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Description

Emulsions can be visualised in simple terms as one discontinuous internal phase or fluid enveloped in a second dissimilar continuous external phase or fluid. In general, emulsions fall into two broad categories, oil-in-water emulsions wherein the oil is the discontinuous internal phase and the water is the continuous external phase, or a water in oil emulsion, where the above rules are reversed. In addition there can be multiple emulsions such as water-oil-water emulsion wherein there is a discontinuous external oil phase suspended in a continuous water external phase; or an oil-water-oil multiple emulsion wherein the above roles are reversed, i.e. in all liquid membrane systems.

Emulsions, whether they are water-in-oil or oil-in-water are further characterised as being low ratio or high ratio. Low ratio emulsions are generally no higher than 4/1 internal phase to external phase whereas high ratio emulsions are normally greater than 4/1, preferably greater than 8/1 internal phase to external phase. Low ratio emulsions possess very small droplet sizes, usually of the order of 1 micron, while high ratio emulsions possess relatively larger particle sizes of the order of 20 micron or more.

To make the low ratio type emulsions, many kinds of emulsification devices are available commercially, such as Tekmar Super Dispax, colloid mill, ultrasonic vibrator, etc. These devices are, however, very expensive. To make the high ratio type emulsions, especially the very high ratio ones, such as 17/1 w/o emulsion, there is no simple, effective, and inexpensive device currently available. The inability of the currently available emulsification machines in making the latter type emulsions is largely because the machines are too powerful to produce and maintain large droplets. They are made basically to produce emulsions composed of very fine droplets.

Methods of preparing emulsions are disclosed *inter alia* in GB—A—684926 and Fr—A—1572021. However neither of these specifications disclose the specific packing materials used in the method and apparatus of this invention through which one obtains a simple, inexpensive, yet effective, method and apparatus for preparing emulsions (which term includes multiple emulsions).

According to the invention there is provided a method of preparing an emulsion by the emulsification of immiscible fluids, characterised in that

(a) the immiscible fluids are introduced into an enclosure through at least one entrance orifice;
(b) the fluids are thereafter flowed through the enclosure in a substantially axial direction, passing through a zone or bed packed with at least one material selected from steel metal sponge, metal shavings, ceramic chips, cannon packing, animal hair or plastic brush, metal tubes shorter than the internal diameter of the enclosure, and Berl saddle, to cause rapid and repeated mixing and remixing of the immiscible fluids in the enclosure so forming the desired emulsion; and
(c) the emulsion so formed is removed from the enclosure through one or more outlet orifices.

The immiscible fluids which are introduced into the packed enclosure through the entrance orifice or orifices may be fed into the enclosure by fluid feeding means selected from pumping means, gravity conduit means, syringe means and combinations thereof, in communication with fluid storage means such as tanks or reservoirs, etc. Preferably single or multiple pumps are used. The fluids fed into the packed enclosure may be introduced into the enclosure either through the same entrance orifice serviced by the fluid feeding means or each fluid through individual entrance orifices in close proximity one to another so as to ensure maximum intermixing of the different fluids.

Any number of packed enclosure emulsion generators can be used, with each generator mixing two or more fluids, or a single generator can be used with the fluids introduced either simultaneously through a single entrance orifice or with each fluid fed into the packed enclosure through individual entrance orifices situated on the apparatus, it being preferred that all fluids desired to be mixed are fed into the enclosure simultaneously. If necessary, however, the individual fluids can be fed into the enclosure sequentially. The packed enclosure can also be equipped with a return loop conduit whereby either all or part of the emulsion leaving the exit orifice is reintroduced into the entrance orifice for recirculation through the packed enclosure either alone or along with added component fluids. In this way a higher degree of emulsification can be obtained if desired. It is most preferred that separate packed enclosure emulsifiers be used to prepare individual emulsions when the final emulsion comprises a multiple emulsion, such as a water/oil/water system.

Further according to the invention, the emulsion is formed using an apparatus which as for example illustrated in the accompanying drawing, comprises an emulsion-forming enclosure (3) which has (a) at least one inlet orifice (1) for the introduction of immiscible fluids, (b) a zone or bed (4) in communication with the inlet orifice(s) and packed with at least one material selected from steel metal sponge, metal shavings, ceramic chips, cannon packing, animal hair or plastic brush, metal tubes shorter than the internal diameter of the enclosure and Berl saddle, and (c) at least one outlet orifice (2) in communication with the zone or bed (4) for removal of formed emulsion; said enclosure (3) preferably having a cross-sectional profile, when viewed at right angles to the flow-path therethrough, of a regular or irregular figure having at least three sides.

