

The present invention relates to an ultrasonic device for the continuous production of particles having as uniform a particle size distribution as possible, and more particularly microdroplets of controlled diameter and sphericity.

The problem of producing finely divided microdroplets is encountered in many fields where it is necessary to spray or atomize organic and/or inorganic liquids, for example in the pharmaceutical field to deliver a product (a drug or the like) in suspension, by means of an aerosol, or a product coated with a matrix (slow-release drug). In the same connection, mention may be made of the field of cosmetics, for example in the production of what may now be called liposomes.

Ultrasonic devices are known for this purpose, such as those disclosed in DE-A-2 537 772 or DE-A-3 036 721, which respectively describe devices having the characteristics referred to in the preamble to Claim 1, for the continuous production of microdroplets specifically from liquids, these devices, which comprise a vibrating surface, atomizing the liquid, which is brought up from the inside, by means of their orthogonal ultrasonic vibratory mode. However, due to the configuration of the means for delivering the liquid in these devices referred to, the latter only operate if the liquid does not have too high a viscosity. In addition, the rate at which the liquid is delivered can be quite irregular and the distribution can be in the form of a sheet of variable thickness.

On the other hand, carburettors of internal combustion engines or boiler injectors, such as described in EP 0 202 381, are known which operate by ultrasound to atomize liquid fuel. Once again, taking into account the very small flow cross sections involved, these atomizers can only operate properly with volatile fuels.

Now, many parts are currently manufactured from fine metal powders or granules, particularly by sintering operations. The problem of particle size distribution is seen to be significant since it is desirable to produce batches of microbeads all having essentially the same size.

In molten metal technology, an ultrasonic device is known which makes it possible to obtain microbeads having a diameter of the order of 5 to 200 microns. A substance in the liquid state is allowed to fall drop-wise onto the end surface of a vibrator oscillating at a frequency of 5 to 50 kilohertz. An ultrasonic vibrator comprises, for example, a piezoelectric transducer extended by a concentrator, that is to say a part whose particular shape allows resonance to occur on the end surface having an order of magnitude greater than that of the oscillation of the transducer. Such a vibrating device is also known in industry as a "sonotrode". This process is preferably carried out in a chamber which enables the atmosphere to be controlled, either under vacuum or an inert gas.

When a drop of liquid falls onto the ultrasonic vibrating surface, the formation of a film is observed, the upper surface of which assumes the form of a network of stationary waves having a wavelength of for example 200 microns. When the amplitude of these waves reaches a sufficient value, drops of liquid become detached from the crest of the waves and, by cooling, form solid microbeads having a diameter of the order of 50 microns. It is noted that the diameter of microbeads thus obtained depends to a large extent on the frequency of vibration of the surface. The distribution of diameters about a mean value depends in part on the amplitude of the vibration, but more especially on the rheological properties of the material which varies appreciably between a liquid and a molten material. For its part, the hourly production rate of microparticles depends essentially on the amplitude of the vibration and the dynamic viscosity coefficient of the material.

However, the ultrasonic device described above exhibits interference effects due to the configuration and arrangement of the elements which bring about a particle size distribution which is still unsatisfactory since the microbeads have to be graded by being passed through a series of screens. It has been found on the one hand that the network of waves on the sheet of liquid is unstable on the vibrating surface and, on the other hand, that the thickness of this sheet was variable, being thick near the place where the material arrives and too thin towards the edges of the surface.

The aim of the present invention is to obviate the aforementioned disadvantages due to a large extent to the excessively irregular flow and distribution of liquid on the vibrating surface, in order to produce microdroplets having a more controlled diameter with an improved yield.

These aims have been achieved by means of an ultrasonic device comprising a vibrating surface which by its orthogonal ultrasonic vibratory mode atomizes a material in the liquid state brought up from the interior of the device by a means of a delivery, characterized in that the material in the liquid state is distributed over the vibrating surface by an annular channel or several channels of which one of the dimensions of the flow cross section is of the order of a millimetre or is submillimetric, either from an intermediate flow-regulating and/or heat regulating chamber/crucible or directly from the said annular channel serving as an intermediate chamber/crucible, the said intermediate chamber being subjacent to the vibrating surface, as well as a process for its use. The means of delivery advantageously comprise one or more delivery channels for the material in the liquid state and the base of the said intermediate chamber, where the said delivery channel or channels opens or open out is situated in the plane of a wave node of the vibratory regime.

