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CONVENTION

AUSTRALIA

Patents Act 1990

NOTICE OF ENTITLEMENT

We, **ITALTRACO S.r.l.** of Via Terraggio, I-20123 Milano, Italy state the following in connection with Australian Application No. 40634/93:

1. We are the nominated person.
2. The nominated person is the assignee of the actual inventor.
3. The nominated person is the applicant of the basic application listed in the declaration under Article 8 of the PCT.
4. The basic application is the application first made in a Convention country in respect of the invention.

Dated: 20 October 1994

By **PHILLIPS ORMONDE & FITZPATRICK**
Patent Attorneys for the Applicant

By:

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To: The Commissioner of Patents

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- (56) Prior Art Documents
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- (57) Claim

1. Device to separate two components of a mixture of at least partially non-miscible liquids, of the type consisting of a hollow body have an inlet port to feed said mixture, a first outlet port and a second outlet port to respectively discharge the separate heavier and lighter components of said mixture, and a filter positioned between said outlet ports,

wherein said filter:

is thoroughly and permanently soaked - so that substantially no air is trapped therein - with one of the components of said mixture; and is positioned between said inlet port and the outlet port of the soaking component.



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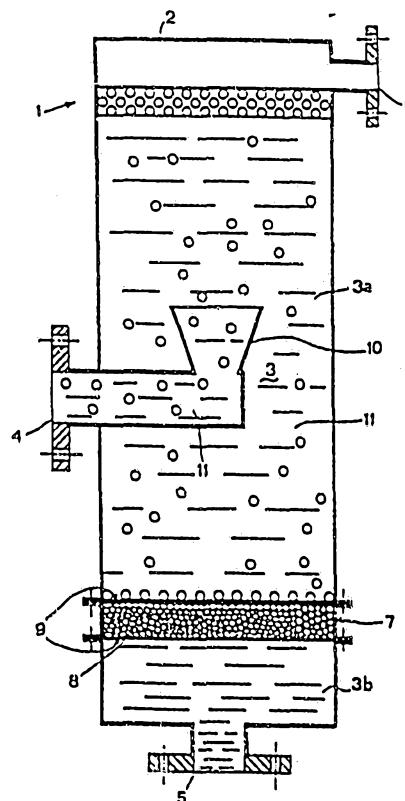
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673671

(54) Title: DEVICE AND METHOD TO SEPARATE THE COMPONENTS IN MIXTURES OF NON-MISCIBLE LIQUIDS

(57) Abstract

The present invention concerns a device (1) to separate two components of a mixture (11) of non-miscible liquids, especially water or salt water and oils. Said device (1) consists of a hollow body (2), having an inlet port (4) to feed the mixture (11) and outlet ports (5, 6) to discharge the separated components, and a filter (7) formed of a loosely packed hydrophilic material (8) in fibers, and positioned between said inlet port (4) and the outlet port (5) of the separated water. Said filter (7), when soaked with clean water, lets through only the water component of said mixture (11), while the oil component is repelled therefrom. The present invention also concerns a method to separate the components of a mixture of non-miscible liquids, making use of the device specified heretofore.



"DEVICE AND METHOD TO SEPARATE THE COMPONENTS IN MIXTURES OF NON-MISCIBLE LIQUIDS"

===oOo===

5 The problem of separating oily or hydrocarbon substances (hereinafter simply referred to as "oils") dispersed into aqueous solutions, namely of the total separation of the insoluble oily phase from the aqueous phase containing the dispersed fraction of the oily phase, has been examined by various experts, and different techniques have been proposed and used up to date.

10 In the separation process, the difficulty notably increases when wishing to reduce to a minimum the residual amount of oils in water, after separation. This difficulty determines also an increase in the operating costs.

15 The known techniques adopted so far include, for example, absorption on active substances (eg. active carbon), distillation, decantation through packed blades, ultrafiltration and centrifugation.

20 All the aforecited techniques show some inconveniences which greatly limit their feasibility, especially when operating on large quantities of water which must be "cleaned" from oil residues to the greatest possible extent: gravimetric separations, for instance, are limited by the availability of sufficient differences of density between the aqueous phase and the oily phase, especially when treating very fine emulsions: thermic separations involve considerable energy costs and are unpropos-
25 able for application on a vast scale, as in oil separation from surface waters; the techniques making use of membranes have the drawback of low productivity and suffer from fouling problems.