The apparatus comprises an enclosure, typically a pipe or column. This enclosure can be of any cross-sectional profile, i.e., any regular or irregular multi-sided configuration of n sides wherein n ranges from 3 to infinity (i.e. circular). The enclosure has orifices so as to permit the entrance of the fluids and the exit of said fluids. These orifices can be either the normal open ends of a piece of pipe or, if the enclosure has no "normally" open end the orifice can be specially constructed in the wall of the enclosure. What is necessary is that there be at least one entrance orifice and one exit orifice. Preferably these entrances and exit orifices are situated at the maximum possible distance away from each other along the axis of fluid flow in the enclosure so as to ensure maximum mixing between the fluids introduced into the enclosure. It is possible, and in some instances desirable, that there be multiple entrance orifices in which case each individual fluid can be introduced into the enclosure through its own entrance orifice. When multiple entrance orifices are employed they can be either serially located parallel to the fluid flow or radially in the enclosure wall in the perimeter of the enclosure defined by a plane passing perpendicular to the direction of flow in the enclosure.

The enclosure is packed with a material which causes the fluids introduced into the enclosure through the entrance orifice to split into many fine streams and to re-mix rapidly and repeatedly resulting in the formation of the desired emulsion. This material is packed into the enclosure in a random manner to as high a degree of density as is possible, short of plugging the enclosure, i.e. the fluid pressure drop between the entrance and exit may not equal zero. Suitable packing material includes steel metal sponge (such as Kurly Kate), metal shavings, ceramic chips, Berl Saddle (e.g. certain porcelain forms available from Fisher Scientific Company—their catalogue Stock No. 9—191—5), animal hair or plastic brush, metal tubes shorter than the internal diameter of the enclosure and mixtures of the above, preferably metal shavings, metal sponge (such as Kurly Kate) and "Cannon" packing. The phrase "Kurly Kate" is a registered Trade Mark at least in the United Kingdom. The proper choice of packing material is critical since it has been discovered that numerous seemingly attractive materials will not function to give emulsions. Some that will not work are perforated glass beads, metal Fenske rings, Raschig rings (glass), steel wool, wooden straw. The usual guidelines for selecting materials to construct emulsification machines may be followed, i.e. it is better to use the material which is wetted by the continuous phase rather than the discontinuous phase of the emulsion to be formed. However, this consideration may not be critical if the fluids are sent into the packed tube by way of a pump to give strong mixing in the tube or the surfactants used are potent ones to produce the desired type of emulsion.

The length of the enclosure from entrance orifices to exit orifices, the amount of packing, the density of the packing, and the type of material packed is left to the discretion of the practitioner, depending on the type of emulsion desired, the density of the fluids used and the final ratio of internal to external phase desired.

The component fluids fed into the packed enclosure are fed into the enclosure by fluid feed means. These fluid feed means are typically selected from pumps for each individual fluid or group of fluids or gravity feed tanks and conduits or syringes for each fluid or group of fluids or any combination of the above. The preferred fluid feed means comprises pumps for the component fluids.

When preparing multiple emulsions of the water-oil-water or oil-water-oil type it is possible to use one enclosure wherein two dissimilar components are added simultaneously to the enclosure through relatively closely situated orifice (or through the same orifice) while the third component is added further downstream. For example, a water and oil combination can be added to the enclosure in sufficient ratio to give a water in oil (w/o) emulsion. Further downstream a separate water stream can be introduced, in sufficient quantity to result in the w/o emulsion being suspended in a continuous water phase resulting in a water/oil/water (w/o/w) emulsion.

Alternatively separate packed enclosures can be used to prepare each emulsion, enclosure 1 preparing the w/o emulsion and enclosure 2, using the w/o emulsion from enclosure 1 as a feedstream, adding water to the emulsion to yield the w/o/w emulsion. Many variations in this basic theme can be envisaged and all are included in the scope of this invention.

The fluids typically used in preparing a water-oil-water emulsion include an internal water phase wherein is dissolved or suspended any desirable material such as medicinals, acids, bases, etc. The oil phase typically comprises an oil component, such as paraffin oil, mineral oil, petroleum distillate, etc. or animal or vegetable oils, depending upon the use to which the ultimate composition will be put. In addition, the oil phase may contain a surfactant, i.e. an oil soluble surfactant of HLB smaller than 8, and/or a strengthening agent. This surfactant and/or strengthening agent may be the same material. The final water component is the suspending phase and may comprise the aqueous phase upon which the basic water-in-oil emulsion is to act (i.e. detoxification, minerals recovery, etc.) or it may comprise a diluent phase permitting easy injection either into the body (if in medicinal use) or into a well (if in drilling use).

The uses to which emulsions and liquid membranes can be put and the materials used in preparing emulsions and liquid membranes are discussed in detail in U.S. 3,389,078, U.S. 3,454,489, U.S. 3,617,546, U.S. 3,637,488, U.S. 3,719,590, U.S. 3,733,776, U.S. 3,740,315, U.S. 3,740,329, U.S. 3,779,907, U.S. 3,897,308, U.S. 3,942,527, U.S. 3,959,173, U.S. 3,969,265, U.S. 4,014,785, RE 27,888 and Re 28,002.

The emulsion prepared by use of the present apparatus may have internal phase to external phase ratios ranging from 1:1 to 32:1, preferably 1:1 to 3:1 for the low ratio type emulsions and 10:1 or greater, more preferably 17.1 or greater for the high ratio type emulsions. These apply to both water-in-oil and oil-in-water type emulsions. The emulsions prepared by the use of the present apparatus may have droplet size from 0.1 micron to greater than 50 micron, preferably from about 0.5 micron to 5 micron for the low ratio type emulsions and 6 micron to 20 micron for the high ratio type emulsions.

