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Production of liquid fuel by pyrolysis of waste polyolefins

ABSTRACT

Growth in the manufacture of easily accessible oil, the main source of high energy liquid transportation fuels, will not match the projected rate of demand growth, especially in developing countries. Waste polyolefins can replace part of fossil petroleum as a feedstock for liquid fuel. The article considers technical and technological solutions for the utilization of polyolefins, primarily low-density polyethylene (LDPE) and polypropylene (PP) into liquid fuel using slow pyrolysis in a flow reactor at a temperature 590°C and pressure 1.0 MPa. The reactor design allowed the removal of volatiles from the reaction zone in accordance with the Le Chatelier-Brown principle and yield of liquid products from LDPE reached 55.9-93.5%. The thermogravimetric and chromatography-mass-spectrometry characteristics of the obtained plastic derived fuel oil were close to the corresponding characteristics of kerosene and diesel fuels. Gross calorific values of plastic derived fuel samples were also close to those for conventional mineral-derived liquid fuels. Thus, liquid fuels produced by pyrolysis of waste polyolefins can be a credible source of energy for isolated built environment areas with low own energy resources.

Keywords: *liquid fuel, polyolefins, slow pyrolysis, waste plastics.*

1. INTRODUCTION

The predominance of renewable energy sources is a priority task of our time. But the reality is that humanity is not able yet to provide itself with clean energy, therefore, methods of energy generation that reduce environmental pollution or based on waste disposal remain relevant. The incineration of combustible organic municipal wastes for energy generation have undeniable advantages over their landfilling [1]. Energy generation at waste incineration is close to traditional fossil fuels combustion technology, therefore it is devoid of such disadvantages of renewable energy challenges as low operation efficiency and electricity quality problems [2]. It is estimated [3] that more than 300 million tons of plastics are manufactured annually, of which more than 200 million tons later end up in municipal solid wastes and approximately 50 million tons of reused waste plastics are burned annually in one form or another [4]. A plastics calorific values are usually about 30-40 MJ/kg that is typical for hydrocarbons [5]. Thus, they can be used for high calorific source of energy.

Obviously, it is more technologically feasible to burn waste plastics in the forms that are similar to traditional fuels. The mixed plastics could be converted into gases, gasoline, and middle distillate by slow pyrolysis [6] and thereof the polyolefins can be considered as an alternative to fossil petrol feedstock for liquid fuel manufacture.

Recycling of plastic after consumption is a serious problem because of non-uniform of wastes, but it is fundamentally solved for large streams by sorting process [7]. In this case, the streams of individual polyolefins can be processed, usually into granular secondary polymers, which have market demand. Another frequently used technical solution is the oxidation of polyolefins together with other combustible materials in waste incineration plants to generate energy [8, 9]. Even landfilled plastics are considered as urban stocks and resource reservoirs for potential energy recovery [10].

The energy potential of any polymers can be preserved by conversion of polymers into liquid fuels. For example, the process of joint pyrolysis of biomass and solid waste, especially plastics, is attractive as a means of increasing the quantity and quality of fuels obtained [11]. The resulting bio-oil is a high-quality liquid fuel and can be used for energy generation. Moreover, liquid fuel is one of the most technologically effective for processing and convenient for storage form of accumulated

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energy. All the plastics are also polymers and this way thermal pyrolysis is often used an effective way of various plastics conversion into liquid oil. All the plastic waste types used in by researchers [12] were converted into liquid oil at 450 °C and 75 min retention time. As a result of pyrolysis, it is possible to convert polymeric wastes into various types of liquid fuels [12-15]. In this case, as the chemical structure of polymers is predominantly preserved their energy potential is kept and formed energy potential of the fuel. Slow pyrolysis in a periodic reactor of polypropylene (PP), as well as high- and low-density polyethylene (HDPE and LDPE), makes it possible to obtain liquid products corresponding in characteristics to gasoline and diesel fuel [16].

