

FORM - 2
THE PATENT ACT 1970
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COMPLETE SPECIFICATION
(See section 10 and rule 13)

Title of invent

Catalytic Conversion of Waste High Density Polyethylene to liquid fuel.

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA – 769008, INDIA, an Indian registered body incorporated under the Registration of Societies Act, (Act XXI of 1860).

The following specification particularly describes and contains the nature of this invention and the manner in which it is to be performed:-

STATEMENT OF INVENTION:

The present invention discloses a simple method for the conversion of waste high density polyethylene (HDPE) to liquid fuel. A stainless steel reactor is used to convert the waste HDPE to liquid fuel in presence of an acid treated kaolin catalyst. This catalyst improves the yield of oil and increases the rate of reaction. The physical properties analysis of the oil obtained in the optimum condition shows that it is a mixture of different petroleum fractions like gasoline, diesel and kerosene.

BACKGROUND OF INVENTION:

Plastic materials encompass a gradually increasing share of the municipal and industrial waste going into landfill. Recycling of plastics has become a predominant subject in today's plastics industry due to the huge amount of plastic wastes and environmental pressures. Development of technologies for reducing plastic waste, which are acceptable from the environmental standpoint and are cost-effective, has proven to be a difficult challenge due to the complications innate in the reuse of polymers. Ascertaining most advantageous processes for the recycling of plastic materials thus leftovers a worldwide challenge in the new century. Plastic materials find applications in agriculture as well as in plastic packaging, which is a high-volume market owing to the many advantages of plastics over other traditional materials. High density polyethylene (HDPE) is the third largest commodity plastic material in the world, after polyvinyl chloride and polyethylene in terms of volume. The demand for HDPE is increasing day by day. The increased demand and production of HDPE has led to accumulation of large amount of its waste due to its low useful life. A thermal approach for dealing with waste plastics is the so-called chemical feedstock or chemical recycling. The most attractive technique for the chemical feed stock recycling is pyrolysis. A number of studies have been reported in which a range of catalysts and reaction conditions have been employed to convert waste plastics into the hydrocarbon liquid using pyrolysis during the past four decades. This is also imitated by a number of pilot and commercial plants processing various types of plastic wastes in the world.

Comprehensive patent search reveals that no patent has been filed in any country in this trend using the waste high density polyethylene and acid treated kaolin clay as catalyst.

OBJECTIVE OF THE INVENTION:

The objective of the invention is to make a simple process for conversion of waste high density polyethylene to liquid fuel. The basic purpose of this invention is to develop a suitable acid treated kaolin catalyst for the degradation of waste HDPE.

SUMMARY OF INVENTION:

Waste high density polyethylene is subjected to thermal and catalytic pyrolysis. Pure kaolin clay and different acid treated kaolin clays are used as the catalysts for conversion of waste HDPE to liquid fuel. The reaction is carried out in a stainless steel reactor in the temperature range of 400-550 °C with an objective to optimize the oil yield, optimize the amount of catalyst. In the catalytic pyrolysis, a mixture of catalyst and the Waste HDPE in different proportion were pyrolysed in the reactor at a desired temperature. The highest yield of liquid product obtained is 51% by weight at 450 °C in thermal pyrolysis. Use of kaolin as catalyst increased the yield of liquid fraction to 58.8%. Nitric acid (optimum concentration 3M) treated kaolin clay further increased the oil yield to 79%. Study of FTIR and GC-MS confirmed the presence of different hydrocarbons (mostly alkanes and olefins) in the oil. The composition of the oil significantly narrowed in presence of kaolin catalyst and also in acid treated kaoline as compared to thermal pyrolysis. The fuel properties of the oil obtained by using 3M concentration nitric acid treated catalyst were analyzed to find its suitability as a substitute of fossil fuel. Kaolin is a cheap clay material and acid treatment technique is very simple process. The catalyst used can be regenerated for further use. So this method is very simple and eco-friendly.

DRAWINGS (if any)

DETAILED DESCRIPTION

The present invention provides a process for the conversion of waste HDPE to liquid fuel using suitable kaolin clay based catalyst. The different acid treated kaolin is prepared by treating kaolin with four different acids (acetic acid, hydrochloric acid, nitric acid and phosphoric acid) and one base (sodium hydroxide) of different concentrations, which is used in the experiment.