Reproductibility of the packed tube device and the effect of the amount of packing materials

When metal sponge was used to pack the tube connected to a gear pump, the amount of the metal sponge used is important in determining the number of recycles needed to make a high ratio emulsion. Table I shows that when 9.5 g of the metal sponge were used, 3 cycles of the feed phase (oil and water) were required to make an emlusion of 18/1 ratio (94% internal phase), whereas only 2 cycles were required when 28.5 gm of the metal sponge were used and 1 cycle was needed to emulsify more than 90% of the feed when 57 g of the metal sponge were used. A cycle is defined as a once-through operation.

Table II shows the results of the duplicate runs. The drop sizes obtained are identical or close to those in Table I, indicating the excellent reproductibility of the packed tube device. In addition to drop size, flow rate (ccm/min.), pressure drop across the tube, and viscosities at various shear rates were measured and summarized in Tables II and III.

When the surfactant was changed from ENJ—3029 to ECA—4360, ECA is a registered Trade Mark in the United Kingdom, the emulsions made were quite similar in terms of drop size, time needed for complete emulsification, and viscosities at various shear rates (Table IV). Since these two polyamine surfactants are very close in chemical structure, these data further illustrate the reproducibility of the device's performance.

Packed tube vs. Kenics and pump

Although the packed tube, like Kenics mixer, is a type of static or motionless mixer, it is much more effective in making high ratio emulsions than Kenics because of the structure difference between the two devices. As discussed previously, the packed tube is much more densely packed in a random manner as compared to Kenics (Kenics is a registered Trade Mark in the United Kingdom).

As shown in Table V, while it took 2 cycles to make a 17/1 w/o emulsion with a 1 or 2 metal sponge-packed tube, it took as many as 18 cycles to produce a similar emulsion with Kenics and 22 cycles with a gear pump alone (without connecting to the packed tube). The centrifugal pump tested simply could not produce such desired high ratio emulsion (Table VI).

It is interesting to note that the centrifugal pump was able to make the relatively low ratio emulsions in the class of the high ratio emulsions, such as 4/1 or 5/1, by first making a 2/1 ratio emulsion and then gradually increasing the ratio to 3/1, 4/1 and 5/1 with slow addition of the internal phase during the recirculation of the feed phase through the centrifugal pump. The ratio of 5/1 was the highest that could be achieved. When the not-completely-emulsified 6/1 ratio emulsion was recycled many times through the pump, a large portion of the emulsion was broken and the remaining emulsion had a ratio of roughly 2/1. The standard lab emulsification equipment used in the liquid membrane project—fluted beaker with marine propeller type stirrer was proved incapable of making high ratio emulsions.

Packing materials

Besides metal sponge, nylon brush, animal hair brush and "cannon" type packing were found to be equally effective packing materials for making emulsions. The emulsions of 10/1 and 20/1 w/o ratios made with a tube packed with Nylon brush were quite similar to those made with metal sponge-packed tube as demonstrated by the viscosity vs. shear rate data (Table VII). The packed tube of 25.4 mm (1 inch) in diameter and 127 mm (5 inch) in length was attached to the discharge end of a 100—400 RPM gear pump. When the pump was used alone, it took 10 times longer than the packed tube in making the 10/1 w/o emulsion. It was totally unsuccessful in making 20/1 ratio emulsion even in a prolonged 1 hr. operation, whereas using a tube packed with either metal sponge or Nylon brush or animal hair brush made the 20/1 ratio emulsion in several minutes (Table VII).

"Cannon" packing is a small, half-cylindrical shape material. It is also very effective in forming high ratio emulsions, such as 17/1 w/o emulsion.

Using Berl Saddle, an emulsion of 20/1 ratio was made: whereas using stainless steel sponge, "Cannon" packing, and Nylon brush and bristle brush, emulsions of 33/1 ratio were successfully made.

Using the same experimental set-up and procedure, it was found that the metal Fenske rings with 152.4 mm (6 inch) diameter, steel wool packing, wooden straw packing, and perforated glass beads, and Raschig rings did not work, i.e., they did not produce any emulsion with high internal to external phase ratio.

Use of a packed tube to make low ratio emulsions

The packed tube is also effective in making low ratio emulsions with uniform droplet size. As shown in Table VIII, when a tube which was packed with 2 metal sponges and connected to a centrifugal pump was used, drop size distribution of 2 to 3 micron was observed after 2 cycles and 1—2 micron after 3 cycles. When 3 metal sponges were used, 1—2 micron drop size distribution was obtained in 1 cycle. In contrast, 4—14 micron drop size distribution was produced when a centrifugal pump was used alone. (Table VIII). Similar wide drop size distribution was obtained with the lab standard set-up of fluted beaker and marine propeller type stirrer.

Making oil-in-water emulsions

The following example shows that a metal sponge-packed tube is also effective in making oil-in-water emulsions.

