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[54] **METHOD FOR DETECTING SMALL PARTICLES IN A TANK AND AN APPARATUS FOR CARRYING OUT THE SAME**

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[30] **Foreign Application Priority Data**

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[51] **Int. Cl** **H04b 11/00**

[58] **Field of Search**....................340/1 R, 15; 73/61, 73/67.5, 67.6; 181/.5 NP

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[57] **ABSTRACT**

An ultra-sonic transmitting means and an ultra-sonic receiving means are provided in fluidized region of a tank at a fixed interval. An ultra-sonic wave is transmitted from the transmitting means to the receiving means and bubbles are prevented from entering the space between the transmitting means and the receiving means, and the presence or absence of small particles is detected by measuring the attenuation of the ultra-sonic wave.

10 Claims, 9 Drawing Figures

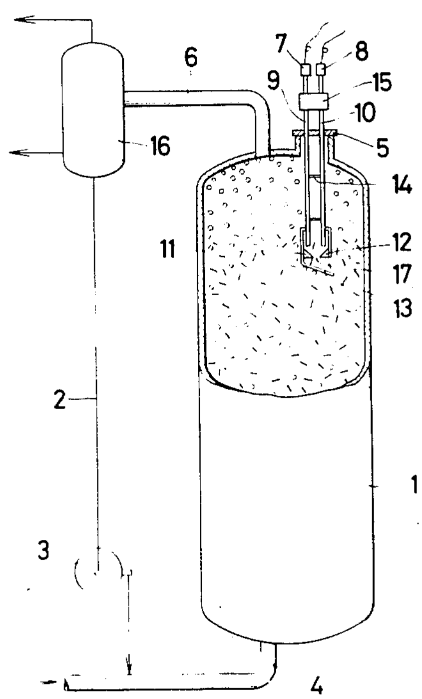


Fig.1

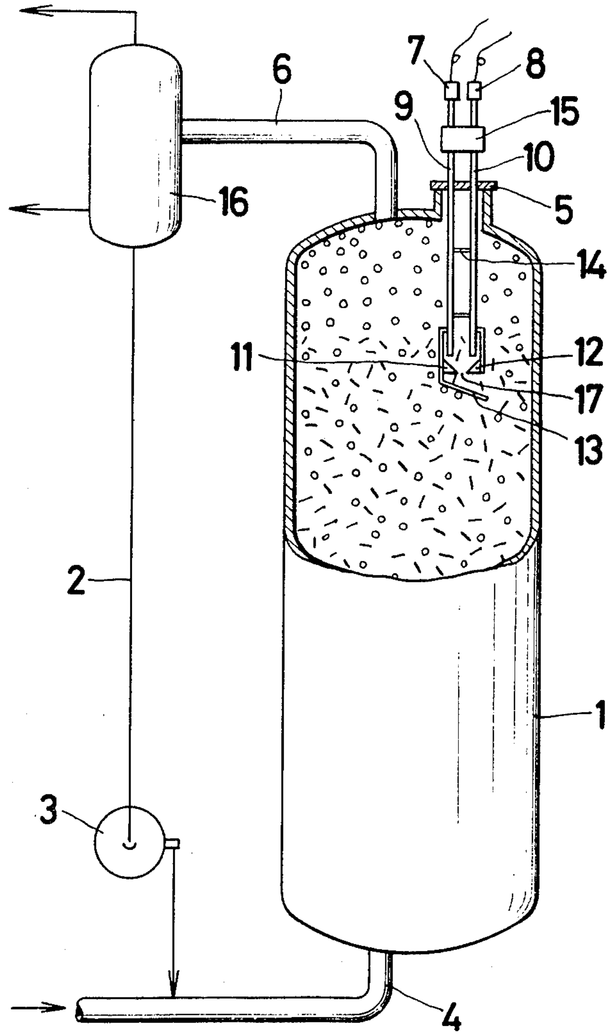


Fig.3

