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PRODUCTION OF GAS OIL COMPONENTS FROM WASTE FATS

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The modern-minded man has discovered that it is necessary to substitute a part of the fossil-derived energy sources with renewable energy sources to cover the energy demand of mobility, which sustains and accelerates the human society and economy. Nowadays, the transportation sector tries to achieve this through the development and utilisation of bio-derived motor fuels. In terms of Diesel-engines the biodiesel has been utilized in great volumes already, which is made from triglycerides via esterification (fatty-acid-methyl-ester, FAME). The FAME or biodiesel, due to its molecular structure, has some unfavourable properties. Therefore, it was necessary to develop a new generation of bio-derived motor fuel for Diesel-engines. The most promising product of these efforts is the bio gas oil, which is a mixture of n- and i-paraffins and obtained via hydroconversion of triglycerides. These compounds are the best components of conventional gas oils, too. Nowadays, mainly different vegetable oils are used as triglyceride source, but for the hydroconversion any feedstock with high triglyceride content can be used (e.g. brown greases of sewage works, used cooking oils, animal fats, etc.). The waste feedstocks can be especially beneficial. Hence, during the experimental work, our aim was to investigate the possibilities of the production of bio gas oil and bio gas oil containing gas oils on waste fat basis via the hydroconversion of waste rancid lard itself and as a 50% mixture with gas oils. We applied a $CoMo/Al_2O_3$ catalyst in sulphide and in non-sulphide state for our experiments. We studied the effects of the process parameters (temperature: 300–380°C, pressure: 40–80 bar, LHSV: 1.0–2.0 h^{-1}, H_2/feedstock ratio: 600 Nm^3/m^3) on the quality and yield of the products. The obtained main product fraction at the process parameters (360–380°C, 60–80 bar, LHSV: 1.0 h^{-1}, H_2/feedstock rate: 600 Nm^3/m^3) found to be favourable by us which were met the valid diesel gas oil standard EN 590:2009 + A1:2010 without additivation, except for its cold flow properties.

Keywords: lard, hydrogenation, renewable, diesel gas oil, animal fat

Introduction

The modern society and economy are increasingly dependent on mobility. Mobility of people and products is usually realized using vehicles. Their number is growing with each day and almost every one of them (>95%) operates with fossil derived fuels. At the same time, the limited amount and the unequal distribution of the available oil reserves is a source of international tensions, because most countries rely on import. Besides, there are more and more vehicles, which means more and more intense environmental pollution. For this reason, in the whole world, research has been started for the development and utilization of cleaner and more available energy sources. Among these alternative fuels, there are the renewable, agricultural-derived fuels from biomass and amongst them the bio-derived motor fuels (agro-motor fuels), which are the primary bio-derived motor fuels of the present and the near future.

Because of environmental considerations and its intense rely on import energy sources, the European Union treats the research of agriculture derived, renewable energy sources with great attention and urges their utilization in greater volumes [1–4], besides attending to the agricultural production, too.

In this context, the Union literally declared the necessity of the production of bio motor fuels and their extensive utilization [1] and allows the blending of bio-components (in the case of diesel gas oil this means maximum 7v/v biodiesel) to conventional fuels. In addition, the Union treats the effort to expand the feedstock supplies of bio motor fuels with great importance too [2].

The actual objective of the European Union is to raise the average share of the renewable energy sources to 20% until 2020. The average content of renewable fuels, particularly, must be raised to 10% in all automotive fuels in the European market until 2020 [1–4]. However, it has been proposed recently that the share of the motor fuels derived from feedstocks that can also be used in food processing could be maximum 5%.

Before the worldwide economic crisis, the tendency of the automotive fuel market in the European Union *(Figure 1)* showed a probable increase in the demand [5] for diesel fuels and this tendency will restore despite the feasible spread of hybrid vehicles. Therefore, the research and development of the agriculture derived bio motor fuels are coming to the front.

