Thermocatalytic cracking of fat from fat boxes with activated red mud

Rachamento termocatalítico de gordura de caixas de gordura com lama vermelha ativa

DOI:10.34117/bjdv6n4-237

Recebimento dos originais: 16/03/2020
Aceitação para publicação: 16/04/2020

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ABSTRACT
In this study, the residual fat from fat boxes from the university restaurant at the Federal University of Pará was used as biomass to produce biofuels by thermocatalytic cracking, using as catalyst the red mud chemically activated with hydrochloric acid solution and thermally at 1000°C. The cracking process of the residual fat with the activated red mud was carried out at a mass ratio of 10% w/w at a fixed temperature of 550°C and the product generated was collected in a fractionated way by the reaction time at 20, 40, 60 and 80 minutes after the cracking reaction starts. Subsequently, the products were subjected to fractional distillation in a 12-stage vigreux column and the condensed fractions were collected according to the gasoline distillation temperature range (45°C-175°C), kerosene (175°C-235°C), light diesel (235°C-305°C) and heavy diesel (305°C-370°C). Considering the effects of temperature, catalyst ratio and acid solution concentration in the chemical treatment of red mud, the characteristics and compositions of the biofuels produced were systematically investigated by physical-chemical and compositional analyzes. The highest yield obtained was 97.46% at point 4 of the collection, also showing the lowest acid index of gasoline at point 4 with 9.4 mgKOH/g.

Keywords: Cracking, Fat boxes, Biomass, Red mud, Fractional distillation.

INTRODUCTION
With the advent of the industrial revolution that started in the United Kingdom in the middle of the 18th century, the global energy demand has also grown exponentially, with the main source of energy being fuels derived from oil, in addition to mineral coal and natural gas.
For decades, oil products were the main sources of energy used in industries and in people's daily lives, however, this resource in the long run is limited in addition to being a potential cause of the greenhouse effect, due to the emission of gases harmful to the environment such as carbon monoxides and sulfur compounds [1].

In order to alleviate or even exhaust these problems, scientific communities in partnership with private and state companies have been investing new technologies in order to obtain renewable sources of fuels, which do not harm the environment and with the lowest economic cost, thus generating a search for innovations and ratification of methodologies for the synthesis of biofuels through technological routes, among which transesterification, esterification, fermentation and thermal transformations such as cracking are being intensively studied [2].

The cracking process of renewable raw materials, which can be a thermal or catalytic process, has as main objective to break the triglyceride molecules found in animal and vegetable oils and fats, at high temperatures, forming compounds with low hydrocarbon chains molecular weight, similar to petroleum derivatives (gasoline, kerosene and diesel), in addition to oxygenated compounds such as carboxylic acids, ketones, aldehydes, monoxide and carbon dioxide. Although the first studies on the cracking process of vegetable oils date from approximately 1890, effective and systematic studies on the subject intensified in the period between the first war and the second world war, in which it was used as a source of obtaining alternative fuels to oil, due to the scarcity of this in the international market [2, 3, 4].

In this context, with several study possibilities, which expand the applicability of several raw materials and catalysts with defined potentials for each region of the country, the present work investigated the feasibility and improvement of the production of biofuels similar to gasoline, kerosene and diesel, via thermocatalytic cracking of the residual fat from the fat boxes of the UFPA university restaurant, using in the process the proportion 10% (w/w) of the catalyst produced from a by-product of the Bayer process, the red mud, treated with a solution of HCl a 1 molar.

2 MATERIALS AND METHODS

2.1 METHODOLOGY

The first stage of the pilot scale process consisted of weighing 20 kg of the residual material on a BALMAK model BK50 scale, then the material was introduced into the cracking reactor (R-01) made of stainless steel and 125 L operating capacity, together with 10% w/w of
catalyst produced from red mud. Subsequently, the cooling water pumping system was activated and the operational process parameters were established on the central panel.

With the stirrer on, the heating process started with the activation of the LPG gas burner, carried out on the control panel, the heating of the reactor (R-01) was pre-established up to a temperature of 550°C and, during the process, the cracked product was collected fractionally for reaction time after the beginning of the condensation of organic liquid products at 20, 40, 60 and 80 minutes in a 30 L stainless steel collection vessel, in which a cogeneration of non-condensable gases as shown in figure 1.

