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Sonochemistry: principles, effects and good laboratory practices

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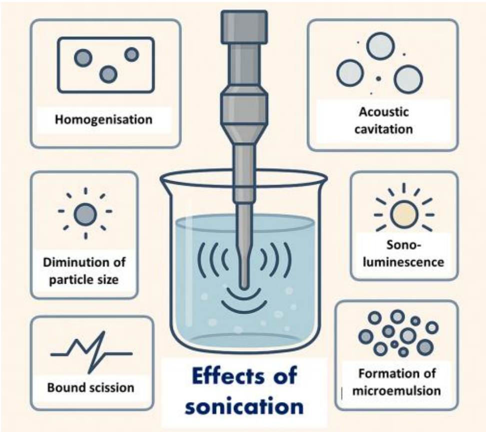
ABSTRACT. Sonochemistry relies on the use of sound, and more specifically ultrasound, to trigger or improve a chemical process. Ultrasonic waves of high frequency, ranging from 20 kHz to several MHz, induce different physical and chemical phenomena that can accelerate a chemical reaction and/or alter its mechanism and, ultimately, improve the efficiency of a chemical procedure. Such technique is used in many fields of chemistry including, for instance, organic chemistry, nanotechnologies, or material science and have various applications for nanomaterials syntheses, extraction processes, polymerisation, wastewater treatment, drug delivery, A few industrial examples will be provided. The versatility of sonochemistry makes it a valuable tool for green industrial processes.

This technical note aims at providing a brief introduction to sonochemistry for persons totally new to the field and to explain how physical waves can impact a chemical medium. It will explain briefly the different specific effects that will be induced by the propagation of the waves, both on the medium (physical effects) and on the chemicals (chemical effects). Finally, it will present different points concerning good laboratory practices regarding sonochemistry based on the type of ultrasonic generator used as well as the key information that must be provided whenever an ultrasonic process is used in chemistry.



1. Introduction

1.1. Introduction to sonochemistry

Ultrasound, a sound which cannot be heard by the human ear, is widely known in the society for being emitted by different animals such as bats or dolphins for orientation or communication as well as for traditional applications such as sonar or foetal imaging. Sound is generally divided in different categories based on its frequencies, in hertz (Hz), and where it stands compared to what a human being can hear:

- **Infrasound**, which frequencies are below the lower limit of human audibility (i.e. below 16 Hz)
- **Audible sounds**, which frequencies range in between 16 and 16.000 Hz
- **Ultrasounds**, with frequencies above 16 kHz, consisting in so-called power ultrasound (16-100 kHz) and diagnostic ultrasound (5 to 10 MHz)

Generally, sonochemical applications are performed within a range of frequencies between 16 kHz and 2 MHz. Figure 1 represents the classification of sound as a function of its frequency.

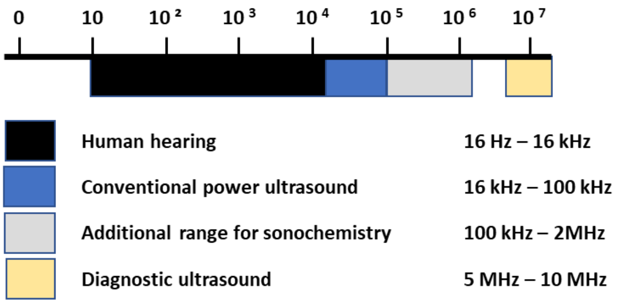


Figure 1. Division of sound as a function of its frequency in hertz (© Certech).

Ultrasound, as any sound, consists in mechanical and elastic waves that propagate through supporting fluids, such as liquids, gas, or solids. It possesses all the traditional properties of such waves in terms of propagation, diffusion attenuation, and reflection. While progressing through a viscoelastic medium, the wave induces a vibrational motion of its molecules, the latter returning to their initial position thereafter. In other terms, when propagating in a fluid such as water, the wave moves along water, but the molecules of water oscillate around their initial position. By doing so, the ultrasonic waves generate, at a given instant, zones of compression (where the density of molecules is higher) and zones of rarefaction (where less molecules are present). Periodically, as the ultrasonic wave is sinusoidal, zones of compressions become zones of rarefaction (and vice versa); the associated variation of pressure following the formula $P_t = P_{max} \sin(2\pi t + \varphi)$, where P_{max} represent the maximal pressure, t the time and φ the phase. Figure 2 shows a representation of an ultrasonic wave as well as the physical effect it involves on a medium, with λ being the wavelength.

