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(71) Applicant: **NV BEKAERT SA** [BE/BE]; Bekaertstraat 2, B-8550 Zwevegem (BE).

(71) Applicant (for CN only): **BEKAERT NEW MATERIALS (SUZHOU) CO., LTD.** [CN/CN]; Ting Lan Xiang 9, Suzhou Industrial Park, Suzhou, Jiangsu 215026 (CN).

(72) Inventors: **WANG, Zhong**; Ting Lan Xiang 9, Suzhou Industrial Park, Suzhou, Jiangsu 215026 (CN). **YAO, Xi-angdong**; Ting Lan Xiang 9, Suzhou Industrial Park, Suzhou, Jiangsu 215026 (CN). **LV, Hongbin**; Ting Lan Xiang 9, Suzhou Industrial Park, Suzhou, Jiangsu 215026 (CN). **GOUX, Aurelie**; Rue Pierre 18, B-7540 Kain (BE). **DE BAERDEMAEKER, Jeremie**; Statiestraat 20, B-8570 Anzegem (BE).

(74) Agent: **KING & WOOD MALLESONS**; 20th Floor, East Tower, World Financial Centre, No. 1 Dongsanhuan Zhonglu, Chaoyang District, Beijing 100020 (CN).

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(54) Title: FILTRATION MEDIA AND COALESCING FILTER TO REMOVE WATER FROM LIQUID HYDROCARBONS

(57) Abstract: A filtration media comprises a nonwoven web of austenitic stainless steel fibers. The austenitic steel fibers have an equivalent diameter less than 25µm. The austenitic stainless steel fibers of the nonwoven web are metallurgically bonded to each other at contacting points between stainless steel fibers. The surface of the austenitic stainless steel fibers comprises Fe₂O₃ in a hematite crystallographic structure.

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FILTRATION MEDIA AND COALESCING FILTER TO REMOVE WATER FROM LIQUID HYDROCARBONS

Description

Technical Field

- 5 [1] The invention relates to the technical field of filtration media and to the field of coalescing filters that are used for the removal of water from liquid hydrocarbons.

Background Art

- 10 [2] US 6395184B1 discloses a method and apparatus for water removal from oil, oil products, gas condensate, and liquid hydrocarbons. The water removal is implemented via the selective filtration of hydrocarbons through a filtering element consisting of three tilted beds. The first and the third beds are made of a porous-cellular metal or alloy with hydrophobic surface, while the intermediate bed is made of hydrophilic materials such as porous-cellular metal or alloy with modified surface, celluloid foam or the cartridges filled with glass balls or glass fiber.

15 Disclosure of Invention

- [3] The objective of the invention is to provide an improved filter media for the removal of water from liquid hydrocarbons.
- 20 [4] The first aspect of the invention is a filtration media comprising - and preferably consisting out of - a nonwoven web of austenitic stainless steel fibers. The austenitic steel fibers have an equivalent diameter of less than 25 μm ; preferably of less than 20 μm , more preferably less than 10 μm ; even more preferably less than 5 μm ; even more preferably less than 4 μm . The austenitic stainless steel fibers of the nonwoven web are metallurgically bonded to each other at contacting points between stainless steel fibers. With metallurgically bonded nonwoven web of austenitic stainless steel fibers is meant that metallurgical bonds are formed between austenitic stainless steel fibers contacting each other in the nonwoven web. The metallurgical bonds can be sinter bonds or welded bonds, e.g. welded bonds established by means of Capacity Discharge Welding (CDW). The surface of the austenitic stainless steel fibers comprises Fe_2O_3 in a hematite crystallographic structure.
- 30 [5] The filtration media provides excellent results over a long lifetime, even in discontinuous operation, in coalescing applications for the removal of water from liquid hydrocarbons. With discontinuous operation is meant that the filtration media is not used during a certain time frame, after which its use is resumed. Although the functioning is not well understood, it is believed that the specific surface of the austenitic stainless steel fibers is such that for coalescing of water from liquid hydrocarbons, an improved balance of affinity of the surface for water and for liquid hydrocarbons is obtained.
- 35 [6] With equivalent diameter of a fiber is meant the diameter of the circle having the same surface area as the area of the cross section of the fiber; cross section which is not necessarily circular.

