Microbubble Generation

William B Zimmerman[1*], Václav Tesař[2], Simon Butler[1], HC Hemaka Bandulasena[1]
[1]Department of Chemical and Process Engineering, University of Sheffield, Mappin Street, Sheffield S1 3JD, United Kingdom
[2]Institute of Thermomechanics, Academy of Sciences of the Czech Republic, Prague, Czech Republic.
[*]Corresponding author. Email: w.zimmerman@shef.ac.uk

Abstract
In general, there are three ways of generating microbubbles. The most common class uses compression of the air stream to dissolve air into liquid, which is subsequently released through a specially designed nozzle system, to nucleate small bubbles as potentially nanobubbles, based on the cavitation principle. These bubbles subsequently grow into much larger bubbles through the rapid dissolution of the supersaturated liquid. The second class uses power ultrasound to induce cavitation locally at points of extreme rarefaction in the standing ultrasonic waves. The third class uses an air stream delivered under low offset pressure, and airs to break off the bubbles due to an additional feature, whether it be mechanical vibration, or flow focussing, or fluidic oscillation. Conventional air diffusers rely on the structure of porous material for the nozzles to generate small bubbles, but fluidic oscillation in general promises to break off the forming bubble while it is still a hemispherical cap – the smallest shape for which bubble formation from a pore is likely to occur given the strong adverse affect of surface tension at higher curvatures. The first two classes of microbubble generation are usually associated with high power densities and power consumption by either the compression or ultrasonic treatment. The third class should have the lowest power consumption, provided it achieves the application targets of bubble size distribution, air phase holdup, and bubble dispersion. In this paper, recent patents in microbubble generation are categorized into the first and the third classes above. The subject area is reviewed for its importance in several fields of application, particularly generalized flotation processes and bioreactor treatments.

1. Introduction

1.1 The challenge of small bubble generation.
Naively, one would expect that when blowing small bubbles through an aperture, making the aperture as small as possible would be sufficient for generating bubbles correspondingly small . But there are a number of reasons why this does not suffice. For instance, when a bubble is formed from a single aperture, the liquid attached to the perimeter of the aperture serves as an "anchor" as the wetting force attaches the growing bubble to the solid surface. Unless this anchoring force is disrupted, the bubble will grow until the buoyant force on the bubble (which is proportional to its volume) exceeds the anchoring restraint on the bubble (typically proportional to its contact perimeter), and therefore breaks off. In this low pressure offset scenario, the force balance usually breaks off the bubble at a size an order of magnitude larger than the diameter of the aperture. Furthermore, the wetting properties of the solid surface are extremely important. Should the bubble contact the surface over a larger region

than the aperture perimeter, if the solid surface is hydrophobic, the gas phase of the growing bubble will form a second anchor force with the solid surface over a wider area, increasing the buoyant force and thus bubble volume required to overcome it. If the surface is hydrophilic, then this attractive force is absent.

A second reason for forming larger bubbles from small apertures is polydispersity of bubble sizes and irregularity of the spacing between bubbles leading to quick coalescence of the bubble cloud. Even if small bubbles are formed, then coalescence can rapidly reduce the benefit.

The third reason for not forming small bubbles from small apertures is channelling in a nozzle bank of pores or through a porous ceramic material. This is described in Figure 1. The largest bubble that forms then provides the path of least resistance, preferentially growing against all other bubbles in the parallel percolation process in a nozzle bank or porous ceramic material.

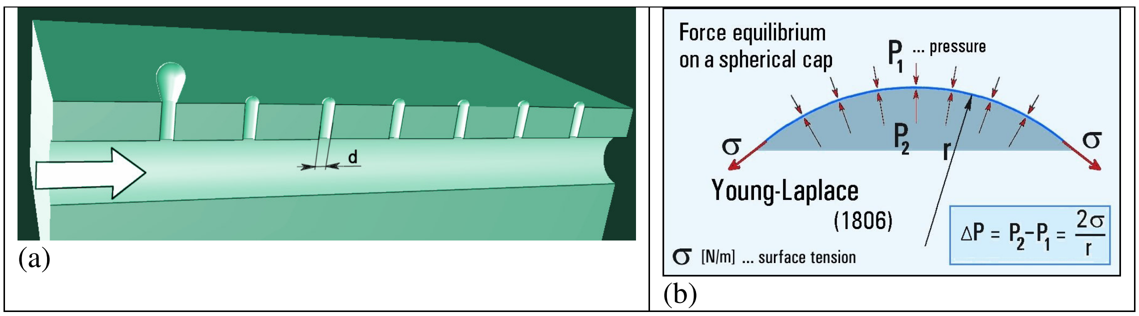


Figure 1 Instability of parallel percolation. (a) If one of the bubbles increases beyond the hemispherical shape, its growth becomes easier: air entering overcomes a lower pressure difference ΔP. It grows at the expense of the other bubbles. (b) The basic reason: Young-Laplace surface tension law. Pressure difference across the air/water surface is inversely proportional to the curvature radius r.

