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SCIENTIFIC PAPER

UDC 66.096.5:628.475.043

LIQUEFIED FUEL FROM PLASTIC WASTES USING NITRO CRACKING METHOD WITH REFINERY DISTILLATION BUBBLE CAP PLATE COLUMN

Article Highlights

- Using nitrogen gas in the pyrolysis process gives more yield at low temperature
- Zeolite catalyst accelerates the performance of reactor
- Catalyst increases 10% of pyrolysis oil yield
- Compared with standard diesel fuel, the properties are relatively high in pyrolyzed oil

Abstract

Development and modernization have resulted in an immense increase in the production of all kinds of goods, which indirectly produce waste to the globe. Plastic is one of the materials that produces more waste due to its wide range of applications that is due to its versatility and relatively low cost. In most cases, thermoplastic polymers make up a high proportion of waste and steadily increasing worldwide pollution to the environment. As a result, waste plastics pose a severe environmental challenge due to their non-biodegradable properties and disposal problems. Diverse innovations are being developed to address plastic drawbacks, which can boost the profits of the recycling industry and shrink the world plastic waste landfills. As a part of recycling, the present work is aimed at producing liquefied fuel through the nitro cracking method using a pyrolysis reactor induced with bubble cap plate column with Y zeolite as a catalyst. The liquefied fuel results produced from plastic wastes (plastics bags, plastic bottles, packing materials, and medical plastics) are compared with fuel produced from virgin plastics. The 8% higher pyrolytic oil yield is achieved compared with the results of oil produced without catalyst, and 82% of total waste plastic material is converted into liquefied fuel by the presence of a catalyst. FTIR, GC-MS, and bomb calorimeter characterized the obtained fuel results by adopting the standard ASTM methods, and the results were compared with virgin and waste plastics.

Keywords: pyrolysis, waste plastics, catalyst, LDPE.

Plastics have turned into a part of humans' day-to-day life and provide a remarkable contribution to society. According to the report of Plast India organization, the total polymer consumption is 155000 kilotons, and polymer imports during 2016-2017 were 4200 kilotons, as shown in Figure 1. Also, they have

estimated that the growth of the world plastic consumption rate may increase 9% during 2019-2020. Polyethylene terephthalate (PET), high-density polyethylene (HDPE), low-density polyethylene (LDPE), polyvinyl chloride (PVC), polypropylene (PP) and polystyrene (PS) are majorly used plastics. Based on the report, polyethylene, polypropylene, and low-density polyethylene are widely used in various applications such as grocery bags, water hoses, garbage cans, film, containers, child products, cellular phones, television, automotive and other electronic equipment [1]. These plastics are versatile, hygienic, highly durable, lightweight, and flexible where they can be produced with a simple technique.

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Paper received: 7 September, 2020

