United States Patent [19]

Kuerten et al.

[54] METHOD AND APPARATUS FOR MIXING LIQUIDS

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- [58] Field of Search 259/4, 18, 36; 137/604

[56] References Cited UNITED STATES PATENTS

945,143	1/1910	Szamek	
2,831,754	4/1958	Manka	259/4
3,251,550	5/1966	Lippert	
3,502,103	3/1970	Verschuur	
3,755,452			

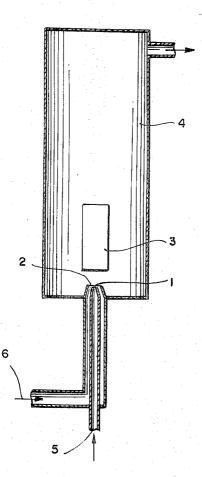
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[57] ABSTRACT

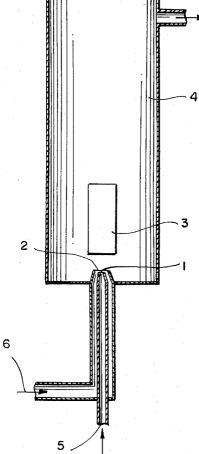
A method of rapidly mixing liquids which differ greatly from one another as regards their volume and-/or density, to form emulsions or homogeneous mixtures. One or more jets of the liquid dispersing agent are passed through nozzles at a velocity up to 100 m/s to an impulse exchange chamber, which is located in the liquid medium and extends in the downstream direction, together with the liquid to be dispersed, which latter liquid is ejected in the immediate proximity of the orifice of the nozzles providing the propulsive jet. The average hydraulic diameter of the impulse exchange chamber is equal to from two to 20 times the diameter of that nozzle which is equal in crosssectional area to all the nozzle orifices present, and its length is equal to from two to 30 times its hydraulic diameter.

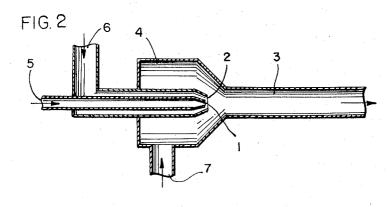
11 Claims, 2 Drawing Figures



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METHOD AND APPARATUS FOR MIXING LIQUIDS

This invention relates to a method of rapidly mixing liquids which differ greatly from one another as regards their volume and/or density to form emulsions or ho- 5 mogeneous mixtures and apparatus therefor.

In industry, mixing of two miscible liquids or the production of liquid-liquid dispersions or liquid-solid dispersions is generally carried out by means of agitators. However, the use of agitators is not satisfactory when 10 the ratio is high, for example, when it is required to make a homogeneous mixture from two liquids flowing together in a ratio of 1:100 or more, by volume, and when it is also required to make said homogeneous mixture or dispersion in a short time. 15

If it is desired to manufacture emulsions of nonmiscible liquids having droplet diameters of a few μm , dispersion can only be carried out in zones of high shear. Care must also be taken to ensure that the liquid to be dispersed reaches said zone of high shear forces. 20 In a stirred vessel, the shear stress is greatest close to the impeller and falls off rapidly toward the vessel walls. The shear forces, i.e., the energy applied by the stirrer, are dissipated over the total volume of the liquid contained in the vessel. Thus the energy required is rel- 25 atively high compared with the dispersing effect obtained. In the case of high throughputs, the mixing time increases due to the fact that the liquid to be dispersed can only be subjected to maximum shear when it is passed through the zone close to the agitator a number 30of times.

It is particularly difficult to mix two liquids which differ greatly in density or of which one contains solid material dispersed therein. Where mechanical agitators are used, satisfactory dispersion is usually only possible ³⁵ by predispersing the heavy liquid in a small volume of the lighter liquid by means of a special impeller capable of producing zones of high shear, this being carried out in a separate small vessel located upstream of the main stirred vessel. The resulting emulsion is transferred to the large stirred vessel and distributed therein with a second mechanical agitator.

