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**PROCESS FOR THE PREPARATION OF GROUP II
AND GROUP III LUBE BASE OILS**

Field

[0001] This disclosure relates to the preparation of Group II and Group III lube base oils wherein liquid-continuous hydrotreating is used to treat a lube oil raffinate. The hydrotreated lube oil raffinate is then sent to a dewaxing stage that can be either a solvent or catalytic dewaxing stage.

Background

[0002] Crude petroleum is distilled and fractionated into many products such as gasoline, kerosene, jet fuel, asphaltenes, and the like. One portion of the crude petroleum forms the base of lubricating baseoils used in, inter alia, the lubricating of internal combustion engines. Lube oil users are demanding ever increasing base oil quality, and refiners are finding that their available equipment is becoming less and less able to produce base oil that meet these higher quality requirements. New processes are required to provide refiners with the tools for preparing high quality modern base oils particularly using existing equipment at lower cost and with safer operation.

[0003] Finished lubricants used for such things as automobiles, diesel engines, and industrial applications are generally comprised of a lube base oil and additives. In general, a few lube base oils are used to produce a wide variety of finished lubricants by varying the

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mixtures of individual lube base oils and additives. Typically, lube base oils are simply hydrocarbons prepared from petroleum or other sources. Lube base oils are normally manufactured by making narrow cuts of vacuum gas oils from a crude vacuum tower. The cut points are set to control the final viscosity and flash point of the lube base oil.

[0004] Group I base oils, those with greater than 300 ppm sulfur and 10% aromatics are generally produced by first extracting the vacuum gas oils (or waxy distillates) or deasphalted vacuum residuum with a polar solvent, such as N-methyl-pyrrolidone, furfural, or phenol. The resulting waxy raffinates produced from solvent extraction process are then dewaxed, either catalytically with the use of a dewaxing catalyst such as ZSM-5, or through traditional solvent dewaxing. The resultant base oils may be hydrofinished to improve color and other lubricant properties.

[0005] Group II base oils, those with less than 300 ppm sulfur and 10% aromatics, and with a viscosity index range of 80-120, are typically produced by hydrocracking followed by selective catalytic dewaxing then hydrofinishing. A second, less common method for producing Group II base oils is to integrate a high-pressure hydrotreating step into a conventional solvent refining train in order to reduce base oil aromatics to below 10 wt.%.

[0006] Group III base oils have the same sulfur and aromatics specifications as Group II base oils but have viscosity indices above 120. These materials are produced with the same type of catalytic technology employed to produce Group II stocks but with

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either the hydrocracker being operated at much higher severity, or with the use of very waxy feedstocks.

[0007] Group II or III base oil specifications limit total aromatics content to less than 10 wt.%. The processing of heavier, more aromatics feedstocks requires a higher degree of aromatics conversion in the hydrocracking and dewaxing zones, which is difficult for conventional lube processing technology. There is a need in the art for improved process technology to allow for the use of heavier feeds for the production of Group II and Group II base oils.

Summary

[0008] In accordance with the present disclosure there is provided a process for the production of lube base oils, which process comprising:

i) solvent extracting a lube oil feedstock containing heteroatoms and aromatics and having a viscosity index with an extraction solvent, at solvent extraction conditions, wherein an extract stream and a raffinate stream are produced, and wherein the raffinate stream contains a smaller fraction of heteroatoms and aromatics and has a higher viscosity index than the lube oil feedstock;

ii) hydrotreating at least a portion of said raffinate in the presence of hydrogen and a hydrotreating catalyst under effective hydrotreating conditions in a liquid-continuous reactor to form a hydrotreated raffinate stream; and

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iii) dewaxing said hydrotreated raffinate stream under solvent dewaxing conditions in the presence of a dewaxing solvent to obtain a dewaxed lube base oil comprised of at least 90 wt.% saturates, a sulfur content of 0.03 wt.% or less, and a viscosity index of at least 80.

[0009] In another embodiment of the present disclosure dewaxing is accomplished by catalytic dewaxing.

[0010] In a preferred embodiment, a portion of the hydrotreated raffinate is recycled and hydrotreated with fresh raffinate.

[0011] In another preferred embodiment a portion of the hydrotreated raffinate from the liquid-continuous reactor is withdrawn and saturated with hydrogen then recycled back to the liquid-continuous reactor.

[0012] In still another embodiment of the present disclosure a Group I base oil is treated by a process comprising hydrotreating at least a portion of said Group I base oil in the presence of hydrogen and a hydrotreating catalyst under effective hydrotreating conditions in a liquid-continuous reactor to form a hydrotreated Group I base oil.

Brief Description of the Figures

[0013] Figure 1 hereof is a simplified flow diagram of a preferred embodiment of the present disclosure showing a solvent extraction stage followed by a liquid-continuous hydrotreating stage followed by a solvent dewaxing stage.

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[0014] Figure 2 hereof is a simplified flow diagram of another preferred embodiment of the present disclosure showing a solvent extraction stage followed by a liquid-continuous hydrotreating stage followed by a catalytic dewaxing stage followed by a hydrofinishing stage.

Detailed Description

[0015] All numerical values within the detailed description and the claims herein are modified by “about” or “approximately” the indicated value, and take into account experimental error and variations that would be expected by a person having ordinary skill in the art.

[0016] The present disclosure is directed to the preparation of Group II and Group III lube base oils. API Publication 1509: Engine Oil Licensing and Certification System, "Appendix E-API Base Oil Interchangeability Guidelines for Passenger Car Motor Oil and Diesel Engine Oils" describes base oil categories. A Group II base oil will contain greater than or equal to 90 wt.% saturates and less than or equal to 0.03 wt.% sulfur and will have a viscosity index greater than or equal to 80 and less than 120. A Group III base oil will contain greater than or equal to 90 wt.% saturates and less than or equal to 0.03 wt.% sulfur and will have a viscosity index greater than or equal to 120. The term “viscosity index” (VI) refers to the measurement defined by ASTM D2270.

