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Characterization and Hydrocarbon Identification of Liquid Fuel from Mixed Waste Plastics at Different Heating Time Intervals

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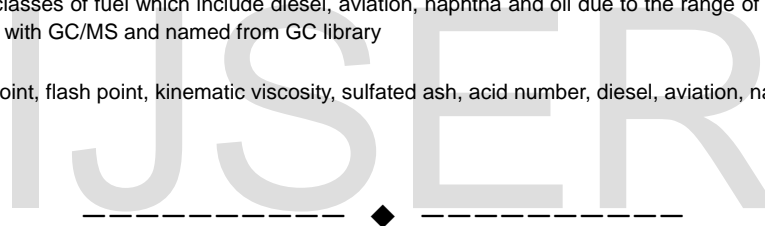
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ABSTRACT: Mixed waste plastics which included High Density Polyethylene ((HDPE), Low Density Polyethylene (LDPE), Polystyrene (PS), Polypropylene (PP), Polyethylene Terephthalate (PET) were investigated for production, characterization and hydrocarbon identification of fuel obtained from the plastics by catalytic pyrolysis. Silica-alumina was used as the catalyst. The mixed waste plastics were heated for one hour and two hours of contact time. The operation temperatures ranged between 250°C to 370°C giving a yellowish liquid after condensing the vapors with a chiller at 5°C. The reactor was connected to a fractionating column of one meter long and then to the condenser. The production ranged between 58 to 73% liquid, 18 to 34% cake and the rest was estimated as gas. The samples were investigated with different parameters based on diesel standards due to the wide range of hydrocarbon content. The parameters showed acceptable average ranges; cloud point 4°C, flash point 79.4 °C, kinematic viscosity 2.18 mm²/s, sulfated ash 4.7x10⁻⁵%, copper strip corrosion 1a, cetane number 63, acid number 72.9 µg and densities of 0.785 g/cm³. The identification of the hydrocarbons showed that the fuel falls into four classes of fuel which include diesel, aviation, naphtha and oil due to the range of hydrocarbons from C8 to C28 when analyzed with GC/MS and named from GC library

KEYWORDS: Cloud point, flash point, kinematic viscosity, sulfated ash, acid number, diesel, aviation, naphtha, oil



1. INTRODUCTION

In order to uplift the standards of living man has drastically increased the use of plastics and plastic products. This has caused a remarkable impact on the environment. Plastics have now become indispensable materials and the demand is continually increasing due to their diverse and attractive applications. Mostly thermoplastics polymers make up a high proportion of waste and this amount is increasing around the world. The disposal of mixed waste plastics is a serious environmental problem, because of their huge quantity and disposal challenges as thermoplastics do not biodegrade for a very long time [1-4].

Plastics are produced from petroleum derivatives and are composed primarily of hydrocarbons and other additives. Among the different recycling methods of waste plastics, thermal or catalytic degradation of waste plastics to fuel oil and valuable chemicals is regarded as the most promising methods to realize commercial use [5]. Pyrolysis is the major method used to convert waste plastics to fuel oil. When waste plastics are pyrolyzed, they can be converted into valuable hydrocarbon products. Different types of waste polymers could be converted into hydrocarbons with favorable properties for further application like fuel. The chemical recycling process (pyrolysis) yields hydrocarbon distillate comprising of straight and branched chain aliphatic, cyclic aliphatic and aromatic hydrocarbons. The resulting mixture is essentially the equivalent to petroleum distillate. The thermal pyrolysis of mixed waste plastics takes place through a complex free radical mechanism, giving rise to a wide product distribution that depends on the pyrolysis conditions like heating rate, temperature and residence time [6-10]. The heart of the pyrolysis system is the prime chamber reactor which performs the essential functions of homogenization controlled decomposition and out gassing in a single process. The process requires minimal maintenance apart from carbon residue removal and produces consistent quality distillate from mixed and low grade waste plastic. The key to an efficient pyrolysis process is to ensure that the plastic is heated uniformly and rapidly. If temperature gradients develop in the molten plastic mass, then different degree of cracking will occur and products with a wide distribution of chain lengths will be formed [11-13].

