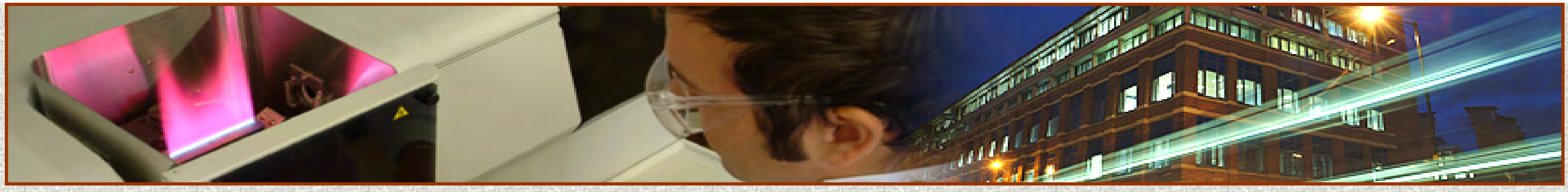

Introduction

Home
Introduction
Research
Electro
Therapy
Food
Synthesis
Green
Materials
Group

THE SONOCHEMISTRY CENTRE AT COVENTRY UNIVERSITY

'The Home of Sound Science'

INTRODUCTION To SONOCHEMISTRY

Publications
Advances in Sono
Books
Chapters
Links
Contact information

1. ULTRASOUND

If you were asked what you knew about ultrasound you would almost certainly start with the fact that it is used in animal communications (e.g. bat navigation and dog whistles). You might then recall that ultrasound is used in medicine for foetal imaging, in underwater range finding (SONAR) or in the non-destructive testing of metals for flaws. A chemist would probably not consider sound as the type of energy that could be used for the excitation of a chemical reaction. Indeed up to a few years ago the use of ultrasound in chemistry was something of a curiosity and the practising chemist could have been forgiven for not having met the concept. To increase chemical reactivity one would probably turn towards heat, pressure, light or the use of a catalyst. And yet, if one stops for a second to consider what is involved in the transmission of a sound wave through a medium it is perhaps surprising that for so many years sound was not considered as a potential source of enhancement of chemical reactivity. The only exception to this being the green-fingered chemist who, in the privacy of his own laboratory, talks, sings or even shouts at his reaction. After all, sound is transmitted through a medium as a pressure wave and the mere act of transmission must cause some excitation in the medium in the form of enhanced molecular motion. However, as we will see later, in order to produce real effects the sound energy must be generated within the liquid itself. This is because the transfer of sound energy from the air into a liquid is not an efficient process.

1.1 HISTORICAL BACKGROUND

The basis for the present-day generation of ultrasound was established as far back as 1880 with the discovery of the piezoelectric effect by the Curies [l-3]. Most modern ultrasonic devices rely on transducers (energy converters) which are composed of piezoelectric material. Such materials respond to the application of an electrical potential across opposite faces with a small change in dimension. This is the inverse of the piezoelectric effect. If the potential is alternated at high frequencies the crystal converts the electrical energy to mechanical vibration (sound) energy – rather like a loudspeaker. At sufficiently high alternating potential high frequency sound (ultrasound) will be generated.

The earliest form of an ultrasonic transducer was a whistle developed by Francis Galton (1822-1911) in 1883 to investigate the threshold frequency of human hearing [4]. A diagram of the whistle is to be found in the section on transducers. Galton himself was a remarkable man. As well as inventing the whistle that carries his name he explored and helped map a portion of the African interior, invented the weather map and developed the first workable system for classifying and identifying fingerprints. His whistle was part of his study of sensory perception, in this case to determine the limits of hearing in terms of sound frequencies in both humans and animals.

The first commercial application of ultrasonics appeared around 1917 and was the first “echo-sounder” invented and developed by Paul Langévin (1872-1946). He was born in Paris and was a contemporary to Marie Curie, Albert Einstein and Hendrik Lorentz. He was noted for his work on the molecular structure of gases, analysis of secondary emission of X-rays from metals exposed to radiation and for his theory of magnetism. However Langévin is more generally remembered for important work on piezoelectricity and on piezoceramics. The original “echo-sounder” eventually became underwater SONAR for submarine detection during World War 2. The transducer was a mosaic of thin quartz crystals glued between two steel plates (the composite having a resonant frequency of about 50 kHz), mounted in a housing suitable for submersion. The early "echo sounder" simply sent a pulse of ultrasound from the keel of a boat to the bottom of the sea from which it was reflected back to a detector also on the keel. For sound waves, since the distance traveled through a medium = 1/2 x time x velocity (and the velocity of sound in seawater is accurately known) the distance to the bottom could be gauged from the time taken for the signal to return to the boat. If some foreign object (e.g. a submarine) were to come between the boat and the bottom of the seabed an echo would be produced from this in advance of the bottom echo. In the UK this system was very important to the Allied Submarine Detection Investigation Committee during the war and became popularly known by the acronym ASDIC. Later developments resulted in a change in the name of the system to SONAR (SOund Navigation And Ranging) which allowed the surrounding sea to be scanned. The original ASDIC system predated the corresponding RAdio Detection And Ranging system (RADAR) by 30 years.

Essentially all imaging from medical ultrasound to non-destructive testing relies upon the same pulse-echo type of approach but with considerably refined electronic hardware. The refinements enable the equipment not only to detect reflections of the sound wave from the hard, metallic surface of a submarine in water but also much more subtle changes in the media through which sound passes (e.g. those between different tissue structures in the body). It is high frequency ultrasound (in the range 2 to 10 MHz) which is used primarily in this type of application because by using these much shorter wavelengths it is possible to detect much smaller areas of phase change i.e. give better 'definition'. The chemical applications of high frequency ultrasound are concerned essentially with measurements of either the velocity of sound through a medium or the degree to which the sound is absorbed as it passes through it. These applications will be discussed in more detail in. Such measurements are diagnostic in nature and do not effect the chemistry of the system under study.

When more powerful ultrasound at a lower frequency is applied to a system it is possible to produce chemical changes as a result of acoustically generated cavitation. Cavitation as a phenomenon was first identified and reported in 1895 by Sir John Thornycroft and Sidney Barnaby [5]. This discovery was the result of investigations into the inexplicably poor performance of a newly built destroyer HMS Daring. Her top speed was well below specifications and the problem was traced to the propeller blades that were incorrectly set and therefore not generating sufficient thrust. The rapid motion of the blades through water was found to tear the water structure apart by virtue of simply mechanical action. The result of this was the production of what are now called cavitation bubbles. The solution to this problem lies in using very wide blades covering about two-thirds of the disc area of the propeller, so as to present a very large surface contact with the water. This helps to prevent disruption under the force necessary to propel the vessel. As ship speeds increased, however, this became a serious concern and the Royal Navy commissioned Lord Rayleigh to investigate. He produced a seminal work in the field of cavitation which confirmed that the effects were due to the enormous turbulence, heat, and pressure produced when cavitation bubbles imploded on or near to the propeller surface [6]. In the same work, he also observed that cavitation and bubble collapse was also the origin of the noise made when water is heated towards boiling point.

Since l945 an increasing understanding of the phenomenon of cavitation has developed coupled with significant developments in electronic circuitry and transducer design (i.e. devices which convert electrical to mechanical signals and vice versa). As a result of this there has been a rapid expansion in the application of power ultrasound to chemical processes, a subject which has become known as “Sonochemistry”.

1.2 THE POWER OF SOUND

Sound, as a general subject for study, is traditionally found in a physics syllabus but it is not a topic which is met in a chemistry course and so is somewhat unfamiliar to practising chemists. Sound is transmitted through a medium by inducing vibrational motion of the molecules through which it is travelling. This motion can be visualised as rather like the ripples produced when a pebble is dropped into a pool of still water. The waves move but the water molecules which constitute the wave revert to their normal positions after the wave has passed. An alternative representation is provided by the effect of a sudden twitch of the end of a horizontal stretched spring. Here the vibrational energy is transmitted through the spring as a compression wave which is seen to traverse its whole length. This is just a single compression wave and it does not equate to sound itself which is a whole series of such compression waves separated by rarefaction (stretching) waves in between. The pitch (or note) of the sound produced by this series of waves depends upon their frequency i.e. the number of waves which pass a fixed point in unit time. For middle C this is 256 per second. In physics sound waves are often shown as a series of vertical lines or shaded colour where line separation or colour depth represent intensity, or as a sine wave where intensity is shown by the amplitude (Figure 1.1).

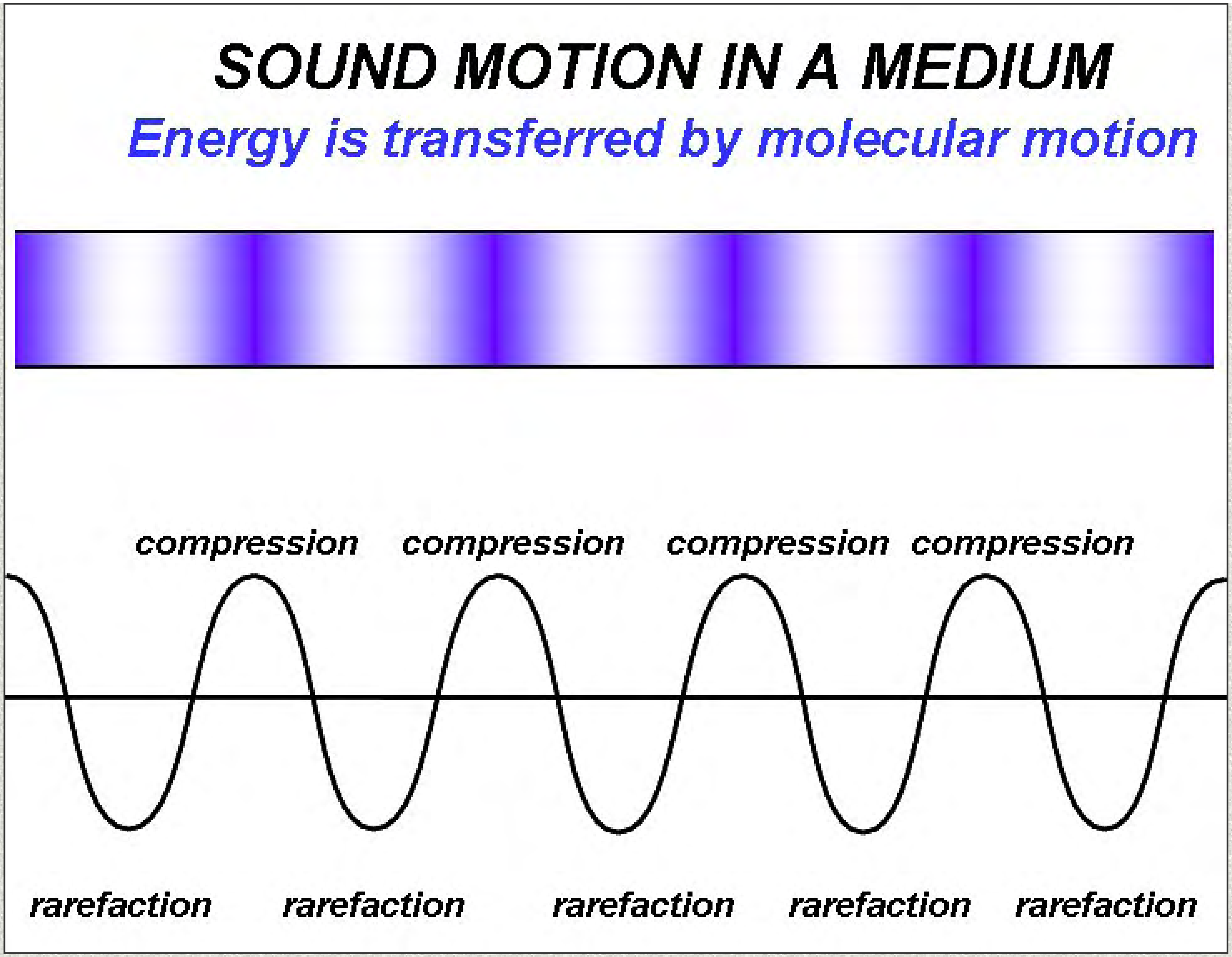

Figure 1.1: Sound transmission through a medium

The physical effects of sound vibrations are most easily experienced by standing in front of a loudspeaker playing music at high volume. The actual sound vibrations are transmitted through the air and are not only audible but can also be sensed by the body through the skin. The bass notes are felt through the body more easily than the high notes and this is connected with the frequency of the pressure pulse creating the sound. Low frequency sound becomes audible at around 18Hz (1Hz = 1 Hertz = 1 cycle per second) but as the frequency of the sound is raised (becoming more treble) it becomes more difficult for the body to respond and that sensation is lost. High frequency sound, while not noticeably effecting the body does cause severe annoyance to hearing e.g. feed back noise from a microphone through a loud speaker. At even higher frequencies the ear finds it difficult to respond and eventually the human hearing threshold is reached, normally around 18-20kHz for adults, sound beyond this limit is inaudible and is defined as ultrasound. The hearing threshold is not the same for other animal species thus dogs respond to ultrasonic whistles (so called "silent" dog whistles) and bats use frequencies well above 50kHz for navigation (Figure 1.2).

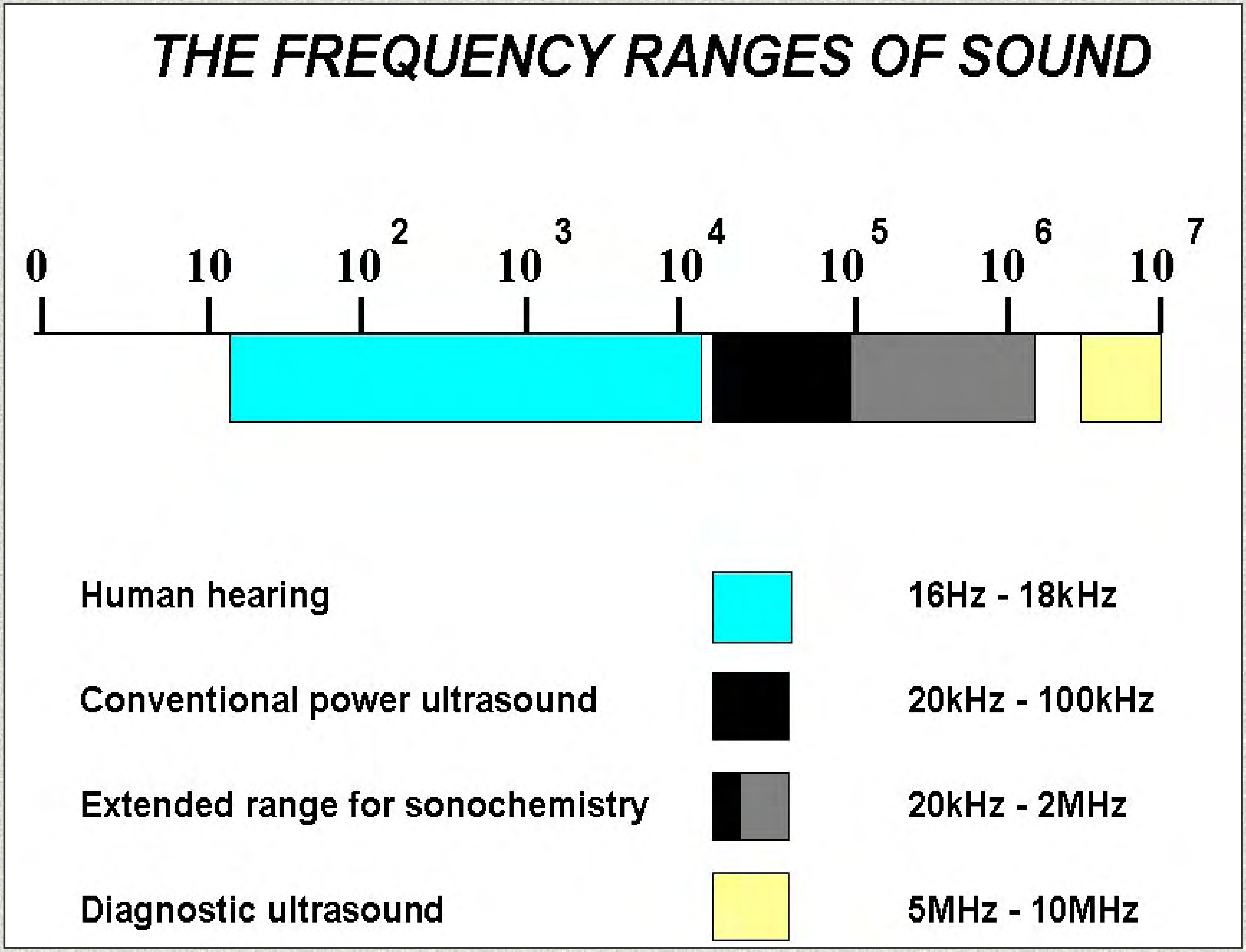

Figure 1.2: Frequency ranges of sound

The broad classification of ultrasound as sound above 20kHz and up to 100MHz can be subdivided into two distinct regions Power and Diagnostic. The former is generally at lower frequency end where greater acoustic energy can be generated to induce cavitation in liquids, the origin of chemical effects. Sonochemistry normally uses frequencies between 20 and 40kHz simply because this is the range employed in common laboratory equipment. However since acoustic cavitation in liquids can be generated well above these frequencies, recent researches into sonochemistry use a much broader range (Figure 1.2). High frequency ultrasound from around 5MHz and above does not produce cavitation and this is the range used in medical imaging.

A whistle which generates a frequency 20kHz is inaudible to humans but perfectly audible to a dog - and produces no physical harm to either. It is however in the correct FREQUENCY range to affect chemical reactivity (Power Ultrasound). Yet such a whistle blown in a laboratory will not influence chemical reactions in any way. This is because the whistle is producing sound energy in air and airborne sound cannot be transferred into a liquid.

1.3 REFERENCES

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2. ACOUSTIC CAVITATION

Power ultrasound enhances chemical and physical changes in a liquid medium through the generation and subsequent destruction of cavitation bubbles. Like any sound wave ultrasound is propagated via a series of compression and rarefaction waves induced in the molecules of the medium through which it passes. At sufficiently high power the rarefaction cycle may exceed the attractive forces of the molecules of the liquid and cavitation bubbles will form. Such bubbles grow by a process known as rectified diffusion i.e. small amounts of vapour (or gas) from the medium enters the bubble during its expansion phase and is not fully expelled during compression. The bubbles grow over the period of a few cycles to an equilibrium size for the particular frequency applied. It is the fate of these bubbles when they collapse in succeeding compression cycles which generates the energy for chemical and mechanical effects (Figure 2.1). Cavitation bubble collapse is a remarkable phenomenon induced throughout the liquid by the power of sound. In aqueous systems at an ultrasonic frequency of 20kHz each cavitation bubble collapse acts as a localised "hotspot" generating temperatures of about 4,000 K and pressures in excess of 1000 atmospheres [1-3].

