

SPECIFICATION

Light lubricating base oils

The invention relates to processes for the production of light lubricating base oil from vacuum gas oil alone or in admixture with deasphalted oil.

5 The invention provides a process for the production of light lubricating base oil, the process comprising hydrodesulphurizing a feed oil which is vacuum gas oil or vacuum gas oil admixed with deasphalted oil of vacuum residue, solvent dewaxing and vacuum redistilling the hydrodesulphurized oil, the solvent dewaxing and vacuum redistillation being carried out in either order, and solvent extracting the oil fractions obtained by the solvent dewaxing and vacuum redistillation. 5

10 The crude oil used in this process is not limited and can be selected appropriately according to the kind of light lubricating base oil to be produced and the hydrodesulphurization conditions etc. Usually, any crude oil such as Kuwait, Arabian Light, Umm Shaif, Basrah and the like can be used alone or in admixture. 10

As a feed material, vacuum gas oil, which can be obtained by distilling the above crude oil in atmospheric distillation equipment followed by distilling the atmospheric distillation residue remaining in the bottom in vacuum distillation equipment, can be used alone or in admixture with oil deasphalted from vacuum distillation residue. The properties of the aforescribed vacuum gas oil are not limited and can be appropriately selected according to conditions relevant to subsequent processes. Usually, vacuum gas oil with a specific gravity of from 0.87 to 0.91 and a sulphur content of from 1.9% to 3.5% by weight can be used. The properties of deasphalted oil which can be used are not limited. Usually deasphalted oil having an ASTM colour of 6.5 or less, a sulphur content of 3.5% by weight or less and a carbon residue of 3.5% by weight or less can be used. If a mixture of vacuum gas oil with deasphalted oil is used, the mixture preferably comprises from 90% to 95% by volume of vacuum gas oil and from 5% to 10% by volume of deasphalted oil. 15 20

25 In the process according to the invention, the feed material is first subjected to hydrodesulphurization. This may be performed under conventional conditions and also under milder conditions. The preferable conditions for the hydrodesulphurization are a temperature of from 340°C to 425°C, a pressure of from 30 to 80 kg/cm² Gauge, a liquid hourly space velocity of from 0.5 to 5 hr⁻¹ and a hydrogen feed rate of from 100 to 1000 NI/l oil, the hydrogen feed containing at least 60% of hydrogen. As catalyst for the hydrodesulphurization, there is preferably used a conventional catalyst comprising mainly from 1% to 9% by weight of cobalt oxide or from 0.5% to 6% by weight of nickel oxide and from 10% to 25% by weight of molybdenum oxide supported on an alumina or silica-alumina carrier. Examples of such a catalyst include Nalcome 477 and 479 (trade name: manufactured by Nalco Chemical Co.) and CDS—D9 (trade name: manufactured by Shokubai Kaisei Kogyo KK.). The hydrodesulphurization is preferably carried out until the sulphur content of the oil is decreased to 0.3% by weight or less. 25 30 35

According to the invention, both solvent dewaxing and vacuum redistillation are carried out after the hydrodesulphurization. The solvent dewaxing may precede or follow the vacuum redistillation. The solvent dewaxing is carried out with a solvent such as a mixture of methyl ethyl ketone and toluene under conventional conditions. The vacuum redistillation is carried out according to well-known vacuum distillation techniques so as to collect the fractions having boiling points of 250°C or above. 40

The solvent extraction is carried out after the solvent dewaxing and/or the vacuum redistillation mentioned above. Suitable solvents are those having an affinity for aromatic compounds, for example phenol, furfural and N-methyl-2-pyrrolidone. The solvent extraction may be performed according to conventional methods, and is usually performed according to the counter-current contacting method. In such an operation, the lubricating oil is recovered from the top of the column as the raffinate. The components which are undesirable in a lubricating oil are removed from the bottom of the column as an extract which is then distilled so as to separate the solvent. As an example of operating conditions, when furfural is used as the solvent the extraction is preferably carried out using a solvent: oil ratio of from 0.5:1 to 4:1, the temperature at the top of the column being from 60°C to 110°C and at the bottom of the column being from 40°C to 80°C. 45 50

The process of the invention produces a stable high quality light lubricating base oil. Furthermore, this process can be very economically practised since the hydrodesulphurization apparatus used for refining fuel oil also can be used as the apparatus for the practice of the invention. The process does not required a hydrogenation unit designed exclusively for the refining of lubricating oil. 55

The invention is illustrated by the following Examples.