[0017] Feedstocks suitable for use herein are preferably one or a combination of refinery streams having a normal boiling point of at least 600° F (316° C), although hydrocarbon refinery streams having initial boiling points as low as 435° F (224° C) can also be

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used. By having a normal boiling point of at least 600° F (316° C) is meant that 85% by volume of the feedstock has a boiling point at atmospheric pressure of at least 600° F (316° C). While higher boiling lube oil feedstocks can be processed in accordance with the present disclosure, the preferred feedstock will have a boiling range such that at least 85% by volume of the feedstock has a normal boiling point of at most 1250° F (677° C), and more preferably at most 1100°F (593°C). Such feedstocks, particularly vacuum gas oils, will contain from 35 wt.% to 70 wt.% aromatics, at least 40% of them being 2-ring and higher aromatics. Representative feedstocks that can be treated in accordance with the present disclosure include gas oils and vacuum gas oils (VGO), hydrocracked gas oils and hydrocracked vacuum gas oils, deasphalted oils, reduced crude, vacuum tower bottoms, deasphalted vacuum resids. The nitrogen, sulfur and saturate contents of these feeds will vary depending on a number of factors. The preferred feedstocks for the present disclosure will have an entrained oil viscosity of greater than 40. In a more preferred embodiment, the entrained oil in the feedstock will have a viscosity index in the range of 50 to 110.

[0018] Lube refineries are continually challenged to increase throughput and to process more refractory feedstocks. Limitations with respect to conventional solvent-based lube plants to accomplish these objectives are the need for cost-effective debottlenecking to handle increased rate, and the poor yields associated with the extraction of very refractory feeds. Additionally, conventional solvent-based lube refining are typically unable, without high-pressure hydrotreating capacity, to meet the Group II aromatics specification (10 wt.% max).

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[0019] The process of the present disclosure represents a cost-effective means for incorporating hydrotreating into a conventional solvent-based lube refinery. This disclosure is better understood with reference to the Figures hereof that illustrate the primary pieces of equipment for practicing a preferred embodiment of the present disclosure. Ancillary equipment, such as valves, pumps, compressors, heat exchanger, heaters and the like are not shown for simplicity reasons. Such ancillary equipment are well known to those having ordinary skill in the art. A lube oil feedstock is conducted via line 10 to solvent extraction stage **100**.

[0020] Solvent extraction is a physical separation process that uses a solvent to preferentially dissolve and remove aromatic and other polar compounds from the lube oil feedstock that cause large changes of viscosity with temperature. Solvent extraction removes a portion of these components and improves viscosity index (VI), oxidation stability, color, and oxidation inhibitor response. The VI of an oil is an arbitrary relative measure of its change in viscosity with temperature. The smaller the change in viscosity of an oil with a given change in temperature the higher the VI value of the oil. A high VI is desirable in high quality motor oils. The amount of material extracted depends on the increase in VI required. Extraction also reduces the Conradson carbon and sulfur content. Low aromatic and sulfur contents are conducive to good oxidation stability and color of the resulting base oils.

[0021] Solvent extraction is suitably carried out with solvents such as N-Methyl-2-pyrrolidone, phenol, or furfural. The solvents are chosen for their relative solubilization of aromatic-type petroleum molecules, and for their relatively low boiling point,

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which permits ease of separation of the solvent from the extract. The extraction takes place in a solvent extractor. Any suitable solvent extractor can be used in the practice of the present disclosure. Non-limiting examples of solvent extractors that can be used in the practice of the present disclosure include rotating disc contactors, packed towers, baffle trayed towers, and centrifugal contactors. If an asphalt-containing feedstock is used in the practice of the present disclosure it is preferably deasphalted prior to solvent extraction. Preferred solvents for deasphalting include lower-boiling paraffinic hydrocarbons such as ethane, propane, butane, pentane, or mixtures thereof. Propane is a preferred deasphalting solvent and pentane is a most suitable solvent if high yields of deasphalted oil are desired. These lower-boiling paraffinic solvents can also be used as mixtures with alcohols, such as methanol and isopropanol. Solvent extraction severity is typically maintained at sufficient conditions to produce an extracted oil product when dewaxed having a viscosity index of at least 80, preferably at least 95.

[0022] The solvent extraction process for the preparation of a lube oil feedstock useful in the present disclosure can be run at lower severity than is commonly employed in the preparation of high quality lubricating oil base stocks. Reduced solvent extraction severity is seen in reduced solvent usage and/or in reduced solvent extraction temperatures and can allow for increased throughput through the extraction device. Decreasing the severity of the solvent extraction step also results in higher yield, but it reduces the VI of the entrained oil in the resulting raffinate. The “under-extracted” raffinate has a higher concentration of aromatics and heteroatoms, hence resulting in the need for an additional step to

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increase VI to acceptable levels. Hydrotreating raffinates offers the potential benefits of increasing the VI at low yield penalty while reducing base oil aromatics to Group II levels. Very severe hydrotreating operation can result in a VI increase large enough to produce a Group III base oil.

[0023] Solvent extraction conditions can be maintained to produce an oil product having a viscosity index which is at least 5 less, and preferably in the range of 5 to 20 less than the desired viscosity index of the lubricating base oil prepared by the present process. If the desired viscosity index of the Group II lubricating base oil is 80, the solvent extraction pre-treatment step of the present process is maintained to produce a lubricating oil feedstock having a viscosity index of less than 75, preferably in the range from 60 to 75. Likewise, if the desired viscosity index of the Group II lubricating base oil is 95, solvent extraction is maintained to produce a lubricating oil feedstock having a viscosity index of less than 90, preferably in the range from 75 to 90.

[0024] Returning now to Figure 1 hereof, the extract from solvent extraction **100** is sent, via line **11**, for solvent recovery (not shown). Solvent recovery technology is well known in the art and a detailed discussion of it is not warranted in this application. Solvent is typically recovered by conducting the extract to equipment such as flash columns or steam strippers (not shown). Multiple flash columns can improve overall heat utilization as solvent recovered in higher pressure flash columns can be used effectively to transfer heat content to hydrocarbon streams. Process variables that affect solvent recovery include such things as reflux ratios, pressure, temperature, and stripping steam within

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the constraints of solvent content of the raffinate and extract streams.