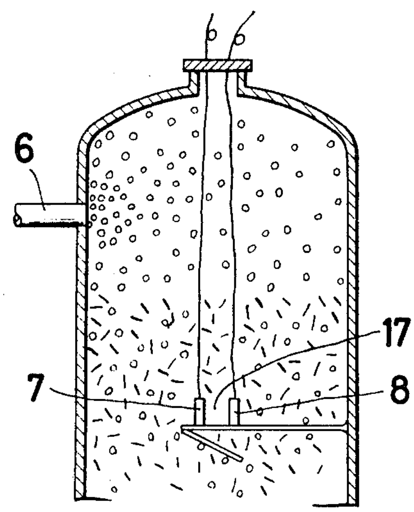


Fig.2

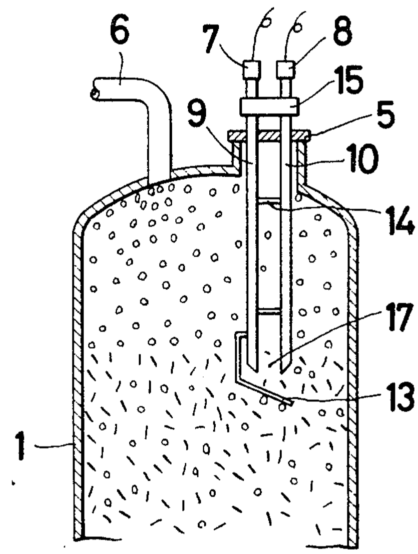


Fig.4

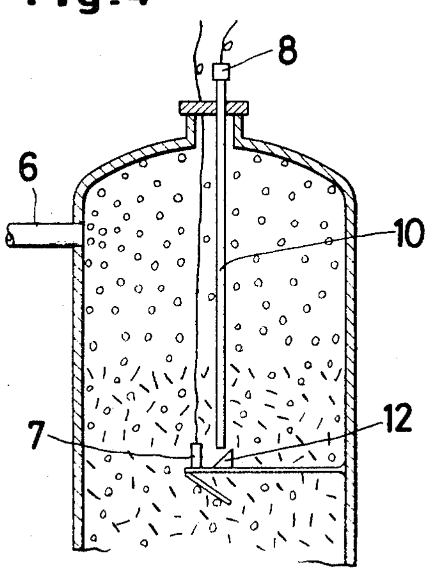


Fig.6

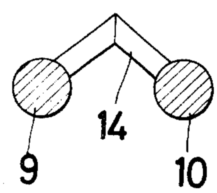


Fig.5

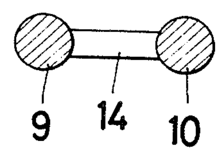


Fig.8

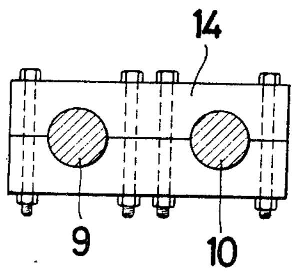
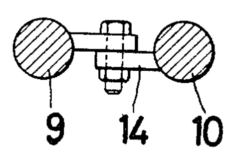


Fig.7



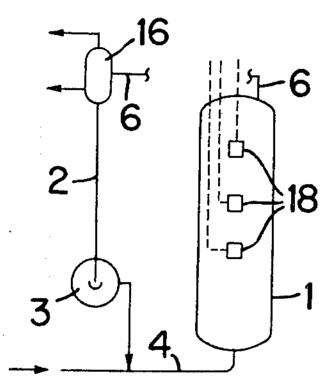


Fig.9

METHOD FOR DETECTING SMALL PARTICLES IN A TANK AND AN APPARATUS FOR CARRYING OUT THE SAME

The present invention relates to a method for detecting small particles and a device for carrying out the same.

In general, the small particles contained in the liquid in a reactor sink under the pull of gravity and accumulate at the bottom of the reactor as a fixed bed. This fixed bed is converted to a fluidized bed by supplying a fluid at a predetermined rate of flow from the lower portion of the reactor. As there is good material mobility and heat transfer in the fluidized bed, this type of bed is used widely in carrying out chemical reaction, heating, cooling, drying etc..

