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Influence of catalyst conditioning on products in a continuously operated FCC pilot plant

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INFLUENCE OF CATALYST CONDITIONING ON PRODUCTS IN A CONTINUOUSLY OPERATED FCC PILOT PLANT

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ABSTRACT

Until recently, the tried and proven refinery process of Fluid Catalytic Cracking (FCC) had primarily been used to increase the product yields of gasoline in order to provide the majority of gasoline demands. While this is still true for the United States, where the majority of motor fuel is still gasoline and diesel fuel plays a minor role, the opposite applies to Europe. In Europe, gasoline consumption stagnates, while diesel fuel demands are on the rise. Along with political and other economic considerations, this caused a shift of interest towards these refinery products. Among the possible process alterations to achieve this goal are different catalysts which are optimized for Light Cycle Oil (LCO) production.

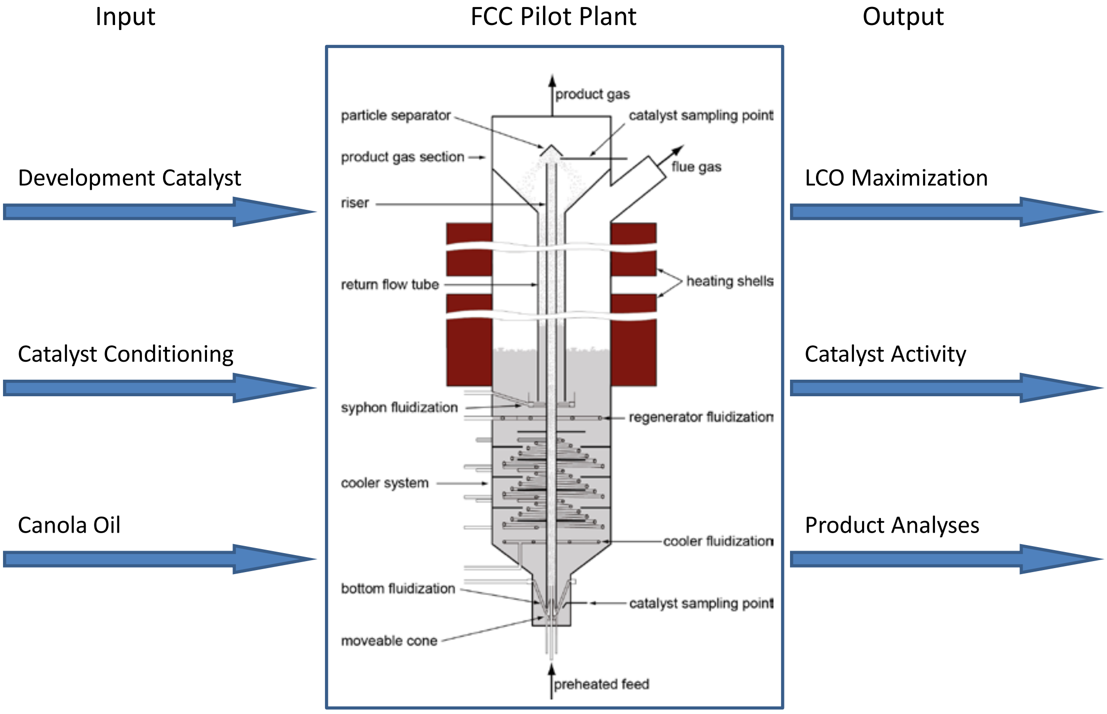
One point of interest therein is the correlation between catalyst activity and the related product distribution, which is the core of the experiments conducted for this paper. One goal was to maximize the LCO yield, the other was the comparison of these results in relation to catalyst activity. The experiments began with a fresh catalyst with unrestrained activity. This activity was lowered step by step through thermal and hydrothermal conditioning at a temperature of 830°C of the catalyst. Achieving an inert material even by violent conditioning is likely to be very difficult due to the composition and stability of a modern catalyst. The destruction of the crystalline matrix would likely occur before complete deactivation. Experiments with an inert substitute, resulting in thermal cracking exclusively, were conducted by Fimberger [1].

The experimental test program was conducted in a continuously operated FCC pilot plant at Vienna University of Technology. To allow for comparability of the results, all experiments were conducted at the same reaction temperature of 550°C and a feed rate of 2.5 kg/h. The results clearly show the desired shift in the resulting product spectra with continued conditioning, while no negative impact on Total Fuel Yield was observed.

KEYWORDS

FCC, Pilot Plant, Catalytic Cracking, Catalyst Conditioning, Fluidized Bed, Vegetable Oil, Canola Oil, Refinery technology, LCO

GRAPHICAL ABSTRACT



HIGHLIGHTS

- Continuously operated medium scale pilot plant
- Use of canola oil – CO_2 neutral FCC Process
- Stepwise conditioning of an FCC fresh catalyst
- Comparison of related product spectra
- Maximization of LCO

1. Introduction

The FCC process had for the most time been seen as the premier refining step in gasoline production. Until today, gasoline is the primary product of existing FCC-plants. Without it, it would be impossible to cover the demand of gasoline in our society. In the recent years however, with the fast development and improvements of diesel engines, a shift in product demand has occurred.

While in the U.S. the majority of vehicles still are gasoline driven, there is a remarkable trend towards diesel driven vehicles in Europe. For example, in the United States only approximately 25 per cent of the Net Production were Distillate Fuel Oils (Diesel) [2] [3]. In contrast, European demands are quite the opposite, where Distillate Fuel Oils account for approximately 70 % of the total fuel demand [4]. The trend in Europe continues towards the stagnation of petroleum / gasoline and an increased demand in Diesel fuels.

For this reason, refineries in Europe are, by various means, attempting to shift their product spectra toward the liquid products in general and the diesel fractions in particular.

During the experimental work canola oil was used as feedstock, as there is a big political and economical focus on renewable energies from vegetable oils in Europe. In this scope, canola oil is for several reasons the most significant vegetable oil. The catalyst used was an as of yet non-commercial FCC catalyst. Proceeding from its fresh, unconditioned state, the catalyst was conditioned in several steps by means of thermal (temperature only) and hydrothermal (steaming) treatment at temperatures of up to 830°C. To allow for such catalyst treatment, the plant had to be adapted to allow for alternating between conditioning and catalytic cracking.