The above drawbacks occuring when mixing liquids with the aid of agitators may be obviated and homogeneous mixtures may be rapidly produced using liquids which differ greatly from each other as regards volume and/or density by passing one or more jets of the dispersing agent through nozzles at a velocity of from 5 to 100 m/s and in particular of from 10 to 30 m/s to an im-50 pulse exchange chamber, which is located in the liquid medium and extends in the direction in which the jets enter said medium, together with the liquid to be dispersed, which latter liquid is ejected in the immediate proximity of the orifice of the nozzle providing the propulsive jet, provided that the average hydraulic diameter of the impulse exchange chamber is equal to from two to 20 times the diameter of the nozzle which is equal in cross-sectional area to all of the nozzle orifices present and its length is equal to from two to 30 times 60 the hydraulic diameter.

It is an object of the method and apparatus of the invention to confine the region of shear forces to a small volume and thus to achieve extremely high densities of energy dissipation. Furthermore, the liquid to be dispersed should be fed to the zone of energy dissipation so that said liquid has the desired droplet size after only one pass.

The method of the invention is carried out by means of apparatus consisting of a multi-stream ejector which projects, for example, upwardly into a reactor or vessel and comprises a nozzle for providing the jet of propulsive liquid, a feed pipe for the liquid to be dispersed, which feed pipe surrounds said nozzle coaxially in spaced relationship thereto, and a mixing tube having a diameter greater than that of the feed pipe and located at a distance therefrom, the mixing tube serving as impulse exchange chamber.

When this mixing apparatus is arranged inside another vessel, the flow of liquid leaving the propulsive nozzles has an entraining effect on the liquid contained in said vessel and thus causes it to flow into the impulse exchange chamber where the liquid to be dispersed is mixed with the entrained medium within fractions of a second. Where miscible liquids are used, there is virtually no concentration gradient in the mixture as it leaves the impulse exchange chamber, and in the case of immiscible media, a homogeneous emulsion leaves said mixing chamber. Since the entire mixing and dispersing operation takes place in the impulse exchange chamber, it is possible in some cases to do without the surrounding vessel. In such a case, the relatively slow stream of liquid otherwise entrained from the vessel may be produced by pump metering. This prevents back-flow of the liquid and the residence times of the liquid obtained are similar to those in a tubular reactor.

The purpose of the impulse exchange chamber is to cause the mechanical energy which is supplied to the reactor by the propulsive jets to be distributed in a very small volume, mainly within the said exchange tube, due to mixing of the slow stream of liquid with the other liquid and dispersion of the latter. This produces high local energy dissipation densities ensuring fine dispersion, even at small absolute outputs.

In general, the impulse exchange chamber should have a constant cross-section of a cross-section with in-40 creases in the direction of flow. The said chamber should be oriented in the direction of ingress of the liquid and may take a variety of shapes, the design preferably being adapted to suit the shape of nozzle used. In general, use is made of cylindrical tubes or frustoconical members. Where a cylindrical tube is used as impulse exchange chamber, its length should be equal to from two to 30 times its diameter. If the impulse exchange chamber is not circular in cross-section or its cross-section varies along its length, its length should be equal to from two to 30 times its hydraulic diameter. The impulse exchange chamber should have an average inlet diameter equal to from two to 20 times the diameter of the propulsive nozzle or, where a number of nozzles are used, the inlet diameter should be equal to from two to 20 times the diameter of a nozzle having a cross-sectional area equal to the sum of said areas of said nozzles.

Embodiments of the invention are illustrated in FIGS. 1 and 2. FIG. 1 illustrates a multi-stream ejector installed in a large vessel. The nozzles and impulse exchange chamber are drawn on an enlarged scale compared with the vessel.

In a vessel 4, filled with liquid to a certain level, there is located a nozzle 1 for the introduction of liquid, which nozzle projects upwardly into vessel 4 with its orifice just above the bottom of the vessel. A feed pipe 6 for the liquid to be dispersed surrounds said nozzle coaxially to form an annular channel thereabout, the end of which takes the form of an annular orifice 2 likewise immersed in liquid. Below the surface of the liquid and at a distance therefrom there is located a mixing tube 3 which is disposed coaxially in relation to nozzle 1. In 5 the embodiment shown in FIG. 2, the mixing apparatus is a tubular mixer. The reference numerals denote the following: 1 is the outlet orifice for the propulsive jet, 2 is the outlet orifice for the liquid to be dispersed, 3 is the feed channel for the slow stream of liquid, 4 is the 10 impulse exchange chamber or mixing chamber and 5, 6 and 7 are the inlet lines for the propulsive liquid, the liquid to be dispersed and the slow liquid medium respectively.