[0025] The raffinate stream from solvent extraction **100** is sent via line **12** to liquid-continuous hydrotreating stage **200** that will primarily be a suitable reactor. Make-up hydrogen, as needed, can be introduced via line **14**. It will be understood that makeup hydrogen can be added at any suitable point along the feed line or even directly into reactor **200**. It is also within the scope of this disclosure that the gas-liquid flow to liquid-continuous hydrotreating reactor **200** be blended under static mixing conditions. By static mixing conditions we mean one or more, preferably more, of geometric mixing elements fixed within a pipe that use the energy of the moving stream to create mixing between two or more fluids. Thus, the static mixers themselves have no moving parts. The advantage of the static mixers of the present disclosure over dynamic mixers, other than the fact that static mixers have no moving parts, is that static mixers split the stream hundreds, or even thousands of times, thus resulting in a continuous phase containing very fine droplets of discontinuous phase. This results in a much larger surface area when compared with dynamic mixers. The gas-liquid mixture can also be flashed in a suitable vessel before entering reactor **200** to remove at least a portion of any excess gas. Alternatively, excess gas can be vented (not shown) directly from reactor **200**.

[0026] It may be necessary to recycle liquid product from the liquid-continuous hydrotreating reactor to ensure that sufficient hydrogen is present in the liquid phase for the reaction. The recycled liquid serves as a carrier for additional solubilized

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hydrogen. Alternatively, or in combination with this liquid recycle, hydrogen may also be added to the reactor by withdrawing liquid at one or more points, preferably at one or more axial points, along the reactor, resaturating the liquid with hydrogen, then reinjecting it back into the reactor. This approach can be used to reduce the amount of required liquid recycle.

[0027] Because the liquid effluent from **200** will contain only dissolved gas, it is not necessary to have a high-pressure separation step downstream of the reactor. Only a low-pressure flash step is needed to vent dissolved and excess gas before product fractionation. Elimination of high-pressure product recovery equipment significantly reduces the cost, particularly if this disclosure is used for debottlenecking in an existing lubes plant.

[0028] As previously mentioned, reactor **200** used in this disclosure is operated such that the liquid phase represents the continuous phase in the reactor. Traditionally, hydroprocessing, including hydrotreating, is conducted in trickle-bed reactors where an excess of gas results in a continuous gas phase in the reactor. In a liquid-continuous reactor, the feedstock is exposed to one or more beds of catalyst. The liquid raffinate preferably enters from the top or upper portions of the reactor and flows downward through the catalyst beds of the reactor. This downward liquid flow can assist in allowing the catalyst to remain in place in the catalyst bed. An advantage of liquid-continuous reactors is that they operate near isothermally. Because there are substantially no hot spots within the reactor, this allows one to tune the operation of the reactor to more precisely meet product quality needs.

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[0029] A hydroprocessing process typically involves exposing a feed to a suitable catalyst in the presence of hydrogen at effective hydroprocessing conditions. Without being bound by any particular theory, in a conventional trickle-bed reactor, the reactor is typically operated so that three “phases” are present in the reactor. The hydroprocessing catalyst corresponds to the solid phase. Another substantial portion of the reactor volume is occupied by a gas phase. This gas phase (second-phase) includes the hydrogen for hydroprocessing, optionally some diluent gases, and other gases such as contaminant gases that form during hydroprocessing. The amount of hydrogen gas in the gas phase is typically present in substantial excess relative to the amount required for the hydroprocessing reaction. In a conventional trickle-bed reactor, the solid hydroprocessing catalyst and the gas phase can occupy at least 80% of the reactor volume, or at least 85%, or even at least 90%. The third “phase” corresponds to the liquid feedstock. In a conventional trickle-bed reactor, the feedstock will typically only occupy a small portion of the volume, such as less than 20%, or less than 10%, or less than 5%. As a result, the liquid feedstock will not form a continuous phase. Instead, the liquid “phase” will include, for example, thin films of feedstock that coat the hydroprocessing catalyst particles.

[0030] Without being bound by any particular theory, a liquid-continuous reactor provides a different type of processing environment as compared to a trickle-bed reactor. In a liquid-continuous reactor, the reaction zone is primarily composed of only two phases. One phase is a solid phase corresponding to the hydroprocessing catalyst, in this case a hydrotreating catalyst. The second phase is a liquid phase corresponding to the raffinate

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feedstock. The liquid feedstock phase will be present as a continuous phase in the liquid-continuous reactor of the present disclosure. In an embodiment, the hydrogen that will be consumed during the hydrotreating reaction is dissolved in the liquid phase. Depending on the quantity of hydrogen used, a portion of the hydrogen can also be in the form of bubbles of hydrogen in the liquid phase. This hydrogen corresponds to hydrogen that is in addition to the hydrogen dissolved in the liquid phase. In another embodiment, hydrogen dissolved in the liquid phase can be depleted as the reactions progress in the liquid-continuous reactor. In such an embodiment, hydrogen initially present in the form of gaseous bubbles can dissolve into the liquid phase to resaturate the liquid phase and provide additional hydrogen for the reactions taking place in the reactor. In various embodiments, the volume occupied by a gas phase in the liquid-continuous reactor can be less than 10% of the reactor volume, or even less than 5%.

[0031] The liquid feed to the reactor **200** is preferably mixed with a hydrogen-containing treat gas. The hydrogen-containing treat gas will preferably contain at least 50 vol% of hydrogen, more preferably at least 80 vol%, even more preferably at least 90 vol%, and most preferably at least 95 vol%. Excess gas can be vented from the mixture before it enters the reactor, or excess gas can be vented directly from the reactor. The liquid level in the reactor is preferably controlled so that the catalyst in the reactor is completely wetted.