The goals in this study were twofold: One aspect was to analyze the correlation between catalyst activity and product spectra. The set goal of the undertaken experimental runs was to maximize of the desired liquid products, minimizing production of light gaseous products, while at the same time avoiding an increased production of residue and a reduction of product quality. Hand in hand with the aforementioned goal went the attempt to maximize the production of middle distillates. Both aspects were investigated during the test runs. The obtained products were analyzed and the results were used as a measure for catalyst deactivation.

Experimental Setup

2.1 Pilot Plant

Fig. 1 shows a schematic of the FCC-Pilot Plant used in the experiments. Table 1 provides key data of the pilot plant. Constructed as an internally circulating fluidized bed reactor (in contrast to industrial plants, which are usually constructed as externally circulating systems), the plant has several unique design features. On one hand, the design provides for a very compact device with comparatively little space requirements, while on the other hand this fact causes a strong thermal coupling between the reactor and regenerator sections for example.

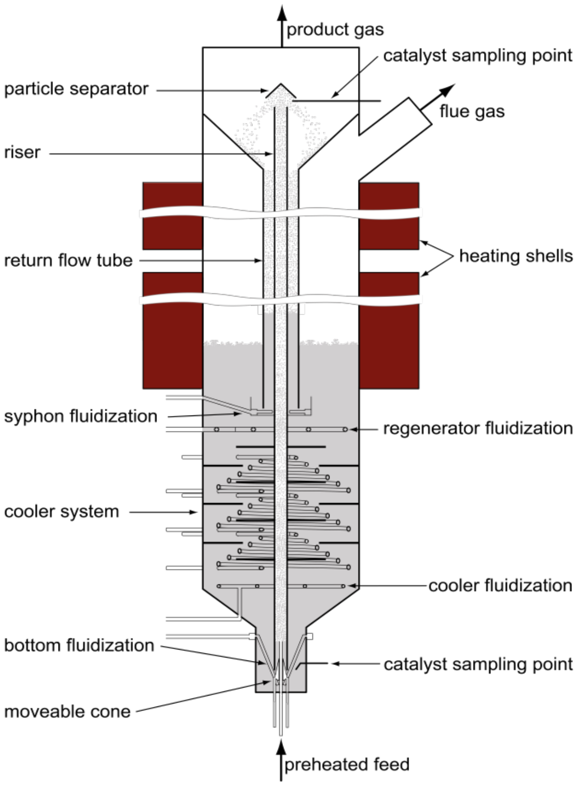


Fig. 1 - Pilot Plant Schematic

Table 1 - Pilot Plant Key Data

Total Height	3,2 m
Riser length	2,505 m
Riser diameter	0,0215 m
Regenerator diameter	0,33 m
Catalyst	shape selective zeolite
Catalyst mass	45-65 kg
Pressure	ambient
Riser temperature	400-700°C
Regenerator temperature	550-800°C
Feed rate	2-8 kg/h
Riser residence time	~1 s
C/O-ratio (adjustable)	10-50

During the experiments, the feed is preheated to a temperature high enough to provide for near-instantaneous evaporation in the plant, yet low enough to prevent coke formation due to thermal cracking of the feed in the feeding system. The preheated feed then enters the plant through the feed inlet pipe, the end of which being located in the riser reactor. Upon contact with the hot catalyst, the feed evaporates and the cracking reactions begin. The enormous increase in volume due to the evaporation and cracking causes a very fast upwards stream of gaseous feed, cracking products and catalyst particles alike. This pneumatic motion also causes a pressure differential, causing the catalyst particles to be continuously sucked from the bottom into the riser. During the residence time in the riser, the catalytic cracking reactions take place. Insufficient presence of hydrogen (required for the formation of short-chained hydrocarbons) causes the formation of coke and its deposition on the catalyst particles' surface, causing rapid degradation of the catalyst's catalytic properties. Upon exiting the riser, the product gas is then separated from the spent (coke-loaded) catalyst. It exits the pilot plant on top and most of it is burnt in a flare.

A small portion of the product gas is gathered for analytic purposes. The increase in diameter at the riser outlet causes a rapid deceleration of the stream, allowing the spent particles to fall back into a return flow tube. At the bottom of this tube, they are transported into the regenerator via the syphon. The Regenerator is operated as a bubbling fluidized bed and the only area in the plant running on air. This allows the coke to be burned off, effectively regenerating the catalyst by freeing its active surface and also providing a part of the process heat needed for the endothermic cracking reactions. The regenerated catalyst then reaches the bottom of the plant, passing through a cooler system, from where it is once again sucked into the riser and the cycle thus repeated continuously as long as feed is provided.

All other areas are fluidized with nitrogen. This is necessary, because the bottom and syphon fluidizations not only act to facilitate catalyst circulation, but also as a gas barrier between reactor and regenerator. For obvious reasons, gas leakages in both directions are less than desirable during operation.

Note that feed rate, riser and regenerator temperatures and C/O ratio can be adjusted within the limits defined in Table 1 during operation.

2.2 Feedstock and Catalyst

The feedstock used for the experimental work was canola oil. The use of vegetable oils in general is in part based on the fact that their side chains are chemically more similar to the Diesel and LCO factions than Vacuum Gas Oil (VGO). Moreover, canola oil in particular was chosen because of its domestic importance.

Canola oil itself is a triglyceride, combining three fatty acids with a glycerol body thus forming an ester (Fig. 2).

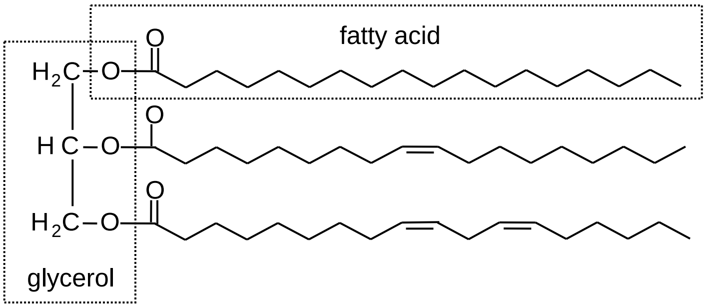


Fig. 2 - Example of a triglyceride

The oil was obtained from Rapso Österreich GmbH. A list of the oil's components and properties is provided in Tables 2 and 3.