The membrane phase was an aqueous solution of 1% Saponin, 70% glycerol and 29% water. The phase to be encapsulated was a mixture of toluene and heptane at a wt. ratio of 1/1. The wt. ratio of the encapsulated phase to the membrane phase was 4/1. Both of these phases blended at 4/1 ratio were sent to the packed tube via a gear pump. Specification of the pump is given in Table I.

A very stable emulsion of the o/w type was made by the pump-packed tube combination. Drop size range of the emulsion was from 4 to 12 micron with an average drop size of 8 micron.

TABLE I

Effects of recycling and amount of packing material on emulsification

Membrane Phase (M)=8% ENJ—3029, 7% S100N, 85% Diesel Fuel
Internal Phase (IP)=2% KCl
M/IP Wt. Ratio=1/17.6
Gear Pump used to connect with the packed tube:

Gearchem Model No. G 6ACT2KT Made by ECO Pump Corp. Capacity 1200 RPM driven by air; 5.3 GPM at 10 psig.

Packing Material=Metal sponge (M.S.), "Kurly Kate", No. 207, made by Kurly Kate Corporation, Chicago
t=25°C

Wt. of Packing (g)	9.5 (1/3 of 1 M.S.)			28.5 (1 M.S.)		57 (2 M.S.)	
No. of Cycle	1	2	3	1	2	1	2
% Emulsification	70	90	100	80	100	90—95	100
Drop Size (micron)	—	10,14,24	8,10,20	—	10,12,20	—	8,14,18

TABLE II

Pressure drop, flowrate, and drop size studies

M, IP and M/IP=Same as in Table I
Packed Tube connected to ECO gear pump. ("ECO" is a registered Trade Mark at least in the United Kingdom)

(Ia) 1 Metal Sponge (M.S.), wt.=28.5 gm, packing length (p.l.)=12.5 cm, packing diam. (p.d.)=2.54 cm, packing volume (p.v.)=63.3 cm.

Cycle	p (psv)	(kg/cm^2) (N/m^{3}x10^{-5})	Flowrate (ml/min)	Drop Size (μ) (smallest, avg., largest)
1st	5.8	0.4	24.00	40, 80, 120
2nd	2.9—4.4	0.2—0.3	200	10, 12, 20
3rd	5.8	0.4	17	8, 10, 18

(Ib) 1 M.S., wt.=28.5 gm, p.l.=45 cm, p.d.=1.6 cm, p.v.=90.5 cm^3

Cycle	p (psv)	(kg/cm^2) (N/m^{3}x10^{-5})	Flowrate (ml/min)	Drop Size (μ) (smallest, avg., largest)
1st	5.8—7.3	0.4—0.5	183.3	8, 18, 22
2nd			81	6, 12, 12

(II) 2 M.S., wt.=63 gm, p.l.=28 cm, p.d.=2.54 cm, p.v.=141.6 cm^3

Cycle	p (psv)	(kg/cm^2) (N/m^{3}x10^{-5})	Flowrate (ml/min)	Drop Size (μ) (smallest, avg., largest)
1st	9.4—10.2	0.66—0.7	1320	14, 40, 52
	5.8	0.4	75	8, 12, 18

TABLE III
Viscosity of emulsions vs. shear rate

Shear rate (sec^{-1})	Viscosity (cp) Emulsion Ia	Emulsion Ib	Emulsion II
5.1	6300	5000	4800
10.2	3000	3750	3150
170.0	450	540	435
240	300	345	278
510	20	>300	200
1020	10	>300	>150
5.1	7500	7200	8000
10.2	4250	5000	5500

TABLE IV
Emulsification with different membrane formulations

M_1=8% ENJ 3029, 92% Diesel Oil (D.O.)
M_2=8% ECA 4360, 92% D.O.
IP=2% KCl sol'n
M/IP=1/20
Packed Tube=1 metal sponge
t=25°C.

	Emulsion No. 1 (Using M_1)		Emulsion No. 2 (Using M_2)
Drop Size	10—20 micron		10—30 micron
Emulsification Time (Min.)	3		3
Viscosity	rpm	cp	cp
	3	3700	2400
	6	2800	2100
	100	405	330
	200	270	225
	300	200	190
	600	>150	150
	3	5500	4500
	6	4000	3250

TABLE V
Emulsification by Kenics and Gear Pump

M=8% ENJ 3029, 7% S100N, 85% D.O.
IP=2% KCl sol'n
M/IP=1/16.7
Gear Pump=see Table I

(I) Kenics (50.8 mm diam. 6 stages) and gear pump

No. of Cycles	% Emulsification	Drop size (micron)
16th	80	6—20
17	98	
18	100	6—10

(II) Gear Pump

No. of Cycles	% Emulsification	Drop size (micron)
20th	95	
22nd	100	6—20

TABLE VI
Emulsification by centrifugal pump alone

M=10% ENJ 2039, 90% Diesel Oil
IP=2% KCl
Centrifugal pump=Century, 3/4 HP, 3450 RPM

(I) M/IP=1/4 (M and IP were mixed at this ratio and fed into the pump).