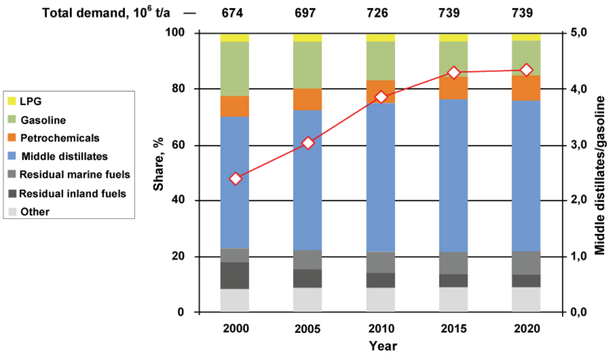

Figure 1: The tendency of the fuel market of the EU
(before the economic crisis)

The research and development of the agriculture-derived bio motor fuels, therefore, is extremely important.

Bio-derived motor fuels of Diesel-engines

The primordial fuel of the Diesel-engine was a vegetable oil (peanut oil), thus for the sake of satisfying the continuously increasing gas oil demand, vegetable oils and their different percentage mixtures with gas oil are attempted to be utilized [6]. The main components of the vegetable oils (and animal fats, etc.) are the triglycerides, which are esters of a polyvalent alcohol, the glycerine, and natural fatty acids (the carbon number of the chain is always paired and they contain unsaturated bonds in a different measure) with different carbon numbers *(Figure 2)*.

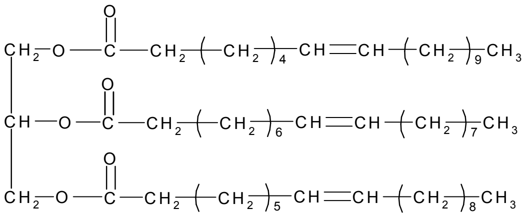

Figure 2: Typical triglyceride molecule

However, the differences between the physical and application properties of these compounds are not suitable to simply replace the conventional gas oils. Therefore, it is necessary to convert them with different conversion pathways. These conversion pathways can be:

- thermal and
- catalytic pathway.

In practice, the more important is the latter one.

Biodiesel

Nowadays, the agriculture derived bio motor fuel and bio blending component that are produced and utilized in the greatest volume is the FAME (fatty-acid-methyl-ester) or biodiesel. This is made by the catalytic esterification of vegetable oils and other fats [7–10].

However the technologies producing biodiesel and the product itself also have numerous disadvantages.

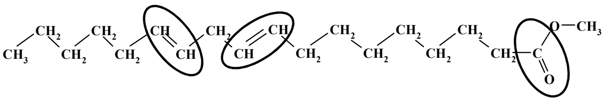

Figure 3: Typical FAME chemical structure and the reactive sites in the molecule

The disadvantages are caused by the chemical structure, which can be seen in *Figure 3* [10, 15, 21]:

- high unsaturated content (causing bad thermal, oxidation, and thus storage stability),
- high water content (corrosion problems)
- sensitivity to hydrolysis (poor storage stability),
- methanol content (toxic),
- reactive OH-group (corrosion of coloured metals),
- low energy content that results in greater fuel consumption (~ 10–15%),
- unfavourable cold properties (cold-start and pulverizing, CFPP).
- high production costs compared to the applicational value,
- limited feedstock supplies, etc.

Bio gas oil

The most suitable for the utilization in Diesel engines and the most valuable compounds of the fossil derived gas oils are the normal- and iso-paraffins with high cetane number and with good cold flow properties. Therefore, intense research has started to produce products with a similar chemical structure on triglyceride base, which do not have the disadvantages of the already utilised biodiesel because of their different (more stable) chemical structure and thus they can be blended to conventional fuels with no limitations [11–24].

One of the alternatives to produce such a product rich in iso-paraffins on triglyceride base is the catalytic hydrogenation *(Fig. 1)* and if necessary, their isomerisation [25–27].