- [7] A surface of the austenitic stainless steel fibers comprising Fe_2O_3 in a hematite crystallographic structure can be obtained by a thermal treatment of the nonwoven web in air atmosphere at a temperature between 200°C and 400°C (more preferably between 200°C and 350°C), preferably after metallurgically bonding the stainless steel fibers to each other.
- [8] Using high resolution XPS (X-ray photoelectron spectroscopy) the different types of oxides of iron in the oxide layer of austenitic stainless steel can be quantitatively analysed over the thickness of the oxide layer; because the peak positions of iron depend on the ionic states of iron. To this end, appropriate curve fitting techniques are used; using specific curve fitting software as provided by suppliers of high resolution XPS equipment. In such analysis, care should be taken: as there could always be some surface contamination; the first measurement point should be disregarded.
- [9] In preferred embodiments, the hematite structure has an alpha-hematite structure.
- [10] In a preferred embodiment, the thickness of the oxide layer of the stainless steel fibers is less than 15 nm, preferably less than 12 nm, more preferably less than 10 nm. The thickness of the oxide layer is determined as the depth from the surface at which the amount of metallic iron is equal to the amount of iron in iron oxides as can be determined via XPS (X-ray photoelectron spectroscopy) measurement.
- [11] In preferred embodiments of the invention, the contact angle of water on the filtration media is less than 50°.
- [12] In a preferred filtration media, more than 40 atomic percent - and preferably more than 45 atomic percent; even more preferably more than 50 atomic percent - of the Fe in the oxide layer is present as Fe^{3+} .
- [13] In preferred embodiments of the invention, the surface of the austenitic stainless steel fibers does not comprise a polymeric coating. It also means that the surface of the austenitic stainless steel fibers does not comprise a fluorocarbon coating.
- [14] In embodiments of the invention, the austenitic stainless steel fibers are preferably made via bundled drawing. Bundled drawing is described e.g. in US-A-2050298, US-A-3277564 and in US-A-3394213. Austenitic stainless steel fibers produced via bundled drawing have a typical polygonal cross section allowing identification that the fibers have been produced by bundled drawing.
- [15] A preferred austenitic stainless steel alloy for use in the invention is alloy 316 according to ASTM A240 (and more specifically according to ASTM A240 / A240M-15a, Standard Specification for Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels and for General Applications, ASTM International, West Conshohocken, PA, 2015).
- [16] For embodiments of invention, the porosity of the nonwoven web of austenitic stainless steel fibers is preferably more the 80%; and preferably less than 90%. Such embodiments provide best results in coalescing applications.

[17] A second aspect of the invention is a coalescing filter for the separation of water out of liquid hydrocarbons. The coalescing filter comprises at least one filtration media as in any embodiment of the first aspect of the invention.

[18] Preferably, the coalescing filter comprises a plurality of filtration media as in any embodiment of the first aspect of the invention. More preferably, the coalescing filter comprises at least two filtration media as in any embodiment of the first aspect of the invention. The two filtration media are spaced in the coalescing filter by a drainage layer. The drainage layer is provided out of another material than austenitic stainless steel. As an example, the drainage layer can be provided out of organic fibers. As an example, the drainage layer can comprise or consist out of a polyester fiber nonwoven fabric.

[19] A preferred coalescing filter comprises a first filtration media as in the first aspect of the invention and a second filtration media as in the first aspect of the invention. The first filtration media comprises - and preferably consists out of - austenitic stainless steel fibers having an equivalent diameter less than 3 μm . The second filtration media comprises - and preferably consists out of - austenitic stainless steel fibers having an equivalent diameter less than 6 μm and wherein the equivalent diameter differs from the equivalent diameter of the austenitic stainless steel fibers of the first filtration media. Such coalescing filters provide improved efficiency of coalescence, especially in discontinuous operation of the coalescing filter.

[20] A preferred coalescing filter comprises a filtration layer of organic fibers - preferably a nonwoven web of organic fibers, e.g. polyester fibers - in fluid flow direction downstream from a filtration media according to the first aspect of the invention. Preferably, the filtration layer of organic fibers is in contact with the filtration media according to the first aspect of the invention. The filtration media out of austenitic stainless steel fibers is provided for coalescing water drops out of the liquid hydrocarbon, thereby increasing the size of the water drops. The filtration layer of organic fibers is provided to drain the water drops out of the coalescing filter.

