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(54) Title: TREATMENT OF WAXES

(57) Abstract

A process for treating a wax includes thermally cracking, in a cracking stage, a feed wax having a congealing point X °C, to produce, as a primary product, a lighter wax having a congealing point Y °C, where X > Y. The feed wax is typically a Fischer–Tropsch derived hard wax.

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TREATMENT OF WAXES

THIS INVENTION relates to the treatment of waxes. It relates in particular to a process for treating a wax, and to a wax when produced by the process.

According to the invention, there is provided a process for treating a wax, which process includes thermally cracking, in a cracking stage, a feed wax having a congealing point X°C, to produce, as a primary product, a lighter wax having a congealing point Y°C, where $X>Y$.

By "primary product" is meant that the lighter wax forms at least 52% m/m of the product produced by the cracking of the feed wax.

The feed wax may be a hard wax of which the congealing point, X, may be above 60°C. Preferably, the congealing point, X, of the hard wax is above 90°C.

In particular, the feed wax may be a Fischer-Tropsch derived hard wax.

By "Fischer-Tropsch derived" in relation to the hard wax is meant a wax fraction or cut obtained by subjecting a synthesis gas comprising mainly carbon monoxide and hydrogen to a low temperature

(200 - 250°C) or high temperature (300 - 360°C) Fischer-Tropsch synthesis reaction in a fixed or slurry catalyst bed, the catalyst of which is a Fischer-Tropsch catalyst, which is normally iron and/or cobalt based. Typically, the Fischer-Tropsch derived hard wax comprises mostly normal paraffins, most of which have a carbon number greater than 18, i.e. greater than C_{18}, and some isomers, olefins and lighter components. Generally, the individual components of the wax have a large spread of carbon numbers and thus a large spread of boiling points.

In particular, the Fischer-Tropsch derived hard wax may be a product of a low temperature Fischer-Tropsch synthesis process, using either a slurry bed reactor or a fixed bed reactor. A significant portion of the hard wax, e.g. at least about 95% m/m, may comprise hydrocarbons of C_{32} and heavier and may consist predominantly of normal paraffins, but may also include isomers and olefins.

The process may include pre-treating the feed wax, and in particular the Fischer-Tropsch derived hard wax to remove a lighter fraction, prior to cracking the feed wax in the cracking stage. The hard wax may thus be a wax produced by the Fischer-Tropsch synthesis process, from which a lighter fraction has been removed. Removal of the lighter fraction may be by any suitable method known to a person skilled in the art.

If desired, at least a portion of the feed wax may be hydrogenated prior to cracking it. Hydrogenation may be effected by contacting the feed wax with hydrogen in the presence of a suitable catalyst at an elevated temperature and an elevated pressure, using any suitable method known to those skilled in the art.

The feed wax may be pre-heated to a temperature of at least 230°C, prior to cracking the feed wax in the cracking stage. Preferably, the feed wax is preheated to a temperature of at least 260°C, prior to cracking the feed wax in the cracking stage.

The thermal cracking is thus effected at an elevated temperature, which may be in the range of 360°C to 600°C. Preferably, the thermal cracking is effected at a temperature in the range of 420°C to 520°C, e.g. at about 460°C.

The cracking of the hard wax may be conducted at an elevated pressure. The elevated pressure may be in the range of 100kPa$_g$ to 1500kPa$_g$, more preferably in the range of 350kPa$_g$ to 600kPa$_g$, e.g. at about 500kPa$_g$. It will be appreciated that, by raising the pressure in the cracking stage, the equilibrium between vapour present or formed during the cracking of the feed wax and the liquid wax in the cracking stage, is effected. At lower pressures, removal of lighter components from the cracking stage is thus facilitated.

The cracking may be effected for a sufficient period of time to obtain a desired degree of cracking, e.g. from about 30 seconds to about 30 minutes. However, it is to be appreciated that the residence time of the feed wax in the cracking stage, i.e. the cracking time, required to obtain a desired degree of cracking, is dependant on the temperature at which the cracking occurs, and the residence time may thus fall outside the range provided above.

The cracking may be effected in the presence of an inert gas, e.g. steam or nitrogen. The inert gas may be used to control the residence time of the feed wax in the cracking stage.

A light fraction of the lighter wax formed during the cracking stage and the inert gas may be separated from any uncracked feed wax and/or from a heavier fraction of the lighter wax formed during the cracking stage by any suitable separation method known to those skilled in the art, e.g. stripping and/or distillation. At least a portion of any uncracked feed wax and/or at least a portion of the heavier fraction of the lighter wax may be recycled to the cracking stage, where it is cracked or recracked together with fresh feed wax, thus increasing the yield of the process.

The products from the cracking stage may be passed to a quenching stage, to lower the temperature and thus to halt the cracking.