The chemical structure of the samples obtained by slow pyrolysis of LDPE and HDPE was found to be close to fossil kerosene and diesel fuel [17]. The resulting product, plastic derived fuel oil (PDFO), can be considered as a replacement for other liquid fuels derived from fossil raw material. Pyrolysis was carried out in a batch reactor in the abovementioned samples, but even in this case, the process was energetically and economically efficient [18].

The chemical process usually proceeds more efficiently in a flow reactor than in a batch one [19, 20]. The productivity, controllability and energy efficiency of the flow reactor is higher, and the product is usually characterized by more stable properties. Besides, it is known that the pressure during the pyrolysis process has a significant effect both on the rate of the process and products composition. So, with increasing pressure distribution of decomposition products of polyethylene shifted towards the lower molecular weight hydrocarbons [21], since the reaction pressure during thermal decomposition is directly involved in breaking C-C bonds. The experience of using a slow pyrolysis flow reactor with an increased pressure equipped with a screw for the production of PDFO from LDPE and PP is quite limited.

There are currently no technical solutions for low-capacity facilities on the market for pyrolysis processing of LDPE, HDPE, PP and polystyrene into hydrocarbon fuels. Such facilities could be virtually valuable technology for underdeveloped or poor communities with poor waste management infrastructure and considerable remoteness from large urban areas. At the same time, PDFO can be a credible source of energy for isolated built environment areas with low own energy resources.

Therefore, the goal of this research was to convert LDPE and PP into PDFO in bench scale flow rector under pressure and to determinate the properties of obtained liquid fuel in the comparison with traditional fuels.

2. MATERIALS AND METHODS

2.1. Materials

Recycled polymers were used as a raw material. Among them there are low-density polyethylene LDPE and polypropylene PP which underwent primary preparation in LLC “Bumatica” (Perm, Russia). LDPE raw material was obtained from colorless and colored films, that after decontamination, washing and extrusion molding, it has been transformed into recycled material granules 3-5 mm sizes. PP raw material was obtained from bags containers packages, etc., it has been separated from impurities, washed and shredded to pieces of 5-8 mm sizes.

2.2. Equipment

The auger reactors well established themselves for flow slow pyrolysis of polymers because of simplicity of construction and operation [22]. Such reactors are characterized by low energy consumption and by easily controlled residence time of reagents inside the reactor. The modified auger reactor with separated screw extruder, reactor and receiver was proposed and fabricated for the experiments. The appearance of the equipment is shown in Figure 1.

Figure 1. The equipment for pyrolysis of polyolefins

The principle of the equipment operation is illustrated by the diagram shown in Figure 2.

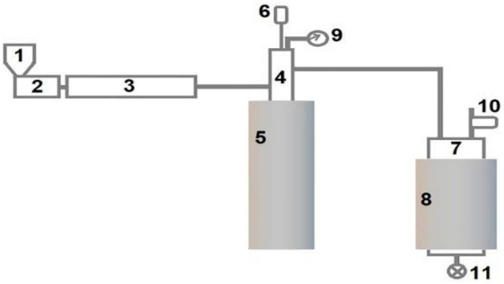

Figure 2. Diagram of the polyolefin pyrolysis equipment: a raw material hopper 1, an extruder 2, a polyolefin plasticization oven 3, a pyrolysis reactor 4, a pyrolysis oven 5, a mixer motor 6, a product receiver 7, a refrigerator 8, a pressure sensor 9, a pressure reducing valve 10, a liquid product drain valve 11

The equipment for pyrolysis of polyolefins works as follows. The polymer granules from the raw material hopper 1 are captured by the extruder screw 2 and passing through the polyolefin plasticizing oven 3 are transferred to the thermoplastic state. The polymer melt enters the pyrolysis reactor 4, where the set temperature is maintained by the pyrolysis furnace 5. As for its hydrodynamic regime, the reactor is close to the ideal mixing reactor due to the continuous mixing of the melt carried out by the mixer motor 6. The low molecular weight hydrocarbons in the gaseous state formed as a result of pyrolysis enter the product receiver 7, which has the room temperature, as it is equipped with an external water cooler 8. Throughout the pyrolysis, the pressure of hydrocarbon vapors in the pyrolysis reactor and in the receiver is the same and controlled by the pressure sensor 9, and is set by the pressure reducing valve 10. After finishing of the experiment, the finished product is drained from the receiver through the liquid product drain valve 11.