Given these difficulties of forming small bubbles from air flow through a nozzle or nozzle bank, high power density techniques have been developed. In general, there are three ways of generating microbubbles. The most common class uses compression of the air to dissolve air into liquid, which is subsequently released through a specially designed nozzle system, to nucleate small bubbles as potentially nanobubbles, based on the cavitation principle. These bubbles subsequently grow into much larger bubbles through the rapid dissolution of the supersaturated liquid. The second class uses power ultrasound to induce cavitation locally at points of extreme rarefaction in the standing ultrasonic waves.

The third class uses an air stream delivered under low offset pressure, and aims to break off the bubbles due to an additional feature, whether it be mechanical vibration, or flow focussing, or fluidic oscillation. Conventional air diffusers rely, fruitlessly, due to the instability described in Figure 1, on the structure of porous material used for the nozzles to generate small bubbles, but fluidic oscillation in general promises to break off the forming bubble while it is still a hemispherical cap – the smallest shape for which bubble formation from a pore is likely to occur given the strong adverse affect of surface tension at higher curvatures. The first two classes of microbubble generation are usually associated with high power densities and power consumption by either the compression or ultrasonic treatment. The third class should have the lowest power consumption, provided it achieves the application targets of bubble size distribution, air phase holdup, and bubble dispersion. In this paper, we review recent patents in microbubble generation, giving substantial exposition to a new technique for generating microbubbles with low power density – just offset pressure – linked with fluidic oscillation to overcome the three major difficulties highlighted above creating bubbles of the scale of the aperture diameter, rather than an order of magnitude higher.

1.2 The benefit of microbubbles.

In many instances, miniaturization is sought for the purposes of convenience – smaller devices are more portable, or require fewer uses of resources. Why would microbubbles be a benefit? In the case of consumer products which use microbubbles, it might be the texture of the product (frequently a foam) is perceived better; possibly the processing of microfoams is better – with lower viscosity or better rheological features. Separations processes such as for minerals or biotech materials might be enhanced, or for the flotation or air-lift of wastes or oil recovery. A common thread among the benefits of microbubbles is in their transport behaviour – mass, momentum, and heat transport at the interface of microbubbles is influenced by the interfacial surface area. Figure 2 depicts the key feature of high surface area to volume ratio.