The invention extends to a lighter wax when produced by the process as hereinbefore described. Preferably, the congealing point or temperature, Y, of the lighter wax is in the range 60°C to 90°C.

The lighter wax may be separated into different product fractions by using methods known to those skilled in the art, such as distillation. The product fractions include, but are not limited to

- the light fraction as hereinbefore described, which may be used for example as a solvent, a fuel or a feedstock for other processes;
- a number of medium wax fractions having a congealing temperature of between 45°C and 70°C which typically are useful in candles and other applications;
- a number of hard wax fractions having a congealing temperature of between 70°C and 110°C which are useful in hot melt adhesives, polymer processing, inks and other applications; and/or
- any combination of the foregoing products for which many commercial applications exist *e.g.* as polishes, emulsions and specialized applications.

Some or all of these product fractions may be hydrotreated or oxidised to modify the product fraction characteristics.

The Applicant has surprisingly found that by subjecting a hard wax, particularly a Fischer-Tropsch derived hard wax to a mild thermal cracking process, a lighter wax is produced as a primary product. The lighter wax has a similar range of hydrocarbons than the hard wax, but has higher concentrations of the lighter hydrocarbons in the range, and thus has a lower congealing temperature than the hard wax. The Applicant has also surprisingly found that the process is very flexible and that the composition (carbon distribution) of the lighter wax can be controlled to a large extent through manipulation of the temperature at which the cracking of the hard wax is effected, the residence time of the hard wax in the cracking stage, the use of an inert, and the use of a recycle as described above.

The invention will now be described, by way of example, with reference to the single accompanying diagrammatic drawing showing a simplified flow diagram of a process according to the invention for converting a Fischer-Tropsch derived hard wax to a lighter wax.

Referring to the drawing, reference numeral 10 generally indicates a process according to the invention for converting a Fischer-Tropsch derived hard wax to a lighter wax having a congealing temperature which is less than the congealing temperature of the hard wax.

The process 10 includes a feed line 12 leading to a pre-heater 14. A flow line 16 leads from the pre-heater 14 to a cracker 18. An inert feed line 20 leads to the cracker 18, and a flow line 22 leads from the cracker 18 to a cooler 24. A flow line 26 leads from the cooler 24 to a distillation column 28.

An overhead withdrawal line 30 leads from the top of the distillation column 28, and a bottom withdrawal line 32 leads from the bottom of the distillation column 28.

A recycle flow line 34 leads from the bottom withdrawal line 32 into the flow line 16. However, it is to be appreciated that other tie-in points for the recycle flow line 34, e.g. into the feed line 12, are feasible. A quench line 36 leads from the recycle flow line 34 into the flow line 22.

In use, a feedstock comprising a Fischer-Tropsch derived hard wax, typically having a congealing temperature, X, above about 90°C, enters the process 10 along the feed line 12 and is pre-heated to a temperature of 280°C in the pre-heater 14 to provide a pre-heated hard wax feedstock. If desired, the pre-heated hard wax feedstock is mixed with recycled uncracked hard wax and/or a heavier fraction of the lighter wax fed from the recycle flow line 34, before the resultant wax mixture enters the cracker 18.

In the cracker 18, the wax mixture is cracked at an elevated temperature of 480°C and at an elevated pressure of $400kPa_g$, in the presence of an inert gas such as steam or nitrogen, fed to the cracker 18 by means of the inert feed line 20. The wax mixture has a residence time in the cracker 18 of about 15 minutes. In the cracker 18, at least a portion of the wax mixture is cracked to produce a wax product which includes, as a primary product, a lighter wax having a congealing temperature, Y, which is less than the congealing temperature of the hard wax feedstock. Typically, Y is in the range of 60 - 90°C. The wax product may also include uncracked hard wax.

It is to be appreciated that, at a lower temperature in the cracker 18, a longer residence time for the hard wax in the cracker 18 would be required to obtain the same degree of cracking as when a higher temperature is used.

The wax product leaves the cracker 18 through the flow line 22, and is mixed with recycled uncracked hard wax and/or a heavier fraction of the lighter wax withdrawn from the recycle flow line 34 by means of the quench line 36. The quenched wax product enters the cooler 24, where its temperature is further reduced to about 300°C, before it is fed along the flow line 26 into the distillation column 28.

In the distillation column 28, the wax product (and thus also the lighter wax) is separated into a light hydrocarbon fraction, which includes the inert gas, and which leaves the distillation column 28 along the overhead withdrawal line 30, and a heavier hydrocarbon fraction which includes any uncracked hard wax and which leaves the distillation column 28 along the bottom withdrawal line 32. If desired, the heavier hydrocarbon fraction to be recycled is withdrawn from the bottom withdrawal line 32 and recycled into the flow line 16 by means of the recycle flow line 34, as described above.

