⑫ EUROPEAN PATENT APPLICATION

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�54 **Method of improving lube oil yield by wax isomerization employing low treat gas rates.**

�57 Increased yields of 5.6-5.9 cSt (5.6 to 5.9 Mm²/s)/100°C lube oil are obtained in wax isomerization processes by utilizing low treat gas rates during the wax isomerization step. The isomerate boiling in the lube oil range obtained by using low treat gas rates during isomerization possess lower dry wax content in addition to possessing a higher fraction of 5.6-5.9 cSt (5.6 to 5.9 Mm²/s)/100°C oil.

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Description

METHOD OF IMPROVING LUBE OIL YIELD BY WAX ISOMERIZATION EMPLOYING LOW TREAT GAS RATES

5 DESCRIPTION OF THE INVENTION

It has been discovered that the yield of lube oil in the viscosity grade 5.6-5.9 cST/100°C produced by the hydroisomerization of wax can be improved by practicing the isomerization process under low treat gas rate. In addition to the improved yield of lube oil on the isomerate the wax content of lube oil fraction of the isomerate is lower when the isomerization is practiced under a low treat gas rate.

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DETAILED DESCRIPTION OF THE INVENTION

A method is disclosed for improving the yield of high viscosity index lube oil obtained by the isomerization of waxes. The improvement comprises operating the wax isomerization process at low treat gas rates, rates in the range of 500 to 5000 SCF/bbl, H_2, preferably 2000 to 4000 SCF/bbl, H_2, most preferably about 2000 to 3000

15 SCF/bbl, H_2. All other isomerization process conditions are maintained in their typical, standard ranges, i.e. temperature in the range of 270-400°C, preferably 300 to 360°C, pressures of 500 to 3000 psi H_2, preferably 1000 to 1500 H_2, and a space velocity in the range of 0.1 to 10 V/V/hr, preferably 1 to 2 V/V/hr.

Operating in the low treat gas rate mode produces an increased yield of isomerate oil boiling in the 370°C$^+$ range possessing a viscosity grade in the range of about 5.6-5.9 cST/100°C. This oil fraction also possesses a

20 reduced wax content and therefore should lead to a reduced load on the dewaxer.

The wax which is isomerized may come from any of a number of sources. Synthetic waxes from Fischer-Tropsch processes may be used, as may be waxes recovered from the solvent or autorefrigeration dewaxing of conventional hydrocarbon oils, as well as mixtures of these waxes. Waxes from dewaxing conventional hydrocarbon oils are commonly called slack waxes and usually contain an appreciable amount of

25 oil. The oil content of these slack waxes can range anywhere from 0 to 45 percent or more, usually 1 to 30 percent oil. For the purposes of this application, the waxes are divided into two categories: (1) light paraffinic waxes boiling in the range about 300 to 580°C; and (2) heavy microwaxes having a substantial fraction ($\geqq$ 50 percent) boiling above 600°C.

Hydroisomerization is performed over any of the standard hydroisomerization catalysts which contain a

30 hydrogenation metal selected from Group VI and Group VIII mixtures, and Group VIII metals, preferably the Group VIII metals, more preferably the noble Group VIII metals, most preferably platinum. Metal loading ranges between 0.1 to 5.0 wt% metal, preferably 0.1 to 1.0 wt% metal most preferably 0.2 to 0.6 wt% metal.

The hydrogenation metal component is supported on a halogenated refractory inorganic metal oxide support, preferably alumina or silica-alumina, most preferably the transition aluminas, e.g. gamma alumina. The

35 halogen is usually chlorine or fluorine or mixture thereof, preferably fluorine, net halogen content in the range 1 to 10 wt%, preferably 2 to 8 wt%.

