydrodenitrogenation, and forming H_2S and NH_3. This also gives way that fuels are upgraded to environmental standards in this instance by eliminating sour components.

Past research efforts on upgrading pyro-oils have been dedicated to studying the impact of hydroprocessing on polyolefin thermal cracking products. Mangesh et al.[24] used a Ni−Mo catalyst for hydroprocessing polypropylene pyrolysis oils in a setup of 70 bar and 350 °C to produce a n-alkane, isoalkane, and aromatic mixture with a 95% match with diesel fuel. In our work (Figures S4 and S5 of the Supporting Information), the hydrotreatment was conducted on much milder operating pressures, resulting in high recovery of the diesel fuel range for the studied pyrolysis oils from linear low-density polyethylene (LLDPE) (Figure S9 of the Supporting Information). Both Escola et al.[25] and Serrano et al.[26] hydrotreated thermal pyrolysis (i.e., non-catalytic) oils using Ni−beta zeolite and Pd/h-ZSM-5, respectively. Enhanced gasoline range fuels were obtained in their case, which could lead to future work in ours, to target low-olefin gasoline fuels.

4. CONCLUSION

Upgrading fuels from different processes aims at reaching market standards, more specifically, environmental standards. This is quite essential for energy suppliers in industry to enforce a balance between the composition of aromatics and hydrocarbon content present in fuel distillates. In this work, we report the effect of hydrotreatment on the products of plastic (polyethylene) pyrolysis, namely, oils and wax, extracted at different operating temperatures. The density of the products was measured and noted to increase proportionally as the pyrolysis operating temperature, resulting in the derived oils, increased from 0.947 ± 0.007 g mL^{-1} (600 °C) to 0.947 ± 0.007 g mL^{-1} (700 °C). The density of hydrocarbon products is a clear indication, along with boiling point measurements, of the composition and properties of the hydrocarbon mixture, indicating a heavier mixture that was eliminated with the temperature. The pressure rates (0.29−0.62 bar h^{-1}), which decreased with the temperature (140−200 °C), were also recorded. Pt/Al$_2$O$_3$ was prepared and used for the hydrotreatment process, which was successful in eliminating all naphthalene and paraffinic compounds from the oils and cracked olefins present in the wax. The hydrotreatment process was conducted for a total time of 6−8 h (depending upon the

sample type), with temperatures reaching 350 °C. The Pt present provides an added value for eliminating the heavier hydrocarbons and saturating the bonds, as we have depicted prior. The GC/MS analysis of the untreated feed sample resulted in a very paraffinic content. The carbon number of the molecules were divided as di-unsaturated, mono-unsaturated, and saturated. All di-unsaturated and mono-saturated paraffins were eliminated post-hydrotreatment. The pyro-wax derived and studied in this work from the pyrolysis process possessed the majority of its constituents as olefinic in nature. These were all converted in the final hydrotreated sample, with prominent peaks possessing double saturated bonds, as $=C−H$, $C=C$, and $−CH=CH_2$. These show that, with hydrotreatment, the saturation of olefins and aromatics (peak C−H bending aromatics) occurs in a maximized case. It is therefore necessary, after reporting the basic and fundamental work in this communication, to continue with characterizing the catalyst and start using different carbon-based catalysts by going by previous studies on bio-oils. This should provide more rationale for techno-economic and scale-up operations in the future, which are also recommended for our case.

■ ASSOCIATED CONTENT

ⓈI Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.energyfuels.3c03152.

Additional experimental details, materials, and methods, including photographs of the experimental setup (PDF)

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Sanaa Hafeez, writing (initial draft), data analysis, and review; Maarten Van Haute, review; George Manos, review; Hajar Jawad Karam, data analysis; Achilleas Constantinou, review; and Sultan Majed Al-Salem, writing (initial draft), data analysis, review, conceptualization, and fund acquisition.

Notes

The authors declare no competing financial interest.

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