Production of biocomponent containing jet fuels

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Abstract: - Recent demands for low aromatic content jet fuels have shown significant increase in the last 20 years. This was generated by the growing of aviation. Further than quality requirements were more aggravated in front of jet fuels. This was generated by the more severe environmental regulations and the increasing requirements. Nowadays reduced aromatic hydrocarbon fractions are necessary for the production of the jet fuels with good burning properties, what is contributing to the less harmful material emission.

In the nearly past the continously severed properties of gasolines and diesel gas oils, they will severe the properties of jet fuels too. So now some people study the possibilities of produce low aromatic and low sulphur content in heterogen catalytic way. There is only a few publication, that handle with investigation of biocomponent containing jet fuel production.

So we studied during our experimental work hydrogenation of sunflower oil containing kerosene fraction on a $CoMo/Al_2O_3$ heteroatom remover and Pt/HZSM-22/Al_2O_3 isomerizer and partially cracking catalyst system. We studied the yield, analytical and application properties of the jet fuel boiling point range hydrocarbons. At the joint application of the chosen catalysts, we found process parameter combinations, that resulted jet fuel boiling point range products, that have excellent flaming properties (smoke point: 37 mm) and good low temperature properties (crystallization point: <-53°C). This contains clear burning iso-and n-paraffins in high concentration, that are not limited for quantity by standards.

Key-Words: - aromatic content, biocomponent, crystallization point, kerosene, jet fuel, vegetable oil

1 Introduction

Recent demands for low aromatic content jet fuels have shown significant increase in the last 20 years (Figure 1.) [1, 2]. This was generated by the growing of aviation. Compared to the beginning of this period, the 2% of the front of the reviewed session approaches 15%, if we calculate in the point of passanger kilometers the drived passages with vehicles, buses, railroad and jets. Further than quality requirements were more aggravated in front of jet fuels. This was generated by the more severe environmental regulations and the increasing quality requirements. Nowadays reduced aromatic hydrocarbon fractions should be used necessary for the production of the jet fuels with good burning properties too, what is contributing to the less harmful material emission [1,3,4].



Fig 1.: Quantity demand of fuels (EU-27) [1]

It can not leave from focus, that aviaton generates only 2% of the CO_2 emission of the world. This value can grow only for 3% to 2050 [6, 7]. Moreover it generates 12% of the CO_2 emisson of the full transportation section. For comparison the public way transport generates 76% of the CO_2 emission [5, 8, 9].

One of the greatest problems are the jets, that flies at one time more than 1500 kilometers, because aviation produce the 80% of greenhouse gases. Because at the end of the passage they let the emergency fuel out, so they rising the forming of the greenhouse effect. But there are no any alternatives for bridging solution of these distances in the transport section [10, 11]. The harmful material emission is available in this case with fuels with good biodegradational property. From the different hydrocarbons biodegradation of n- and i-paraffins is the best [6].

In the nearly past the continously severed properties of gasolines and diesel gas oils, they will severe the properties of jet fuels too. So now some people study the possibilities of produce low aromatic and low sulphur content in heterogen catalytic way [2,3,8,9].

In aviation by safety aspects the resistance is higher in front of alternative fuels, compared to the alternative fuels in road transport. This is generated by the extraordinary conditions of aviation (low temperature, great distances with one take-off). Further more alternative fuels must be compatible with the conventional fuels, with do not make problem in the logistics of airports. Of course alternative fuels must be compatible with the actual jet engines too. By this causes aim can be to develop a fuel, what is available as itself for aviation [12].

Present days the jet fuels are produced from different crude oils. For the reason of the growing demand it can take into count more feedstock sources, using them it can be produce environmentat-friendly (low sulphur- and aromatic content), preferable application technically fuel (Figure 2.) [1-7, 12].