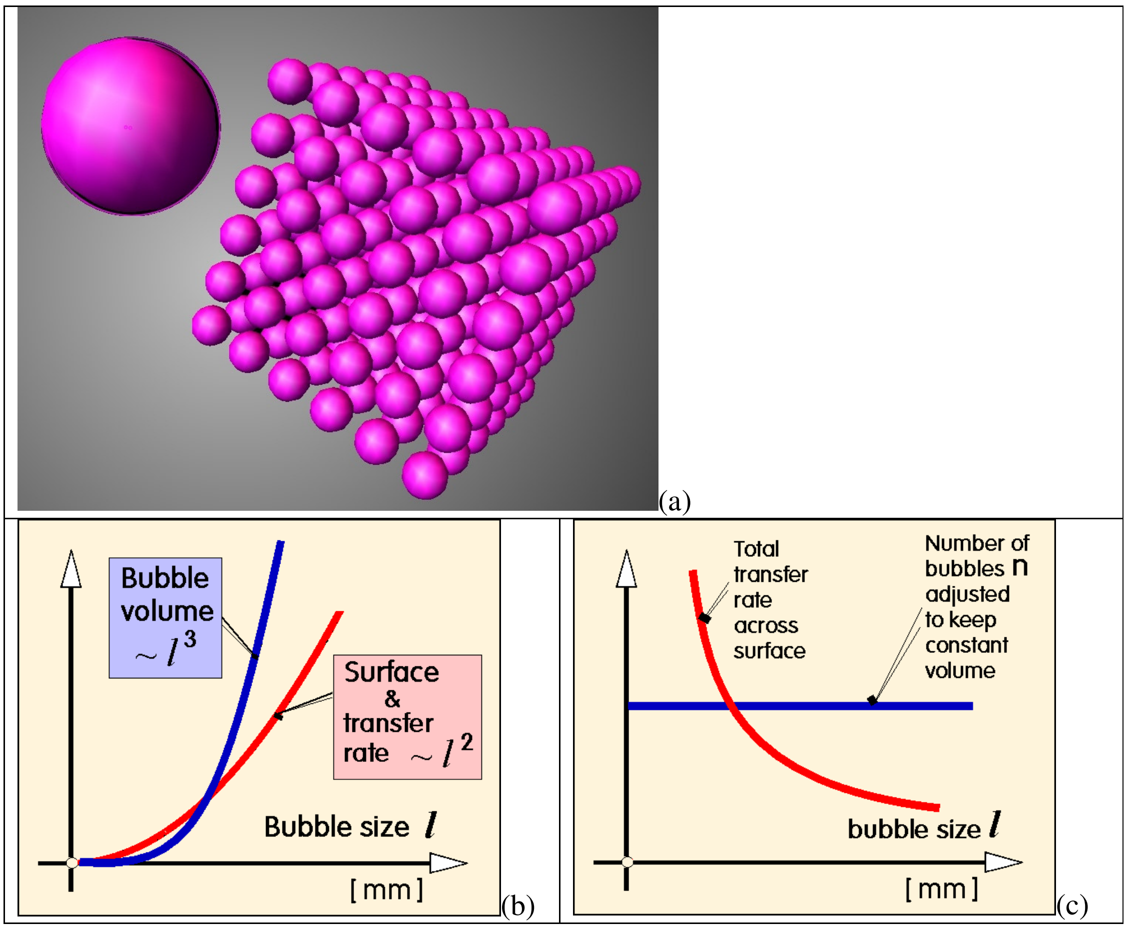


Figure 2. The transfer benefit of microbubble generation. (a) Division of a volume into n smaller, equally sized objects, productes additional surface area that scales with the cube root of the dividing number. (b) For a single bubble, the surface area and transfer rate scale as the square of the bubble size *l*, but bubble volume scales with its cube. (c) Therefore, the total transfer rate with the number of bubble adjusted to keep the air phase volume constant, scales inversely with the bubble size *l* –smaller bubbles lead to greater transfer.

The argument given in Figure 2 for the benefit in transfer efficiency is typified by the common chemical engineering phenomenological description of interphase mass transfer flux J (moles per second):

$$J = K_l a \left(c_g - c_l \right) \quad (1)$$

where K_l is the mass transfer coefficient (units of velocity), a is the interfacial area, and c_g and c_l are molar concentrations. There is a direct analogy to heat transfer flux Q where the roles of the concentrations are played by temperature, i.e. Newton's Law of Cooling. What is not so intuitive, however, is that there is a similar transfer effect for momentum, where the role of J is taken by the force F in the vertical direction induced by velocity changes in the horizontal direction, which follows from Newton's law of viscosity:

$$F = -\mu a \frac{\partial w}{\partial x} \quad (2)$$

The interpretation of equation (2) is that the momentum transfer by a cloud of rising bubbles increases with the surface area of the cloud dragging more of the ambient liquid with it than a larger bubble with less surface area. This feature opposes the more intuitive feature that smaller bubbles rise less quickly than a single larger bubble that matches its volume. The rise velocity is a linear effect with bubble size as shown in Figure 3.

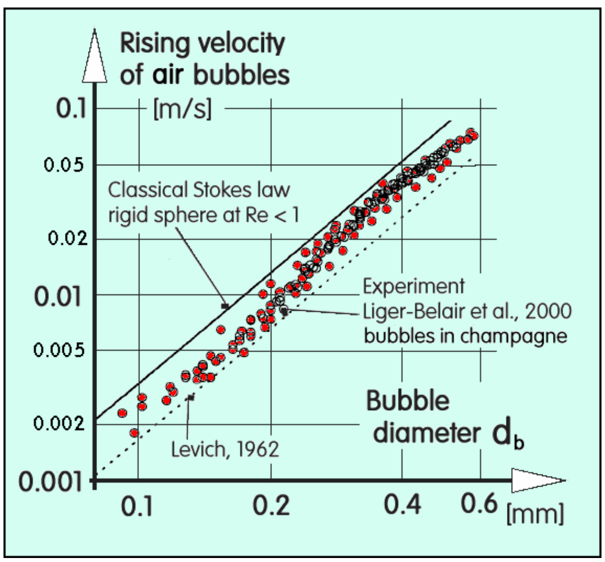


Figure 3. The rise velocity of bubbles as shown theoretically (Levich) and experimentally. As transfer rates are shown to increase inversely proportionally to bubble size, but the velocity difference rises proportionally to bubble size, one would expect momentum transfer by the cloud of bubbles to be relatively constant. The total amount of momentum transferred, however, should be larger by the smaller bubbles due to the finite height of the liquid layer. The slope of the above graph shows that bubbles three fold smaller stay in the liquid ten fold longer, thus having longer time to transfer the same momentum rate.