No. of Cycles	Unemulsified IP (2%)
1	63
2	45
3	50
4	40
5	48
10	65

the above data indicate that the emulsion made had a M/IP ratio 2 1/2.

TABLE IV (Cont'd)

(II) MIP=1/2→1/3→1/4→1/5→1/6 (M and IP were mixed at the 1/2 ratio and fed into the pump. When emulsion was formed, additional IP was added to change the ratio to 1/3, 1/4, etc.)

M/IP	No. of cycles	Unemulsified IP	Diam. of Emulsion drop (micron)
1/2	1	10	
	2	0	0.5—2
1/3	1	0	1—2
1/4	1	0	—
1/5	1	0	1—12
1/6	1	100 (additional IP was not emulsified)	

When the existing emulsion was recycled many times, almost half of the emulsion was broken, the emulsion left had a M/IP ratio 1/2.

TABLE VII

M=8% ENJ 3029, 7% S100N 85% Diesel Oil
IP—2% KCl Sol'n
(I) M/IP—1/10
1) Gear Pump and Tube packed with Nylon needles (Brush)

Time needed to make emulsion (min)	Drop size (micron)	Shear rate (sec.$^{-1}$)	Viscosity (cp)
3	8—12	5	2800
		10	1600
		170	420
		340	270
		510	225
		1020	150
		5	3900

2) Gear Pump and tube packed with metal sponge

Time needed to make emulsion (min)	Drop size (micron)	Shear rate (sec.$^{-1}$)	Viscosity (cp)
3—4	8—12	5	2800
		10	1600
		170	420
		340	270
		510	220
		1020	145
		5	4500
		10	2750

3) Gear Pump

30	10—20	5	1500

(II) M/IP=1/20

1) Gear Pump and tube packed with nylon needles

7	8—12	5	700
		10	4200
		170	510
		340	270
		510	190
		1020	145
		5	10000
		10	6500

2) Gear Pump and tube packed with metal sponge

Time needed to make emulsion (min)	Drop size micron	Shear rate (sec^{-1})	Viscosity (cp)	t°C (°F)	cp at 5 sec^{-1}
3	8—22	5	3300	26.7 (80)	6500
		10	2350	30.0 (86)	5000
		170	360	39.0 (102)	4300
		340	233	45.0 (114)	4000
		510	220	58.0 (138)	3500
		1020	>150	69.0 (154)	2800
		5	6000	74.0 (164)	2500
		10	4250	82.0 (180)	2800
				88.0 (190)	4800
				99.0 (196)	4900

3) Gear Pump

Time needed to make emulsion (min)	Drop size (μ)	Shear rate ($sec.^{-1}$)	Viscosity (cp)
60	no emulsion	—	—

Notes:

(1) Animal hair brush and "Cannon" packing were also found to be effective in making high ratio emulsions. "Cannon" packing is half-cylindrical shell with 4 mm height, 3.2 mm diam. and 0.5 mm diam. holes on shell.

(2) The standard lab equipment, fluted beaker with marine propeller-type stirrer, was ineffective in making high ratio emulsions.

TABLE VIII

Using packed tube to make low ratio of W/O emulsions

M=1% ENJ—3029, 5% Lix 64 N, 11% S 100N, 83% Isopar M

("Isopar" is a registered Trade Mark at least in the United Kingdom)

Internal Reagent for Cu Extraction, IR=14% H_2SO_4, 13% $CuSO_4 \cdot 5H_2O$, 73% H_2O

M/IR wt. Ratio=1/1

The packed tube was connected to the Century centrifugal pump (3/4 H.P.)

(I) Packed tube=2.54 cm diam., 14 cm length Packing Materials-a=Metal sponge b="Cannon" packing (half cylindrical shells with 4 mm height, 3.2 mm diam. 0.5 mm diam. holes on shell)

No. of cycles	Δp a (psv)	Δp a kg/cm²	Δp b (psi)	Δp b kg/cm²	Drop size (μ) a	Drop size (μ) b
1	1.5	0.1	1.5	0.1	2—5	2—5
	2.9	0.2	2.9	0.2	2—3	2—3
	2.9—4.4	0.2—0.3	2.9	0.2	1—2	1—2
	2.9—4.4	0.2—0.3	2.9—4.4	0.2—0.3	1—2	1—2

(II) Packed tube=2.54 cm diam., 28 cm length, wt.=63 gm (2 m.s.)

Cycle	Δp (psi)	Velocity (cc/min)	Drop size (micron)
1	0.194 bar (2.9)	1200	2—5
2	0.194 (2.9—4.4)	—	2—3
3	0.194—0,33 (2.9—4.4)	784	1—2
4	0.194—0,33 (2.9—4.4)	775	1—2
5	0,33 (4.4)	—	1—2

Note: p=0,1 bar (1.5 psi) when pure water was recirculated.

(III) Packed tube=3 metal sponges with a total weight of 85.5 g.