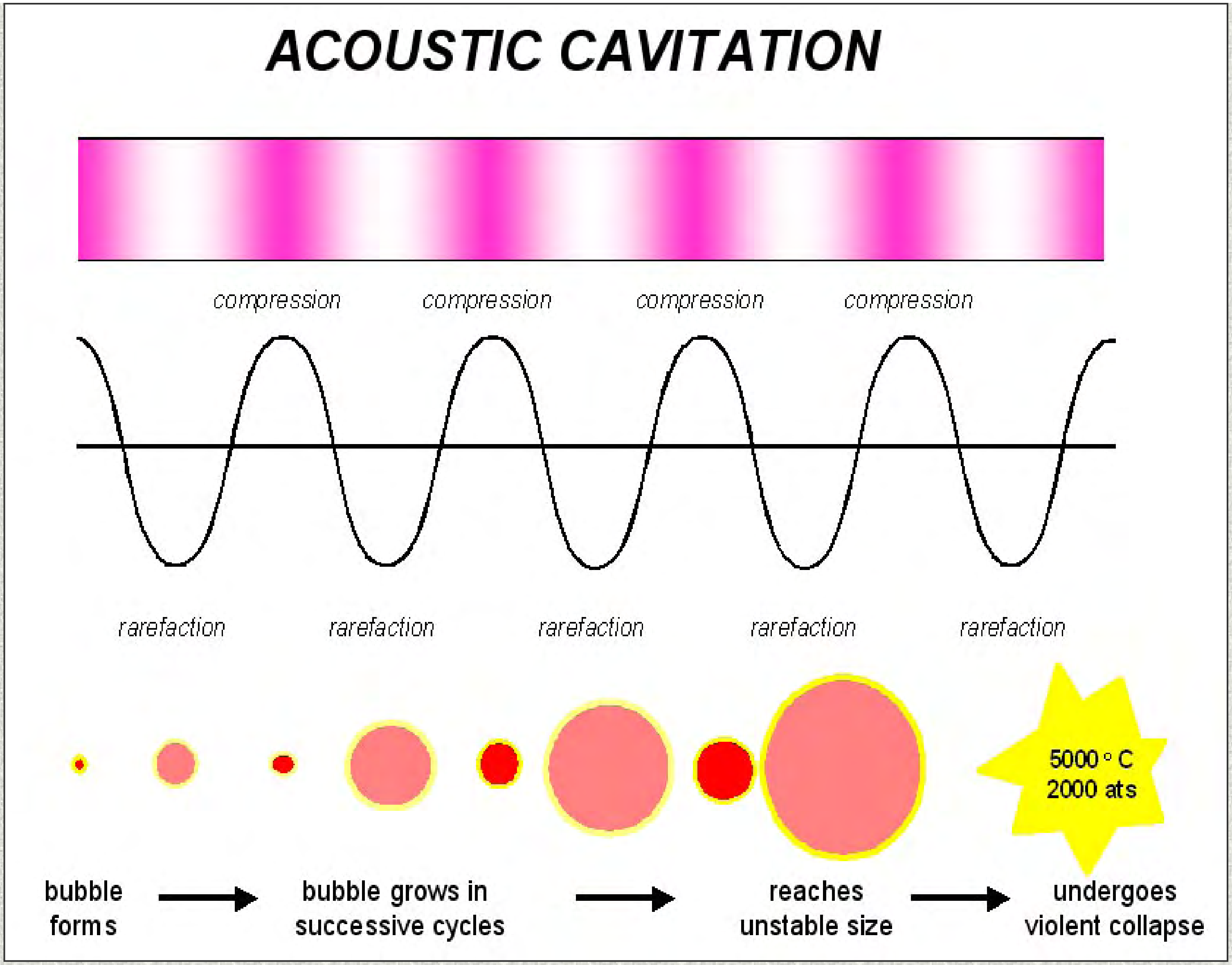


Figure 2.1: Generation of an acoustic bubble

The cavitation bubble has a variety of effects within the liquid medium depending upon the type of system in which it is generated. These systems can be broadly divided into homogeneous liquid, heterogeneous solid/liquid and heterogeneous liquid/liquid. Within chemical systems these three groupings represent most processing situations.

2.1 HOMOGENEOUS LIQUID-PHASE REACTIONS

(i) in the bulk liquid immediately surrounding the bubble where the rapid collapse of the bubble generates shear forces which can produce mechanical effects and
(ii) in the bubble itself where any species introduced during its formation will be subjected to extreme conditions of temperature and pressure on collapse leading to chemical effects. (Figure 2.2).

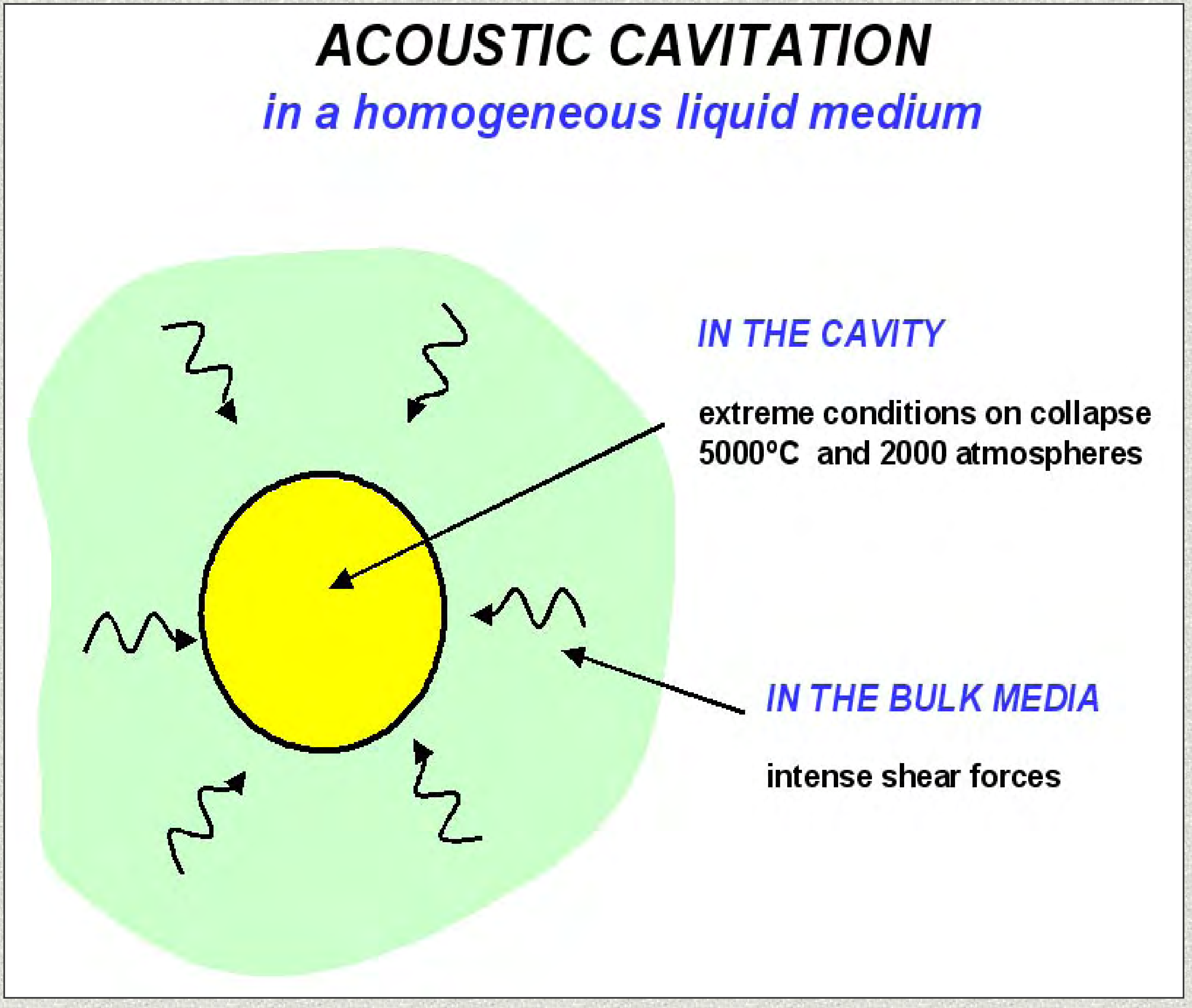
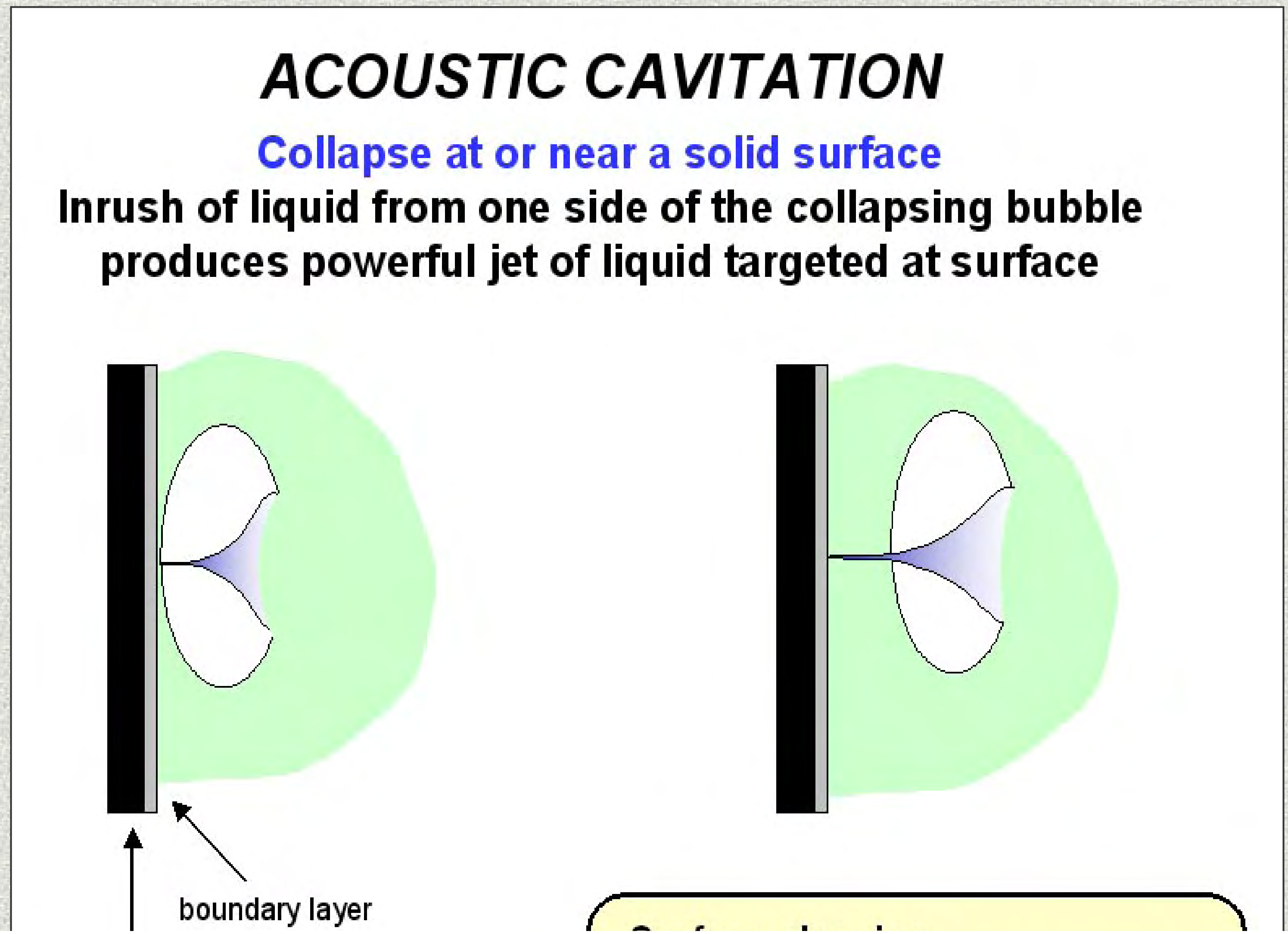


Figure 2.2: Acoustic cavitation in a homogeneous liquid

2.2 CAVITATION NEAR A SURFACE

Unlike cavitation bubble collapse in the bulk liquid, collapse of a cavitation bubble on or near to a surface is unsymmetrical because the surface provides resistance to liquid flow from that side. The result is an inrush of liquid predominantly from the side of the bubble remote from the surface resulting in a powerful liquid jet being formed, targeted at the surface (Figure 2.3). The effect is equivalent to high pressure jetting and is the reason that ultrasound is used for cleaning. This effect can also activate solid catalysts and increase mass and heat transfer to the surface by disruption of the interfacial boundary layers.



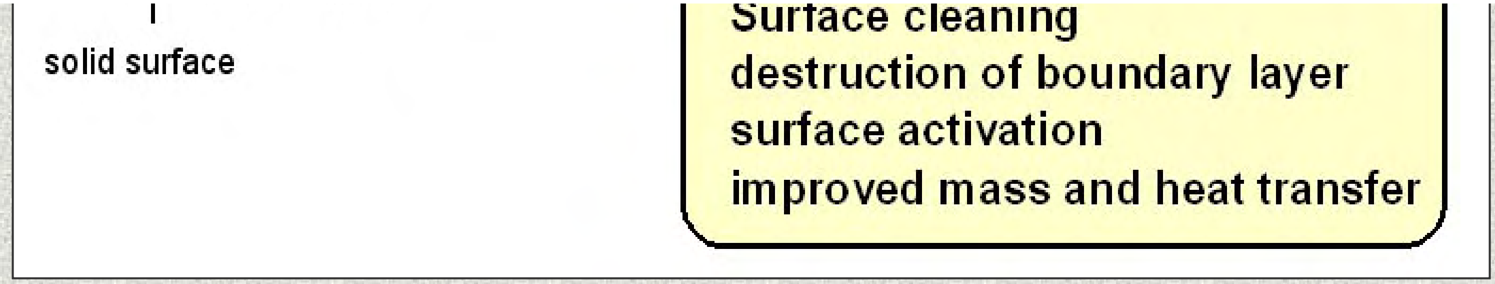


Figure 2.3: Cavitation bubble collapse at or near a solid surface

2.3 HETEROGENEOUS POWDER-LIQUID REACTIONS

Acoustic cavitation can produce dramatic effects on powders suspended in a liquid (Figure 2.4). Surface imperfections or trapped gas can act as the nuclei for cavitation bubble formation on the surface of a particle and subsequent surface collapse can then lead to shock waves which break the particle apart. Cavitation bubble collapse in the liquid phase near to a particle can force it into rapid motion. Under these circumstances the general dispersive effect is accompanied by interparticle collisions which can lead to erosion, surface cleaning and wetting of the particles and particle size reduction.

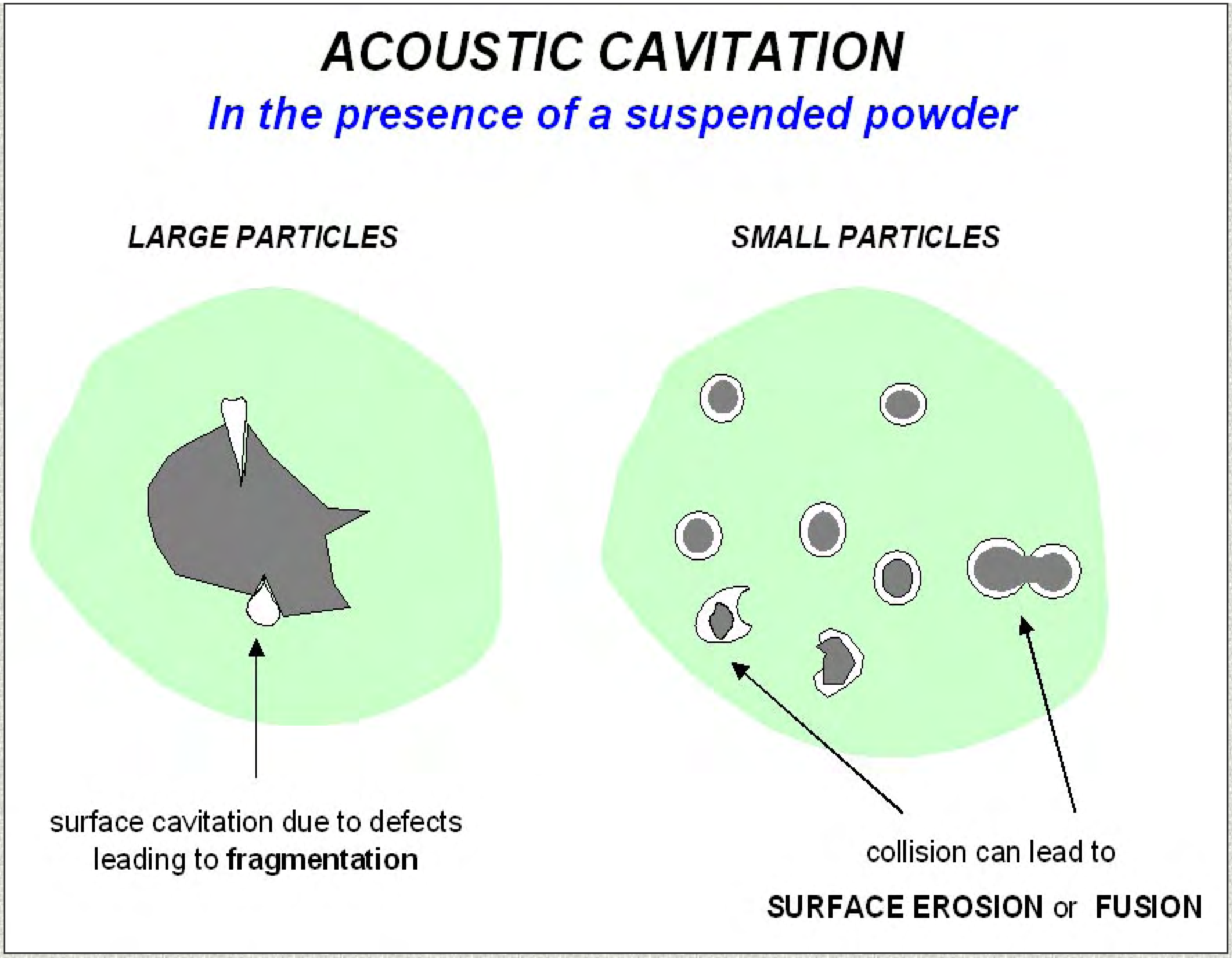


Figure 2.4: Acoustic cavitation in a liquid with a suspended powder

In heterogeneous liquid/liquid reactions, cavitational collapse at or near the interface will cause disruption and mixing, resulting in the formation of very fine emulsions (Figure 2.5).

