

Pilot plant studies of hydrotreating catalysts

Studies of catalysts for hydrotreating lubricant base oil delivered similar results from conventional and high throughput pilot plants

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Proposed catalyst systems for a lubricant base oil hydrotreater were evaluated with two pilot plant studies. Both studies compared two different catalyst loading schemes – System A and System B – where System A outperformed System B for hydrodesulphurisation (HDS) and hydrodenitrogenation (HDN).

One pilot plant is a conventional unit with a single reactor pilot and an available catalyst volume of above 500ml. The second is an Avantium Flowrence unit with 16 parallel single pellet string reactors (SPSRs), each of which has an internal diameter (ID) of 2.6mm and an available catalyst volume of 1.0ml. In the conventional unit, the catalyst schemes were tested one at a time, without replication, while in the Avantium Flowrence unit, the schemes were tested in parallel – at two different space velocities and in quadruplicate for increased accuracy.

The SPSRs in the Avantium unit were fitted into a commercially available Flowrence XR¹ system, which ensures stable and highly accurate control of gas flow, liquid flow, and pressure across all reactors. Performance data like hydrogen consumptions and liquid product properties were determined independently per reactor. For this, the products from each SPSR were collected separately and various offline analyses performed, for instance for distillation, sulphur, nitrogen, and aromatics.

Due to the excellent hydrodynamics,^{2,10} of the SPSR and sophisticated process control, the Avantium unit achieved high reproducibility, resulting in average deviations of

Comparison of lubricant base oil groups				
Group	Sulphur, wt%		Saturates, wt%	VI
I	>0.03	and/or	<90	80-119
II	≤0.03	and	≥90	80-119
III	≤0.03	and	≥90	≥120
IV	All polyalphaolefins (PAOs)			
V	All stock not included in Groups I-IV. Includes pale oils and non-PAO synthetics			

Table 1

less than 0.2 wtppm for HDS and HDN across the quadruple reactors with the same loading scheme.

Results from the conventional pilot plant corresponded closely to results from the Avantium pilot plant. For Catalyst System A, the relative average deviations were less than 1% for HDS and HDN. For Catalyst System B, all relative HDS deviations and two of three HDN deviations were less than 1%.

These observations indicate transitively that the Avantium unit is a suitable alternative to the conventional pilot plant for the customer's lube oil hydrotreater. It is in fact preferable if one accounts for the advantages of high throughput technology: parallel testing, lower cost and feed amounts, and increased flexibility on testing more options. Moreover, due to the small scale of testing, safe operation can be accomplished in a laboratory setting which would be difficult to achieve with the same number of reactors at conventional scale.

This particular study considered base oil hydrotreating for a single feedstock, but Avantium equipment and methodology also can be employed to evaluate several feedstocks. Moreover, the same technology has been used successfully

to examine other fixed bed catalytic processes, including hydrocracking, hydrodewaxing, catalytic reforming, and hydroisomerisation.

Lubricant base oil classification and preparation routes

Depending on the preparation, lubricant base stocks are classified into different groups.³ Groups I, II, and III are manufactured from paraffinic crudes in refineries. They are commonly called mineral base oils or petroleum base stocks to differentiate them from synthetic base stocks, such as those prepared with polyalphaolefins (PAO, Group IV). Group V stocks include all remaining pale oils (naphthenic base oils), which are manufactured from naphthenic crudes, and other synthetic base materials (see Table 1).

The vast majority of lubricants contain Group I or II base stocks. In 1999, the National Advertising Division of the United States Better Business Bureau declared that automotive lubricants made using Group III base stocks could be labelled 'synthetic' due to the very severe processing conditions required to produce them, and because the performance of Group III lubricants was comparable to that provided by PAO. Table 1 compares important properties: sulphur content, satu-

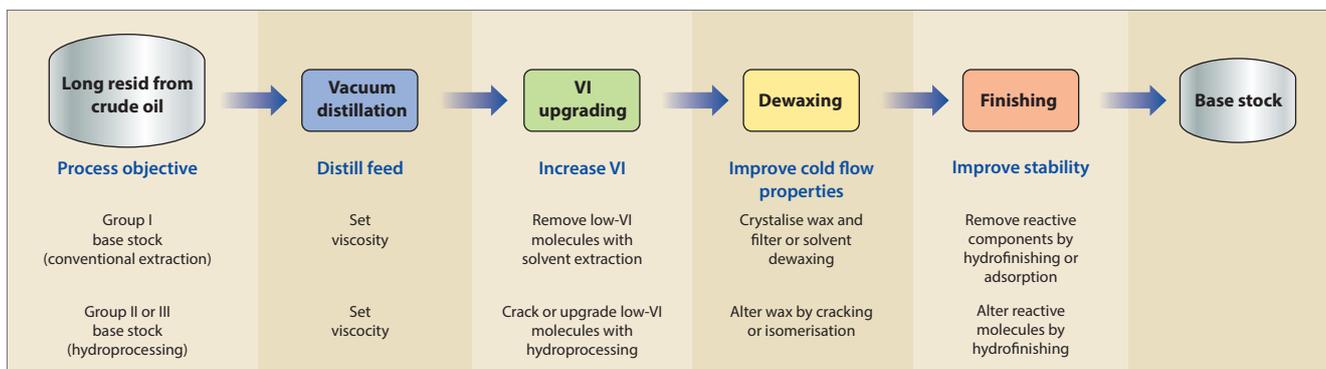


Figure 1 Preparation routes for Groups I, II, and III lubricant base stocks. Adapted from Reference 1, Figure 34.5

rates content, and viscosity index (VI) for all five major groups.