Table 2 - Canola Composition by fatty acids

Acid	Structure	Amount [wt%]
Palmitic	C16:0	4.5
Stearic	C18:0	1.8
Oleic	C18:1	60.8
Linoleic	C18:2	18.5
Linolenic	C18:3	8.0
Rest		6.4

Table 3 - Canola Oil properties

Density (20°C)	915 kg/m³
Viscosity (40°C)	35.9 mm²/s
Bromine Number	69.1
Acid Number	0.1 mg/g
Oxygen Content	10.85 wt%
Water Content	< 0.1 wt%

Regarding the fatty acids, the composition of the canola oil used as feedstock for the experimental work can be seen in Fig. 3.

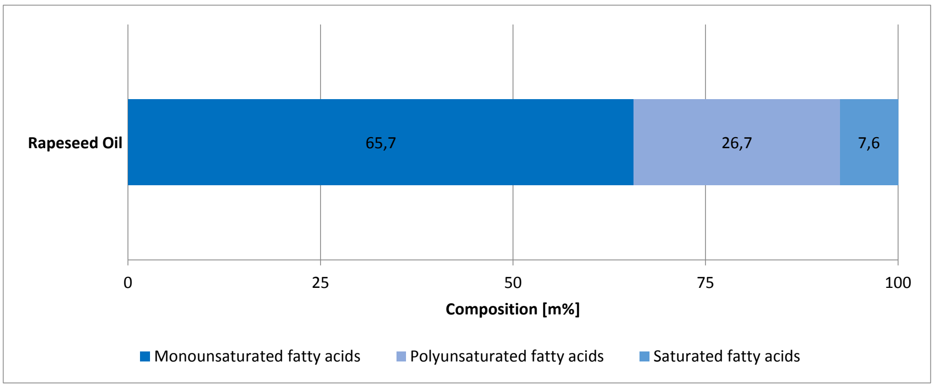


Fig. 3 - Canola oil composition

The catalytic reactions taking place between canola oil and catalyst will not be described in this paper. The reader is advised to refer to reference literature, as the feasibility of canola oil as FCC feedstock and the related reactions of vegetable oils have been proven by other research projects (e.g. [5] or [6]).

The catalyst used in the experiments is an as of yet non-commercial Zeolite-based FCC catalyst. For reasons of confidentiality, the exact properties cannot be included in this paper, and some properties were not even known to the authors. Suffice to say that this particular catalyst had been developed with a decreased activity compared to other catalysts. In order to be able to study the effects of catalyst conditioning during our experiments, this catalyst needed to be in its fresh unconditioned state.

In this state, the catalyst usually has a Si/Al ratio of close to 1 and therefore has a very high catalytic activity due to the high amount of aluminum atoms bound in the crystalline structure, which act as strong catalytic acid sites [7]. This promotes severe cracking, which results in the formation of small, gaseous hydrocarbon products and large amounts of coke, while gasoline and LCO were of low content in the products. This fact also led to problems during the first experiments and is the main reason for the industrial conditioning of commercial FCC catalysts. This hydrothermal process "damages" the crystalline lattice by forcibly removing aluminum atoms from it. This reduces the catalytic activity of the catalyst to a point, where, depending on the catalyst, the products contain large amounts of gasoline (up to and beyond 50%) and gas (up to 40%), a moderate amount of LCO (10-20%) and relatively little coke (5-10%) [7].

On the other end of the spectrum is a completely inert bed material like quartz sand, leading to thermal cracking only, which has also been tested by Fimberger [1]. While pure thermal cracking caused the formation of large amounts of LCO, product quality was impaired, mainly due to oxygen still present in the product molecules [1].

Comparing the results of the tests from both these extremes, it was theorized that there could be a point of catalyst conditioning, where LCO production would reach a maximum while still retaining a high product quality through catalytic deoxygenation, effectively removing oxygen from the products as water.

For this reason, a test program was established during which every conditioning step was followed by a cracking experiment to allow for the comparison of the related product yields and thereby draw a conclusion to catalyst activity. The catalyst activity itself is closely connected to the Unit Cell Size [8], which in turn is strongly influenced by the conditioning [9]. In this context, conditioning causes the Unit Cell Size to shrink, which in turn causes a reduction of catalyst activity. Furthermore gasoline selectivity is also reduced thereby, leading to a larger LCO yield [8] [9].

2.3 Analyses

The products obtained from the FCC process are comprised of countless hydrocarbon species. For this reason, a simplification is necessary to characterize the products with reasonable effort and accuracy. The model used (also in industrial refining processes) is the so called lump model. In this model, the entire product is divided into several groups with similar properties (e.g. boiling ranges; see Table 4).

Table 4 - The Lump Model

Fraction	Lump	Composition, Boiling range	Analysis method
Gas fraction	Crack gas	C1-C4	GC
	CO_x		infrared
Liquid fraction	Gasoline	<190°C	GC (SimDist)
	LCO	190-360°C	GC (SimDist)
	Residue	>360°C	GC (SimDist)
	Water		gravimetric
Solid fraction	Coke		Gas analyzer (flue gas)

By means of cooling the product gas, a gaseous and a liquid fraction is obtained in the first step. When using vegetable oils in the FCC process, water is formed as one more liquid byproduct which can easily be separated from the organic compounds by phase separation. The water phase is not discarded, but rather used in the calculations to establish the complete feedbased lump model. The amount of the solid byproduct of the FCC process – coke – which is burned in the regenerator is calculated based on the carbon oxide measurement data from the online flue gas measurement. The carbon oxides in both the flue gas and the product gas are measured online by two redundant Rosemount NGA2000 gas analyzers. In the same way as the CO_x content in the flue gas can be used to determine the amount of formed coke, the CO_x content in the product gas can furthermore be used as an indicator for a reduced catalytic cracking activity, as the catalytic reactions usually form water if oxygen is present in the feed molecules, thereby leading to a significant CO_x reduction in the product gas.