30 It is of common knowledge, now-a-days, that huge volumes of oil-polluted surface waters have to be treated so as to restore the proper environmental conditions, since the presence of oils is harmful for the environment, the water life and the general use of the water resources. There is hence at present an unsatisfied demand for an innovating technology, apt to be economically applied - in removing oils from water - on a vast scale.

An object of the present invention is to provide a device and method for separating two components of a mixture of at least partially non-miscible liquids which overcome or at least alleviate one or more disadvantages of the prior art.

According to the present invention, there is provided a device to separate
 5 two components of a mixture of at least partially non-miscible liquids, of the type consisting of a hollow body have an inlet port to feed said mixture, a first outlet port and a second outlet port to respectively discharge the separate heavier and lighter components of said mixture, and a filter positioned between said outlet ports,

10 wherein said filter:

is thoroughly and permanently soaked - so that substantially no air is trapped therein - with one of the components of said mixture; and is positioned between said inlet port and the outlet port of the soaking component.

Preferably, one of said liquids is water - or a liquid similar to water or apt to be mixed therewith - and at least another one is an oily liquid, and said filter is formed of hydrophilic material.

The present invention also provides a method to separate two components of a mixture of at least partially non-miscible liquids, carried out in a chamber having an inlet port to feed same mixture and outlet ports to discharge the separated components, wherein said method includes the following preliminary discontinuous step:

a) dipping a filter positioned between said inlet port and one of said outlet ports into one of the components of said mixture, up to fully expelling any air trapped in the filter, and thoroughly soaking its filtering bed;

25 and the subsequent continuous steps:

b) causing said mixture to pass through said previously soaked and deaerated filter under a predetermined hydrostatic head;

c) recovering one of the separated components, and precisely the one used in the soaking step a), from the outlet port facing the side of the filter opposite to that facing the inlet port;

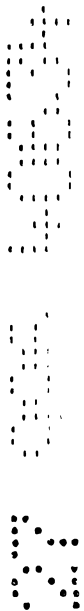
30



2a

- d) recovering the other separated component from the outlet port facing the same side of the filter as the inlet port.

Further characteristics and advantages of the present invention



will anyhow be more evident from the following detailed description, given with reference to the accompanying drawing, which is a diagrammatic cross-section view of a preferred embodiment of the device according to the invention.

5 As shown on the drawing, the device 1 consists of a static hollow body 2, which defines a chamber 3 and is provided with a side inlet port 4 to feed the mixture 11 to be separated, and with a lower port 5 and an upper lateral port 6 to respectively let out the heavier (water) and the lighter (oil) components of the mixture 11 which have been separated. To
10 improve the efficiency of the device 1 and make its construction more economic, the hollow body 2 is preferably of elongated cylindrical shape - as can be seen from the drawing - and has a circular or rectangular cross-section.

 The chamber 3 is divided into an upper chamber 3a and a lower chamber 3b by a filter 7, sealed to the walls of the hollow body 2 in a position set between the inlet port 4 and the lower outlet port 5. The filter 7 is fixed to the walls of the hollow body 2 in any suitable way, as for
15 example by flanges, so as to prevent any channelizing between the lateral surface of the filter 7 and the walls of the hollow body 2. The filter 7 is formed of a hydrophilic material 8, preferably consisting of short
20 fibres, housed between a pair of wire or plastic nets 9, or any other means allowing to keep in position the filtering material 8. but letting through the liquid flow. The meshes of the nets 9 should be sufficiently close not to let through the hydrophilic material 8, and sufficiently
25 wide to let through the water at the wanted flow rate. The mechanical structure of the filter 7 supporting the nets 9 is formed in known manner and must be apt to withstand the hydraulic stresses determined by the normal filtering flow, as well as by the reverse-flow of the water initially used to prepare and, thereafter, to possibly clean the filter 7.
30 without impairing or unduly packing the hydrophilic material 8 contained therein.

 It has been found by the inventor that said hydrophilic material 8

should preferably be in finely divided form, the single particles preferably having one of their dimensions larger than the other two. Said hydrophilic material 8 is stratified or otherwise casually positioned in the filter 7 and, when dipped into water, it is apt to form an almost
5 continuous system of micro-channels through which flows the water during use of said filter 7.