Figure 1 illustrates different routes for preparing Groups I-III base stocks; note that hydroprocessing plays at least some role in each route. Preparation of Group I base oils entails distillation to set viscosity, solvent extraction to remove aromatics and other low-VI molecules, wax removal – by extraction or crystallisation – to improve cold-flow properties, and finishing, which removes remaining impurities and improves both colour and colour stability. Preparation of Groups II and III stocks, often called premium base stocks, relies extensively on hydroprocessing where aromatics are removed with saturation, and wax is removed either with selective hydrocracking or hydroisomerisation.

'Plus' categories are recognised informally for marketing reasons. Group I+ has VIs from 103 to 108. Group II+ has VIs from 111 to 119. Group III+ has VIs >130 for light neutral base stocks, and Group IV+ has VIs from 5 to 15 higher than conventional 1-decene.

Premium base stocks are preferred because they have:

- Lower viscosity, which increases fuel economy during cold starts and reduces engine friction
- Lower volatility, which reduces oil losses and, consequently, reduces emissions
- Improved oxidative and thermal stability
- Improved lubricant performance across a wide range of temperatures, allowing an engine to crank at sub-zero temperatures and also provide superior lubrication during high temperature operation

Hydroprocessing in lubricant feedstock conversion

Traditional solvent based lube oil plants are designed for a specific range of crude oils due to the inherent limitations of solvent extraction units.⁴ If the aromatics content of a crude oil is too high, aromatics extraction will be a bottleneck, and the distillation, dewaxing, and hydrofinishing sections of the production train will be under-utilised, resulting in a low base stock yield. On the other hand, if the wax content is too low, a wax crystalliser may not function efficiently.⁵

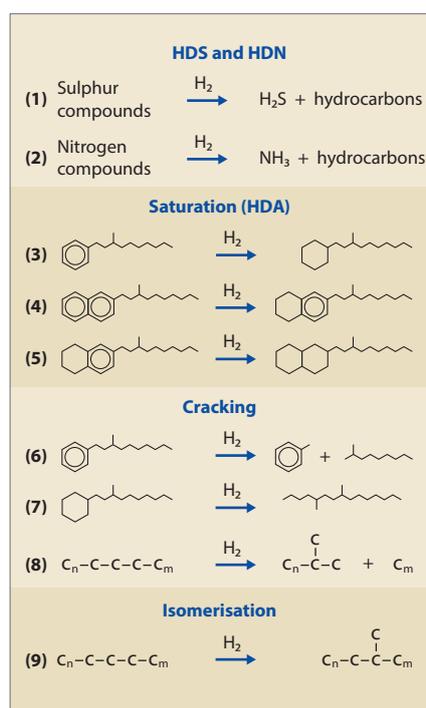


Figure 2 Important chemical reactions in base oil hydrocracking.

- (1) hydrodesulphurisation (HDS).
- (2) hydrodenitrogenation (HDN).
- (3), (4), and (5) hydrodearomatisation (HDA).
- (6) hydrocracking (ring dealkylation).
- (7) hydrocracking (ring opening).
- (8) hydrocracking (hydrodewaxing).
- (9) catalytic isomerisation

Applying hydroprocessing technologies adds feedstock flexibility by increasing the practical range of crude oil properties.

Figure 2 illustrates important chemical reactions in base oil hydroprocessing. While HDS and HDN remove reactive heteroatoms, which accelerate oxidative degradation, saturation and ring opening convert low VI molecules into high VI molecules. Hydrodewaxing removes waxy n-paraffins by converting them into lighter molecules, such as diesel and naphtha constituents, over catalysts containing ZSM-5 or similar materials.⁶ Isomerisation converts n-paraffins into i-paraffins and by this removing waxy molecules.

One considers a lube stock preparation plant in which aromatics removal is accomplished by saturation, that is hydrodearomatisation (HDA). Up to a point – below the aromatics crossover temperature⁷ and within guidelines for safe operation – HDA can be increased simply by raising the average catalyst temperature in the hydroprocessing unit. The same applies if wax removal is accomplished by hydrodewaxing. Higher temperature is not necessarily beneficial in hydroisomerisation, where cracking is undesirable.⁸

Base oil pilot plant studies

For lube base stock production, modest differences in feedstocks, catalysts, and process configuration can have a major impact on product quality. Refiners and catalyst vendors conduct pilot plant studies to ensure that changes are practical and economically viable. Relevant and scalable test data are required

and test results from pilot plant studies should enable the reliable prediction of the performance of commercial scale units.

Conventional hydroprocessing pilot plant studies are relatively expensive to build and operate. They employ relatively large reactors, typically with an inside diameter (ID) ranging from 1.2-2.5cm and a catalyst bed length of 30-80cm. Consequently, it becomes impractical to evaluate more than a few alternative catalyst loading schemes or different sets of process conditions within a given time frame. Moreover, replicate tests are seldom even considered, which makes these tests simply spot measurements of catalyst activity.

