Synthesis of C5-C22 Hydrocarbon Fuel From Ethylene-Based Polymers

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Abstract— The current study tested the hypothesis as to whether or not polyethylene-based plastic can be converted to liquid fuel by thermal catalytic cracking over metal oxide catalysts, including $Ca(OH)_2$, Al_2O_3 , and ZnO. To determine the optimum condition for the highest yield of low molecular weight (C5-C22, MW=100-300) liquid fuel production, the catalyst-to-polymer ratio was investigated at various temperature and reaction times. The initial liquid product obtained from each catalyst was further cracked to yield hydrocarbons of lower molecular mass. The obtained final products were analyzed by gas chromatography-mass spectrometry (GC-MS), high performance liquid chromatography (HPLC), infrared resonance (FT-IR), and elemental analysis (EA). The GC/MS analysis demonstrated that the fuel obtained had constituents ranging from carbon numbers of 5-22. The FT-IR spectra showed the expected functional group and the element analyzer indicated the gross and net heating values of the fuel to be within the range 61-383 MJ/Kg. Comparative analysis showed that $Ca(OH)_2$ has the highest total conversion yield followed by Al_2O_3 and then ZnO.

Index Terms-Catalytic conversion, gasoline, hydrocarbon fuel, metal oxide, plastic, polyethylene, thermal cracking

1 INTRODUCTION

ETHYLENE-BASED polymers are vital in the production of film wraps, plastic bags, electrical insulators, and bottles. The demand for ethylene-based materials is growing fast. World plastic production data shows an increase from 1.7 million tons in 1950 to 280 million tons in 2011 [1].

With increased consumption and disposal of plastic materials, the need to manage plastic wastes is apparent. Plastic waste materials are largely composed of high density and low density polyethylene (HDPE/LDPE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC), polyethylene terephthalate (PET) and acrylonitrile butadiene styrene (ABS). We hypothesize that thermal catalytic conversion of waste plastics will generate low molecular weight hydrocarbon fuel. Our aim is to create an alternative energy source as well as ensure the protection of the environment through effective waste collection and conversion [2], [3]. HDPE and LDPE are identified as suitable plastic waste materials for pyrolysis and are the most commonly found plastic materials littered around the world [4]. Polyethylene is resistant to thermal degradation and catalysts are required to enhance pyrolysis. Pyrolysis of LDPE using a catalyst is reported to have a high conversion rate of 86%

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[4]. Various catalysts, including silica alumina and zeolites, have been reported to give high yields of liquid fuel through the thermo-catalytic conversion, and BaCO₃ is reported to have a low production of wax during the process [5], [6], [7], [8], [9].

We tested our hypothesis further that the liquid collected after catalytic conversion was distilled to produce hydrocarbon fractions that have similar properties as gasoline and diesel. However, various factors affect the conversion of polyethylene-based polymers to liquid fuels. We found that an important factor is the catalyst-to-polymer ratio. Catalyst-topolymer ratio controls yield and optimum liquid hydrocarbon products using various catalysts [5], [10]. The current studies tested catalyst-to-polymer ratio and reaction conditions to maximize hydrocarbon liquid yield. Our data demonstrated that reaction temperature affected the product yield with high temperature (450°C-550°C) to be ideal for total conversion; however, various catalysts accelerated reactions under low reaction temperature (250°C) [4], [5], [10], [11], [12]. The current study tested the hypothesis with the following aims: (1) to determine the catalytic conversion of carbon-carbon bond cleavage of polymers under 200°C and within a 1-hour time frame using several catalysts, including Ca(OH)₂, ZnO and Al₂O₃; (2) to determine the optimum catalyst-to-polymer ratio at which the maximum yield of C6-C16 hydrocarbon can be obtained; (3) to determine distillation by-products from fuel produced from LDPE/HDPE/PET. The by-products in question include terephthalic acid and benzoic acid that are biodegradable. Our data demonstrated that polymer waste can be converted into low molecular weight hydrocarbons, including gasoline and diesel, by thermal catalytic cracking.

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2 MATERIALS AND METHODS

2.1 Reaction Conditions

LDPE packaging material was obtained from the local market and shredded into small pieces. The pieces were weighed and prepared for the pyrolysis. In all pyrolysis experiments, the catalysts used included Al₂O₃, ZnO, and Ca(OH)₂ obtained from Acros Organics, Kermel and BDH Chemicals Ltd., respectively.