According to the invention, the hydrophilic material 8 apt to form the bed of said filter 7 preferably consists of short portions - hereinafter referred to as "fibrils" - of natural fibers (for instance cotton,
10 wool, flax, etc.), either on their own or mixed with other natural or synthetic fibers containing polar groups having hydrophilic properties. In particular, a material suited for the purpose could consist of spinning wastes or of the fiber "tuft" produced as waste or spinning dust in the spinning operations.

It is an important characteristic of the invention that the hydrophilic material 8 be made of fibrils of predetermined size. The size of the fibrils should not be too small, or it would cause the packing of the filter bed of hydrophilic material and thereby drastically reduce the specific water flow rate through the filter 7. On the other hand, it
15 should not be too large, as this could cause channelizing and thereby reduce the efficiency of the filter 7. The fibrils should thus preferably have a diameter between 10 and 3.000 micron and a length from 2 to 50 mm.
20

Since the material 8 has to be hydrophilic, the fibers of vegetable origin - as cotton, flax, hemp - and some fibers of animal origin - as
25 wool - are the most suited for the purpose. Among the synthetic fibers, use can be made of those deriving from resins which have a sufficient degree of polarity, as acrylic, polyamidic, cellulosic and polyurethanic resins.

The thickness of the layer of hydrophilic material 8 forming the filter 7 will be for example between about 1 cm and 10 cm (preferably 2-5
30 cm). For an efficient separation at high flow rates, the layer of fibrils should preferably remain very soft; in other words, the fibrils should be

loosely packed, to the extent that the single fibrils are simply kept in mutual contact without being unduly compressed. The proportion of voids in the packed filter 7 (intended as ratio between the volume of voids in the filter and the overall volume of the filter) is thus preferably in the range from 5 to 80%. To prevent an excessive packing of the filter bed, the fibrils of natural origin (eg. cotton) should preferably be mixed with acrylic or synthetic fibrils which, undergoing no mechanical changes with water, form the anti-packing agent for the natural fibrils. The added synthetic fibrils can also be in the form of "tufts". As an alternative to said mixture, the filter 7 can be formed by alternating layers of natural fibrils with layers of synthetic fibrils. The filter bed can be obtained also with the techniques of the non-woven cloths.

Other important advantages of the hydrophilic material 8 of the present invention are its chemical resistance in the different working environments and the possibility to be recycled with non-polluting methods, as through composting techniques.

The fibrils of hydrophilic material 8 can be used in their natural state, namely as they are produced as spinning wastes, but they can also be more conveniently pretreated, so as to supply a filter bed of higher quality and, above all, of further homogeneity.

A first pretreatment consists in a washing step with surface-active agents - so as to eliminate any fatty matters and dressings adhering to the fibrils - followed by a light oxidation with 5-10% hydrogen peroxide, at 30-40°C, which is meant to activate the surface of the fibrils, giving them higher hydrophilic properties. The "water carding" operation is subsequently carried out, to make the fibrils soft and smooth. There follows a drying operation, and then the mixing into a fast ploughshare mixer of the Lodige type, in order to obtain a homogeneous mixture between the various types of fibrils, according to the requirements of the invention.

According to the separation method of the present invention, the aforescribed device 1 is used as follows. To start with, clean water is introduced from the lower port 5, until the filter 7 is soaked with water

up to complete filling of each interspace between the fibrils of hydrophilic material 8 and up to forming a water layer above the filter 7 thus soaked. The dipping of the filter 7 into clean water continues until all the air present in the filter has been fully expelled and the single
5 fibrils have totally swollen.

This preliminary step of the separation method according to the invention is particularly important and has to be carried out correctly for the device 1 to work efficiently in separating the water/oil mixture 11. In fact, according to the inventor, the soaking water should substantially occupy three different positions - the water absorbed inside the
10 fibrils; the water adhering to the surface of the fibrils; the water forming a bridge between one fibril and the next - thereby creating a hydrophilic barrier.

According to a plausible explanation for the extremely efficient
15 performances of the device according to the present invention - explanation given by mere way of example and in no way to be considered as limiting the invention itself - the particular nature of the filter 7 and its thorough soaking before starting the separation, allow in fact to form over its surface a laminar layer of water molecules. strongly bound both
20 one to the other and to the hydrophilic material 8. Said laminar layer thus forms an impenetrable hydrophilic barrier for the oil drops contained in the mixture 11 to be separated. The water molecules can in fact flow through said barrier, while the oil drops are prevented from passing through.