The preferred catalyst contains a hydrogenation metal component which is a Group VIII metal or mixture thereof, preferably noble Group VIII metal, most preferably platinum on a fluorided alumina or material containing alumina, preferably alumina or material consisting predominantly (i.e. >50%) or alumina, most

40 preferably gamma or eta alumina wherein said catalyst in its as introduced to waxy feed form is characterized by possessing (1) a hydrate level of 60 or less, preferably 10 to 60 determined as the relative amount of hydrate represented by a peak in the X-ray diffraction (XRD) pattern at $2\theta = 5.66$Å when a hydrate level of 100 corresponds to the XRD peak height exhibited by a standard material constituting 0.6 wt% pt on 150 m²/g γ alumina containing 7.2 wt% F wherein the fluorine has been deposited using an aqueous solution containing a

45 high concentration of HF, i.e. 10 wt% HF, and greater, preferably 10 to 15 wt% HF and the material dried at 150°C for 16 hrs; (2) a surface nitrogen content N/Al ratio of 0.01 or less, preferably 0.007 or less, most preferably 0.004 or less as determined by X-ray photoelectron spectroscopy (XPS); (3) a bulk fluorine concentration of about 2 to 10 wt% and (4) a surface fluorine concentration, defined as the amount of fluorine present in a layer extending from the surface of the particle (e.g. 1/16 inch extrudates) to a depth of 1.100 inch

50 of less than 3 wt%, preferably less than 1 wt%, most preferably less than 0.5 wt% fluorine in that zone provided that the surface fluoride concentration is less than the bulk fluoride concentration.

The fluoride content of the catalyst can be determined in a number of ways.

One technique analyzes the fluorided catalysts using oxygen combustion methodology which is well established in the literature. Approximately 8-10 mgs of sample is mixed with 0.1 g benzoic acid and 1.2 gms of

55 mineral oil in a stainless steel combustion capsule which is mounted in a 300 mL. Parr oxygen combustion bomb. The "sample" is purged of air and subsequently combusted under 30 Atms of pure oxygen. Combustion products are collected in 5 mL. of deionized water. Once the reaction has gone to completion (about 15 minutes), the absorbing solution is quantitatively transferred and made to fixed volume.

Fluoride concentration of the sample is determined by ion chromatography analysis of the combustion

60 product solution. Calibration curves are prepared by combusting several concentrations of ethanolic KF standards (in the same manner as the sample) to obtain a 0-10 ppm calibration range. Fluoride concentration of the catalyst is calculated on an ignition-loss-free-basis by comparison of the sample solution response to that of the calibration curve. Ignition loss is determined on a separate sample heated to 800 degrees F for at

least 2 hours. Ion chromatographic analysis uses standard anion conditions.

Another procedure employs the use of fluoride distillation with a titrimetric finish. Fluorides are converted into fluorosilicic acid (H_2SiF_6) by reaction with quartz in phosphoric acid medium, and distilled as such using super heated steam. This is the Willard-Winter-Tananaev distillation. It should be noted that the use of super heated, dry (rather than wet) steam is crucial in obtaining accurate results. Using a wet steam generator yielded results 10-20% lower. The collected fluorosilicic acid is titrated with standardized sodium hydroxide solution. A correction has to be made for the phosphoric acid which is also transferred by the steam. Fluoride data are reported on an ignition-loss-free-basis after determination of ignition loss on a sample heated to 400 degrees C for 1 hour.

A preferred catalyst is a catalyst prepared by a process involving depositing a hydrogenation metal on an alumina or material containing alumina support, calcining said metal loaded support typically at between 350 to 500°C, preferably about 450 to 500°C for about 1 to 5 hrs, preferably about 1 to 3 hrs and fluoriding said metal loaded support by using a high pH fluorine source solution to a bulk fluorine level of about 8 wt% or less (e.g., 2 to 8 wt%), preferably about 7 wt% or less, said high pH source solution being at a pH of 3.5 to 4.5 and preferably being a mixture of NH_4F and HF followed by rapid drying/heating in a thin bed or rotary kiln to insure thorough even heating in air or oxygen containing atmosphere, or inert atmosphere to a temperature between about 350 to 450°C, in about 3 hours or less, preferably 375 to 400°C and holding, if necessary, at the final temperature for a time sufficient to reduce the hydrate and nitrogen contents to the aforesaid levels (e.g. 1 to 5 hours), or by using a low pH fluorine source solution having a pH of less than 3.5 using aqueous solutions of HF or appropriate mixtures of HF and NH_4F to a bulk fluorine level of about 10 wt% or less (e.g., 2 to 10 wt%), preferably about 8 wt% or less followed by drying/heating in a thin bed or rotary kiln to a temperature of about 350 to 450°C, preferably 375 to 425°C in air or oxygen containing atmosphere, or an inert atmosphere and holding at that temperature, if desired, for 1 to 5 hours. The alumina or alumina containing support material is preferably in the form of extrudates and are preferably at least about 1/32 inch across the longest cross sectional dimension.