Paper revised: 3 May, 2021

Paper accepted: 11 May, 2021

<https://doi.org/10.2298/CICEQ200907014S>

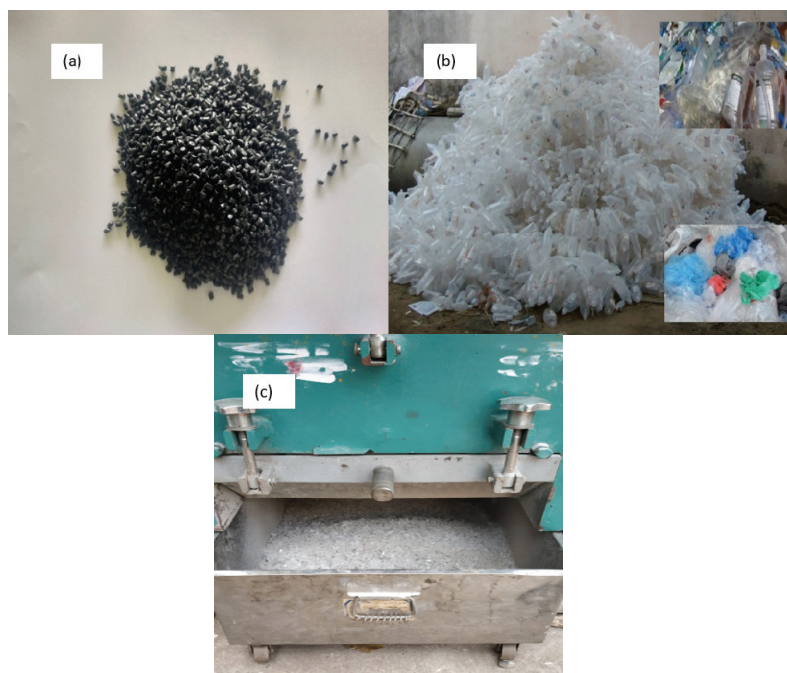


Figure 1. Photographic image: a) virgin LDPE; b) waste LDPE material; c) shredder machine.

The current scenario describes the plastics produced over the past 65 years. Only 12% has been incinerated, and 9% has been recycled. The remaining 79% has ended up in landfills or the oceans [2]. These plastics degrade slowly and release toxic micro-plastics and chemicals into the land and the water. This is a tragedy to the seas because once these pollutants are dispersed, they cannot be completely retrieved. The predicted increase in the consumption of plastics materials over the next decade will increase the efforts of the pressure groups objecting to plastics waste. This objection will tend to encourage the development of recycling and recovery options for plastics. On the other hand, an appropriate waste management strategy is another crucial parameter for sustainable development since plastic waste creates environmental problems [3-4].

Most of the works of literature conducted experiments to convert waste into some other product, especially plastic waste into fuel. Due to the lack of availability of fossil fuels, humankind has to focus on emerging alternative energy sources. The waste-to-energy conversion is investigated as one of the potential methods to deliver the replacement of fossil fuel. The recent trends in plastic waste management and its conversion techniques of plastics to fuel/oil were discussed by [5,6]. They have depolymerized the waste plastics by utilizing the thermal cracking method (pyrolysis). Pyrolysis is a process of thermal degrading of long-chain polymer molecules into shorter, less complex molecules through heat and

pressure. This process can produce a high amount of liquid oil, up to 80 wt.%, at a moderate temperature of around 300-500 °C [7]. Without sulphur and chlorinated polymers in the pyrolysis feed mixture, it does not produce much environmental pollution. The crude oil produced through this process can be used in multiple applications such as furnaces, boilers, turbines, and diesel engines, *etc.* [8]. Additionally, the gaseous byproduct has a substantial calorific value where it can be reused to compensate for the overall energy requirement of the pyrolysis plant.

Detailed analysis of the gas composition of various plastic products also showed and discussed the different reactor types used in the pyrolysis. It provided the data for the assessment of the feedstock recycling process. Furthermore, the process handling is also much more accessible and flexible. An intense sorting is not required because the mixture of different plastics can be fed into the reactor so it is less labor-intensive [9]. Quesada *et al.* examined the plastic film from municipal solid waste into fuel by non-catalytic pyrolysis. Heating rates and residence times had less influence on the yield at high temperatures [10]. At the lowest temperature (450 °C), higher yield is obtained when the time is longer. These results were similar to others found in the literature [11-14].

Manos obtained the pyrolysis technique to extract the oil from domestic waste plastics [15]. Catalyst materials are used to improve the process and product yield from pyrolysis. They reduce the pyrolysis temperature to its optimum condition and increase the

reaction rate and hydrocarbon distribution. The most widely used catalysts are zeolite, FCC and silica-alumina catalyst. Among these catalysts, zeolite catalysts are mainly used in the plastic pyrolysis process. Miandad *et al.* conducted the catalytic pyrolysis process with the help of natural and synthetic zeolite catalysts. They obtained the liquid oil from plastics and reported that catalyst material improved the liquefied oil quality by increasing the light hydrocarbon compounds [16]. Williams *et al.* [17] assessed the processing conditions of different types of plastics and provided the data related to derived oil/wax and gas products from the plastics. Thangaraj *et al.* [18] reviewed the role of various catalytic systems typically used in the transesterification reaction of plastic oils derived from pyrolysis in biodiesel generation.