In a useful embodiment, the material in the liquid state is distributed over the vibrating surface by one or more channels of which the total flow cross-section is greater than 8mm^2 , the flow of the material taking place by capillarity and/or pressure gradient induced by the vibratory mode.

A "material in the liquid state" is understood to be both a liquid per se at ambient temperatures as well as a molten material, that is to say one which is solid at these temperatures and made liquid as required. This material may be inorganic and/or organic. By virtue of the presence of the intermediate chamber, it becomes possible on the one hand to control the viscosity of the liquid on the vibrating surface through its temperature, and on the other hand to control losses in pressure more effectively by making the ejection channels, which are now shorter, with greater precision. Thus, a material which is initially solid may be kept in the liquid state or may be liquified by heating in the intermediate chamber acting as crucible very close to the vibrating surface.

According to a first preferred embodiment, the internal intermediate chamber is cylindrical and parallel to the vibrating surface. Material in the liquid state is thus distributed by several channels situated at regular intervals along the line of the chamber closest to the surface.

According to a second preferred embodiment, the internal intermediate chamber is central and has a tubular shape orthogonal to the vibrating surface onto which it opens directly at one of its ends, which may be tapered if so desired.

According to a third preferred embodiment, the tubular internal intermediate chamber is situated at the periphery of the vibrating device, sub-adjacent and orthogonal to the convex vibrating surface, at the periphery of which it opens.

Advantageously, the vibrating surface has a series of parallel or crisscross grooves, or a network of circular and/or radial grooves which tend to stabilize the position of the undulating regime of the film. The grooves may have a rectangular cross section, a trapezoidal cross section, a U-shape etc.

In order to improve the spread of the material in the liquid state over the vibrating surface, the channels may open at an angle of between 25 and 60 degrees in relation to the surface. Alternatively, a cover parallel to the surface is arranged above and close to the exit of the channel or channels.

The invention is described below in a more detailed manner with the help of working examples without these being limiting in nature, illustrated by the accompanying drawings in which :

- figure 1 is a partially exploded perspective view of a first embodiment of the ultrasonic device,
- figure 2 is a partially exploded perspective view of a second embodiment of the ultrasonic device,
- figure 3 is a partially exploded perspective view of a third embodiment of the ultrasonic device.

Figure 1 shows the end of a vibrating element 10, in the present case the concentrator of a sonotrode, which ends in the vibrating surface forming an atomizer 11. In the partial cross section through the vibrating element 10, an internal intermediate chamber 22 may be observed, used as an internal crucible, which is substantially a cylinder the side of which is parallel to the atomising surface 11. This internal intermediate chamber 22 is supplied with material in the liquid state, for example in the molten state, by a feed channel 20. Since the vibrating element 10 is heated, the molten material also remains heated inside the intermediate chamber 22. A series of ejection channels 24, for example of

the order of 15, connects this intermediate chamber 22 to the surface of the atomizer 11. These ejection channels are preferably arranged at regular intervals along the line of the intermediate chamber closest to the surface. The diameter of each of these channels is of the order of a millimetre.

The vibratory mode of the sonotrode creates a difference in pressure between the channels 24 and the outside, causing the molten material to issue from these channels. Taking into account the narrow flow cross section of each of these channels, the exit flow rate of the molten material is influenced by capillarity effects, which are themselves dependant on the one hand on the ultimate shape and quality of the machining of the channels, and on the other hand on the rheological properties of the molten material which are themselves dependant on the value of the final temperature of the material.

As may be clearly seen in figure 1, the atomizing surface 11 comprises a series of regular parallel serrations or grooves of the order of one millimetre wide and approximately 0.25 mm deep for a spacing of the order of 2 millimetres. The ejection channels 24 respectively open out at the bottom of a groove thus formed. The object of these grooves is to stabilise the spread sheet of material in a lateral position.

In order to limit the projection of material from the exits of the channels beyond the vibrating surface 11, a shoe is provided on the vibrating element 10 the end of which constitutes a cover 30 parallel to the atomizing surface 11, this cover being disposed above the ejection channels 24. The shape, dimensions and weight of this shoe and cover 30 are obviously gauged to avoid too sensitive modifications to the vibratory regime of the vibrating element 10, particularly the concentrator.