If the low pH prepared catalyst is first charged to a unit, the catalyst must be held at the final activation temperature for longer than 5 hours, preferably longer than 10 hours and preferably at temperatures of 400 to 450°.

The above catalysts typically contain from 0.1 to 5.0 wt% metal, preferably 0.1 to 1.0 wt% metal, most preferably 0.2 to 0.6 wt% metal.

The dried/heated catalyst has a surface nitrogen content of 0.01 or less N/Al by X-ray photoelectron spectroscopy (XPS) preferably 0.007 N/Al (by XPS).

The catalyst, following the aforesaid heating, can be charged to the isomerization reactor and brought quickly up to operating conditions. Alternatively the catalyst following the aforesaid heating prepared using the high pH solution technique can be hydrogen activated preferably in pure or plant hydrogen (60-70 vol% H_2), at 350 to 400°C, care being taken to employ short activation times, from 1 to 24 hours, preferably 2 to 10 hours being sufficient. Long activation times (in excess of 24 hours) have been found to be detrimental to catalyst performance. By way of comparison, catalysts made using the low pH solution technique can be activated in pure or plant hydrogen at 350 to 500°C for from 1 to 48 hours or longer. In fact, if such low pH prepared catalysts are not heated first, then it is preferred that they be subsequently activated at more severe conditions, i.e., for longer times and/or at higher temperatures. On the other hand, if they are heated first, then moderate activation procedures similar to those employed with catalysts made from the higher pH solution treatment will suffice.

A typical activation profile shows a period of 2 hours to go from room temperature to 100°C with the catalyst being held at 100°C for 0 to 2 hours then the temperature is raised from 100 to about 350 over a period of 1 to 3 hours with a hold at the final temperature of from 1-4 hours. Alternatively the catalyst can be activated by heating from room temperature to the final temperature of 350-450°C over a period of 2-7 hours with a hold at the final temperature of 0-4 hours. Similarly activation can be accomplished by going from room temperature to the final temperature of 350-450°C in 1 hour.

It is possible to dispense with a separate activation procedure entirely (provided the catalyst has first been heated in air). In these instances, the calcined catalyst is simply charged to the reactor, heated to just above the melting point of the wax feed, feed and hydrogen introduced onto the catalyst, and thereafter the unit brought quickly up to operation conditions.

Another preferred catalyst comprises a hydrogenating metal on fluorided alumina or material containing alumina support made by depositing the hydrogenation metal on the support and fluoriding said metal loaded support using acidic fluoride sources such as HF by any convenient technique such as spraying, soaking, incipient wetness, etc. to deposit between 2-10% F preferably 2-8% F. Following halogenation the catalyst is dried, typically at 120°C and then crushed to expose inner surfaces, the crushed catalyst is double sieved to remove fines and uncrushed particles. This sized catalyst is 1/32 inch or less and typically from 1/64 to 1/32 inch in size across its largest cross-sectional dimension.

The starting particle or extrudate may be of any physical configuration. Thus particles such as cylinders, trilobes or quadri lobes may be used. Extrudates of any diameter may be utilized and can be anywhere from 1/32 of an inch to many inches in length, the length dimension being set solely by handling considerations. It is preferred that following sizing the particle have a length smaller than the initial extrudate diameter.

Following deposition of the hydrogenation metal and the fluoriding of the particle or extrudate, the particle

or extrudate is crushed or fractured to expose inner surfaces.

The crushing is conducted to an extent appropriate to the particle or extrudate with which one is starting. Thus, an extrudate which is 1 foot long and 1/16 inch in diameter would be sized into pieces which range anywhere from 1/64 to 1/32 inch across its longest cross-sectional dimension. Similarly, if the extrudate is only 1/16 inch to begin with it will be enough simply to break it in half, into two 1/32 inch pieces, for example.