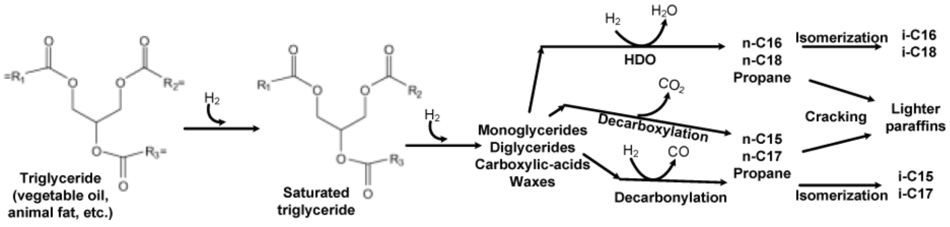

Figure 4: The reaction pathway of the bio gas oil production (R_1, R_2, R_3: carbon chains with C_{11}-C_{23} carbon number)

Through the reaction pathway, in the first step the hydrogenating of the unsaturated bonds of the triglycerides takes place, then deoxygenating reaction occurs by three different pathways: decarboxylation, decarbonylation, and hydrodeoxygenation (reduction, HDO). As the next possible step in the process, isomerisation reactions can occur, of which measure depends on the applied catalyst and process parameters. Cracking reactions may occur in the course of the whole process [11–24].

The product of the reaction is the so called bio gas oil, which is a mixture of n- and i-paraffins in the gas oil boiling point range, made with specific catalytic hydrogenating process of raw materials with high triglyceride content (vegetable oils, animal fats, used frying oils, brown greases of sewage works etc.) [11, 13, 15, 21].

To produce bio gas oil and products containing it, there are different technological methods *(Figure 5)*.

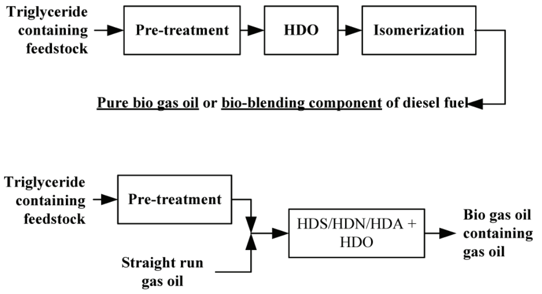

Figure 5: Possible technological solutions to produce bio gas oil

It is possible to pre-treat the triglyceride containing feedstock in a pre-treater reactor and then hydrodeoxygenate it, in other words, convert it in a second HDO reactor as a stand-alone unit. The obtained product in this technology is rich in normal-paraffins, has very high cetane number (90–105), but besides it, the product needs to be isomerised after the separation to improve its poor cold flow properties (18–26°C) [14, 20–21, 25, 27]. The bio gas oil obtained with such a technology can be blended to deeply desulphurized gas oil stream and thus gas oil with bio component content can be made. Beyond that, through blending the pre-treated triglyceride containing feedstock to a straight run gas oil stream and process this feedstock mixture in an existing (or slightly modified) desulphurization plant, bio-component containing gas oil can be obtained *(Fig. 2)* [15–19, 24].

As we mentioned before, a wide range of publications deal with the examination of the conversion of triglycerides per se, or the so called co-processing. However, these articles deal mostly with the hydroconversion of vegetable oils (sunflower oil and rapeseed oil mostly). The bio gas oil – as we already mentioned – can be produced from animal fats and any kind of fats and oils beside vegetable oils, too. This is important, because the increasing degree of the cultivation of oilseeds takes the land from the cultivation of food and fodder, and thus it can have an influence on the food prices. However, as a by-product or waste of the food industry and the agricultural sector, streams rich in triglycerides (rendered fats from slaughterhouse by-products and animal carcasses) forms, which can be applied for the production of bio gas oil, and thus they do not affect the food prices.

In this paper, we investigate the hydroconversion of rancid lard, which can adumbrate the possibility of the application of such animal derived feedstocks.