The catalytic pyrolysis reaction was carried out in a batch Pyrex round-bottom glass. A fixed amount of the LDPE and catalyst was weighed and placed in the reactor. The glass reactor with known weight of feedstock was placed on a 200-Watt heating mantle and heated to $200 \circ C$. The reactor delivery tube was connected to a condenser leading to the receiving flask. The products from the reaction were in the form of non-condensable gases, condensable oil, wax and solid residues. Condensable oil and wax were collected while the solid residues were left in the bottom of the reactor.

The condensable liquid obtained from catalytic pyrolysis was placed in a fractional distillation apparatus and distilled into various fractions at boiling point ranges of gasoline and diesel. The experiment was performed using catalyst-to-polymer ratios from 1:1 to 1:5.

2.2 Analysis

The total percent conversion was calculated as follows:

Total % conversion = (weight of LDPE waste material – (1) weight of residue) x 100 /weight of LDPE waste material

The liquid percent conversion was calculated as follows:

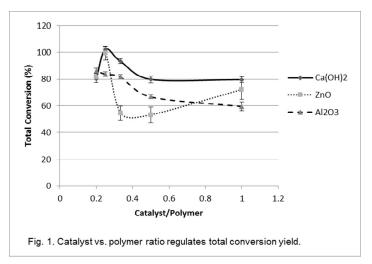
Liquid % conversion = weight of liquid x 100 /weight of (2) LDPE waste material

Thermo Nicolet IR100 FT-IR was used to determine the functional groups in the various distillates recovered after fractionation. Agilent 7890A GC with 5975C inert MSD was used to analyze the components of the fractions. The oven of the GC operated at a pressure of 7.070 psi and had an initial temperature of 40 °C for 10 minutes and was gradually increased to 300 °C at a rate of 10 °C/min.

3 RESULTS AND DISCUSSION

3.1 Effect of Catalyst Ratio

The optimum ratio was determined by testing various catalyst-to-polymer ratios (1:1, 1:2, 1:3, 1:4 and 1:5) for maximum liquid conversion. Experiments showed that increased catalyst downregulated liquid conversion [13]. Catalyst:polymer ratio of 1:4 appeared to yield the optimum total conversion as well as optimum liquid conversion as shown in Fig. 1.



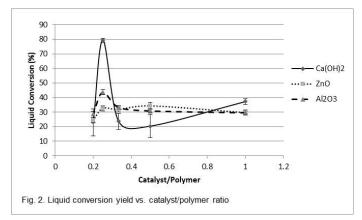
3.2 Fractional Distillation

The first liquid products were separated into three fractions based on boiling point. Two distillates were obtained from each of the parent oils for a specific catalyst type. Table 1 presents the boiling temperature, color, and the respective mass (as a per cent of parent mass) of distillate fractions obtained from the parent oil for each catalyst. Catalyst-to-polymer ratio also affected total liquid conversion yield as shown in Fig. 2.

TABLE 1

DISTILLATES OF PARENT OIL AND THEIR CORRESPONDING MASS PERCENTAGE

Catalyst	Mass of parent oil (g)	Distillate type	Mass of distillate (g)	Mass (%)
Са(ОН)2 21.8		first distillate (90-200 °C) light yellow	10.4	47.7
	21.8	second distillate (200-230 °C) dark yellow	10.5	48.2
Al2O3 11.9	first distillate (30-100 °C) dark yellow	16.3	82.9	
	11.9	second distillate (100-230 °C) dark brown	2.36	12.0
ZnO	10 (first distillate (80-112 °C) light yellow	9.47	79.9
2110	19.6	second distillate (112-230 °C) dark brown	1.92	16.2



FT-IR spectrum analysis showed that the fuel fractions that were obtained from the parent oil catalyzed by Ca(OH)₂ had the following functional groups: cis-RCH=CHR, =C-H (trans), =CH₂ (trans), -OH bending, -CH₃ bend, -C=C-, C_{sp3} -H and C_{sp2}-H (Table 2). Previous reports from others also showed the domination of olefins in liquid hydrocarbons by solid base catalysis [14].

TABLE 2

FTIR SPECTRA OF FRACTIONS FROM Ca(OH)₂ CATALYSIS

Peak number	Wave number (cm ⁻¹)	Functional group
1	3077.95	Csp2-H
2	295 7.65	Csp3-H
3	2925.55	Csp3-H
4	2855.01	Csp3-H
5	1641.01	-C=C-
6	1463.74	-CH3
7	1378.01	-OH bending
8	992.01	-HC=CH ₂ -
9	965.20	-HC=CH ₂ -(trans)
10	909.37	-HC=CH ₂
11	721.88	RCH=CHR (cis)

Analysis from fuel fractions obtained by Al₂O₃ catalysis displayed the following functional groups: C_{sp3} -H, Si-H, C=C, $-CH_3$ bend, -OH bend, cis-RCH=CHR, $-NH_2$ (Table 3).