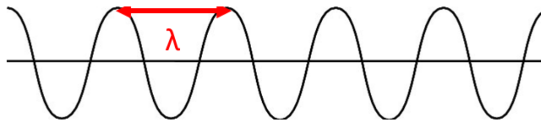


Figure 2. Representation of a sonic wave, (© Certech).

These variations are at the origin of a phenomenon known as acoustic cavitation. When the local pressure in the liquid is low enough – below the vapor pressure of the liquid, which is feasible when ultrasounds of high intensity are applied – the liquid starts to boil locally and bubbles of gas start to form in these zones of low pressure (or can be generated from dissolved gases). These cavitation bubbles will subsequently grow via rectified diffusion, meaning that a small amount of additional gas will enter the bubbles during the expansion phase. The cavitation bubbles will then grow up to a critical size, which is inversely proportional to the frequency of the ultrasonic wave – with an approximation of $R \approx 3/f$ with f being the frequency of the ultrasonic wave (in Hz) and R the radius of the cavitation bubble (in m). Once this size is obtained, two different phenomena can occur, known as stable cavitation and transient cavitation. Stable cavitation occurs for low variation of pressure,[1] meaning at low ultrasonic intensity (1-3 W/cm²).[2] In these conditions, the size of the bubbles oscillates in a sinusoidal way around an equilibrium with a low amplitude. Such cavitation will generate pressure waves that can lead to microstreaming around the bubbles. Transient cavitation occurs at a high ultrasonic intensity (> 100 W/cm²).[1] In the case of transient transition, the linearity of the oscillation is broken and, after a few acoustic cycles, the bubbles collapse generating a large amount of energy in their vicinity. Such a collapse is a particularly remarkable phenomenon as it can lead to extremely high temperature and pressure, that have been estimated to be around 5000K and 1000 atm respectively (in water and for 20 kHz ultrasound). This violent collapse is at the origin of the theory of the "hotspot", generating a large range of consequences that are the source of sonochemistry,[3,4,5] Figure 3 represents the life of a bubble during transient cavitation.

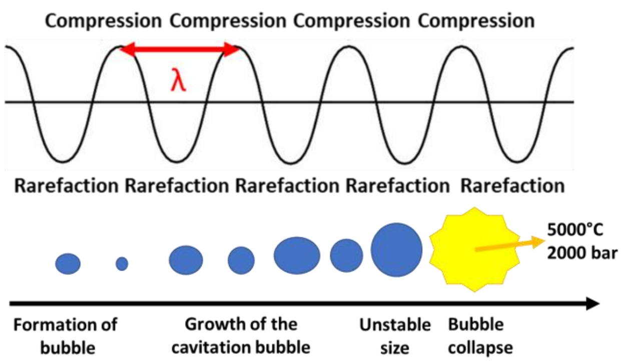


Figure 3. Life of a cavitation bubble, from formation to collapse. (© Certech)

1.2. Effects induced by cavitation

Whenever transient cavitation occurs, various physical and chemical phenomena will happen in the fluid that will influence the chemistry happening. Generally, these effects are classified in two categories, namely physical effects and chemical effects.[6]

The bubble implosion generates a lot of specific physical effects that will influence the chemical medium. This phenomenon will generate important shear forces that will induce microstreams within a liquid phase. For a homogeneous system, it will lead to a very efficient mixing. In the case of a heterogeneous liquid/liquid system, sonication can create projections of liquids around the interface, therefore propelling one phase into the other and increasing significantly mass transfer. In a sense, ultrasound acts here as a physical phase-transfer agent. This capacity to create a very efficient mixing locally can also be valorised for the preparation of emulsions for instance.[7] Sonication can also increase the mass transfer between a solid and a liquid by reducing the particle size of the former. When collapsing near a solid, a cavitation bubble can create high speed flows that can impact the solid surface,[8] as described on the figure below.