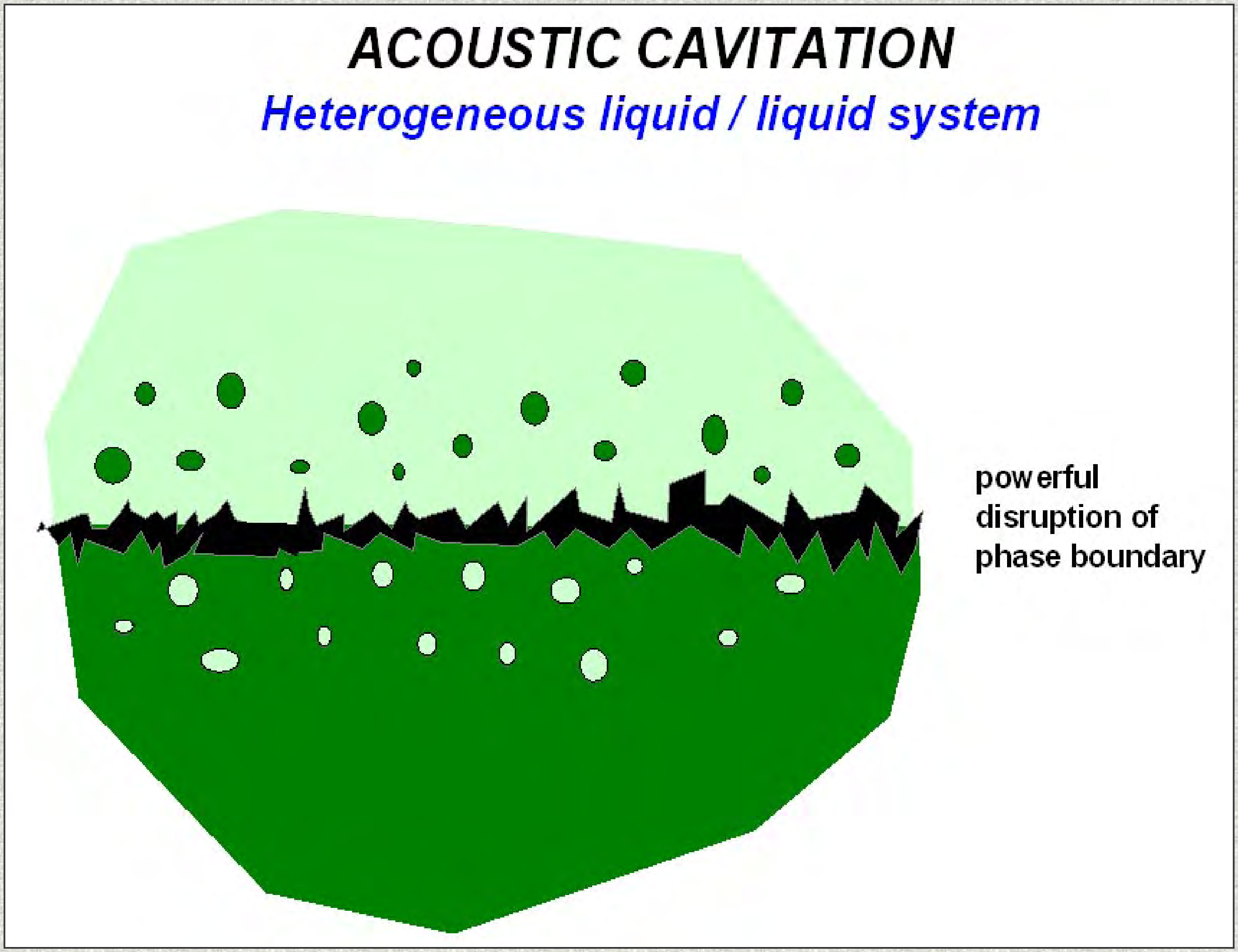


Figure 2.5: Cavitation effects in a heterogeneous liquid/liquid system

2.4 REFERENCES FOR CAVITATION

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3. TRANSDUCERS

A transducer is the name for a device capable of converting one form of energy into another, a simple example being a loudspeaker which converts electrical energy to sound energy. Ultrasonic transducers are designed to convert either mechanical or electrical energy into high frequency sound and there are three main types: gas driven, liquid driven and electromechanical.

3.1 GAS-DRIVEN TRANSDUCERS

These are, quite simply, whistles with high frequency output (the dog whistle is a familiar example). The history of the generation of ultrasound via whistles dates back 100 years to the work of F.Galton who was interested in establishing the threshold levels of human hearing. He produced a whistle that generated sound of known frequencies and was able to determine that the normal limit of human hearing is around 18kHz. Galton's whistle was constructed from a brass tube with an internal diameter of about two millimetres (Figure 3.1) and operated by passing a jet of gas through an orifice into a resonating cavity. On moving the plunger the size of the cavity could be changed to alter the "pitch" or frequency of the sound emitted. An adaptation of this early principle is to be found in some dog whistles that have adjustable pitch.

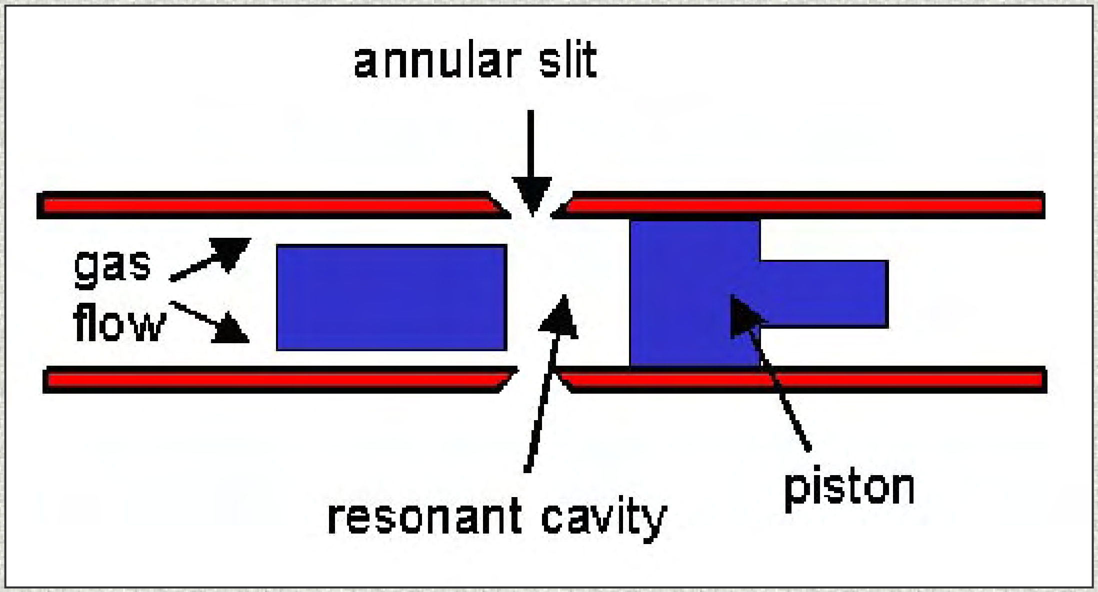


Figure 3.1: Galton Whistle

An alternative form of gas generated ultrasound is the siren. When a solid object is passed rapidly back-and-forth across a jet of high pressure gas it interferes with the gas flow and produces sound of the same frequency at which the flow is disturbed. A siren can be designed by arranging that the nozzle of a gas jet impinges on the inner surface of a cylinder through which there are a series of regularly spaced perforations. When the cylinder is rotated the jet of gas emerging from the nozzle will rapidly alternate between facing a hole or the solid surface. The pitch of the sound generated by this device will depend upon the speed of rotation of the cylinder. Neither type of transducer has any significant chemical application since the efficient transfer of acoustic energy from a gas to a liquid is not possible. However whistles are used for the atomization of liquids.

The conventional method of producing an atomized spray from a liquid is to force it at high velocity through a small aperture. (A typical domestic examples being a spray mist bottle for perfume). The disadvantage in the design of conventional equipment is that the requirement for a high liquid velocity and a small orifice restricts its usage to low viscosity liquids and these atomizers are often subject to blockage at the orifice.

Figure 3.2 shows a schematic gas driven atomizer. The system comprises of an air or gas jet, which is forced into an orifice where it expands and produces a shock wave. The result is an intense field of sonic energy focused between the nozzle body and the resonator gap. When liquid is introduced into this region it is vigorously sheared into droplets by the acoustic field. Air by-passing the resonator carries the atomized droplets downstream in a fine soft plume shaped spray. The droplets produced are small and have a low forward velocity. Atomized water sprays have many uses including dust suppression in industry and humidifiers for horticultural use under glass.

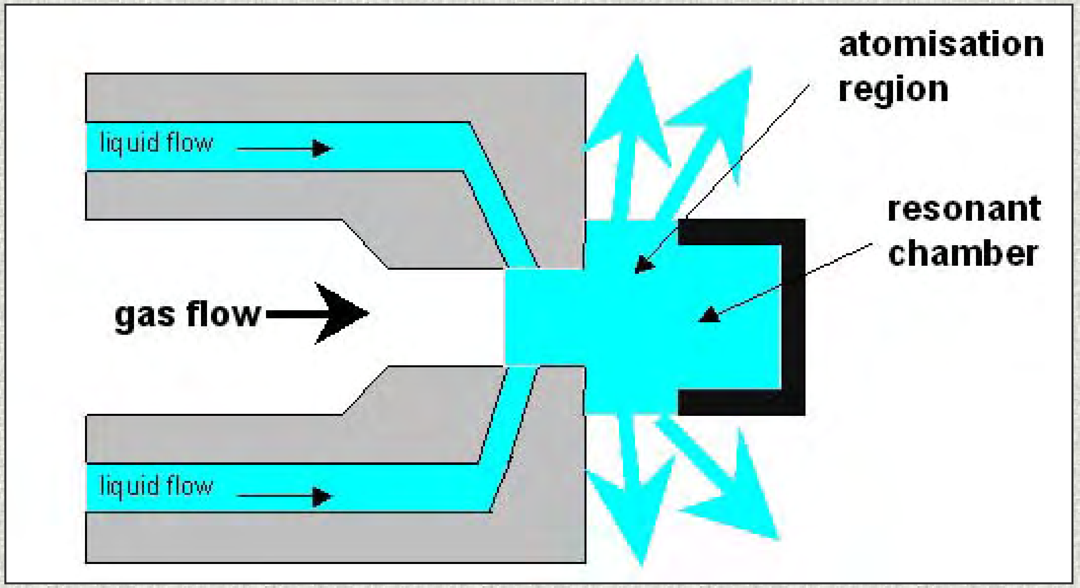


Figure 3.2: Gas Driven Atomizer

3.2 LIQUID-DRIVEN TRANSDUCERS

In essence this type of transducer is a "liquid whistle" and generates cavitation via the motion of a liquid rather than a gas. Process material is forced at high velocity by the homogeniser pump through a special orifice from which it emerges as a jet which impacts upon a steel blade (Figure 3.3). There are two ways in which cavitational mixing can occur at this point. Firstly through the Venturi effect as the liquid rapidly expands into a larger volume on exiting the orifice and secondly via the blade which is caused to vibrate by the process material flowing over it. The relationship between orifice and blade is critically controlled to optimise blade activity. The required operating pressure and throughput is determined by the use of different sizes and shapes of the orifices and the velocity can be changed to achieve the necessary particle size or degree of dispersion. With no moving parts, other than a pump, the system is rugged and durable. When a mixture of immiscible liquids is forced through the orifice and across the blade cavitational mixing produces extremely efficient homogenization.

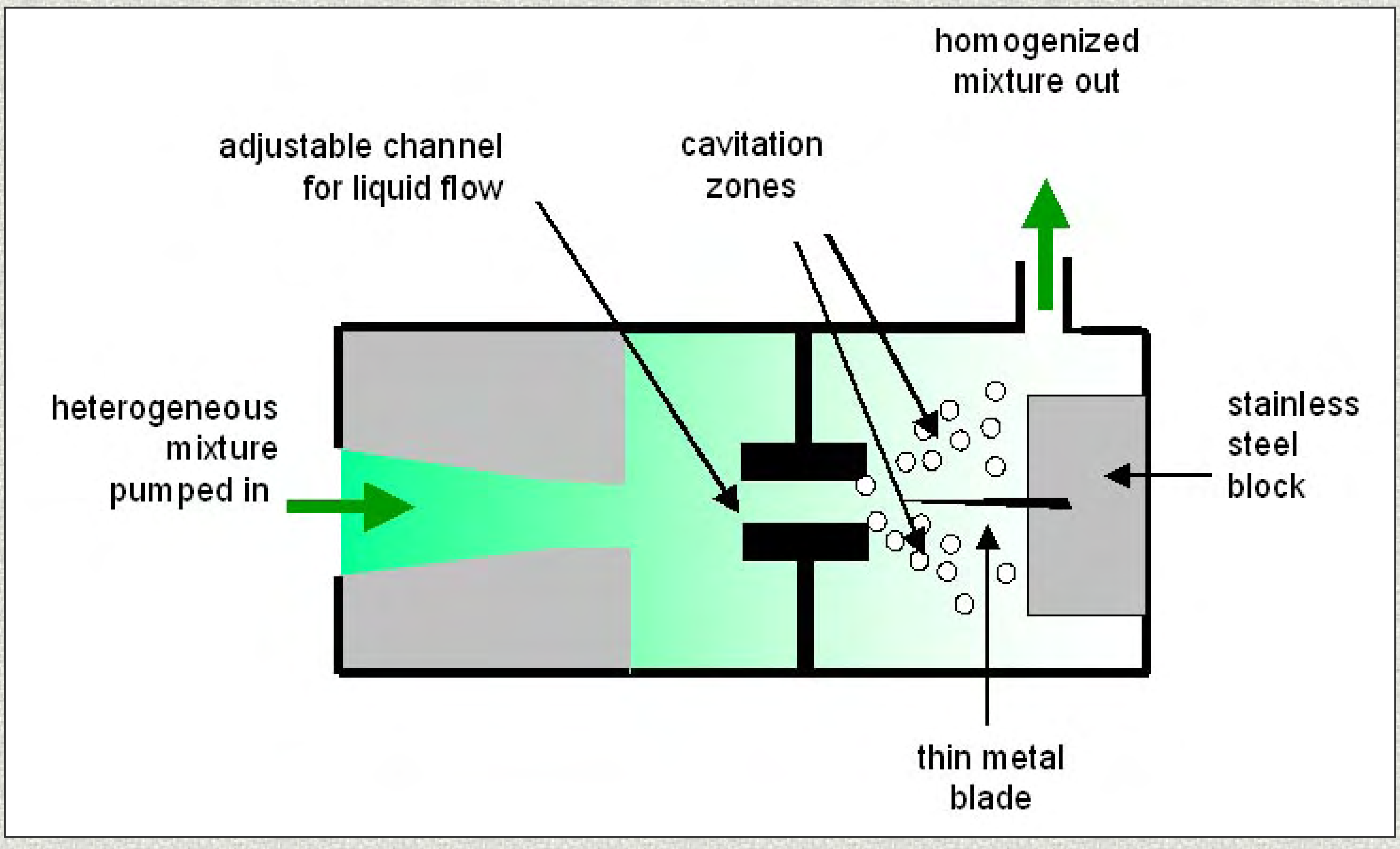


Figure 3.3: Liquid Whistle

3.3 ELECTROMECHANICAL TRANSDUCERS

The two main types of electromechanical transducers are based on either the piezoelectric or the magnetostrictive effect. The most commonly used of which are piezoelectric transducers, generally employed to power the bath and probe type sonicator systems. Although more expensive than mechanical transducers, electromechanical transducers are by far the most versatile.

3.3.1 Magnetostrictive Transducers

Historically magnetostrictive transducers were the first to be used on an industrial scale to generate high power ultrasound. These are devices which use an effect found in some materials e.g. nickel which reduce in size when placed in a magnetic field and return to normal dimensions when the field is removed (magnetostriction). When the magnetic field is applied as a series of short pulses to a magnetostrictive material it vibrates at the same frequency. In simple terms such a transducer can be thought of as a solenoid in which the magnetostrictive material (normally a laminated metal or alloy) forms the core with copper wire winding. To avoid magnetic losses two such solenoids are wound and connected in a loop (Figure 3.4).

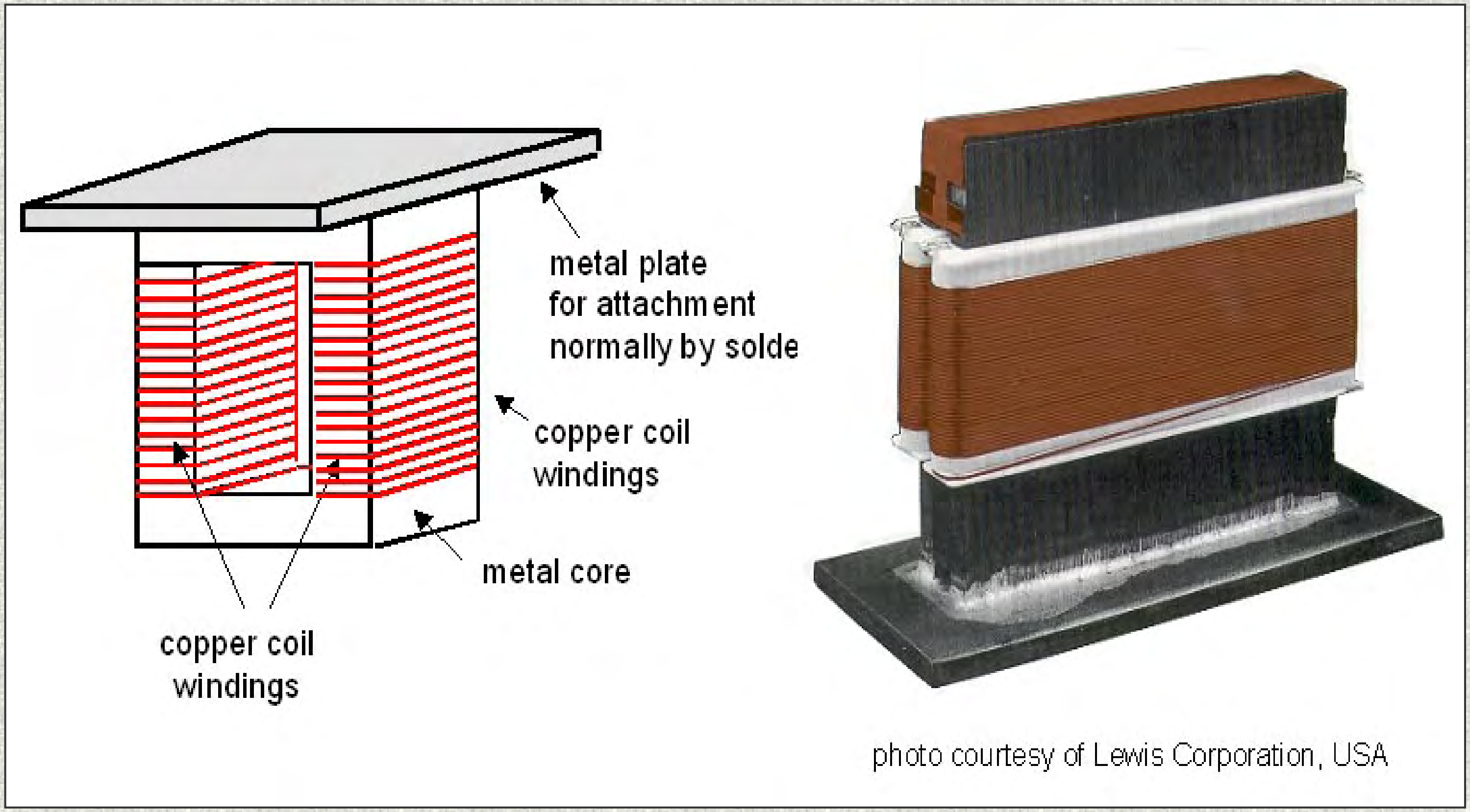


Figure 3.4: Piezoelectric Sandwich Transducer

The major advantages of magnetostrictive systems are that they are of an extremely robust and durable construction and provide very large driving forces. This makes them an attractive proposition for heavy duty industrial processing. There are however two disadvantages, firstly the upper limit to the frequency range is 100kHz, beyond which the metal cannot respond fast enough to the magnetostrictive effect, and secondly the electrical efficiency is less than 60% with significant losses emerging as heat. As a result of the second of these problems all magnetostrictive transducers subject to extended use are liquid cooled. This has meant that piezoelectric transducers (see below) which are more efficient and operate over a wider frequency range are generally considered to be the better choice in sonochemistry, especially in laboratory situations. However now that a range of industrial applications for sonochemistry are under consideration, particularly those requiring heavy duty continuous usage at high operating temperatures, the magnetostrictive transducer is coming back into consideration.