One then proceeds to feed into the device 1, through the port 4,
25 the mixture 11 to be separated, for instance water (dashed lines in the drawing) and oil (small circles in the drawing). For this purpose, the port 4 extends into the hollow body 2 with a duct ending into a conical diffuser 10 facing upwards, so as to prevent any eddy flow from reaching
30 the surface of the filter 7, which could hamper the proper forming of the aforescribed laminar water layer. In fact, thanks to this configuration. the flow of mixture 11 to be treated in the part overlying the

filter 7 is a laminar flow with maximum speeds between a fraction and a few cm/sec, preferably between 0.1 and 10 cm/sec.

The mixture 11 thereby fills the chamber 3, the level of its surface being determined by the height of the free outlet port 6, thereby creating an hydrostatic head on the filter 7. Preferably, the purified liquid is discharged from the port 5 through a trap device (not shown) which easily guarantees a constant head on the filter 7, equal to the weight of the liquid column between the port 6 and the outlet of said trap device. The outlet of the trap device will preferably be positioned higher than the filter 7, so as to maintain the chamber 3b constantly filled with liquid.

Said hydrostatic head is by itself sufficient to allow the flow of liquid through the filter 7. When the single tiny emulsified oil drops reach the laminar water layer formed on the filter 7, they are stopped from passing through said filter, until - coalescing with other oil drops - they reach dimensions sufficient to create a buoyancy force higher than the entrainment force of the water flow, whereby the oil collects at the top of the hollow body 2 and is let out through the port 6. Suitable control devices of known type provide to keep the oil/mixture separation surface, in the chamber 3a, at a level below the height of the port 6, so as to prevent any mixture outlets from said port.

It is thus surprising to note that, in the device 1 of the present invention, the oil drops are stopped from penetrating into the filter 7, but remain over its surface. The filter 7 does not hence require to be regenerated, nor periodically replaced, unless - due to a wrong preparation of said filter, or to any other accidental causes - the laminar water layer formed on the surface of the filter 7 should break, thereby creating inside said filter channelizings of non-purified mixture. Said separation effect is schematically shown on the drawing by the oil drops (circles) collecting above the laminar water layer of the filter 7. In the meantime, the water of the mixture 11, separated from the oil, penetrates through the laminar water layer and then through the soaked filter

7, and finally flows out of the port 5.

As it can be understood from the above description, the present invention realizes a physical separation system, wherein the mixture 11 to be treated - comprising a continuous aqueous phase having an oily phase dispersed therein - is separated into the two water and oil components, the aqueous phase - practically free from oil - penetrating through the filtering unit of the system, while the dispersed oily phase gradually coalesces into a continuous oily phase, up to allowing its full recovery in an economic way.

The motive power required to separate the water and oil components can be limited to that generated by the free hydrostatic head of the mixture 11 to be treated. Said hydrostatic head on the filter bed can vary from 0.05 to 5 meters of water, with a flowing speed from 0.1 to 10 cm/sec. Otherwise, the flow can be obtained by pumping the mixture 11 to be filtered at the desired pressure.

In synthesis, the device and method according to the present invention allow water to be filtered from the oils thanks to a system which behaves like a very high flow-rate filter (up to $100 \text{ m}^3 / \text{m}^2 \text{ h.}$ with 0.1-0.2 bar of hydrostatic head on the filter), essentially based on a continuous water layer permanently formed above a support consisting of a layer of fully soaked hydrophilic material.

The operation to separate water from oil, through the barrier formed by said water layer supported by the layer of fibrils of hydrophilic material 8, takes place like a true and proper filtration.

Use can thus be made of any type of static or dynamic filtering devices available on the market, modified by replacing the conventional filtering cloths or panels with a layer of the hydrophilic material 8, previously soaked - until all air is expelled therefrom - according to the present invention.

There follow some practical examples of the method used to carry out the invention, which are by no means meant to limit the same.

EXAMPLE 1

Separation of fresh water from lubricating oil

A circular filtering panel, about 4 cm high, was introduced by flanging into a static separation device according to the present invention, having a cross-section of 0.125 m^2 and consisting of a cylinder with an inside diameter of 400 mm.