[0032] In some embodiments, the hydrotreating reactions in a bed, stage, and/or reactor can require more hydrogen than can be dissolved in the fresh liquid feed. In such embodiments, one or

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more techniques can be used to provide additional hydrogen for the hydrotreating reaction. One option is to recycle a portion of the product from the reactor. A recycled portion of product that has already passed through a hydrotreating stage will likely have a reduced hydrogen consumption as it passes again through the hydrotreating stage. Additionally, the solubility of the recycled feed can be higher than a comparable unprocessed feed. As a result, including a portion of recycled product with fresh feed can increase the amount of hydrogen available for reaction with the fresh feed.

[0033] Another option is to introduce additional streams of hydrogen into the hydrotreating reactor directly. One or more additional hydrogen streams can be introduced at any convenient location in the reactor. The additional hydrogen streams can include a stream of make-up hydrogen, a stream of recycled hydrogen, or any other convenient hydrogen-containing stream. In some embodiments, both product recycle and injection of additional hydrogen streams along the axial dimension of the reactor can be used to provide sufficient hydrogen for a reaction.

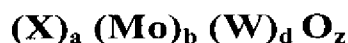
[0034] In embodiments involving recycle of the liquid-continuous hydrotreated product for use as part of the input to reactor **200**, the ratio of the amount by volume of product recycle to the amount of fresh feed into reactor **200** will be at least 0.5 to 1, or at least 1 to 1, or at least 1.5 to 1. The ratio of the amount by volume of product recycle to the amount of fresh feed can be 5 to 1 or less, or 3 to 1 or less, or 2 to 1 or less.

[0035] The hydrotreating catalyst of the present disclosure will contain at least one of Group VIB and/or Group VIII metals

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optionally on a support. Any suitable refractory support material can be used in the practice of this disclosure. Non-limiting examples of such suitable support materials include alumina, silica, silica alumina, titania, zirconia, silica-alumina, combinations of the above. Examples of Group VIB metals that can be used herein include molybdenum, tungsten, or a combination thereof. Examples of Group VIII metals that can be used herein include nickel, cobalt, iron, or combinations thereof. All Groups referred to herein are as found in the Sargent-Welch Periodic Table of the Elements copyrighted in 1968 by the Sargent-Welch Scientific Company. Preferred catalyst compositions contain in excess of 5 wt.% Group VIB metals, preferably 5 to 40 wt.% molybdenum and/or tungsten, and at least 0.5 wt.%, and generally 1 to 15 wt.% of nickel and/or cobalt determined as the corresponding oxides. Hydrotreating catalysts of this type are readily available from catalyst suppliers. These catalysts are generally presulfided using H₂S or other suitable sulfur containing compounds.

[0036] Bulk multimetallic catalysts can also be used for aromatics saturation in the practice of the present disclosure. Such catalysts are described in U.S. Patent Nos. 6,156,695; 6,162,350; and 6,299,760, all of which are incorporated herein by reference. The catalysts described in these patents are bulk multimetallic catalysts comprised of at least one Group VIII non-noble metal and at least two Group VIB metals, wherein the ratio of Group VIB metal to Group VIII non-noble metal is from 10:1 to 1:10. These catalysts are prepared from a precursor having the formula:



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where X is a Group VIII non noble metal, wherein the molar ratio of and a, b, and c, are such that $0.1 < (b+c)/b < 10$, and $z = [2a + 6(b+c)]/2$. The precursor has x-ray diffraction peaks at $d = 2.53$ and 1.70 Angstroms. The precursor is sulfided to produce the corresponding activated catalyst.

[0037] The degree of aromatics saturation and desulfurization activity of the catalyst may be found by experimental means, using a feed of known composition under fixed hydrotreating conditions.

[0038] Control of the reaction parameters of the hydrotreating step also offers a useful way of varying product properties. As hydrotreating temperature increases the degree of desulfurization increases; although hydrogenation is an exothermic reaction favored by lower temperatures, desulfurization usually requires some ring-opening of heterocyclic compounds to occur and these reactions being endothermic, are favored by higher temperatures. If the temperature during the hydrotreating step can be maintained at a value below the threshold at which excessive desulfurization takes place, products of improved oxidation stability are obtained. When a bimetallic such as nickel-molybdenum for the hydrotreating catalyst is used, temperatures of 400°F to 800°F (205°C to 427°C), preferably 600°F to 750°F (316°C to 399°C) are recommended for good oxidative stability. Space velocity in the hydrotreater also offers a potential for desulfurization control with the higher velocities corresponding to lower severities resulting in a reduction in the degree of desulfurization. The hydrotreated product preferably has an organic sulfur content of less than 300 wppm, preferably less than 200 wppm.

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[0039] Variation of hydrogen pressure during the hydrotreating step also enables the desulfurization to be controlled with lower pressures generally leading to less desulfurization as well as a lower tendency to saturate aromatics, and eliminate peroxide compounds and nitrogen, all of which are desirable. A balance may therefore need to be achieved between a reduced degree of desulfurization and a loss in the other desirable effects of the hydrotreating. Generally, pressures of 200 to 2200 psig (1480 to 15300 kPa abs) are satisfactory with pressures of 1000 to 1500 psig (7000 to 10450 kPa abs) giving good results with appropriate selection of metal function and other reaction conditions made empirically by determination of the desulfurization taking place with a given feed.

[0040] Hydrotreating is performed by exposing a feedstock to a hydrotreating catalyst under effective hydrotreating conditions. Effective hydrotreating conditions include temperatures of at least 600°F to 750°F, pressures from 200 to 2200 psi, a liquid hourly space velocity (LHSV) over the hydrotreating catalyst of 0.2 to 5, and a treat gas rate of 500 to 10,000 standard cubic feet per barrel (scf/bbl). In still another embodiment, the temperature, pressure, and LHSV for a liquid-continuous reactor can be conditions suitable for use in a trickle-bed reactor.

[0041] In embodiments where excess gas is vented from the liquid, the available hydrogen in the reactor corresponds to the amount of hydrogen dissolved in the liquid. Thus, a higher treat gas rate may not lead to an increase in the amount of available hydrogen. In such a situation, the effective treat gas rate within a reactor may be dependent on the solubility limit of the feedstock.

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The hydrogen solubility limit for a typical hydrocarbon feedstock is 30 scf/bbl to 200 scf/bbl.