The proportion of gas, liquid and solid products depend very much on the pyrolysis technique used and on the reaction parameters. Basing on the operating condition, the pyrolysis process can be divided into three sub-classes (a) conventional pyrolysis, (b) fast pyrolysis and (c) flash pyrolysis [13]. The preferred technology for production of oily products is fast or flash pyrolysis at high temperatures with very short residence time [14].

In this study, catalytic pyrolysis was used in the production of fuel with a silica-alumina catalyst using two residence heating time, (i) one hour (ii) two hours at a temperature range of 250 °C – 370 °C. This was done by

conducting at low temperature to acquire high quality product. Catalytic degradation was done at low temperature with addition of the catalyst to achieve a high quality hydrocarbon [8]. In the degradation of the polymer chain using acidic catalyst the molecular weight of polymer chain could be rapidly reduced through cracking reaction and then carbonium ion intermediates would be rearranged by hydrogen and carbon atoms shifts producing the isomers of high quality [15]. The hydro-cracking involves the reaction with hydrogen over a bimetallic catalyst at moderate temperature and pressure which is focused onto obtaining a high quality hydrocarbon product. The silica-alumina catalyst used in hydro cracking helped in cracking heavy hydrocarbons in the mixed waste plastic incorporating both cracking and hydrogenation [16].

This study is concerned with the investigation of the physical properties and identification of hydrocarbons of Ugandan fuel produced from mixed waste plastic with a temperature range of 250 – 370 °C. Uganda is one of the major consumers of plastic products in East Africa and this puts the solid waste generated in the country to contain a high percentage of waste plastics. In Uganda, 300 tons of plastic bags are dumped into soils annually and Kampala, the capital city, generates about 900 metric tons per day of solid waste. The solid waste generated contains up to 15% waste plastics. Out of 900 metric tons solid waste generated, about 400 metric tons are collected per day [17-18]. This means the rest of the uncollected waste are washed away by rain and end up into drainage systems causing blockage which includes the plastic which is non-bio-gradable which permanently clogs up the channels causing water floods in the rainy season.

2. MATERIALS AND METHODS

Materials

The waste plastics used in the study were collected around Kampala, the capital city of Uganda, where most business activities are carried out. The city has the highest waste disposal challenge in the country due to its population density with no waste disposal management. The plastics are used as supermarket shopping bags, carrier/package, soft drinks containers, wrappers/covers of products domestic among others. These included low density polyethylene (LDPE) and high density polyethylene (HDPE). The mixed plastics were subjected to wet cleaning process to remove soil/ labels and inserts, then dried and shredded into pieces of 1 cm. The cracking silica-alumina catalyst which was synthesized in the laboratory was used to lower the operational temperatures during production. The temperatures were monitored at a range of 250°C – 370°C. The production was assembled in the laboratory with different apparatus which included; a batch reactor, a fractionating column, a condenser/chiller and a temperature logger. Sodium carbonate was used as a scavenger for the dioxin gases.

Methods

Production

The pyrolysis production was carried out in a batch reactor of 200g capacity, in each batch; 100 grams of mixed waste plastic were used with 10% of the catalyst and 10% of scavenger in reference to the waste plastic used. The reactor was subjected to heat in two categories; heating for one hour and heating for two hours and the mixed waste plastics cracked into vapour through a fractionating column to the condenser/chiller at 5 °C to liquefy the vapours into a liquid. The liquid then collected, measured, weighed and the yields were recorded. The experiments were repeated ten times for one hour and two hours heating. The quantification was done basing on the input (feedstock) versus the output of the liquid collected and the residue weight. The gas was estimated in the quantification as an approximation of the remaining output.

Characterization of the fuel

For each fraction of the cracked liquid were analyzed using standardized methods. A list of fuel parameters were characterized with the following methods; Acid number (ASTM D664), sulphated ash (ASTM D874), water and sediments (ASTM D2709), kinematic viscosity (ASTM D445), flash point (ASTM D93), distillation temperature (ASTM D1160), cetane number (ASTM D613), cloud point (ASTM D5773) and corrosion (ASTM D130) [19]

Hydrocarbon identification

Identification of the components of each fraction was carried out by means of a Gas Chromatography (GC) 6890N coupled with a mass spectrometer (5975 Inert XL MSD) Agilent technologies in reference with its library. The samples were run on the machine along with regular diesel from the local market. These samples were diluted with n-Hexane (Analytical grade from BDH) as a solvent in a ratio of 1:20. An HP-5MS column of 30 meters length, 0.25 mm inner diameter, and 0.25µm thickness was used in the GC. The column was used to separate the products according to their molecular weight [20]. The temperature sequence of the chromatograph oven was 40°C for 2min, ramp of 4°C/min to 300°C for 6 min. The chromatographic analysis conditions were as follows, detected mass interval 40 – 400, solvent delay 4.5 min, injector pressure 7.07Psi, total flow rate 93.2 mL/min, column flow rate 1.0 mL/min and split 59.9:1 [20].