The filtering panel was protected and supported by two stainless steel nets with a 3 mm mesh. The hydrophilic material used for the filtering panel consisted of cotton fibrils, washed and pretreated as described heretofore. The separation device was fed with a mixture of 3% oil (FIAT VS oil) in water, up to forming a constant hydrostatic head on the filter bed of about 1 m of the mixture. The whole system was kept in circulation by recycling the filtered water, after having mixed the same with the separated oil.

The test was continued for 48 hours and gave, on an average, the following results:

- flow rate through the filter: $40 \text{ m}^3 / \text{m}^2 \text{ h}$;
- lubricating oil content in the filtered water: less than 3 mg/kg.

EXAMPLE 2

Separation of petroleum from sea water

A rectangular filtering panel, about 5 cm high, was introduced by flanging into a static separation device according to the present invention, having a cross-section of 0.5 m^2 .

The filtering panel was protected and supported by two stainless steel nets with a 3 mm mesh. The hydrophilic material consisted of a bottom layer of acrylic fibrils, to which were subsequently superposed layers of cotton fibrils up to reaching the desired height of 5 cm.

The separation device was fed with a mixture of 10% petroleum in sea water, up to forming a constant hydrostatic head on the filter bed of about 1.2 m of the mixture. The whole system was kept in circulation by recycling the filtered water, after having mixed the same with the separated petroleum.

The test was continued for a long while and gave, on an average, the following results:

- flow rate through the filter: $32 \text{ m}^3/\text{m}^2\text{h}$;
- content of CCl_4 extractable substances in the filtered sea water: below 5 mg/kg.

EXAMPLE 3

Separation of Diesel oil from water

A square filtering panel, about 3 cm high, was introduced by flanging into a static separation device according to the present invention, having a 20x20 cm cross-section.

The filtering panel was protected and supported by two stainless steel nets with a 3 mm mesh. The hydrophilic material consisted of a bottom layer of acrylic fibrils, to which were subsequently superposed layers of cotton fibrils up to reaching the desired height of 3 cm.

The separation device was fed with a mixture of 5% Diesel oil in water, up to forming a constant hydrostatic head on the filter bed of about 0.7 m of the mixture. The whole system was kept in circulation by recycling the filtered water, after having mixed the same with the separated Diesel oil.

The test was continued for a long while and gave, on an average, the following results:

- flow rate through the filter: $48 \text{ m}^3/\text{m}^2\text{h}$;
- no residual Diesel oil in the filtered water.

As can be understood from the above description, the invention allows to separate mixtures based on water and oil, with a very high selectivity and efficiency, in a definitely economic way, and by means of a very simple device.

For what concerns the mixtures to be treated, these can evidently be of various types, even containing many different components, provided that one of the components is a hydrophilic component and that at least another component cannot be mixed therewith.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. Device to separate two components of a mixture of at least partially non-miscible liquids, of the type consisting of a hollow body have an inlet port to feed
5 said mixture, a first outlet port and a second outlet port to respectively discharge the separate heavier and lighter components of said mixture, and a filter positioned between said outlet ports,

wherein said filter:

is thoroughly and permanently soaked - so that substantially no air is trapped
10 therein - with one of the components of said mixture; and is positioned between said inlet port and the outlet port of the soaking component.

2. Device as in claim 1), wherein one of said liquids is water - or a liquid similar to water or apt to be mixed therewith - and at least another one is an oily
15 liquid, wherein said filter is formed of an hydrophilic material, is soaked with clean water, and is positioned between said inlet port and said first outlet port.

3. Device as in claim 2), wherein said hydrophilic material is in the form of a particulate.
20

4. Device as in claim 3), wherein the single particles of said particulate have one of their dimensions larger than the other two.

5. Device as in claim 3), wherein the single particles of said particulate have
25 the largest dimension of their cross-section in the range from 10 to 3.000 μ m, and their length in the range from 2 to 50 mm.

6. Device as in any one of claims 1) to 5), wherein the percentage of voids in the filter is in the range from 5 to 80% in volume.

30



7. Device as in any one of claims 2) to 6), wherein said hydrophilic material is selected in a group formed by vegetal, natural or synthetic fibres, or mixtures thereof.

5 8. Device as in claim 7), wherein said hydrophilic material is formed of short portions of said fibres, having a length in the range from 2 to 50 mm.

9. Device as in claim 7), wherein said hydrophilic material is formed of spinning wastes or spinning dust.

10

10. Device as in claim 7), wherein said hydrophilic material is formed of non-woven cloths of said fibres.

15

11. Device as in claim 2), wherein the walls of said filter of hydrophilic material through which is apt to flow one of said components, are formed by nets.