The "stirring effect" by a rising cloud of small bubbles, according to Figure 2, exceeds that of the passage of a single larger bubble if nonlinearity is neglected (Stokes regime). The canonical chemical engineering application for such microbubble dispersal is called surface aeration, frequently the most important process in bioreactors and fermentors (see Grammatika and Zimmerman, 1999).

The paper is organized as follows. In section 2, the two classes of high power consumption formation of microbubbles – compression followed by release, and power ultrasound – are discussed, with an eye to the high value added products from the processes that utilize microbubbles. In section 3, the third class of low power consumption microbubble generation is discussed. In recent patents, this class has included the use of porous materials, flow focussing, and fluidic oscillation actuated microbubble generation. In section 4, speculations about current and future trends are discussed, particularly with regard to the future promise of miniaturization and power efficiency.

2. Applications and high power nucleation of microbubbles.

Applications of small bubbles

As the Introduction made clear the generic benefits of microbubbles for transfer processes, one of the key application areas for microbubbles are generalized flotation processes (see e.g. Grammatika and Zimmerman, 2001) for which microbubbles collect on larger particle, forming a floc which is lense dense than the surrounding fluid, thus rising due to buoyancy. The larger particle may be a solid waste or an oil droplet in the common application area of dissolved air flotation.

Generating small bubbles is important in many industrial applications. Low density particulates are separated from potable water and wastewaters by small bubbles produced by either dissolved air flotation or dispersed air flotation. Also biological treatment of wastewater requires aeration systems with high oxygen transfer efficiencies. Electroflotation is commonly used in mineral industry to separate fine particles from solutions and oil industry to separate oil-water emulsions. In adsorbing colloid flotation, heavy metals are removed by fine microbubbles. In yeast industry, bio mass production mainly depends of oxygen transfer efficiency of the aeration system. In the production process of some biopharmaceutical products, bioreactors require fine bubble aeration systems.

For biomolecular separations, very small microbubbles, termed aphrons, have shown particularly useful extraction properties. Lye and Stuckey (2001), for instance, report large mass transfer coefficients in the extraction of erythromycin using colloidal *liquid* aphrons, an emerging technique for the recovery of microbial secondary metabolites, such as antibiotics in pre-dispersed solvent extraction (PDSE) processes. More typically, microbubbles at the micron scale form colloidal *gas* aphrons are used for gas phase extraction, such as for lactoferrin and lactoperoxidase from sweet whey (Fuda et al. 2004). Protein separations are typically by interfacial affinity, rather than phase transfer. For instance, Noble et al. (1998) showed that protein affinity to foams was largely chemical interactions, but to gas aphrons, electrostatic and hydrophobic interactions dominated. Oil recovery could benefit from gas-lift technology if sufficiently small microbubbles can be generated in situ downhole (Guet and Ooms 2006), driving such innovations as new pump cavitation mechanisms (Samuel and Saveth, 2006).

Burns et al.(1997) compared three common bubble generation mechanisms namely , DAF, electroflotation and air spraying, for bubble size and surface area produced per time as a function of power input to the system. The study revealed that DAF produced the narrowest bubble size distribution with largest average bubble size, but promised highest surface area/unit time/power compared to other two methods. Also

DAF experiments showed slight improvement of bubble size with increasing pressure requiring 60-90 psi to produce 46 to 57.5 μm bubbles.

Makuta et al. (2006) reported an ultrasonic method to produce microbubbles of uniform diameter from 4 to 15 μm at a constant periodic rate. A bubble protruded at the tip of a needle was oscillated by ultrasonic waves and projections were formed by the surface waves to give a continuous stream of tiny bubbles. The stability of bubble generation was limited by the gas viscosity to approximately 20.0 μPa s in a highly viscous liquid of kinematic viscosity between 5 and $100mm^2\ s^{-1}$ and surface tension between 20 and 34 mNm^{-1}.

In all these generalized flotation processes, the high value added of the substance, typically an economically viable product, to be separated justifies the use of high power techniques to generate microbubbles. But given that high power is required to generate microbubbles with conventional techniques of nucleation from compression or power ultrasound, all of the processes mentioned above would benefit from inventions and innovations that are energy efficient, while still achieving target bubble sizes and hold-ups.