The pyrolysis of polymers processing is the following. Raw polymers are directed to the pyrolysis reactor by extruder through polyolefin plasticization oven. The temperature in the pyrolysis reactor is constant because of heating by a pyrolysis oven. Moreover, the pressure in the pyrolysis reactor is also constant because of control by a pressure reducer situated at the receiver. So, the pressure inside the pyrolysis reactor and inside the receiver is equal and it is determinated by the receiver located in cold zone. Such reactor design allows to interrupt the following cracking of polymers after reaching of the hydrocarbon molecule the length acceptable for removing from reactor to receiver.

PP and LDPE pyrolysis of the polymer yield mainly volatiles and the minimum amount of fixed carbon and ash. The ratio of fixed carbon and ash at pyrolysis is known [23] to amount 0.03% and 1.40% for HDPE and 0.16% and 1.99% for PP correspondently. Our preliminary STA analysis shows an insignificant amount of solid residue after pyrolysis, which was 1.2% for raw LDPE and 1.9% for raw PP Therefore, it was decided not to remove the solid residue from the reactor during its operation in order not to complicate the reactor design. A small amount of solid residue was ejected from the bottom of the reactor after the completion of each experiment and this amount corresponded to the calculated amount based on the abovementioned thermal analysis.

The working volume of the reactor is 2.85 liters, reactor`s diameter is 82 mm, height is 540 mm. It was revealed previously at the batch reactor that the optimal conditions for pyrolysis were 1.0 MPa pressure and 590°C temperature, therefore all experiments in the flow reactor were carried out under these conditions.

2.3. Products analysis

Thermogravimetric analysis was provided with a STA 449 F1 device for synchronous thermal analysis (NETZSCH-Gerätebau GmbH, Selb, Germany). The flow of high-purity gaseous argon was used and the heating rate was 20 degrees per minute. The appropriate software NETZSCH Proteus was processed for the resulting data.

The pyrolysis products were determined by chromatography-mass spectrometry on the Agilent Technologies instrument (USA): 7890BGS System chromatograph, 5977AMSD mass spectrometer, HP-1MS capillary column. The GC/MS results were processed using the software package supplied with the instrument for multi compounds organic mixtures. The individual compounds were identified using the NIST 2017 library supplied with the program.

The capillary method of melting point detected was used:

- in the temperature area from 0 to 400°C - using a melting point instrument SMP30 (Stuart Bibby Scientific), UK);
- in the area below 0°C - using the laboratory unit equipped with a liquid nitrogen bath and a LT-300-N electronic thermometer (TERMEKS, Russia).

The samples gross calorific value at a constant volume was determined using an IKAC6000 isoperibol 1/12 calorimeter (Germany).

3. RESULTS AND DISCUSSION

3.1. The staying time effect in the reactor and the physical properties of the products

Attempts to use continuous flow pyrolysis reactor have already been made by a number of authors. So, Al-Salem [24] showed the effectiveness of such reactors use for pyrolysis of mixed polymers excavated from a landfill for conversion into liquid fuel. The yields oil, light and heavy wax were 5.5 and 93.2 wt%, respectively. Continuous microwave-assisted pyrolysis system was used for fuel production from HDPE [25]. Pyro-oil yield was about 73.5 wt%.