Fig 2.: Classification of jet fuel production possibilities

During the production of jet fuels must be effort to produce high hydrogen/carbon ratio component, so production of n- and i-paraffins, and cycloparaffins. Isoparaffins have the lowest freezing point, they burn – together with n-paraffins – clean. The cycloparaffins have lower hydrogen/carbon ratio, but their energy content rises the energy content of n- and i-paraffins. Together with isoparaffins, they improve the low temperature properties, this is one of the most important quality property. So, aromatic hydrocarbons must be saturated, or possibility of their formulation must be limited. Hydrogen/carbon ratio of these compounds is lower, so as a consequence they have lower energy content, moreover their freezing point is unfavourable (Table 1.) [12-18].

Crude oil based jet fuels contain heteroatomic (S, N, O) compounds too. Nowadays their quantity is reduced (at the most strict case <10 mg/kg) toward harmful material emission [12, 19, 20].

The role of producing jet fuels from natural triglycerides in a catalytic way will be more important in the near future (Figure 3.) [14]. During the hydrogenation, the formed normal- and isoparaffin hydrocarbons have suitable energetical and low temperature properties [8].

In the future, beside the hydrogenation of triglycerides, two other processes operating with renewable source feedstock can get a role; one produces motor fuels with transformation and hydrogenation of lignocelluloids, while the other is the Fischer-Tropsch synthesis, which processes synthesis gas from biomass, and which is applied in our present days (Figure 4. and 5.) [9, 15, 21].



Fig 3.: Jet fuel production from triglycerides



Fig 4.: Jet fuel production from lignocelluloids

TABLE I

Carbon		Boiling	Heating	value	Freezing point, °C	
number	Compound	point, °C	MJ/dm ³	MJ/kg		
	n-hexyl-benzene	258	32.8	39.5	-42	
C	n-hexyl-cyclohexane	254	36.5	45.0	-52	
C_{12}	n-dodecane	264	33.2	44.1	-10	
	4-methyl-undecane	205	33.2	44.1	-68	
	n-heptyl-benzene	262	33.4	39.7	-40	
C	n-heptyl-cyclohexane	260	36.6	44.9	-47	
C_{13}	n-tridecane	268	33.6	44.1	-6	
	3-methyl-dodecane	213	33.5	44.1	-60	
	n-octhyl-benzene	284	33.9	39.8	-39	
0	n-octyl-cyclochexane	280	35.5	43.5	-20	
C_{14}	n-tetradecane	279	33.8	44.1	6	
	5-methyl-tetradecane	222	33.8	44.1	-51	
C ₁₅	7-methyl-tetradecane	238	33.7	44.0	-48	
C ₁₆	5-methyl-penthadecane	273	33.7	44.1	-31	
C ₁₇	2-methyl-hexadecane	280	33.6	44.0	-23	

BOILING POINTS, HEATING VALUES AND FREEZING POINTS OF AROMATICS, CYCLOPARAFFINS, I- AND N-PARAFFINS WITH THE SAME CARBON NUMBER



Fig 5.: Jet fuel production with Fischer-Tropsch synthesis during isomerizer hydrocracking

Some company handle with investigation of produce jet fuels from synthetic crude oil, produced with Fischer-Tropsch synthesis. During this process carbon number of product mixture is good controllable, so synthetic jet fuel can be developed. On figure 6. is plotted gas chromatographic hydrocarbon group distribution of jet fuels, produced with Fischer-Tropsch process of different companies [12]. These products are very favourable, because they do not contain aromatics, but at the same time isoparaffin content of products is significant. This means the mentioned favourable properties. It must be note, with application of Sasol IPK process, which is processing coal, differently from other GtL technologies, cycloparaffin content of product is significant, what is favourable than nparaffins, by its low temperature properties [12].



Fig 6.: Distribution of hydrocarbons in case of different Fischer-Tropsch processes

2 Experimental

The aim of our experimental work, was to investigate production possibilities of biocomponent containing, low sulphur and aromatic jet fuels. During this, we studied transformation of different sun flower oil containing kerosene fractions. Inside this we studied the effect of process parameters to the yield of liquid products, and to the quality properties. Further more, our aim was to study the kerosene boiling point range paraffin mixture using as jet fuel blending component.

2.1 Experimental apparatus and test method

The heterogeneous catalytic hydrogenation experiments for aromatic saturation were carried out in a 2 reactor contained system which contained all of the important apparatus and units that can be found in a reactor loop of an industrial hydrogenation plant. The simplified process flow diagram of the apparatus is shown in Figure 7. The effective volume of reactors was 100-100 cm³. Table 2. contains the conditions of the catalytic experiments.