TABLE 3

groups: C_{sp3}-H, Si-H, -C=C-, -CH₃ bend, -OH bend, cis-RCH=CHR, -NH₂, -HC=CH₂- (trans), (Table 4).

TABLE 4

FTIR SPECTRA OF FRACTIONS FROM ZnO CATALYSIS

Peak number	Wave number (cm ⁻¹)	Functional group
1	3077.13	C _{sp2} –H
2	3013.69	Csp2-H
3	2931.21	C _{sp3} –H
4	2882.77	Csp3-H
5	1648.55	-C=C-
6	1548.96	-NH2
7	1464.90	−CH3
8	1376.35	–OH bending
9	964.27	-HC=CH ₂ -(trans)
10	909.23	-HC=CH ₂
11	721.88	RCH=CHR (cis)

Gas chromatography and mass spectrometry (GC-MS) analysis revealed hydrocarbon chains ranging from C₅-C₁₅ in the first (90-200 °C) and second (200-230 °C) distillate fractions from Ca(OH)₂ catalysis (Table 5). Analysis of first (30–100 °C) and second (100-230 °C) distillate fraction from Al₂O₃ indicated hydrocarbon chains from C5-C16. The fraction obtained from the first (80-112 °C) and second (112-230 °C) distillate of ZnO catalysis contained hydrocarbons ranging from C5-C22.

It was observed that the chromatogram obtained from the 90-200 °C distillate via Ca(OH)₂ catalysis revealed a higher relative abundance of light hydrocarbons over heavier types, while the chromatogram of the second distillate (200-230 °C) showed predominantly heavier hydrocarbons. We attribute this result to the fact that the heavier fractions and higher boiling hydrocarbons, including octadecane and nonadecane, were obtained in the second distillate. Similarly, the chromatograms of the first distillates via Al₂O₃ and ZnO catalysis showed that the relative abundance of the light hydrocarbons was greater compared to the heavier types (Table 6, 7). The second distillates showed high percentage of heavier hydrocarbon chains as expected.

TABLE 5

HYDROCARBON COMPOSITION OF FUEL DISTILLATES BY Ca(OH)2

FTIR SPECTRA OF FRACTIONS FROM AI_2O_3 Catalysis		Compound Name	Compound Formula	Molecular Weight	%
Wave number (cm ⁻¹)	Functional group	1-Decene	C10H20	140.27	9.02
2931.21	C _{sp3} –H	1-Decyne	C10H18	138.25	0.07
2882.77	C _{sp3} –H	1-Dodecyne	C12H22	166.30	0.03
2348.63	Si-H	2-nonene	C9H18	126.24	2.42
1739	C=O	3-nonene	C9H18	126.24	1.93
1557.99	-NH ₂	(E)-Cyclopropane, 1-methyl, 2-	Caller	106.04	1 1/
1460	-CH3	pentyl	C9H18	120.24	1.46
1399.21	-OH bending	(E,Z)-Propylidencyclohexane	C9H16	124.22	0.02
720	RCH=CHR (cis)	7-Formylbicyclo [4.1.0] hep-	0.11.0	104.10	0.07
		tane	C8H12U	124.18	0.07
py carried out on the fr	actional distillates ob-	3, 4-Octadiene	C8H14	110.20	0.02
tained from ZnO catalysis indicated the following functional		3-Dodecyne	C12H22	166.30	0.01
	Wave number (cm ⁻¹) 2931.21 2882.77 2348.63 1739 1557.99 1460 1399.21 720 py carried out on the fr	Wave number (cm ⁻¹) Functional group 2931.21 Csp3-H 2882.77 Csp3-H 2348.63 Si-H 1739 C=O 1557.99 -NH2 1460 -CH3 1399.21 -OH bending 720 RCH=CHR (cis)	Wave number (cm ⁻¹)Functional group1-Decene2931.21Csp3-H1-Decyne2882.77Csp3-H1-Dodecyne2348.63Si-H2-nonene1739C=O3-nonene1557.99-NH2(E)-Cyclopropane, 1-methyl, 2-1460-CH3pentyl1399.21-OH bending(E,Z)-Propylidencyclohexane720RCH=CHR (cis)7-Formylbicyclo [4.1.0] hep- tanepy carried out on the fractional distillates ob-3, 4-Octadiene	Wave number (cm ⁻¹) Functional group 1-Decene C10H20 2931.21 Csp3-H 1-Decyne C10H18 2882.77 Csp3-H 1-Dodecyne C12H22 2348.63 Si-H 2-nonene C9H18 1739 C=O 3-nonene C9H18 1557.99 -NH2 (E)-Cyclopropane, 1-methyl, 2- C9H18 1399.21 -OH bending (E,Z)-Propylidencyclohexane C9H16 720 RCH=CHR (cis) 7-Formylbicyclo [4.1.0] hep- C8H12O tane 3, 4-Octadiene C8H14 C9H14	Wave number (cm ⁻¹) Functional group 1-Decene C10H20 140.27 2931.21 Csp3-H 1-Decyne C10H18 138.25 2882.77 Csp3-H 1-Dodecyne C12H22 166.30 2348.63 Si-H 2-nonene C9H18 126.24 1739 C=O 3-nonene C9H18 126.24 1557.99 -NH2 (E)-Cyclopropane, 1-methyl, 2- C9H18 126.24 1399.21 -OH bending (E,Z)-Propylidencyclohexane C9H16 124.22 720 RCH=CHR (cis) 7-Formylbicyclo [4.1.0] hep- C8H12O 124.18 pt carried out on the fractional distillates ob- 3, 4-Octadiene C8H14 110.20