Experimental

During the experimental work our aim was to investigate the possibilities of bio gas oil and bio gas oil containing gas oils on waste fat basis via the hydroconversion of waste rancid lard in itself and as a 50% mixture with gas oils. We investigated the application of a CoMo/Al_2O_3 catalyst in sulphide and in non-sulphide state. In the course of the experiments, we studied the effects of the process parameters on the quality and yield of the products and in addition the utilisation possibilities of the main product.

Experimental equipment

The experiments were carried out in experimental equipment (tubular reactor of 100 cm^3 active volume capacity) in continuous mode [15, 17, 19, 21].

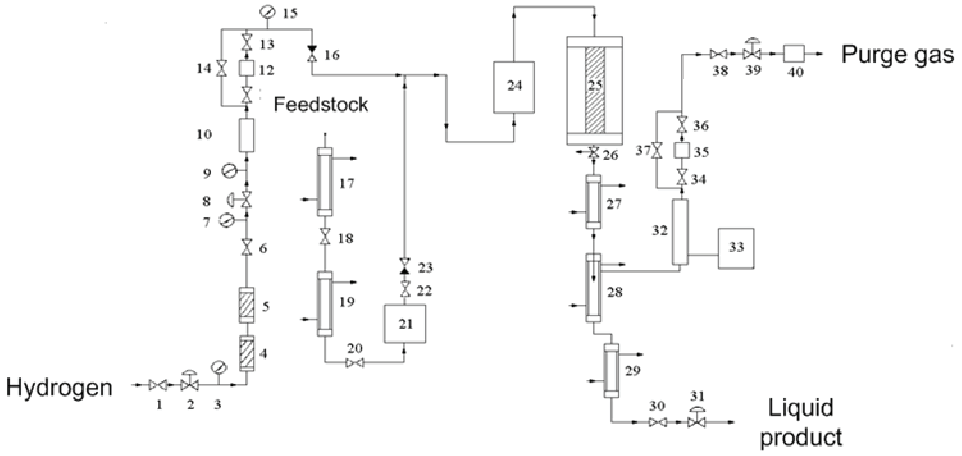

Figure 6: Experimental equipment

(Legend: 1, 6, 11, 13, 14, 18, 20, 22, 26, 30, 34, 36, 37, 38: throttle valves; 2, 8, 31, 39: controlling valves; 3, 7, 9, 15: manometers; 4: deoxygenizer; 5: gasdrier; 10, 28: gasfilter; 12: gasflow measurer; 16, 23: non-return valve; 17: holding burette; 19: feeder burette; 21: pump; 24: preheater; 25: reactor; 27, 29: chillers; 28: separator; 33: pressure register; 35: pressure controller; 40: gas-meter)

Applied feedstocks and catalyst

As the base stock of the heterogeneous catalytic hydrogenating experiments we used lard (fatty acid composition is in *Table 1*) of Hungarian origin and a deep desulphurized gas oil stream (properties in *Table 2*) – derived by MOL Plc. – obtained from Russian crude. As feedstock we used pure lard, pure gas oil, and their mixtures of 50%.

Table 1: The typical fatty acid composition of the applied lard feedstock

Fatty Acid	Lard
C14:0	1.19
C16:0	21.35
C16:1	2.04
C18:0	11.54
C18:1	45.40
C18:2	12.50
C18:3	0.78
C20:1	1.06
C22:2	0.67

*CX:Y, where
X: carbon number of the fatty acid,
Y: number of the unsaturated bonds in the fatty acids.

Table 2: The heteroatom and aromatic content of the feedstocks

Properties	Gas oil	Lard
Boiling point range, °C	197.8–383.2	-
Sulphur content, mg/kg	5	20
Nitrogen content, mg/kg	<1	61
Aromatic content, %	22.5	0.0
Polyaromatic content, %	2.4	

We investigated the application of a $CoMo/Al_2O_3$ catalyst in sulphide and in non-sulphide state in the course of the experiments. In the first case, the catalyst had been sulphided "in-situ", and the sulphur content of the feedstock was adjusted to 1000 mg/kg with the use of a sulphur containing chemical (dimethyl-disulphide, which is easily degradable in the investigated process conditions) to preserve the sulphide state of the catalyst.