After the collections, the distillation step took place in the form of batch separation operations, in which, initially weighed on an analytical balance, on average 400 grams of the produced PLOs, then the product was packaged in a volumetric flask consisting of borosilicate with a capacity of 1 liter, which was positioned in a thermal forest with 380W of power, in this a Vigreux distillation column consisting of six stages was connected, which was connected to a hull-type condenser and everything, aiming to condense the desired fractions. The liquid fractions were stipulated at temperatures of (45°C-175°C) for gasoline, (175°C-235°C) for kerosene and (235°C-305°C) for light oil and above 305°C for heavy oil. The fractions were collected, weighed, stored and cataloged according to the temperature ranges, for later physical-chemical and compositional analyzes to be carried out.

Figure 01 - Illustration of the Pilot Cracking Unit
3 RESULTS AND DISCUSSION

Analyzing the results after applying the principle of conservation of matter, for the material currents entering and leaving the catalytic cracking process, one can calculate the yields through Equation 1, as shown in Table 1, one can also observe that there was success in the experimental procedure since the input raw materials were converted into PLO, this fact is confirmed by observing the results of the physical-chemical analyzes (Table 2) performed on the post-cracking product.

\[ n = \left( \frac{MPLOs}{MOe} \right) \times 100 \]  

(1)

Where, MPLOs is the mass of organic liquid product that leaves the system, MOe is the mass of crude palm oil that enters the Cracking reaction.

| PARAMETERS                              | RED MUD 10% WITH 1 MOLAR |
|-----------------------------------------|--------------------------|
| Cracking temperature                    | 550                      |
| Fat mass (Kg)                           | 20                       |
| Catalyst mass (Kg)                      | 2                        |
| Cracking time (min)                     | 120                      |
| Starting Cracking temperature (°C)      | 406                      |
| Cracking start time. (min)              | 40                       |
| First collection (°C)                   | 454                      |
| Second collection (°C)                  | 529                      |
| Third collection (°C)                   | 538                      |
| Fourth collection (°C)                  | 490                      |
| Total OLP mass (Kg)                     | 13.15                    |
| Residues (Kg)                           | 2.3                      |
| Mass of gas generated (Kg)              | 4.55                     |
| Residual water mass (Kg)                | 0                        |
| OLP yield (%)                           | 65.5                     |
| Total yield (%)                         | 88.5                     |

Source: Authors

Analyzing the table, it is noted that the experiment carried out with the catalyst showed a OLP yield of 65.5% and an initial cracking temperature of 406°C. Table 2 below shows the results obtained from the physical-chemical analyzes carried out on the organic liquid products after the cracking reaction.
Table 02. Result of physical-chemical analyzes carried out on liquid organic products (OLP).

| PARAMETERS                          | COLLECTION POINTS |
|-------------------------------------|-------------------|
|                                     | 20 min | 40 min | 60 min | 80 min |
| Density g/cm³                        |   0.8201 | 0.8129 | 0.7819 | 0.7798 |
| Viscosity cSt                        |   15.989 | 14.712 |  9.997 |   6.644 |
| Acidity index mgKOH/g               | 149.25  | 118.21 |  25.85 |   19.34 |

Source: Authors

When analyzing the results in table 2, it is noted that the acidity index parameter significantly reduced from 149.25 mgKOH/g to 19.34 mgKOH/g with the course of the cracking process, as the catalyst provided a lower reaction rate formation of oxygenated compounds in the second half of the cracking process. The gradual reduction can also be observed in the viscosity parameter from 15.989 at point 1 at 20 minutes to 6.644 cSt at point 4 at 80 minutes after the start of the reaction. Table 3 below shows the operational parameters used in the fractional distillation experiments.

Table 03. Operational parameters of bench-scale distillation processes.

| PARAMETERS                          | COLLECTION POINTS |
|-------------------------------------|-------------------|
|                                     | 20 min | 40 min | 60 min | 80 min |
| Collect point                       | 401.8   | 404.0  | 402.4  | 401.8  |
| Sample mass (g)                    | 33      | 33     | 32     | 33     |
| Initial temperature (°C)           | 69      | 45     | 40     | 50     |
| Drip (°C)                          | 300     | 302    | 307    | 328    |
| Final temperature (°C)             | 59.91   | 29.34  | 75.52  | 85.70  |
| Mass (45°C - 175°C) (g)            | 35.65   | 28.97  | 62.33  | 68.55  |
| Mass (235°C - 305°C) (g)           | 65.16   | 100.80 | 147.08 | 158.49 |
| Mass (> 305°C) (g)                 | 0.00    | 0.00   | 0.00   | 44.12  |
| Body of water (g)                  | 163.00  | 0.00   | 0.00   | 0.00   |
| Background material (g)            | 218.68  | 217.82 | 97.06  | 22.24  |
| Mass of gas (g)                    | 22.41   | 27.08  | 20.43  | 22.69  |
| Yield fractions (%)                | 40.00   | 39.38  | 70.80  | 88.81  |
| Yield (%)                          | 45.58   | 46.09  | 75.88  | 94.46  |