Alternatively, the effect of the cover 30 may be replaced by oblique openings of the ejection channels 24, that is to say opening on to the vibrating surface 11 at an angle of between 15 and 75 degrees with relation to that surface. However, in practice the formation of such angled channels is more difficult and may in addition create losses of pressure within these channels.

As will be appreciated, the flow of molten material over the atomizing surface 11 is determined on the one hand by the flow cross section of each of the channels 24 and on the other hand by the number of these channels.

Figure 2 shows a second variant of the end of the sonotrode in which the main components are coaxial to the cylindrical vibrating element 60. To this end, a blind hole 75 is first drilled at the middle of the vibrating element 60. A component 76 is forcibly inserted into this blind hole, the lower part having a diameter corresponding to that of the blind hole 75 and the upper part having a restriction 74.

The base of the component 76 is advantageously situated in the plane of a wave node, that is to say at the height of the sonotrode where the amplitude of vibration is at a minimum, thus voluntarily limiting the more or less controlled resonance of this component 76.

Together with the upper part of the blind hole 75, the restriction 74 creates a cylindrical, elongated channel 72, in other words an annular or toric channel, opening directly onto the upper atomizing surface 61. This annular or toric channel acts here both as an internal chamber/crucible and an ejection channel. If the volume of this internal chamber has to be larger for reasons of flow regulation by a buffer effect, the restriction 74 is then reduced at its upper end to form a tapered ejection channel.

A transverse feed channel 70 intersects the channel 72. This feed channel 70 can open into the blind hole 75 at any height, but preferably at the base or at the level of the wave node plane.

As may be noted in figure 2, the upper atomizing surface 61 also has a series of parallel grooves 64 having dimensions essentially identical to the grooves described in figure 1. It may prove useful to stabilize the sheet of molten material in the two axes of the plane of the surface 61, which is done by cutting supplementary transverse grooves 62.

In addition, the element 76 is completed at its upper end by a cover 80 projecting beyond the exit from the channel 72. As before, the function of this cover 80 is to direct the ejected molten material back towards the atomizing surface 61. Alternatively, the blind hole 75 may have a conical opening at the level of the surface 61 with an apex angle which may vary between 30 and 150°. Thus, instead of a cover 80, the element 76 is conically widened at a corresponding angle.

Figure 3 illustrates a third variant of the end of the sonotrode comprising a cylindrical vibrating element 100, which may even be in the shape of a truncated cone towards its lower end, and having in its upper part, sub-adjacent to the vibrating surface 105, a peripheral groove 127 which forms an intermediate chamber 128 in conjunction with a detachable collar 110. The internal diameter of the collar 110 is larger than the external diameter of the vibrating element by one to two millimetres. This collar 110 is preferably fixed to the vibrating element 100 at the level of a wave node plane to limit possible vibrations in the manner of a bell.

The material may be brought to the intermediate chamber 128, preferably near to the wave node, that is to say at the base of the chamber, through internal channels 122, 124 and 126, and even by an external channel. For machining reasons,

the central channel 124 may be drilled from the lower surface, and the inlet opening is consequently reclosed.

An ejection channel 130 is formed at the periphery of the vibrating surface 105 by the small difference between the external diameter of the vibrating element 100 and the internal diameter of the collar 110. As may be seen in figure 3, the vibrating surface 105 is slightly convex, and in particular is rounded at the exit of the ejection channel 130. This rounded shape avoids any disruption of the film of liquid material progressing radially from the channel to the centre of the surface. If necessary, the upper edge of the collar 110 may also be slightly turned radially inwards to accompany the movement of the liquid material over the periphery of the vibrating surface.

The outer part 106 of the vibrating surface 105 also has a certain slope, of the order of 10 to 20°, relative to the horizontal, both to soften the rounded part at the exit from the ejection channel and also to direct the majority of the microdroplets which are forming there to the exterior of the device. Since this peripheral zone 106 proves to be the most important from the point of view of the formation of microdroplets, it may be an advantage to cut a network of radial circular grooves in this zone to regulate the distribution of liquid material.

As may be readily appreciated, the sonotrode with its vibrating surface according to the invention is installed within an enclosed chamber to enable the surrounding medium to be controlled, that is to say to enable operations to be carried out under vacuum or under an inert gas.