12. Device as in claim 2), wherein said port extends into said hollow body with a duct ending into a conical diffuser facing the side opposite to that of the filter.

20

13. Device as in claim 2), wherein said port is connected to a trap device.

14. Device as in claim 13), wherein said trap device has its outlet at a level higher than the filter.

25

15. Device as in claim 2), wherein said port is a spillway outlet of said hollow body.

16. Device as in any one of claims 1) to 15), wherein said hollow body is formed by a cylinder having a circular or rectangular cross-section.

30

17. Method to separate two components of a mixture of at least partially non-miscible liquids, carried out in a chamber having an inlet port to feed same mixture and outlet ports to discharge the separated components, wherein said method includes the following preliminary discontinuous step:

- 5 a) dipping a filter positioned between said inlet port and one of said outlet ports into one of the components of said mixture, up to fully expelling any air trapped in the filter, and thoroughly soaking its filtering bed;

and the subsequent continuous steps:

- 10 b) causing said mixture to pass through said previously soaked and deaerated filter under a predetermined hydrostatic head;
- c) recovering one of the separated components, and precisely the one used in the soaking step a), from the outlet port facing the side of the filter opposite to that facing the inlet port;
- 15 d) recovering the other separated component from the outlet port facing the same side of the filter as the inlet port.

18. Method as in claim 17), wherein one of said liquids is water - or a liquid similar to water or apt to be mixed therewith - and at least another one is an oily liquid, and in that said filter is formed of hydrophilic material, is soaked with clean water, and is positioned between said inlet port and the outlet port through which is discharged the separated aqueous component.

20

19. Method as in claim 18), wherein said hydrophilic material is in the form of a particulate.

25

20. Method as in claim 19), wherein the single particles of said particulate have one of their dimensions larger than the other two.

21. Method as in claim 19), wherein the single particles of said particulate have the largest dimension of their cross-section in the range from 10 to 3.000 μ m, and their length in the range from 2 to 50 mm.

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22. Method as in any one of claims 17) to 21), wherein the percentage of voids in the filter is in the range from 5 to 80% in volume.

23. Method as in any one of claims 18) to 22), wherein said hydrophilic material is selected in a group formed by begetal or natural fibres, or synthetic fibres, or mixtures thereof.

24. Method as in claim 23), wherein said hydrophilic material is formed of short portions of said fibres, having a length in the range from 2 to 50 mm.

25. Method as in claim 23), wherein said hydrophilic material is formed of spinning wastes or spinning dust.

26. Method as in claim 23), wherein said hydrophilic material is formed of non-woven cloths of said fibres.

27. Method as in claim 17) or 18), wherein said hydrostatic head is in the range from 0.05 to 5 m of water.

28. Method as in claim 27), wherein said hydrostatic head is obtained by controlling the level of the mixture above said filter at a predetermined height.

29. Method as in claim 27), wherein said hydrostatic head is obtained by means of a pumping device.

30. Method as in claim 17) or 18), wherein the flow of said mixture on the filter is a laminar flow.

31. Method as in claim 17) or 18), wherein the flowing speed of said mixture on the filter is in the range from 0.001 to 0.1 m/s.

32. A device to separate two components of a mixture of at least partially non-miscible liquids, substantially as herein described with reference to the accompanying drawing.