The applied process parameters							
Temperature, °C	320-380						
Pressure, bar	35, 50						
H_2 /hydrocarbon volume ratio, Nm^3/m^3	250-600						
Liquid hourly space velocity, cm ³ /cm ³ h	0.8 - 2.0						

TABLE II



Figure 7.: Experimental apparatus (1 reactor; 2 preheater; 3 oxygentransformer; 4, 5 gas dryer; 6,10 gasfilter; 7 gasflow meter;

8 gasflow meter; 9 dropholder; 11 compressor; 12, 13 feedstock vessel; 14 pump;

15, 18 cooler; 16 separator; 17 level detector; 19 controlling valve; 20 blocking valve; non-return valve; 22 pressure indicator; 23 feedstock dryer)

At the choosing of process parameters we considered the followings: the studied CoMo/Al₂O₃ catalyst can desuplhurize the kerosene fraction itself on 320-340°C, 35 bar pressure, 2.0 h⁻¹ liquid hourly space velocity and 250 Nm³/m³ H₂/HC ratio under to 10 mg/kg. But the same time at choosing of temperature it must be considered, in case of the 10-20-30-40% vegetable oil containing kerosene the oxygen content of feedstock changes between 1.1-4.4%. We studied the value of pressure, near the mentioned 35 bar, on 50 bar. Application of lower pressure than 35 bar is not suitable, because total desulphurization of kerosene do not work. Investigation of effect of liquid hourly space velocity is expedient only lower value than 2.0 h^{-1} based on the same causes, the higher heteroatom content of feedstock mixtures (this caused by the blending of high oxygen content sunflower oil). Based on preexperimental results detailed investigation of $0.8-1.2 \text{ h}^{-1}$ range seems to be fnm3 avourable. We increased the H2/feedstock volume ratio in case of feedstock mixtures 400-600 Nm³/m³ feedstock, to assure enough quantity of hydrogen to the hydrogen consumer reactions. In case of pure sunflower oil hydrogenation we chosen 600 Nm3/m3, to assure higher hydrogen quantity with ca. 40% than the theoretical value. We carried out the experiments constant active catalyst and in continuous mode.

We mixed H_2 gas to the liquid product mixture, that came out the first reactor, after the removal of H_2S containing hydrogen rich gas, to hold the designed H_2 /feedstock ratio.

We separated product mixtures from the sunflower oil containing feedstock hydrogenation experiments reached to gas phase, water phase and organic phase (Figure 8.). In the separator of experimental apparatus, the separated gas phase contained carbon-dioxide, carbon-monoxide, that forms from the triglyceride molecules, hydrogensulphide, ammonia, that form from the sulphur- and nitrogen content of feedstock beyond hydrogen, moreover as side products (valuable side product), other light hydrocarbons (C_1-C_4) from cracking reactions. The liquid product mixture from separator contains water, hydrocarbons and oxygen containing compounds. After the separation of water, we reached the light fraction (mixture of C5-C9 hydrocarbons) with distillation of organic phase. After this we reached the kerosene fraction with distillation to 260°C, so it was the target product (jet fuel). The higher boiling point range part was separated to a gas oil and a residue fraction with vacuum distillation. Residue fraction contains the unconverted triglycerides. the formed. or unconverted diglycerides and monoglycerides, and the intermediate products, or the unconverted higher carbon number carboxyl acids, esthers.



Fig 8.: Theoretical scheme of separation of product mixtures produced by hydrogenation of sunflower oil contained kerosene fractions with catalytic hydrogenation

2.2 Materials

We carried out our experiments on an "in situ" sulphided Co(2.8%)Mo(11.2%)/Al₂O₃ heteroatom remover and a Pt(0.5%)/HZSM-22/Al₂O₃ isomerizer and partially hydrocracker catalysts. Before the beginning of experiments we loaded in to the middle section of first reactor 80 cm³ from the heteroatom remover catalyst, and in to the second reactor 40 cm³ Pt(0.5%)/HZSM-22/Al₂O₃ catalyst. This catalyst assured, we can isomerising and partially

cracking n-paraffins, that formed from sunflower oil on the heteroatom remover catalyst. The aim of it was to produce hydrocarbons, that are in jet fuel boiling point range.