Alternatively, one can take a metal loaded support particle which is already about 1/32 inch in size or smaller and fluoride it as described above using HF.

Generally, therefore, the sized material will range in size between about 1/64 to 1/32 inch in size.

The uncalcined sized catalyst is activated in a hydrogen atmosphere such as pure hydrogen or plant hydrogen containing 60 to 70 vol% H_2 by heating to 350 to 500°C, preferably 350 to 450°C for from 1 to 48 hours or longer. The hydrogen activation profiles previously described may similarly be employed here.

This sized catalyst is unexpectedly superior for wax isomerization as compared to the uncrushed particle or extrudate starting material. It has also been discovered that 370+ oil products made using the sized catalyst as compared to the uncrushed or extrudate material starting with wax possessing about 5-10% oil exhibit higher viscosity indexes (VI's) than do 370°C+ oil products made starting with wax possessing 0% oil (on the one hand) and about 20% oil (on the other). Therefore, to produce products having the highest VI one would isomerize wax having from 5-15% oil, preferably 7-10% oil using the "sized" catalyst produced using UF.

As one would expect, isomerization catalysts are extremely susceptible to deactivation by the presence of heteroatom compounds (i.e. N or S compounds) in the wax feed so care must be exercised to remove such heteroatom materials from the wax feed charges. When dealing with high purity waxes such as synthetic Fischer-Tropsch waxes, some precautions may not be necessary. In such cases, subjecting such waxes to very mild hydrotreating may be sufficient to insure protection for the isomerization catalyst. On the other hand, waxes obtained from natural petroleum sources contain quantities of heteroatom compounds as well as appreciable quantities of oil which contain heteroatom compounds. In such instances the slack waxes should be hydrotreated to reduce the level of heteroatom compounds to levels commonly accepted in the industry as tolerable for feeds to be exposed to isomerization catalysts. Such levels will typically be a N content of about 1 to 5 ppm and a S content of about 1 to 20 ppm, preferably 2 ppm or less nitrogen and 5 ppm or less sulfur. Similarly, such slack waxes should be deoiled prior to hydrotreating to an oil content in the range of 0 to 35% oil, preferably 5 to 25% oil. The hydrotreating step will employ typical hydrotreating catalyst such as Co/Mo or Ni/Mo on alumina under standard, commercially acceptable conditions, e.g., temperature of 280 to 400°C, space velocity of 0.1 to 2.0 V/V/hr, pressure of from 500 to 3000 psig H_2 and hydrogen gas rates of from 500 to 5000 SCF/bbl.

It is preferred that the isomerization reaction be conducted to a level of conversion such that about 40% or less, preferably 15 to 35%, most preferably 20 to 30%, unconverted wax remains in the fraction of the isomerizate boiling in the lubes boiling range sent to the dewaxing unit. The fraction of unconverted wax is calculated as unconverted wax/(unconverted wax + dewaxed oil) X 100. The amount of unconverted was in the 370°C+ oil fraction is taken to be the amount of wax removed or recovered from said oil fraction upon dewaxing. The total product from the isomerization unit is fractionated into a lube oil fraction boiling in the 330°C+ range, preferably in the 370°C+ range or even higher. This lube oil fraction is solvent dewaxed, preferably using a 20/80 v/v mixture of MEK/MIBK, and unconverted wax is recycled for further isomerization by being fed either to the fresh feed reservoir or directly to the isomerization unit.

In principle a wax extinction process for maximizing lube yields would involve operation at a very low severity, i.e. where conversion to fuels is at a minimum. Under these circumstances the amount of unconverted wax recycled to the isomerization reactor would be large and differences in catalyst selectivity would be less important.

In practice, however, it is not practical to operate in a low conversion mode. Instead, the operating severity is governed by the need to make a low pour (<-21°C pour point) oil. It has been discovered that low pours cannot be achieved from isomerates made at low conversion. This is unexpected since with natural oils the amount of wax present did not affect the ability to dewax the oil to low target pour point. A critical determinant in reaching low pours is that the amount of wax remaining in the 370°C+ fraction obtained from isomerization should not exceed 40% and for lower pour points may have to be as little as 15 to 25%. To maximize yield in this situation, the choice of catalyst becomes important.