[21] A preferred coalescing filter comprises a succession of pairs of layers. Each pair of layers comprises a filtration media as in the first aspect of the invention; and a filtration layer of organic fibers - preferably a nonwoven web of organic fibers, e.g. polyester fibers - in fluid flow direction downstream from the filtration media; and preferably in contact with the filtration media. The filtration media out of austenitic stainless steel fibers are provided for coalescing water drops out of the liquid hydrocarbon, thereby increasing the size of the water drops. The filtration layers of organic fibers are provided to drain the water drops out of the coalescing filter. More preferably, the filtration media of a first pair of layers comprises austenitic stainless steel fibers of lower equivalent diameter than the filtration media of a pair of layers in fluid flow direction downstream from the first pair of layers. Such arrangement has shown to provide improved efficiency of the coalescing filter.

[22] A preferred coalescing filter comprises a metal mesh, e.g. a woven wire mesh. The metal mesh can e.g. be provided at the fluid flow exit of the coalescing filter; or as layer inside the coalescing filter.

[23] In a preferred embodiment, the coalescing filter is a pleated filter.

5 [24] In a preferred embodiment, the coalescing filter is a pleated candle. Preferably, the coalescing filter is provided for fluid flow from the inside to the outside of the pleated candle.

[25] A third aspect of the invention is a method for removing water from liquid hydrocarbons. The method uses a coalescing filter as in any embodiment of the second aspect of the invention. Liquid hydrocarbon comprising water is flown through the coalescing filter, in order to coalesce and remove water.

Mode(s) for Carrying Out the Invention

[26] A first filtration media that has been made is a sintered nonwoven web consisting out of 2 μm equivalent diameter bundled drawn austenitic stainless steel fibers out of alloy 316. The first filtration media has a specific mass of 750 g/m^2 and 88% porosity. The sintered nonwoven web has been treated at 300°C . The surface morphology of the austenitic stainless steel fibers is not modified by the thermal treatment compared to the morphology after sintering the nonwoven web. The surface of the austenitic stainless steel fibers comprises Fe_2O_3 in a hematite crystallographic structure. The thickness of the oxide layer was 10 nm. The thickness of the oxide layer is determined as the depth from the surface at which the amount of metallic iron is equal to the amount of iron in iron oxides, as determined via XPS measurement.

Contact angle measurements have been performed using a dynamic contact angle goniometer and tensiometer DCAT 11/21. The contact angle to water on the first filtration media has been measured; the result was a contact angle below 40° . If the thermal treatment at 300°C was maintained longer, e.g. 10 minutes after gradual heating up to 300°C , the contact angle was 0° . The contact angle to water has also been measured for the sintered nonwoven web before performing the thermal treatment at 300°C , the contact angle in such condition was more than 90° .

30 The contact angle to diiodomethane on the first filtration media has been measured; the result was a contact angle of about 45° . If the thermal treatment at 300°C was maintained longer, e.g. 10 minutes after gradual heating up to 300°C , the contact angle was about 37° . The contact angle to diiodomethane has also been measured on the sintered nonwoven web before performing the thermal treatment at 300°C , the contact angle is such condition was about 50° .

[27] A second filtration media that has been made is a sintered nonwoven web consisting out of 4 μm equivalent diameter bundled drawn austenitic stainless steel fibers out of alloy 316. The first filtration media has a specific mass of 750 g/m^2 and 88% porosity. The sintered nonwoven web has been treated at 300°C . The surface morphology of the austenitic stainless steel fibers is not modified by the thermal treatment compared to the

morphology after sintering the nonwoven web. The surface of the austenitic stainless steel fibers comprises Fe_2O_3 in a hematite crystallographic structure.

[28] Without further treatment of the surface of neither the first filtration media nor the second filtration media; a coalescing filter has been made with the first filtration media and the second filtration media. The built up of the coalescing filter in the order as listed is:

- at the inflow side of the coalescing filter, a layer of the first filtration media (750 g/m^2).