The kaolin clay used in this experiment is procured commercially from Chemtex Corporation, Kolkata, India. The modification of kaolin using four different acids and one base of a concentration was carried out by adding 50g of the kaolin clay to 500ml of acetic acid, hydrochloric acid, phosphoric acid, nitric acid and sodium hydroxide solution of different concentrations and refluxing at 110 °C under the atmospheric pressure in a round bottomed flask equipped with a reflux condenser for four hours. The resulting clay suspension was then quickly quenched by adding 500ml ice cold water. The content was then filtered, repeatedly washed with distilled water to remove any residual acid, dried in an oven, calcined at 650 °C for four hours and ground in a mortar pestel to powder form. The untreated sample is referred to as KC and acid treated samples after calcination at 650 °C are referred to as KC (HCl), KC (CH₃COOH), KC (HNO₃), KC (H₃PO₄) and KC (NaOH). The clay samples were characterized by using XRD, XRF, TGA, SEM, BET surface area analyzer, TPD (ammonia), FTIR to understand the effect of acid treatments. The acid treatment of catalyst increases the Si/Al ratio, surface area and pore volume of the kaolin clay sample. The composition and properties of the modified kaolin clay samples are summarised in the Table 1.

Table 1 Analysis of clay.

| Material | Chemical Content (% weight) | | | | | | | | Si/Al Ratio | Surface Area m ² /g | Pore Volume cc/g |
|--------------------------------------|-----------------------------|--------------------------------|-------|-------|------------------|--------|------------------|-------------------------------|-------------|--------------------------------|------------------|
| | SiO ₂ | Al ₂ O ₃ | MgO | CaO | K ₂ O | ZnO | TiO ₂ | V ₂ O ₅ | | | |
| KC | 43.12 | 46.07 | 0.027 | 0.030 | 0.010 | 0.0064 | 0.74 | Nil | 0.82 | 23 | 0.361 |
| KC (HCl) | 47.80 | 37.61 | 0.016 | 0.017 | 0.01 | 0.0064 | 0.26 | 0.001 | 1.122 | 183 | 1.783 |
| KC (HNO ₃) | 44.83 | 41.51 | 0.02 | 0.008 | 0.008 | 0.0064 | 0.23 | 0.002 | 0.95 | 230 | 1.924 |
| KC (H ₃ PO ₄) | 60.42 | 21.88 | Nil | 0.01 | 0.007 | 0.0064 | 0.44 | 0.001 | 2.438 | 81 | 0.658 |
| KC (CH ₃ COOH) | 40.83 | 42.81 | 0.026 | 0.017 | 0.01 | 0.0065 | 0.20 | 0.003 | 0.84 | 70 | 0.504 |
| KC (NaOH) | 56.14 | 29.30 | 0.070 | 0.186 | 0.017 | 0.0064 | 0.12 | Nil | 1.962 | 76 | 0.591 |

The pyrolysis reaction is carried out in a stainless steel reactor set up. The waste HDPE is pyrolysed in the reactor along with different clay catalyst samples using different catalyst to feed ratio (1:4, 2:4, 3:4, 4:4) in the temperature range of 400-550 °C. The oil yield and reaction time for the pyrolysis reaction carried out using different catalysts in optimum condition (catalyst to feed ratio 1:4) is summarized in Table 2.

Table 2 The results of the pyrolysis (Temperature=450 °C, Catalyst: Feed=1:4) using different types of catalysts of optimum concentration (3M concentration)

| Catalyst | Yield of oil in wt.% | Reaction time in minute |
|--------------------------------------|----------------------|-------------------------|
| No catalyst (Thermal) | 51 | 175 |
| KC | 58.8 | 157 |
| KC (HCl) | 69.2 | 135 |
| KC (CH ₃ COOH) | 59.8 | 148 |
| KC(HNO ₃) | 78.7 | 90 |
| KC (H ₃ PO ₄) | 62.9 | 145 |
| KC (NaOH) | 59.4 | 135 |

The properties of obtained liquid fuel are summarized in Table 3. The distillation report of the oil infers that oil is in the boiling range of 58-376 °C, so it is a mixture of different petroleum products like gasoline, kerosene and diesel.