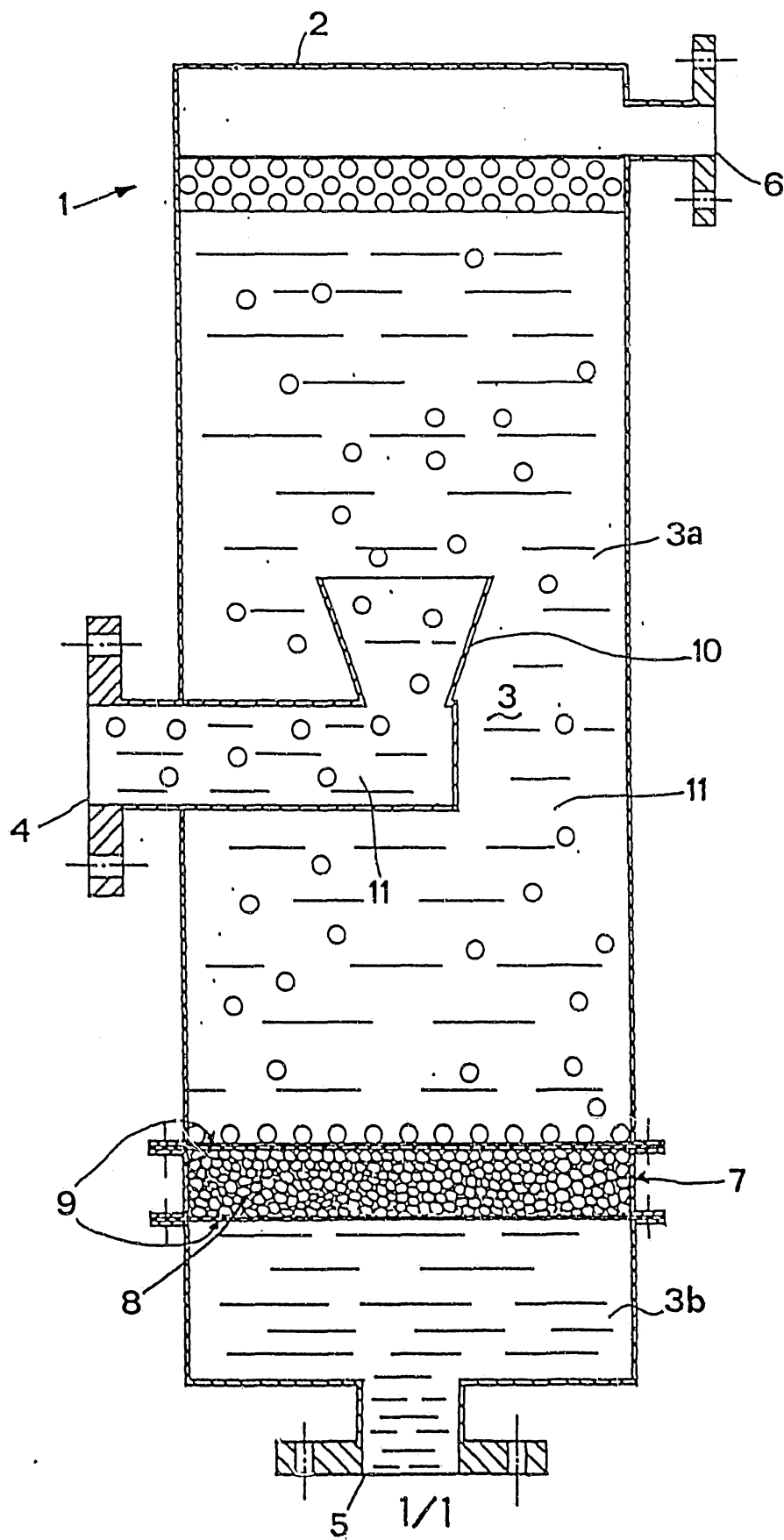
5 33. A method to separate two components of a mixture of at least partially non-miscible liquids, substantially as herein described with reference to the accompanying drawing.

34. A device to separate two components of a mixture of at least partially non-miscible liquids, substantially as herein described with reference to any one of the Examples.

35. A method to separate two components of a mixture of at least partially non-miscible liquids, substantially as herein described with reference to any one of the Examples.

20 DATED: 26 September, 1996
PHILLIPS ORMONDE & FITZPATRICK
Attorneys for:
ITALTRACO S.R.L.





INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 93/01053

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 B01D17/022; B01D39/04		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	B01D	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS C IDERED TO BE RELEVANT⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
Y	DE,A,3 644 665 (DIDIER-WERKE AG) 14 July 1988 see column 1, line 28 - line 40 see column 2, line 10 - line 40; claim 5 ---	1,4,7,8, 16-20, 23,27-31
Y	FR,A,895 586 (FAUDI) 29 January 1945 see page 2, line 89 - page 3, line 3; figure 1 ---	1,4,7,8, 16-20, 23,27-31
A	US,A,3 925 202 (GENE HIRS) 9 December 1975 see column 3, line 22 - line 30; figure see column 2, line 66 - column 3, line 10 --- -/--	5,6,21, 22
¹⁰ Special categories of cited documents : ¹⁰ "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document 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 "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		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
14 JULY 1993	0 2. 08. 93	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	MARZENKE J.	

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