Following isomerization, the isomerate is fractionated into a lubes cut and fuels cut, the lubes cut being identified as that fraction boiling in the 330°C+ range, preferably the 370°C+ range or even higher. This lube fraction is then dewaxed. Dewaxing is accomplished by techniques which permit the recovery of unconverted wax, since in the process of the present invention this unconverted wax is recycled for further isomerization. It is preferred that this recycled wax by sent to the feed wax reservoir and passed through the hydrotreating unit to remove any quantities of entrained dewaxing solvent, which solvent could be detrimental to the isomerization catalyst. Alternatively, a separate stripper can be used to remove entrained dewaxing solvent or other contaminants. Since the unconverted wax is to be recycled, dewaxing procedures which destroy the wax such as catalytic dewaxing are not recommended. Solvent dewaxing is utilized and employs typical dewaxing solvents. Solvent dewaxing utilizes typical dewaxing solvents such as C_3 to C_6 ketones (e.g. methyl ethyl ketone, methyl isobutyl ketone and mixtures thereof), aromatic hydrocarbons (e.g. toluene), mixtures of ketones and aromatics (e.g. MEK/toluene), autorefrigerative solvents such a liquified, normally gaseous C_2-C_4 hydrocarbons such as propane, butane and mixtures thereof, etc. at filter temperatures of -25 to -30°C.

The preferred solvent to dewax the isomerate under miscible conditions and thereby produce the highest yield of dewaxed oil at a high filter rate is a mixture of MEK/MIBK (20/80) used at a temperature in the range of -25 to -30°C. Pour points lower than -21°C can be achieved using lower filter temperatures and other ratios of said solvent, but a penalty is paid due to operation under immiscible conditions, the penalty being lower filter rates. Further, when dewaxing isomerate made from a microwax, e.g. Bright Stock slack wax, it has been found to be preferred that the fraction of the isomerate which is dewaxed is the "broad heart cut" identified as the fraction boiling between about 330 and 600°C, preferably about 370 to 580°C. The heavy bottoms fraction boiling above about 580°C-600°C contains appreciable wax and can be recycled for further isomerization by being sent to the isomerization unit directly, or if any hydrotreating or deoiling is deemed necessary or desirable then the fractionation bottoms may be sent to the fresh feed reservoirs and combined with the wax therein.

It has also been found that prior to fractionation of the isomerate into various cuts and dewaxing said cuts the total liquid product (TLP) from the isomerization unit can be advantageously treated in a second stage at mild conditions using the isomerization catalyst or simply noble Group VIII on refractory metal oxide catalyst to reduce PNA and other contaminants in the isomerate and thus yield an oil of improved daylight stability.

In that embodiment the total isomerate is passed over a charge of the isomerization catalyst or over just noble Group VIII on transition alumina. Mild conditions are used, e.g. a temperature in the range of about 170 to 270°C, preferably about 180 to 220°C, at pressures of about 300 to 1500 psig H_2, preferably 500 to 1000 psig H_2, a hydrogen gas rate of about 500 to 10,000 SCF/bbl, preferably 1000 to 5000 SCF/bbl and a flow velocity of about 0.25 to 10 V/V/hr, preferably about 1 to 4 V/V/hr. Higher temperatures than those recited may be employed if pressures in excess of 1500 psi are used, but such high pressures may not be practical.

The total isomerate can be treated under these mild condition in a separate, dedicated unit or the TLP from the isomerization reactor can be stored in tankage and subsequently passed through the aforementioned isomerization reactor under said mild conditions. It has been found to be unnecessary to fractionate the first stage product prior to this mild second stage treatment. Subjecting the whole product to this mild second stage treatment produces an oil product which upon subsequent fractionation and dewaxing yields a base oil exhibiting a high level of daylight stability and oxidation stability. These base oils can be subjected to subsequent hydrotreating under conventional conditions to remove undesirable nitrogen and/or sulfur compounds using conventional catalysts such as KF-840 or HDN-30 (e.g., Co/Mo or Ni/Mo on alumina).

Those skilled in the art believe hydroisomerization to occur through the formation of trace reactive olefins on the surface of the isomerization catalyst. For this reason, the catalyst must contain a hydrogenation/dehydrogenation component (for example, a Group VIII metal such as platinum) and the reaction is carried out in a hydrogen-rich atmosphere.