The proposed flow reactor and the diagram for organizing the pyrolysis allow to remove irreversibly the light products from the reaction zone, which condense in the receiver. Thus, the target products are continuously and irreversibly removed from the reaction zone as a result of condensation in the receiver. As a result, in accordance with the Le Chatelier-Brown principle

[26, 27], the equilibrium of the cracking reaction of polymer molecules shifts towards the formation of light hydrocarbons C6-C18, and their removal from the reaction zone avoids further crushing of hydrocarbons to low molecular weight products.

The experiments for LDPE pyrolysis at 590°C, 1.0 MPa and various feed rates were carried out in order to confirm this assumption. The products obtained are liquids and oily or waxy products, the properties of which and the reaction conditions are shown in Table 1.

Table 1. Production conditions and melting point of LDPE pyrolysis products

Raw material feed rate, kg/h	Yield of liquid products, wt.%	Product melting point, °C
3.80	93.5	88
2.50	77.6	70
1.74	72.1	50
0.71	55.9	-10

It is obvious that an increase in the reaction time promotes deeper cracking of hydrocarbons and forming products with lower molecular mass. Therefore, the fixed melting point of products naturally decreases with a decrease in the molecular mass of hydrocarbons. In its appearance and melting point, the product obtained at high contact time is a light brown liquid reminiscent of mineral fuel. So, the pyrolysis of polypropylene was carried out under similar conditions, that is, at the feed rate of 0.71 kg/hour, the temperature of 590°C and the pressure of 1.0 MPa. The product yield was 49.4 wt.%, melting point -30°C.

Plastic derived fuel oils obtained under these conditions from LDPE and PP are further designated as PDFO-LDPE and PDFO-PP. Their appearance and raw materials are shown in Figure 3.

Figure 3.Samples of PDFO-LDPE (left) and PDFO-PP (right) and corresponding raw materials

Melting points of PDFO-LDPE and PDFO-PP are close to the melting point of diesel fuel. It can be assumed that the chemical composition of PDFO-LDPE and PDFO-PPis also close to diesel fuel and, in this case, their volatility is possible similar to that of diesel fuel. This assumption was confirmed experimentally. Thermo-gravimetric analysis of organic compounds in an inert atmosphere makes it possible to approximately estimate the properties of various fuels to pass into a gaseous state [28]. The thermo-gravimetric curves of PDFO-LDPE and PDFO-PP are shown in the Figure 4 as also the similar curves of diesel fuel and kerosene.

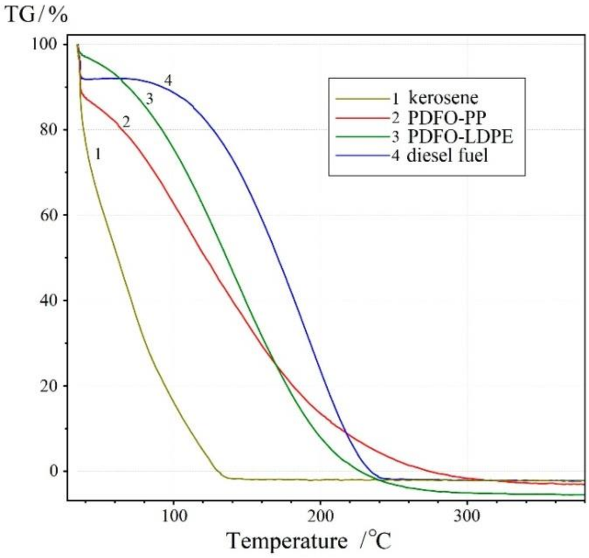

Figure 4. Thermogravimetric analysis of PDFO-LDPE and PDFO-PP pyrolysis products in comparison with diesel fuel and kerosene

TGA, which shows the weight loss of oil at temperature in a constant flow of inert gas, can be regarded as a miniature distillation process and measure the boiling point distribution of the oil. Using TA data the authors [29] distinguish fractions of bio-oil similar as gasoline-like, kerosene-like, diesel-like and fuel oil-like.