Process Parameters

The process parameters were based on previous experimental results, considering both the physical and chemical properties of the feedstock. The series of the experiments were carried out at the following process parameters:

- temperature: 300–380°C,
- pressure: 40–80 bar,
- LHSV: 1.0–2.0 h^{-1},
- H_2/feedstock ratio: 600 Nm^3/m^3.

Analytical methods

The properties of the feedstock and the products were specified according to the specifications of the valid EN 590:2009 + A1:2010 diesel fuel standard, and with standardised calculation methods. The obtained liquid organic product's composition was identified by high temperature gas chromatography [15, 25].

Obtaining the main product fraction

The fractionating of the product mixture was carried out as it can be seen in *Figure 7*. In the course of the experiments, the product mixture was separated into gaseous and liquid phases in the separator unit of the experimental equipment. After separating the water from the obtained liquid product mixture, we separated the light, C_5-C_9 hydrocarbon products by distillation up to 180°C from the organic liquid phase. The fraction above the boiling point of 180°C was separated to gas oil boiling point range main product (C_{10}-C_{22} hydrocarbons up to the boiling point of 360°C) and to residual fraction by vacuum-distillation.

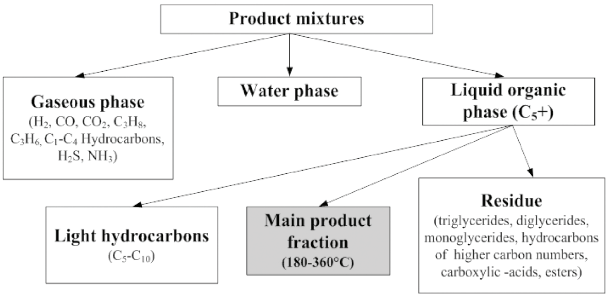

Figure 7: The method of the product fractionating

All product yields are based on the amount of the feedstock.

Results and discussion

The gaseous phase, besides hydrogen, contained carbon-oxides formed during the deoxygenation, propane originating from the triglyceride molecule, hydrogen sulphide and ammonia formed in the course of heteroatom removal, and a very small amount of lighter hydrocarbons (C_{4-}) originating from the hydrocracking reactions. With increasing the temperature, the yield of the gaseous product slightly increased because the cracking reactions came to the front and the deoxygenation, desulphurisation, and aromatic saturation reactions took place increasingly.

The yield of the main product fractions increased with increasing temperature *(Figure 8–12)* in the case of both sulphide and non-sulphide state catalyst, which refer to the increasing conversion of the feedstock triglyceride content.

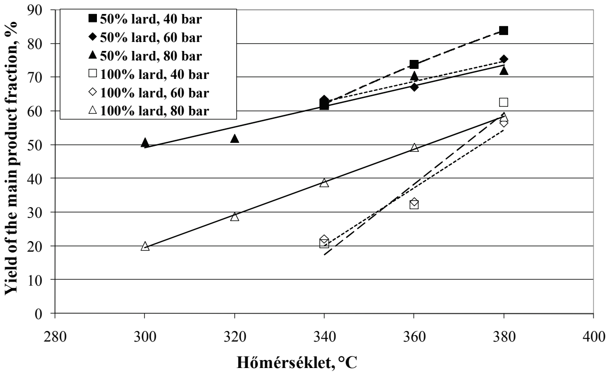

Figure 8: Yield of the main product fraction as a function of temperature and pressure
(catalyst: non-sulphided CoMo/Al_2O_3; LHSV = 1.0 h^{-1}; H_2/feedstock ratio: 600 Nm^3/m^3)