The volume ratio of hydrogen gas to wax feed (the TREAT GAS RATE) has been shown in the past to affect the reactivity of the wax, but it has not been shown to affect the hydroisomerate product distribution.

For example, it has been observed that selectivity for 370°C⁺ oil depends only on the amount of conversion to 370°C⁻ material and not on any specific process parameter such as temperature, pressure or treat gas rate. This observation implied that the distribution of high boiling wax and oil were also independent of parameters such as treat gas rate.

However, it has now been discovered that the treat gas rate has an effect on the relative yields of wax and oil in the high boiling fractions. Thus, when a specific viscosity grade of oil such as 5.6-5.9 cST/100°C is considered, lower treat gas rates give rise to increased yields of 5.6-5.9 cST/100°C oil and decreased amounts of wax in that fraction.

The increased high boiling oil yield and decreased wax yield mean several things:

    (1) Wax is more efficiently converted to oil. The overall oil yield is increased and there is less wax to recycle (plant size saving).

    (2) The concentration of wax in the stream sent to the dewaxing unit is decreased. This means a reduced loan on the dewaxer, and there is less wax to entrain oil. Thus, less valuable hydroisomerate oil will be recycled to hydroisomerization.

The preferred slack wax conversion level is such that between 25 and 40 wt% unconverted wax remains in that fraction of the isomerate sent to the dewaxing unit. The amount of wax present in the stream sent to dewaxing can be controlled by the conversion level. Thus, hydroisomerization can be carried out to produce more fuels, and a lower overall residual wax yield. However, this leads to destruction of valuable wax and a reduction in the overall oil yield. The present invention is a method of minimizing the amount of wax present in the stream to be dewaxed without operating at higher conversion levels and this serves to preserve overall oil yields. Further, at the same level of conversion to 370°C⁻ material, lower treat gas rates unexpectedly give rise to higher yields of 5.6-5.9 cSt/100°C oil and lower dry wax contents of these oils.

The slack wax is preferably hydrotreated prior to hydroisomerization to remove nitrogen and sulfur compounds which may be harmful to the hydroisomerization catalyst.

Isomerization is carried out between 270 and 400°C, pressures of 500 to 3000 psig (preferably 800 to 1500 psig), and space velocities of 0.1 to 10 V/V/hr, preferably 0.5 to 2 V/V/hr. The preferred treat gas rate is between 500 and 5000 SCF/bbl, H_2, preferably 2000 to 4000 SCF/bbl, H_2, most preferably about 2000 to 3000 SCF/bbl, H_2. This range of treat gas rates allows advantage to be taken of the increased reactivity of the slack wax at higher treat gas rates and the increased oil yields and lower dry wax contents which result at lower treat gas rates. Following isomerization the isomerate is fractionated to recover the improved yield of oil in the 5.6 to

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5.9 cSt @ 100°C viscosity range.

The hydroisomerate product produced at lower treat gas rates can be isomerized in a second low temperature hydroisomerization to improve daylight stability.

This invention will be better understood from the following examples.

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EXAMPLES

A hydrotreated slack wax was used in a series of hydroisomerization experiments. Pertinent details for the wax source, pre-treatment and hydroisomerization catalyst history are summarized below:

10   Crude Source: 58% Brent System
                                          Mix
                                14% Parentis
                                 9% Basrah
                                 9% Arab Light
15   10% Chammoy

20   Slack Wax Obtained from 600N Distillate

            Wax Pre-treatment:

Hydrotreating Catalyst	Sulfided KF-840 (Ketjen, Ni/Mo alumina catalyst)
Hydrotreating Temperature	370°C
Hydrotreating Pressure	1000 psig
Hydrotreating Treat Gas Rate	1500 SCF H$_2$/B feed
Hydrotreating Liquid Space Velocity	0.42 V/V/hr
Hydrotreated Wax Oil Content	20.0 wt%
Hydrotreated Wax Sulfur Content	<10 wppm
Hydrotreated Wax Nitrogen Content	<1 wppm
Hydroisomerization Catalyst	0.6 wt% Pt/8.1 wt% F/alumina 1/16" extrudates
Method of Fluorination	HF impregnation of reforming catalyst with 10% aqueous HF.
Catalyst Volume	3600 cc
Mode	down flow, trickle bed