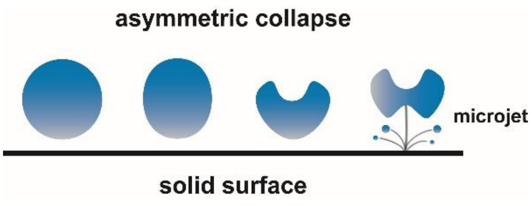


Figure 4. Physical effect of ultrasound next to a solid surface. (© Certech)

As another physical effect, the sonication of a liquid media will also lead to an increase of its temperature. A control of temperature must therefore be implemented in any sonochemical processes

Contrary to other techniques, such as microwave irradiation for instance, ultrasounds can also directly impact the molecules present in the media. In addition to the mechanical effects, the collapse of a cavitation bubble can generate enough energy to break chemical bounds, leading to the formation of free radicals that can subsequently induce new reactional pathways. These phenomena are often referred to as the chemical effects of ultrasound. These effects were unambiguously proven by the pioneering work of Ando et al. in the sono-organic synthesis described on Figure 5.[9] Starting from the same reactive mixture, his group obtained two different products when the medium was stirred (benzyl toluene isomers via an ionic pathway) or when it was sonicated (benzyl cyanide through a radical mechanism).

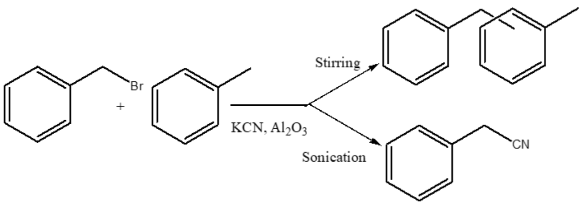


Figure 5. Ando's sonochemical switching.[9]

This chemical effect can be beneficial in different applications, such as in the degradation of pollutants in water. For such processes, sonication will lead to the homolytic cleavage of water into hydroxyl (HO•) and hydrogen (H•) radicals that will then induce the degradation processes.

The nature of the induced effects – physical or chemical – is mainly driven by the frequency of the ultrasonic wave applied. For relatively low frequencies (20-80 kHz), transient bubbles are relatively rare but large, generating mainly physical effects when collapsing. At higher frequencies (150 kHz to 2 MHz), bubbles are more numerous and of smaller size and their collapse favour chemical effects.

A representation of the different effects inducted by sonication are presented in Figure 6 as a conclusion of this part of the note.[10]

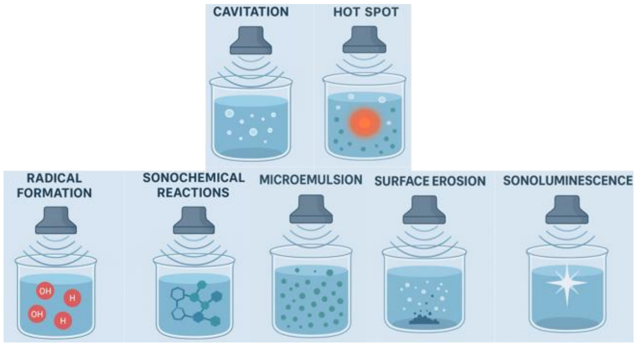


Figure 6. Different effects of sonication.[11]

2. Good laboratory practices

2.1. Equipment

Historically, the first ever ultrasonic transducer, following the work of Pierre and Jacques Curie on piezoelectricity,[11] was developed by Galton in 1883 and consists in a whistle that he used to determine the range a human ear can hear.[12] From these days, many pieces of equipment have been introduced in the market and nowadays, three main technologies are mostly used, namely ultrasonic bath, ultrasonic probe, and cup-horns systems. Each of these systems should be used according to good laboratory practices as described below to ensure an optimal and reproductible use. Noteworthy, the temperature must be controlled for any of these systems for reproducibility reasons. Additionally, any piezoelectric material loses its transducing properties above a certain temperature, known as Curie Temperature. The Curie temperature being different for different materials, it is vital to maintain the temperature of your medium below it (with a margin of generally 25%) to prevent any degradation of your equipment. Lastly, the use of sono-reactors can produce high-pitched noise and the use of acoustic protection is highly recommended.