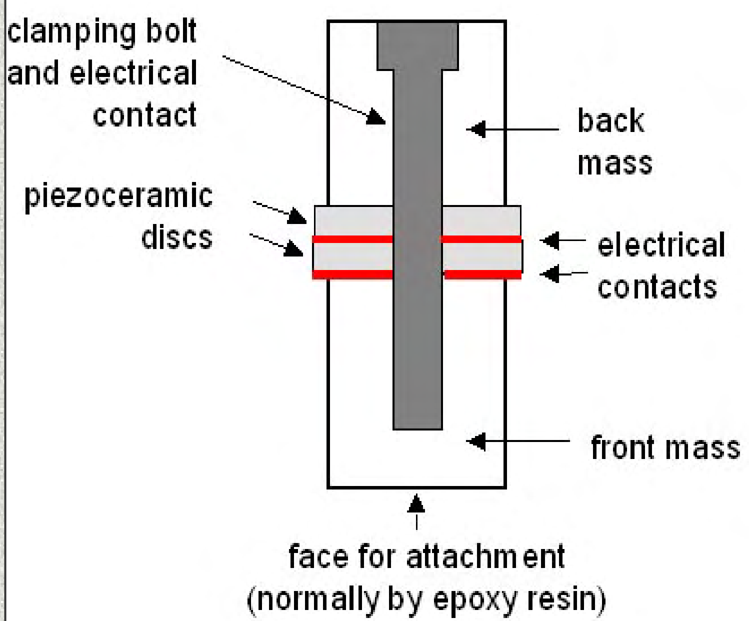
Many improvements in the operating efficiency of this type of transducer have been made all of which are based on finding a more efficient magnetostrictive core. The original nickel based alloys have been replaced by more electrically efficient cobalt/iron combinations and, more recently, aluminium/iron with a small amount of chromium. One of the latest developments in magnetostrictive technology has been the introduction of a new material called TERFINOL-D. This is an alloy of the rare earths terbium and dysprosium with iron which is zone refined to produce a material almost in the form of a single crystal. It can be produced in various forms, rods, laminates, tubes etc and has several major advantages over the more conventional alloys used.
A magnetostrictive transducer based on this material can generate more power than a conventional piezoelectric transducer, it is compact (about 50% smaller) and lighter than other magnetostrictives. It does have the same problem as other such devices in that it has an upper limit of frequency response - in this case 70kHz.

3.3.2 Piezoelectric Transducers

The most common types of transducer used for both the generation and detection of ultrasound employ materials that exhibit the piezoelectric effect, discovered over a century ago. Such materials have the following two complementary properties:

1. The direct effect - when pressure is applied across the large surfaces of the section a charge is generated on each face equal in size but of opposite sign. This polarity is reversed if tension is applied across the surfaces.
2. The inverse effect - if a charge is applied to one face of the section and an equal but opposite charge to the other face then the whole section of crystal will either expand or contract depending on the polarity of the applied charges. Thus on applying rapidly reversing charges to a piezoelectric material fluctuations in dimensions will be produced. This effect can be harnessed to transmit ultrasonic vibrations from the crystal section through whatever medium with which it is in contact.

Quartz was the piezoelectric material originally used in devices such as the very early types of ASDIC underwater ranging equipment. Quartz is not a particularly good material for this purpose because of its mechanical properties, it is a somewhat fragile and difficult to machine. Modern transducers are based on ceramics containing piezoelectric materials These materials cannot be obtained as large single crystals and so, instead, they are ground with binders and sintered under pressure at above 1000oC to form a ceramic. Cooling from above their ferroelectric transition temperature in a magnetic field then aligns the crystallites of the ceramic. Such transducers can be produced in different shapes and sizes. Nowadays the most frequently employed piezoceramic contains lead zirconate titanate (commonly referred to as PZT where the P represents plumbum - the chemical term for the element lead - and the Z and T are initials from the name of the salts). The most common form is a disk with a central hole. In a power transducer it is normal practise to clamp two of these piezoelectric disks between metal blocks which serve both to protect the delicate crystalline material and to prevent it from overheating by acting as a heat sink. The resulting "sandwich" provides a durable unit with doubled mechanical effect (Figure 3.5). The unit is generally one half wavelength long (although multiples of this can be used). The peak to peak amplitudes generated by such systems are normally of the order of l0-20 microns and they are electrically efficient. Generally piezoelectric devices must be cooled if they are to be used for long periods at high temperatures because the ceramic material will degrade under these conditions.



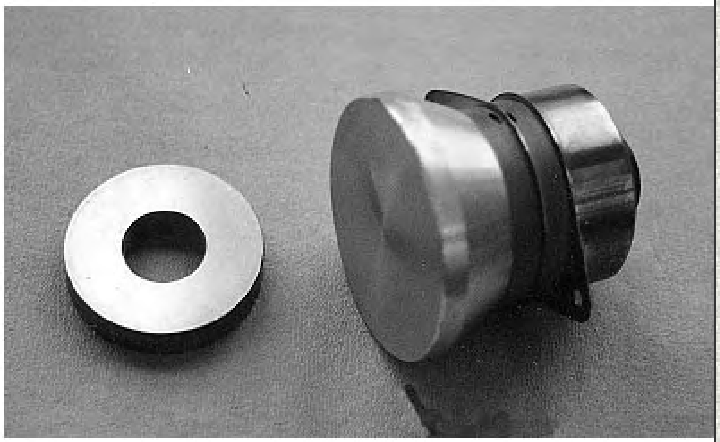


photo courtesy of Langford Ultrasonics, UK

Figure 3.5: Piezo electric Transducer

Such transducers are highly efficient (>95%) and, depending on dimensions, can be used over the whole range of ultrasonic frequencies from 20kHz to many MHz. They are the exclusive choice in medical scanning which uses frequencies above 5MHz.

3.3.3 Low frequency vibrating bar transducer system

A significantly different system has been introduced to large scale processing and this involves audible frequency vibrations generated in a large cylindrical steel bar6. The bar is driven into a clover leaf type of motion by firing three powerful magnets in sequence which are located at each end of the bar. The bar is supported by air springs so that the ends and the centre are then caused to rotate at a resonance frequency depending on its size (Figure 3.6). One such unit, operating at a power of 75kW, drives a bar which is 4.1 metre long and 34 cm in diameter at its resonance frequency of 100Hz. The bar itself weighs 3 tonnes and produces a vibrational amplitude at each end of 6mm considerably larger than the amplitudes available through sonochemical processing and hence better for the dispersal of materials in liquids. This type of system can be used in chemical processing applications by fixing a robust cylindrical steel cell to each end of the bar. Material in the form of a liquid or slurry can then be pumped through the cells in order to perform operations such as mixing, grinding and the destruction of hazardous waste. Hard spherical grinding balls are often added to the cells to assist in these processes. The combination of the large vibrational energy together with the motion of grinding balls appears to provide an extremely good alternative to conventional mixers and grinders. Other units using smaller sized bars operating at higher, though still audible, frequencies have also been built.

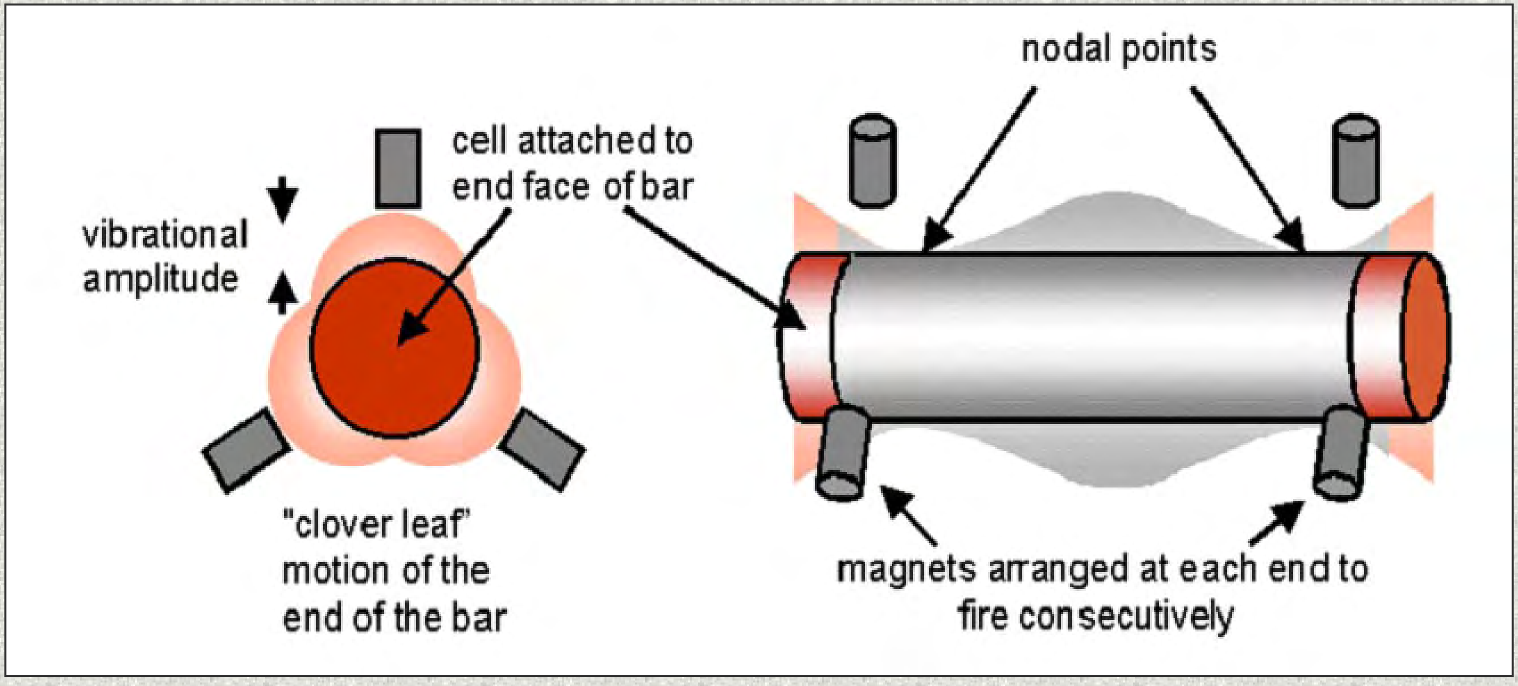


Figure 3.6: Vibrating Bar Transducer

4. REACTOR DESIGN AND SCALE UP

The design of sonochemical reactors and the rationale for the scale up of successful laboratory ultrasonic experiments are clear goals in sonochemistry and sonoprocessing. Indeed the progress of sonochemistry in green and sustainable chemistry is dependent upon the possibility of scaling up the excellent laboratory results for industrial use. The first step in the progression of a sonochemical process from laboratory to large scale is to determine whether the ultrasonic enhancement is the result of a mechanical or a truly chemical effect. If it is mechanical then ultrasonic pre•treatment of slurry may be all that is required before the reacting system is subjected to a subsequent conventional type reaction. If the effect is truly sonochemical however then sonication must be provided during the reaction itself. The second decision to be made is whether the reactor should be of the batch or flow type. Whichever type is to be used there are only three basic ways in which ultrasonic energy can be introduced to the reacting medium.

- Immerse reactor in a tank of sonicated liquid *(e.g. flask dipped into a cleaning bath)*

- Immerse an ultrasonic source directly into the reaction medium *(e.g. probe placed in a reaction vessel*)
- Use reactor constructed with vibrating walls *(e.g. a tube operating through radial vibrations)*

4.1 Batch Treatment

The obvious batch treatment processor is the ultrasonic cleaning bath which is a readily available source of low intensity ultrasonic irradiation generally at a frequency of around 40kHz. A reactor based on this design might require adaptation to provide chemically resistant walls, a sealed lid for work under an inert atmosphere and mechanical stirring. Using this system for large volume treatment the acoustic energy entering the reaction would be quite small and any stirrer and fittings in the bath would cause attenuation of the sound energy.

An alternative configuration would involve using a submersible transducer assembly which have been used for many years in the cleaning industry. It consists of a sealed unit within which transducers are bonded to the inside of one face and can be designed to fit into any existing reaction vessel.

4.2 Flow Systems

Flow Systems are generally regarded as the best approach to industrial scale sonochemistry. The general arrangement would consist of a flow loop outside a normal batch reactor which acts as a reservoir within which conventional chemistry can occur. Such an arrangement allows the ultrasonic dose of energy entering the reaction to be controlled by transducer power input and flow rate (residence time). Temperature control is achieved through heat exchange in the circulating reaction mixture.

Pipes of various cross•sectional geometry can be converted to flow processors by generating ultrasonic vibrations through their walls. The length of pipe must be accurately designed so that a null point exists at each end and it can then be retro•fitted to existing pipework. Such systems are capable of handling high flow rates and viscous materials. There are four common cross-sectional geometries: rectangular, pentagonal, hexagonal and circular. The pentagonal pipe provides a fairly uniform ultrasonic field since the energy from each irradiating face is reflected at an angle from the two opposite faces. The other configurations provide a "focus" of energy in the centre where direct energy and that reflected from the opposite wall meet.

4.3 References

1 *Practical Considerations for Process Optimisation*, by T.J.Mason and E.Cordemans de Meulenaer, *Synthetic Organic Sonochemistry*, ed J-L.Luche, Plenum Press, 301-328 (1998).

2 *The design of ultrasonic reactors for environmental remediation*, T J Mason, Advances in Sonochemistry, 6, Ultrasound in Environmental Protection, ed. T.J.Mason and A.Tiehm, Elsevier, 247-268 (2001).

3 *High Powered Ultrasound in Physical and Chemical Processing*, T.J.Mason, New Acoustics – Selected Topics, eds C.Ranz-Guerra and J.A.Gallego-Juarez, Biblioteca de Ciecias, 7, Consejo Superior de Investigaciones Cientificas, 105-138, (2003).

4 *A novel angular geometry for the sonochemical silver recovery process at cylinder electrodes*, B.G. Pollet, J.P. Lorimer, S.S. Phull, T.J. Mason and J.-Y. Hihn, Ultrasonics Sonochemistry 10, pp 217-222 (2003).