EXAMPLE 1

100 l of feed stock, which was a mixture of 7.5 l of vacuum gas oil with 92.5 l of heavy gas oil obtained from 70% by volume of Umm Shaif crude oil and 30% by volume of Basrah Heavy crude oil, was hydrodesulphurized in a reaction tube filled with 300 ml of a catalyst consisting of 14.0% by weight of molybdenum oxide and 3.3% by weight of cobalt oxide supported on alumina, at a reaction temperature of 350°C a pressure of 55 kg/cm² Gauge, a hydrogen feed rate of 500 NI/l oil and a liquid hourly space velocity of 1.5 hr⁻¹. 60

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The resulting desulphurized oil was subjected to vacuum redistillation to obtain 5.9 l of desulphurized 60 neutral fraction and 79.5 l of desulphurized 150 neutral fraction.

The 5.9 l of the desulphurized 60 neutral fraction was mixed in the ratio of 3:1 with a mixed solvent of methyl ethyl ketone and toluene (hereinafter referred to as "MEK-toluene"), cooled to -27°C and then dewaxed by filtration through a filter. Thus 5.3 l of dewaxed oil (yield: 90%) was obtained.

8.0 l of the desulphurized 150 neutral fraction were mixed in a ratio of 3:1 with the MEK-toluene solvent, cooled to -27°C and then dewaxed by filtration through a filter. 6.3 l of dewaxed oil were obtained (yield: 79%). The 6.3 l of dewaxed oil was batchwise extracted with furfural at 60°C to obtain 4.4 l of refined 150 neutral oil (yield: 70%).

The properties of the feed stock, desulphurized neutral oil fraction, dewaxed oil and refined neutral oil obtained after the extraction are reported in Tables 1 to 4.

EXAMPLE 2

100 l of feed stock, which was a mixture of 80% by volume of vacuum gas oil, 10% by volume of deasphalted oil and 10% by volume of heavy gas oil obtained from 90% by volume of Arabian Light crude oil and 10% by volume of Kuwait crude oil, was hydrodesulphurized in a reaction tube filled with 300 ml of a catalyst consisting of 19.0% by weight of molybdenum oxide and 4.4% by weight of cobalt oxide supported on alumina, at a temperature of 380°C, a reaction pressure of 55 kg/cm² Gauge, a hydrogen feed rate of 500 NI/l oil and a liquid hourly space velocity of 1.9 hr⁻¹.

The resulting desulphurized oil was vacuum redistilled to obtain 35.3 l of desulphurized 60 neutral fraction and 50.3 l of desulphurized 150 neutral fraction.

The 35.3 l of the desulphurized 60 neutral fraction was mixed in a ratio of 3:1 with MEK-toluene solvent, cooled to -27°C and dewaxed by filtration through a filter. 31.6 l of the thus obtained dewaxed oil (yield: 89.7%) was batchwise extracted with furfural at 60°C to give 24.7 l of refined 60 neutral oil (yield: 78.1%).

The 50.3 l of the desulphurized 150 neutral fraction was treated as described hereinbefore to obtain 28.6 l of refined 150 neutral oil. The yield after the dewaxing was 79.9% and the yield after the refining by extraction with furfural was 71.2%. The properties of the resulting oils are reported in Tables 1 to 4.

TABLE 1

Property	Test Method	Example 1		Example 2	
		Feed Stock for hydro- desulphurization	Desulphurized Heavy Gas Oil	Feed Stock for hydro- desulphurization	Desulphurized Heavy Gas oil
Specific Gravity (15/4 °C)	ASTM-D-1298	0.9026	0.8862	0.8998	0.8808
Kinematic 50 °C	ASTM-D-445	13.19	18.06	13.10	14.08
Viscosity (cst) 98.9 °C	—		4.862		4.152
Pour Point (°C)	ASTM-D-97	+30.0	+35.0	+30.0	+27.5
Carbon Residue (wt %)	ASTM-D-189	0.22	0.01	0.35	0.06
Flash Point (°C) COC	ASTM-D-92	144	190	148	180
Sulphur Content (wt %)	ASTM-D-129	2.23	0.21	2.24	0.14
Nitrogen Content (ppm)	ASTM-D-3431	720	440	—	380
Colour	ASTM-D-2500	—	L 2.5	—	L 3.0

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TABLE 2

	Example 1		Example 2	
	60 Neutral fraction	150 Neutral fraction	60 Neutral fraction	150 Neutral fraction
Percentage distilled (vol. %)	9.0-14.9	14.9-94.4	5.0-40.3	40.3-90.5
Cut-Off Point (°C)	383-403	403-503	324-418	418-513
Specific Gravity (15/4 °C)	0.8820	0.8869	0.8740	0.8832
Kinematic @ 37.8 °C	10.52	-	9.936	-
Viscosity (cst) @ 98.9 °C	2.558	5.306	2.411	5.284
Viscosity Index (by ASTM-D-97)	73	-	58	-
Pour Point (°C)	+7.5	+30.0	+7.5	+30.0

