

SYSTEM AND METHOD FOR PROVIDING MIXED MODE ENERGIES DURING A CONTINUOUS CHEMICAL FLOW PROCESS

Field of the Invention

5 This invention relates to a continuous flow chemical process. More particularly, this invention relates to simultaneously providing ultrasonic energy and a second type energy to a continuous flow chemical process to increase the efficiency of the process. Still more particularly, this invention relates to providing ultrasonic energy from an ultrasonic transducer in direct contact with a first side of a flow reactor in the absence
10 of a carrying medium while providing a second type of energy from a second side of the reactor.

Prior Art

 Chemical transformations in pharmaceutical and other chemical facilities are predominately performed using traditional batch processing. However, batch
15 processing is not desirable because of the inefficient mixing and heat transfer.

 In view of the above limitations with batch processing, those skilled in the art are constantly striving to provide better forms of processing for chemical transformations. One alternative type of processing is continuous flow processing.
20 However, continuous flow processing has heretofore not been widely adapted because of drawbacks in continuous flow processing system. One drawback is that the efficiency of the current continuous flow processing technology is not at an acceptable level that makes such processes economically feasible. A second drawback is that channels in a flow reactor may become easily clogged slowing the processing and
25 requiring additional monitoring.

 Several articles have described ways to increase the efficiency of continuous flow processes. Articles including "The Combined Use of Microwaves and Ultrasound: Improved Tools in Process Chemistry and Organic Synthesis" from Chem. Eur.
30 Journal 2007 pages 1902-1909 by Cravotto et al. published in 2007; "Microwave Reactions Under Continuous Flow Conditions" from Combinatorial Chemistry and High Throughput Screening Volume 10, by Baxendale et al. published in 2007; and "Palladium-catalyzed Amination Reactions in Flow: Overcoming the Challenges of Clogging via Acoustic Irradiation" from Chemical Science Volume 2 by Buchwald et al.
35 published in 2011 discuss applications of various types of energies to flow processes to improve the efficiency of the process.

Several patent publications have put forth manners of applying certain types of energies to flow process systems to increase the efficiency of the reactions. The publications include US Patent Publication 2003/0049820 A1 titled "Apparatus and Method for Ultrasonic Treatment of a Liquid", published in the name of Chandler et al. on 13 March 2003 in which the use of ultrasonic energy to improve efficiency is disclosed; US Patent Publication 2006/0180500 A1 titled "Upgrading Petroleum by Combined Ultrasound and Microwave Treatments" in the name of Gunneman published 17 August 2006 which discloses treating an emulsion of oil and aqueous liquid with ultrasonic energy in a chamber and then applying microwave energy to the emulsion as the emulsion flows through a pipe; US Patent Publication US2009/0044700 A1 titled "Process for Producing Injectable Solutions by Degassing Liquids and the Use Thereof for Stabilizing Oxidation-Sensitive Substances" in the name of Dietlin et al. Published 19 February 2009 which discloses a method for degassing aqueous solutions of dispersions of oxygen sensitive phenolic substances by subjecting these liquid substances to ultrasonic energy and a second type of energy in large volume tanks during bulk preparation of the substances; Chinese Patent Publication 101319405 titled Production Method of TiO₂ Nanotube and/or TiO₂ Nanowhisker" in the name of Suzhou Institute of Nanotechnologies published 10 December 2008 which discloses a method of TiO₂ nanotube and/or TiO₂ nanowhisker production in which ultrasonic energy is applied to a solution followed by heating of the solution before applying further treatments to make the desired product; WO publication 2011/016744 titled "Device for Processing Aqueous Media in a Flow" in the name of Nikolavich published 10 February 2011 which discloses the use of ultraviolet and ultrasonic energy on an aqueous solution; US Patent Number 6,079,508 titled "Ultrasonic Processors" issued to Caza published 27 June 2000 which discloses a ultrasonic processor that may be used for dispersion, emulsifying, dissolving mixing or deagglomerating a material in a chamber with transducers affixed around the outer diameter of the chamber; and US Patent 7, 322, 431 titled "Advanced Ultrasonic Processor" issued to Ratcliff on 29 January 2008 which discloses an ultrasonic processor for the continuous processing of material that includes an enclosed chamber through which the material passes that has ultrasonic transducers placed on the walls substantially perpendicular to the inputs and outputs of the chamber to apply ultrasonic energy to the material that passes through the chamber.