5. EXAMPLES OF RESEARCH PROJECTS

“Prospects for scale-up in the ultrasonic extraction of natural materials”
“Large scale sonochemical processing”

"Ultrasonic intensification of chemical processing and related operations"
"Sonic and ultrasonic removal of chemical contaminants from soil in the laboratory and on a large scale"

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information

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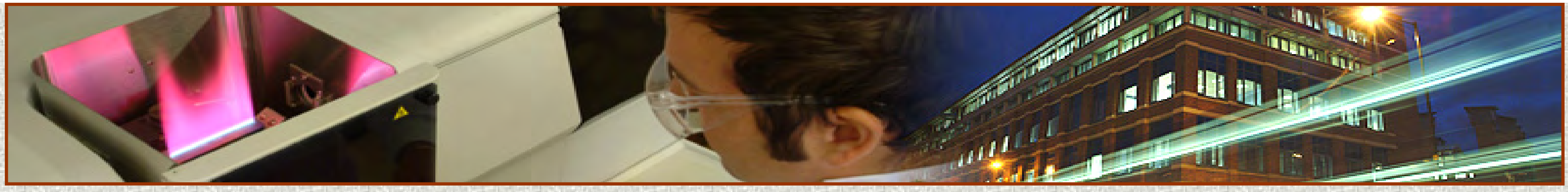

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- Home
- Introduction
- Research
- Electro
- Therapy
- Food
- Synthesis
- Green
- Materials
- Group

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Publications

Advances in Sono

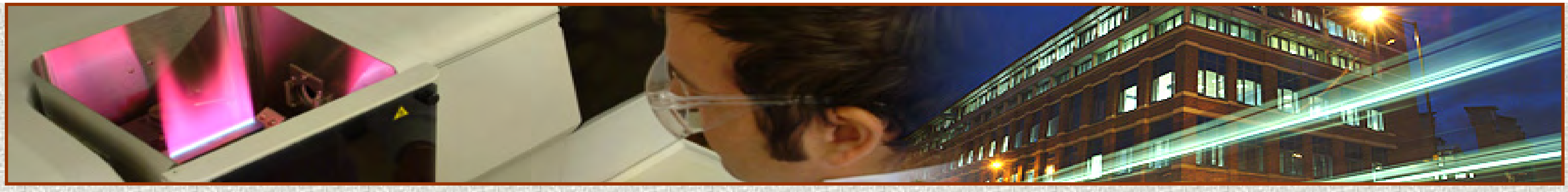
Books

Chapters

Links

Contact information

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Research

- Home
- Introduction
- Research
- Electro
- Therapy
- Food
- Synthesis
- Green
- Materials
- Group

AT THE SONOCHEMISTRY CENTRE AT COVENTRY UNIVERSITY

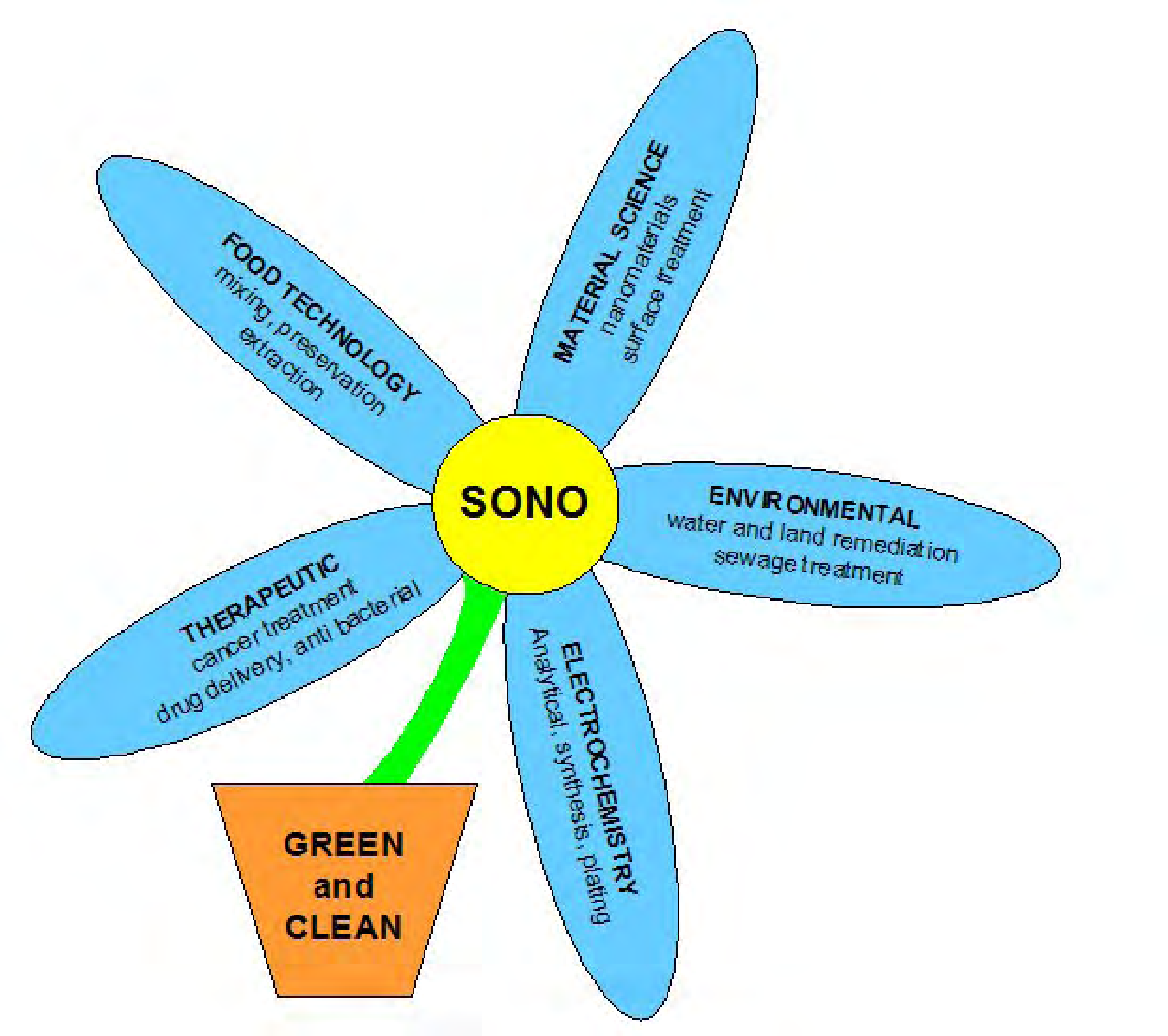
'The Home of Sound Science'

The Sonochemistry Centre in Coventry University was established in 1994 it has achieved a position of international excellence in a variety of applications of power ultrasound in chemistry and processing technologies. Amongst these applications are: Environmental Protection, Electrochemistry, Food Processing, Material Science and Therapeutic Ultrasound.

The Centre is a national and international resource base for topics related to power ultrasound and will provide expertise on applications of sonochemistry to academic institutions, companies and government organizations.

Since the end of the 1980's Sonochemistry has grown and expanded well beyond what might be considered to be "pure" chemistry as shown below.

- Publications
- Advances in Sono
- Books
- Chapters
- Links
- Contact information



Background information on sonochemistry can be found in this website below under the headings:

- ***General introduction to ultrasound and sonochemistry***
- ***Cavitation – the driving force in sonochemistry***
- ***Transducers – the devices which provide acoustic energy***

The ***team of researchers*** within the sonochemistry group (May 2008):

Left to right: Bilal Mkhlef (MSc Student), Dr Andy Cobley (Team Leader, Materials), Professor Tim Mason (Director of the Sonochemistry Centre), Dr Larysa Paniwynk (Associate Director of the Sonochemistry Centre), Anas Al Abachi (PhD student), Dr Eadaoin Joyce (Team Leader, Environment), Amna Al-Hashimi (PhD Student), Dr John Graves (Research technician), Veronica Saez Bernal (Research technician), On-anong Larpparisudthi (PhD student), Mohammed Alarjah (Research technician), Xiaoge Wu (PhD student), Dr. Fakhradin Mirkhalaf (Team Leader, Electrochemistry)

The team are highly-qualified and experienced specialists in a wide range of applications of Sonochemistry, some of which are shown below:

- ***Chemical Synthesis*** new methods, green chemistry and catalysis
- ***Electrochemistry*** analysis, plating, synthesis
- ***Environmental Protection*** remediation of air, land and water
- ***Food Technology*** drying, mixing, preservation ***and extraction of raw materials from plants***
- ***Materials Science*** ***nanoparticles, polymer science and technology***
- *Microbiology* **modification of cells and enzyme action**
- ***Reactor Design and Scale-Up*** optimization of effects
- ***Therapeutic Ultrasound*** cancer treatment and drug delivery

Most of these topics have received UK and EU funding together with industrial sponsorships that have generated both high-quality research, review papers and textbooks, and new, safer and cost-effective chemical processes in Industry. We have carried out many projects worldwide for our clients, many of whom are household names (e.g. DEFRA, DTI, Unilever, Yorkshire Water) and implemented some new concepts and processes throughout Industry.

Any enquires about M.Sc and Ph.D programes in the above topics are available in the Sonochemistry Centre and for enquires please contact: t.mason@coventry.ac.uk

General reading material can be found in the following texts:

Sonochemistry, by T.J.Mason, Oxford University Primer Series No 70, Oxford Science Publications, pp 92, 1999, ISBN 0 19 850371 7.

Applied Sonochemistry, by T.J.Mason and J.P.Lorimer, Wiley VCH (2002) ISBN 3-527-30205-0

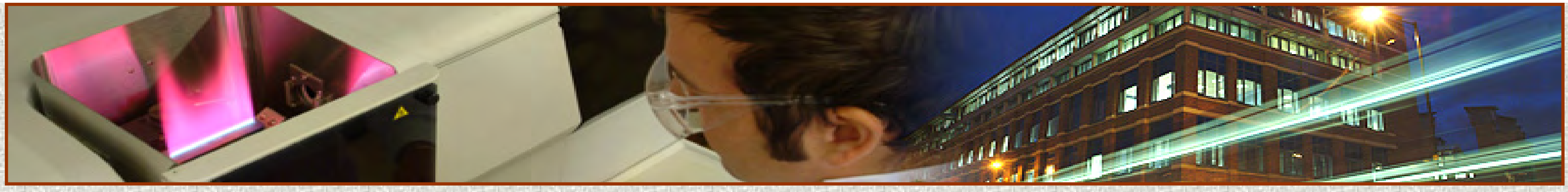
Practical Sonochemistry, (2nd Edition) by T.J.Mason and D Peters, Ellis Horwood Publishers (2002) ISBN 1-898563-83-7.

Other publications from the group can be found under:

- ***Advances in Sonochemistry a series of monographs***
- ***Books and chapters on sonochemistry***
- ***Research papers in sonochemistry***

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Electro

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ULTRASOUND IN ELECTROCHEMISTRY: SONOELECTROCHEMISTRY

Recent studies have demonstrated that there are several aspects of ultrasound which recommend its use in conjunction with electrochemical processes

- Ultrasonic degassing limits gas bubble accumulation at the electrode.
- Ultrasonic agitation (*via* cavitation) disturbs the diffusion layer and stops the depletion of electroactive species.
- Ultrasonic agitation provides more even transport of ions across the electrode double layer.
- Ultrasonic irradiation continuously cleans and activates the electrode surfaces.

These improvements include enhanced diffusion processes, increased yields, increased current efficiencies, increased limiting currents, lower overpotentials and improved electrodeposition rates. Whilst there may be different origins for the variety of these effects, one well-characterized effect of ultrasonic irradiation is the generation and subsequent collapse of cavitation bubbles both within the electrolyte medium and near to the electrode surface of the electrochemical cell. The electrode surface causes asymmetrical collapse of a bubble which in turn leads to the formation of a high velocity jet of liquid which is directed toward the surface. This jetting is thought to lead to the destruction of the mass transfer boundary layer at the electrode. This improves the overall mass transfer of the system and, as a consequence, the reaction rates at the electrodes.

Electroplating

Publications

Advances in Sono

Books

Chapters

Links

Contact information

Early research into the field of sonoelectrochemistry seems to have been carried out mainly by metallurgists concerned with improving the efficiency of electroplating. Using the simple method of directly sonicating the plating bath considerable savings are possible in processing costs through improvements *via* a shortening in process time, an increase in the deposition rate and a reduction in the plating current which occurs in conventional electroplating due to polarisation. Research in this domain continues towards improvements in both electroplating and electroless plating.

Electrosynthesis

Investigations into the influence of ultrasound on electrode reactions and electrosynthesis are of more recent origin. For the reasons outlined above the interfacing ultrasound with electrochemistry appears to hold a lot of potential and the field of sonoelectrochemistry is set to make new strides. For example, the electrochemical oxidation of Fe^{2+} to Fe^{3+}, $Fe(CN)_6{}^{4-}$ to $Fe(CN)_6{}^{3}$ and Cr^{3+} to Cr^{4+} have been investigated and the yields and current efficiencies for the reactions were studied at a current density of 0.25 A/mm^2. It was found that ultrasound accelerates the process and increases the current efficiency and also raises the limiting current density considerably, thus causing a reduction of the diffusion layer thickness resulting in the increased efficiency of the process.

The application of ultrasound on electrochemical polymerisation of conducting polymers has also been studied. In the case of the electrochemical polymerisation of thiophene ultrasonic irradiation resulted in an improvement of the polymer yield and in a lowering of the anode potential during polymerisation. The polythiophene films that are produced using sonoelectrochemistry have been shown to be flexible and tough in contrast to the more brittle forms produced using conventional technology.

The effects of ultrasound on electrochemical processes suggest significant benefits. These include modifications to the chemistry of reactions at the electrode and greatly increased current efficiencies. One major result of these studies could be that, in the future, industrial electrochemistry might become a more attractive proposition.

1 *Sonoelectrochemistry*, T.J. Mason, J.P. Lorimer and D.J. Walton, Ultrasonics, 28, 333•337 (1990).

2 *The Applications of Ultrasound in Electroplating*, J.P. Lorimer and T.J. Mason, Electrochemistry, 67, 924-930 (1999).

3 *The Effect of Ultrasonic Frequency and Intensity upon Electrode Kinetic Parameters for the $Ag(S_2O_3)_2{}^{3-}$/Ag Redox Couple,* B. Pollet, J.P. Lorimer, S.S. Phull, T.J. Mason and D.J. Walton, The., J. Applied Electrochem, 29, 1359 (1999).

4 *Sonoelectrochemical Recovery of Silver from Photographic Processing Solutions*, B. Pollet, J.P. Lorimer, S.S. Phull and J. Y. Hihn, , *Ultrasonics Sonochemistry,* 69, 7 (2000).

5 *Sonoelectrochemical effects in electro-organic systems*, D.J. Walton, J. Iniesta, M. Plattes, T.J. Mason, J. P. Lorimer, S. Ryley, S.S. Phull, A. Chyla, J. Heptinstall, T. Thiemann, H. Fuji, S. Mataka and Y. Tanaka, Ultrasonics Sonochemistry 10, 209-216 (2003).

6 *The sonoelectrooxidation of thiophene s-oxides*, J. Iniesta Valcarel, D.J. Walton, H. Fujii, T. Thiemann, Y. Tanaka, S.Mataka, T.J.Mason, J. P. Lorimer, Ultrasonics Sonochemistry 11, 227-232 (2004).

Examples of projects

“Ultrasonically assisted removal of metals from wastewater”

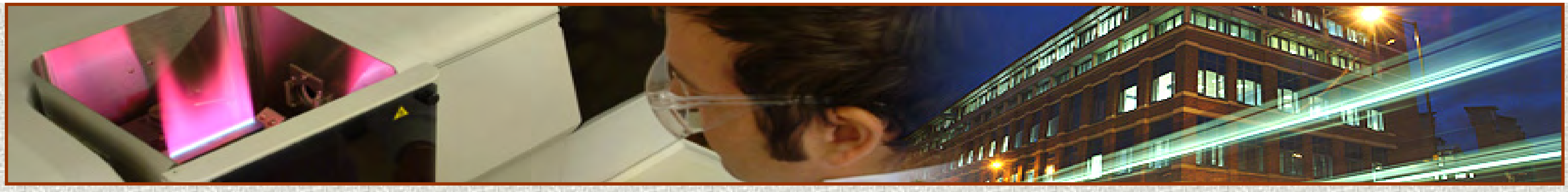
“The effect of ultrasound in combination with uv radiation and/or electrolysis for the biological decontamination of potable water”

“The effect of ultrasound on trivalent chrome plating”

“Preparation of a carbon-based composite electrode for the purpose of electrochemical degradation of chlorinated phenols”

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Therapy

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THERAPEUTIC ULTRASOUND

The use of high frequency ultrasound (around 5MHz) in medical imaging is a routine part of antenatal medicine to obtain an ultrasonic scan of the foetus in the womb. Such "diagnostic" ultrasound uses a refined pulse echo technique based upon the fact that sound waves are reflected to varying degrees from the interfaces between different tissue, blood and bone in the body. The reflections are processed to give a visual image.

At lower ultrasonic frequencies energy inputs can be increased to a level where chemical effects become possible and this has given rise to a new field of medicine called therapeutic ultrasound. Such is the interest and expansion in this field of medicine that a new society devoted to the promulgation of the general area of ultrasound in non-diagnostic medicine has been established under the title "International Society for Therapeutic Ultrasound". The society has a scientific committee chaired by Dr. G. ter Haar, Head of Therapeutic Ultrasound, Royal Marsden Hospital, Sutton, UK and Professor Mason is a member of the committee.

Two purely mechanical applications of ultrasound have been in use for many years.

- In Dentistry a common piece of equipment is an ultrasonic device used for cleaning and descaling teeth. There has been research into the use of the same equipment for assisting in the curing of (glass ionomer) white filling material.
- In the hospital operating theatre another ultrasonic device is proving to be most useful – the ultrasonic scalpel. This device has a scalpel blade which vibrates ultrasonically and as a result reduces significantly the overall force required to cut. It also gives precise cutting and induces coagulation with

minimal lateral tissue damage

Ultrasonic Tooth Descaler

Photo courtesy of Cavitron

Ultrasonic Scalpel

Photo courtesy of Ethicon Endo-Surgery

>

Sono-Dynamic Therapy (SDT)

Some chemicals e.g. porphyrins give out free radicals on treatment with light (Photo Dynamic Therapy) and so cancers which absorb the chemical can be treated wih light to accelerate kill. Ultrasound can do a similar job with the advantage that ultrasound can penetrate the body and so reach tumours without the need for them to be exposed directly to light. This is known as Sono-Dynamic Therapy (SDT). For such treatment the patient has a chemotherapy drug administered, the drug is taken up by the cancer cells and these are then targeted with therapeutic ultrasound to provide enhanced kill.

Transdermal drug delivery

Physiotherapists use ultrasound at frequencies of between 1 and 3 MHz to treat muscle injury because applying ultrasound to the skin surface provides a localised heating/massage effect to the injury site. However the accompanying vibration also causes a temporary weakening of the barrier formed by the stratum corneum. Ultrasound can alter the barrier function of the skin to permit the administration of drugs not normally permeable through the skin layer. This approach to drug delivery shows potential for the transdermal delivery of:

- antibiotics - where oral administration would not deliver the appropriate dose to the affected area, e.g. in severe acne or gangrene.
- non-steroidal anti-inflammatory drugs - when taken orally over a long period may cause gastro-duodenal ulcers in many patients.

- protein drug molecules - where enhancement of drug penetration through the skin could eliminate the need for injectable forms of insulin which currently 125 million people deal with on a daily basis.
- Activation of dermal patches – for a rapid and instantaneous extra release of drugs through the skin

Improved uptake of drugs into cells

Several in vitro studies show that low-intensity ultrasound can increase the uptake of chemotherapeutic agents to cancer cells. This increased intracellular drug accumulation is believed to be due to an alteration in the cell membrane permeability mediated by ultrasound.

HIFU

The use of a focused array of transducers for use in cancer therapy (High-Intensity Focused Ultrasound known as HIFU) has been under investigation for many years. In principal the array is constructed to produce a focus within the body in the approximate shape of an elongated rugby football a few millimetres or so in cross section and several millimetres long. The focus can be targeted accurately on cancerous tissue within the body and, through the energy intensity generated at the focus, thermally destroy it. In a sequence of exposures the focus can be moved to cover the whole of the affected region. At lower powers the focused ultrasound can also be used to enhance the action of chemotherapy agents such as a porphyrin which are known to be affected region and is thought to promote the types of radical reaction which are generally considered to be involved in chemotherapy.
To obtain an accurate focal point within the body which is accurately targeted at the correct point in space is not easy because the sound must pass through various different tissues.

"Review of research into the uses of low level ultrasound in cancer therapy" Tinghe Yu, Zhibiao Wang and T. J. Mason (2004) *Ultrasonics Sonochemistry*, 11, 95-103.

"The use of a microbubble agent to enhance rabbit liver destruction using high intensity focused ultrasound" Tinghe Yu, Shuhua Xiong, T.J. Mason and Zhibiao Wang (2006) *Ultrasonics Sonochemistry* 13, 143-149.

"Ultrasound: a chemotherapy sensitizer", Tinghe Yu, Shugang Li, Jie Zhao and T.J.Mason (2006) *Technology in Cancer Research and Treatment*, (2006) 5, 51-60 .

