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(54) PROCESS TO PREPARE MIDDLE DISTILLATES AND BASE OILS

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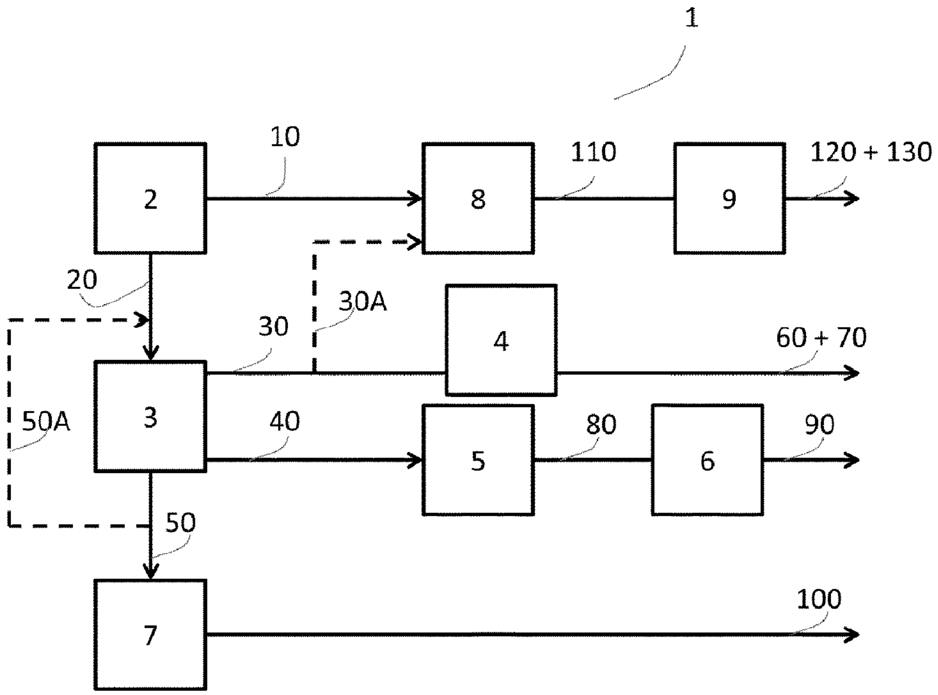
(57) **ABSTRACT**

A process to prepare a first middle distillates fraction, a second middle distillates fraction, a distillate base oil and a residual base oil by providing a Fischer-Tropsch product stream; separating the Fischer-Tropsch product stream to obtain at least a low boiling fraction, boiling below a temperature ranging from 300 to 450° C., and a high boiling fraction, boiling above a temperature ranging from 300 to 450° C.; subjecting the high boiling fraction to a hydrocracking/hydroisomerization step to obtain a partially isomerised product stream; separating the partially isomerised product stream to obtain a first middle distillates fraction, a heavy distillates fraction and a residual fraction, the residual fraction having a T5 wt. % boiling point between 400 and 650° C.; dewaxing the low boiling fraction to obtain a second middle distillates fraction; dewaxing the heavy distillates fraction to obtain a distillate base oil; and dewaxing the residual fraction to obtain a residual base oil.