Of course, the TGA curves significantly differ from the real distillation curve made in the distillation system. But the joint study of various hydrocarbons by the STA method gives a reference idea of the ratio of volatile components of various liquids. The product of distillation of oil obtained from plastics by pyrolysis is known to be close in its consumer properties to diesel fuel [30], in particular, the rectification temperatures of waste plastic oil derived from pyrolysis of plastic debris are characteristic of the rectification temperatures of the diesel fuel fraction obtained from fossil petroleum. For example, investigation of polyethylene wax non-catalytic pyrolysis at 400-

500°C showed the possibility of liquid fuel producing [31].

In our experiments, such properties of the obtained samples of PDFO-LDPE and PDFO-PP, as melting point and volatility, turned out to be comparable with the corresponding characteristics of standard diesel fuel. The obtained fuel samples PDFO-LDPE and PDFO-PP contain more light fractions of hydrocarbons than in diesel fuel, but less than in kerosene. Therefore, the DTA curves of the PDFO samples are intermediate between the corresponding curves for kerosene and diesel fuel.

3.2. Composition and heat of combustion

Determination of the gross calorific value for the PDFO-LDPE and PDFO-PP samples showed the values of 57.30 MJ/kg and 56.03 MJ/kg, respectively, which are close to those for conventional mineral-derived liquid fuels.

The results of gas chromatography-mass spectrometry demonstrate a predominantly linear structure of PDFO-LDPE hydrocarbons (Figure 5 and Table 2).

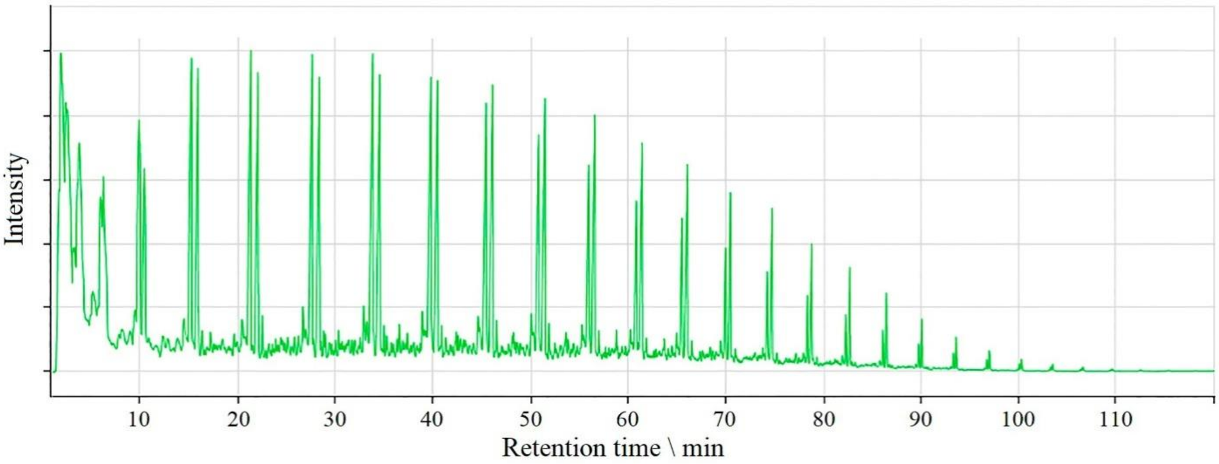

Figure 5.Chromatogram of PDFO-LDPE

Table 2. Results of the sample PDFO-LDPE analysis

Peak, №	Retention time, min	wt, %	Substance
1	1.896	7.77	1-Hexene
2	2.523	3.67	2-Heptene
3	3.788	3.42	3-Octene
4	9.886	3.2	1-Decene
5	15.268	3.46	1-Undecene
6	21.339	3.29	1-Dodecene
7	22.069	2.74	Dodecane
8	27.628	3.14	1-Tridecene
9	28.366	3.1	Tridecane
10	34.546	3.1	Tetradecane
11	40.476	3.16	Pentadecane
12	45.434	2.64	Cetene
13	46.094	3.12	Hexadecane
14	51.47	2.87	Heptadecane
15	56.57	2.52	Octadecane

The data presented reveal two fundamental features of the product. Firstly, the product contains only linear hydrocarbons. Secondly, paraffin hydrocarbons are formed in pair with ethylene hydrocarbon, which has the same number of atoms in the chain as paraffin and a double bond, mainly in the α-position.