tained from ZnO catalysis indicated the following functional

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7-methyl-3,4-octadiene	C9H16	124.22	0.09
3-Decen-1-ol	C10H20O	156.27	0.02
(Z)-Ethylidenecycloheptane	C9H16	124.223	0.30
Undecane	C11H24	156.31	9.29
1-hexyl-2-propyl trans-cyclopropane	C12H24	168.32	0.22
2-Tridecene	C13H26	182.35	2.89
Cyclotetradecane	C14H28	196.37	0.36
1-Docosene	C22H44	308.58	0.03
3,7,11-Trimethyl-1-Eicosanol	C23H51O	343.55	0.01
2-Undecene	C11H22	154.29	2.75
1-Hexyl-2-propyl cis-2-octene	C8H16	122.21	0.72
1-butyl-2-pentyl trans- cyclopropane	C12H24	168.32	0.01 -
5-Undecene	C11H22	154.29	0.66
1,12-Tridecadiene	C13H24	180.33	0.10
4-nonene	C9H18	126.24	0.10
9-oxabicylo [6.1.0] nonane	C8H14O	126.20	0.11
13-Octadecenal	C18H34O	266.46	0.34
1-Tridecene	C13H26	182.35	9.53
n-Heptadecanol-1	C17H36O	256.47	0.54
5-Tetradecene	C14H28	196.38	0.29
1-Tetradecene	C14H28	196.38	8.85
2-Tetradecene	C14H28	196.38	3.11
Cyclohexadecane	C16H32	224.43	0.07
n-pentadecanol	C15H32O	228.42	0.01
8-heptadecene	C17H34	238.27	0.01
Dodecyl propyl ester Oxalic acid	C17H32O34	300.43	0.58

TABLE 6

HYDROCARBON COMPOSITION OF FUEL DISTILLATES BY AI2O3

Compound Name	Compound	Molecular	%
Compound Name	Formula	Weight	70
Cis-2-nonene	C9H18	126.24	0.16
4-Nonene	C9H18	126.24	0.07
Cyclopropane, 1-methyl-2-	C9H18	126.24	2.35
pentyl-	C9118	120.24	2.55
(Z)-5- Decene	C10H20	140.27	0.66
4-Decene	C10H20	140.27	0.30
2-Decene	C10H20	140.27	0.19
Cis-3-decene	C10H20	140.27	4.10
2,7-Octadiene	C8H14	110.20	0.30
4-Undecene	C11H22	154.29	0.71
(E)-7-oxabicyclo [4.1.0] hep-	C7H12O	112.17	1.14
tane, 3 methyl-	C/H12O	112.17	1.14
5-Undecene	C11H22	154.29	0.36
2-Undecene	C11H22	154.29	5.29
1-Hexadecanol	C16H34O	242.44	0.53
2-Dodecene	C12H24	168.32	1.78
3,7,11-trimethyl -1-Dodecanol	C15H32O	228.41	1.16
2-butyl-Dodecane	C16H34	226.44	5.25
5-Octadecene	C18H36	252.48	1.02
Trichloroacetic acid, undecyl	C13H23CI3O2	317.68	2.54
ester	C131 123 C13 C2	517.00	2.34
Cis-4-Undecene	C11H22	154.29	0.96
Tridecane	C13H28	184.22	5.71
3-Tetradecene	C14H28	196.38	1.57
5-Tetradecene	C14H28	196.38	0.64
Oxalic acid	C2H2O4	90.03	3.74