50 Catalyst Pre-treatment: The catalyst was dried at 220°C (4 hours in vacuum, then overnight in a lab oven at atmospheric pressure). The catalyst was charged to the reactor, and heated from ambient to 400°C in flowing H$_2$, pressure 300 psig, as follows: held at 100°C for 24 hours; from 100 to 400°C at 10°C/hour; held at 400°C for 3 hours; reactors cooled to 250°C.Details for the hydroisomerization experiments are given in the Table. Three hydroisomerate products, produced at treat gas rates 2573, 5035 and 9465 SCF H$_2$/B, were assayed.

55 Traditionally, the amount of 370°C$^-$ material produced has been used as an indicator of conversion. The conversion levels were very similar for the products from the two highest gas rates. The conversion level was slightly lower for the lowest gas rate case (22 versus 25 wt% 370°C$^-$).

The yields of 370°C$^+$ oil and 370°C$^+$ wax based on total slack was feed were also very similar for the three treat gas rates. Thus, the yield of 370°C$^+$ oil was between 49.1 and 49.8 wt%/feed for the three cases and the

60 similarity would not lead one to expect any significant differences in the oil products obtained. There is no reason to expect that there would be any difference between the yields of 5.6-5.9 cST/100°C oil which could be recovered from the three 370°C$^+$ oil products produced.

Each of the products was topped and the topped material dewaxed, to produce dewaxed oil with approximately 5.85 cST/100°C viscosity. Pour points of the dewaxed oils were measured as Westcan

65 auto-pours, for maximum accuracy. The yield of topped material was found not to be the same for the three

products. For dewaxed oils with the same pour point, the amount of dry wax removed also differed for the three cases. These effects gave rise to different yields of oil and wax using the three treat gas rates.

Auto-pour is a more sensitive indicator of pour point than is ASTM pour point. The data for dewaxing condition A indicates that the oils obtained at 2573 and 9565 SCF/B treat gas rates were dewaxed to lower auto-pour points than the oil obtained at 5035 SCF/B treat gas rate. It is ordinarily understood that more wax would have to be removed to reach the lower pour point. However, oil produced at 2573 SCF/B had the least wax of the three cases.

On comparing the three cases in the Table, it must be pointed out that the lowest treat gas rate experiment was carried out at a slightly lower conversion level (22 wt% 370°C⁻ material produced). This means that less wax has been converted to oil and light material. If this experiment had been carried out at 25 wt% 370°C⁻ conversion, as had the other two cases, the yield of 5.85 cST/100°C oil would have been higher still, and the amount of wax present in that fraction would have been even lower.

In any event, even though the lowest treat gas rate experiment was carried out at a lower conversion level, more dewaxed oil was obtained (2 to 3 wt% on slack wax feed to hydroisomerization). As well, less dry wax was removed from that oil during dewaxing than from products produced at the higher treat gas rates. This means that hydroisomerization can be carried out at a lower conversion severity and a high yield of oil will still be obtained, or hydro isomerization can be carried out at a high conversion level, resulting in a lower dry wax content in the stream to be dewaxed.

Two separate dewaxings were carried out for each of the three cases to confirm the effect. MIBK (methyl isobutyl ketone) is a better solvent than MEK (methyl ethyl ketone), and this results in less wax being removed when the dewaxing solvent is pure MIBK.

Dewaxings were carried out in a simple batch dewaxing apparatus. The waxy oil was heated to total fluidity, diluted with ketone solvent, then cooled with agitation in a cold bath to the required temperature. The mixture was filtered using a Buchner filter which was itself cooled by circulating solvent. Solvent was stripped from the wax and oil individually.