Table 3 Physical properties of liquid fuel obtained by catalytic pyrolysis of waste HDPE using nitric acid (3M concentration) treated kaolin clay

| Tests | Results Obtained | Test method |
|------------------------------------|------------------|---------------|
| Specific Gravity @ 15°C/15°C | 0.7906 | IS:1448 P:16 |
| Density @ 15°C in kg/cc | 0.7900 | IS:1448 P:16 |
| Kinematic Viscosity @ 40°C in Cst | 2.1 | IS:1448 P:25 |
| Kinematic Viscosity @ 100°C in Cst | 1.0 | IS:1448 P:25 |
| Conradson Carbon Residue | Less than 0.01% | IS:1448 P:122 |
| Flash Point by Abel Method | Minus 2°C | IS:1448 P:20 |
| Fire Point | Plus 5°C | IS:1448 P:20 |
| Cloud Point | Plus 12°C | IS:1448 P:10 |
| Pour Point | Minus 1°C | IS:1448 P:10 |
| Gross Calorific Value in Kcal/Kg | 10,567 | IS:1448 P:6 |
| Sulphur Content | 0.05% | IS:1448 P:33 |
| Calculated Cetane Index (CCI) | 66 | IS:1448 P:9 |
| <u>Distillation:</u> | | IS:1448 P:18 |
| Initial Boiling Point | 58°C | |
| Final Boiling Point | 376°C | |

The present invention is further described in the following examples.

EXAMPLE 1

Waste HDPE when subjected to thermal pyrolysis at 450 °C yields 51% by weight of oil in 175 minutes reaction time.

EXAMPLE 2

Waste HDPE when pyrolysed using kaolin catalyst with 1:4 catalyst to waste HDPE ratio at 450 °C yields 58.8% by weight of oil in 157 minutes reaction time.

EXAMPLE 3

Waste HDPE when pyrolysed using 3M concentration hydrochloric acid treated kaolin clay catalyst with 1:4 catalyst to waste HDPE ratio at 450 °C yields 69.2% by weight of oil in 135 minutes reaction time.

EXAMPLE 4

Waste HDPE when pyrolysed using 3M concentration acetic acid treated kaolin clay catalyst with 1:4 catalyst to waste HDPE ratio at 450 °C yields 59.8% by weight of oil in 148 minutes reaction time.

EXAMPLE 5

Waste HDPE when pyrolysed using 3M concentration nitric acid treated kaolin clay catalyst with 1:4 catalyst to waste HDPE ratio at 450 °C yields 78.7% by weight of oil in 90 minutes reaction time.

EXAMPLE 6

Waste HDPE when pyrolysed using 3M concentration phosphoric acid treated kaolin clay catalyst with 1:4 catalyst to waste HDPE ratio at 450 °C yields 62.9% by weight of oil in 145 minutes reaction time.

EXAMPLE 7

Waste HDPE when pyrolysed using 3M concentration sodium hydroxide treated kaolin clay catalyst with 1:4 catalyst to waste HDPE ratio at 450 °C yields 59.4% by weight of oil in 135 minutes reaction time.

CLAIMS

We claim:

1. A process for conversion of waste High-density polyethylene (HDPE) to liquid fuel using modified kaolin clay catalyst at 400-550 °C with 1:4 catalyst to feed ratio.
2. A process according to claim 1, wherein the pyrolysis process is carried out in a reactor made up of stainless steel.
3. A process according to claim 1, wherein the waste HDPE is pyrolysed using nitric acid treated kaolin clay to obtain liquid fuel.
4. A process according to claim 1, wherein the reaction time decreased (rate of reaction increased) in presence of acid treated kaolin catalyst.
5. A process according to claim 1, wherein the yield of oil was 79% by weight using 3M concentration nitric acid treated kaolin clay catalyst at 450 °C with 1:4 catalyst to feed ratio.
6. A process according to claim 1, acid treated kaoline can be regenerated at 750 °C after use and reused with same efficiency.

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Dated this _____.

Title of invent

CATALYTIC CONVERSION OF WASTE HIGH DENSITY POLY ETHYLENE TO LIQUID FUEL

Abstract

The present invention discloses a simple method for the conversion of waste high density polyethylene (HDPE) to liquid fuel. A stainless steel reactor is used to convert the waste HDPE to liquid fuel in presence of an acid treated kaolin catalyst. This catalyst improves the yield of oil and increases the rate of reaction. The physical properties analysis of the oil obtained in the optimum condition shows that it is a mixture of different petroleum fractions like gasoline, diesel and kerosene.