Several recent patents fall into this category of using high power compression effects to dissolve air and then, upon releasing the pressure, nucleating or otherwise creating small bubbles. For example, In US20070108640, Takahashi et al. (2007) claim a device which first dissolves air under pressure and then draws, using suction through a mixer unit, the dissolved air enriched water and a stream of air, using a nozzle and a bubble generating cartridge. The key feature of this is that the dissolved air enriched water stream becomes supersaturated and requires less pressurization overall in creating microbubbles. This patent is specific for a hair-washing unit, and therefore can be termed a process patent – the fundamental mechanisms may well have been known already in, say, wastewater treatment, but then have never been applied, in such an embodiment, for a hair washing system.

In US20070119987, Vion (2007) demonstrates a three staged compression system with a prerelease stage with modest decompression, a nozzle release stage, and a transition chambre which brings the pressure to saturation, before a final outlet tube which confines cavitation and limits the reattachment to the tube walls, thus opposing initial coalescence. By staging the releases, this technology should reduce the initial compression required so that energy costs for supersaturation of the dissolved air are minimized. This is a promising technology for retrofitting existing dissolved air flotation plant for separating solids and immiscible oily effluent.

In US7199085, Rea et al. (2007) do not claim, per se, a microbubble generation methodology, but rather a recipe for creating a colloidal liquid and gas aphron composite fluid which has a long enough “shelf-life” to be pumped downhole, which in particular is valued for its thixotropic rheological properties. Given our discussion at the beginning of this section of the value of microbubbles for enhanced oil recovery, one possible delivery method would be through a sufficiently stabilized composite liquid with colloidal emulsion and or aphron constituents which would release the microbubbles as the complex liquid stability fails in the oil sands. This latter claim is not made in the patent, but rather it does not limit the range of downhole activities for which such a complex fluid could be applied.

In US7214508, Hucklenbroich and Mueller (2007) do not actually claim a microbubble generation technique. They describe a technique for cell lysate coarse level separation that involves compression of the lysate which forms flocs, and then decompression which results in a coarse phase separation between the flocs and a lower, clear liquid layer. This clarification technique appears rapid, and it is difficult to imagine that the compression-decompression cycle does not nucleate microbubbles that integrate into the organic material flocs, thus improving the buoyancy difference between the floc and clear liquid phases. Analysis suggests that this separation is a microbubble mediated flotation process.

In WO/2007/068446: Eichler et al. (2007) describe an integrated biogas production and separation process, where the biogas generated is combined with the process liquid in a microbubble generation stage to use the biogas enriched bubbles in a recycle stage to separate the biodegradable material, by flotation, from which it was formed. As the major idea of this patent is process integration – simultaneous use of the product in its own processing – it is not necessary to specify any novel microbubble generation scheme to achieve this. Consequently, one would expect that in the first instance, implementation has been done with convention compression methods for dissolved gas flotation.

3. Low power *microfluidic* microbubble generation.

In this section, three low power methods of generating microbubbles are discussed. In general, the use of porous ceramic materials or slits in membranes is common in aeration used in wastewater treatment. Flow focussing, particularly in microdroplet formation, is known as a route to create microbubbles/microfoams as well. Finally, fluidic oscillation has recently been demonstrated as a methodology for producing microbubbles. All three methods of these methods are implemented by geometric features with characteristic dimensions on the microscale. The latter two are likely to be engineered microfluidic devices (see Zimmerman, 2005), whereas the random porous nature of porous materials is likely to be a property of the material for which it was selected.

Porous materials.

In US20070114176, Yamasaki et al. (2007) introduce a porous wood charcoal material prior to a diaphragm in a diffuser system, which has the interesting biochemical property of activating microorganisms on the wood charcoal which digest wastewater faster. In this case, the micro/nano bubble generation mechanism is subsidiary to the microorganism activation as the economic driver. If the sole goal were microbubble generation, then porous materials generally suffer from the difficulties discussed in the Introduction. However, porous materials do create a range of bubble sizes, with sufficiently high population of the microbubble regime to achieve the desired activation of the microorganisms for digestion.