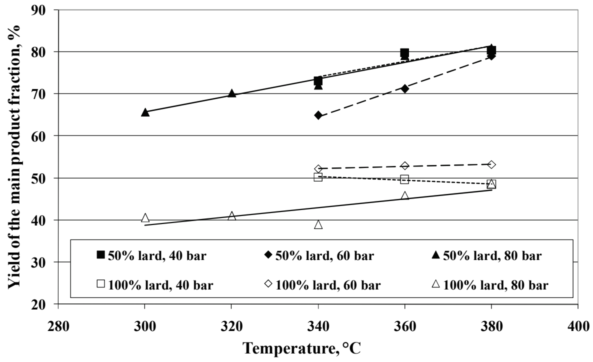

Figure 9: Yield of the main product fraction as a function of temperature and pressure
(catalyst: sulphided CoMo/Al_2O_3; LHSV = 1.0 h^{-1}; H_2/feedstock ratio: 600 Nm^3/m^3)

As a function of the pressure *(Fig. 8, 9)* – in the investigated parameter range – there were no clear trends; in the case of sulphided catalyst and pure lard feedstock, the higher pressure (80 bar) seemed to be favourable, but in the case of mixture feedstock, the lower pressure seemed to be favourable; in the case of non-sulphided catalyst and pure lard feedstock, the lower pressure (40 bar) seemed to be favourable, but in the case of mixture feedstock, the pressure of 60 bar seemed to be favourable.

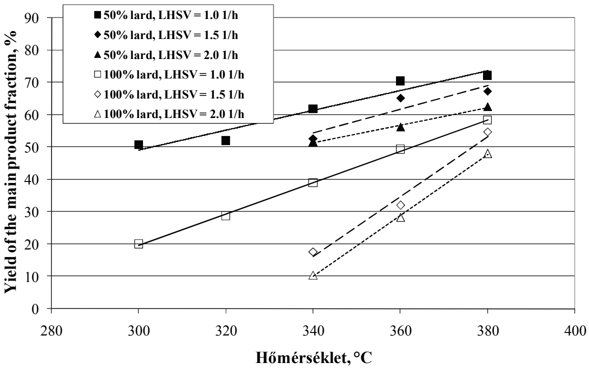

Figure 10: Yield of the main product fraction as a function of temperature and LHSV
(catalyst: non-sulphided CoMo/Al_2O_3;P = 80 bar; H_2/feedstock ratio: 600 Nm^3/m^3)

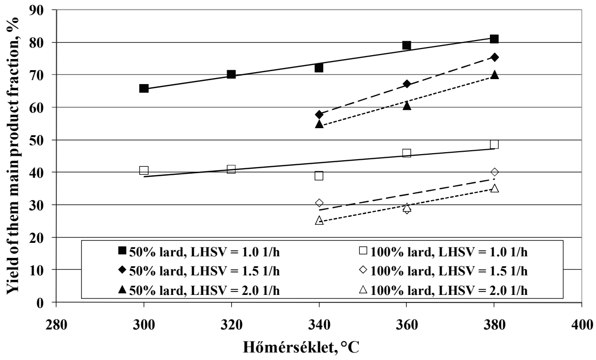

Figure 11: Yield of the main product fraction as a function of temperature and LHSV
(catalyst: non-sulphided CoMo/Al_2O_3;P = 80 bar; H_2/feedstock ratio: 600 Nm^3/m^3)

Decreasing the liquid hourly space velocity favoured the triglyceride conversion to paraffins in the case of both feedstock and the state of the catalyst, so the yield of the main product fraction increased with decreasing the liquid hourly space velocity *(Fig.* 10, 11*)*. This was caused by the higher retention time of the reactants on the active sites.

Based on the yields of the main product fractions in the cases of both states of the catalyst *(Fig. 12)*, we determined that in the case of the sulphide state catalyst, the yield of the main product fraction was higher, but not even in this case was the conversion of the triglycerides full, because the yield of the residual fraction was still significant *(Figure 13)*.