Regardless of size, pilot plant reactors have inherent limitations for testing catalysts with commercial sizes and shapes. To compensate for channelling, wall effects, and back-mixing, catalysts are diluted with non-porous inert particles of suitable size. The impact of diluent size has been identified experimentally.⁹ Even with a suitable diluent, reactors must be carefully packed to ensure even flow of fluids through the bed, to approximate plug-flow behaviour.

It is intuitive to expect that larger reactors are less susceptible to size-related limitations. However, recent research by Moonen *et al.* shows that SPSRs are no more susceptible to wall effects, channelling, and back-mixing than properly utilised bench-scale reactors.¹⁰ With experiments, they showed an excellent correspondence for gasoil HDS between an Avantium SPSR unit and a bench-scale unit with a catalyst volume of 225ml – more than 300 times the volume of an SPSR. With rigorous modelling of the corresponding hydrodynamics, they explained why results from the smaller unit are so similar to the larger. Moreover, the book chapter by van der Waal *et al.* discusses the engineering concepts of the Flowrence parallel small-scale reactor systems. This included the influence of catalyst particle size, flow patterns, pressure drop, and temperature profiles on the quality of catalytic results and, as is exem-

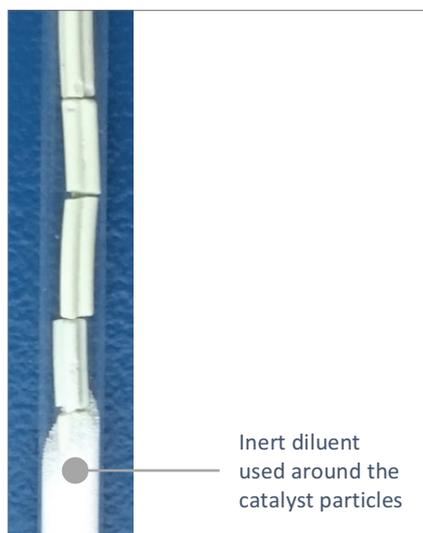


Figure 3 Extrudates automatically line up in a string when loaded into a single pellet string reactor

plified by several case studies on Fischer-Tropsch, oxidative coupling of methane and hydrotreating to obtain ultra low sulphur diesel.

This particular study looked at base oil hydrotreating – specifically HDS, HDN, and HDA. It is also possible to apply high throughput pilot plant equipment and methodology to other base oil processes, including hydrocracking, hydrode-waxing, catalytic reforming, and hydroisomerisation.

Advantages of single pellet string reactors

SPSR units have many advantages. They require far less catalyst and feed. They provide excellent temperature control and reproducible reactor loading due to the fact that the diameter of the extrudates is just slightly smaller than the reactor diameter. In addition, extrudates automatically align as a string of extrudates (see **Figure 3**) which, in combination with the narrow reactor, avoids maldistribution of gas and liquid over the catalyst bed, thereby eliminating catalyst bed channelling and incomplete catalyst wetting. When an inert diluent is used, it can be introduced after catalyst pellets are loaded over the full length of the tube, resulting in embedded extrudates while not going between them.

Due to the size and feedstock consumption of such small-scale reactor systems, it becomes feasible to implement these in a compact platform while still being able to operate under relevant conditions. This enables not only testing of multiple options under identical conditions, but also allows true replication of tests. This increases data quality and allows the estimation of

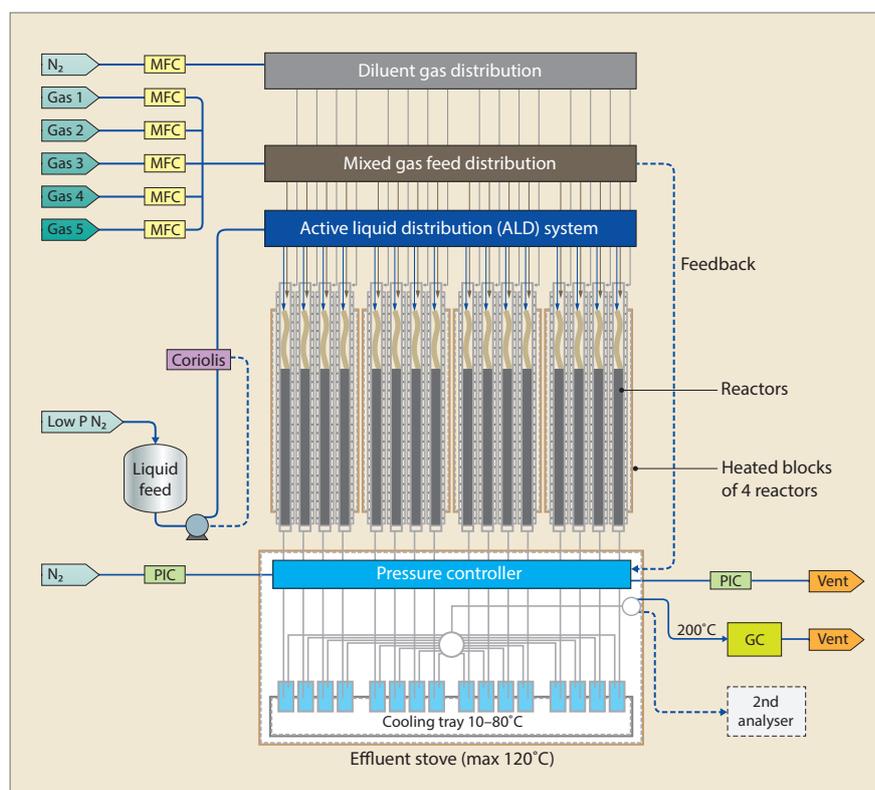


Figure 4 Schematic representation of the Avantium pilot plant with 16 parallel SPSRs

confidence intervals, thus improving over the more common 'single point' tests.