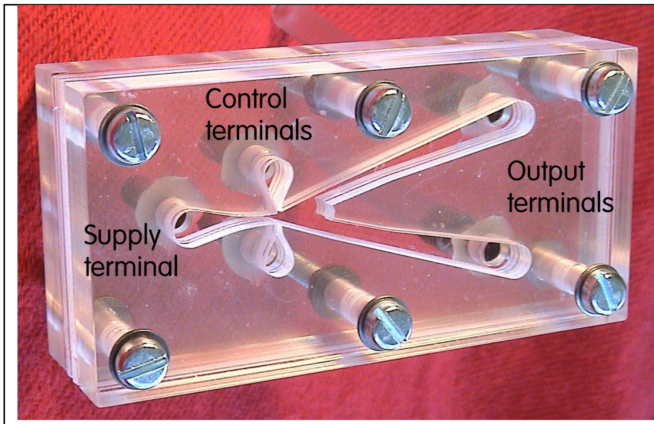
Flow focussing.

In US20070114183: Lee and Muir (2007) focus on the use of microbubbles in separating dispersions of immiscible liquids, such as oil and water, which is conventionally done by dissolved air flotation with high power usage, but is done with low power usage by clarification tanks (lowest) or by structured packings or parallel plate packs, without the use of microbubbles. Combining the use of interesting internal contacting patterns in their vessel to help induce buoyant segregation and microbubble dispersal, with microbubbles generated by flow focussing, this patent ingeniously joins the major features of the two extreme conventional techniques (high energy dissolved air flotation and low energy parallel plate packs) to achieve a low energy and relatively quick, active separation.

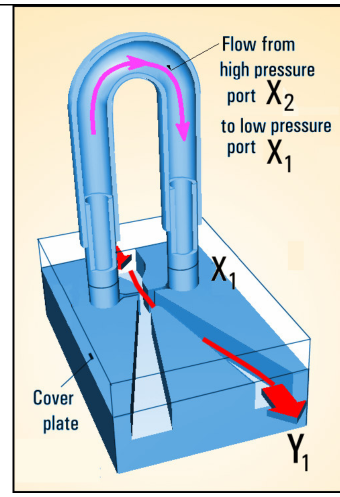
In US20070095937, Noguchi and Chuang (2007) demonstrate a series of devices which use restrictions in flow, partitions in flow, flanges, and entrainment of air from recesses in the walls to generate microbubbles laden fluid, which are then launched against bouncing surfaces. They claim particular configurations, which achieve the microbubble formulation. Flow focussing in general is know for foam (microbubbles) and dispersion (microdroplet) generation (see Lorenceau et al. 2006), but any particular configuration and process for which flow focussing has not yet been shown to generate dispersions of microbubbles or microdroplets is fair game for a process patent based on novel usage.

Fluidic oscillation.

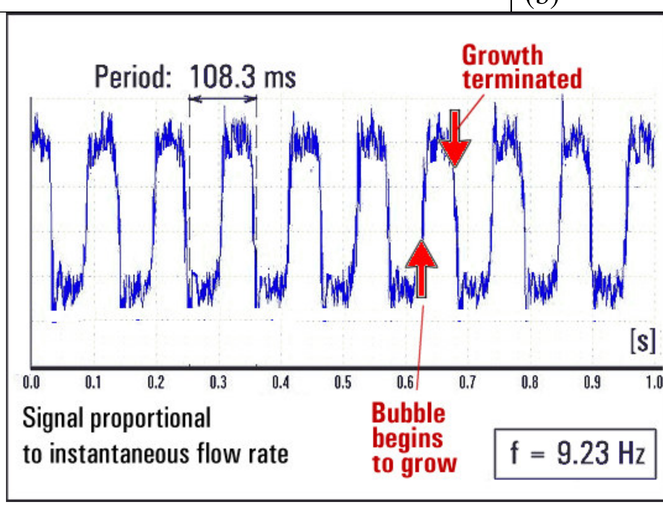
Prior art in microbubble generation included a number of systems that cause mechanical vibration – oscillating of solid bodies and their surfaces – but to our knowledge, only in UK0621561, Tesař and Zimmerman (2006), it is the air that is oscillated, particularly by its taking a completely different spatial path to alternative exit nozzles. The device performing the vibration of the air flow is a fluidic oscillator (or a bank of oscillators). It handles a continuous flow input, switching it into one of two output channels with a regular frequency. Typically, oscillator is a no-moving part amplifier based on the Coanda effect (see Tesař, 2007), Fig. 4a, provided with a feedback loop. Although in UK0621561, Tesař and Zimmerman (2006) claim not to be limited to any particular implementation of a fluidic oscillator as a methodology for actuating microbubble formation from a hemispherical cap formed from an aperture or nozzle bank, the oscillator of Tesař et al. (2006), Fig. 4b, was used in demonstrator experiments for applications to wastewater treatment (Hu 2006), yeast growth (Zhang, 2007), and oil recovery (Varma, 2007).