3. RESULTS AND DISCUSSION

The production was based on heating contact time of the waste plastics with two categories; one hour and two hours. The overall condensed liquid for all the batches was a yellowish liquid with little transparency. The average production was 58% liquid oil and 34% cake for one hour category and 73% liquid oil and 18% cake for

the two hours heating contact time. The rest of the percentage for the both categories was estimated to be the gas generated in the system (Figure 1).

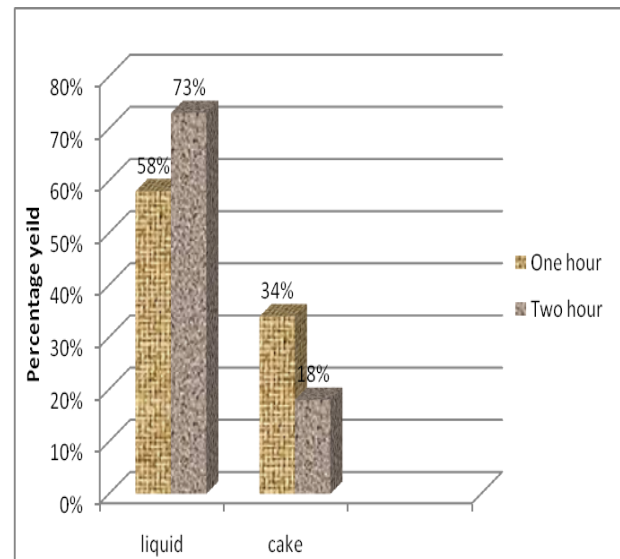


Figure 1: Percentage yield for liquid and cake

In the cracking of waste plastics, the yield (Figure 1) indicated the increase in the percentage with increase in the heating contact time. In doubling the heating time from one hour to two hours, the yield averagely increased by 15% and reduced the cake formation by 16% which is almost to half of the cake with one hour heating. This implies that when waste plastics are heated at longer time than one hour, the yield increases. Lee [23] showed a higher yield of liquid oil (66.98%), gas (28.95%) and cake (4.08%) for contact time of one hour. This was due to higher temperature of 450°C used compared to the temperature used in the study ranging from 250 – 370°C for the experiments.

The produced fuels were characterized based on the parameters for their applicability. These parameters are key factors for the fuel applicability. Since acid number is a measure of free fatty acids in the fuel, the two fuel categories showed very low values with the average of 72.90±0.31 µg of KOH/g for both and they were lower than 0.80µg of KOH/g of fuel standard by ASTM D664 [19]. This evidently rules out the possibilities of the fatty acid to be in the fuel samples. This means that the fuels are free from corrosion possibility of the systems or storage, because fatty acids in fuel lead to corrosion and fuel oxidation caused by higher concentration of free fatty acids. The densities of the two fuel samples were measured at the temperature of 15°C as average of 0.784 g/cm³ for one hour heating time and the average of 0.786 g/cm³ for two hours heating. The average values are less dense than 0.880 g/cm³ of diesel standard. The results are in the same range of 0.771 – 0.916 g/mL for fuel obtained from HDPE [21] and lower than 0.921 g/cm³ [27] for LDPE and 0.913 g/cm³ [28] for light cycle oil. The sulphated ash from the two categories was 4.80x10⁻⁵% and 4.5x10⁻⁵% for one heating time and two hours, respectively. They are lower than the maximum standard value of diesel of 0.020% wt [19]. The fuel should not

contain more than 15ppm of sulphur [22]. In comparison with Achyut [24] and Arands [28] which showed 0.002% wt and 0.6% respectively; the samples have very minimal levels of sulphur as fuel. This means that the fuel can be used with no exhaust particulate levels. This implies that the effect on the quality of residual metals in the fuel samples is almost negligible resulting in less or no danger which can be caused on the engine.