The vibrating element 10 in figure 1 is advantageously inclined upwards at an angle of 45° with the feed channel 20 also directed upwards. A stream or sheet of material or a rod of preheated metal can thus be introduced into the channel 20

from above, this metal then melting in the internal intermediate chamber 22. Alternatively, the metal is previously heated in a main crucible which is then brought in the molten state into the entry channel 20. It has been noted that the grooves 12 ensure lateral stabilization of the sheet and, to a certain extent, ensure deliver of material coming from the channels.

The vibrating element 60 according to figure 2, or the element 100 in figure 3, may be installed within the chamber at an angle of between 0 and 180° relative to the horizontal. Even when the element is installed at an angle greater than 90°, the sheet will still remain on the vibrating surface 61 under the effect of surface tension. This phenomenon is further confirmed when the vibrating surface is directed downwards. Detached microdroplets are projected and fall directly downwards by gravity whilst cooling and forming microbeads.

If necessary, particularly for achieving higher vibration frequencies, and hence smaller microbead diameters, the vibrating element may also consist of a vibrating plate, the intermediate chamber with its ejection channels being then arranged in the plate beneath the upper surface.

The invention may be applied in various fields where it is desirable continuously to produce particles having as uniform a size distribution as possible and those which have been mentioned above may be usefully recalled, that is to say molten metals, as well as pharmaceuticals, cosmetics and internal combustion engines.

CLAIMS

1. An ultrasonic device for the continuous production of microdroplets having a uniform particle size distribution, comprising a vibrating surface (11,61,105) which by its orthogonal ultrasonic vibratory mode atomizes a material in the liquid state brought up from the interior of the device by a means of delivery (20,22,24; 70,72; 122,124,126,128,130) characterized in that the material in the liquid state is distributed over the vibrating surface (11,61,105) by an annular channel (72,130) or several channels (24) of which one of the dimensions of the flow cross section is of the order of a millimetre or is submillimetric, either from an intermediate flow-regulating and/or heat regulating chamber/crucible (22,128) or directly from the said annular channel (72) serving as an intermediate chamber/crucible, the said intermediate chamber (22,72,128) being subjacent to the vibrating surface (11,61,105).
2. Ultrasonic device according to claim 1, characterized in that the said means of delivery comprises one or more conduits (20,70,126) for delivering the material in the liquid state, the base of the said intermediate chamber (22,72,128) where the said delivery conduit or conduits emerge (20,76,126) being situated in the plane of a wave node of the vibratory mode.
3. Ultrasonic device according to claims 1 or 2, characterized in that the total cross flow section of the channel or channels (72,130,24) is greater than 8 mm^2 , the flow of the material taking place by capillarity and/or by the pressure gradient induced by the vibratory mode.
4. Ultrasonic device according to one of the preceding claims, characterized in that the channels open out onto the vibrating surface (11) at an angle of between 25 and 75 degrees to the surface.



5. Ultrasonic device according to one of claims 1 to 4, characterized in that the internal intermediate chamber (22) is cylindrical and parallel to the vibrating surface (11) and in that the material in the liquid state is distributed through several channels (24) situated at regular intervals along the line of the chamber nearest to the surface (11).

6. Ultrasonic device according to one of claims 1 to 4, characterized in that the internal intermediate chamber (72) is central and has a tubular shape orthogonal to the vibrating surface (11) on which it opens directly at one of its ends which may be tapered if so desired.

7. Ultrasonic device according to one of claims 1 to 4, characterized in that the tubular internal intermediate chamber (128) is situated at the periphery of the vibrating device (100), subjacent and orthogonal to the vibrating surface (105), having a convex shape with respect to the said vibrating device, at the periphery of which it emerges.

8. Ultrasonic device according to one of the preceding claims, characterized in that the vibrating surface (11) has a network of parallel or crisscross straight grooves (12) or a network of circular and/or radial grooves.

9. Ultrasonic device according to claim 8, characterized in that the grooves have a rectangular cross section.

10. Process for the manufacture of microbeads from thermofusible material according to which the material is introduced into the intermediate chamber/crucible of an ultrasonic device according to one of claims 1 to 8, such that the said material is introduced in the fused state, the microdroplets produced by the device and detached from the vibrating surface falling by gravity whilst cooling to form the said microbeads.