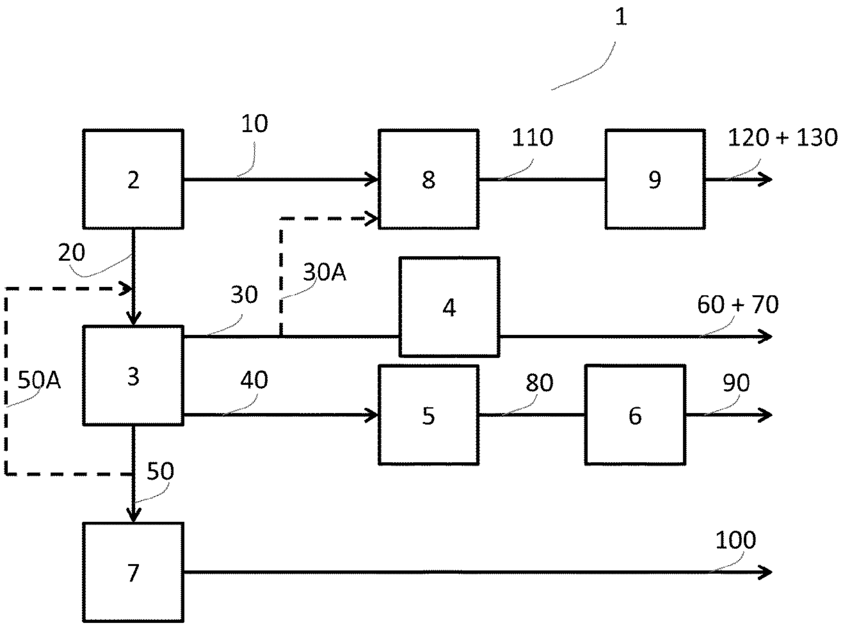


Figure 1

PROCESS TO PREPARE MIDDLE DISTILLATES AND BASE OILS

[0001] The present invention relates to a process to prepare a first middle distillates fraction, a second middle distillates fraction, a distillate base oil and a residual base oil.

[0002] It is known that Fischer-Tropsch derived middle distillates fractions typically have a high normal paraffin content (and low degree of isomerisation). Hydrocracking/hydroisomerization and/or dewaxing of the middle distillates fractions is accordingly desirable, to lower the normal paraffin content.

[0003] A lower normal paraffin content (and higher degree of isomerisation) has a positive impact on the cloud point and pour point of the middle distillates fraction as described for instance in US 2009/0200203 and WO 2011/064236.

[0004] Sufficient isomerisation is of importance, as for fuels, especially those resulting from the middle distillates fraction, good cold flow properties, such as pour point or cloud point, are desired.

[0005] WO 2009/080681 discloses a process to prepare a gas oil fraction and a residual base oil fraction, by hydrocracking/hydroisomerization a Fischer-Tropsch derived feedstock, followed by a separation step to obtain a gas oil fraction, a heavy fraction and a residual fraction, which residual fraction is dewaxed to obtain a residual base oil.

[0006] A problem of the process disclosed in WO 2009/080681 is that although this process delivers a high amount of residual base oil, the overall gas oil yield is low. Moreover, the cold-flow quality of the gas oil prepared in above process is limited by process design, i.e. the quality cannot be improved without negatively impacting the overall residual base oil yield. This is caused by the use of a hydrocracking/hydroisomerization step to control the gas oil quality, which step also affects residual base oil yield. Increasing cracking reactivity of Fischer-Tropsch derived paraffins upon increasing chain length, means that the residual fraction will be cracked faster when attempting to increase the gas oil quality according to the process described in WO 2009/080681.

[0007] The relation between cracking reactivity and chain length is for example described in Catalysis Today, 8 (1991), page 385-389.

[0008] As a result, the yield of residual base oil will always suffer when attempting to increase the gas oil cold-flow quality by enhancing isomerisation in the hydrocracking/hydroisomerization step. This is not favourable as both products are of premium economic interest.

[0009] It is an object of the invention to solve or minimize at least one of the above problems. It is a further object to provide a more efficient method for preparation of a high yield of middle distillates with excellent cold flow properties and a high yield of base oils.

[0010] One of the above or other objects may be achieved according to the present invention by providing a process to prepare a first middle distillates fraction, a second middle distillates fraction, a distillate base oil and a residual base oil, the process at least comprising the steps:

[0011] (a) providing a Fischer-Tropsch product stream;

[0012] (b) separating the Fischer-Tropsch product stream of step (a), thereby obtaining at least a low boiling fraction, boiling below a temperature in the range of from 300 to 450° C., and a high boiling fraction, boiling above a temperature in the range of from 300 to 450° C.;

[0013] (c) subjecting the high boiling fraction of step (b) to a hydrocracking/hydroisomerization step to obtain an at least partially isomerised product stream;

[0014] (d) separating the product stream of step (c), thereby obtaining a first middle distillates fraction, a heavy distillates fraction and a residual fraction, wherein the residual fraction has a T**5** wt. % boiling point of between 400 and 650° C.;

[0015] (e) dewaxing the low boiling fraction of step (b) to obtain a second middle distillates fraction;

[0016] f) dewaxing the heavy distillates fraction of step (d) to obtain a distillate base oil; and

[0017] g) dewaxing the residual fraction of step (d) to obtain a residual base oil.