EXAMPLE 1

Mixing of two mutually soluble liquids

Since chemical reactions may be affected in an undesirable manner by uneven mixing of the reactants, it is often desirable in chemical industrial plant to effect 20 therewith had a diameter of 15 mm and a length of 100 continuous and even mixing, in a single pass, of two streams of liquid flowing at greatly different rates, for example at rates differing by as much as from 500:1. According to the present invention, mixing may be carried out using an apparatus such as is illustrated in FIG. 25 2. It has been found particularly effective to divide the liquid which flows at the higher rate into two partial streams, for example, at a ratio of 3:1. The smaller of these streams is passed through nozzle 1 at a velocity of 10 m/s, whilst the larger stream is passed slowly 30through feed line 7 to mixing tube 3 at a velocity of about 1 m/s. In an experimental plant, mixing tube 3 has a diameter of 30 mm and a length of 300 mm. The diameter of propulsive nozzle 1 is 5 mm, through which 0.7 m³ of 1/10N HCl solution is passed per hour. The ³⁵ amount of 1/10N HCl solution passed through chamber 4 is 2.1 m³/hr. The second liquid, which it is desired to mix evenly with the first liquid, is in this case 5.6 1/hr of 5N NaOH solution and is passed through line 6 to 40 leave nozzle 2 at a low velocity to the zone of shear between the fast stream leaving nozzle 1 and the slow stream from feed line 7 and is evenly mixed with these streams in impulse exchange chamber 3 in fractions of a second (about 0.05 sec.). There are no dead spots in 45 the mixing zone and back-flow is possible only in small regions thereof, which means that the overall mixing times may be very short.

EXAMPLE 2

Production of a liquid/liquid dispersion

An oil-in-water emulsion is to be prepared. The ejector reactor has a diameter of 150 mm and a length of 800 mm. The reactor, as shown in FIG. 1, is filled with water. Nozzle 1 has a diameter of 2 mm and through 55 this nozzle there is passed water at a velocity of 20 m/s into impulse exchange chamber 3 having a diameter of 15 mm and a length of 100 mm. This jet entrains a slow stream of water from the vicinity of the impulse exchange chamber. The fast and slow streams mix in the 60 impulse exchange chamber and produce a zone of high shear. Paraffin oil is caused to flow at a velocity of 1.5 m/s through annular nozzle 2, which has an outlet area of 22 mm², into said zone of high shear. The oil is broken up into very fine droplets having diameter of be- 65 liquid and second liquid form a homogeneous solution. tween 1 and 30µm. These droplets produced in the impulse exchange chamber are not generally stable. They are therefore stabilized by the addition of a dispersing

agent such as sodium lauryl sulfate and are then measured under a microscope. This method produces stable dispersions showing a narrow spectrum of droplets diameters.

EXAMPLE 3

Production of a liquid/liquid dispersion with liquids showing large density differences

One of the most difficult mixing problems is the dispersion of mercury in an organic solution or water, since the differences in density are extremely great and the heavy mercury tends to collect at the bottom of the reactor.

In one test, water was placed in a glass reactor having 15 a diameter of 150 mm and a length of 800 mm and a zone of high shear was produced using a multi-stream ejector having a two-component nozzle of glass. The diameter of the propulsive jet was 2 mm, whilst the mixing tube disposed above the nozzle orifice coaxially mm. The nozzle orifices were only 2 mm above the bottom of the reactor. The zone of shear was produced by circulating the contents of the reactor. The velocity of the propulsive jet was 35 m/s. The mercury to be dispersed was passed through the annular gap of the nozzle at a rate of 20 kg/hr and a velocity of 0.01 m/s into the shear zone of the mixing chamber. The mercury was broken up into very fine droplets having diameters of between 3 and 90 μ m, a peak being found at a diameter of 12 μ m. In this case, the ejector served not only to break up the mercury but also to distribute it evenly throughout the reactor. Hardly any mercury settled at the bottom of the reactor.