From the various research carried out by the researchers, after careful studies, the multiple properties of conventional fuels compared to plastic-derived oil showed that the plastic-derived oil can be used as an alternate fuel to serve alternate energy. The pyrolysis process becomes the best alternative for waste management. It converts the waste plastics into a value-added product like liquefied fuel [19]. The present work is aimed to investigate and compare the yield of virgin LDPE and waste LDPE plastic material collected from municipal waste. In this work, liquefied fuel is produced through the nitro cracking method using a pyrolysis reactor induced with a bubble cap plate column using Y zeolite as a catalyst for enhancing fuel yield.

MATERIALS AND METHOD

The experiment was conducted with the help of a fixed-bed pyrolysis reactor setup integrated with a bubble cap plate column. The pyrolyzed oil produced from virgin and waste LDPE plastic materials is characterized by using Fourier-transform infrared spectroscopy (FT-IR), gas chromatography-mass spectrometry (GC-MS) and bomb calorimeter by adopting the standard ASTM methods.

The plastic materials used in this study are commercially available virgin LDPE and waste LDPE plastics collected from various sources such as plastic bags, plastic bottles, packing materials, and medical plastics segregated by their types. The photographic view of virgin, waste plastic, and the shredder machine is shown in Figure 1. Collected plastic wastes were washed, dried and separated. A mechanical shredder was used to shred virgin and waste plastics in the size range of 2-5 mm². The catalyst used in this study is a Y zeolite catalyst supplied by a

commercial catalyst supplier (Sakthi Group, Chennai), and nitrogen gas was used as an inert gas.

The catalyst composition cannot be disclosed due to a non-disclosure agreement with the supplier. The catalyst was stored in a glove box to prevent the absorption of moisture from the atmosphere. The shredded plastics are used in the catalytic pyrolysis process along with the catalyst in the feedstock.

Pyrolysis experiment and procedure

The pyrolysis was carried out in the fixed-bed reactor with the minimum operating temperature of 800 °C with the Y zeolite as a catalyst. The pyrolysis reactor is made up of stainless-steel material which holds a capacity of 5000 grams. Initially, 1000 g of waste LDPE plastic fed into the reactor with the addition of 100 g of Y zeolite. In continuation with the initial stage, 2000 g of plastics and 150 g of Y zeolites were mixed and fed into the reactor under the nitrogen atmosphere. Meanwhile, the reactor was initiated when the temperature reached 350 °C. The pyrolytic gas from the reactor flowed through the bubble cap column to remove wax and ash residue content in the vapor. The vapor outcomes from the pyrolysis reactor were condensed with a water-cooled condenser's help, and the condensed oil was collected in the collector flask. The water temperature used in the condenser was maintained below room temperature with the support of ice blocks. A photographic view of the pyrolysis setup with and without a bubble cap column is presented in Figures 2 and 3, respectively, along with the schematic representation of the pyrolysis reactor setup as illustrated in Figure 4. An FT-IR, Jasco, Japan instrument analyzed the functional groups of pyrolytic oil. It has an operating range of 7800 to 350 cm⁻¹ having a maximum resolution of 0.7 cm⁻¹ and an S/N ratio of 25,000:1. The FT-IR analysis was conducted using a minimum of 32 scans with an average 4 cm⁻¹ IR signal within the frequency range of 400-4000 cm⁻¹.



Figure 2. Photographic view of pyrolysis setup.

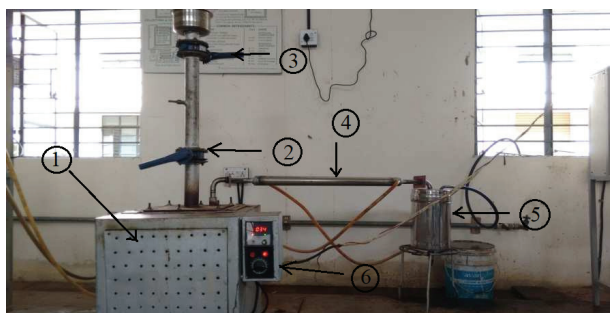


Figure 3. Photographic view of pyrolysis setup with distillation bubble cap column: 1. reactor chamber; 2. tray I; 3. tray II; 4. condenser; 5. collecting tank; 6. temperature controller.

The mass percentage of product composition could then be calculated for % conversion, % oil, % residue and % gas, according to the formula given below.