[0018] It has now surprisingly been found according to the present invention that the preparation of a high yield of middle distillates with any desired quality, such as good cold flow properties, need not go at the expense of the preparation of a high yield of base oils.

[0019] As explained above, it has been found that in a Fischer-Tropsch process to prepare middle distillates and base oils, the use of a hydrocracking/hydroisomerization step to upgrade the middle distillates goes at the expense of the base oil yield.

[0020] Typically, a low boiling fraction, boiling below a temperature in the range of from 300 to 450° C. at atmospheric conditions, preferably comprises a C_3 to C_{30} fraction, more preferably comprising a C_3 to C_{23} fraction. Thus a low boiling fraction comprises Fischer-Tropsch derived middle distillates range products. Thus, separating the Fischer-Tropsch product stream into a low boiling fraction and a high boiling fraction, followed by dewaxing the low boiling fraction, results in a source of highly isomerised middle distillates, which may be used alone or blended with middle distillates obtained in step (e) to obtain a high yield of a wide range of desired middle distillate products.

[0021] In this way only the high boiling fraction needs to be subjected to the full hydrocracking/hydroisomerization step, which step is thus no longer needed to control the middle distillates quality, and results therefore in a high yield of base oils. Thus high yields of both desirable middle distillates and base oils are achieved.

[0022] Another advantage of the present invention is that since the lower boiling fraction is not subjected to a hydrocracking/hydroisomerization step, the required hydrocracking/hydroisomerization unit size can be reduced.

[0023] In step (a) of the process according to the present invention a Fischer-Tropsch product stream is provided. The Fischer-Tropsch product as provided in step (a) is derived from a Fischer-Tropsch process. Fischer-Tropsch product is known in the art. By the term "Fischer-Tropsch product" is meant a synthesis product of a Fischer-Tropsch process. In a Fischer-Tropsch process synthesis gas is converted to a synthesis product. Synthesis gas or syngas is a mixture of hydrogen and carbon monoxide that is obtained by conversion of a hydrocarbonaceous feedstock. Suitable feedstock include natural gas, crude oil, heavy oil fractions, coal, biomass and lignite. A Fischer-Tropsch product may also be referred to a GTL (Gas-to-Liquids) product.

[0024] The preparation of a Fischer-Tropsch product has been described in e.g. WO 2003/070857.

[0025] The Fischer-Tropsch product of the Fischer-Tropsch process is usually separated into a water stream, a gaseous stream comprising unconverted synthesis gas, carbon dioxide, inert gases and C1 to C2, and a C3+ product stream by distillation. Commercially available equipment can be used. The distillation may be carried out at atmospheric pressure, but also reduced pressure may be used. By Fischer-Tropsch product stream in step (a) is preferably meant the C3+ product stream.

[0026] In step (b) the Fischer-Tropsch product stream provided in step (a) is separated to obtain at least a low boiling fraction boiling below a temperature in the range of from 300 to 450° C. at atmospheric conditions and a high boiling fraction boiling above a temperature in the range of from 300 to 450° C. at atmospheric conditions.

[0027] Preferably, in step (b) the Fischer-Tropsch product stream provided in step (a) is separated to obtain at least a low boiling fraction in the distillate range with a T95 wt. % atmospheric boiling point in the range of from 300 to 450° C. and in a high boiling fraction in the atmospheric residue range with a T5 wt. % atmospheric boiling point in the range of from 300 and 450° C.

[0028] By boiling points at atmospheric conditions is meant atmospheric boiling points, which boiling points can be determined using methods such as ASTM D2887 or ASTM D7169.

[0029] The separation is preferably performed by means of a distillation at atmospheric or slightly elevated pressure conditions.

[0030] The low boiling fraction, boiling below a temperature range of 300 to 450° C. at atmospheric conditions, preferably comprises a C_3 to C_{30} fraction, more preferably comprising a C_3 to C_{23} fraction. The high boiling fraction, boiling above 300 to 450° C. at atmospheric conditions, preferably comprises a C_{30} to C_{200} fraction, more preferably a C_{23} to C_{200} fraction, but may also contain C200+ molecules depending on the Fischer-Tropsch synthesis conditions.