The analyses of both the gaseous and the liquid products were conducted by gas chromatography utilizing two separate GC's . The gaseous product was analyzed with a classic gas chromatograph (GC) in which the hydrocarbons are identified and quantified by a flame ionization detector (FID), while Nitrogen is measured by a thermal conductivity detector (TCD). Refer to Table 5 for a more detailed equipment description.

Table 5 - Gas Chromatograph configuration

Gas-GC - Shimadzu GC-17A		
Columns	I:	Varian CP-Al2O3/Na2SO4
	II:	CP CarboPLOT P7
Phase	I:	100% Polydimethylsiloxan
	II:	Carbon Porous Layer
Dimensions	I:	50 m x 0,25 mm ID x 4 µm
	II:	27,5 m x 0,53 mm ID x 25 µm
Temperature Program	50°C to 200°C; Dwell time: 30min	
Carrier gas	Helium 1,46 mL · min-1 constant flow	
Injector	Splitless	
Injection volume	50 µL @ 200°C	
Detectors	I:	Flame Ionization Detector (FID) @ 200°C
	II:	Thermal Conductivity Detector (WLD) @ 105°C

Table 6 SimDist GC configuration

SimDist – GC Shimadzu GC-17A	
Column	Zebron ZB-1 30m x 0.32mm x 0.25µm
Temperature Program	2min at 35°C, ramp 10min to 350°C, dwelltime 24min
Carrier Gas	Hydrogen, 1.68ml/min
Injector	Split Injector, 350°C, 1:30 Split
Injection Volume	1.5µl
Detector	Flame Ionization Detector (FID) at 350°C

The liquid organic phase was analyzed by means of a simulated distillation (SimDist). This is a GC with a single capillary column and an FID (refer to Table 6 for a more detailed description). Unlike the gas GC, individual species cannot be determined due to the exponentially growing number of isomers the longer the hydrocarbon molecule chains get.

However, the correlation between retention time and boiling temperatures can be used to determine the distillation range. Furthermore, a manual distillation of the liquid fraction was carried out as per Table 4. The obtained lumps are then used to determine the octane numbers of the gasoline fraction and the cetane number of the diesel/LCO fraction.

Note that C5 and C6 hydrocarbons cannot be condensed entirely, for this reason they are present in both the gaseous and liquid fractions. These hydrocarbons are attributed to the liquid fraction.

Besides the individual lumps another very important parameter is the total fuel yield (TFY), which is defined as the sum of valuable products (crackgas, gasoline, and in our case LCO) in relation to the amount of feed processed:

$$TFY = \frac{\dot{m}_{crackgas} + \dot{m}_{gasoline} + \dot{m}_{LCO}}{\dot{m}_{feed}}$$

The TFY can therefore be seen as a measure of the overall process performance.

RESULTS

This first part of experiments revolved around the relation between catalyst activity and product spectra obtained. The first experiment was conducted with the fresh catalyst. Due to the extremely high activity of the catalyst material, mainly gas and a large amount of coke were produced. In a matter of seconds the coke accumulation inside the pilot plant forced the abort of the experiment and shutdown of the plant. For the same reason, only a minuscule amount of data and product was obtained and thus the data had to be discarded .The next step was the conditioning of the catalyst by means of both thermal (heat only) and hydrothermal (H_2O steam and heat) treatment. Starting with thermal treatment and continuing with hydrothermal treatment in 2 separate steps of different conditions respectively (see Table 1).

Table 7 Catalyst conditioning – treatment properties

Step	Thermal Conditioning		Steaming	
	Temperature [°C]	Time [h]	Temperature [°C]	Time [h]
1	795	15	-	-
2	830	10	-	-
3	-	-	830	6
4	-	-	830	23

The means to judge the progress of the conditioning was the comparison of the products that were obtained during the experiments that followed each conditioning step. To ensure comparability of the results, the relevant process parameters were maintained constant during each Cracking experiment. The main parameters were the constant federate of 2.5 kg/h and a constant reaction temperature of 550°C. Fig. 4 clearly shows the changes in the results.

The shift of total conversion and product composition clearly indicates movement in the desired direction. The reduction of total conversion and gas production showing an increase in gasoline and LCO is congruent to many other experiments related to catalyst deactivation, confirming our theoretical expectations. The product yields in relation to the time of conditioning are provided in Table 8 and Fig. 4.

Table 8 - Product yields in relation to conditioning time

Lump [wt%], feedbased	Conditioning time (hours total)			
	15	25	31	54
Crack Gas	20,33	23,57	21,68	16,13
Gasoline	41,80	41,32	41,45	45,972
LCO	17,08	16,31	16,87	17,60
Residue	2,34	2,042	1,95	1,54
Coke	9,53	8,86	8,06	7,49
Water	6,56	5,60	8,241	9,37
Carbon Oxides	2,32	2,27	1,71	1,87
Total fuel yield	79,22	81,21	80,01	79,71