Conversion of thermal cracking:

$$\text{Conversion (wt.\%)} = 100 \frac{\text{Mass of LDPE} - \text{Mass of residue}}{\text{Mass of LDPE}} \quad (1)$$

Liquid yield:

$$\text{Oil (wt.\%)} = 100 \frac{\text{Mass of oil}}{\text{Mass of LDPE}} \quad (2)$$

Residue (char) yield:

$$\text{Residue (wt.\%)} = 100 \frac{\text{Mass of residue}}{\text{Mass of LDPE}} \quad (3)$$

Gas yield:

$$\text{Gas (wt.\%)} = 100(\text{oil} + \text{residue})$$

The resulting fuel product is based explicitly on density (ρ -30 °C), specific weight (Sg) [20]:

$$\text{API Gravity} = \frac{141.5}{\text{Sg}} - 131.5 \quad (4)$$

RESULTS AND DISCUSSION

The various ratios of waste and virgin plastic materials used in this experiment with the amount of catalyst and the yield outcomes are shown in Table 1. The yield outcome clearly shows that increasing the catalyst percentage after a particular stage will not increase the oil yield. Sharratt *et al.* [21] analyzed the performance of the pyrolysis process with the existence of catalyst that gives more HDPE oil yield at a lower temperature compared with the absence of catalyst materials. Also, the addition of more catalyst does not change the degradation pattern. During the presence of a catalyst, the degradation occurs at lower temperatures and more gradually.

FT-IR analysis

The FT-IR result outcome of liquefied oil extracted from virgin and waste LDPE plastics is shown in Figure 5, and those results are compared with the FT-IR result of standard diesel.

The FT-IR findings confirmed that the presence of hydrocarbons spread in wide ranges mainly distributed with C₆-C₁₆. The paraffinic, olefinic and naphthenic distribution in liquefied fuel produced from virgin LDPE is 58.90, 32.20 and 8.90%. Also, it was 59.70, 31.90 and 8.40% for the liquefied oil produced from waste LDPE materials. The values are nearer to the diesel values.

The FT-IR results of virgin and waste LDPE produced pyrolysis oil (LDPEPO) are compared with the standard diesel FT-IR result and its functional groups taken by [22,23]. It shows the functional group of

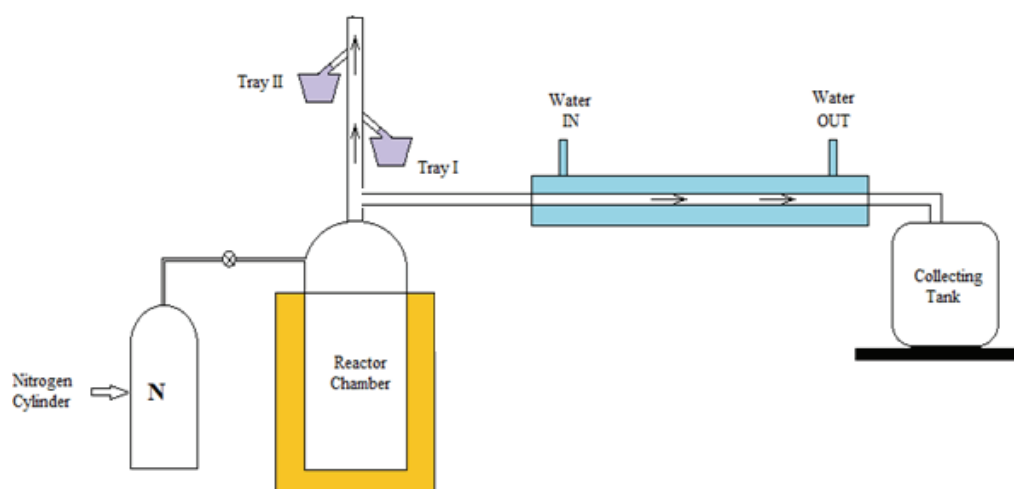


Figure 4. Schematic of thermal cracking pyrolysis integrated with distillation bubble cap column.