[0031] In step (c) the high boiling fraction of step (b) is subjected to a hydrocracking/hydroisomerization step to obtain an at least partially isomerised product. It has been found that the amount of the isomerised product is dependent on the hydrocracking/hydroisomerization conditions. Hydrocracking/hydroisomerization processes are known in the art and therefore not discussed here in detail. Hydrocracking/hydroisomerization and the effect of hydrocracking/hydroisomerization conditions on the amount of isomerised product are for example described in Chapter 6 of "Hydrocracking Science and Technology", Julius Scherzer; A. J. Cruia, Marcel Dekker, Inc, New York, 1996, ISBN 0-8247-9760-4.

[0032] The preparation of the at least partially isomerised feedstock in step (c) has been described in e.g. WO 2009/080681.

[0033] In step (d) the isomerised feedstock of step (c) is separated by means of distillation into a middle distillates fraction, a heavy distillates fraction and a residual fraction. The middle distillates fraction may preferably be obtained via multiple distillation steps.

[0034] Preferably, in step (d) the isomerised feedstock of step (c) is initially separated by means of distillation into a light fraction, the heavy distillates fraction and the residual fraction.

[0035] The distillation may be performed in one or more steps.

[0036] The light fraction may be obtained from the isomerised feedstock by distillation at atmospheric or at near atmospheric conditions.

[0037] The heavy distillates fraction and the residual fraction may be obtained from the isomerised feedstock by vacuum distillation. Preferably this vacuum distillation is performed at a pressure of between 1 and 250 mbar, more preferably between 10 and 100 mbar and most preferably between 10 and 75 mbar.

[0038] The light fraction typically has a T95 wt. % between 200 and 420 C, more in particular between 300 and 400° C. T95 wt. % is the temperature corresponding to the atmospheric boiling point at which a cumulative amount of 95% of the product is recovered. A gas chromatographic method such as ASTM D2887 can be used to determine the level of recovery.

[0039] The light fraction comprises a first middle distillates fraction, which middle distillates fraction may be separated by distillation. This may preferably be by atmospheric distillation.

[0040] The first middle distillates fraction of step (d) preferably has a T10 wt. % boiling point from **150** to 250° C., more preferably from 175 to 225° C. and a T90 wt. % boiling point from 300 to 400° C., more preferably from 330 to 370° C.

[0041] Suitably, the first middle distillates fraction has a cetane number according to ASTM D-613 greater than 70, suitably from 74 to 85.

[0042] The cloud point of the first middle distillates fraction as obtained in step (d) according to the International Standard ISO 3015 is preferably below −15° C. and more preferably below −20° C.

[0043] The cold filter plugging point (CFFP) of the first middle distillates fraction of step (d) according to European Standard EN 116 is preferably below −10° C., more preferably below −15° C.

[0044] Optionally, the first middle distillates fraction may be further dewaxed to obtain a dewaxed first middle distillates fraction. Preferred dewaxing conditions are the same as those described below for step (e)

[0045] Further it is preferred that the first middle distillates fraction comprises Fischer-Tropsch derived gas oil and Fischer-Tropsch derived kerosene.

[0046] The heavy distillate preferably has a T10 wt. % boiling point of between 200 and 450° C. and preferably between 300 and 420° C. The heavy distillate fraction will have an intermediate boiling range. Such a fraction preferably has a T90 wt % boiling point between 400 and 650° C., preferably between 450 and 600° C.

[0047] In an alternative embodiment of the process according to the present invention, a part of the heavy distillate fraction of step (d) is combined with the high boiling fraction of step (b) before step (c). In this way, at least part of the heavy distillate fraction obtained in step (d) may be recycled to step (c).

[0048] Suitably, between 20 to 70 wt. % of the heavy fraction of step (d) is recycled to step (c), more preferably between 30 to 60 wt. % and most preferably between 30 to 50 wt. %.

[0049] The residual fraction of step (d) typically has a low pour point of below 150° C., more preferably below 130° C. and even more preferably below 110° C. The residual fraction has a T5 wt. % boiling point of between 400 and 650° C., preferably between 450 and 600° C.