Method of making emulsion (no recycle)	Drop size (micron)
(1) By centrifugal pump along	4—14
(2) By centrifugal pump and packed tube	1—2

Claims

1. A method of preparing an emulsion by the emulsification of immiscible fluids, wherein

(a) the immiscible fluids are introduced into an enclosure through at least one entrance orifice;
(b) the fluids are thereafter flowed through the enclosure in a substantially axial direction, passing through a zone or bed packed with at least one material so as to cause rapid and repeated mixing and remixing of the immiscible fluids in the enclosure so forming the desired emulsion; and
(c) the emulsion so formed is removed from the enclosure through one or more outlet orifices;

characterised in that the material is steel metal sponge, metal shavings, ceramic chips, cannon packing, animal hair or plastic brush, metal tubes shorter than the internal diameter of the enclosure or Berl saddle.

2. A method as claimed in claim 1, wherein the immiscible fluids are introduced into the enclosure through the entrance orifice(s) by fluid feeding means, preferably pumping means, gravity conduit means, syringe means or combinations thereof, in communication with fluid storage means.

3. A method as claimed in either of claims 1 and 2, wherein all or part of the emulsion removed from the exit orifice(s) is recirculated through the enclosure.

4. A method as claimed in any preceding claim, wherein the emulsion formed is of the water-in-oil type or is of the oil-in-water type.

5. A method as claimed in any preceding claim, wherein the emulsion produced has an internal phase to external phase ratio of from 1:1 to 32:1.

6. A method as claimed in any preceding claim, wherein the emulsion produced has a droplet size of from 0.1 micron to at least 50 micron.

7. A method as claimed in claim 6, wherein the emulsion produced has an internal phase to external phase ratio of from 1:1 to 3:1, and a droplet size of preferably 0.5 to 5 micron, or has an internal phase to external phase ratio of at least 10:1 and a droplet size of preferably from 6 to 20 micron.

8. A method as claimed in claim 5, wherein the emulsion produced has an internal phase to external phase ratio of at least 17:1.

9. A method as claimed in either of claims 1 and 2, wherein the emulsion discharged from the exit orifice(s) is fed to the entrance orifice(s) of a second packed enclosure to which is fed a third immiscible fluid, where a multiple phase emulsion is formed and is then collected at the outlet orifice(s) of the second packed enclosure.

10. A method as claimed in claim 9, wherein the multiple phase emulsion formed is of the oil-in-water-in-oil type or is of the water-in-oil-in-water type.

11. A method as claimed in any one of the preceding claims wherein the emulsion is formed by using an apparatus which comprises an emulsion-forming enclosure (3) which has (a) at least one inlet orifice (1) for the introduction of immiscible fluids, (b) a zone or bed (4) in communication with the inlet orifice(s) and (c) at least one outlet orifice (2) in communication with the zone or bed (4) for removal of formed emulsion; said enclosure (3) preferably having a cross-sectional profile, when viewed at right angles to the flow-path therethrough, of a regular or irregular figure having at least three sides, in which method the zone or bed (4) is packed with at least one material as defined in claim 1.