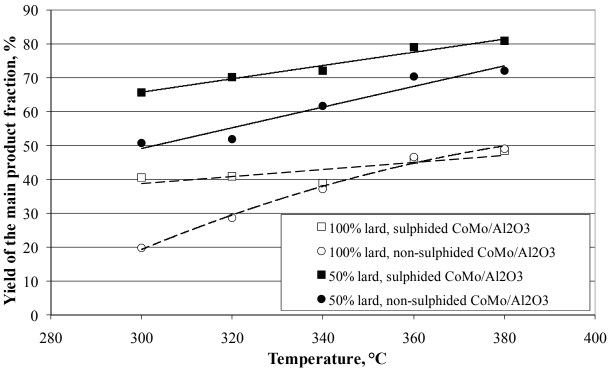

Figure 12: Yield of the main product fractions as a function of the temperature in the case of different feedstock and states of the catalyst
(P: 80 bar; LHSV: 1.0 h^{-1}; H_2/feedstock ratio 600 Nm^3/m^3)

The decreasing of the yield of the residual fraction *(Fig. 13)* with increasing the temperature also provides information about the increasing conversion of the triglyceride content of the feedstock, but at the strictest process parameters, the yield of the residual fraction was still significant.

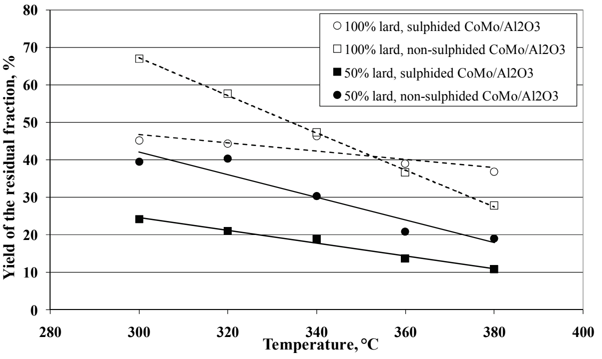

Figure 13: Yield of the residual fractions as a function of the temperature in the case of different feedstocks and states of catalyst
(P: 80 bar; LHSV: 1.0 h^{-1}; H_2/feedstock ratio 600 Nm^3/m^3)

Figure 14 provides information about how the different deoxygenating reactions (mechanisms) take place and about the relationship between them. In the course of the deoxygenation of the natural – and so containing fatty acids with conjugated carbon numbers in their carbon chains – triglycerides, during the hydrodeoxygenation (HDO) reaction pathway paraffins with the same carbon numbers as the fatty acids form next to water. In the course of decarboxylation-decarbonylation (DCO) reaction pathways, paraffins with one less carbon numbers as the fatty acids form next to carbon oxides. The HDO/DCO ratio of the hydroconversion pathways, which is favourable for the yield of the main product fraction, was higher in the case of non-sulphided catalyst than in the case of the sulphided catalyst.

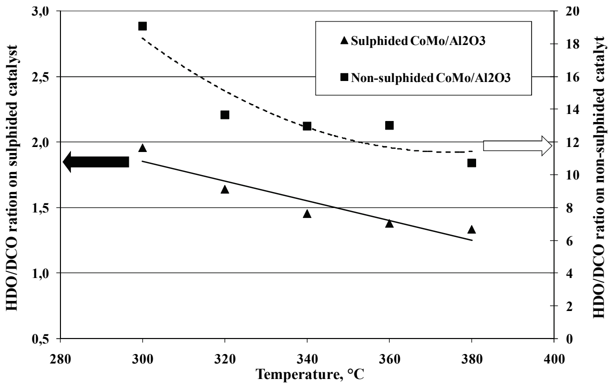

Figure 14: The change in the ratio of the different routes of oxygen removal as a function of the temperature
(P: 80 bar; LHSV: 1.0 h^{-1}; H_2/feedstock ratio: 600 Nm^3/m^3)

The isoparaffin content of the main product fractions (high isoparaffin content is favourable for the good cold flow properties) was the highest in the case of the non-sulphided state $CoMo/Al_2O_3$ catalyst *(Figure 15)*, because of the high isomerisation activity of the non-sulphided CoMo bimetallic system [25].