(a)



(b)



(c)

Figure 4. Fluidic oscillator used in experiments. (a) The amplifier made as cavities in a stack of laser cut Perspex plates (b) In an oscillator, the feedback loop connects the two control terminals of the amplifier. The pressure difference between them generates the flow in the loop than causes the main air flow from the supply terminal to switch from one Coanda-effect attachment wall to the opposite one. This is repeated periodically at 1-100Hz depending on the length of the feedback loop. (c) Flow rate time history for the fluidic oscillator connected to a parallel percolation nozzle bank with apertures of 600μ diameter. The bubble growth is limited by the duration of the oscillation pulse. Growth stops while all generated bubbles are still smaller than the hemispherical stability limit (see Figure 1).

This fluidic oscillator was connected to two identical parallel percolation nozzle banks, with fixed size apertures, Fig. 5a. One set of nozzle banks was used for aeration tests to find the oxygen transfer efficiency with apertures of 600 micron diameter (Hu, 2006). Oscillation, with careful selection of the materials for construction to avoid strong hydrophobic forces anchoring bubbles, and selection of the orientation with flow, lead to generation of small bubbles and order. Specific measures were necessary to ensure forced separation of the not yet "mature", small bubbles from the percolation orifices. One layout, Fig. 5b achieves the separation by the action of water pulses synchronised with the air pulsation. Another layout, Fig. 5c, uses alternating liquid and air columns moving in a reciprocating manner in the manifold.