Examples of Projects

"The influence of ultrasound on the uptake of chemotherapeutic agents into cells"

"Effect of ultrasound on transdermal drug delivery systems"

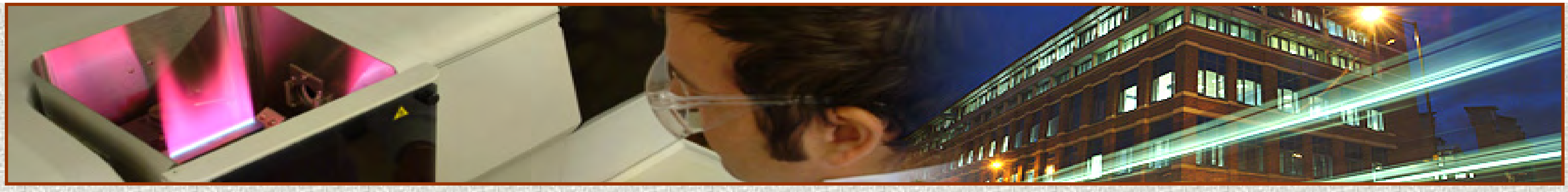
"Measurements of power output for therapeutic ultrasound devices"

"The effect of ultrasound on the permeability of diclofenac through cellulose membrane and EpiDerm tissue from a customized gel formulation in comparison to Voltarol Emugel"

“Ultrasonically assisted curing of glass ionomer cements”

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Food

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ULTRASOUND IN FOOD TECHNOLOGY

Nowadays, power ultrasound is considered to be an emerging and promising technology for industrial food processing. Probably the first question that might be asked about applications of ultrasound in food technology is why use ultrasound. For the answer to this we need only think of two properties of sound to appreciate the possibilities. The first is the use of sound as a diagnostic tool e.g. in non-destructive evaluation and the second is the use of sound as a source of energy e.g. in sonochemistry. These applications involve different frequency ranges of ultrasound and the uses of both ranges in the food industry are an active subject for research and development.

Until recently the majority of applications of ultrasound in food technology involved non-invasive analysis with particular reference to quality assessment. Such applications use techniques that are similar to those developed in diagnostic medicine, or non-destructive testing, using high frequency (>1 MHz) low power (<1 W/cm^2) ultrasound. Examples of the use of such technologies are to be found in the location of foreign bodies in food the analysis of droplet size in emulsions of edible fats and oils and the determination of the extent of crystallization in dispersed emulsion droplets.

In recent years food technologists have discovered that it is possible to employ a more powerful form of ultrasound (>5 W/cm^2) at a lower frequency (generally around 40 kHz). This is usually referred to as power ultrasound and its history can be traced back to 1927 when a paper was published entitled "The chemical effects of high frequency sound waves: a preliminary survey" which described the development of

Publications
Advances in Sono
Books
Chapters
Links
Contact information

power ultrasound for use in a range of processing including emulsification and surface cleaning (Richards and Loomis, 1927). By the 1960's the uses of power ultrasound in the processing industries were well accepted and this interest has continued to develop (Abramov 1998, Mason 2000, Mason and Lorimer 2002}. In this chapter we will concentrate on possible applications of power ultrasound in the food industry, an indication of the breadth of which is shown in the Table below

Mechanical Effects	Chemical and Biochemical Effects
crystallisation of fats, sugars etc degassing destruction of foams extraction of flavourings filtration and drying freezing mixing and homogenisation precipitation of airborne powders tenderisation of meat	bactericidal action effluent treatment modification of growth of living cells alteration of enzyme activity sterilisation of equipment

The potential use of this novel technology to produce permanent changes in food materials in liquid systems is through the generation of intense cavitation. This can lead to the inactivation of microorganisms and enzymes for food preservation or decontamination by ultrasonic irradiation demonstrates the benefits of ultrasound (alone or combined with heat and high-pressure techniques) as a food preservation tool. In addition there are an increasing number of industrial processes that employ power ultrasound as a processing aid including the mixing materials; foam formation or destruction; agglomeration and precipitation of airborne powders; the improvement in efficiency of filtration, drying and extraction techniques in solid materials and the enhanced extraction of valuable compounds from vegetables and food products.

1 *The use of ultrasound in food technology*, T.J.Mason, L.Paniwnyk and J.P.Lorimer, Ultrasonics Sonochemistry, 3, pp253-260 (1996).
2 *Ultrasound as a Preservation Technology*, T.J.Mason, L.Paniwnyk; F.Chemat, Chapter 16 of Food Preservation Techniques, eds P.Zeuthen and L.B˝gh-S˝rensen, pp 303-337, Woodhead Publishers (2003).
3 *Potential for the use of ultrasound in the extraction of antioxidants from Rosmarinus officinalis for the*

food and pharmaceutical industry, S.Albu, E.Joyce, L.Paniwnyk, J.P.Lorimer and T.J.Mason,. Ultrasonics Sonochemistry 11, pp 261-265 (2004).

4 *Applications of Ultrasound*, T.J.Mason, Enrique Riera, Antonio Vercet and Pascual Lopez-Buesa, Chapter 13 of Emerging Technologies for Food Processing, ed Da-Wen Sun, pp 323-352, Elsevier (2005).

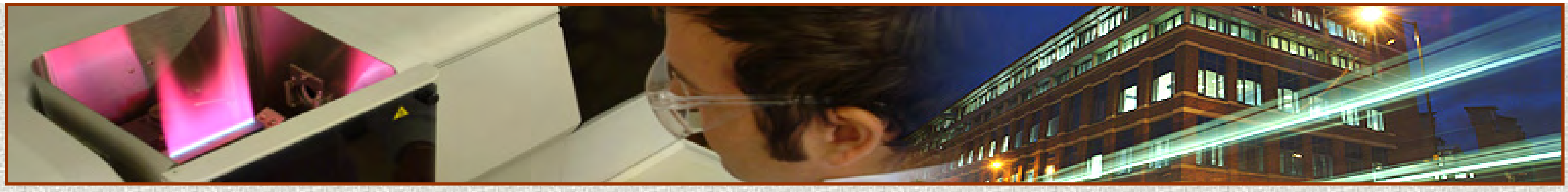
Examples of projects

“Effect of ultrasound on the physicochemical and functional properties of whey and soy protein isolate and concentrate, alpha-lactalbumin and whey protein hydrolysate suspensions”

“Accelerated Drying of Mushrooms, Brussels sprouts and Cauliflower by Means of Power Ultrasound and its impact on Food Quality”

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Synthesis

Home
Introduction
Research
Electro
Therapy
Food
Synthesis
Green
Materials
Group

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SONOCHEMISTRY IN CHEMICAL SYNTHESIS

The chemical effects of ultrasound cannot be the result of any direct coupling of the sound field with the chemical species involved on a molecular level since the sound frequencies most commonly employed (20•40kHz) are several orders of magnitude too low even for the excitation of rotational motion. Thus, there is no direct interaction between the ultrasonic wave and matter and so there must be an indirect interaction via a process of energy concentration that can then affect molecules. Such a process is acoustic cavitation which provides bubbles which act as microreactors (see introduction to sonochemistry). Here there are four possible reaction sites associated with the collapse (Table):

Reaction site	Chemical effects
Hot gas phase (inside the bubble)	(a) sonolysis of solvent or volatile compounds (formation of radicals/activated molecules) (b) radical reactions
Liquid shell (around the bubble)	(a) pressure/temperature gradients and/or electrical fields cause the sonolysis of non-volatile compounds (b) radical reactions caused by radicals expelled from the bubble interior

Publications
Advances in Sono
Books
Chapters
Links
Contact information

Liquid (surrounding the bubble)	(a) solid or dissolved reactants which may react with reactive species expelled from the bubble interior (b) consecutive radical reactions (c) emulsion formation of non-miscible liquids (d) intense mixing of bubbled gas and liquid (e) mechanical effects on solids and metals (surface erosion and cleaning, activation of metals, degradation of polymers, enhanced particle transport) (f) accelerated mass and heat transfer as well as fluid flow (g) disturbing of solvation layers (h) single electron transfer (SET) seems to be favoured
Liquid droplets (inside the bubble)	(a) high pressure/temperature cause sonolysis of non-volatile compounds (b) liquid is heated (there is a suggestion that supercritical fluids may be involved but this a matter of controversy. It is argued that the collapse time is too short for a supercritical state to evolve) (c) radical reactions caused by radicals expelled from the bubble interior

Based on the physical effects just described and observed during bubble collapse (or to some extent during stable cavitation), there are several possible chemical effects at the reaction sites. These may be divided into two groups: radical effects and mechanical effects.

(1) Radical formation

The drastic conditions inside the bubble just described stimulate the generation of radicals. The source of the radical formation may be the solvent vapour or volatile compounds in the liquid. Primary radicals may induce secondary reactions: they may be converted to other radicals, may initiate a radical chain, can react with substrates (including the solvent itself) or attack other radicals (including recombination). Such radical reactions may occur in the bubble interior, in the bubble liquid shell/interface or in the bulk solution. Examples are the generation of OH-radicals in water, the formation of hydrogen peroxide in water, the hydroxylation of aromatic compounds by OH-radicals or the acceleration of the initiation of radical polymerisation. Furthermore, volatile substrates may be thermally degraded inside or at the interface of a collapsing bubble. Organic solvents will also slowly decompose on sonication but solvent decomposition normally provides only a minor contribution to any sonochemical reaction that is taking place in the medium.

However, it should be noted that the primary formation of radicals inside the bubble is a relatively slow process having reaction rates that are of the order of 10^{-4} to 10^{-5} $mol.l^{-1}.min^{-1}$. Moreover, secondary radical attacks on a substrate are not influenced by ultrasound. Consequently, only those radical reactions causing a radical chain or those requiring only a small number of radicals compared to the substrate (e.g. destruction of polymers) are of interest to a synthetic chemist.

(2) Mechanical effects

Results indicate that ionic chemical reactions in solution are not influenced by sonication unless they involve solids, metals or multi-phase conditions however the physical effects generated in solution around the collapsing bubble can effect chemical species in solution, solid particles in suspension or surfaces.

Homogenous systems

The following effects in a homogeneous solution are of relevance:

- enhanced mass and heat transfer due to microstreaming or acoustic streaming
- degradation of large molecules such as polymer chains due to shear forces induced by shock waves and microstreaming
- disturbance of the solvation layer around neutral and charged molecules
- degassing of liquids occurs if dissolved gas is present or if gaseous reaction products are generated in the course of a chemical reaction.

Heterogenous systems

The most successful applications of ultrasound have been found in the field of heterogeneous chemistry involving solids and metals. This is due to the mechanical impact of ultrasound on solid surfaces. In conventional chemistry there are several problems associated with conventional reactions involving solids or metals

- small surface area of the solid/metal,
- penetration of reactants into deeper areas is not possible,
- oxide layers or impurities can cover the surface,
- reactants/products have to diffuse onto and from the surface,
- reaction products can act as deposit on the surface and prevent further reactions.

The mechanical effects of ultrasound offer an opportunity to overcome these problems:

- hard oxide layers on soft metals are broken by plastic deformation of the surface
- oxide layers on hard metals (low cohesion) are removed
- impurities are removed in the same manner (surface cleaning)
- “break up“ of the surface structure allowing penetration of reactants and/or release of materials from surface
- degradation of large solid particles due to shear forces induced by shock waves and microstreaming leads to reduction of particle size and increase of surface area
- microstreaming produces collisions of those particles smaller in size than a cavitation bubble
- accelerated motion of suspended particles
- erosion of solid surfaces due to jetting, cleaning of solid surfaces due to shock waves and/or jetting
- intensification of mass transfer from and onto the surface by microstreaming (removing of impurities and small particles, accelerated transportation of reactants/products)
- reduction of the induction time

A problem when dealing with syntheses in heterogeneous systems involving immiscible liquids (e. g. aqueous/organic mixtures) is that the reagents are often dissolved in different phases. Any reaction between these species can only occur in the interfacial region between the liquids and this is a very slow process. Sonication can be used to produce very fine emulsions from immiscible liquids. This is the result of cavitational collapse at or near the interface which causes disruption and impels jets of one liquid into the other to form the emulsion.

The normal method of inducing a reaction between species dissolved in different immiscible liquids (usually water and an organic solvent) is through the use of a phase transfer catalyst (PTC) which will bring both reactants into the same, usually organic, phase. There are however two drawbacks to the use of such catalysts in that some of the more specialised PTC reagents are expensive and all PTC's are potentially dangerous since they can, by their very nature, transfer chemicals from water into human tissue. Sonication of immiscible liquids generates extremely fine emulsions which result in very large interfacial contact areas between the liquids and a corresponding dramatic increase in the reactivity between species dissolved in the separate liquids. This effect can be used to either replace the need for a PTC or reduce dramatically the quantity required.

The following effects in liquid/liquid or gas/liquid systems are of relevance:

- rapid emulsification due to intense mixing by microstreaming/jetting/shock waves and interaction of the sound field with the liquid/liquid boundary
- accelerated transportation of reactants or products onto/from the phase boundary due to radiation forces and microstreaming
- intense mixing of bubbled gas and liquid
- intensification of mass and heat transport

Sonochemistry has some important *environmental* connotations. Thus, the accelerating effect of sound waves often reduces the formation of side products (*waste minimisation*), and the enhanced activation of catalysts and reagents enables the replacement or control of hazardous, highly reactive substances. Overall this results in simplified and milder procedures accompanied by *energy savings*. Essentially ultrasound can often be considered to offer a cheaper “green” route.

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Sonochemistry, T.J.Mason and P Cintas, Handbook of Green Chemistry and Technology, ed J.Clark and D.Macquarrie. 372-396, Blackwell (2002).
Practical Sonochemistry, Power ultrasound uses and applications, (2nd Edition) by T.J.Mason and D Peters, Ellis Horwood Publishers, (2002).

Examples of projects

“ The use of ultrasonic irradiation for the modification of chemical reactivity”

“The effect of ultrasound and other physical parameters on the reactivity of powders and catalysts”

“The effect of ultrasound on the polymerisation of N-vinylcarbazole”

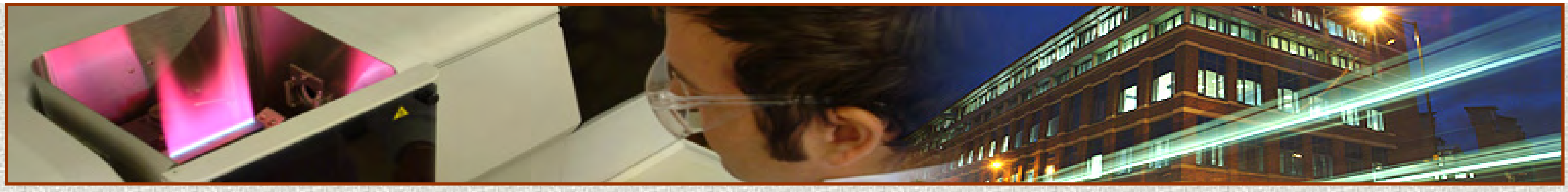
"The effect of ultrasound on the emulsion polymerisation of styrene"

“The effect of ultrasound on organic synthesis and processing from laboratory to large scale”
“Reactions of lead tetra acetate on alcohols and styrenes in the presence and absence of ultrasound”

“Sonochemistry on the surface and at the interface of materials”

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Green

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ULTRASOUND IN ENVIRONMENTAL PROTECTION AND WASTE CONTROL

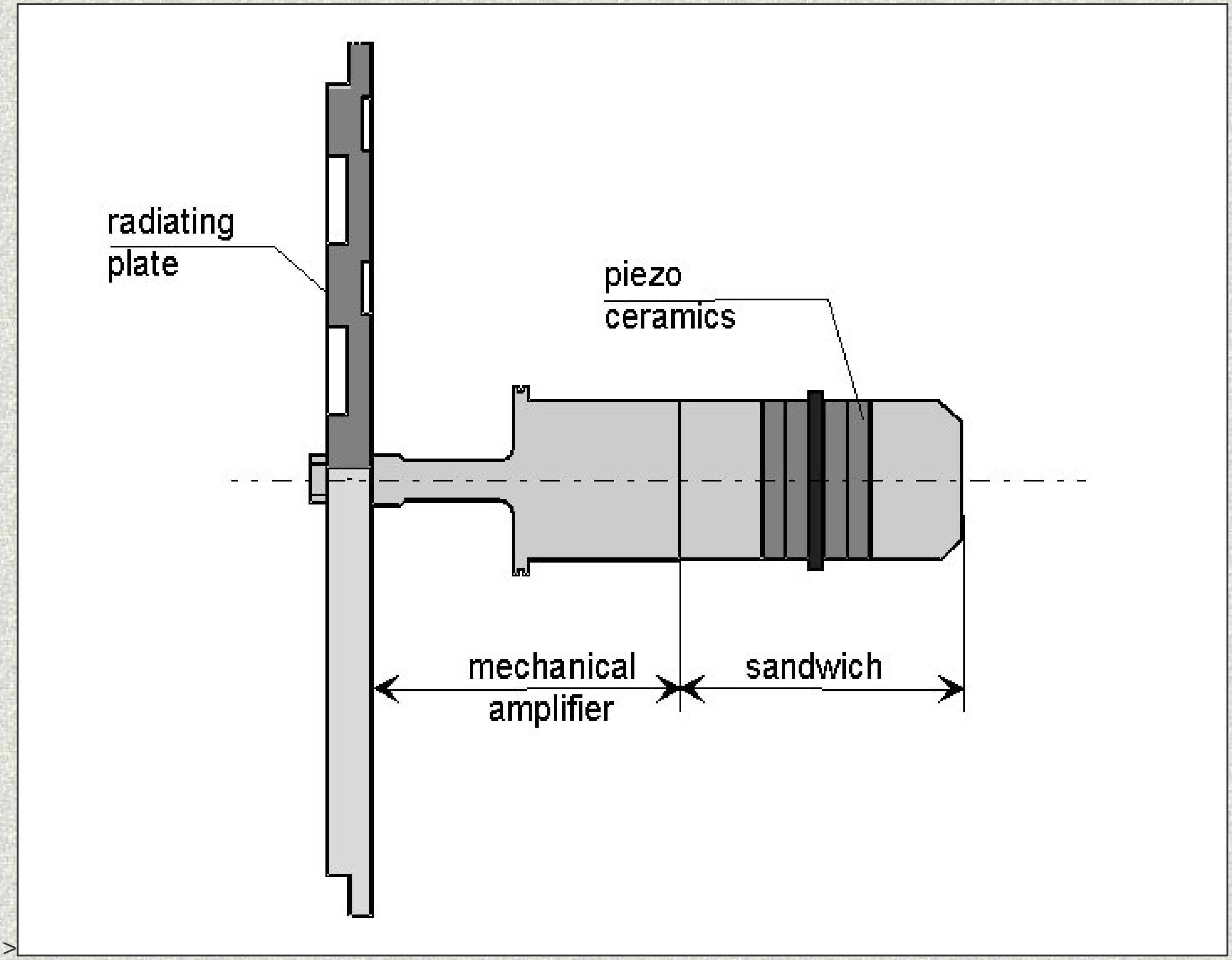
Research into the use of ultrasound in environmental protection has received a considerable amount of attention with the majority of investigations focusing on the harnessing of cavitational effects for the destruction of biological or chemical pollutants in water and the processing of sewage. The field is much broader than this however and a summary of topics is given in the Table.