Heretofore known methods for measuring the small particles in a reactor employing the fluidized bed include those utilizing the difference in density of the materials in the reactor and those utilizing the change in electrostatic capacitance. In the conventional methods, although it is possible to obtain relatively correct measurement when bubbles do not exist in the reactor, it is impossible to measure the small particles accurately when the fluid to be supplied to the reactor includes both gas and liquid phase materials because the gas phase materials, i.e., the bubbles are detected indistinguishably from the small particles. A method is also known in which an ultrasonic transmitting apparatus and an ultra-sonic receiving apparatus are provided in the tank and the small particles are detected by measuring the time duration between transmission and reception of the ultra-sonic wave. In this method, however, it is very difficult to distinguish the ultra-sonic waves reflected by the small particles from those reflected by the bubbles because, where the small particles coexist with the bubbles, the ultra-sonic wave is also reflected by the bubbles. In particular, where the reaction must be performed under severe conditions, such as in the case of the hydrogenolysis of the reduced pressure residual oil which is performed at 200 atms and at 450°C, the existence of the small particles in the tank is detected by utilizing gamma rays. In this method the tank is irradiated by gamma rays from the inside and the gamma rays penetrating the tank are measured by a counter. The thickness of the tank in the vicinity of the area where the measuring is performed is made thinner in order to minimize the absorption of the gamma rays by the tank. As a result, the ability of the tank to withstand high pressure is reduced thus increasing the danger of explosion of the tank.

One object of the present invention is to provide a method for easily detecting the existence of small particles in a fluidized bed containing bubbles particularly when the fluidized bed is under severe conditions.

Another object of the present invention is to provide an apparatus for carrying out this method.

Other objects and features of the present invention will become apparent from the following description with reference to the attached drawings, in which:

FIG. 1 is a side view partially in section of an embodiment of the apparatus of the present invention;

FIGS. 2, 3 and 4 are side views partially in section of another embodiment of the apparatus of the present invention;

FIGS. 5, 6, 7 and 8 are plan views showing methods for securing the transmitting and receiving wave guide rods and

FIG. 9 diagrammatically illustrates the apparatus of FIG. 1 having a plurality of detection elements.

FIG. 1 shows a conventional reactor provided with a detector constructed in accordance with the present invention. A raw material supply pipe **4** is provided at a lower portion of the tank **1** to fluidize the small particles (catalyzer) in the tank by feeding therethrough raw material (in gas phase and liquid phase) pressurized to a predetermined level by a circulating pump **3.** The reaction products are removed from the system through an outlet pipe **6** and separated into gas and liquid by a gas-liquid separator **16.** A portion of the liquid is fed back to the tank **1** by a pipe **2** and the circulating pump **3.** The tank **1** has a supporting body **5** at the upper portion thereof which supports a transmitting wave guide rod **9** and a receiving wave guide rod **10** passing therethrough at a fixed distance from one another. Each wave guide rod extends into the interior of the tank **1** by a predeterminated length. The wave guide rod **9** has extending therefrom a transmitting reflector plate **11** inclined by 45° while the wave guide rod **10** has extending therefrom a receiving reflector plate **12** inclined by 45°. The plates **11** and **12** are disposed so that the inclined faces thereof are in opposing relation. The opposite ends of the wave guide rods **9** and **10** which are outside of the tank are connected respectively to an ultra-sonic transmitter **7** and an ultrasonic receiver **8.** The ultra-sonic wave transmitted from the ultra-sonic transmitter **7** is guided by the transmitting wave guide rod **9** to the transmitting reflector plate **11,** deflected 90° thereby, transmitted through the tank to the receiving reflector plate **12,** deflected 90° thereby, guided out of the reactor by the receiving wave guide rod **10** and detected by the ultra-sonic receiver **8.** Accordingly, when there are bubbles or small particles in the space **17** between the receiving reflector plate **11** and the transmitting reflector plate **12,** the ultra-sonic wave is scattered in the space or absorbed by the small particles or the bubbles, so that the strength thereof is considerably reduced.

In the present invention, in order to avoid the attenuation of the ultra-sonic wave by the bubbles, a plate **13** for preventing the bubbles from entering into the space **17** is provided at the lower portion of the space **17.** The plate **13** may be of any material which is not damaged by the liquid reactant and the shape thereof may be of any shape which does not disturb the formation of the fluidized bed.

The plate for preventing the bubbles from entering the space prevents the bubbles from entering the space **17** from the bottom thus ensuring that no bubbles are present between the transmitting reflector plate and the receiving reflector plate. Thus, if the ultra-sonic wave received by the ultra-sonic receiver **8** is considerably attenuated, the existence of the small particles is affirmed and if there is no attenuation of the ultra-sonic wave, the small particles have not reached the predetermined level yet.