Experimental

Purpose

The primary aim of this experiment was to evaluate two catalyst systems for a commercial lubricant base oil hydrotreater. A secondary aim was to compare the performance of a single reactor conventional pilot plant with an Avantium pilot plant system employing 16 SPSRs.

Equipment, feedstock and reactor loading

Figure 4 presents an overview of the Avantium Flowrence XR unit. Hydrogen feed gas was equally distributed to 16 reactors, and the pressure of each channel was measured before and after the catalyst bed by using electronic pressure sensors. Based on these measurements, the reactor pressures were individually controlled to ensure equal process conditions. The oil feed (see properties in Table 2) was uniformly distributed to all 16 channels. The exact liquid flow rate of each of the individual liquid feed lines was measured and actively controlled to ensure even distribution of the oil feed to each of the reactors within 0.5% relative standard deviation between the reactors. When necessary for sulphiding or passivation, the feed can be dosed with additives from a separate pump.

The SPSRs are made of a stainless steel tube with 2.6mm inside diameter, a maximum catalyst bed volume of 1.0ml, and 560mm length includ-

Selected properties of the tested base oil feedstock

Parameter	Value
API @ 60 F	20
Sp.gr. @ 60 F	0.9300
Sulphur, wt%	0.560
Nitrogen (total), wtppm	1000
Aniline Point, °F	170
Viscosity cSt @ 100 F	170
Viscosity Index (calc)	140
ASTM D-2887 (SimDis)	°F
IBP	565
5%	697
20%	760
40%	803
60%	840
80%	891
90%	829
95%	958
EP	1024

Table 2

ing a conditioning zone at the top of the reactor.

As mentioned above, the loaded extrudates automatically aligned as a string of extrudates (see Figure 3), and porous ceramic beads with diameters of 0.07mm were used to embed them.

The reactor tubes were inserted into four separate isothermal heating blocks. Each reactor block was loaded with four reactor tubes which can be operated at the same temperature without impact on any other block. All process control data were recorded per reactor while liquid products were collected in separate sample vials for 12 reaction conditions (2 catalyst systems * 3 temperatures * 2 LHSVs). Online gas chromatography was used to analyse and quantify the light gas content.

In both studies, two catalyst configurations were tested: System A and System B. The commercial catalysts were loaded to match the recommendations of the catalyst vendors. Note that for each condition catalysts were loaded in quadruplicate. Total catalyst volume was varied to achieve the low and high desired LHSV.

Experimental protocol

The experimental protocol is summarised in Figure 5 and is based on customer requirements. The first step is sulphidation to convert the catalyst (typically metal oxides supported on a high surface area Al_2O_3) into sulphides via reactions with hydrogen and H_2S .¹¹ For this study, a liquid activation was chosen, where the H_2S for sulphiding comes from organic sulphur compounds in feedstocks augmented by dimethyldisulphide (DMDS) or butyl sulphides such as Lubrizol's SulphrZol 54.

In this case, for sulphiding the test feedstock was spiked with sufficient DMDS to bring the sulphur concentration to 2.0 wt%. The reactor pressure (hydrogen partial pressure) was maintained constant in a moderate-to-high range. Temperatures were as specified by the catalyst vendor.

During the 14-day catalyst conditioning phase, normal test feed passed through the reactors under vendor specified conditions. At the end of the conditioning phase came three successive periods of line out (24 hours) at a given temperature followed by liquid product collection (88 hours) at that temperature. Compositions of gaseous products were determined online. Liquid effluent was collected at each condition for fractionation and analysis. Daily sulphur and nitrogen analyses were performed to track HDS and HDN activity.

Customer's single reactor experimental protocol

The customer's conventional single reactor protocol is very similar to the Avantium unit, differing in the number of parallel reactors, scale of testing/volume processing, and process complexity. Like in the