During our experiments we hydrotreated pure kerosene and sunflower oil/kerosene mixtures. The quality properties of the applied kerosene fraction, that is produced with distillation of Russian crude oil, which we used during the experiments, are given in Table 3. We changed the sunflower oil content of feedstocks between 10 and 40%.

We used conventional sunflower oil, that is pressed from Hungarian sunflower for the experiments to investigation of catalytic transformation of triglycerides. We summarized its main quality properties and fatty acid composition in Table 4.

TABLE III

Properties of used kerosene								
Property	Value							
Appearance	Clear, bright and							
	sediment free							
Densitiy on 15°C, g/cm ³	0.8078							
Aromatic content, %								
Mono	16.8							
Di	3.1							
Merchaptane sulphur content, %	0.01							
Total sulphur content, mg/kg	1690							
Crystallization point, °C	-49							
Heating value, MJ/kg	42.33							
Smoke point, mm	23.8							

TABLE IV								
Properties of used sunflower oil								
Property	Conventional sunflower oil							
Kinematic viscosity, 40°C,	31.55							
mm ² /s								
Density (15°C), g/cm ³	0.9182							
Acid number, mgKOH/g	1.8							
Iodic number, g I ₂ /100g	132							
Conradson number, %	0.16							
Flashpoint, °C	>250							
Cold filter plugging point, °C	32							
Cetane number	37							
Fatty acid composition*, %								
C14:0	0.09							
C16:0	6.33							
C18:0	3.45							
C18:1	21.64							
C18:2	67.78							
C18:3	0.09							
C20:0	0.23							
C20:1	0.13							
C22:0	0.22							
Other	0.04							
Oxygen content, %	10.9							

2.3 Test methods

The main quality properties of the petroleum fraction and sunflower oil, that was used as feedstock and the liquid products of heterogen catalytic experiments were determined according to the standards with the prescribed tolerance given in Table 5.

TAI	BLE V							
Test methods of feedstocks and liquid products								
Property	Standard							
Density	EN 12185:1998							
Sulphur content	EN 14596:2007							
Sulphui Content	EN 20846:2004							
Aromatic content	EN 12916:2000							
Smoke point	EN 3014:1993							
Crystallization point	EN 2047:2002							
Gas								
chromatographic	Shimadzu GC 2010							
analysis								
Distillation	EN 2405-2000							
properties	EN 3403.2000							

We determined the composition of the sorganic product mixtures from catalytic experiments with high temperature gas chromatographic method (HT-GC), with a HP 6980A and a Shimadzu 2010 GC devices, with application of special, high temperature stable columns, with the following measuring conditions: Shimadzu 2010 GC, Zebron DB-1HT (30m x 0.32mm x 0.1 □m) column,PTV injector (temperature program: $100^{\circ}C \rightarrow 400^{\circ}C$ 30°C/min heating velocity), column space temperature program: 40°C (4 min) \rightarrow 240°C, 15° C/min heating velocity $\rightarrow 400^{\circ}$ C 8°C/min heating velocity, 11 min on 400°C, FID detector (400°C), carrier gas: $H_2(5.0 \text{ pure})$.