One can assume the polymer molecule breakage as a result of cracking occurs with the transfer of hydrogen from one of the formed radicals to another one with the formation of a paraffinic hydrocarbon. The second radical, after leaving the hydrogen radical, forms a double bond in the α-position to compensate for the electronic structure. The PDFO-PP sample contains a significant amount of branched hydrocarbons, unlike the PDFO-LDPE sample, which follows from the results of gas chromatography-mass spectrometry (Figure 6 and Table 3). This fact directly follows from the differences in the structure of the initial polymers. The observed significant amount of branched hydrocarbons is consistent enough with the data in Table 1, since the fuels containing methyl-substituted alkanes have lower pour points than the fuels based on conventional alkanes [32].

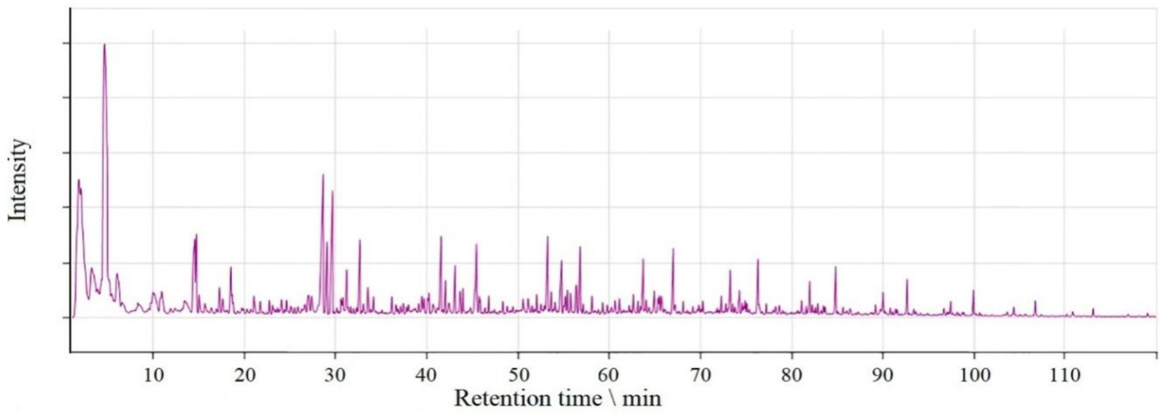

Figure 6.Chromatogram of PDFO-PP

Table 3. Results of the sample PDFO-PP analysis.

Peak, №	Retention time, min	wt, %	Substance
1	1.846	8.02	2-Hexene
2	2.084	8.22	1-Hexene, 2-methyl-
3	3.259	1.94	1-Heptene, 4-methyl-
4	4.7	16.51	2,4-Dimethyl-1-heptene
5	6.048	1.55	2,4,6-Trimethyl-3-heptene
6	14.565	2.64	1-Decene, 2,4-dimethyl-
7	14.759	1.47	1-Dodecene
8	28.64	4.77	1-Pentadecene
9	29.055	1.53	1-Pentadecene
10	29.673	3.38	1-Pentadecene
11	32.669	1.46	1,15-Hexadecadiene
12	41.568	1.62	1-Octadecene
13	45.434	1.79	9-Nonadecyne
14	53.256	1.58	10-Heneicosene
15	54.76	1.36	Henicos-1-ene

The presence of branched hydrocarbons is preferable in liquid fuels because it reduces its melting point and increases the octane number. However, the existing practice of Fischer-Tropsch synthesis leads to the manufacture of mainly ordinary alkanes, which must then be isomerized for their usage as fuel [33].