In determining the combined volume of free water and sediments in the samples, the average values were 0.025% and 0.024% for one hour and two hours of heating, respectively. These average values were much lower than 0.05% maximum limit for diesel fuel [19]. This means that the fuels are within the acceptable standard as per ASTM D2707. This indicates that the fuel is free from microbial growth caused by free water reaction with esters to form fatty acids which supports microbial growth in the system storage tanks. The results are slightly higher than 0.01% from Moinuddin [25] for the high density polyethylene. This may be due to poor control of air flow in the batch reactor of our experiments. The resistance to flow of the fluid under gravity at 40°C is the kinematic viscosities of the two average categories were found to be 2.17 mm²/s and 2.19 mm²/s of one hour and two hours, respectively. The average values are within the range of 1.9 – 6.0 mm²/s of diesel fuel as per ASTM D445. This means that the ratio between the applied shear stress and rate of shear of the fuel is minimal and causes no failure in the injector's engine performance. In comparison with 1.2mm²/s of Sharma [26] for polyethylene fuel, the values

were slightly higher. This may be due to the effects of mixed LDPE and HDPE for our experiments.

The ability of the fuel to form a combustible vapours above the liquid is determined by flash point value. Acceptable diesel fuel must have a flash point above 38°C [19]. This study showed an average of 78.35°C for the fuel of one hour heating and 80.4°C for two hour heating. The results from this study were slightly higher than that of regular diesel fuel of 52°C from the local fuel stations and lower than that of biodiesel standards of 150 °C. The data from our experiments were in agreement with the range of 76 – 86°C from Miskolczi [8] and close to 93°C from Arandes [28]. This means that the studied fuel has a minimal tendency to form a flammable mixture with the air circulation of the global or local ignition systems.

The lowest temperature operability of the experimental fuels was studied as cloud point at which the fuel first forms a cloud of wax crystals. The fuels averagely formed waxy cloud at 4°C. This means that the fuels can be used well in regions where temperatures hardly drop to 4°C like the tropics. In other regions where temperature drops below four degrees, the fuel can be improved by pour point depressants additives which pushes the fuel to form waxy clouds at -21°C [19].

Table 1. Characterization data for one hour and two hours heating

Parameter	Values		unit	Test method
	One hour	Two hours		
Appearance	Yellowish	Yellowish		
Flash point	78.35	80.4	°C	D93
Water and sediment	0.025	0.024	% volume	D2709
Kinematic viscosity, 40°C	2.17	2.19	mm ² /s	D445
Sulfated ash	4.80x10 ⁻⁵	4.50x10 ⁻⁵	Wt %	D874
Copper strip corrosion	1a	1a		D130
Cetane number	62.96	63.03		D613
Cloud point	4	4	°C	D2500
Acid number	72.90	72.90	µg KOH/g	D664
Distillation test			% distilled (°C)	D1160
10%	150	150		
50%	232.5	235.6		
90%	316	316		

Hydrocarbon identification

The yield compositions of the samples were analyzed with GC (6890N) coupled with mass spectrometer for identification of hydrocarbons. The samples were analyzed alongside regular diesel for comparison of the hydrocarbons and naming of the structures was done by use of the GC/MS Library for the

identification of the hydrocarbons in the samples and diesel. Spectrum responses were produced for each sample and at different retention time, different compounds were revealed. In the samples of one hour heating elution of the compounds averagely started at 5.33 minutes as the retention time with octane (C₈H₁₈) as the first

hydrocarbon and the run ended at 55.16 minutes as retention time with Hexacosane(C₂₆H₅₄) according to the GC/MS (6890N) library. With the retention time between 8 to 19 minutes the spectrum response were showing the highest peak abundance (Table 2.1) and started decreasing in abundances up to the last elute. There was a steady increase in the hydrocarbons from C₈H₁₈ up to C₂₆H₅₄ although there was an overlap of hydrocarbons with the increase in the retention time (Table 2.2). Based on the GC/MS library, the sample contained non aromatic compounds mainly aliphatics with carbons from C₁₀ to C₂₆. In the samples of two hour heating time, the trend of elution was almost the same as in one hour heating time (Table 3.1). In the two hour heating, samples had Eicosane (C₂₀H₄₂) which was missing in the samples of one hour heating. The two hour samples eluted Heptacosane (C₂₇H₅₆) as the last compound at 56.99 minute retention time (Table 3.2).