Ultrasonic bath

Ultrasonic baths are present in many R&D laboratories where they are commonly used to clean glassware or hasten the solubilisation of one compound. It is an indirect way to sonicate your media as the flask to be submitted to ultrasound is introduced into a water bath that is itself sonicated. The bath must be filled, generally with water, up to the level that is indicated by the supplier, as using a smaller or larger amount of water will alter the efficiency of sonication. The sample shall ideally be introduced in a flat-bottomed flask that must not be placed in contact neither with the bottom nor the walls of the ultrasonic bath. The ultrasonic field generated by an ultrasonic bath is non-homogeneous and can vary greatly depending on the position in the bath. It is possible to determine the optimal position in the bath by introducing an aluminium foil in the water and find the position where it gets the most eroded. Once determined, all experiments have to be performed at the same spot to ensure repeatability. Additionally, the level of the solvent present in the flask should be approximately at the same level as the water in the bath to maximise physical effects. Finally, ultrasonic baths must not be switched on when empty.

Ultrasonic probe

Ultrasonic probes enable a direct sonication by immerging their tips into the medium to be sonicated. It is generally a highly energetic system that leads to a significant increase of temperature, especially with viscous media, and therefore double-jacketed reactors should be favoured to maintain the temperature constant via cooling. If not possible, pulsed irradiation should be used to prevent this important increase in temperature. The state of the tip should be controlled prior to any use as it tends to corrode, which leads to a decrease in acoustic power and possibly to the contamination of the medium via the metal constituting the tip. If degradation is observed, the tip can – in some cases – be replaced or should be polished. One of the potential issues of using such a system can come from the reflection of the sonic wave that can lead to the degradation of the probe. Therefore, in the case of small flasks, it is recommended to introduce the probe roughly in between the liquid level and the bottom of the flask to minimise this effect. It is also recommended to favour, if possible, round-bottomed flask to flat-bottomed ones, to prevent a direct reflection of the sonic wave onto the tip. Generally, it is recommended to always use the same geometry of flasks for repeatability. Precipitation of the medium would also lead to a very fast degradation of the sonotrode once again due to this direct reflection as well as a fast heating of the probe. Finally, for heterogeneous liquid/liquid systems, it is recommended to place the probe slightly above the liquid/liquid interface to "force" the liquid above to go into the other phase as efficiently as possible.

Cup-horn system

Cup-horn devices are designed to irradiate the system from the bottom to the top. They are highly energetic systems, and a strict control of the temperature is mandatory; double-jacketed systems as well as pulsed irradiation are also recommended to manage thermal build-up. Both direct and indirect irradiations are possible but the direct one should be favoured to maximise efficiency. In either case, corrosivity of the medium should be assessed. The acoustic efficiency highly depends on the height of liquid in the cup-horn reactor. An optimisation of the system – when the formation of radicals are expected. – can be done using chemical dosimeters, that would enable to estimate their number, and once determined, the volume of liquid should be kept constant.

2.2. Data

One of the main critics made to sonochemistry is that it can be difficult to replicate experiments described in published studies. This perception is fuelled by the publication of many papers or reports in which the experimental conditions are poorly described making these studies genuinely difficult to reproduce. Such a lack of reliable data is even more stringent for a domain like sonochemistry where the energy distribution is far from uniform. Therefore, it is extremely important to provide, for any sonochemical test, a very specific and accurate set of data regarding the equipment used as well as the experimental conditions of the process.

Equipment

Quite obviously, the reference of the system used for the irradiation (bath, probe…) must be provided, including brand, model and commercial name, if available. Information specific to this system such as its frequency and the diameter/shape of the irradiating zone must be provided. The mode of irradiation selected, continuous or pulsed (with duration of the irradiation/silent steps) must be indicated. The geometry and volume of the reactor used for the sonication will influence the efficiency of the sonication and must therefore also be provided. If a cooling system is used, it must be documented.