A cooling device **15** may be provided to protect the ultrasonic transmitter **7** and the ultra-sonic receiver **8** from damage due to high temperature where the reaction is performed at a high temperature.

FIG. 2 shows another embodiment wherein the transmitting wave guide rod **9** and its ultra-sonic transmitting reflector plate **11** are formed as one body as are the receiving wave guide rod **10** and its integrated ultra-sonic receiving reflector **12.** The ends of the wave guide rods 9 and 10 in the tank are cut to form end faces inclined by 45°. The inclined end faces are in back-to-back relation. The ultra-sonic wave transmitted from the ultra-sonic transmitter is deflected by 90° at the tip of the wave guide rod 9, guided through the reactant to the inclined portion of the wave guide rod 10 deflected by 90° thereby and detected by the ultra-sonic receiver through the wave guide rod 10. In this embodiment, although the loss is slightly larger than that of the embodiment shown in FIG. 1 because the ultra-sonic wave transmitted is not fully reflected at the reflecting plate, the construction itself becomes simpler than that of the arrangement in FIG. 1.

Where the reaction temperature of the tank is relatively low, an arrangement as shown in FIG. 3 may be used in which the ultra-sonic transmitter 7 and the ultra-sonic receiver 8 are disposed in the tank 1 at a fixed interval. In this arrangement, the ultra-sonic wave is transmitted directly from the transmitter to the receiver, the detected output is converted into an electric output and the electric signal is transmitted through a lead wire to the exterior of the tank 1.

FIG. 4 shows another embodiment which is a combination of the embodiment shown in FIG. 1 and FIG. 3. In this embodiment, the ultra-sonic transmitter 7 is provided in the tank 1, the ultra-sonic wave is deflected by 90° by the receiving reflector plate 12 and detected through the receiving wave guide rod 10 by the ultra-sonic receiver 8.

By providing the ultra-sonic transmitter in the tank as in the embodiments shown in FIGS. 3 and 4, the transmitting output power can be minimized. However, this method cannot be used where the reaction temperature is higher than the transition temperature of the crystal used in the ultra-sonic transmitter, i.e., 570°C.

Although the transmitting wave guide rod 9 and the receiving wave guide rod 10 are held fast by supporting member 5 through which they pass, they may, if several meters in length, be caused to vibrate by the motion of the liquid and particles. This will make it difficult to measure the ultra-sonic wave as the rods may come into contact, be damaged or give rise to noise. For this reason, the vibration of the transmitting wave guide rod and the receiving wave guide rod is prevented by connecting them to each other by a supporting means 14. In this case, however, if the supporting means 14 is in the form of a single rod, as shown in FIG. 5 and the wave guide rods are secured to the respective ends of the supporting rod by welding etc., the ultra-sonic wave will be transmitted from the transmitting wave guide rod 9 through the supporting rod 14 to the receiving wave guide rod 10 and thus this construction may constitute a source of noise. In particular, when the supporting rod 14 is provided in the vicinity of the top of the wave guide rods, it becomes difficult to distinguish the ultra-sonic wave passing through the reactant from that passing through the supporting rod because these waves are received at substantially the same time. In order to avoid this problem, the supporting rod is secured to the wave guides 9 and 10 in a suitable manner using at least two supporting members, as shown in FIGS. 6, 7 and 8. When the connection of the wave guides 9 and 10 to the supporting means is made by welding, it is preferable that the supporting means be constituted with two separate members 14 connected with each other by welding, screws etc., in such a manner that the wave guide 9 is secured to the other member as shown in FIGS. 6 and 7. Alternatively, the above problem may be avoided by an unwelded connection. In this case, the wave guides 9 and 10 are pinched and held by two supporting pieces 14 at the respective ends thereof, the pieces being assembled in such a way that they oppose each other as shown in FIG. 8.

The wave guide rods used in the present invention may be of any material which can withstand the liquid reactant and generally stainless steel is used as the wave guide material. When the ultra-sonic wave passes through the stainless steel to the liquid and through the liquid to the stainless steel, the wave is reflected at the interface between the stainless steel and the liquid and between the liquid and the stainless steel, and thus about 85 percent of the strength thereof is lost. Furthermore, when the small particles exist, the ultra-sonic wave is further attenuated to 1/1000 of its original value. Since the relation between the length of the stainless steel and the amount of attenuation is fixed, any large attenuation of the ultra-sonic wave beyond the calculated amount of attenuation indicates the existence of small particles.