The invention is further illustrated by the following non-limiting example:

EXAMPLE

A Fischer-Tropsch derived hard wax having a congealing temperature of 101°C and comprising 59% m/m of hydrocarbons having a carbon number above C_{49} and 21% m/m of hydrocarbons having a carbon number between C_{19} and C_{41} (See Table 1) was cracked in a cracking stage at about 440°C and about 500kPa$_g$ for a period of about 15 minutes. The cracking of the hard wax was conducted in the absence of an inert gas. A lighter wax having a congealing temperature of 88°C and comprising 24% m/m of hydrocarbons having a carbon number above C_{49} and 54% m/m of hydrocarbons having a carbon number between C_{19} and C_{41} (see Table 2) was produced, as a heavier hydrocarbon fraction of the primary product, after a light hydrocarbon fraction was removed.

TABLE 1: Composition of feedstock

Carbon number	m %
<18	0.21
18	0.07
19	0.09
20	0.12
21	0.18
22	0.24
23	0.24
24	0.24
25	0.24
26	0.25
27	0.36
28	0.41
29	0.55
30	0.80
31	1.02
32	1.24
33	1.47
34	1.66
35	1.77
36	1.95
37	2.03
38	2.07
39	2.10
40	2.11
41	2.13
42	2.12
43	2.14
44	2.15
45	2.20
46	2.25
47	2.28
48	2.33
49	2.31
50	2.35
51	2.41
52	2.46
53	2.53
54	2.47
55	2.49
56	2.48
57	2.44
58	2.39
59	2.34
60	2.29
61	2.21
62	2.13
63	2.06
64	1.97
65	1.88
66	1.83
67	1.68
68	1.58
69	1.48
70	1.39
71	1.31
72	1.21
73	1.12
74	1.07
75	1.00
76	0.94
77	0.88
78	0.82
79	0.78
80	0.72
81	0.63
82	0.58
83	0.54
84	0.52
85	0.47
86	0.38
87	0.33
88	0.30
89	0.21

TABLE 2: Composition of product

Carbon number	m %
13	0.16
14	0.19
15	0.21
16	0.25
17	0.29
18	0.47
19	0.65
20	1.50
21	2.36
22	2.83
23	3.15
24	3.28
25	3.26
26	3.23
27	3.29
28	3.34
29	3.38
30	3.40
31	3.34
32	3.40
33	3.32
34	3.32
35	3.22
36	3.20
37	3.08
38	2.94
39	2.83
40	2.68
41	2.54
42	2.40
43	2.27
44	2.11
45	1.92
46	1.80
47	1.66
48	1.53
49	1.37
50	1.19
51	1.12
52	1.13
53	1.09
54	0.95
55	0.73
56	0.71
57	0.72
58	0.77
59	0.76
60	0.71
61	0.58
62	0.59
63	0.59
64	0.54
65	0.52
66	0.52
67	0.45
68	0.43
69	0.35
70	0.32
71	0.27
72	0.22
73	0.13
74	0.16
75	0.11
76	0.11
77	0.06

From Tables 1 and 2, it is clear that the lighter wax produced has a similar range of hydrocarbons as the Fischer-Tropsch derived hard wax, but has higher concentrations of the lighter hydrocarbons in the range, which explains the lower congealing temperature of the lighter wax.

It is an advantage of the process 10 of the invention, as illustrated, that it can be used to convert a hard wax, particularly a Fischer-Tropsch derived hard wax to a lighter wax, and even more particularly that it can covert a Fischer-Tropsch derived hard wax having a congealing temperature of 90°C or higher, to a lighter wax. It is yet a further advantage of the process 10, that it is flexible, allowing the composition of the lighter wax produced to be easily controlled.

CLAIMS

1. A process for treating a wax, which process includes thermally cracking, in a cracking stage, a feed wax having a congealing point X°C, to produce, as a primary product, a lighter wax having a congealing point Y°C, where X>Y.

2. A process as claimed in claim 1, in which the feed wax is a hard wax of which the congealing point, X, is above 60°C.

3. A process as claimed in claim 2, in which the congealing point, X, of the hard wax is above 90°C.

4. A process as claimed in claim 2 or claim 3, in which the hard wax is a Fischer-Tropsch derived hard wax.

5. A process as claimed in claim 4, in which the Fischer-Tropsch derived hard wax is a product of a low temperature Fischer-Tropsch synthesis process.

6. A process as claimed in any one of claims 2 to 5 inclusive, in which at least 95% of the hard wax comprises hydrocarbons of C_{32} and heavier and in which the hard wax consists predominantly of normal paraffins.

7. A process as claimed in any one of the preceding claims, which includes pre-treating the feed wax to remove a lighter fraction, prior to cracking the feed wax in the cracking stage.