Ultrasonic energy has also been used to reduce the clogging problem as described in WO 2011/023236 titled " Method For Preventing Plugging of a Continuous-Reaction Channel-System and Micro-Reactor for Carrying Out the

Method", published in the name of Lonza AG on 3 March 2011. In this document, a flow reactor is described that provides a custom manufactured ultrasound system that emits ultrasonic energy in a flow direction of the feed flow of chemical reagents. The ultrasound system provides a sonotrode to transmit and emit ultrasonic waves in the direction of the flow channel of the material. The ultrasonic waves enter the material and travel in the direction of the material flow to prevent the materials from clogging in the flow channel.

The above described publications do not provide adequate process for applying ultrasonic energy and other types of energies to a continuous flow process. The publications that do apply energies to continuous flow processes apply the energies sequentially. This reduces and limits the amount of improvement in efficiency that may be achieved in these described systems. Thus, those skilled in the art are continuously striving to improve the methods in which energies are applied to increase the efficiency of continuous flow systems.

Summary of the Invention

The above and other problems are solved and an advance in the art is made by a system and method for providing mixed mode energies during a continuous chemical flow process in accordance with this invention. A first advantage of a system and method in accordance with this invention is that the efficiency of a continuous chemical flow process is increased by simultaneously applying ultrasonic energy and a second type of energy to the process. A second advantage of a system and method in accordance with this invention is that the system and method may be scaled for larger scale production. A third advantage of a system and method in accordance with this invention is that the system and method do not require a carrier medium for the ultrasonic energy. A fourth advantage of a system and method in accordance with this invention is that the system can offer reaction controls that encourage more efficient mixing; and mass and heat transfer.

In accordance with embodiments of the system of this invention, an ultrasonic transducer is configured in the following manner. The transducer includes a housing plate. A piezo-ceramic disk is affixed to the housing plate. A coupling secures the housing plate in a position such that the housing is in direct contact with a flow reactor. Circuitry applies current to the piezo-ceramic disk to cause the piezo-ceramic disk to generate ultrasonic waves that are emitted through the housing plate towards the flow reactor. In accordance with some of these embodiments, multiple piezo-ceramic disks

are affixed to the housing plate and the circuitry applies current to each of the piezo-ceramic disks to generate ultrasonic waves that are emitted through the housing plate towards the flow reactor. In accordance with some of these embodiments, the ultrasonic waves are emitted in the absence of carrying medium. In accordance with
5 some of these embodiments, the ultrasonic waves are emitted by having the piezo-ceramic disk in direct contact with a horn; and the entire piezo-ceramic disk and horn system being affixed to the housing plate.

10 In accordance with some embodiments of this invention, the transducer is positioned on a first side of the flow reactor and a second apparatus for applying a second type of energy to the flow reactor is on a second side of the flow reactor. In accordance with some of these embodiments of the invention, the second type of energy is selected from a group of energies consisting of microwave energy, thermal energy, and ultraviolet radiation.

15 In accordance with embodiments of this invention, the method of generating a product from a continuous flow product is performed in the following manner. Reagents flow into a flow reactor. Ultrasonic energy and a second energy are simultaneously applied to the reagents in the flow reactor. The generated product then
20 flows out of the flow reactor. In accordance with some of these embodiments, the second energy is thermal energy. In accordance with some of these embodiments, the second energy is microwave energy. In accordance with some of these embodiments, the second energy is ultraviolet light.

25 In accordance with some embodiments of the method of this invention, the simultaneous application of the ultrasonic energy and the second energy is performed by transmitting ultrasonic waves from a first side of the flow reactor; and transmitting the second energy from a second side of the flow reactor. In accordance with some of these embodiments, the ultrasonic waves are transmitted from an ultrasonic
30 transducer that is in direct contact with the flow reactor. In accordance with some of these embodiments, the step of simultaneously applying the ultrasonic energy and the second energy is performed in the absence of a carrying medium.

Brief Description of the Drawings

35 The above and other features and advantages of systems and methods in accordance with this invention are described in the following detailed description and are shown in the following drawings:

Figure 1 illustrating a top view of an ultrasonic transducer in accordance with an embodiment of system of this invention;

Figure 2 illustrating a cross sectional view of an ultrasonic transducer connected to control circuitry in accordance of an embodiment of system of this invention;

Figure 3 illustrating a schematic side view of a continuous chemical flow process system in accordance with an embodiment of this invention; and

Figure 4 illustrating a flow diagram of a method for performing a continuous flow process in accordance with embodiments of this invention.

Detailed Description

This invention relates to a continuous flow chemical process. More particularly, this invention relates to simultaneously providing ultrasonic energy and a second type energy to a continuous flow chemical process to increase the efficiency of the process. Still more particularly, this invention relates to providing ultrasonic energy from an ultrasonic transducer in direct contact with a first side of a flow reactor in the absence of a carrying medium while providing a second type of energy from a second side of the reactor.