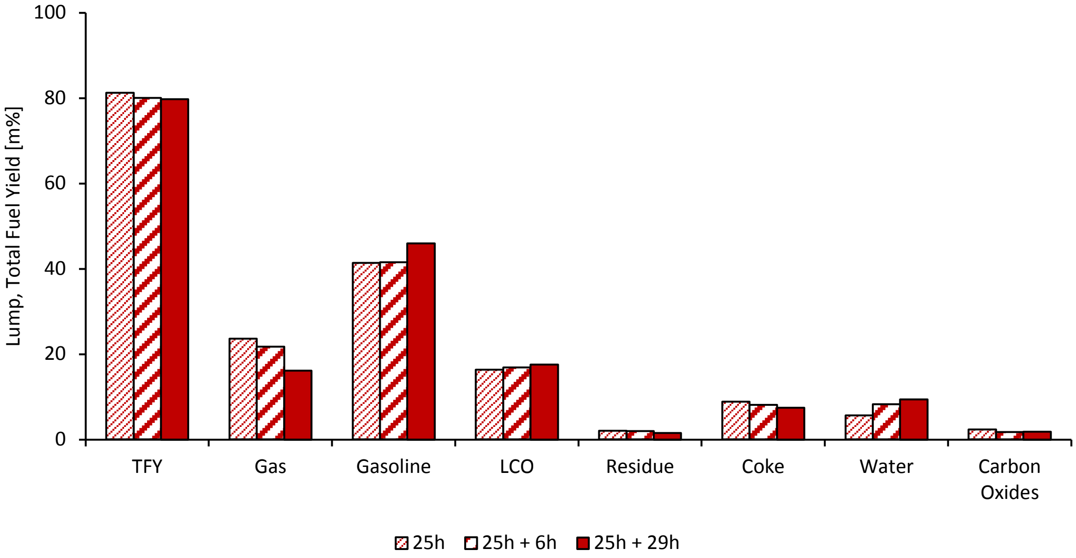


Fig. 4 - Change in TFY and product lumps by conditioning step

The interesting observations here however, clearly were that the change in the product yields and total conversion show a nearly linear progression. That fact is of interest because the total of 29 hours of steaming were by far the more aggressive means of conditioning compared to pure thermal conditioning. This clearly points in the direction that catalyst deactivation is not a linear process, but much rather follows an exponential pattern [7] [8] [9].

While the first, although quite mild, steps showed relatively great impact, the following, more severe steps had comparably less influence on catalyst activity. This observation is in accordance with the results of industrial treatment of FCC-catalysts during their manufacturing. For a detailed description of these steps in catalyst manufacturing, extended reference literature is recommended (eg. [7]).

Regarding the composition of the crack gas according to Fig. 5, the impact of the conditioning steps is also evident and in accordance with expectations. While the amount of ethylene remains relatively constant, those of propylene and butylene rise at the expense of the remaining gases, which contain mostly methane, ethane and propane. This is a clear indication for a more controlled catalytic cracking process following an ionic mechanism. This also clearly increases the quality of the gas lump overall.

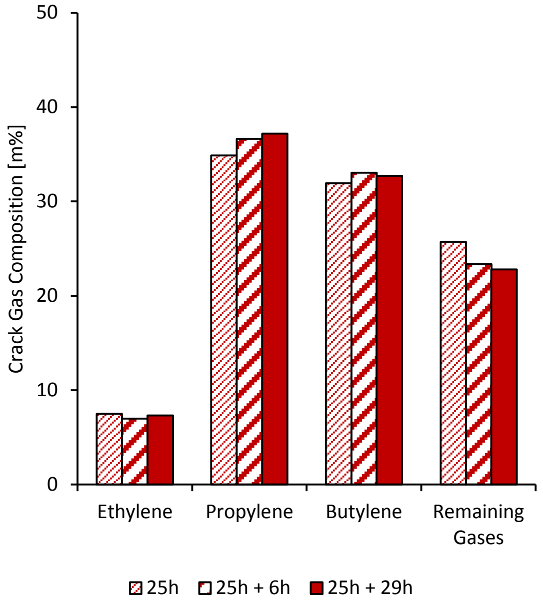


Fig. 5 - Crack Gas Composition by conditioning step

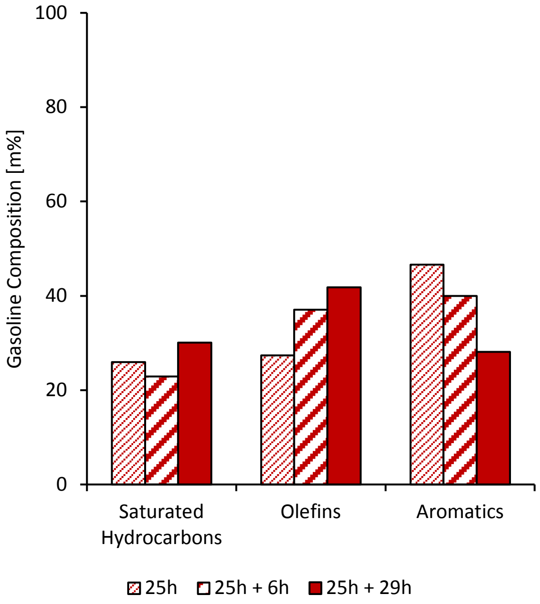


Fig. 6- Gasoline Composition by Conditioning Step

Fig. 6 shows the change in gasoline composition. While the amount of saturated Hydrocarbons shows no distinct tendencies, olefins and aromatics as important indicators of gasoline quality clearly do. The amount of olefins rises at the cost of aromatics content. While this is not unexpected, it has a negative impact on product quality, as high olefin contents negatively influences storage stability due to their tendency for polymerization and oxidation. The reduction in the aromatics content also leads to a loss in quality through the reduction of the octane number of the gasoline lump.

Regarding LCO, on a positive note, a slight increase of the amount of LCO was observed. The increase in LCO was less than hoped for, but still readily measurable. This is not entirely unexpected, as most current commercial FCC catalysts are optimized for olefin and gasoline production. Furthermore they are usually based on Ultra Stable Zeolites and are therefore extremely resilient to thermal and hydrothermal damage/conditioning once they have reached their so called equilibrium (E-Cat).

Also, because of the change in reaction mechanisms due to the continued conditioning, a reduction in coke formation was observed, along with a significant increase in water formation. Especially the increased water formation leads to an increase in overall product quality, because the oxygen contained in the feed triglycerols is removed as water instead of remaining bound in the liquid product molecules. Oxygen in the products has a significant negative impact on product quality. On one hand it reduces product stability and on the other hand it reduces the products' overall heating value.

CONCLUSION

The adaption of the pilot plant for use as a catalyst conditioning device posed little difficulty and was carried out successfully. The challenge in this case was to adapt the plant in such a way, that it could be used alternating between conditioning and catalytic cracking without further effort.

Conditioning the catalyst neither posed a problem. The advance of catalyst conditioning can easily be measured by the related change in the resulting product spectra and therefore the catalyst activity. What could not be measured directly were the UCS and the grade of dealumination. However, judging from the change in product spectra, the conditioning clearly occurred as described in [7-9].