Source: Authors
Analyzing the data presented in table 3, the behavior of the samples is observed with respect to the initial drip temperatures, relatively low, ranging from the minimum of 40°C to the maximum of 69°C, a behavior that may be related to the presence odd number of short-chain hydrocarbons C5Hn to C9Hn. Another important characteristic that can be highlighted by observing the table is in terms of the mass of gas generated in the distillations, achieving mass values of up to 27.08 grams corroborating the effectiveness of the catalyst with regard to the generation of short chain compounds of non-condensable gases, as well as in the generation of fuels in the temperature range of gasoline, kerosene and diesel, with a fraction yield of up to 88.81% for point 4 (80 minutes after the start of the reaction). Table 4 below shows the physical-chemical characteristics of the distilled fractions of the products obtained from cracking residual fat.

Table 4. Physico-chemical properties of fuels obtained from the distillation of OLP (red mud 10% at 1 molar).

| COLLECTION POINT | FRACTIONS | PARAMETERS |
|------------------|-----------|------------|
|                  |           | Density (g/cm³) | Viscosity (cSt) | Acidity (mgKOH/g) | Index | Refraction Index |
| 1                | (45°C - 175°C) | 0.7554 | 0.7346 | 106.1076 | 1.4 |
|                  | (175°C - 235°C) | 0.861 | 2.322 | 209.5973 | 1.422 |
|                  | (235°C - 305°C) | 0.8441 | 3.69 | 146.8342 | 1.433 |
| 2                | (45°C - 175°C) | 0.7562 | 0.7835 | 271.3491 | 1.409 |
|                  | (175°C - 235°C) | 0.8428 | 2.875 | 160.7319 | 1.424 |
|                  | (235°C - 305°C) | 0.8397 | 4.1281 | 140.2817 | 1.443 |
| 3                | (45°C - 175°C) | 0.7506 | 0.8012 | 17.8849 | 1.416 |
|                  | (175°C - 235°C) | 0.7917 | 1.5184 | 16.3385 | 1.432 |
|                  | (235°C - 305°C) | 0.8034 | 2.3505 | 15.613 | 1.44 |
| 4                | (45°C - 175°C) | 0.7137 | 0.7986 | 20.4917 | 1.418 |
|                  | (175°C - 235°C) | 0.8017 | 1.351 | 13.619 | 1.437 |
|                  | (235°C - 305°C) | 0.8242 | 2.4372 | 11.6054 | 1.449 |

Source: Authors

When analyzing table 4, it can be seen that the acidity index parameter of biofuels showed a marked decrease from the distillation of point 3, which represents the material collected over 40 minutes from the beginning of the cracking reaction, and accentuating the reduction in point 4, where the value of 11.60 mgKOH/g oil was obtained, for light diesel.
It should be noted that the acidity rates of the experiment are high, but tangent, when compared with the values presented by Almeida [1] who cracked the residual fat from fat boxes using red mud 10% w/w as a catalyst, and presented results in the acidity indexes of fuels 70.203 mgKOH/g oil, for light diesel.

Table 5 below shows the results of the chemical composition of the gasoline (45°C - 175°C) obtained, it is noted that the material, presents in its composition a large amount of hydrocarbons with carbons in the range of C8-C15, in the form of paraffins, olefins, naphthenics and aromatics, which are the main components present in the fractions derived from petroleum.