In the spectrum response of both samples, similar hydrocarbon compounds appeared in both samples at almost the same retention time. This introduced a fraction classification based on the number of carbons present; the naphtha fraction with C₈ to C₁₄ with compounds range from octane (C₈H₁₈) to tetradecane (C₁₄H₃₀). The aviation fraction with carbon ranging from C₈ to C₂₀ ending with

Eicosane (C₂₀H₄₂) as shown in Table 3. The diesel fraction where carbons range from C₈ to C₂₈ ending with Octacosane (C₂₈H₅₈). Lastwerethe oil fractions which slightly differ from diesel with carbon range of C₈ to C₂₇ ending with heptacosane (C₂₇H₅₆) as in Tables 2 and 3. Both samples of our study have all the four classes of fuel.

In comparison with the diesel fuel spectrums (Table 4.1), the trend of eluting were the same for all although there was an overlap of Heptane (C₇H₁₆) in diesel at 54.39 minute retention time (Table 4.2). The high responses for diesel concentrated more between 22 to 42 minutes of the retention time which was not the case with the studied samples (Table 4.1). Looking at all the spectrum response of the samples and that of diesel, there was at least a compound in one sample groups which was missing in the other groups (Table 5). Eicosane (C₂₀H₄₂) in the two hour heating samples and diesel samples but was missing in one hour heating samples. Heptane (C₇H₁₆) appeared in diesel samples and was missing in the other sample groups. Heptacosane (C₂₇H₅₆) appeared in two hour samples and was missing in other sample groups. This gives a simple uniqueness to each group samples in the study.

Table 2.1. GC/MS Chromatogram response/abundance of mixed plastic wastes heated for one hour

Compound Name	Response/Abundance (10 ⁷)	Compound Name	Response/Abundance (10 ⁷)
Octane	0.42	Heptadecane	0.27
Nonane	2.20	Octadecane	0.25
Decane	1.81	Nonadecane	0.21
Undecane	1.42	Heneicosane	0.19
Dodecane	1.12	Docosane	0.16
Tridicane	0.81	Tricosane	0.15
Tetradecane	0.52	Tetracosane	0.13
Pentadecane	0.43	Pentacosane	0.09
Hexadecane	0.36	Hexacosane	0.07

Table 2.2. GC/MS Chromatogram compounds list of mixed plastic waste heated for 1 hr.

Retention time (minutes)	Compound Name	Formula	Retention time (minutes)	Compound Name	Formula
5.33	Octane	C ₈ H ₁₈	35.00	Heptadecane	C ₁₇ H ₃₆
8.43	Nonane	C ₉ H ₂₀	37.66	Octadecane	C ₁₈ H ₃₈
12.07	Decane	C ₁₀ H ₂₂	40.19	Nonadecane	C ₁₉ H ₄₀
15.81	Undecane	C ₁₁ H ₂₄	44.91	Heneicosane	C ₂₁ H ₄₄
19.44	Dodecane	C ₁₂ H ₂₆	47.13	Docosane	C ₂₂ H ₄₆
22.90	Tridecane	C ₁₃ H ₂₈	49.26	Tricosane	C ₂₃ H ₄₈
26.17	Tetradecane	C ₁₄ H ₃₀	51.30	Tetracosane	C ₂₄ H ₅₀
29.27	Pentadecane	C ₁₅ H ₃₂	53.27	Pentacosane	C ₂₅ H ₅₂
32.21	Hexadecane	C ₁₆ H ₃₄	55.16	Hexacosane	C ₂₆ H ₅₄

Table 3.1. GC/MS Chromatogram response/abundance for mixed plastic waste heated for two hours

Compound Name	Response/Abundance (10 ⁷)	Compound Name	Response/Abundance (10 ⁷)
Octane	0.32	Octadecane	0.37
Nonane	2.21	Nonadecane	0.23
Decane	1.72	Eicosane	0.20
Undecane	1.43	Heneicosane	0.19

Dodecane	1.25	Docosane	0.17
Tridecane	0.72	Tricosane	0.16
Tetradecane	0.58	Tetracosane	0.24
Pentadecane	0.54	Pentacosane	0.14
Hexadecane	0.42	Hexacosane	1.84
Heptadecane	0.39	Heptacosane	0.09