[0042] One advantage of a liquid-continuous reactor is that a large excess of hydrogen does not have to be fed to the reactor. The use of a large excess of hydrogen typically requires complex and expensive separation equipment to allow for recovery, and often recycling, of the excess hydrogen. Typically the recycle compressor used for hydrogen recycle in a trickle-bed reactor corresponds to 10 to 15% of the total cost of the erected processing unit. Instead, it is desirable for a liquid-continuous reactor will desirably supply only an amount of hydrogen comparable to the amount needed for a hydroprocessing reaction and to mitigate catalyst coking. For example, a hydrotreating process can consume from 150 scf/bbl (27 sm^3/m^3) of hydrogen to 1000 scf/bbl (180 sm^3/m^3).

[0043] Returning now to Figure 1 hereof, the effluent stream from **200** is conducted via line **16** to separation zone **300** wherein a gaseous phase, which is primarily comprised of excess hydrogen and contaminant gases such as ammonia and H_2S , is separated from the hydrotreated liquid raffinate phase. The gaseous phase can be vented or sent via line **18** for further processing or recycle. The hydrotreated liquid raffinate phase is conducted via line **20** to dewaxing stage **400**. Although dewaxing stage **400** can be either a solvent dewaxing or catalytic dewaxing process, for purposes of this Figure 1, the dewaxing stage is solvent dewaxing.

[0044] Solvent dewaxing typically involves mixing a raffinate feed from the solvent extraction unit with chilled dewaxing solvent to form an oil-solvent solution and precipitated wax is thereafter

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separated by, for example filtration. The temperature and solvent are selected so that the oil is dissolved by the chilled solvent while the wax is precipitated. In this case, the raffinate feed is hydrotreated before being sent to dewaxing.

[0045] A preferred solvent dewaxing process involves the use of a cooling tower where solvent is prechilled and added incrementally at several points along the height of the cooling tower. The oil-solvent mixture is agitated during the chilling step to permit substantially instantaneous mixing of the prechilled solvent with the oil. The prechilled solvent is added incrementally along the length of the cooling tower so as to maintain an average chilling rate at or below 10° F per minute, usually between 1 to 5°F per minute. The final temperature of the oil-solvent/precipitated wax mixture in the cooling tower will usually be between 0 and 50°F (-17.8 to 10°C). The mixture may then be sent to a scraped surface chiller to separate precipitated wax from the mixture.

[0046] In general, the amount of solvent added will be sufficient to provide a liquid/solid weight ratio from 5 to 1 to 20 to 1 at the dewaxing temperature and at a solvent/oil volume ratio at 1.5 to 1 to 5 to 1. The solvent dewaxed oil is typically dewaxed to an intermediate pour point, preferably less than +10°C.

[0047] Non-limiting examples of dewaxing solvents that can be used in the practiced of the present disclosure include aliphatic ketones having 3-6 carbon atoms such as methyl ethyl ketone and methyl isobutyl ketone, low molecular weight hydrocarbons such as propane and butane, and mixtures thereof. These solvents can be mixed with one or more other solvents such as benzene, toluene or xylene. Further descriptions of solvent dewaxing processes

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useful herein are disclosed in U.S. Pat. Nos. 3,773,650 and 3,775,288 both of which are incorporated herein in their entirety by reference.

[0048] Returning again to Figure 1 hereof two streams are collected from solvent dewaxing stage **400**. A precipitated wax via line **22** and a Group II or Group III base oil via line **24**.

[0049] Figure 2 hereof is a schematic flow diagram of another embodiment of the present disclosure wherein catalytic dewaxing is used instead of solvent dewaxing. All components and numbers of this Figure 2 are identical to that of Figure 1 hereof up to and including hydrotreating zone **200**. The hydrotreated raffinate stream from separation zone **300** is passed via line **20** to catalytic dewaxing stage **400**. The dewaxed stream is passed via line **30** to second separation zone **600** where a gaseous effluent stream is removed as an off-gas via line **32** and the dewaxed liquid effluent stream is passed via line **34** to stripper **700** to remove any remaining gaseous moieties. The resulting Group II or Group III base oil is collected via line **36**, which base oil will at least meet the API Group II base oil requirements as previously discussed.

[0050] Instead of conducting the effluent stream from hydrotreating stage **200** to separation zone **300** it can alternatively be conducted, via lines **17** and **20** directly to dewaxing stage **500**. Because contaminant gases are not removed with this alternative, the dewaxing catalyst would operate in a sour environment, thus hydrogen consumption would be relatively low, potentially obviating the need for liquid recycle to the dewaxing reactor if it were a liquid-continuous reactor. It is preferred that the effluent

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stream from hydrotreating stage **200** be first passed to separation zone **300** instead of being directly passed to dewaxing stage **500**.

[0051] Catalytic dewaxing is performed by exposing the hydrotreated raffinate to a dewaxing catalyst under effective (catalytic) dewaxing conditions. Effective dewaxing conditions can include a temperature of at least 500°F (260°C), or at least 550°F (288°C), or at least 600°F (316°C), or at least 650°F (343°C). Alternatively, the temperature can be 750°F (399°C) or less, or 700°F (371°C) or less, or 650°F (343°C) or less. The pressure can be at least 200 psig (1.4 MPa), or at least 400 psig (2.8 MPa), or at least 750 psig (5.2 MPa), or at least 1000 psig (6.9 MPa). Alternatively, the pressure can be 2200 psig (15.3 MPa) or less, or 1500 psig (10.4 MPa) or less, or 1000 psig (6.9 MPa) or less, or 800 psig (5.5 MPa) or less. The liquid hourly space velocity (LHSV) over the dewaxing catalyst can be at least 0.1 hr⁻¹, or at least 0.2 hr⁻¹, or at least 0.5 hr⁻¹, or at least 1.0 hr⁻¹, or at least 1.5 hr⁻¹. Alternatively, the LHSV can be 10.0 hr⁻¹ or less, or 5.0 hr⁻¹ or less, or 3.0 hr⁻¹ or less, or 2.0 hr⁻¹ or less. In still another embodiment, the temperature, pressure, and LHSV for a liquid-continuous reactor can be the same conditions typically used for a trickle-bed reactor.