We claim:

1. A method of rapidly mixing liquids which differ greatly from one another as regards volume and/or density, to form dispersions, emulsions or homogeneous mixtures, wherein one or more jets of a first liquid are passed through a nozzle or nozzles at a velocity of from 5 to 100 m/s into an impulse exchange chamber open at both ends, which is located in a liquid medium, extends longitudinally in the direction in which the jet or jets enter said medium and has a volume equal to one hundredth to one ten-thousandth of the volume of a vessel containing said medium, together with a second liquid to be dispersed, emulsified or mixed with said first liquid, which latter liquid is ejected in the immediate proximity of the orifice of the nozzle providing the propulsive jet, the average hydraulic diameter of the impulse exchange chamber being equal to from two to 20 times the diameter of a theoretical, single nozzle which is equal in cross-sectional area to all of the nozzle orifices present, and its length being equal to from two to 30 times the hydraulic diameter.

2. A method of mixing liquids as claimed in claim 1, wherein a fast stream of the first liquid is passed through its nozzle or nozzles at a velocity of from 5 to 100 m/s with the second liquid being ejected into the immediate proximity of said nozzles providing the propulsive jets, and also together with a considerably slower stream of liquid, into said impulse exchange chamber.

3. A method as claimed in claim 1 wherein said first

4. A method as claimed in claim 1 wherein said first liquid and second liquid form an emulsion.

5 5. A method as claimed in claim 1 wherein said first liquid and second liquid form a liquid dispersion.

6. A method of rapidly mixing liquids which differ greatly from one another as regards volume and/or density, to dispersions, emulsions or homogenous mix- 5 tures, wherein one or more jets of a first liquid are passed through a nozzle or nozzles at a velocity of from 5 to 100 m/s into an impulse exchange chamber open at both ends, said chamber being immediately downstream of said nozzle or nozzles, and extending longitu- 10 dinally in the direction of said jet or jets, together with a second liquid to be dispersed, emulsified or mixed with said first liquid, which second liquid is ejected in the immediate proximity of the orifice of the nozzle providing the propulsive jet, the average hydraulic di- 15 rected, liquid streams, one of which is a fast propulsive ameter of the impulse exchange chamber being equal to from two to 20 times the diameter of a theoretical, single nozzle which is equal in cross-sectional area to all of the nozzle orifices present, and its length being equal to from two to 30 times the hydraulic diameter. 20

7. A method of mixing liquids as claimed in claim 6, wherein a fast stream of the first liquid is passed through its nozzle or nozzles at a velocity of from 5 to 100 m/s with the second liquid being ejected into the 25 immediate proximity of said nozzles providing the propulsive jets, and also together with a considerably

slower stream of a liquid, into said impulse exchange chamber.

8. A method as claimed in claim 6 wherein said first liquid and second liquid form a homogeneous solution.

9. A method as claimed in claim 6 wherein said first liquid and second liquid form an emulsion.

10. A method as claimed in claim 6 wherein said first liquid and second liquid form a liquid dispersion.

11. An apparatus for the intermixing of liquids in an impulse exchange zone of high liquid/liquid shear forces, which comprises a vessel adapted to contain a liquid, upwardly directed nozzle means for introducing into the lower portion of said vessel two upwardly dijet stream having a velocity of 5 to 100 m/s and the other of which is a slower stream, and an impulse exchange chamber which extends in the direction of ingress of the liquids ejected by said nozzle means and which is located immediately above and downstream of said nozzles, the hydraulic diameter of said chamber being equal to from two to 20 times the diameter of a single, theoretical nozzle which is equal in crosssectional area to all of the nozzle orifices present, and the length of said chamber being equal to from two to 30 times its hydraulic diameter.

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UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

PATENT NO. 3,847,375 DATED November 12, 1974

INVENIOR(S) Heribert Kuerten and Otto Nagel

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Page 1 should read:

--[30] FOREIGN APPLICATION PRIORITY DATA

October 14, 1971 Germany..... P 2151206.1

Signed and sealed this 20th day of May 1975.

(SEAL) Attest:

RUTH C. MASON Attesting Officer C. MARSHALL DANN Commissioner of Patents and Trademarks