Control of air-borne contamination	agglomeration of smokes and aerosols defoaming of liquids
Washing of soils	Removal of organic and inorganic contamination
Water treatment	biocidal action • *direct mechanical action e.g. cell rupture and the break-up of bacterial clumps* • *indirect mechanical action e.g. increased cell permeability to bactericide* • *stabilization and dewatering of sludge*
	Removal of chemical contamination • *direct oxidation of chemical and pesticide residues* • *in combination with other techniques e.g. ozonation, uv light*

Publications
Advances in Sono
Books
Chapters
Links
Contact information

	Removal of surface contamination and biofilms

Air Cleaning



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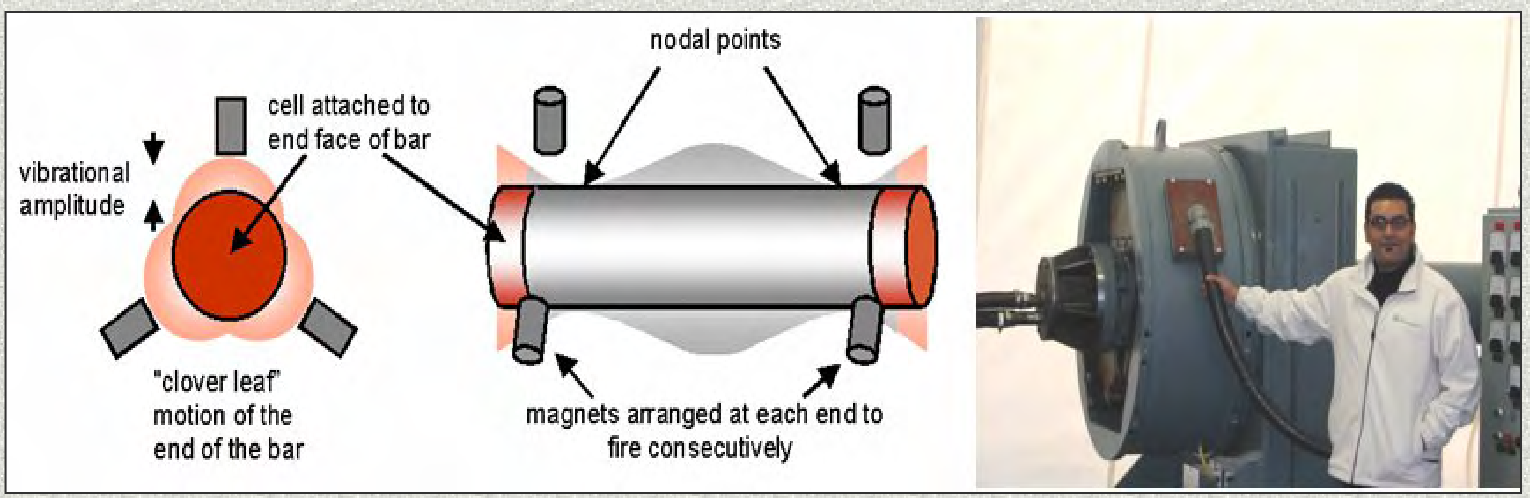
The inhalation of airborne particles is now recognized as a serious public
Using this type of device airborne ultrasound has been used for both the precipitation of airborne powders and defoaming.

Land Remediation

For contaminated soil wastes the currently available options for management and disposal are principally:

- Permanent storage in a secure landfill. This will result in a permanent retained liability by the waste generator.
- Incineration in a permitted waste incinerator. This is costly and entails the risk of atmospheric emissions.
- Soil washing to produce bulk soil with low-level contamination. However the washing process itself will produce a volume of solvent that must be treated before disposal.

For many years ultrasound has been considered as a technology to promote the process of soil washing and if subsequent disposal of the washings was considered at all this was perhaps to be a separate treatment. An integrated system has been developed in Canada (by Sonic Environmental Solutions Inc.) for large scale continuous processing using acoustic frequencies in the audible range that incorporates the clean-up of the washings and recycling of the solvent. The equipment itself affords vibrational amplitudes considerably larger than those available using ultrasound and it has proved to be particularly efficient for the removal and destruction of PCB contaminants in soils. The equipment generates vibrational energy through the use of resonant bending modes in a large cylindrical steel bar. The bar is driven into a cloverleaf type of motion by firing six powerful magnets (three at each end of the bar) in sequence. The bar is supported by air springs so that the ends and the centre are then caused to rotate at a resonance frequency depending on its size.



One such unit, operating at a power of 75kW, drives a bar that is 4.1 metre long and 34 cm in diameter at its resonance frequency of 100 Hz. The bar weighs 3 tonnes and produces an amplitude of vibration at each end of 6 mm. For the washing of soils a mixing chamber is rigidly mounted on each end of the bar and these are used in three process areas: PCB extraction, PCB destruction and solvent recovery. The use of this generator for pilot testing has proved that processing can be achieved at a commercial scale of around 3 to 4 tonnes of soil/hour.

Water Remediation

Removal of biological contamination

Some species of bacteria produce colonies and spores, which agglomerate in spherical clusters (e.g. Bacillus subtilis). The use of a biocide can destroy microorganisms on the surface of such clusters but often leaves the innermost bacteria intact. Flocs of fine particles e.g. clay can entrap bacteria which can also protect them against disinfection [Mir, 1997]. Due to these problems alternative methods of purifying water are being investigated and amongst these the application of ultrasound is proving to be of considerable interest. Ultrasound is able to inactivate bacteria, make them more susceptible to biocides and/or deagglomerate bacterial clusters or flocs depending upon the power and frequency applied through a number of physical, mechanical and chemical effects arising from acoustic cavitation.

Removal of Chemical contamination

The mechanical effects of cavitational collapse together with the production of radical species combine to provide the essential elements for water decontamination. The primary radicals produced during the sonication of water are OH· and H· and the fate of these is quite complex (Scheme 18). The HO· radical is extremely reactive and is capable of oxidising most chemical compounds dissolved in the water. This oxidation is mainly responsible for the degradation of organic pollutants in sonicated aqueous media. The efficient generation of HO· is therefore an important goal in waste treatment.

1. *Degradation of dye effluent*, J.P.Lorimer, T.J.Mason, M.Plattes, S.S.Phull, and D.J.Walton, Pure and Applied Chemistry, 73, 1957-1968 (2001).
2. *Potential uses of ultrasound in the biological decontamination of water*, T.J.Mason, E.Joyce, S.S.Phull, and J.P.Lorimer, Ultrasonics Sonochemistry 10, pp 319-324 (2003).
3. *Ultrasound in Advanced Oxidation Processes*, T.J.Mason and C.Petrier, Chapter 8 in Advanced Oxidation Processes for Water and Wastewater Treatment, pp 185-208, ed S Parsons, IWA Publishing (2004).
4. Application of UV radiation or electrochemistry in conjunction with power ultrasound for the disinfection of water Eadaoin M. Joyce, Timothy J. Mason and John P. Lorimer , Int. J. Environment and Pollution 27, 222-230 (2006)
5. Oxygen-induced concurrent ultrasonic degradation of volatile and non-volatile aromatic compounds Christian Pétrier, Evelyne Combet and T.J.Mason, Ultrasonics Sonochemistry 14, (2007) in press.

Examples of projects

Water purification:

Advanced oxidation methods involving sonochemistry

“Degradation of water pollutants using ultrasound”

Biological decontamination

“The effect of ultrasound in combination with uv radiation and/or electrolysis for the biological decontamination of potable water”

“The effect of sonication at different frequencies on microbial disinfection usinghypochlorite”

“Controlling algae in reservoirs with ultrasound”

“Assessment of hydrodynamic cavitation methods compared with sonochemistry for the decontamination of water”.

Soil remediation

“Sonic and ultrasonic removal of chemical contaminants from soil in the laboratory and on a large scale”

Airborne pollution

“Ultrasound for the removal of dust, suppression of foam”

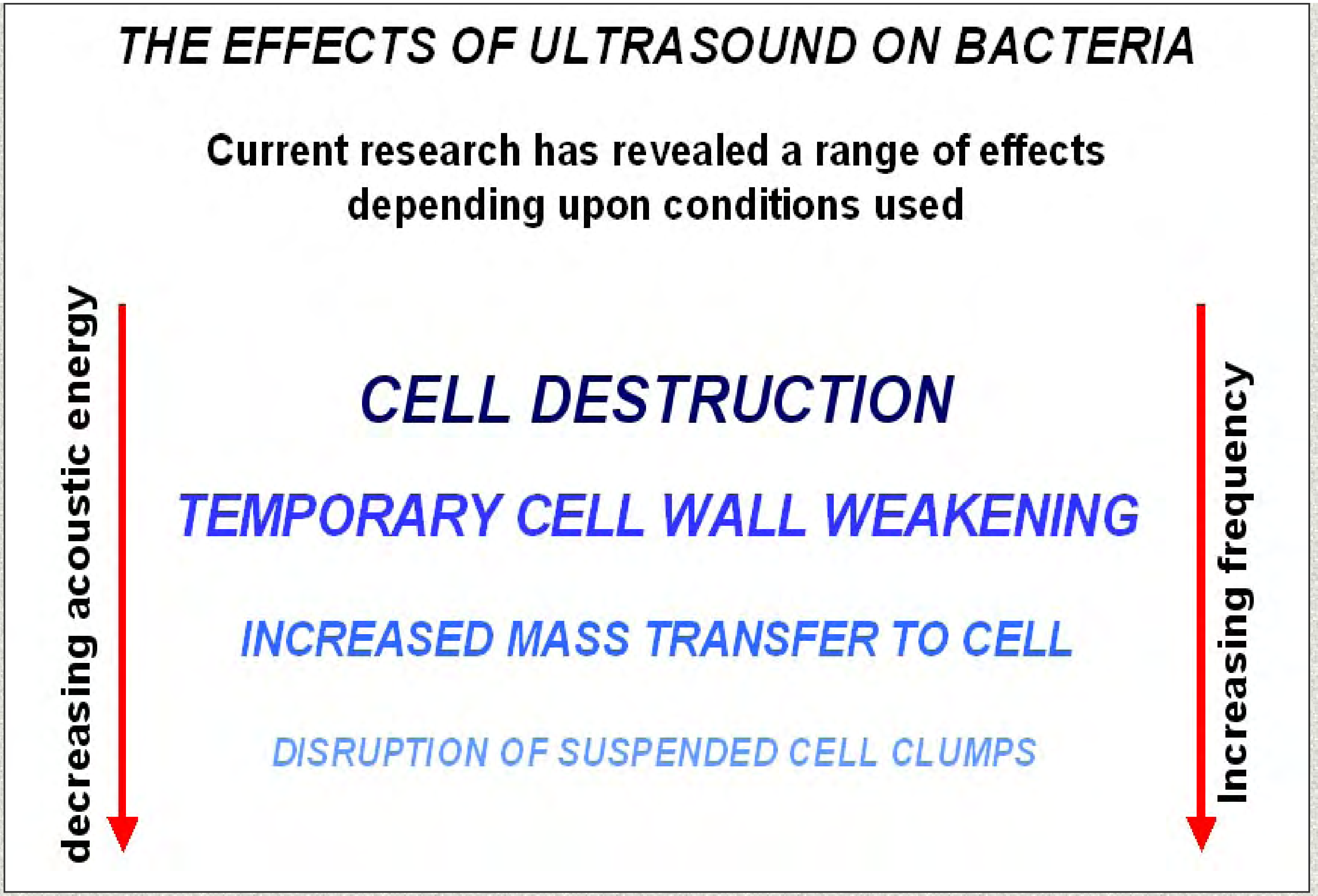
Surface Cleaning

“Membrane fouling and integrity in the municipal sector: a multi-faceted approach to their amelioration”

“Surface decontamination in the food industry”

MICROBIOLOGY

The effect of ultrasound on biological systems and biotechnological processes depends strongly on frequency, intensity and sonication time.



Low intensity effects (i.e. under conditions which occur below the cavitation threshold) are the result of microstreaming and acoustic streaming. At these intensities, where no cavitation damage will occur, the beneficial effects are:

- activation of enzymes in enzymatic reactions
- improvements in microbial reactions (e.g. fermentation)
- improvement of the bioavailability of contaminants in environmental remediation using microorganisms

Higher intensity effects are the result of cavitational damage and may be summarised as follows:

- destruction of cell walls and release of cell components into the surrounding solution (damage to cell components e.g. DNA, proteins is limited if sonication time is short)
- extraction of organic substances from plants
- emulsification of food (see Food section)
- damage of cell walls and cell components at very high intensity
- killing of microorganisms (see Environmental Remediation)
- improvement of the conventional bacterial decontamination (disinfection) of water
- destruction of biological tissue e.g. tumours or kidney stones (see Therapeutic Ultrasound)

1. *The use of ultrasound in microbiology - Sonomicrobiology*. S.S.Phull and T.J.Mason, *Advances in Sonochemistry*, Vol 5, ed. T.J.Mason, JAI Press, 175-208 (1999)
2. *Potential uses of ultrasound in the biological decontamination of water*, Mason, T.J., Joyce, E., Phull, S. S. and Lorimer, J.P., Ultrasonics Sonochemistry 10, pp 319-324 (2003).
3. The effect of sonication on microbial disinfection using hypochlorite, H. Duckhouse, T.J. Mason, S.S. Phull, and J.P. Lorimer, Ultrasonics Sonochemistry 11, 173-176 (2004).
4. A review of research into the uses of low level ultrasound in cancer therapy, Tinghe Yu, Zhibiao Wang and T.J.Mason, Ultrasonics Sonochemistry 11, 95-103 (2004).

Examples of Projects

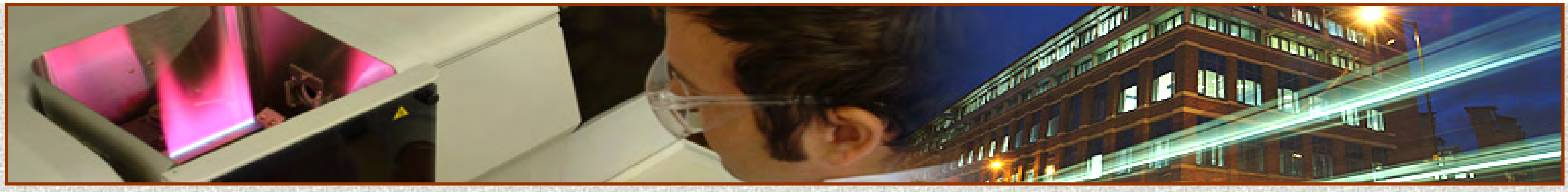
“The effect of ultrasound and ultraviolet radiation on bacterial suspensions”

“The effect of ultrasound and ultraviolet radiation on gram positive and gram negative bacteria”

“The influence of ultrasound on the uptake of chemotherapeutic agents into cells”

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Materials

Home
Introduction
Research
Electro
Therapy
Food
Synthesis
Green
Materials
Group

THE SONOCHEMISTRY CENTRE AT COVENTRY UNIVERSITY
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MATERIAL SCIENCE: THE EXTRACTION OF RAW MATERIALS FROM PLANTS

The use of plants not only as food but also as flavouring, colouring or in medicine has a long history. The interest in aromatic and medicinal plants has declined over the last half century, mainly due to the tremendous developments in the production of synthetic substitutes. Nowadays however there is a resurgence of interest in natural remedies which is in part due to some disillusionment with modern medicines and the hope that new treatments can be resurrected from ancient remedies.

Medicinal and aromatic plants provide an inexhaustible resource of raw materials for the pharmaceutical, cosmetic and food industries and more recently in agriculture for pest control. People have learned to increase the power or usefulness of herbs, by preparing medicinal compounds from them, by preserving them so that they are always available and by finding new ways to release their active constituents.

Increased efficiency in extraction leads directly to a reduction in material wastage and power ultrasound has been shown to improve extraction from plant materials. The classical techniques for extraction are mainly liquid-solid extraction by means of steam and/or organic solvents. All such techniques use relatively high temperatures and thus the energy consumption is very high and decomposition of some compounds may also occur. The use of ultrasound avoids these high temperatures and can result in enhanced component extraction at lower temperatures and in a faster time.

Publications
Advances in Sono
Books
Chapters
Links
Contact information

Plants are a source of raw chemical materials and there are real possibilities for the growing crops for specific extracts. One of the best known examples is the rubber tree. It also well known that oil plants like sunflower, rape, castor, could be not only a source of food material but also a bulk source of chemicals for the cosmetic and chemical industries. Some examples are: linalool from coriander, limonene and carvone from dill seeds, anethole from fennel seeds and α-pinene which can be separated from turpentine oil extracted from coniferous trees in quite large amounts.

1. *Comparison of conventional and ultrasonically assisted extractions of pharmaceutically active compounds from* Salvia Officinalis, M.Sali¨ovل, ¦.Toma and T.J.Mason, Ultrasonics Sonochemistry, 4, pp 131-134 (1997).
2. *Ultrasonically assisted extraction of bioactive principles from plants and their constituents*, M.Vinatoru, M. Toma and T.J.Mason, Advances in Sonochemistry, 5, ed. T.J.Mason, JAI Press, pp 209-248 (1999)
3. *Towards the industrial production of medicinal tincture by ultrasound assisted extraction*, P.Valachovic, A. Pechova, T.J.Mason, *Ultrasonics Sonochemistry*, 8, pp 111-118 (2001).
4. *Potential for the use of ultrasound in the extraction of antioxidants from* Rosmarinus officinalis *for the food and pharmaceutical industry*, S.Albu, E.Joyce, L.Paniwnyk, J.P.Lorimer and T.J.Mason,. Ultrasonics Sonochemistry 11, pp 261-265 (2004).

Examples of Projects

“The extraction of Rutin from *Sophora Japonica* using ultrasound”

“The effect of various parameters and techniques on the efficiency of extraction of antioxidant materials from the herb Rosmarinus Officinalis”

“Extraction and analysis of anti-inflammatory agents from blueberries”

“Improved Extraction of Antioxidants and Flavonoids from Natural Materials”

MATERIALS SCIENCE: POLYMER SCIENCE AND TECHNOLOGY

There are several ways in which ultrasound has been used in Polymer science and technology.