[0050] T**5** is the temperature corresponding to the atmospheric boiling point at which a cumulative amount of 5% of the product is recovered, determined using for example a gas chromatographic method such as ASTM D7169.

[0051] In step (e) the low boiling fraction of step (b) is dewaxed to obtain a second middle distillates fraction.

[0052] Typically dewaxing processes are catalytic dewaxing and solvent dewaxing. Catalytic and solvent dewaxing processes are known in the art and therefore not described here in detail. Typical catalytic and solvent dewaxing processes are for example described in Chapter 7 and 8 of "Lubricant base oil and wax processing", Avilino Sequeira, Jr., Marcel Dekker, Inc, New York, 1994, ISBN 0-8247-9256-4.

[0053] Dewaxing of the low boiling fraction in step (e) is preferably performed by means of a catalytic dewaxing process. Typical catalytic dewaxing processes are for example described in WO 2009/080681 and WO 2012/055755.

[0054] Suitably, catalytic dewaxing is performed in the presence of a catalyst comprising a molecular sieve and a group VIII metal.

[0055] Suitable dewaxing catalyst are heterogeneous catalysts comprising molecular sieve, more suitably intermediate pore size zeolites and optionally in combination a metal having a hydrogenation function, such as the Group VIII metals. Preferably, the intermediate pore size zeolites have a pore diameter of between 0.35 and 0.8 nm.

[0056] Preferably, catalytic dewaxing is performed in the presence of a catalyst comprising a molecular sieve and a group VIII metal, wherein the molecular sieve is selected from a group consisting of a MTW, MTT, TON type molecular sieve, ZSM-48 and EU-2.

[0057] In the present invention, the reference to ZSM-48 and EU-2 is used to indicate that all zeolites can be used that belong to the ZSM-48 family of disordered structures also referred to as the *MRE family and described in the Catalog of Disorder in Zeolite Frameworks published in 2000 on behalf of the Structure Commission of the International Zeolite Assocation. Even if EU-2 would be considered to be different from ZSM-48, both ZSM-48 and EU-2 can be used in the present invention. Zeolites ZBM-30 and EU-11 resemble ZSM-48 closely and also are considered to be members of the zeolites whose structure belongs to the ZSM-48 family. In the present application, any reference to ZSM-48 zeolite also is a reference to ZBM-30 and EU-11 zeolite.

[0058] Besides ZSM-48 and/or EU-2 zeolite, further zeolites can be present in the catalyst composition especially if it is desired to modify its catalytic properties. It has been found that it can be advantageous to have present zeolite ZSM-12 which zeolite has been defined in the Database of Zeolite Structures published in 2007/2008 on behalf of the Structure Commission of the International Zeolite Assocation.

[0059] Suitable Group VIII metals are nickel, cobalt, platinum and palladium. Preferably, a Group VIII metal is platinum or palladium.

[0060] The dewaxing catalyst suitably also comprises a binder. The binder can be non-acidic. Examples of suitable binders are clay, silica, titania, zirconia, alumina, mixtures and combinations of the above and other binders known to one skilled in the art.

[0061] Preferably the catalyst comprises a silica or a titania binder.

[0062] The second middle distillates fraction of step e) preferably has a T10 wt. % boiling point from 150 to 250° C., more preferably from 175 to 225° C. and a T90 wt. % boiling point from 300 to 400° C., more preferably from 330 to 370° C.

[0063] Suitably, the second dewaxed middle distillates fraction has a cetane number according to ASTM D-613 greater than 70, suitably from 74 to 85.

[0064] The cloud point of the second dewaxed middle distillates as obtained in step (e) according to the International Standard ISO 3015 is preferably below −40° C. and more preferably below −50° C.

[0065] The cold filter plugging point (CFFP) of the second middle dewaxed distillates fraction of step (e) according to European Standard EN 116 is preferably below −30° C., more preferably below −40° C.

[0066] Further it is preferred that the second middle distillates fraction comprises Fischer-Tropsch derived gas oil and Fischer-Tropsch derived kerosene.

[0067] Suitably, C_3 to C_9 is separated from the second dewaxed middle distillates fraction by distillation, preferably at atmospheric conditions. By a C_3 to C_9 fraction is meant a Fischer-Tropsch derived naphtha fraction.