As an example, when the diameter and the length of the transmitting wave guide rod of stainless steel are 20 millimeters and 7 meters, respectively, and an ultra-sonic wave of 15 volts and 1 MHz is applied thereto in the absence of small particles, about 85 percent of the energy of the wave is lost during its travel to the receiving rod, so that the receiver detects about 0.05 volts ultra-sonic wave. On the other hand, when small particles exist between the rods the wave is attenuated to about 1/1000 of its original value and thus the strength of the received wave becomes about 0.000054 volts. Of course the degree of the attenuation of the ultra-sonic wave varies depending upon the density of the small particles and the reaction conditions etc. However, as above mentioned, the ultrasonic wave is greatly attenuated when the small particles exist, and therefore the detection is very easy. Furthermore, since the present invention provides separate transmitting and receiving devices, it is possible to distinguish the wave passing through the liquid from that reflected at the end faces of the wave guide rods thus making it possible to simplify the construction of the ultra-sonic receiver.

A tank capable of safe continuous operation can be constructed (FIG. 9) by providing a plurality of the detection apparatuses 18 of the present invention in vertical arrangement and at appropriate intervals within the tank, constantly checking the level of the small particles in the tank and controlling (or stopping) the supply of liquid and the rate of circulation by means of an automatic control device 3 when the small particles reach the predetermined level.

While the drawings show embodiments using a transmitting wave guide rod and a receiving wave guide rod disposed in parallel, it should be noted that many modifications of the rod arrangement can be made within the scope of the present invention, and as an example, these rods may be arranged in such a manner that they are disposed oppositely or one of them makes an angle to the other.

In summary the present invention provides a means for detecting the level of small particles in a fluidized bed containing bubbles and thus easily overcomes a problem unsolved by the conventional devices. In particular the present invention makes possible the detection of the level of the small particles where the reaction is being carried out under very severe conditions so that the reaction can proceed effectively and safely.

What is claimed is:

1. A method for detecting small particles in a tank comprising the steps of providing in said tank an ultra-sonic transmitting means and an ultra-sonic receiving means opposite to each other at a fixed interval, maintaining the space between said transmitting means and said receiving means free of bubbles, transmitting an ultra-sonic wave from said transmitting means to said receiving means, and detecting the presence or absence of small particles in said space by the attenuation or lack of attenuation of said ultra-sonic wave.

2. An apparatus for detecting small particles in a tank, comprising a tank, an ultra-sonic transmitting means provided in said tank, an ultra-sonic receiving means also provided in said tank oppositely to said ultra-sonic transmitting means at a fixed distance therefrom, and a plate for preventing bubbles from entering the space between said transmitting means and said receiving means whereby the presence or absence of said particles between said transmitting means and said receiving means is detected by the attenuation or lack of attenuation of an ultra-sonic wave transmitted from said transmitting means to said receiving means.

3. The apparatus defined in claim **2**, wherein said transmitting means and said receiving means are in the form of rods connected at one end respectively to an ultra-sonic transmitter means and an ultra-sonic receiving means located outside of said tank.

4. The apparatus defined in claim **3**, wherein said plate for preventing the bubbles from entering said space is provided on an extension of a transmitting rod.

5. The apparatus defined in claim **3**, wherein the other end of said transmitting rod and the other end of said receiving rod have inclined faces in opposing relation.

6. The apparatus defined in claim **3**, wherein a member having an inclined face is disposed on said extension of said transmitting rod and on an extension of said receiving rod.

7. The apparatus defined in claim **2**, wherein said transmitting means is an ultra-sonic transmitter connected to a lead wire leading out of said tank.

8. The apparatus defined in claim **2**, wherein said receiving means is an ultra-sonic receiver connected to a lead wire leading out of said tank.

9. The apparatus defined in claim **3**, wherein said rods are supported in parallel relation by at least two members which are welded to said rods and connected with each other in such a manner that the connection between said members absorbs the stress of said rods.

10. The apparatus of claim **2** wherein a plurality of ultrasonic transmitting means and ultra-sonic receiving means are disposed in said tank at different levels.

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