1-Decanol	C10H22O	158.28	0.28
2-Tridecene	C13H26	182.35	0.04
Cyclodecane, methyl-	C11H22	154.29	0.01
2,3,5-trimethyl decane	C13H28	184.36	0.72
Cyclotetradecane	C14H28	196.22	0.20
Dodecane	C12H26	170.33	1.21
Cyclotetradecane	C14H28	196.22	1.18

TABLE 7

HYDROCARBON COMPOSITION OF FUEL DISTILLATES BY ZnO

O	Compound	Molecular	0/
Compound Name	Formula	Weight	%
3-Nonen-2-ol	C9H18O	142.24	0.26
2-Nonene	C9H18	126.24	2.41
4-Nonene	C9H18	126.24	0.21
Trans-4-Nonene	C9H18	126.24	1.91
Cyclohexane, 1,3-dimethyl-2-	C9H16	124.22	0.31
methylene	C9H16	124.22	0.31
6-Tridecane	C13H28	184.22	0.51
E-2-Octadecadecen-1-ol	C18H36O	268.48	0.57
Decane	C10H22	142.28	5.04
Cis-4-Decene	C10H20	140.27	1.77
(E)-Trans-4-Decene	C10H20	140.27	1.97
3, 4-Octadiene	C8H14	110.20	0.55
9-Methylbicyclo[3.3.1] non- ane	C10H18	138.25	1.11
Undecane	C11H24	156.31	6.43
(Z)-4-Undecene	C11H24 C11H22	154.29	0.43
Dodecane	C11H22 C12H26	154.29	5.53
	C12H26 C12H24	168.32	5.53 0.74
(Z)-5-Dodecene 8-Dodecen-1-ol, (Z)-	C12H24 C12H24O	184.32	0.74
Tridecane		184.32	
	C13H28		5.39
2-Tetradecene	C14H28	196.38	4.38
Z-5-Nonadecene	C19H38	266.51	1.48
1-Nonadecene	C19H38	266.51	0.01
(Z)-Oxirane	C ₂ H ₄ O	44.05	0.04
Oxalic acid	C2H2O4	90.03	0.10
(Z)-2-Tridecene	C13H26	182.35	0.24
1-Tridecene	C13H26	182.35	0.09
1-Dodecene	C12H24	168.32	0.09
2,5-dimethyl dodecane	C14H30	198.39	4.97
1,9Tetradecadiene	C14H26	194.36	0.14
1-Eicosanol	C20H42O	298.55	7.77
13-Octadecenal	C18H34O	266.46	0.57
7-Hexadecene	C16H32	224.43	3.85
(Z)-Cyclohexadecane	C16H32	224.43	0.25
3-Hexadecene	C16H32	224.43	0.14
Cyclotetradecane	C14H28	196.37	2.84
Octadecane	C18H38	254.49	0.01
Heptadecanoic acid	C17H34O2	270.45	0.01

Other studies employed the use of a batch reactor which converted a mixture of LDPE, HDPE, PVC and PP into light hydrocarbons at low temperature [2], [15], [16], [17]. The use of Fluidized Catalytic Cracking (FCC) units in converting LDPE to fuel as well as improving the quality and yield of conventional petroleum products has also been reported [13], [18], [19]. Chemical processes that make use of fluidized bed reactors and zeolite-based catalysts which were operated iso-

IJSER © 2014 http://www.ijser.org thermally and at ambient pressure have also been able to produce liquid and volatile hydrocarbons [9], [19]. It is expected that the design of various techniques to improve the conversion process which includes lowering reaction temperature and finding optimum polymer vs. catalyst ratios for various catalysts, including calcium hydroxide. The reaction kinetics is another factor that influences the conversion yield.

4 CONCLUSION

The current study tested the hypothesis of whether or not LDPE can be converted to gasoline fuel type via $Ca(OH)_2$, AI_2O_3 and ZnO catalysis. The results show that $Ca(OH)_2$ proved to be the best catalyst, followed by AI_2O_3 and then ZnO $[Ca(OH)_2 > AI_2O_3 > ZnO]$. That is, $Ca(OH)_2$ gave the highest yield (100%) and liquid conversion (79.8%). In addition, the 1:4 catalyst-to-polymer ratio proved to be the optimum ratio for the synthesis of liquid fuel using any of the catalysts applied in the current study. Study of catalyst regeneration is currently underway in order to fully ascertain the possibility of using $Ca(OH)_2$ on an industrial scale.

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