[0052] Catalytic dewaxing involves the removal and/or isomerization of long chain, paraffinic molecules from feeds. Catalytic dewaxing can be accomplished by selective cracking or by hydroisomerizing these linear molecules. Hydrodewaxing catalysts can be selected from molecular sieves such as crystalline aluminosilicates (zeolites) or silico-aluminophosphates (SAPOs). In an embodiment, the molecular sieve can be a 1-D or 3-D

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molecular sieve. In another embodiment, the molecular sieve can be a 10-member ring 1-D molecular sieve. Examples of molecular sieves which have shown dewaxing activity in the literature can include ZSM-48, ZSM-22, ZSM-23, ZSM-35, Beta, USY, ZSM-5, and combinations thereof. In an embodiment, the molecular sieve can be ZSM-22, ZSM-23, ZSM-35, ZSM-48, or a combination thereof. In still another embodiment, the molecular sieve can be ZSM-48, ZSM-23, ZSM-5, or a combination thereof. In yet another embodiment, the molecular sieve can be ZSM-48, ZSM-23, or a combination thereof. Optionally, the dewaxing catalyst can include a binder for the molecular sieve, such as alumina, titania, silica, silica-alumina, zirconia, or a combination thereof.

[0053] The dewaxing catalyst can also include a metal hydrogenation component, such as a Group VIII metal. Suitable Group VIII metals can include Pt, Pd, Ni, or a combination thereof. The dewaxing catalyst can include at least 0.1 wt% of a Group VIII metal, or at least 0.3 wt%, or at least 0.5 wt%, or at least 1.0 wt%, or at least 2.5 wt%, or at least 5.0 wt%. Alternatively, the dewaxing catalyst can include 10.0 wt% or less of a Group VIII metal, or 5.0 wt% or less, or 2.5 wt% or less, or 1.5 wt% or less, or 1.0 wt% or less.

[0054] In some embodiments, the dewaxing catalyst can also include at least one Group VIB metal, such as W or Mo. Such Group VIB metals are typically used in conjunction with at least one Group VIII metal, such as Ni or Co. An example of such an embodiment is a dewaxing catalyst that includes Ni and W, Mo, or a combination of W and Mo. In such an embodiment, the

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dewaxing catalyst can include at least 0.5 wt% of a Group VIB metal, or at least 1.0 wt%, or at least 2.5 wt%, or at least 5.0 wt%. Alternatively, the dewaxing catalyst can include 20.0 wt% or less of a Group VIB metal, or 15.0 wt% or less, or 10.0 wt% or less, or 5.0 wt% or less, or 1.0 wt% or less. In an embodiment, the dewaxing catalyst can include Pt, Pd, or a combination thereof. In another embodiment, the dewaxing catalyst can include Co and Mo, Ni and W, Ni and Mo, or Ni, W, and Mo.

[0055] In the case where catalytic dewaxing is used, makeup hydrogen-containing treat gas can be added as needed upstream of the catalytic dewaxing reactor. The effluent from the catalytic dewaxing zone can then be sent to a liquid/gas separator wherein the gaseous effluent is separated from the liquid effluent. The gaseous effluent, can be vented or sent to further processing and the liquid effluent can be sent to a stripper to remove light byproducts.

[0056] It is within the scope of this disclosure that there be two dewaxing steps run in parallel. One dewaxing step would be solvent dewaxing and the other catalytic dewaxing. One type of base oil can be solvent dewaxed while another is catalytically dewaxed. If it is desired to produce Group II base oils in a conventional lube plant and to increase base oil product, the addition of both a hydrotreating process unit and a dewaxing unit would be required. In such a case catalytic dewaxing would be preferred because it would be the least costly option and would be well integrated with the hydrotreater.

[0057] It will be understood that a hydrofinishing step can follow either solvent dewaxing or catalytic dewaxing. If catalytic

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dewaxing is used, it is preferred that a hydrofinishing step follow dewaxing. Hydrofinishing is a mild, relatively cold hydrotreating process, that employs a catalyst, hydrogen and mild reaction conditions to remove trace amounts of heteroatom compounds, aromatics and olefins, to improve primarily oxidation stability and color. Hydrofinishing reaction conditions include temperatures from 300°F to 675°F. (149°C to 357°C.), preferably from 400°F to 600°F. (204°C to 316°C.), a total pressure of from 400 to 2200 psig (2860 to 15270 kPa abs), a liquid hourly space velocity ranging from 0.1 to 5 LHSV (hr^{-1}), preferably 0.5 to 3 hr^{-1} . The hydrogen treat gas rate will range from 500 to 5000 scf/bbl (89 to 890 m^3/m^3). Hydrofinishing following solvent dewaxing will normally be conducted at pressures between 200 and 1000 psig while hydrofinishing following catalytic dewaxing will normally be conducted at a pressure similar to that of the dewaxing step. The hydrofinishing catalyst can comprise a support component and one or more catalytic metal components. The one or more metals are selected from Group VIB (Mo, W, Cr) and Group VIII (Ni, Co and the noble metals Pt and Pd) which Groups are found in the Sargent-Welch Periodic Table of the Elements copyrighted in 1968 by the Sargent-Welch Scientific Company. The metal or metals may be present from as little as 0.1 wt% for noble metals, to as high as 30 wt% of the catalyst composition for non-noble metals. Preferred support materials are low in acid and include, for example, amorphous or crystalline metal oxides such as alumina, silica, silica alumina and ultra large pore crystalline materials known as mesoporous crystalline materials, of which MCM-41 is a preferred support component. Un-supported base metal (non-noble metal) catalysts are also applicable as hydrofinishing catalysts.

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[0058] The effluent stream from hydrofinishing can be passed to a separation zone wherein a gaseous effluent stream is separated from the resulting liquid phase lube oil base stock. The gaseous effluent stream, a portion of which will be unreacted hydrogen-containing treat gas can be recycled to hydrotreating stage **200**. The resulting lube oil base stock, will meet Group II or Group III base oil requirements.