Table

Example	1	2	3
Treat Gas Rate, SCF/B, H_2	2573	5035	9565
Catalyst Hours	801-804	403-430	715-719
Temperature, $^{\circ}C$	327	315	317
Pressure, psig	1200	1200	1200
LHSV, V/V/hr	1.00	1.00	0.99
Wt% $370^{\circ}C^{-}$ Conversion/Feed	22.0	25.1	24.7
Wt% $370^{\circ}C^{+}$ Oil/Feed	49.1	49.8	49.8
Wt% $370^{\circ}C^{+}$ Wax/Feed	28.9	25.1	25.5
Wt% Waxy 5.85 cST/$100^{\circ}C$ Oil on Feed	71.0	66.4	69.0

Dewaxing Conditions A

	1	2	3
Solvent	------- 100% MIBK --------		
Solvent:Oil, w/w	---------- 4:1 ----------		
Filter Temperature, $^{\circ}C$	--------- $-25^{\circ}C$ ---------		
Wt% Dry Wax	32.6	34.5	34.9

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	1	2	3
Wt% Oil on Feed	47.8	43.4	44.9
Wt% Wax on Feed	23.2	23.0	24.1

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Viscosity of Dewaxed Oil

	1	2	3
cST, $40^{\circ}C$	29.58	29.79	30.07
cST, $100^{\circ}C$	5.83	5.86	5.90
Viscosity Index	145	145	145
Auto-Pour Point, $^{\circ}C$	-19	-17	-19
ASTM Pour Point, $^{\circ}C$	-18	-18	-18

Dewaxing Conditions B

Solvent			
Solvent	— 10wt% MEK/90wt% MIBK —		
Solvent:Oil, w/w	---------- 4:1 ----------		
Filter Temperature, °C	---------- -25 ----------		
Wt% Dry Wax	34.4	35.5	35.7

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Wt% Oil on Feed	46.5	42.7	44.3
Wt% Wax on Feed	24.5	23.7	24.7

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Viscosity of Dewaxed Oil			
cST, 40°C	29.16	29.88	29.92
cST, 100°C	5.80	5.86	5.88
Viscosity Index	146	144	145
Auto-Pour Point, °C	-20	-21	-20
ASTM Pour Point, °C	-18	-21	-18

NOTES

- "Autopour" is the Trade Mark of Hanovia Limited, Slough, England.

- Temperature in °F is converted to °C by subtracting 32 and then dividing by 1.8.

- 1 inch = 2.54 cm.

- 1 SCF = 28.316 liter

- 1 Bbl = 158.97 liter

- 1 psi = 6.895 kPa

- 1 cSt = 1 Mm^2/s

Claims

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1. A method for producing an improved yield of lube oil in the viscosity grade 5.6 to 5.9 cSt (5.6 to 5.9 Mm^2/s) at 100°C obtained from isomerized wax, said method comprising (1) isomerizing the wax over an isomerization catalyst under typical isomerization temperature, pressure and space velocity conditions, but at a low treat gas rate in the range of from 500 to 5000 $SCFH_2$/bbl (89.05 to 890.53 liter H_2 gas/liter oil), and (2) recovering the oil in the viscosity grade 5.6 to 5.9 cSt (5.6 to 5.9 Mm^2/s) at 100°C from the thus produced isomerate.

2. The method of claim 1 wherein the treat gas rate is in the range of from 2000 to 4000 SCF/bbl (356.21 to 712.42 liter gas/liter oil), H_2.

3. The method of claim 1 or claim 2 wherein the treat gas rate is in the range of from 2000 to 3000 SCF/bbl (356.21 to 534.32 liter/liter oil), H_2.

4. The method of any one of claims 1 to 3 wherein the isomerization conditions include a temperature in the range of from 270 to 400°C, a pressure in the range of from 500 to 3000 psi (3.45 to 20.60 MPa) H_2 and a space velocity in the range of from 0.1 to 10 v/v/hr.

5. The method of any one of claims 1 to 4 wherein the wax which is isomerized is selected from synthetic waxes and slack waxes.

6. The method of any one of claims 1 to 5 wherein isomerization is conducted over a catalyst comprising a Group VI metal, Group VIII metal and mixtures thereof deposited on a halogenated refractory metal oxide support.

7. The method of any one of claims 1 to 6 wherein the catalyst consists of a Group VIII metal on a fluorided alumina support.

8. The method of any one of claims 1 to 7 wherein the Group VIII metal is platinum and the alumina is gamma-alumina.

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