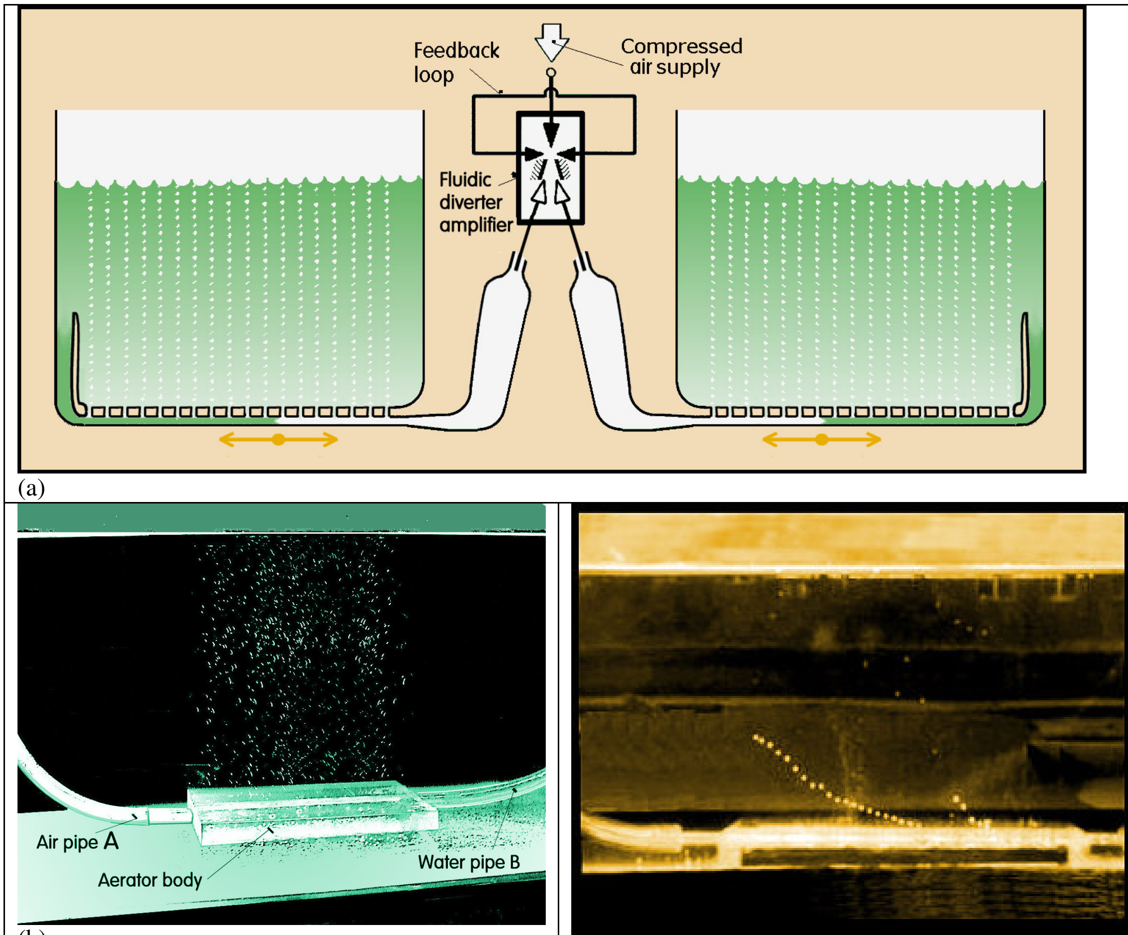


Figure 5. Microbubble generation from nozzle banks with fluidic oscillation. (a) Artist's conception of the dual outlet bank feed by a single fluidic oscillator. (b) Image of fine mist microbubbles generated by opposing nozzle banks for air and water flow. The air pulse separates the bubbles that would otherwise relain attached to the exit apertures (c) Slower operation in oil with air microbubbles generated. In this configuration, the air is injected into the manifold leading to the nozzles and its pulsation leads to alternating filling the nozzle with air and liquid. Clearly shown is the fact that the microbubbles are generated with even spacing. Hydrodynamics of viscous flow ensures that the column of uniformly spaced, monodisperse rising air bubbles is stable in its rise rate – no bubble overtakes its predecessor and thus the cloud is non-coalescent in theory (Crabtree and Bridgewater, 1969). In practise, this approximation appears to hold well in (b).

The 600 micron nozzle bank showed the formation of nearly mono-dispersed, uniformly well spaced clouds of microbubbles, nearly all submillimetre size. Oxygen transfer efficiency rates were shown to be 8-fold higher with the fluidic oscillator operating than without (Hu, 2006). A subsequent study found that yeast cultures had exponential growth rates at least 20% higher with the fluidic oscillation than without (Zhang, 2007). Implementation of nozzle banks in microchips with oriented outlets of 60 micron diameter also demonstrated bubble clouds with sizes of the scale of the aperture diameter when actuated by air flow through the fluidic oscillator with a range of offset pressures from 0.1 to 1.5 bar, but approximately 8-fold larger without the fluidic oscillation (Varma, 2007). The latter result suggests that high hold-ups can be achieved given the slow rise time of the microbubbles and low coalescence rates, with the potential for low power dissipation.

4. Current and future developments

Microbubble generation is a hot topic, but it is possible that the future applications are even smaller. Cameron (2005) reports in the trade press about the development of nanobubble generation in the labs of Masatoshi Takahashi at the Institute for Environmental Management Technology at the National Institute of Advanced Industrial Science and Technology (AIST). Takahashi's 200 nanometer diameter bubbles have been reported to have no propensity to rise and are stable for months, having been formed by a process involves "physical stimulation leading to violent adiabatic collapse." Insufficient information has yet been revealed for primary disclosure to validate the claims, which, however, are striking concerning the possible metabolic and medical uses of nanobubbles, which carry more of the advantages with regards to transfer efficiency than microbubbles, but potentially others as well. Nanoparticles are supposedly highly reactive, which suggests that nanobubbles will make for excellent interfacial reactions in heterogeneous catalysis. Mass transfer will be dominated by interfacial interactions, which are different with different chemical species. Deshpande and Zimmerman (2005) have already demonstrated that such asymmetry in mass transfer kinetics can be exploited in optimizing heterogeneous chemical reactors.

In this review, we categorized microbubble generators into high power consumption processes which are tolerated by the value of the product produced, and low power consumption approaches that attempt to generate microbubbles using little more than the offset pressure driving the air flow to form the microbubbles. Given the economic drivers in industries using high power consumption, the current trends in patents are to incrementally cut the usage of power with improved designs and design concepts.

The major challenge is to shift from high power consumption regimes to low power consumption methodologies without sacrificing the desired microbubble size and distribution through coalescence or channelling instabilities. Fluidic oscillation is one approach that promises minimal power losses and low power consumption with desirable bubble size properties and nearly uniform spacing to oppose coalescence. The approach is not yet sufficiently characterized to know if the concept will work with all types of nozzles used in microbubble formation, whether it scales up to large sparging requirements of fermentors and bioreactors, or scales down to microfluidic bioreactors, as the combination of oscillation frequencies and flow rates must be sufficient to limit the bubble growth to the hemispherical cap and to interact favourably with mechanisms to detach the hemispherical cap semi-formed bubble by overcoming the anchoring force subcritical buoyancy. These studies, as well as potential application areas, are underway.

Many of these questions must simply be answered by experimentation. Multiphase flow and interfacial phenomena at three phase boundaries are insufficiently developed to shed much light on the questions of transient oscillation in three-dimensional, complex geometries.

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