Chemistry and process

As mentioned previously, sonication is an energetic process that will lead to an increase of the temperature of the irradiated media. Temperature must therefore be recorded in-situ all along the process. Noteworthy, the temperature probe must not touch the ultrasonic device, especially the emitting zone(s). The irradiation time must also be recorded. Acoustic power is also a very important parameter to indicate the amount of energy provided to the system. and must be determined experimentally as it depends on many parameters (design of the rector, introduction of the sonotrode in the medium, …). The nominal value, often indicated on the ultrasound generator, is therefore inaccurate for a specific system and methods such as calorimetry must be used to determine the real value of the acoustic powert.[13] The volume of liquid irradiated, as well as the position of the probe must be given, as well as, obviously, the nature and physical form of the media that is irradiated. Pressure as well as the gas dissolved in a given liquid have a direct influence on cavitation. If a closed system is used, a precise control of pressure must be performed. Noteworthy, for a closed system using an ultrasonic probe, the connection between the reactor and the probe has to be done at a nodal point of the probe. For any system, it should be indicated if a degassing of the medium is performed, or not, prior to its sonication.

3. Some applications of sonochemistry

Physical and/or chemical effects of ultrasound can be beneficial in a large range of applications. As explained previously, the former can generate surface erosion/disruption, particle size reduction, efficient mixing and mass transfer, emulsification or nucleation while the latter leads to the formation of new chemical species.

The collapse of the cavitation bubbles near a solid surface will create microstreams that can be used for its cleaning. Ultrasonic cleaning is often used for glassware in chemistry laboratories but is also employed in other applications for example to clean electronic components or industrial equipment as well as expensive pieces such like jewellery or watchmaking.

The implosion of cavitation bubbles also generates extreme conditions that can disrupt cell structures. This phenomenon can be extremely beneficial to extract high value natural compounds from plant, leading to application in the food industry (neutraceuticals,…), cosmetics, pharmaceuticals (bioactive compouds) and others (essential oils, pigments, natural fragrances…). This process, known as ultrasound assisted extraction,[14] also benefits for the efficient mass transfer that ultrasound produces but its parameters (ultrasonic power, time, solvent,…) must be carefully controlled to avoid the potential degradation of the very products to be extracted. The potential of ultrasound to alter surface can also be used for other industries like the leather industry where sonication accelerates the diffusion of chemical species in the materials in processes such as tanning or dyeing.[15]

Reduction of particles size can be interesting for instance for the synthesis of nanomaterials such as metal nanoparticles,[16] nanobiomaterials.[17] or metal oxides[18] Ultrasounds enable the formation of smaller particles as well as a better distribution of size. In addition, chemical effects can also prevent the use of some specific reagents, via the generation of H_2O_2 and H_2 in water, for instance, for the synthesis of metal oxides like magnetite.[19] Smaller and more controlled particles can lead to enhanced overall properties as well as to better dispersion in composite materials like paints or coatings.

The formation of fine and stable emulsion via sonification, in some cases without the use of emulsifier in the formulation, can be valuable in different sectors like the food industry,[20] or cosmetics,[21]. As stable emulsions with smaller droplets are formed, this process can lead not only to a better shelf like of the product but also to an increase of the availability of the ingredients present in the droplets.

Ultrasounds are also used for the controlled formation and growth of crystals via a process often called sonocrystallisation. Many physical effects can explain the advantageous effects of sonocrystallisation including the formation of nuclei via the cavitation bubbles, the initiation of secondary nucleation via erosion or shattering/disruption of nuclei or a control of the size via sonofragmenation or deagglomeration.[22] Such processes are particularly interesting in the pharmaceutical industry, where a fine control of the size and polymorphism is vital. Crystalline active pharmaceutical ingredients such as Paroxetin Hydrochloride IPA Solvate or fenoterol.HBr can for instance be produced *via* sonocrystallisation.[23]

One of the main applications of the chemical effects of ultrasound is in environmental science and more precisely of wastewater treatment *via* sonolysis, either alone or coupled to other techniques.[24] As mentioned previously, the energy generated during the collapse of cavitation bubbles is high enough to form radicals via the homolytic breakage of chemical bounds. In the case of water, it will lead to the formation HO· et H· radicals (and other reactive species depending on the medium et experimental conditions) that could break down complex organic pollutants into simpler species (or even into CO_2 and H_2O).[25]

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