[0059] All patents and patent applications, test procedures (such as ASTM methods, UL methods, and the like), and other documents cited herein are fully incorporated by reference to the extent such disclosure is not inconsistent with this disclosure and for all jurisdictions in which such incorporation is permitted.

[0060] When numerical lower limits and numerical upper limits are listed herein, ranges from any lower limit to any upper limit are contemplated. While the illustrative embodiments of the disclosure have been described with particularity, it will be understood that various other modifications will be apparent to and can be readily made by those skilled in the art without departing from the spirit and scope of the disclosure. Accordingly, it is not intended that the scope of the claims appended hereto be limited to the examples and descriptions set forth herein but rather that the claims be construed as encompassing all the features of patentable novelty which reside in the present disclosure, including all features which would be treated as equivalents thereof by those skilled in the art to which the disclosure pertains.

[0061] The present disclosure has been described above with reference to numerous embodiments and specific examples. Many variations will suggest themselves to those skilled in this art in

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light of the above detailed description. All such obvious variations are within the full intended scope of the appended claims.

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CLAIMS:

1. A process for the production of lube base oil, which process comprising:

i) solvent extracting a lube oil feedstock containing heteroatoms and aromatics and having a viscosity index with an extraction solvent, at solvent extraction conditions, wherein an extract stream and a raffinate stream are produced, and wherein the raffinate stream contains a smaller fraction of heteroatoms and aromatics and has a higher viscosity index than the lube oil feedstock;

ii) hydrotreating at least a portion of said raffinate in the presence of hydrogen and a hydrotreating catalyst under effective hydrotreating conditions in a liquid-continuous reactor to form a hydrotreated raffinate stream; and

iii) dewaxing said hydrotreated raffinate stream under solvent dewaxing conditions in the presence of a dewaxing solvent to obtain a dewaxed lube base oil comprised of at least 90 wt.% saturates, a sulfur content of 0.03 wt.% or less, and a viscosity index of at least 80.

2. The process of claim 1 wherein the lube oil feedstock is selected from the group consisting of vacuum gas oils, hydrocracked gas oils, hydrocracked vacuum gas oils, deasphalted oils, reduced crude, vacuum tower bottoms, and deasphalted vacuum resids.

3. The process of claim 2 wherein the lube oil feedstock is a vacuum gas oil.

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4. The process of claim 1 wherein the extraction solvent is selected from the group consisting of N-Methyl-2-pyrrolidone, phenol, or furfural.
5. The process of claim 1 wherein the extract from solvent extraction is sent to a solvent recovery step.
6. The process of claim 1 wherein a portion of the hydrotreated raffinate is recycled to the liquid-continuous reactor and again hydrotreated with fresh raffinate.
7. The process of claim 6 wherein the volume ratio of recycled hydrotreated raffinate to fresh raffinate to the liquid-continuous reactor is from 0.5 to 1 to 5 to 1.
8. The process of claim 6 wherein the volume ratio of recycled hydrotreated raffinate to fresh raffinate to the liquid-continuous reactor is from 1 to 1 to 3 to 1.
9. The process of claim 1 wherein a portion of the hydrotreated raffinate from the liquid-continuous reactor is withdrawn and saturated with hydrogen then recycled back to the liquid-continuous reactor.
10. The process of claim 1 wherein the hydrotreating catalyst is comprised of one or more catalytic metals selected from Group VIB and Group VIII of the Periodic Table of the Elements on a refractory support.

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11. The process of claim 10 wherein the support is selected from the group consisting of alumina, silica, silica alumina, titania, zirconia, and silica-alumina.

12. The process of claim 1 wherein the hydrotreating process conditions include temperatures from 400°F to 800°F and total pressures from 200 psig to 2200 psig.

13. The process of claim 12 wherein the hydrotreating process conditions include temperatures from 600°F to 750°F and total pressures from 1000 to 1500 psig.

14. The process of claim 1 wherein the raffinate stream, before it is conducted to hydrotreating is sent to a gas-liquid separation stage to remove at least a portion of any excess gas that may be present in the raffinate stream.

15. The process of claim 1 wherein the hydrotreated raffinate stream, before it is conducted to dewaxing is sent to a gas-liquid separation stage to remove excess hydrogen and contaminant gases selected from ammonia and hydrogen sulfide and the remaining liquid hydrotreated raffinate stream is sent to solvent dewaxing.

16. The process of claim 1 wherein dewaxing solvent is selected from the group consisting of aliphatic ketones having 3 to 6 carbon atoms, low molecular weight hydrocarbons having from 2 to 4 carbon atoms.

17. The process of claim 16 wherein the dewaxing solvent is selected from the group consisting of methyl ethyl ketone, methyl isobutyl ketone, propane, butane, and mixtures thereof.

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18. The process of claim 1 wherein the dewaxing solvent is added to provide a liquid to solids weight ratio of from 5 to 1 to 20 to 1 at dewaxing temperatures from 0 to 50°F.

19. The process of claim 1 wherein there catalytic dewaxing is run parallel with solvent dewaxing.

20. The process of claim 19 wherein the dewaxed lube base oil is sent to a hydrofinishing zone wherein it is contacted at hydrofinishing conditions with a hydrofinishing catalyst and in the presence of hydrogen to remove at least a portion of any remaining aromatics and to improve color.

21. A process for the production of lube base oils, which process comprising:

i) solvent extracting a lube oil feedstock containing heteroatoms and aromatics and having a viscosity index with an extraction solvent, at solvent extraction conditions, wherein an extract stream and a raffinate stream are produced, and wherein the raffinate stream contains a smaller fraction of heteroatoms and aromatics and has a higher viscosity index than the lube oil feedstock;

ii) hydrotreating at least a portion of said raffinate in the presence of hydrogen and a hydrotreating catalyst under effective hydrotreating conditions in a liquid-continuous reactor to form a hydrotreated raffinate stream; and

iii) catalytically dewaxing said hydrotreated raffinate in the presence of hydrogen and a dewaxing catalyst under effective dewaxing conditions including a temperature from 500°F to 750°F

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and a pressure up to 2200 psig and at an effective contact time of feed to catalyst that will remove at least a portion of the waxy paraffinic components by isomerization to less waxy iso-paraffinic components, thereby producing a lube base oil comprised of at least 90 wt.% saturates, less than 0.03 wt.% sulfur and a viscosity index of at least 80.