Power ultrasound in polymer technology	
Treatment of polymers	Treatment of plastics
Molecular weight reduction	Welding
Enhanced radical polymerisation	Reduction in Viscosity in Molding
Copolymerisation	Mixing of additives
Encapsulation	Surface treatment

Molecular Weight Reduction - Polymer Degradation

It has been known for some time that long chain molecules are broken down by ultrasonic waves. Although the exact mechanism by which this occurs is open to question, it is generally agreed that it is the hydrodynamic forces that are of primary importance. It is also believed that ultrasonic degradation, unlike chemical or thermal decomposition, is a non-random process with cleavage taking place at roughly the centre of the molecule and with larger macromolecules degrading the fastest. The consequence of this is that the larger molecules are preferentially degraded. It is also known that there is a limiting molecular weight below which degradation does not take place. This limiting molecular weight has the added effect of narrowing the molecular weight distribution.

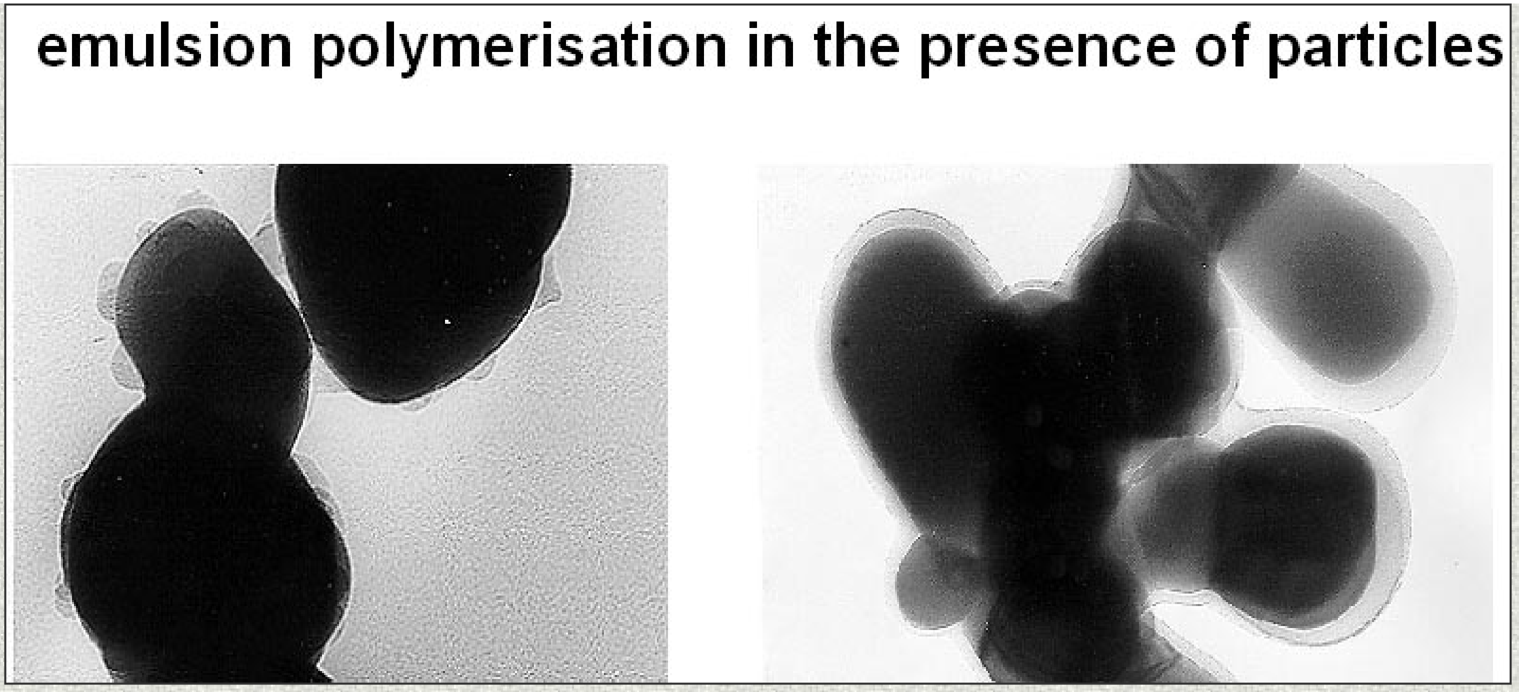
Polymer Synthesis

Early investigations into the use of ultrasound in polymer synthesis involved sonicating solutions containing a polymer and a monomer. Polymerisation was thought to be affected by utilising the shock wave energy, released on bubble collapse, to homolytically break a carbon-carbon bond in the polymer's backbone thereby producing a radical entity which could attack the monomer and polymerise by a conventional mechanism. The sonochemical generation of radicals has also been utilised to improve emulsion polymerisation

Polymer Encapsulation

Over the past 30 or so years there has been a general interest in the development of technologies for the encapsulation of fine inorganic powders with organic polymers. The general aim of encapsulation is to affect the physical properties of such powders particularly in terms of increasing their dispersability in solvents or in composite phases. For example, if the end use of the powder is to be in either the coating field or the production of speciality films, then the two factors which dictate optimum physical properties are firstly an even and small particle size of the original powder, and secondly a uniform coating of each and every particle.

Most paint formulations contain TiO_2 pigment particles produced by ball milling. Unfortunately during storage there is a problem with the re-agglommeration of the TiO_2 pigment which ultimately leads to poor coverage and a patchy appearance of the final paint product. By applying ultrasound to TiO_2 pigment in an emulsion system (water, surfactant and monomer) we were able to show that it was possible to produce the “ideal” pigment for formulation purposes where each particle was separated from its neighbour and was totally covered with polymer and had no tendency to reagglomorate (see figure below).



In the absence of ultrasound in the presence of ultrasound

Plastics Technology

There are at present only a few commercial applications of ultrasound in the plastics industry. The best known is probably the welding of thermoplastics, a process which lends itself readily to automation. In the process ultrasound is applied to two layers of plastic, heat is generated at the interface causing the material to soften, and flow, and the two layers are subsequently glued or joined together.

The effect of ultrasound on the encapsulation of titanium dioxide pigment, J.P.Lorimer, T.J.Mason and D. Kershaw, Colloid and Polymer Science, 269, 392•397, 1991.

The use of ultrasound for the controlled degradation of polymer solutions, Advances in Sonochemistry Vol 1 G. J.Price (pp231-287) ed. T.J.Mason, JAI Press 1990

Sonochemical initiation of polymerization, Advances in Sonochemistry Vol 2 P.Kruus (pp1-22) ed. T.J.Mason, JAI Press 1991

Examples of Projects

"The effect of ultrasound on the polymerisation of N-vinylcarbazole"
"The effect of ultrasound on the emulsion polymerisation of styrene"
"Ultrasonic degradation of dextran in aqueous solutions"

MATERIAL SCIENCE: THE PREPARATION OF NANOMATERIALS

There are close to 20 different methods for the fabrication of nanomaterials, these are regarded as the chemical and engineering materials of the future. What makes the use of power ultrasound effective and different from the other methods of synthesis are properties such as:

- The ability to produce nanomaterials in the amorphous state. This is of particular importance in catalysis, magnetism, coatings etc.
- The shorter reaction times involved e.g. mesoporous materials (MSPM) can be prepared in hours (it normally takes days by the sol–gel method).
- The insertion of nanoparticles into the pores of MSPM without blockage of the pores.
- The syntheses of inorganic fullerenes at room temperature. Other methods normally require high temperatures.

Power ultrasound provides one of the most exciting ways to synthesize pure and supported nanomaterials for research and industry. This is due to the high temperatures and pressures created during the collapse of an acoustic cavitation bubble is on a microsecond time scale and is associated with a rapid cooling rate (> 10^9 K/s) which is much greater than that obtained by conventional rapid cooling techniques (10^5-10^6 K/s). This means that sonochemistry can be used to prepare amorphous nanosized metallic particles. Also, since the thermal conductivities of metal oxides are generally much lower than those of the metals, these faster cooling rates are necessary to prepare amorphous metal oxides.

The Sonochemistry Centre is involved in the preparation of nanoparticles through an EU STREP programme entitled "Development of multifunctional nanometallic particles by Sonoelectrochemistry" (SELECTNANO). SELECTNANO aims to manufacture new metal and transition metal nanoparticles for dedicated new applications, using the novel process of sonoelectrochemistry..
This technique is a specialism of the Centre and combines electrolysis with sonolysis. The sonication horn serves as a cathode for the electrolysis process and as a transducer releasing ultrasonic waves.
A short electric pulse serve to reduce ionic species and deposit seed nanoparticulate metal crystals on the cathode. This is followed by a short ultrasonic pulse causing these nanoparticles to fbe released into the electrolysis mixture. Repeated sequential pulse then provide a semi-continuous method of generating the metallic powders.

This technique will be applied to fabricate nano Mg, Al, Fe, Co, Cr as well as nano alloys such as Fe-Cr, Fe-Mn, Fe-Co, and Cu-Sn which are foreseen to have a wide range of applications. Once formed, these nanoparticles can also be adsorbed onto stabilizing matrices such as colloidal dispersions using surfactants and polymers.

Applications for such materials include:

- Multifunctional printing; (conductive labels and information coding based on a printed pattern for security purposes

- nanostructured metallic coating
- sized shell structures for controlled release of encapsulated active materials
- molecular diagnostics and bio separations
- high intensity color pigments; novel cosmetic ingredients
- Nanoscale conductive structuring materials
- Novel coating additives

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information

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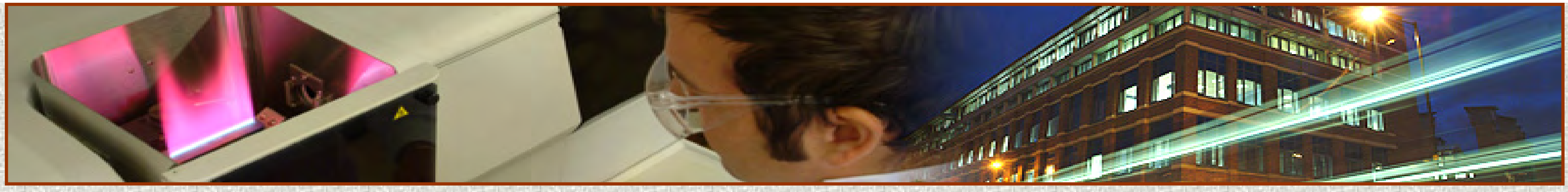

Group

Home
Introduction
Research
Electro
Therapy
Food
Synthesis
Green
Materials
Group

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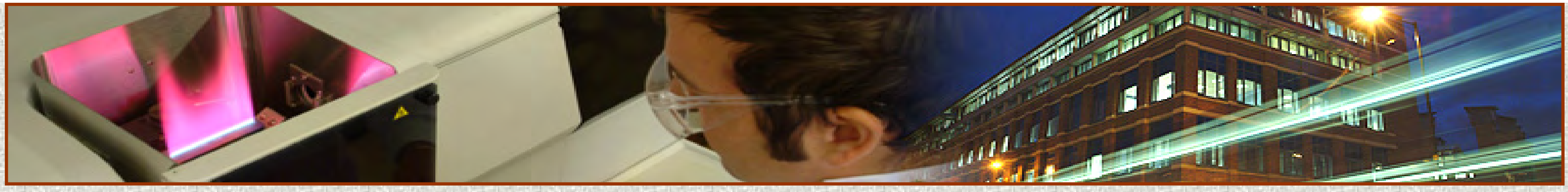
Academic Career

1964 - 1970	B.Sc. in Chemistry then Ph.D. in Physical Organic Chemistry, Southampton University U.K.
1970 - 1972	NATO Fellow at Amherst College, Massachusetts U.S.A.
1972 - 1973	Research Fellow, York University, U.K.
1973 - 1974	Temporary Lecturer in Organic Chemistry, Bradford University, U.K.
1975 - present	Coventry Polytechnic (now University) U.K., Lecturer in Organic Chemistry,
	Senior Lecturer (1985)
	Principal Lecturer (1990)
	Professor of Chemistry (1991)
1996	D.Sc., Southampton University, U.K.
1989 - 1990	Visiting Professor of Chemistry at University Paris Sud, France
1999 - present	Honorary Professor at Chongqing Medical University, P.R.China

Other Professional Activities

1981	Elected Fellow of Royal Society of Chemistry
1987	Co-founder and Secretary of Royal Society of Chemistry Sonochemistry Group
1990 - present	Chairman of RSC Sonochemistry Group
1991 - present	President of European Society of Sonochemistry
	European Editor of "Ultrasonics - Sonochemistry" (Elsevier)

Return to top



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Publications

Advances in Sono

Books

Chapters

Links

Contact information

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Sonovoltammetric studies on copper in buffered alkaline solution, JP Lorimer, T J Mason, Mario Plattes, S S Phull, J Iniesta and D J Walton, Ultrasonics Sonochemistry 11, 223-226 (2004).

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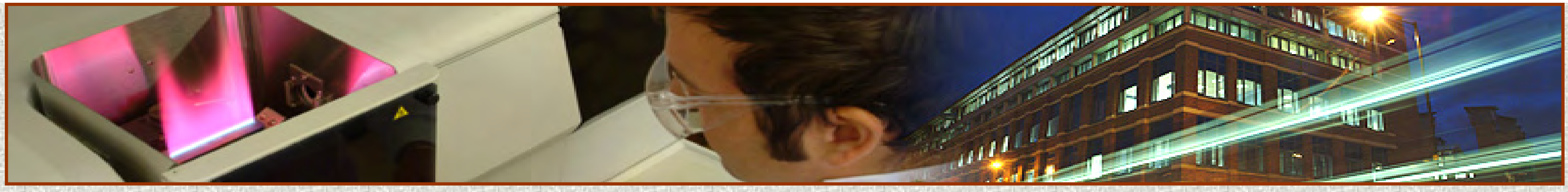
Return to top



Advances in Sono

Home
Introduction
Research
Electro
Therapy
Food
Synthesis
Green
Materials
Group

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Publications
Advances in Sono
Books
Chapters
Links
Contact information

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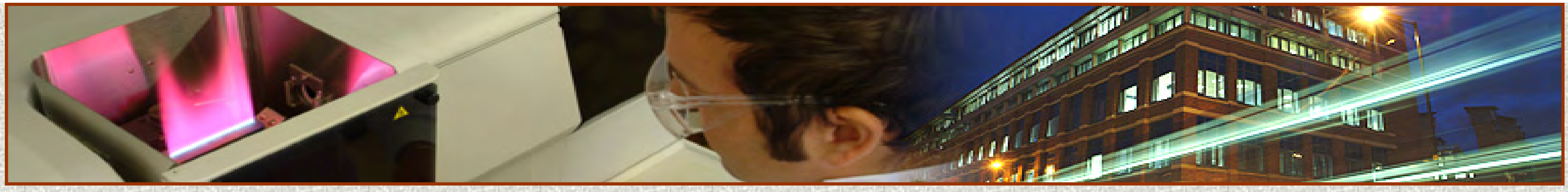
Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications
| Advances in Sono | Books | Chapters | Links | Contact information



Books

Home
Introduction
Research
Electro
Therapy
Food
Synthesis
Green
Materials
Group

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Publications

Advances in Sono

Books

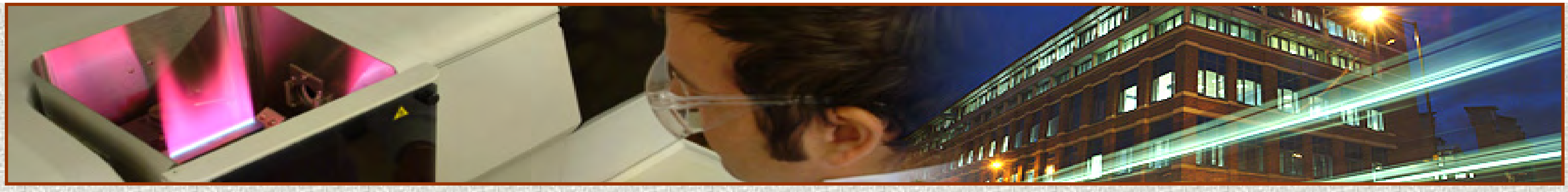
Chapters

Links

Contact information

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Chapters

Home
Introduction
Research
Electro
Therapy
Food
Synthesis
Green
Materials
Group

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Advances in Sono

Books

Chapters

Links

Contact information

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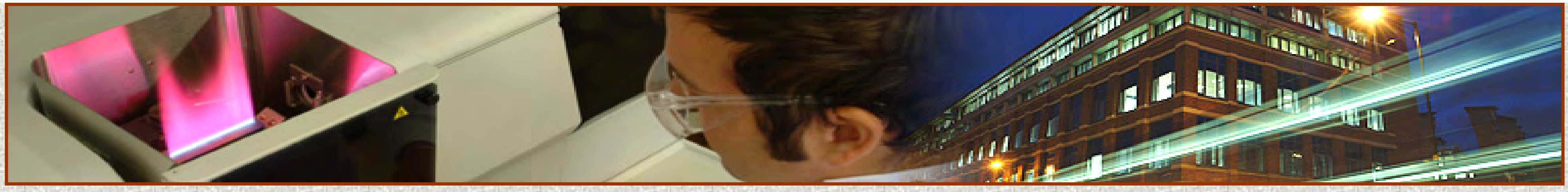
Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



Links

Home
Introduction
Research
Electro
Therapy
Food
Synthesis
Green
Materials
Group

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- Diagnostic Ultrasound
- High Intensity Focussed Ultrasound (HIFU)

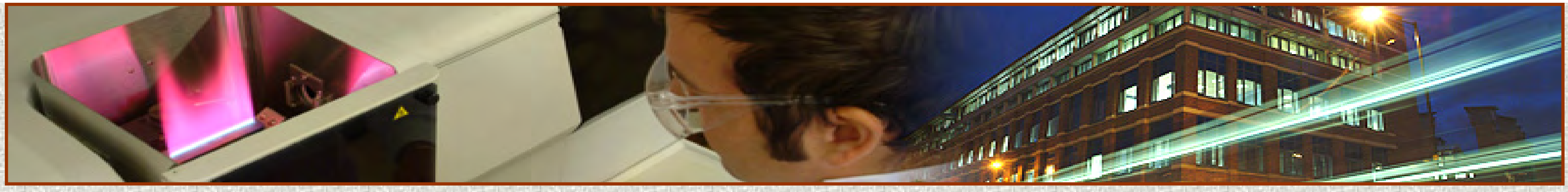
Research Opportunities

Publications
Advances in Sono
Books
Chapters
Links
Contact information

- MSc by Research in Sonochemistry

Return to top

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information



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Publications

Advances in Sono

Books

Chapters

Links

Contact information

Home | Introduction | Research | Electro | Therapy | Food | Synthesis | Green | Materials | Group | Publications | Advances in Sono | Books | Chapters | Links | Contact information

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