[0068] In an another embodiment, the present process suitably comprises a further step (h) wherein prior to performing step (e), the first middle distillates fraction of step (d) or part of it is combined with the low boiling fraction of step (b) to obtain a mixture and wherein this mixture is dewaxed to obtain a third middle distillates fraction. The fraction of the first middle distillates that may be combined with the low boiling fraction of step (b) may cover 0-100%.

[0069] In a preferred embodiment, the process of the present invention comprises a further step (i), wherein the first middle distillates fraction of step (d) or part of it is combined with the second middle distillates fraction of step (e) to obtain a combined middle distillates product. The fraction of the first middle distillates that may be combined with the second middle distillates fraction of step (e) may cover 0-100%.

[0070] In a further aspect the present invention provides a middle distillate product obtainable by the process according to the present invention.

[0071] In step (f) of the process according to the present invention the heavy distillates fraction of step (d) is dewaxed to obtain a distillate base oil.

[0072] Dewaxing of the heavy distillates fraction in step (f) is preferably performed by means of a catalytic dewaxing process. Typical catalytic dewaxing processes are for example described in WO 2009/080681 and WO 2012/055755.

[0073] Preferred catalytic dewaxing conditions are the same as described above for step (e).

[0074] In a further aspect the present invention provides a distillate base oil obtainable by the process according to the present invention.

[0075] In a preferred embodiment a distillate base oil is separated into further base oils by distillation. Suitably, these further base oils have different kinematic viscosities at 100° C. according to ASTM D-445 between 2 to 10 mm^2/s, preferably between 2 to 8 mm^2/s.

[0076] The pour point of the distillate base oil according to ASTM D-5950 is below −5° C., preferably below −10° C., and more preferably below −15° C.

[0077] The distillate base oil has a cloud point according to ASTM D-2500 of below −10° C., more preferably below −15° C. and most preferably below −20° C.

[0078] In step (g) of the process of the present invention, the residual fraction of step (d) is dewaxed to obtain a residual base oil.

[0079] Dewaxing of the residual fraction in step (g) is preferably performed by means of a catalytic dewaxing process. Typical catalytic dewaxing processes are for example described in WO 2009/080681 and WO 2012/055755.

[0080] Preferred catalytic dewaxing conditions are the same as described above for step (e).

[0081] In another aspect the present invention provides a residual base oil obtainable by the process according to the present invention.

[0082] The residual base oil will preferably have a kinematic viscosity at 100° C. according to ASTM D-445 of from 15 to 35 mm^2/s, preferably from 15 to 30 mm^2/s, more preferably from 20 to 30 mm^2/s, and most preferably 23 to 26 mm^2/s.

[0083] The pour point of the residual base oil according to ASTM D-5950 is below 0° C., preferably below −20° C., more preferably below −40° C. and most preferably below −50° C.

[0084] The residual base oil has a cloud point according to ASTM D-2500 of between −50° C. and +80° C.

[0085] In a preferred embodiment the process of the present invention comprises a further step (j) wherein at least part of the residual fraction of step (d) is combined with the high boiling fraction of step (b) before step (c). In this way, at least part of the residual fraction obtained in step (d) may be recycled to step (c).

[0086] Suitably, between 20 to 70 wt. % of the residual fraction of step (d) is recycled to step (c), more preferably between 30 to 60 wt. % and most preferably between 30 to 50 wt. %.

[0087] FIG. **1** schematically shows a process scheme of the process scheme of a preferred embodiment of the process according to the present invention.

[0088] For the purpose of this description, a single reference number will be assigned to a line as well as a stream carried in that line.

[0089] The process scheme is generally referred to with reference numeral **1**.