Patentansprüche

1. Verfahren zur Herstellung einer Emulsion durch Emulgierung von nichtmischbaren Flüssigkeiten, bei dem

(a) die nichtmischbaren Flüssigkeiten durch mindestens eine Eintrittsöffnung in einen Behälter eingeführt werden,
(b) die Flüssigkeiten anschließend in im wesentlichen axialer Richtung durch den Behälter strömen, eine Zone oder ein Bett passieren, das mit mindestens einem Material gefüllt ist, um ein schnelles und wiederholtes Mischen und Wiedervermischen der nichtmischbaren Flüssigkeiten in dem Behälter und somit die Bildung der gewünschten Emulsion zu bewirken, und
(c) die so gebildete Emulsion aus dem Behälter durch eine oder mehrere Austrittsöffnungen entfernt wird, dadurch gekennzeichnet, daß das Material aus Stahlschwamm, Metallspänen, keramischen Splittern, "Cannon"-Material, Tierhaar oder Kunststoffborsten, Metallröhrchen, die kürzer sind als der Innendurchmesser des Behälters, oder Berl-Sätteln besteht.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die nichtmischbaren Flüssigkeiten durch die Eintrittsöffnungen(en) mittels Flüssigkeitseinspeisvorrichtungen, vorzugsweise Pumpen, Fallleitungen Spritzen oder Kombinationen derselben, eingeführt werden, die in Verbindung mit Flüssigkeitslagervorrichtungen stehen.
3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß die gesamte aus der (den) Austrittsöffnung(en) austretende Emulsion oder ein Teil davon nochmals durch den Behälter geleitet wird.
4. Verfahren nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, daß die gebildete Emulsion vom Wasser-in-Öl-Typ oder vom Öl-in-Wasser-Typ ist.
5. Verfahren nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, daß die hergestellte Emulsion ein Verhältnis von interner Phase zu externer Phase von 1:1 bis 32:1 besitzt.
6. Verfahren nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, daß die hergestellete Emulsiion eine Tröpfchengröße von 0,1 μ bis mindestens 50 μ besitzt.
7. Verfahren nach Anspruch 6, dadurch gekennzeichnet, daß die hergestellte Emulsion ein Verhältnis von interner zu externer Phase von 1:1 bis 3:1, eine Tröpfchengröße von vorzugsweise 0,5 μ bis 5 μ oder ein Verhältnis von interner zu externer Phase von mindestens 10:1 und eine Tröpfchengröße von vorzugsweise 6 μ bis 20 μ besitzt.
8. Verfahren nach Anspruch 5, dadurch gekennzeichnet, daß die hergestellte Emulsion ein Verhältnis von interner zu externer Phase von mindestens 17:1 besitzt.
9. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß die aus der (den) Austrittsöffnung(en) austretende Emulsion in die Eintrittsöffnung(en) eines zweiten gefüllten Behälters eingespeist wird, in den eine dritte nicht mischbare Flüssigkeit eingespeist wird, so daß eine Mehrphasenemulsion erzeugt und dann an der (den) Austrittsöffnung(en) des zweiten gefüllten Behälters gesammelt wird.
10. Verfahren nach Anspruch 9, dadurch gekennzeichnet, daß die gebildete Mehrphasenemulsion vom Öl-in-Wasser-in-Öl-Typ oder vom Wasser-in-Öl-in-Wasser-Typ ist.
11. Verfahren nach einem der vorangehenden Ansprüche, dadurch gekennzeichnet, daß die Emulsion unter verwendung einer Vorrichtung hergestellt wird, die einen emulsionsbildenden Behälter (3) aufweist, der (a) mindestens eine Eintrittsöffnung (1) für die Einführung der nicht mischbaren Flüssigkeiten, (b) eine Zone oder ein Bett (4) in Verbindung mit der (den) Eintrittsöffnung(en) und (c) mindestens eine Austrittsöffnung (2) in Verbindung mit der Zone oder dem Bett (4) zur Entfernung der gebildeten Emulsion besitzt, wobei der Behälter (3) bei Blockrichtung rechtwinklich zum Strömungsweg vorzugsweise ein Querschnittsprofil von regulärer oder irregulärer Gestalt mit mindestens drei Seiten besitzt und wobei die Zone oder das Bett (4) mit mindestens einem Material gemäß Anspruch 1 gefüllt ist.

Revendications

1. Procédé de préparation d'une émulsion par émulsification de fluides non miscibles dans lequel:

(a) les fluides non miscibles sont introduits dans une enceinte par l'intermédiaire d'au moins un orifice d'entrée;
(b) les fluides sont ensuite amenés à s'écouler à travers l'enceinte dans une direction sensiblements axiale, à traverser une zone ou lit garni d'au moins une matière pour provoquer un mélange et remélange rapide et répété des fluides non miscibles dans l'enceinte en formant ainsi l'émulsion désirée; et
(c) l'émulsion ainsi formée est retirée de l'enceinte par l'intermédiaire d'un ou de plusieurs orifices de sortie, caractérisée en ce que la matière est une éponge métal acier, des rognures de métaux, des copeaux de céramique, un garnissage "Cannon", des poils d'animaux ou une brosse en plastique, des tubes métalliques plus courts que le diamètre interne de l'enceinte ou "Berl Saddle".

2. Procédé selon la revendication 1, caractérisé en ce que les fluides non miscibles sont introduits dans l'enceinte par l'intermédiaire du ou des orifices d'entrée par un moyen d'amenée de fluide, de préférence, un moyen de pompage, un moyen à conduit à gravité, une seringue ou une combinaison de ceux-ci, en communication avec un moyen de stockage de fluide.

3. Procédé selon l'une des revendications 1 ou 2, caractérisé en ce que toute l'émulsion ou une partie de l'émulsion retirée de l'orifice ou des orifices de sortie est recyclée à travers l'enceinte.

4. Procédé selon l'une quelconque des revendications 1 à 3, caractérisé en ce que l'émulsion formée est du type eau dans huile ou est du type huile dans eau.

5. Procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce que l'émulsion produite a un rapport de phase interne à la phase externe d'environ 1:1 à 32:1.

6. Procédé selon l'une quelconque des revendications 1 à 5, caractérisé en ce que l'émulsion produite a une dimension des gouttelettes d'environ 0,1 μ à au moins 50 μ.