Table 1. Catalyst ratios and the pyrolytic oil outcomes

Sample ID	With catalyst					
	Virgin LDPE			Waste LDPE		
	Material weight (g)	Catalyst weight (g)	Pyrolytic oil yield (g)	Material weight (g)	Catalyst weight (g)	Pyrolytic oil yield (g)
A	3000	250	2000	3000	250	1850
B	3000	300	2150	3000	300	1950
C	3000	350	2100	3000	350	2000
D	3000	400	2080	3000	400	1975
E	3000	450	2030	3000	450	1875

Without catalyst			
Virgin LDPE		Waste LDPE	
Material weight (g)	Pyrolytic oil yield (g)	Material weight (g)	Pyrolytic oil yield (g)
3000	1800	3000	1730

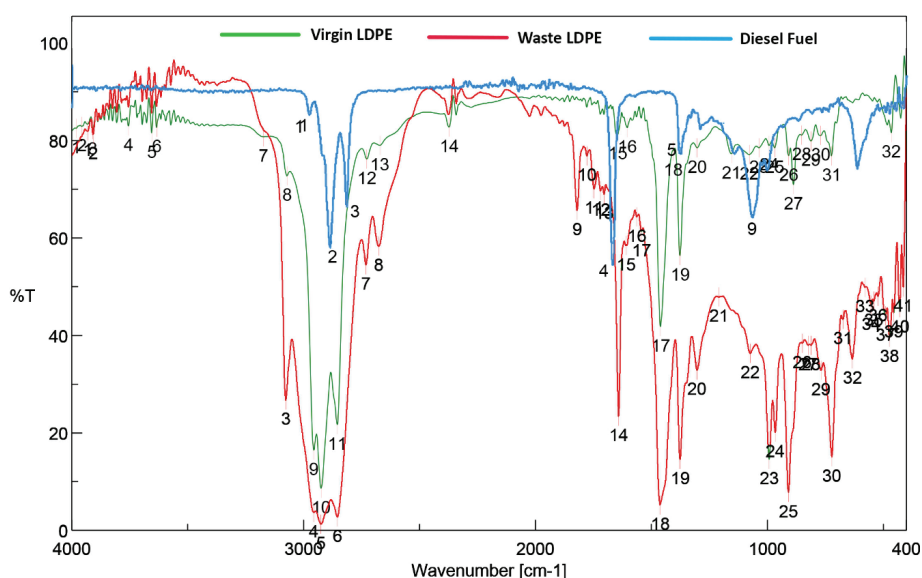


Figure 5. FT-IR result of virgin, waste LDPE and standard diesel fuel.

diesel shows the stretching just below 3000 cm^{-1} implying the absence of sp^3 . The results indicate that most of the hydrocarbons found in the LDPE pyro oils were alkanes, alkenes, and small quantities of other functional groups were present in the spectra of waste LDPE pyro oil. The compounds of all vibration stretching are very near the peak intensities to that of diesel fuel. It is again suggested that all these compounds present in LDPEPO are related to diesel fuel. The properties of fuels were analyzed in Italab private limited, Chennai.

GC-MS analysis

The liquid product review was performed with GC-MS and compounds with peaks of 90% consistency. The amount of carbon in each chain and their preliminary allocations have been calculated. In agreement with the GC-MS data published for comparable products, they were confirmed. Allotted com-

pounds' peak qualities are as shown in Table 2. It is visible from the GC-MS result that the waste LDPE pyrolysis gave very complex mixtures of liquid materials containing many aliphatic and aromatic compounds. Numerous hydrocarbons, alkanes, alkenes, cyclic and halogenated hydrocarbons make up the liquid product from the pyrolysis of waste polyethylene waste. Those results are confirmed with standard diesel results and compared with the literature [24].

Physical properties and characteristics of oils

Pyrolysis-derived fuels are very similar to crude oil. They cannot be used directly as fuel or other energy sources since they must comply with certain standard specifications to ensure combustion engine performance. Plastic pyrolysis products, in the form of naphtha products, are considered as sources of petroleum hydrocarbons.