2.3 Results and discussion

We carried catalytic experiments to producing bio component and low aromatic content petroleum range fractions. Determining the favourable reaction parameters to hydrogenation of the petroleum fractions on CoMo/Al₂O₃ catalyst, these results are summarising in the table 6. The basis on this results we obtained that the yield of the liquid products between 340°C – 360°C (LHSV = 2,0 m³/m³h, H₂/CH ratio = 200Nm³/m³) showed the highest rate(97.8 – 99.1%). The pressure rising from 35bar to 50bar resulted in only 0.1 - 0.2% liquid yield decreasing. Fixing the temperature (on 340°C – 360°C) the products sulphur content was only 1 – 2 mg/kg on any other investigated reaction parameters, consequently hydrogenation of sulphur content molecules have been completely (feedstock sulphur content 1690mg/kg). The desulphurization and the partial dearomatization occurred parallel during the process. On 320°C – 380°C temperature saturation of diaromatics range. the to monoaromatics have been completely, may be saturated towards. The total aromatic content was decreasing with the increasing temperature to near half (of the start concentration), on the abovementioned favourable operation parameters. This is because, the rate of the exoterm aromatic saturation reaction decreased with the increasing temperature due to the thermodynamic hindrance. Increasing the pressure from 35bar to 50bar effected that the aromatics content decreasing further 1.5 - 2.9%. It is can be explained to the pressure increasing favour of the mole number decrease resulting reactions.

TABLE VI.

Hydrogenation of petroleum fraction (sulphur content 1690 mg/kg; aromatic content: 19.9 %) on sulphided CoMo/Al₂O₃ catalyst (LHSV = 2.0 h^{-1} , H₂/CH ratio = 200 Nm^3 /m³); the yield and the products main quality properties

AND THE PRODUCTS MAIN QUALITY PROPERTIES										
Properties		Temperature								
Temperature, °C	320		340		360		380			
Pressure, bar	35	50	35	50	35	50	35	50		
Yield of liquid										
products,%	99.8	99.6	99.1	98.9	98.0	97.8	96.0	95.7		
sulphur content,	14	3	2	<1	<1	<1	<1	<1		
mg/kg										
aromatics	19.8	18.3	13.1	10.2	10.2	8.7	16.3	14.8		
content, %										

The basis of all above-mentioned results we obtained that the hydrogenation of petroleum fraction itself the favourable process parameters was follows, temperature: $340 \text{ }^{\circ}\text{C} - 360 \text{ }^{\circ}\text{C}$, LHSV: 2.0 h⁻¹, H₂/CH ratio = $200 \text{Nm}^3/\text{m}^3$.

We explained previously that during the hydro conversion of sunflower oil – petroleum fraction blend we used such catalytic system that is capable to produce petroleum boiling range fraction from the hydrogenation steps mainly C_{17} C_{18} hydrocarbons containing products. Hence the products that formed on CoMo/Al₂O₃ catalyst (after stripping the water and H₂S) mixed adequate amount of hydrogen and we performed further conversion.

In the table 7. we summarised the favourable results that obtained sulphided $CoMo/Al_2O_3$ and Pt-HZSM-22 catalyst collective use. The basis on this results we found that the yield of the liquid products decreased onwards in the case of use Pt-HZSM-22. This can be explain by this catalyst moderate cracking activity. The sulphur content of the products aromatic contents decreased onwards that the result of Pt-HZSM-22 catalyst hydrogenation activity. The total aromatics content decreasing on two catalyst bed was 75% - 80% on the favourable reaction parameters. (temperature = $340^{\circ}C - 360^{\circ}C$, 50bar, 1.33 total LHSV, H₂/CH ratio = $200Nm^3/m^3$)

TABLE VII.

Hydrogenation of petroleum fraction (sulphur content 1690 mg/kg; aromatic content: 19.9 %) on sulphided CoMo/Al₂O₃ and Pt/HZSM-22/Al₂O₃ catalyst (LHSV = 2.0 h-1, H₂/CH ratio = 200 Nm³/m³); the yield and the products main quality properties

Properties	Temperature							
Temperature, °C	320		340		360		380	
Pressure, bar	35	50	35	50	35	50	35	50
Yield of liquid								
products,%	98.4	99.2	97.1	98.5	95.3	97.2	91.2	93.0
sulphur content, mg/kg	<1	<1	<1	<1	<1	<1	<1	<1
aromatics content (mono aromatic), %	14.3	8.4	8.2	2.6	6.0	5.1	7.1	5.9

The process parameter for the conversion of sunflower oil- jet mixtures were selected based on the previously presented results for Jet fractions and regarding the about 11% oxygen content of sunflower oil. Considering the latter one is important, because the heteroatom content of 10-40% sunflower oil containing mixtures is higher by 1.1-4.4% than that is the pure jet fraction. This essentially decreases the activity of the catalyst. Consequently the value of liquid hourly space velocity (LHSV) was varied between 0.5 and 1.3 h⁻¹, in order to increase the contact time of the feedstock on the catalyst.