The obtained product spectra themselves also show the expected changes. The decrease in coke formation is preferable for a higher product yield, yet care must be taken to ensure the formation of a sufficient amount of coke to sustain the endothermic cracking processes. The increase in water is a good indicator of an improving product quality, as the oxygen contained in the feed molecules is being removed in that form, rather than CO_x or even remaining in the product molecules, which would impair product quality. The increase in LCO can readily be seen, although an even higher increase would be preferable. Such an increase, however, is unlikely to be achievable with a modern ultrastable catalyst by steaming alone, as these catalysts are designed to withstand even severe conditions. Furthermore, most conventional FCC catalysts are optimized for high gasoline and short olefin production, thus making a significant improvement in LCO yields even more difficult. Even though this particular catalyst had already been designed with a reduced activity, it still showed a clear tendency towards the aforementioned conventional behavior.

So, while an increase in LCO yields and an increase in product quality at the same time is possible to a measurable extent, an even higher shift of the product spectra towards LCO seems to be achievable by mixing an inert bed material with a low-activity FCC catalyst. This mixing would likely bring about other challenges however. Among others for example the segregation of the bulk material due to differences in physical properties. Another possible way to proceed in this direction could be the development of a new generation of catalysts, optimized for LCO production.

ACKNOWLEDGMENTS

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Refereces

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Special Field Fluidized Bed Systems and Refinery Technology
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- *The Lump Model*
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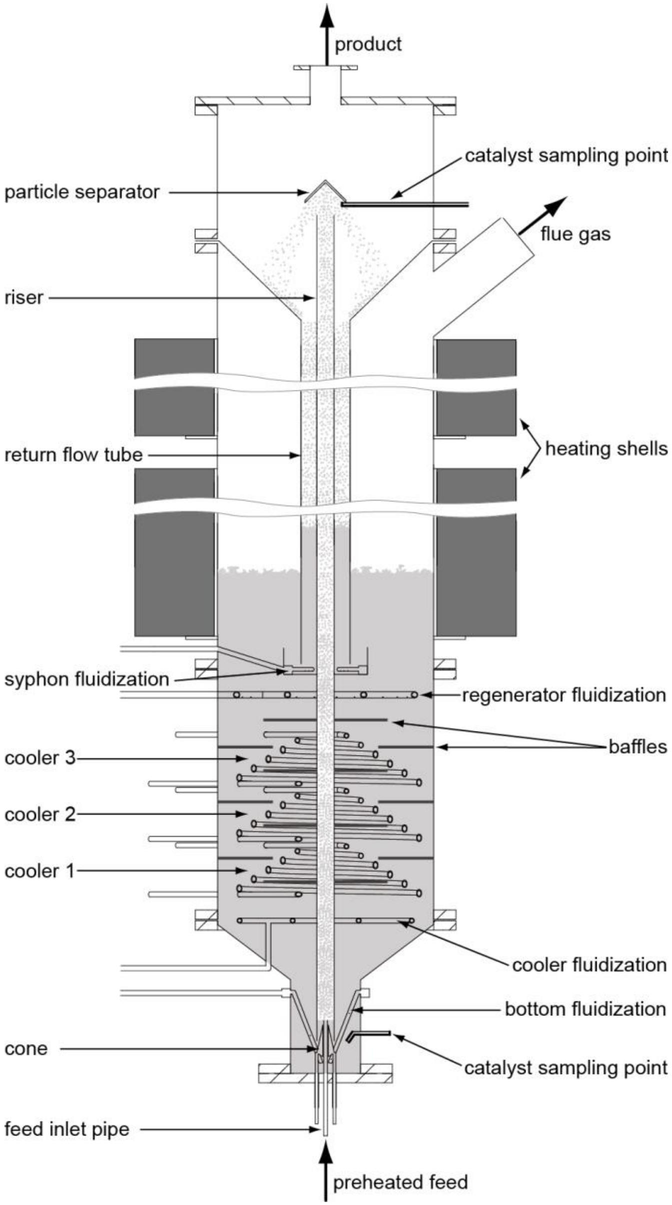
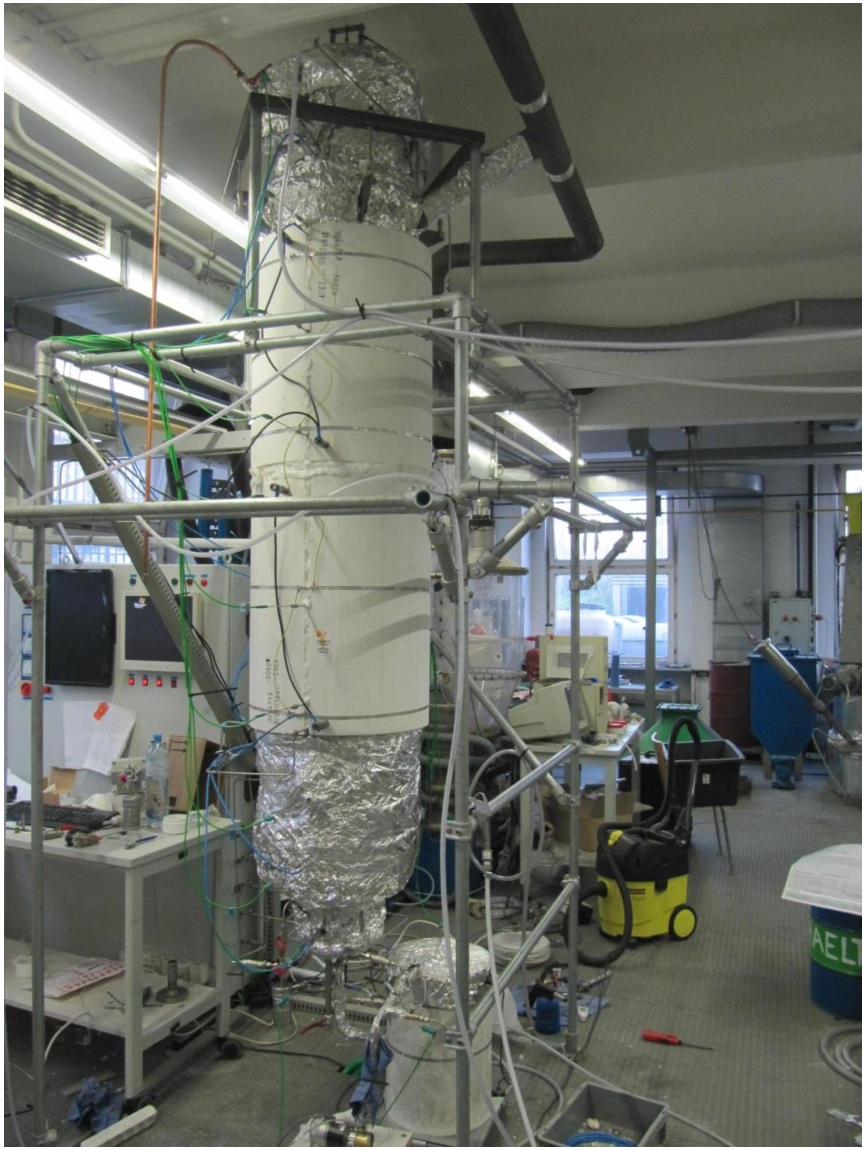
- Conclusion