Table 2. Compounds in pyrolysis oil (GC-MS)

Sl No.	Compound	Chemical Formula	Area, %
1.	Benzene,1-methyl-4-(1methylpropyl)	C ₁₀ H ₁₄ O	20.14
2.	Naphthalene, decahydro-2,6-dimethyl	C ₁₂ H ₂₂	8.37
3.	Benzene,1,2,3,5-tetramethyl	C ₁₀ H ₁₄	20.7
4.	Tridecane	C ₁₃ H ₂₈	47.61
5.	Undecane,2,6-dimethyl	C ₁₃ H ₂₈	12.3
6.	Naphthalene, 1-ethyl	C ₁₂ H ₁₂	2.79
7.	Hexacosane	C ₂₇ H ₅₆	7.32
8.	Heneicosane	C ₂₁ H ₄₄	29.38
9.	Pentacosane	C ₂₅ H ₅₂	7.05
10.	Heptadecane	C ₁₇ H ₃₆	38.6
11.	Tetracosane	C ₂₄ H ₅₀	17.69
12.	Heptadecane	C ₁₇ H ₃₆	16.3
13.	Octadecane	C ₁₈ H ₃₈	16.89
14.	Nonadecane	C ₁₉ H ₄₀	30.71
15.	Eicosane	C ₂₀ H ₄₂	30.33

The fuel characteristics of these products are therefore changed to standard fuel products. Consequently, it needs to be refined; hence the bubble cap plate column that was used in this research. The liquified fuel produced from virgin LDPE and waste LDPE material is collected and used in an engine to characterize the liquified fuel. The analyzed properties of diesel, LDPE, and waste LDPE pyrolytic oil is shown in Table 3.

Characterization of oils by density

Density is one of the parameters which indicates the characteristics of products. According to ASTM D 1298 standard, specific gravity was measured at 60 °F. The relative density (specific gravity) at 60 °F can also be represented by API gravity. The typical densities of transportation diesel fuels at 15 °C (60 °F) are 0.80 to 0.86 gm L⁻¹, or 33-45° API gravity [25]. The density of the virgin and waste pyrolyzed oil extracted from this study shows a lower density parameter compared with standard diesel value analyzed at 30 °C gm/ml by the IS1448, P32 standard.

Characterization of oils by kinematic viscosity

Viscosity is a significant property of oil products that affects the handling or storage, pumping and burning of these products (including the choice of the type of burner used). If the kinematic viscosity value is low, this will affect the quality of the oil fuel, giving it a low heating value [26]. The kinematic viscosity of liquified fuel produced from virgin LDPE and waste LDPE material are calculated at 40 °C according to standard IS1448, P25. Compared with the kinematic viscosity values of standard diesel fuel, it is observed that the kinematic viscosity of diesel is lower than that of virgin and waste LDPE pyrolyzed oils, shown in Table 3.

The high process temperature of pyrolysis accelerates the breaking of the structural polymer bonds. In addition, the termination of the polymer bonds under certain process conditions is imperfect and results in the vapor containing polymers due to pressure and temperature influence in the reactor.

Table 3. Properties of diesel, virgin and waste LDPE pyrolytic oil extracted from the pyrolysis process

Property	Standard	Diesel	Virgin LDPE	Waste LDPE
Density @30 °C gm/ml	IS1448, P32	860	808	802
Kinematic Viscosity @40 °C (cSt)	IS1448, P25	2.107	2.31	2.27
Flashpoint °C by PMCC	IS1448, P21	50	26	24
Fire point °C by PMCC	IS1448, P21	56	32	31
Cetane number	ASTM D976-91	50	69.57	67.64
Gross calorific value Kcals/kg	IS1448, P6	10150	9851	9836
Gross calorific value Kcals/kg	IS1448, P6	10150	9851	9836
Cetane number	ASTM D976-91	50	69.57	67.64

Characterization of oils by calorific value

Calorific or heating value is one of the essential properties of the fuel. The heating value or the energy content affects the power output and fuel economy. The oil produced from virgin and waste LDPE materials is calculated with a bomb calorimeter. The calorific values of each pyrolyzed oil are shown in Table 3. The extracted pyrolytic oils calorific value was nearer to conventionally available fuels. Silvarrey and Phan reported that waste plastic pyrolyzed oil can be an alternate energy source for running engines and turbines. Since the calorific values of liquefied fuel from virgin and waste, LDPE has a nearer calorific value to diesel fuel [27].