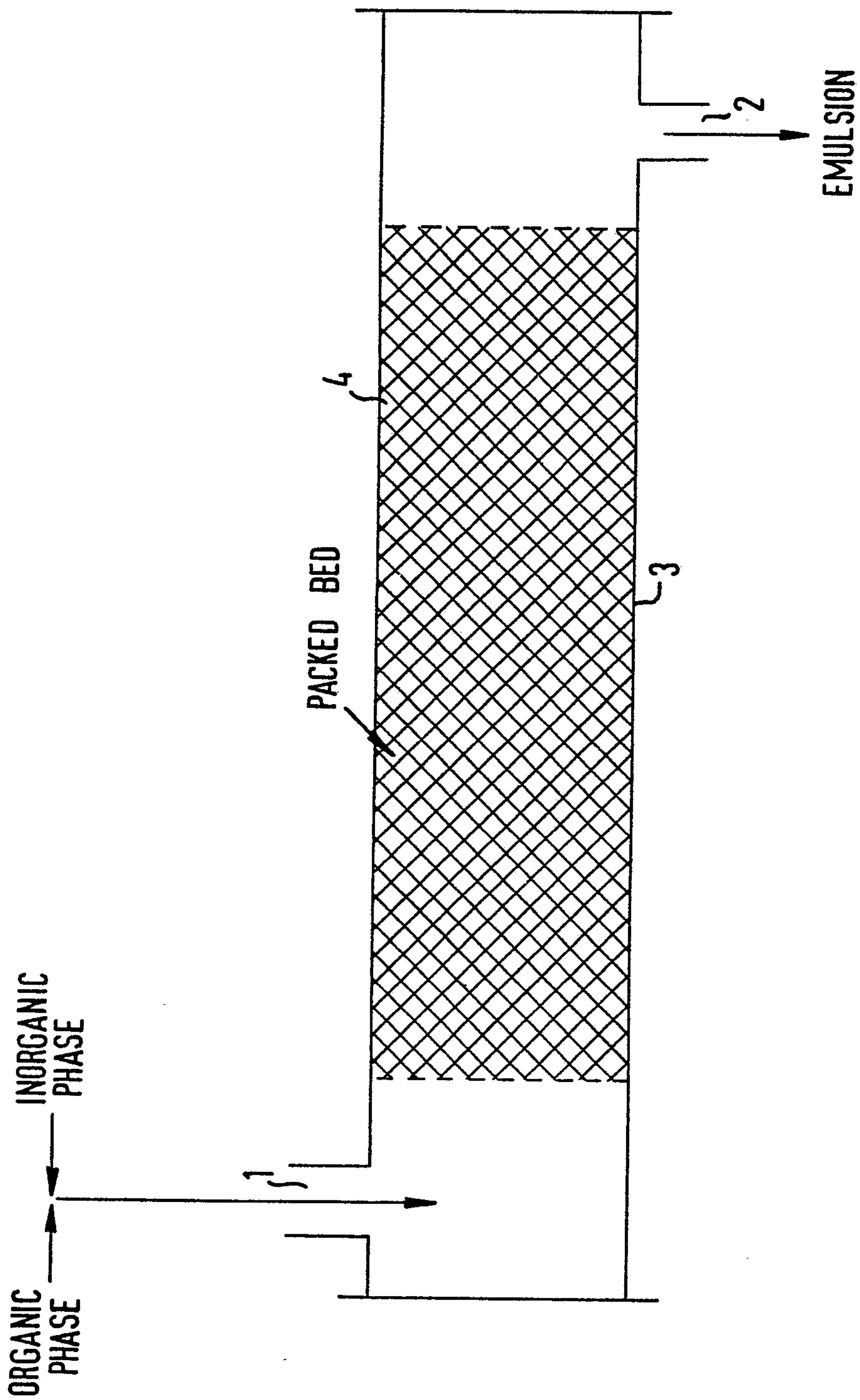
7. Procédé selon la revendication 6, caractérisé en ce que l'émulsion produite a un rapport de phase interne à la phase externe d'environ 1:1 à 3:1 et une dimension des gouttelettes, de préférence, de 0,5 μ à 5 μ en un rapport de phase interne à la phase externe d'au moins 10:1 et une dimension des gouttelettes de préférence de 6 μ à 20μ.

8. Procédé selon la revendication 5, caractérisé en ce que l'émulsion produite a un rapport de phase interne à la phase externe d'au moins 17:1.

9. Procédé selon l'une des revendications 1 ou 2, caractérisé en ce que l'émulsion déchargée du ou des orifices de sortie est amenée à l'orifice ou aux orifices d'entrée d'une seconde enceinte garnie dans laquelle est amené un troisième fluide non miscible où une émulsion à phases multiples est formée et est ensuite recueillie à l'orifice ou aux orifices de sortie de la seconde enceinte garnie.

10. Procédé selon la revendication 9, caractérisé en ce que l'émulsion formée à phases multiples est du type huile dans eau dans huile ou est du type eau dans huile dans eau.

11. Procédé selon l'une quelconque des revendications 1 à 10, caractérisé en ce que l'émulsion est formée en utilisant un appareil qui comprend une enceinte (13) de formation d'émulsion qui a (a) au moins un orifice d'entrée (1) pour l'introduction de fluides non miscibles, (b) une zone ou lit (4) en communication avec le ou les orifices d'entrée et (c) au moins un orifice de sortie (2) en communication avec la zone oulit (4) pour le retrait de l'émulsion formée, ladite enceinte (3) ayant, de preférence, un profil en coupe d'une figure régulière ou irrégulière ayant au moins trois côtés, vu perpendiculairement au trajet d'écoulement et dans ledit procédé la zone ou lit (4) est garnie avec au moins une matière telle que définie dans la revendication 1.


EMULSION
2
PACKED BED
4
3
INORGANIC PHASE
ORGANIC PHASE
1