Table 3.2. GC/MS Chromatogram compound list of plastic wastes heated for two hours

Retention time (minutes)	Compound Name	Formula	Retention time (minutes)	Compound Name	Formula
5.34	Octane	C ₈ H ₁₈	37.65	Octadecane	C ₁₈ H ₃₈
8.43	Nonane	C ₉ H ₂₀	40.18	Nonadecane	C ₁₉ H ₄₀
12.07	Decane	C ₁₀ H ₂₂	42.60	Ecosane	C ₂₀ H ₄₂
15.80	Undecane	C ₁₁ H ₂₄	44.91	Heneicosane	C ₂₁ H ₄₄
19.43	Dodecane	C ₁₂ H ₂₆	47.13	Docosane	C ₂₂ H ₄₆
22.88	Tridecane	C ₁₃ H ₂₈	49.25	Tricosane	C ₂₃ H ₄₈
26.16	Tetradecane	C ₁₄ H ₃₀	51.28	Tetracosane	C ₂₄ H ₅₀
29.27	Pentadecane	C ₁₅ H ₃₂	53.26	Pentacosane	C ₂₅ H ₅₂
32.20	Hexadecane	C ₁₆ H ₃₄	55.16	Hexacosane	C ₂₆ H ₅₄
34.99	heptadecane	C ₁₇ H ₃₆	56.99	Heptacosane	C ₂₇ H ₅₆

Table 4.1. GC/MS Chromatogram response/abundance of regular diesel

Compound Name	Response/Abundance (10 ⁷)	Compound Name	Response/Abundance (10 ⁷)
Octane	0.17	Octadecane	1.93
Nonane	0.38	Nonadecane	1.80
Decane	0.53	Eicosane	1.52
Undecane	0.82	Heneiconane	1.34
Dodecane	0.85	Docosane	1.16
Tridecane	1.12	Tricosane	0.81
Tetradecane	1.52	Tetracosane	0.62
Pentadecane	2.15	Pentacosane	0.38
Hexadecane	2.23	Heptane	0.17
Heptadecane	2.18	Hexacosane	0.09

Table 4.2. GC/MS Chromatogram compound list of regular diesel from local fuel stations

Retention time (minutes)	Compound Name	Formula	Retention time (minutes)	Compound Name	Formula
5.32	Octane	C ₈ H ₁₈	37.71	Octadecane	C ₁₈ H ₃₈
8.41	Nonane	C ₉ H ₂₀	40.24	Nonadecane	C ₁₉ H ₄₀
12.05	Decane	C ₁₀ H ₂₂	42.65	Eicosane	C ₂₀ H ₄₂
15.80	Undecane	C ₁₁ H ₂₄	44.96	Heneicosane	C ₂₁ H ₄₄
19.44	Dodecane	C ₁₂ H ₂₆	47.17	Docosane	C ₂₂ H ₄₆
22.91	Tridecane	C ₁₃ H ₂₈	49.29	Tricosane	C ₂₃ H ₄₈
26.21	Tetradecane	C ₁₄ H ₃₀	51.32	Tetracosane	C ₂₄ H ₅₀
29.32	Pentadecane	C ₁₅ H ₃₂	53.28	Pentacosane	C ₂₅ H ₅₂
32.27	Hexadecane	C ₁₆ H ₃₄	54.37	Heptane	C ₇ H ₁₆
35.05	heptadecane	C ₁₇ H ₃₆	565.17	Hexacosane	C ₂₆ H ₅₄

Table 5. The main identified hydrocarbons from samples

Chemical name	One hour	Two hours	Diesel
Octane	†	†	†
Nonane	†	†	†
Decane	†	†	†
Undecane	†	†	†
Dodecane	†	†	†

Tridecane	†	†	†
Tetradecane	†	†	†
Pentadecane	†	†	†
Hexadecane	†	†	†
Heptadecane	†	†	†
Octadecane	†	†	†
Nonadecane	†	†	†
Eicosane	—	†	†
Heneicosane	†	†	†
Docosane	†	†	†
Tricosane	†	†	†
Tetracosane	†	†	†
Pentacosane	†	†	†
Heptane	—	—	†
Hexacosane	†	†	†

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