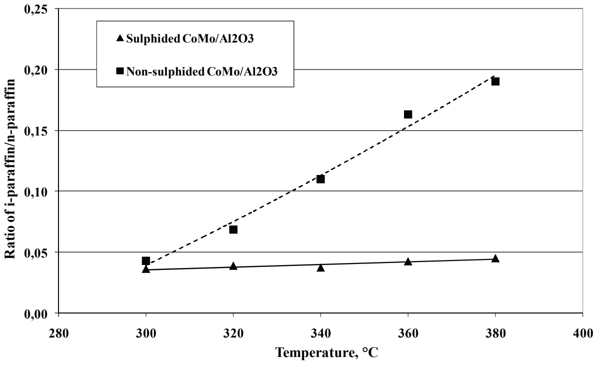

Figure 15: The change in the iso- and normal-paraffin content of the products as a function of the temperature
(P: 80 bar; LHSV: 1.0 h^{-1}; H_2/feedstock ratio: 600 Nm^3/m^3)

Conclusion

In this paper we shortly presented the necessity of the research and development of the bio-derived motor-fuels, the already known bio-derived fuels of Diesel-engines, and the bio gas oil or bio gas oil containing gas oil producing technologies, respectively. During the experimental work, our aim was to investigate the possibilities of bio gas oil and bio gas oil containing gas oils on waste fat basis via the hydroconversion of waste rancid lard in itself and as a 50% mixture with gas oils on a CoMo/Al_2O_3 catalyst in sulphide and in non-sulphide state.

The experimental results adumbrate the industrial application of different animal derived (waste) fats, with which the feedstock supplies can be expanded, for the production of bio derived motor fuel components to expand the feedstock supplies. The properties of the feedstock and the products were specified according to the specifications of the valid EN 590:2009 + A1:2010 diesel fuel standard, and with standardised calculation methods. In the case of mixture feedstock and sulphide state CoMo/Al_2O_3 catalyst, the yield of the main product fraction (81%) was higher than in the case of non-sulphided state CoMo/Al_2O_3 catalyst (72%) at the strictest process parameters. However, in both forms (i.e. sulphided and non-sulphided) of the applied catalyst, the yield of the unconverted residual fraction (boiling point >360°C) was significant. The cause of this was that the oxygen content of the lard is high, about 11%. Besides this, it contains compounds (N-compounds) that strongly adsorb to the catalytically active sites, and thus decrease the possibilities of the deoxygenating reactions to take place.

The HDO/DCO ratio of the hydroconversion pathways which is favourable for the yield of the main product fraction was higher in the case of non-sulphided catalyst than in the case of the sulphided catalyst. The iso-paraffin content of the main product fractions (high iso-paraffin content is favourable for the good cold flow properties) was the highest in the case of non-sulphided state CoMo/Al_2O_3 catalyst, because of the high isomerisation activity of the non-sulphided CoMo bimetallic system. The properties of the main product fractions obtained at these process parameter combinations meet the valid diesel gas oil standard EN 590:2009 + A1:2010, except for their cold flow properties (CFPP). However, these products could be excellent diesel gas oil blending components because of their very low sulphur and nitrogen content, and decreased aromatic content, and because their high cetane number is ensured by the n-paraffins forming during the triglyceride conversion. The cold flow properties of the products can be improved by catalytic hydroisomerisation of the n-paraffins then with additives. Thus, a high quality bio-motor fuel or bio-derived blending component containing Diesel motor fuel can be obtained which meets the valid diesel gas oil standard.

Briefly, we determined that considering the yield of the main product fraction the sulphided state CoMo/Al_2O_3 catalyst is more preferable for the hydroconversion of lard. In the case of non-sulphided state catalyst, the main product fraction contained a higher amount of i-paraffins, which are beneficial from the point of view of cold flow properties. The experiments were carried out on a “once flow” reactor system. Therefore, it may be a possibility for increasing the conversion to separate the residual fraction from the product and recycle it to the reactor. To corroborate, further experiments and tests should be carried out.

Acknowledgement

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