This layer functions to increase the water drops of the water comprising liquid hydrocarbon from which the water needs to be eliminated through coalescing filtration;

- a filtration layer of 40 g/m^2 of a polyester fiber nonwoven. The polyester fibers are 4 denier fibers having a trilobal cross section. This filtration layer acts as drainage layer in the coalescing filter;

- a layer of the second filtration media (750 g/m^2). This layer functions to increase the water drops of the water comprising liquid hydrocarbon from which the water needs to be eliminated through coalescing filtration;

- a filtration layer of 40 g/m^2 of a polyester fiber nonwoven. The polyester fibers are 4 denier fibers having a trilobal cross section. This filtration layer acts as drainage layer in the coalescing filter; and

- a metal wire mesh.

[29] Using this layer construction pleated coalescing filter candles have been made with a pleat height 22 mm and with 15 pleats around the circumference of the pleated candle.

[30] The candles have been tested and have shown excellent performance and long performing lifetime for the removal of water from kerosene.

WHAT IS CLAIMED IS:

1. Filtration media comprising a nonwoven web of austenitic stainless steel fibers;
wherein the austenitic steel fibers have an equivalent diameter less than 25 μm ;
wherein the austenitic stainless steel fibers of the nonwoven web are metallurgically bonded to
each other at contacting points between stainless steel fibers;
and wherein the surface of the austenitic stainless steel fibers comprises Fe_2O_3 in a hematite
crystallographic structure.
2. Filtration media as in claim 1, wherein the thickness of the oxide layer of the stainless steel
fibers is less than 15 nm;
wherein the thickness of the oxide layer is determined as the depth from the surface at which
the amount of metallic iron is equal to the amount of iron in iron oxides as can be determined
via XPS measurement.
3. Filtration media as in claim 1, wherein the contact angle of water on the filtration media is less
than 50° .
4. Filtration media as in claim 2, wherein the contact angle of water on the filtration media is less
than 50° .
5. Filtration media as in claim 1, wherein more than 40 atomic percent of the Fe in the oxide layer
is present as Fe^{3+} .
6. Coalescing filter for the separation of water out of liquid hydrocarbons; comprising at least one
filtration media as in claim 1.
7. Coalescing filter as in claim 6; comprising
 - a first filtration media as in claim 1, wherein the austenitic stainless steel fibers have an
equivalent diameter less than 3 μm ;
 - a second filtration media as in claim 1; wherein the austenitic stainless steel fibers have an
equivalent diameter less than 6 μm ; and wherein the equivalent diameter differs from the
equivalent diameter of the austenitic stainless steel fibers of the first filtration media.
8. Coalescing filter as in claim 6, wherein the coalescing filter comprises a filtration layer of
organic fibers in fluid flow direction downstream from a filtration media as in claim 1.
9. Coalescing filter as in claim 6, wherein the coalescing filter comprises a succession of pairs of
layers, wherein each pair of layers comprises
 - a filtration media as in claim 1;
 - a filtration layer of organic fibers in fluid flow direction downstream from the filtration media.

10. Coalescing filter as in claim 9, wherein the filtration media of a first pair of layers comprises austenitic stainless steel fibers of lower equivalent diameter than the filtration media of a pair of layers in fluid flow direction downstream from the first pair of layers.

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11. Coalescing filter as in claim 6; wherein the coalescing filter comprises a metal mesh.

12. Coalescing filter as in claim 6, wherein the coalescing filter is a pleated filter.

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13. Coalescing filter as in claim 6, wherein the coalescing filter is a pleated candle.

14. Method for removing water from liquid hydrocarbons,
wherein liquid hydrocarbon comprising water is flown through a coalescing filter as in any of the claims 6 – 13 in order to coalesce and remove water.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2016/104918

A. CLASSIFICATION OF SUBJECT MATTER		
B01D 17/022(2006.01)i		
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)		
B01D		
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 practicable, search terms used)		
CPRSABS;CNABS;WPI;EPODOC;SIPOABS;CNKI:austenitic, stainless steel, fiber, sintered, welding, metallurgically bonded, Fe2O3, ferric oxide, hematite		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	CN 103977600 A (SUZHOU ERYUAN CENTURY NANO TECHNOLOGY CO) 13 August 2014 (2014-08-13) abstract, description, paragraphs[0019]-[0031]	1-14
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A	CN 105709459 A (UNIV CHONGQING TECHNOLOGY) 29 June 2016 (2016-06-29) the whole document	1-14
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☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
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Information on patent family members

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