[0090] In a Fischer-Tropsch process reactor **2** a Fischer-Tropsch product stream is obtained (not shown). This product is separated in a distillation column (not shown) into a fraction **10** boiling below a temperature in the range of from 300 to 450° C. at atmospheric conditions and a fraction **20** boiling above a temperature in the range of from 300 to 450° C. at atmospheric conditions. The high boiling fraction **20** is fed to a hydrocracking/hydroisomerization reactor **3** wherein part of the components boiling above a temperature in the range of from 300 to 450° C. are converted to product boiling below a temperature in the range of from 300 to 450° C. The effluent (not shown) of reactor **3** is distilled in a distillation column (not shown) to recover a first middle distillates fraction **30**, a heavy distillates fraction **40** and a residual fraction **50**. The middle distillates fraction **30** is distilled in a distillation column **4** to recover a gas oil **60** and kerosene **70**. The heavy distillates fraction **40** is fed to a catalytic dewaxing reactor **5** to obtain a distillate base oil **80**. The effluent **80** of reactor **5** is distilled in distillation column **6** to recover further base oils **90** with different kinematic viscosities at 100° C. between 2 and 10 mm^2/s, preferably between 2 and 8 mm^2/s. Part of the residual fraction **50** is fed to a catalytic dewaxing reactor **7** to obtain a residual base oil **100** with a kinematic viscosity at 100° C. between 15 and 35 mm^2/s. Preferably, part of the residual fraction **50**A is recycled to reactor **3** by combining **50**A with the high boiling fraction **20**.

[0091] The low boiling fraction **10** is fed to a catalytic dewaxing reactor **8** wherein the low boiling fraction is converted to a second dewaxed middle distillates fraction **110**. The middle distillates fraction **110** is distilled in distillation column **9** to recover a gas oil **120** and kerosene **130**.

[0092] Suitably, a C3-C9 fraction (=Naphtha fraction) (not shown) is distilled from the middle distillates fraction **110** prior to the distillation in column **9**. Optionally, part of first middle distillates fraction **30**A is fed to catalytic dewaxing reactor **8**.

1. A process to prepare a first middle distillates fraction, a second middle distillates fraction, a distillate base oil and a residual base oil, the process comprising the steps:

(a) providing a Fischer-Tropsch product stream;

(b) separating the Fischer-Tropsch product stream of step (a), thereby obtaining at least a low boiling fraction, boiling below a temperature in the range of from 300 to 450° C., and a high boiling fraction, boiling above a temperature in the range of from 300 to 450° C.;

(c) subjecting the high boiling fraction of step (b) to a hydrocracking/hydroisomerization step to obtain an at least partially isomerised product stream;

(d) separating the product stream of step (c), thereby obtaining a first middle distillates fraction, a heavy distillates fraction and a residual fraction, wherein the residual fraction has a T5 wt. % boiling point of between 400 and 650° C.;

(e) dewaxing the low boiling fraction of step (b) to obtain a second middle distillates fraction;

f) dewaxing the heavy distillates fraction of step (d) to obtain a distillate base oil; and

g) dewaxing the residual fraction of step (d) to obtain a residual base oil.

2. A process according to claim **1**, wherein the first middle distillates fraction of step (d) has a T10 wt. % boiling point from 150 to 250° C. and a T90 wt. % boiling point from 300 to 400° C.

3. A process according to claim **1**, wherein dewaxing is performed by means of a catalytic dewaxing process in the presence of a catalyst comprising a molecular sieve and a group VIII metal.

4. A process according to claim **3**, wherein the molecular sieve is selected from a group consisting of a MTW, MTT, TON type molecular sieve, ZSM-48 and EU-2.

5. A process according to claim **3**, wherein the Group VIII metal is platinum or palladium.

6. A process according claim **3**, wherein the catalyst comprises a silica or titania binder.

7. A process according to claim **1**, wherein the second middle distillates fraction of step (e) has a T10 wt. % boiling point from 150 to 250° C. and a T90 wt. % boiling point from 300 to 400° C.

8. A process according to claim **1**, further comprising step (h), wherein prior to performing step (e) the first middle distillates fraction of step (d) or part of it is combined with the low boiling fraction of step (b) to obtain a mixture and wherein this mixture is dewaxed to obtain a third middle distillates fraction.

9. A process according to claim **1**, further comprising step (i) wherein the first middle distillates fraction of step (d) or part of it is combined with the second middle distillates fraction of step (e) to obtain a combined middle distillates product.

10. A process according to claim **1**, further comprising step (j) wherein before step (c) at least part of the residual fraction obtained in step (d) is combined with the high boiling fraction obtained in step (b).

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