Another distinctive feature of the obtained product is the absence of significant amounts of saturated hydrocarbons in the mixture. Indeed, in the series of the first fifteen substances with the largest share in the mixture, not a single saturated hydrocar-bon was recorded. And unsaturated hydrocarbons have a double bond mainly in the α-position.

Decomposition of macromolecules with the formation of active centers of the radical type is a stage of thermal destruction initiation. Polypropylene obtained by stereospecific polymerization of propylene on metal complex catalysts usually is obtained with regularly alternating spatially ordered methyl substituents in the chain. Compared to polyethylene, thanks to tertiary carbon atoms, it is more easily attacked by free radicals that detach hydrogen atoms from macromolecules. Chain termination as a rule occurs as a result of a disproportionation reaction, as a result of which terminal vinyl groups are formed. Therefore, polypropylene is less stable to thermal degradation [34].

Obtained products are similar to the mineral fossil fuels according chemical structure. The petroleum products are considered as light products (C1-C4) products of light naphtha (>C5) and heavy products as heavy naphtha (C6-C9), kerosene (C10-C15) and diesel (>C15). Therefore, the received PDFO is possible to use as liquid fuels corresponded to mineral fuels according distillation temperatures, gross calorific value and chemical structure.

4. CONCLUSIONS

The technical solution has been proposed for processing LDPE and PP waste by slow pyrolysis under pressure into liquid fuels similar in their properties to existing industrial liquid fuels obtained from fossil petroleum. The proposed design of the flow pyrolysis reactor makes it possible to remove products corresponding to the chemical structure of traditional liquid fuels from the pyrolysis zone, which, according to Le Chatelier-Brown principle, allows to avoid deep pyrolysis with the production of pyrocarbon. The resulted hydrocarbons, according to GC/MS results, have the chemical structure C6-C18 that corresponding to the traditional liquid fuels. The gross calorific value for the PDFO-LDPE and PDFO-PP samples showed the values of 57.30 MJ/kg and 56.03 MJ/kg, respectively, which are close to those for conventional mineral-derived liquid fuels. Finally, the distillation temperatures of received hydrocarbons are also cloth to the distillation temperatures of fossil fuels. PDFO produced from waste PP and LDPE can be a credible source of energy for isolated built environment areas with low own energy resources.

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IZVOD

PROIZVODNJA TEČNOG GORIVA PIROLIZAMA OTPADNIH POLIOLEFINA

Rast proizvodnje lako dostupne nafte, glavnog izvora visokoenergetskih tečnih goriva za transport, neće odgovarati projektovanoj stopi rasta potražnje, posebno u zemljama u razvoju. Otpadni poliolefini mogu zameniti deo fosilne nafte kao sirovine za tečno gorivo. Rad razmatra tehnička i tehnološka rešenja za korišćenje poliolefina, prvenstveno polietilena niske gustine (LDPE) i polipropilena (PP) u tečno gorivo korišćenjem spore pirolize u protočnom reaktoru na temperaturi od 590°C i pritisku od 1,0 MPa. Dizajn reaktora omogućio je uklanjanje isparljivih materija iz reakcione zone u skladu sa Le Šatelje-Braunovim principom, a prinos tečnih proizvoda iz LDPE dostigao je 55,9-93,5%. Termogravimetrijske i hromatografsko-maseno-spektrometrijske karakteristike dobijenog lož ulja iz plastike bile su bliske odgovarajućim karakteristikama kerozina i dizel goriva. Bruto kalorijske vrednosti uzoraka goriva iz plastike takođe su bile bliske onima za konvencionalna tečna goriva iz minerala. Stoga, tečna goriva proizvedena pirolizom otpadnih poliolefina mogu biti kredibilan izvor energije za izolovane izgrađene sredine sa niskim sopstvenim energetskim resursima.

Ključne reči: *tečno gorivo, poliolefini, spora piroliza, otpadna plastika.*

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