8. A process as claimed in any one of the preceding claims, in which the feed wax is pre-heated to a temperature of at least 230°C, prior to cracking the feed wax in the cracking stage.

9. A process as claimed in any one of the preceding claims, in which at least a portion of the feed wax is hydrogenated, prior to cracking the feed wax in the cracking stage.

10. A process as claimed in any one of the preceding claims, in which the thermal cracking is effected at an elevated temperature in the range of 360°C to 600°C.

11. A process as claimed in claim 10, in which the thermal cracking is effected at a temperature in the range of 420°C to 520°C.

12. A process as claimed in any one of the preceding claims, in which the thermal cracking is effected at an elevated pressure in the range of $100kPa_g$ to $1500kPa_g$.

13. A process as claimed in claim 12, in which the thermal cracking is effected at a pressure in the range of $350kPa_g$ to $600kPa_g$.

14. A process as claimed in any one of the preceding claims, in which the thermal cracking is effected in the presence of an inert gas.

15. A process as claimed in claim 14, in which a light fraction of the lighter wax formed during the cracking stage and the inert gas are separated from any uncracked feed wax and/or from a heavier fraction of the lighter wax formed during the cracking stage, and in which at least a portion of any uncracked feed wax and/or at least a portion of the heavier

fraction of the lighter wax is recycled to the cracking stage, where it is cracked or recracked together with fresh feed wax.

16. A process as claimed in any one of the preceding claims, in which the products from the cracking stage is passed to a quenching stage, to lower the temperature and thus to halt the cracking.

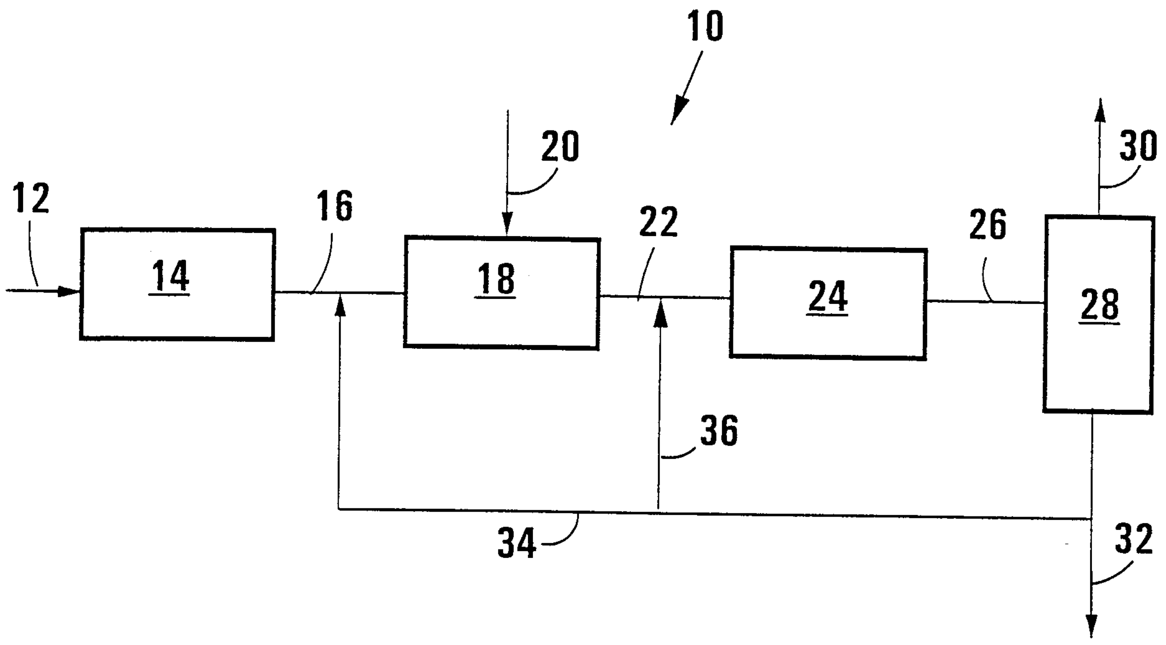
17. A wax when produced by the process as claimed in any one of claims 1 to 16 inclusive.

18. A wax as claimed in claim 17, which has a congealing point, Y, which is in the range 60°C to 90°C.

19. A process as claimed in claim 1, substantially is herein described and illustrated.

20. A wax as claimed in claim 17, substantially as herein described and illustrated.

21. A new process, or a new wax, substantially as herein described.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INTERNATIONAL SEARCH REPORT

Interr nal Application No
PCT/EP 99/00293

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C10G73/42 C10G9/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C10G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 773 237 A (RUHRCHEMIE) see claims 1-4,15-17 see page 1, line 20 - line 41 see examples 1-3 -----	1-4,6, 10-12, 15,17-21

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 773237 A		NONE	

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