Figure 1 illustrates a top view of an ultrasonic transducer in accordance with an embodiment of system of this invention. Ultrasonic transducer 100 includes housing plate 105. Housing plate 105 has top surface 110. Sidewalls 115 extend upwards from a perimeter of top surface 110 at substantially a perpendicular angle to top surface 110. In some embodiments, a second plate may be affixed to the top ends of sidewalls 115 to form an enclosure. Preferably, top surface is made of glass, stainless steel, hastelloy, aluminum, or other such materials in order for top surface to improve the transmission of ultrasonic energy. Couplings 120 are affixed to sidewalls 115 or any other surface of housing plate 105 to secure housing plate 105 in place. As shown in figure 1, couplings 120 are wings extending out of the sides of sidewalls 115 and/or housing plate 105 that include openings for receiving screws, pins or the like to connect housing plate 105 to another structure. However, different coupling structures may be used without departing from this invention.

One or more piezo-ceramic disks 120-125 are affixed to top surface 110 of housing plate 110. As shown, piezo-ceramic disks 120-125 are arranged substantially evenly spaced apart in two rows and three columns. However, other configurations

may be used. The exact configuration depends upon the size and number of disks as well as the size and shape of housing plate 105. Each piezo-ceramic disk 120-125 is made of piezo electric material. The piezoelectric materials that may be used include, but are not limited to, metal zirconate titanates and ceramics. Preferably, each piezo-ceramic disk is approximately 35 – 55 mm in diameter and is 4 – 8 mm thick. However, the size of the diameter, thickness, and frequency will depend on the desired properties of each disk and the type of piezoelectric material from which the disk is made. In accordance to some embodiments, one or more of piezo-ceramic disks 120-125 may be affixed to a horn (Not Shown). The horn (Not shown) is then affixed to top surface 110 of housing plate 105 to aid in the generation of ultrasonic waves.

Figure 2 illustrates a cross sectional view of ultrasonic transducer 100 along line X-X' shown in Figure 1. Sidewalls 115 are shown in Figure 2 as extending upward from top surface 110 of housing plate 105. Furthermore, piezo-ceramic disk 123-125 are shown to be solid disk structures. Furthermore, each of piezo-ceramic disks 120-125 is shown to be connected to control circuitry 205. Control circuitry 205 applies current to each of the piezo-ceramic disks to cause each of piezo-ceramic disks 120-125 to vibrate and generate ultrasonic waves. Preferably, the ultrasonic waves generated are in a range of approximately 40 to 60 KHz. The generated ultrasonic waves pass from piezo-ceramic disks 120-125 through housing plate 105. Housing plate 105 changes to the frequency of the ultrasonic waves to a range from approximately 20 to 100 KHz. Preferably, each piezo-ceramic disk 120-125 has a frequency sweeping function of about 5 KHz, managed by the control circuitry. The ultrasonic waves are emitted from housing plate 105 into reaction chamber as discussed below with regards to Figure 3.

Figure 3 illustrates the components of a continuous flow chemical process system in accordance with this invention. One skilled in the art will recognize that other components not required to operate in accordance with the present invention are omitted. Continuous flow chemical process system 300 includes flow reactor 305. Flow reactor is a tube, pipe, or other enclosed chamber through which a liquid flows. One skilled in the art will note that flow reactor 305 is shown as a straight tube. However, flow reactor may have any type of configuration including, but not limited to a helical shape without departing from this invention. Flow reactor includes one or more inputs 320, 321 for flowing chemical reagents into flow reactor 305. Preferably, one or more of the reagents will be in liquid form to allow the reagents to flow through flow

reactor 305. Flow reactor 305 also includes one or more outputs 325 for allowing a product of the reactions between reagents to flow out of flow reactor 305.

Ultrasonic transducer 100 is secured in a position by couplings 120 in such a manner that at least a portion of ultrasonic transducer 100 is in direct contact with one or more portions of flow reactor 305. As ultrasonic transducer is in direct contract with flow reactor 305, the system does not require a carrier medium between the transducer and the flow reactor to carry or apply the emitted ultrasonic waves to the material in flow reactor 305. Furthermore, ultrasonic transducer 100 is on one side of flow reactor 305 and is positioned in such a manner that the emitted ultrasonic waves are applied to flow reactor 305 at an angle that is substantially perpendicular to the flow through flow reactor 305 when control circuitry 205 applies current to piezo-ceramic disks 120-125.