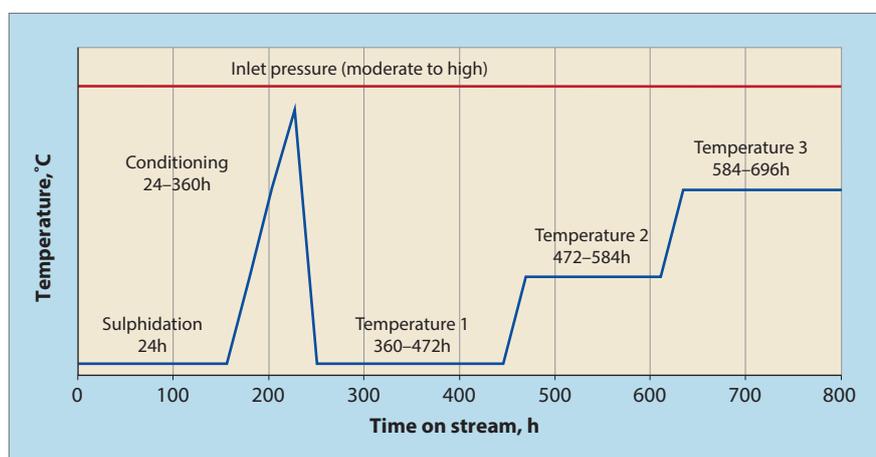


Figure 5 Experimental protocol

Avantium unit, the first step is sulphidation to convert the catalyst (metal oxides supported on a high surface area Al_2O_3) into sulphides via reactions with hydrogen and H_2S . For this study, a liquid activation was chosen, where the H_2S for sulphiding come from organic sulphur compounds in feedstocks augmented by dimethyldisulphide (DMDS) or butyl sulphides such as Lubrizol's SulphrZol 54.

In this case, for sulphiding the test feedstock was spiked with sufficient SulphrZol 54 to increase the sulphur concentration. The reactor pressure (hydrogen partial pressure) was moderate-to-high, while temperatures were as specified by the catalyst vendor.

During the 14-day catalyst conditioning phase, normal test feed passed through the reactors under customer specified conditions. At the end of the conditioning phase came three successive periods of line out at a given temperature followed by liquid product collection at that temperature. Liquid effluent was collected at each condition for further product analysis.

Results and discussion

Temperature and pressure

Figure 6 presents the time on stream, temperatures, and pressures used for the experiment. Note the ramp to a peak temperature during the second stage of sulphiding. All temperatures were controlled within 0.5°C while maintaining target inlet pressure.

Figure 7 presents the box plot of the inlet reactor pressure per reactor. As mentioned above, each reactor pressure was actively controlled using Avantium's proprietary reactor pressure control (RPC) technology, leading to a bandwidth within ± 0.01 bar across all 16 reactors.

Material balance

Figure 8 depicts the mass balances for all 16 reactors separated by the catalyst system with a more detailed statistical analysis in **Figure 9**. The average mass balance obtained was 99.5% with 95% of the data falling within a range of $\pm 1.5\%$ (please note that two reactors have some minor outliers), which indicates that lube oils such as this can be con-

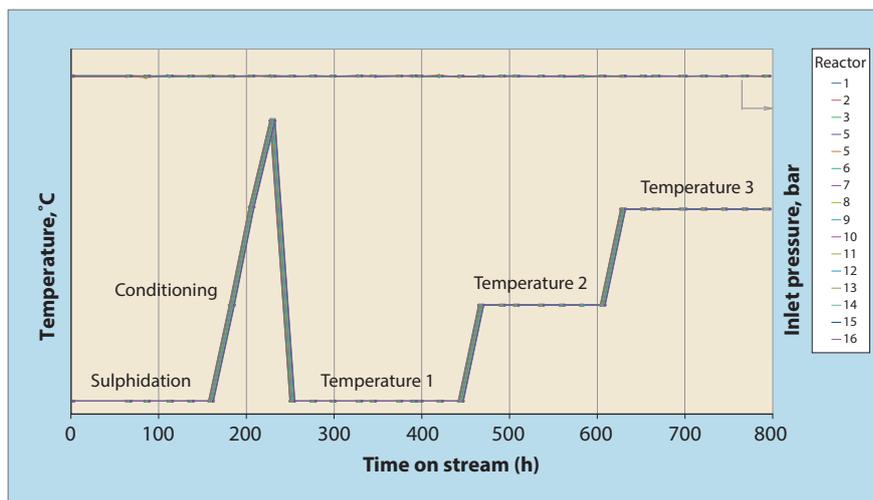


Figure 6 Temperature and pressure profile for all 16 parallel reactors over the duration of the experiment