The 2nd generation FCC pilot plant

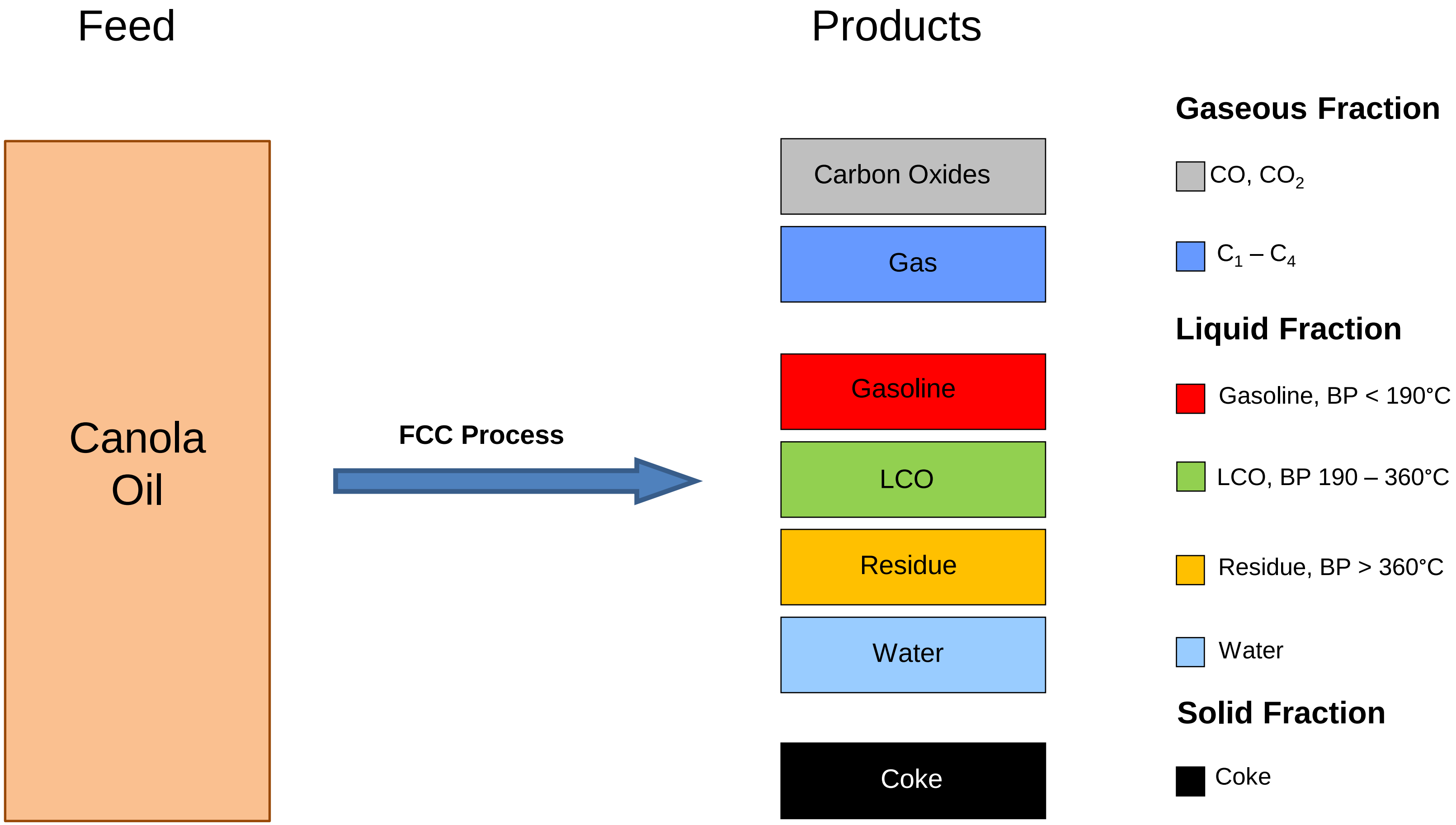


Height	3.2 m
Riser length	2.505 m
Riser diameter	0.0215 m
Regenerator diameter	0.33 m
Catalyst	commercial E-Cat
	shape selective zeolite
Catalyst mass	40-65 kg
Catalyst spectrum	20-200 µm
Riser temperature	400-700°C (range)
Regenerator temperature	550-800°C (range)
Feed rate	2-8 kg/h
Riser residence time	~1 s
C/O-ratio (adjustable)	10-50
Pressure	ambient

The advanced FCC pilot plant



The Lump Model



Feed

Products

Canola Oil

FCC Process

Carbon Oxides

Gas

Gasoline

LCO

Residue

Water

Coke

Gaseous Fraction

- ⬜ CO, CO_2
- 🟦 $C_1 – C_4$

Liquid Fraction

- 🟥 Gasoline, BP < 190°C
- 🟩 LCO, BP 190 – 360°C
- 🟨 Residue, BP > 360°C
- 🟦 Water

Solid Fraction

- ⬛ Coke

$$TFY = \frac{m_{gas} + m_{gasoline} + m_{LCO}}{m_{Feed}}$$

Goals and Objectives

Main objectives

- Use of a low-activity, non-commercial development catalyst in conjunction with Canola Oil
- Stepwise Catalyst conditioning to
- Observe the change in the product spectra with
- focus on LCO