| PEAK | TIME | COMPOUND | FORMULA | AREA(%) |
|------|------|----------|---------|---------|
| 1    | 6.192| 1-Octene | CH3(CH2)4CH=CH2 | 0.99    |
| 2    | 6.430| Octane   | CH3(CH2)6CH3     | 1.92    |
| 3    | 9.280| 1-Nonene | CH3(CH2)6CH=CH2  | 4.34    |
| 4    | 9.488| 4-Nonene | CH3(CH2)4CH=CHCH2CH2CH3 | 0.52 |
| 5    | 9.574| Nonane   | CH3(CH2)4CH3     | 9.57    |
| 6    | 9.755| 2-Nonene | CH3(CH2)4CH=CHCH3 | 1.71    |
| 7    | 10.012| cis-2-Nonene | CH3(CH2)4CH=CHCH3 | 0.72    |
| 8    | 11.151| Cyclopentene. 1-butyl | C6H16 | 0.20    |
| 9    | 11.546| 1-Pentadecyne | HC≡C(CH2)12CH3 | 0.60    |
| 10   | 12.296| Spiro[4.4]non-1-ene | C9H14 | 0.62    |
| 11   | 12.673| 1-Decene | CH3(CH2)6CH=CH2  | 5.14    |
| 12   | 12.973| Decane   | CH3(CH2)4CH3     | 5.30    |
| 13   | 13.153| 4-Decene | CH3(CH2)4CH=CHCH2CH2CH3 | 1.89 |
| 14   | 13.426| cis-3-Decene | CH3(CH2)4CH=CHCH2CH3 | 0.81    |
| 15   | 13.504| 5-Norbornane-2-carboxaldehyde | C9H16O | 0.69    |
| 16   | 13.786| 4.6-Decadiene | CH2=CH(CH2)9CH=CHCH2CH2CH3 | 1.15 |
| 17   | 14.902| Butylibenzene | C6H5(CH2)4CH3 | 0.71    |
| 18   | 16.059| 1-Undecene | CH3(CH2)10CH=CH2 | 8.79    |
| 19   | 16.339| Undecane  | CH3(CH2)10CH3   | 4.71    |
| 20   | 16.511| 5-Undecene | CH3(CH2)10CH=CHCH2CH2CH2CH3 | 16.15  |
| 21   | 16.784| 3-Undecene. (Z) | CH3(CH2)10CH=CHCH2CH3 | 8.00    |
| 22   | 17.227| 1-Pentylcyclopentene | C10H18 | 2.63    |
| 23   | 18.120| 3-Pentyl-1-cyclohexene | C11H20 | 0.76    |
| 24   | 18.242| Pentylibenzene | C6H5(CH2)4CH3 | 0.81    |
| 25   | 19.286| 1-Dodecene | CH3(CH2)12CH=CH2 | 3.03    |
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26  19.550  Dodecane  \( \text{CH}_3(\text{CH}_2)_{10}\text{CH}_3 \)  2.89
27  19.702  4-Dodecene  \( \text{CH}_3(\text{CH}_2)_3\text{CH}＝\text{CHCH}_2\text{CH}_3\text{CH}_3 \)  0.62
28  22.344  1-Tridecene  \( \text{CH}_3(\text{CH}_2)_{10}\text{CH}＝\text{CH}_2 \)  1.75
29  22.590  Tridecane  \( \text{CH}_3(\text{CH}_2)_{11}\text{CH}_3 \)  4.55
30  23.279  1-Ethyl-5-methylcyclopentene  \( \text{C}_9\text{H}_{14} \)  1.81
31  25.234  1-Tetradecene  \( \text{CH}_3(\text{CH}_2)_{11}\text{CH}＝\text{CH}_2 \)  1.12
32  25.455  Tetradecane  \( \text{CH}_3(\text{CH}_2)_{12}\text{CH}_3 \)  0.98
33  26.235  Cyclohexene.1-(2-propenyl)  \( \text{C}_9\text{H}_{14} \)  0.90
34  28.161  Pentadecane  \( \text{CH}_3(\text{CH}_2)_{13}\text{CH}_3 \)  3.59

Source: Authors

Figure 2 below shows the gasoline chromatogram obtained in the distillation process at collection point 1 (20 minutes).

Figure 02. Chromatogram of gasoline at the collection point 1.

Looking at the graph, it is noted the presence of several peaks indicative of a high amount of substances present in the analyzed sample, totaling 34 significant peaks and that the highest concentration of peaks with retention time varies between 10 and 20 minutes.
4 CONCLUSIONS

Based on the development of this work and considering the analyzes contained in this study, it is possible to conclude that it is possible to use an environmental liability without added value, the residual fat from retention boxes, as a source of raw material in the thermocatalytic cracking reaction with 10% w/w of catalyst produced from red mud, confirmed by obtaining a good yield in PLO, reaching 65.5%. It is also necessary to ratify the potential of the catalyst in terms of providing the generation of hydrocarbon molecules of short and medium chains proven by the good yield in fuel fractions with up to 88.81% of products obtained in fractional distillation.

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