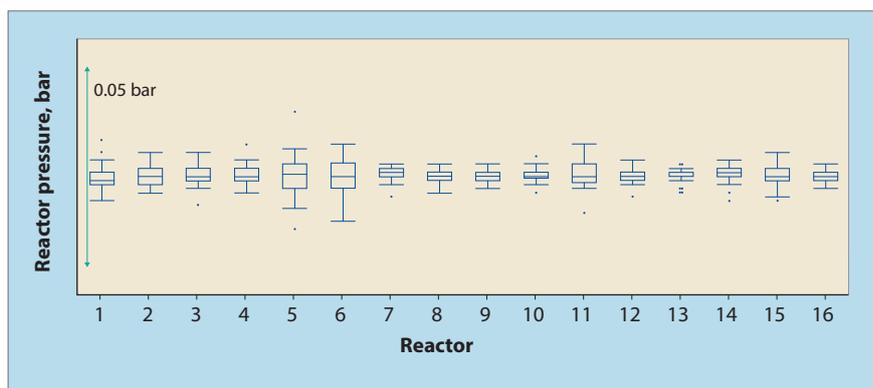


Figure 7 Reactor inlet pressure trend analysis over time per reactor

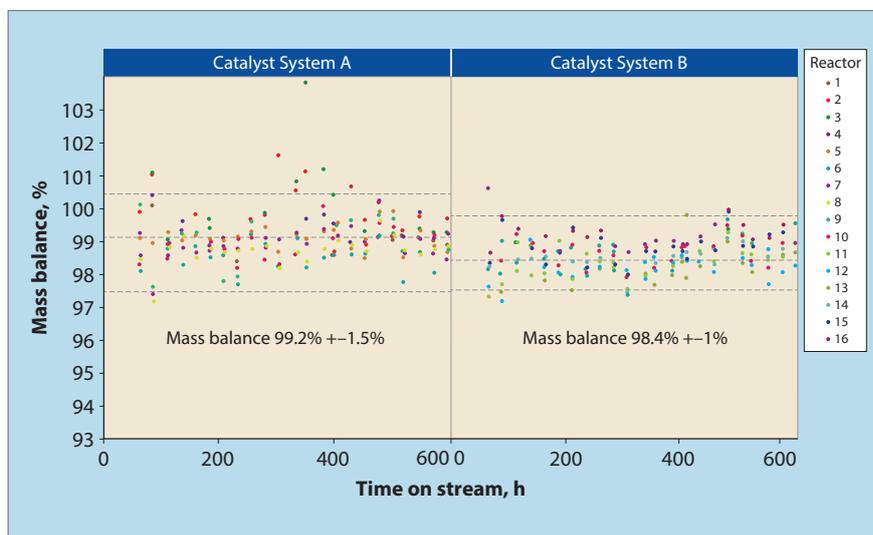


Figure 8 Mass balances for all 16 reactors throughout the entire test programme. The average mass balance was 99.5% with 95% of the data falling within a range of $\pm 1.5\%$

trolled and distributed evenly over all 16 reactors using Avantium's advanced liquid distribution (ALD) technology.

Hydrogen consumption

Figure 10 shows the hydrogen consumption data for all 16 reactors.

The values are based on online gas chromatography measurement. The colours correspond to the different LHSVs applied while the green line shows the temperature profile applied. The data show that H_2 consumption increases with temperature and decreases with LHSV,

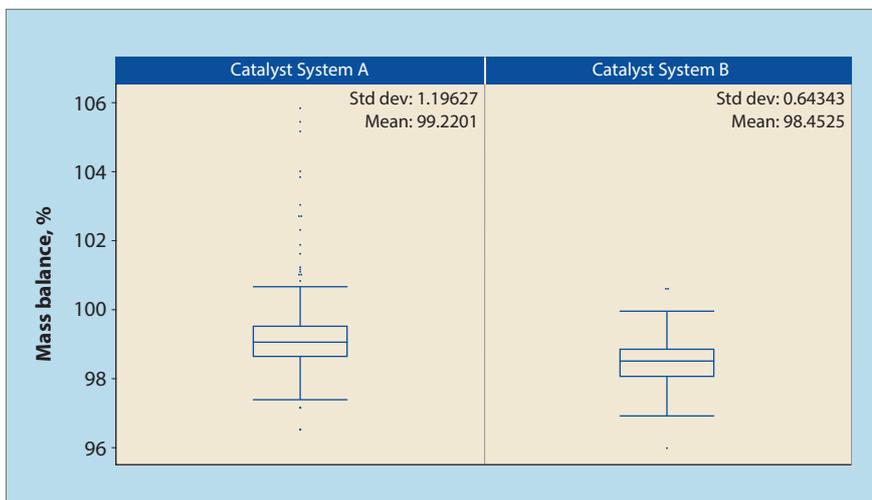


Figure 9 Statistical analysis of the mass balances per catalyst system throughout the entire test programme

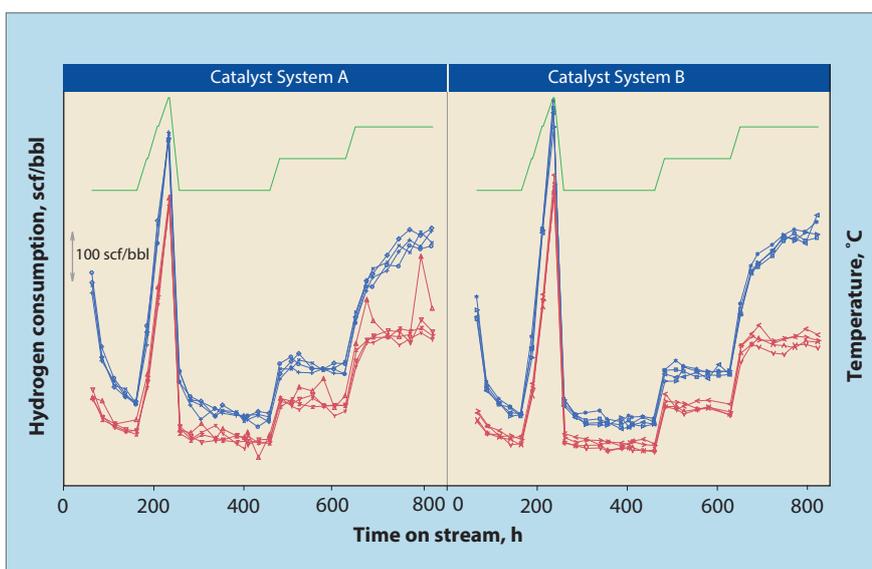


Figure 10 Hydrogen consumption data in dependence of time on stream. The blue line represents the high LHSV while the red one the low LHSV. The green line represents the temperature pattern applied. Note the very high H₂ consumption at the peak conditioning temperature



Figure 11 Liquid product samples for 12 reaction conditions. Lighter colour is observed at higher severity, which corresponds to greater HDS and HDA

which is consistent with the expectations. H₂ consumption is higher for catalyst System B compared to System A, which could indicate a higher activity for HDS, HDN, and HDA. Note the very high H₂ consumption for catalyst System B at the peak sulphiding temperature.