Second energy emitter 315 is positioned proximate flow reactor 305 to emit a second form energy to apply to the reagents in flow reactor 305. Second energy emitter 315 may emit one or more of microwave energy, thermal energy, and ultraviolet energy or any other type of energy desired. If microwave energy is emitted, the microwaves are preferably in a range of approximately 2.45 GHz. If thermal energy is emitted, the temperature is in a range from approximately 30 to 250 degrees Celsius. If ultraviolet energy is emitted, the ultraviolet light is preferably in a range from approximately 190nm to 800nm. As shown, second energy emitter 315 is position on a second side of flow reactor 305 opposite the side where ultrasonic transducer 100 is located. Thus, the second energy is applied at an angle that is substantially perpendicular to the flow of material through flow reactor 305. However, second energy emitter 315 may be placed in other positions and the emitted energy may be applied at another angle with respect to the flow of material without departing from this invention.

Figure 4 illustrates process 400 which is an embodiment of a continuous flow chemical process in accordance with this invention. Process 400 begins in step 405 by flowing the reagents into the flow reactor. In step 410, ultrasonic energy is applied to the reagents in the flow reactor. Preferably, the ultrasonic energy is applied from a transducer, such as transducer 100 described above, that is in direct contact with the flow reactor. Furthermore, transducer 100 is preferably on one side of the flow reactor and the ultrasonic waves are applied at an angle that is substantially perpendicular to the flow of material through the flow reactor.

Step 415 is performed substantially simultaneously to step 410. In step 415, a second type of energy is applied to the reagents in the flow reactor. In accordance with various embodiments, the second type of energy may be microwave, thermal, or ultraviolet energy. Preferably, the second type of energy is applied from an emitter on a second side of the flow reactor that is opposite from the ultrasonic transducer. In step 420, the resulting product is flowed out of the flow reactor and process 400 ends. One skilled in the art will recognize that other steps may be performed during process 400 that are omitted, as the steps are not part of the steps of the process in accordance with this invention.

The above is a description of a method and system for providing ultrasonic and at least one other type of energy to a continuous flow chemical process. From the above description, it is envisioned that those skilled in the art will design other method and systems that infringe on this invention as set forth in the following claims.

What is claimed is:

1. An apparatus for applying ultrasonic energy to a flow reactor comprising:

5 a housing plate;
a piezo-ceramic disk affixed to said housing plate;
a coupling for securing said housing plate in a position wherein said housing plate is in direct contact with a flow reactor; and
circuitry for applying current to said piezo-ceramic disk to cause said
10 piezo-ceramic disk to generate ultrasonic waves that are emitted through the said housing plate towards said flow reactor; and
wherein said ultrasonic waves are emitted in the absence of a carrying medium.

15 2. The apparatus for applying ultrasonic energy to said flow reactor of claim 1 further comprising:

a plurality of piezo-ceramic disks including said piezo-ceramic affixed to said housing plate wherein said circuitry for applying said current applies said current to each of said plurality of piezo-ceramic disks to generate ultrasonic waves
20 that are emitted through the said housing plate towards said flow reactor.

3. The apparatus of claim 1 further comprising:

a horn having a first end affixed to said piezo-ceramic disk and a second end affixed to said housing plate to affix said piezo-ceramic disk to said
25 housing plate.

4. The apparatus of claim 1 wherein said apparatus is on a first side of said flow reactor and a second apparatus for applying a second type of energy to said flow reactor is on a second side of said flow reactor.

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5. The apparatus of claim 4 wherein said second type of energy is selected from a group of energies consisting of microwave energy, thermal energy, and ultraviolet radiation.

35 6. A method for generating a product using a continuous flow process comprising:

flowing a plurality of reagents into a flow reactor;
simultaneously applying a ultrasonic energy and a second energy to said
plurality of reagents in said flow reactor; and
flowing a product generated from said reagents out of said flow reactor; and
5 wherein said ultrasonic energy is applied in the absence of a carrying medium.

7. The method of claim 6 wherein said second energy is thermal energy.

8. The method of claim 6 wherein said second energy is microwave
10 energy.

9. The method of claim 6 wherein said second energy is ultraviolet light.

10. The method of claim 6 wherein said step of simultaneously applying
15 said ultrasonic energy and said second energy comprises:
transmitting ultrasonic waves from a first side of said flow reactor; and
transmitting said second energy from a second side of said flow
reactor.

20 11. The method of claim 6 wherein said step of simultaneously applying
said ultrasonic energy and said second energy comprises:
transmitting ultrasonic waves from a ultrasonic transducer that is in
direct contact with said flow reactor.