Characterization of oils by octane and cetane numbers

The octane number is a measure of the ability of a fuel to “knock” resistance. An engine’s octane requirement varies with compression ratio, geometric and mechanical considerations, and operating conditions. The higher the number of octanes, the more excellent the fuel’s resistance to knocking or pinging during combustion. An essential property of fuel is the anti-knock or octane efficiency, as shown by the research and motor octane numbers (RON and MON). Generally speaking, in a given engine operating situation, the anti-knock quality of a fuel is determined by its octane index $OI = RON - KS$, where K is a constant for that situation, and S is the sensitivity ($RON - MOM$). The higher the index of octane, the greater the fuel’s anti-knock efficiency. K is always believed to be 0.5, so that, on the other hand, $OI = (RON + MOM)/2$ [28], Cetane number (CM) is an empirical parameter associated with an ignition delay time of diesel fuel [29]. The cetane numbers of extracted pyrolyzed oils are shown in Table 3. It is observed that the cetane value for virgin LDPE is high compared with diesel fuel and nearer to the waste LDPE pyrolyzed oil. Hence, waste LDPE oil also generates higher temperature in the combustion chamber and reduced hydrocarbon emission. The virgin and waste LDPE plastics pyrolysis oil and their calorific values and cetane numbers are presented in Table 3.

CONCLUSIONS

The experiment has been conducted to investigate the properties of pyrolytic oil with the addition of a catalyst to produce liquid products through the refinery bubble cap column with the help of nitrogen as an inert gas. The yield % of virgin and waste LDPE plastics differs from the amount of catalyst used in

pyrolysis. The optimum level catalyst will give an 8% higher yield of pyrolytic oil, so the catalyst ratio needs to be calculated before the pyrolysis process.

The characteristics of fuel obtained from plastic, such as density, viscosity, cetane number, ash content and calorific value, have similar properties to those of fossil fuels. From the pyrolysis process, the addition of the catalyst to the reactor will enhance the yield of pyro oil up to a certain level and after that the addition of catalyst will not change the pyrolytic oil yield. The complete conversion happened at 450 °C during the process, and 82% of oil yield was obtained from the total waste plastics.

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NAUČNI RAD

TEČNO GORIVO OD PLASTIČNOG OTPADA DOBIJENO METODOM NITRO-KREKOVANJA U RAFINIRIJSKOJ DESTILACIONOJ KOLONI SA ZVONASTIM PODOVIMA

Razvoj i modernizacija rezultirali su ogromnim povećanjem proizvodnje svih vrsta roba, koje indirektno proizvode otpad. Plastika je bila jedan od materijala koji proizvodi više otpada zbog širokog spektra primene, raznovrsnosti i relativno niske cene. U većini slučajeva, termoplastični polimer čini veliki udeo otpada i stalno povećava zagađenje životne sredine širom sveta. Kao rezultat toga, otpadna plastika predstavlja ozbiljan izazov za životnu sredinu zbog svojih nerazgradivih svojstava i problema sa odlaganjem. Razvijaju se različite inovacije kako bi se rešili nedostaci plastike, kojima se može povećati profit reciklažne industrije i smanjiti svetske deponije plastičnog otpada. Kao deo reciklaže, ovaj rad ima za cilj proizvodnju tečnog goriva metodom nitro-krekovanja korišćenjem piroliznog reaktora tipa kolone sa sa zvonastim podovima i Y zeolita kao katalizatorom. Rezultati tečnog goriva proizvedenog od plastičnog otpada (plastične kese i boce, materijali za pakovanje i medicinska plastika) upoređeni su sa gorivom proizvedenim od nekorišćene plastike. Prinos pirolitičkog ulja je 8% veći u poređenju sa postupkom bez katalizatora, pri čemu se 82% ukupnog otpadnog plastičnog materijala pretvara u tečno gorivo u prisustvu katalizatora. Metode FTIR-a, GC-MS-a i kalorimetrije korišćene su za karakterizaciju goriva prema ASTM standardima, pri čemu je izvršeno poređenje rezultata za nekorišćenu i otpadnu plastiku.

Ključne reči: piroliza, otpadna plastika, katalizator, LDPE.