It is important to note that samples were measured in quadrupli-

cate for statistical purposes. The average bandwidth is well within +/-20 scf/bbl, indicating very good reproducibility.

Sulphur and nitrogen removal

Liquid samples were collected and analysed offline. As mentioned, 12 composite samples were produced (2 catalyst systems * 3 temperatures

* 2 LHSV). An example is shown in **Figure 11** where the lighter colour is observed at higher severity, which corresponds to greater HDS and HDA. Please note that the composites were analysed by the customer for different properties such as distillation, aromatics, and delta aniline point.

Sulphur and nitrogen in the liquid products were analysed daily and used to calculate percent HDS and HDN (see **Figures 12** and **13**). Expected trends are observed: sulphur and nitrogen removal increases with increasing temperature and at lower LHSV. The observed values for conversion are in line with client results from the conventional pilot plant.

It is important to note that the high discriminatory power of Avantium's reactor technology can also be observed in the HDS activity shown in **Figure 14**. For catalyst System B, at the highest temperature the conversion at high LHSV is 99%, whereas at the low LHSV it is 99.999%. Moreover, reproducibility between the quadruples is excellent and consistent with the perfect hydrodynamic achieved in the unit. Please note that the HDN activity shows a similar result.

Comparison of results with the customer's single reactor unit

The customer executed a similar study on a larger scale without duplications. **Figures 15** and **16** show a comparison of the results from these tests and indicate that results from the Avantium unit are in line with client data obtained from much larger scale reactors. Results from the two pilot plants corresponded closely. For catalyst System A, average deviations were less than 1% for HDS and HDN. For catalyst System B, all HDS deviations and two of three HDN deviations were less than 1%.

Conclusions

Two catalyst loading schemes were evaluated for a change-out in a commercial lubricant base oil hydrotreater. The tests were executed in a unit with single pellet string reactors (SPSRs) from Avantium as well as in a conven-

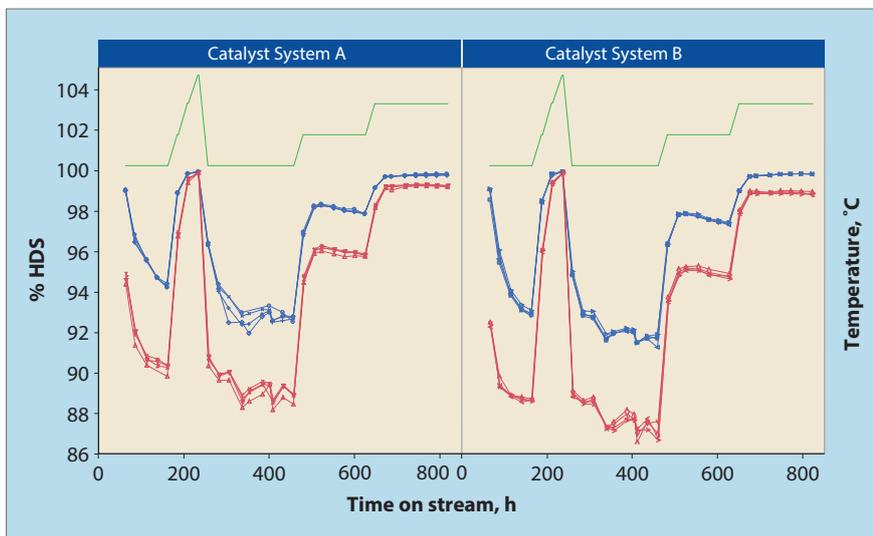


Figure 12 %HDS in dependence of time on stream. The blue line represents the high LHSV while the red one the low LHSV. The green line represents the temperature pattern applied

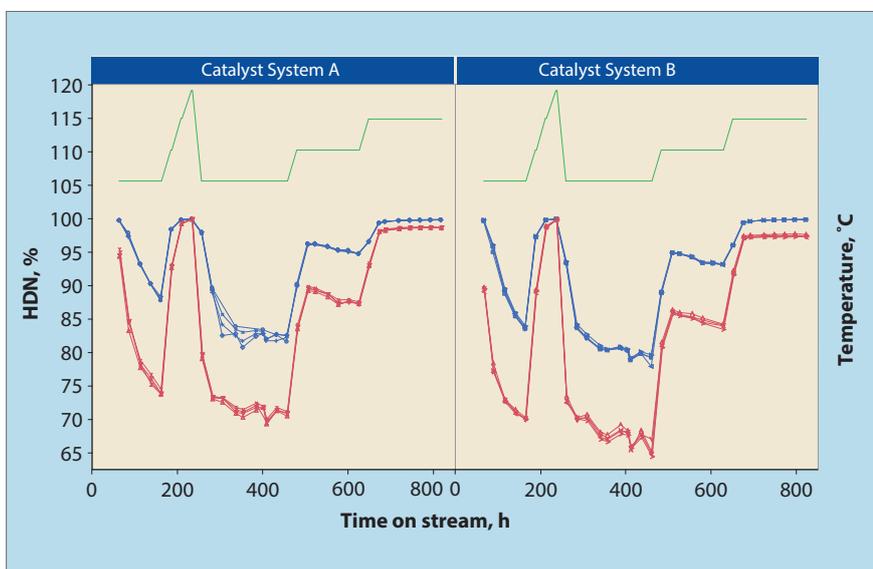


Figure 13 %HDN in dependence of time on stream. The blue line represents the high LHSV while the red one the low LHSV. The green line represents the temperature pattern applied