TABLE 3

	Example 1			Example 2		
	60 Neutral fraction Dewaxed Oil	150 Neutral fraction Dewaxed Oil	Neutral fraction Raffinate	60 Neutral fraction Dewaxed Oil	150 Neutral fraction Dewaxed Oil	Neutral fraction Raffinate
Percentage (Based on the amount of distilled Desulphurized Heavy Gas Oil) (vol %)	9.0-14.9	14.9-94.4		5.0-40.3	40.3-90.5	
Dewaxing Condition	-27 5:5 89.9	-27 5:5 78.6		-27 5:5 89.7	-27 5:5 79.9	
Temperature (°C)						
MEK: Toluene (vol)						
Yield (vol %)						
Extracting Condition						
Total amount of solvent (ratio to the amount of Desulphurized Heavy Gas Oil)						
Number of times of Extraction (Yield (vol %))						
Yield based on Feed Stock (vol %)	5.3	62.5	60	31.7	40.1	60
Specific Gravity (15/4 °C)	0.8855	0.8995	4	0.8806	0.8946	4
Colour ASTM	L 1.5	2.0	2	L 1.0	L 2.5	2
Kinematic Viscosity @ 37.8 °C (cst)	11.38	43.34	70.0	9.704	41.49	71.2
@ 98.9 °C (cst)	2.626	5.789	43.8	2.416	5.750	28.6
Viscosity Index	59	75	0.5	69	82	0.8707
Pour Point (°C)	-15.0	-15.0	0.8750	-17.5	-15.0	0.5
Sulphur Content (wt %)	0.20	0.26	0.5	0.14	0.17	33.70
Total Acid Value (mg KOH/g) by ASTM-D-974)		0.02	35.90	0.02	0.07	5.381
Aniline Point (°C) (by ASTM-D-611)	69.3	83.7	5.511	81	86.2	102
% CA	18.7	16.1	98	-12.5	16.3	-10.0
Ring Analysis (by n.d. M)	25.2	24.1	0.181	0.170	21.2	0.146
% CN	56.1	59.8	0.01>	0.01>	62.5	0.01>
% CP	0.65	0.77	98.0	68.5	0.79	26.2
RA			7.0	20.2		66.9
			28.1	21.7		0.34
			64.9	58.0		
			0.34	0.69		

TABLE 4

	Example 1 (150 Neutral Oil)	Example 2 (150 Neutral Oil)
Solvent Extraction Temperature	60°C	60°C
	Colour %T	Colour %T
Fresh Oil	L 1.0 101	L 1.0 105
Oil weathered for 1 day	L 1.5	L 1.5
„ for 2 days	2.0	2.0
„ for 5 days	2.5	2.5
„ for 7 days	L 3.0	L 3.0
„ for 10 days	L 3.0 (flock)	L 3.0 (flock)
Thermal Stability 170°C (12 hours)	pass	pass
JIS K-2540 170°C (24 hours)	clouding	pass

EXAMPLE 3

The same feed stock as in Example 1 was subjected to hydrodesulphurization, solvent dewaxing, vacuum redistillation and solvent extraction successively in a similar manner as in Example 1 to obtain 5 100 neutral oil in a yield of 42.6% based on feed stock. The yields and properties of the resulting oils in each operation stage are reported in Table 5.

TABLE 5

	Desulphurized Heavy Gas Oil	Dewaxed Oil from Desulphurized Heavy Gas Oil	Vacuum Redistillation of Dewaxed Oil from Desulphurized Heavy Gas Oil	Raffinate from 100 Neutral fraction	Testing Method
Yield (vol %)	88	85.3	72.0	78.8	
Specific Gravity (15/4°C)	0.8761	0.8834	0.8862	0.8631	ASTM-D-1298
Colour			L 2.5	L 1.0	ASTM-D-2500
Kinematic Viscosity @ 40°C	19.56	19.23	23.50	22.33	ASTM-D-445
Viscosity (cst) @ 100°C	3.972	3.863	4.274	4.298	
Viscosity Index			76	96	ASTM-D-2270
Pour Point (°C)		85	-12.5	-10	ASTM-D-97
Aniline Point (°C)		-10	83.5	93.0	
Sulphur content (wt %)			0.20	0.10	ASTM-D-129
Total Acid Value (mgKOH/g)			0.01	0.01	ASTM-D-974
%CA			15.3	12.3	
Ring Analysis %CN			21.4	16.3	n.d.M Method
%Cp			62.9	71.4	
Thermal Stability (170°C x 12 hrs)	-	-	-	pass	JIS K-2540
Weather Resistance				clouding (for 7 days)	Idemitsu Method *

*1: ratio to the amount of Desulphurized Heavy Gas Oil

* This method is carried out as follows. At first, 90 cc of sample are put into a colourless, transparent candle bottle (100 cc), and then a cork with a hole is put on the candle bottle. Thereafter the candle bottle containing sample is allowed to stand outside. Occurrence of cloud is observed by naked eye once a day.