22. The process of claim 21 wherein the lube oil feedstock is selected from the group consisting of vacuum gas oils, hydrocracked gas oils, hydrocracked vacuum gas oils, deasphalted oils, reduced crude, vacuum tower bottoms, and deasphalted vacuum resids.

23. The process of claim 21 wherein the lube oil feedstock is a vacuum gas oil.

24. The process of claim 21 wherein the extraction solvent is selected from the group consisting of N-Methyl-2-pyrrolidone, phenol, or furfural.

25. The process of claim 21 wherein the extract from solvent extraction is sent to a solvent recovery step.

26. The process of claim 21 wherein a portion of the hydrotreated raffinate is recycled to the liquid-continuous reactor and again hydrotreated with fresh raffinate.

27. The process of claim 26 wherein the volume ratio of recycle hydrotreated raffinate to fresh raffinate to the liquid-continuous reactor is from 0.5 to 1 to 5 to 1.

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28. The process of claim 27 wherein the volume ratio of recycle hydrotreated raffinate to fresh raffinate to the liquid-continuous reactor is from 1 to 1 to 3 to 1.

29. The process of claim 21 wherein a portion of the hydrotreated raffinate from the liquid-continuous reactor is withdrawn and saturated with hydrogen then recycled back to the liquid-continuous reactor.

30. The process of claim 21 wherein the hydrotreating catalyst is comprised of one or more catalytic metals selected from Groups VIB and Group VIII of the Periodic Table of the Elements on a refractory support.

31. The process of claim 30 wherein the support is selected from the group consisting of alumina, silica, silica alumina, titania, zirconia, and silica-alumina.

32. The process of claim 21 wherein the hydrotreating process conditions include temperatures from 400°F to 800°F and total pressures from 200 psig to 2200 psig.

33. The process of claim 32 wherein the hydrotreating process conditions include temperatures from 600°F to 750°F and total pressures from 1000 to 1500 psig.

34. The process of claim 21 wherein the raffinate stream, before it is conducted to hydrotreating is sent to a gas-liquid separation stage to remove at least a portion of any excess gas that may be present in the raffinate stream.

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35. The process of claim 21 wherein the catalytic dewaxing temperature is from 550°F to 750°F.

36. The process of claim 21 wherein the catalytic dewaxing catalyst are selected from the group consisting of crystalline aluminosilicates and silicoaluminophosphates.

37. The process of claim 36 wherein the catalytic dewaxing catalyst is a crystalline aluminosilicate selected from the group consisting of ZSM-22, ZSM-23, ZSM-35 and ZSM-48, and combinations thereof.

38. The process of claim 37 wherein the catalytic dewaxing catalyst contains a binder material selected from the group consisting of alumina, titania, silica, silica-alumina, zirconia, and combinations thereof.

39. The process of claim 37 wherein the catalytic dewaxing catalyst contains at least one metal selected from the group consisting of Pt, Pd, and Ni.

40. The process of claim 39 wherein the catalytic dewaxing catalyst also contains a metal selected from W and Mo.

41. The process of claim 21 wherein the dewaxed lube oil is subjected to hydrofinishing in the presence of hydrogen and a hydrofinishing catalyst at a temperature from 450°F to 675°F and total pressures from 400 to 2200 psig.

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42. The process of claim 41 wherein the hydrofinishing catalyst is comprised of one or more metals selected from Group VIII and Group VI of the Periodic Table of the Elements.

43. The process of claim 42 wherein the hydrofinishing catalyst contains at least one metal from Group VIII and at least one metal from Group VIB.

44. The process of claim 42 wherein the hydrofinishing catalyst is comprised of a noble metal selected from Pt and Pd on a mesoporous crystalline support.

45. The process of claim 44 wherein the mesoporous crystalline support is MCM-41.

46. A process for upgrading a Group I lube base oil, which process comprising: hydrotreating said Group I lube base oil having less than 90 wt.% saturates and greater than 0.03 wt.% sulfur in the presence of hydrogen and a hydrotreating catalyst under effective hydrotreating conditions in a liquid-continuous reactor to form a hydrotreated lube base oil having a saturate concentration greater than 90 wt.% and a sulfur concentration less than 0.03 wt.%.

47. The process of claim 46 wherein a portion of the hydrotreated Group I base oil is recycled to the liquid-continuous reactor and again hydrotreated with fresh Group I base oil.

48. The process of claim 46 wherein the volume ratio of recycle hydrotreated Group I base oil to fresh Group I base oil to the liquid-continuous reactor is from 0.5 to 1 to 5 to 1.

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49. The process of claim 48 wherein the volume ratio of recycle hydrotreated Group I base oil to fresh Group I base oil to the liquid-continuous reactor is from 1 to 1 to 3 to 1.

50. The process of claim 46 wherein a portion of the hydrotreated Group I base oil from the liquid-continuous reactor is withdrawn and saturated with hydrogen then recycled back to the liquid-continuous reactor.

51. The process of claim 46 wherein the hydrotreating catalyst is comprised of one or more catalytic metals selected from Groups VIB and Group VIII of the Periodic Table of the Elements on a refractory support.

52. The process of claim 51 wherein the support is selected from the group consisting of alumina, silica, silica alumina, titania, zirconia, and silica-alumina.

53. The process of claim 46 wherein the hydrotreating process conditions include temperatures from 400°F to 800°F and total pressures from 200 psig to 2200 psig.

54. The process of claim 53 wherein the hydrotreating process conditions include temperatures from 600°F to 750°F and total pressures from 1000 to 1500 psig.

55. The process of claim 21 wherein the catalytic dewaxing is also performed in a liquid-continuous reactor.

56. The process of claim 55 wherein a hydrofinishing step is included which conducted in a liquid-continuous reactor.