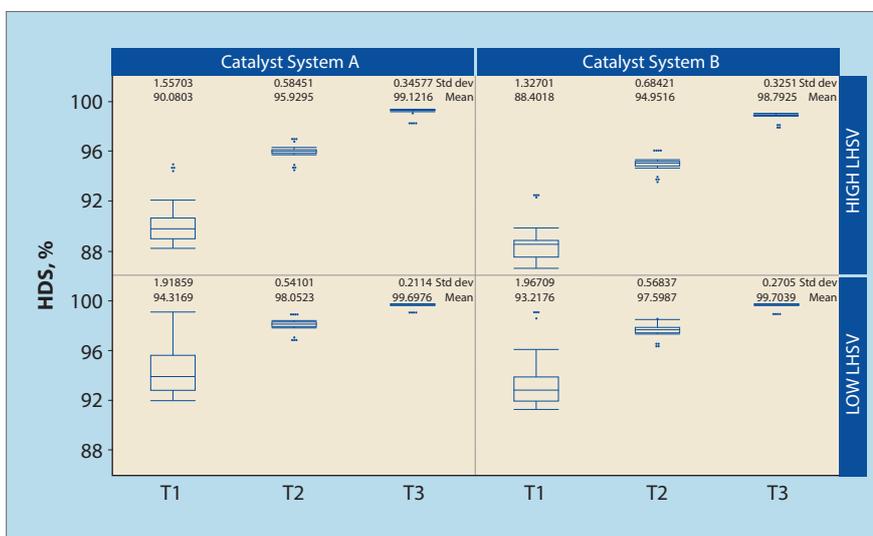


Figure 14 Statistical analysis of HDS capacity at the three selected temperature levels measured

tional pilot plant. Due to the inherent limitation of a single flow unit, only one LHSV was evaluated at large scale, while in the parallel testing unit multiple options were evaluated. A comparison of both units showed that the small-scale results are well in line with the conventional technology, especially for HDS and HDN. The study demonstrated several advantages for SPSR systems, including the following:

- Avantium systems can replicate results from conventional pilot plants
- A feed far heavier than diesel can be handled and processed reliably
- Different catalyst schemes can be tested simultaneously at several sets of process conditions
- Replication provides the means for statistical evaluation of data
- Hydrogen consumption can be determined with high repeatability

With the additional information about the LHSV within the same testing time, the customer could evaluate more economic options and ultimately make a better decision. This particular study considers base oil hydrotreating; however, Avantium's Flowrence equipment and methodology can also be employed to study other base oil processes, including hydrocracking, hydrodewaxing, catalytic reforming, and hydroisomerisation.

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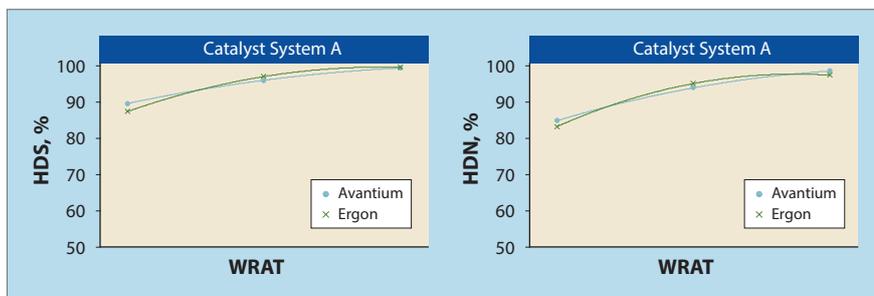


Figure 15 Catalyst System A: Comparison of %HDS and %HDN in one pilot plant run (Ergon) with results from the Avantium unit

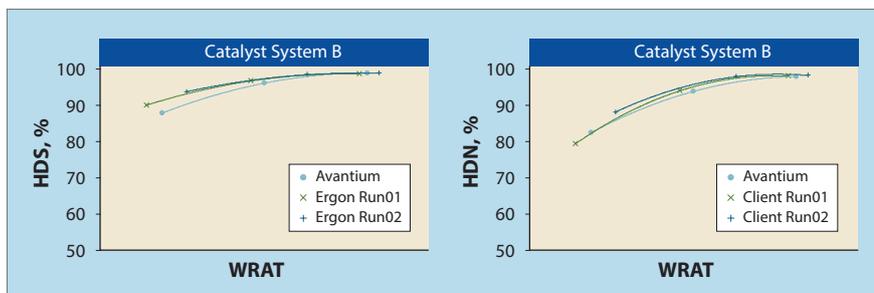


Figure 16 Catalyst System B: Comparison of % HDS and % HDN in two customer pilot plant runs (Ergon Run01 and Run02) with results from the Avantium unit

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