EXAMPLE 4

120 l of feed stock consisting of 29.6% of heavy gas oil obtained mainly from Kuwait crude oil, 59.8% of vacuum gas oil, 6.2% of vacuum distilled oil and 4.3% of deasphalted oil was hydrodesulphurized in a reaction tube filled with 1.0 l of a catalyst consisting of 14.0% by weight of molybdenum oxide and 3.3% by weight of cobalt oxide supported on alumina, at a reaction temperature of 390°C, a pressure of 55 kg/cm² Gauge, a hydrogen feed rate of 500 N/l oil, and a liquid hourly space velocity of 1.5 hr⁻¹.

Then 100 l of the desulphurized oil was mixed in the ratio of 4.0:1 with the MEK-toluene solvent, charged into a cooler at a velocity of 5 l per hour so as to cool and dewax at -27°C continuously by filtration with an Oliver filter. "Oliver" is a Trade Mark. 80 l of the resultant dewaxed oil (yield: 80%) were distilled under reduced pressure to obtain 50.4 l of the 100 neutral fraction. 50 l of the 100 neutral fraction were fed into the lower part of a rotating disc contactor at a rate of 60 ml per minute, contacted countercurrently with furfural with the ratio of solvent of 1.5 and extracted to obtain 39.1 l of refined 100 neutral oil (yield: 78.2%). Table 6 reports the yield in dewaxing and the properties of the product in each operation stage of Example 4.

TABLE 6

	Desulphurized Oil	Dewaxed Oil	100 Neutral Fraction (30-92%)	100 Neutral oil by Furfural extraction
Yield in Dewaxing (vol %)		80.0		82.1
Specific Gravity (15/4°C)	0.8727	0.8806	0.8882	0.8680
Colour		3.0	2.5	L 1.0
Kinematic @ 37.8°C	13.00	12.12	23.76	21.39
Viscosity (cst) @ 98.9°C	3.141	2.946	4.283	4.215
Viscosity Index	102	90	73	99
Pour Point (°C)	+20	-17.5	-12.5	-12.5
Cloud Point (°C)		-18	-11	-9
Sulphur Content (wt %)	0.13		0.2	0.06
Total Acid Value (mg KOH/g)				
Aniline Point (°C)			82.0	93.6
Refractive Index n_D^{20}				1.4803
Mean Molecular Weight			357	371
%CA			17.2	9.5
Ring Analysis % CN			19.9	23.7
% Cp			62.9	66.7
Thermal Stability (170°C×12 hrs)				pass

**CLAIMS**

1. A process for the production of light lubricating base oil, the process comprising hydrodesulphurizing a feed oil which is vacuum gas oil or vacuum gas oil admixed with deasphalted oil of vacuum residue, solvent dewaxing and vacuum redistilling the hydrodesulphurized oil, the solvent dewaxing and vacuum redistillation being carried out in either order, and solvent extracting the oil fractions obtained by the solvent dewaxing and vacuum redistillation. 5
2. A process according to claim 1 wherein the solvent dewaxing precedes the vacuum redistillation.
3. A process according to claim 1 wherein the solvent dewaxing follows the vacuum redistillation. 10
4. A process according to any preceding claim, wherein the vacuum gas oil has a specific gravity of from 0.87 to 0.91 and a sulphur content of from 1.9% to 3.5% by weight. 10
5. A process according to any preceding claim in which the feed oil is vacuum gas oil admixed with deasphalted oil of vacuum residue, and the deasphalted oil has an ASTM colour of 6.5 or less, a sulphur content of 3.5% by weight or less, and a residual carbon content of 3.5% by weight or less. 15
6. A process according to any preceding claim, wherein the hydrodesulphurization is carried out using a catalyst comprising (i) from 1% to 9% by weight of cobalt oxide or from 0.5% to 6% by weight of nickel oxide, and (ii) from 10% to 25% by weight of molybdenum oxide, supported on an alumina or silica-alumina carrier, at a temperature of from 340°C to 425°C, a pressure of from 30 to 80 kg/cm² Gauge, a liquid hourly space velocity of 0.5—5 hr⁻¹ and a hydrogen feed rate of from 100 to 1000 NI/oil, the hydrogen feed containing at least 60% of hydrogen. 20
7. A process according to any preceding claim wherein the vacuum redistillation is carried out to produce fractions which are distilled out at temperatures of at least 250°C under atmospheric pressure.
8. A process according to any preceding claim wherein the solvent used for solvent dewaxing is a mixture of methyl ethyl ketone and toluene.
9. A process according to any preceding claim wherein the solvent used in the solvent extraction stage is phenol, furfural or N-methyl-2-pyrrolidone. 25
10. A process for the production of light lubricating base oil, the process being substantially as described herein with reference to any of the Examples.