During the hydrogenation of sunflower oil- jet mixtures we investigated the yield of jet and gasoil fractions in the obtained liquid products. The increase of the temperature was favorable for the cracking reactions. This resulted the decrease of the yield of jet boiling range compounds coming from the jet feedstock. On the other hand, the yield increased up to 360°C, since hydrocarbons having jet boiling range were formed from vegetable oil. At 380°C the yield of these latter compounds decreased as well, mainly because of the high hydrocracking activity of the Pt/HZSM-22/Al₂O₃ catalyst (Fig. 9 and 10).



Fig 9. Yield of the Jet fuel boiling range products as a function of the temperature and sunflower oil content of the feedstock (P=35 bar, H_2 /CH volume

ratio: $600 \text{ Nm}^3/\text{m}^3$)



Fig 10. Yield of the Jet fuel boiling range products as a function of the temperature and sunflower oil content of the feedstock (P = 50 bar, H₂/CH volume ratio: $600 \text{ Nm}^3/\text{m}^3$)

The gas oil yield increased with the increasing of the vegetable oil concentration. At the same time these values decreased with the decreasing of liquid hourly space velocity, while yield of jet fuel increased (Figure 11.). The reason is at lower liquid hourly space velocity the cracking was higher, moreover at higher liquid hourly space velocity the conversion was lower ont he hydrotreater catalyst, so as its effect the remaining oxygen content component inhibited the cracking activity of $Pt/HZSM-22/Al_2O_3$.



Fig 11. Yield of gas oil as a function of liquid hourly space velocity

 $(T=360^{\circ}C, H_2/HC \text{ ratio: } 600 \text{ Nm}^3/\text{m}^3)$

The aromatic content of the product mixtures obtained from the jet – sunflower oil mixtures varied according a minimum curve as a function of the temperature (Fig. 12.). By increasing the temperature up to 360° C the concentration of the aromatic compounds decreased in the products. However at 380° C the aromatic content of the products were higher because of the thermodynamic inhibition. At 320° C the aromatic content of the products were higher than it was expected. It was due to the oxygenic compound of the sunflower oil, which hindered the hydrogenation of aromatics, since they partially covered the catalytically active sites of the catalysts.



Fig. 12. The concentration of aromatic hydrocarbons as a function of the temperature and pressure (LHSV = 0.8 h^{-1} , H₂/CH volume ratio: 600 Nm³/m³)

The crystallization point and smoke point of the obtain jet products are summarized in Table 8. It can be seen that the smoke point increased, while the crystallization point decreased by increase the severity of the process parameters. These can be explained in some part by the formation of normal and isoparaffins, and in some part by the decrease of the aromatic content. These values were less favorable at 380°C, than values of products at 360°C. The reason is the decreasing of hydrogenation of aromatics. These have unfavorable effect to crystallization point and flaming properties of products.

AFFLICATION PROFERITES OF THE FRODUCED JET FUELS										
Temperature, °C	320		340		360		380			
Pressure, bar	35	50	35	50	35	50	35	50		
LHSV, 1/h	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0		
Crystallization point, °C	-47	-48	-49	-51	-53	-56	-54	-56		
Smoke point, mm	25.9	28.5	30.3	32.9	34.2	36.8	32.4	33.8		

TABLE VIII. Application properties of the produced jet fuels

4 Summary

So we studied during our experimental work hydrogenation of sunflower oil containing kerosene fraction on a CoMo/Al₂O₃ heteroatom remover and Pt/HZSM-22/Al₂O₃ isomerizer and partially cracking catalyst system. We studied the yield, analytical and application properties of the jet fuel boiling point range hydrocarbons. At the joint application of the chosen catalysts, we found process parameter combinations, that resulted jet fuel boiling point range products, that have excellent flaming properties (smoke point: 37 mm) and good low temperature properties (crystallization point: <-56°C). This contains clear burning iso-and nparaffins in high concentration, that are not limited for quantity by standards.

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