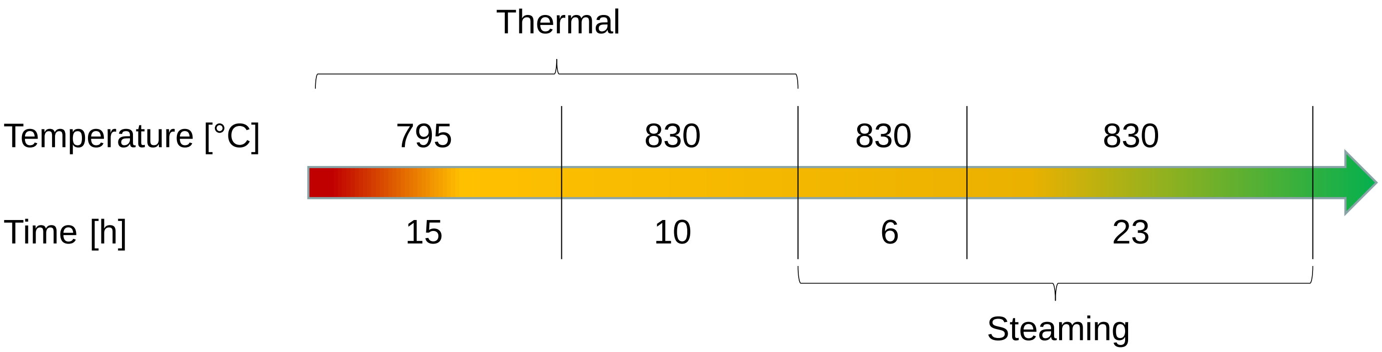
→ Product properties?

→ LCO yields & quality?

Catalyst Conditioning

Stepwise Conditioning by means of

- Thermal Treatment (heat only)
- Hydrothermal treatment (high temperature steam)



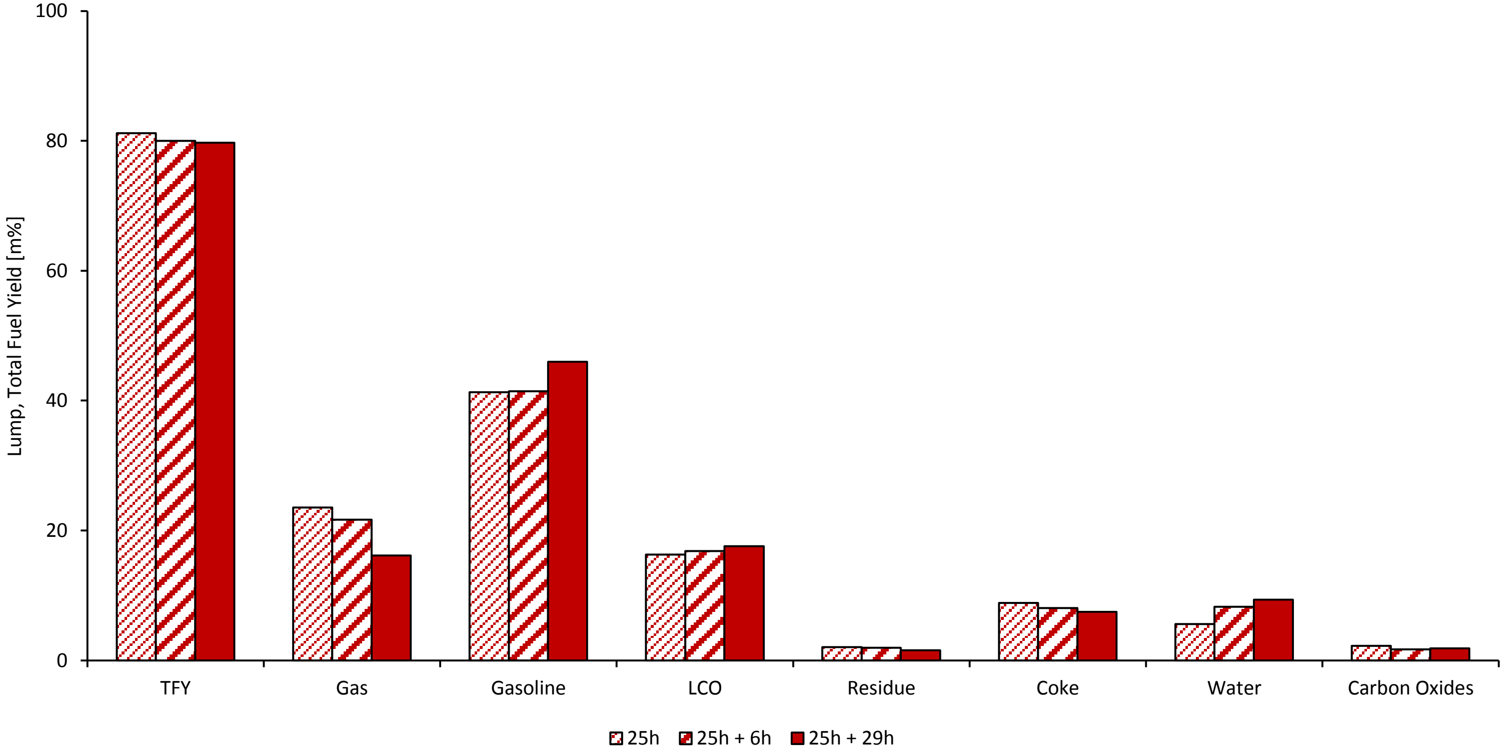
	Thermal			
Temperature [°C]	795	830	830	830
Time [h]	15	10	6	23
			Steaming	

Expected Changes

Affected catalyst properties include

- Non-linear reduction of Activity
- Change in selectivity
- No change in Deoxygenation process

Results


Lump, Total Fuel Yield [m%]
100
80
60
40
20
0
TFY
Gas
Gasoline
LCO
Residue
Coke
Water
Carbon Oxides
▨ 25h
▨ 25h + 6h
■ 25h + 29h

Conclusions & Acknowledgement

- Pure Canola-Oil was processed successfully with the development catalyst
- *Conditioning moves product spectra in the desired direction*
- Obtained LCO yields rise, but a more would be desirable – not feasible with this method

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Thank you for your Attention

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