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Sonochemistry and its Industrial Applications

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Abstract: One of the main scientific and technological concerns at present for industrial processing is the development of green technologies. Sonochemistry or High-power ultrasonics is an emerging green technology that offers a great potential for a wide range of processes. The fundamentals of Sonochemistry rely on the formation of the cavity vacuole in the liquid by the ultrasonic frequency. As a consequence, a series of mechanisms can be activated by the ultrasonic energy such as localised heating, agitation, chemical effects, diffusion, interface instabilities, friction reduction, mechanical rupture etc. Such mechanisms can be utilized to produce or to enhance industrial processes. As the process industries are in need for greater efficiency, many companies can make use of ultrasonic energy in reaction chemistry and the physico-chemical separation of products. The effects of high power ultrasound in liquids can be utilized for greater chemical benefit.

Key words: Ultrasonics · Acoustic cavitation · Homogeneous sonochemistry · Heterogeneous sonochemistry · Sonocatalysis

INTRODUCTION

Sonochemistry is the application of ultrasound energy to chemical reactions. Ultrasound is transmitted through a medium using pressure waves by inducing vibrational motion of the molecules which alternately compress and stretch the molecular structure of the medium due to a time-varying pressure. The distances among the molecules vary as the molecules oscillate around their mean position. If the intensity of ultrasound is increased in a liquid, a point is reached at which the intramolecular forces are not able to hold the molecular structure intact. Consequently, it breaks down and cavitation bubbles are created. This process is called cavitation and the point at which it starts is known as the cavitation threshold [1].

Chemical transformation by ultrasound does not come from direct coupling of a sound field with chemical species but from the phenomena of cavitation leading to the production of oxidants. When an ultrasound is applied to a liquid, the formation of cavitation bubbles is initiated with sufficient amplitude of ultrasound during the rarefaction cycle. By the violent collapse of bubbles, the hot spots are caused by semi-adiabatic compression with temperatures of several thousands Kelvin, pressures of about one thousand atm and heating and cooling rates above 10^{10} K/s. In such extreme condition, water dissociation takes place easily to generate some oxidants of hydroxyl radical, hydrogen peroxide, ozone etc. responsible for various chemical reactions referred to as sonochemical reactions. Also, at the violent collapse, cavitation bubble emits light called sonoluminescence from the inside of the bubble or light called sonochemiluminescence when cavitation-induced oxidants react with some matter such as luminol in bulk liquid or bubble liquid interface [2,3,4].

Sonochemical Reactions: The chemical effects of ultrasound are diverse and include dramatic improvements in both stoichiometric and catalytic reactions [5]. In, some cases, ultrasonic irradiation can increase reactivities by million folds.

Investigations into the chemical effects of ultrasound during the past few years can be delineated in three areas: homogeneous sonochemistry, heterogeneous sonochemistry and sonocatalysis.

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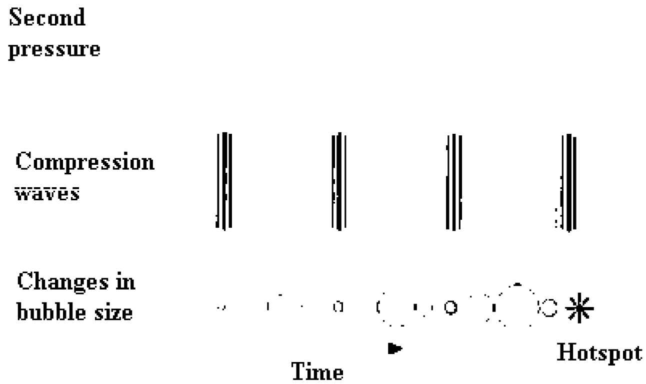

Fig. 1: Representation of bubble growth and collapse in a liquid irradiated by ultrasound and the resulting hot-spot due to adiabatic compression [6].

- The homogeneous sonochemical reactions can be further investigated in aqueous media, organic solvents and in liquid-solid systems.

The sonolysis of water produces both strong reductants and oxidants which further causes secondary oxidation and reduction reactions. Fig. 1.

In organic solvent a variety of unusual activity patterns have been observed during ultrasonic irradiation, including multiple ligand dissociation, novel metal-cluster formation and initiation of homogeneous catalysis at low ambient temperatures with rate enhancements greater than 100,000 fold [7-9].

In Liquid-Solid System cavitation near extended liquid-solid interfaces is very different from cavitation in pure liquids. In these cases the shockwaves created by homogeneous cavitation can create high-velocity interparticle collisions. The turbulent flow and shockwaves produced by intense ultrasound can drive metal particles together at sufficiently high speeds to induce effective melting at the point of collision [10,11].

- The use of high intensity ultrasound to enhance the reactivity of metals as stoichiometric reagents has become a routine synthetic technique for many heterogeneous organic and organometallic reactions [2-5,12-17]. The rate enhancements of more than ten folds are common, yields are often substantially improved and by-products are avoided. Infact, ultrasound is used at room temperature and pressure to promote heterogeneous reactions e.g. synthetic organometallic chemistry and heterogeneous catalysis which normally occur only under extreme conditions of hundreds of atmospheres and degrees [18].

- The sonocatalysis effects can occur during the formation of supported catalysts, in the activation of preformed catalysts, or by the enhancement of catalytic behavior during the catalytic reaction. Dramatic changes in surface morphology and composition lead to depassivation of the catalyst surface causing large increase in activity. This is especially important in the case of metal powders [19-21].

As a consequence of high power ultrasound a series of mechanisms can be activated such as agitation, diffusion, interface instabilities, friction reduction, localised heating, mechanical rupture, chemical effects etc. Such mechanisms can be exploited to produce or to enhance industrial processes. A new family of high power ultrasound transducers with extensive radiators has been recently introduced. It comprises a variety of transducer types designed with the radiators adapted to different specific uses in fluids and multi-phase media. Such transducers implement high power capacity, high efficiency and radiation pattern control. The introduction of such new transducers has significantly contributed to the development at semi-industrial and industrial level of a number of processes in the food and beverage industry, in environment and in manufacturing [22].

Current Industrial Applications

Cleaning and Decontamination: Ultrasonic cleaning technology is generally used in laboratories as ultrasonic cleaning baths. Ultrasonic cleaning can be either applied for the cleaning of micro components under clean-room conditions or used for very large items such as engine blocks in factories. It is particularly effective in the removal of biological contamination because it can reach

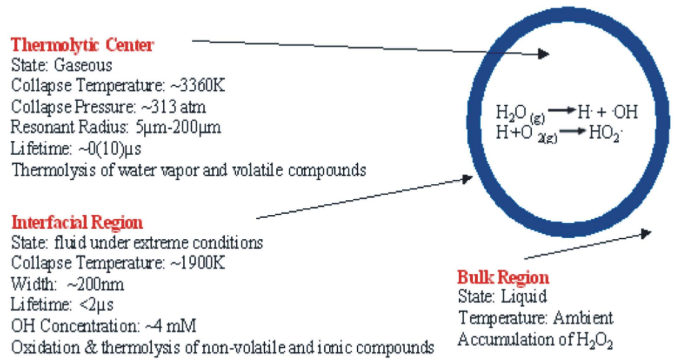

Fig. 2: Shows Cavitation bubble and the chemical reactions taking place at different regions (www.ceegs.ohio-state.edu/~lweavers/images/cav).

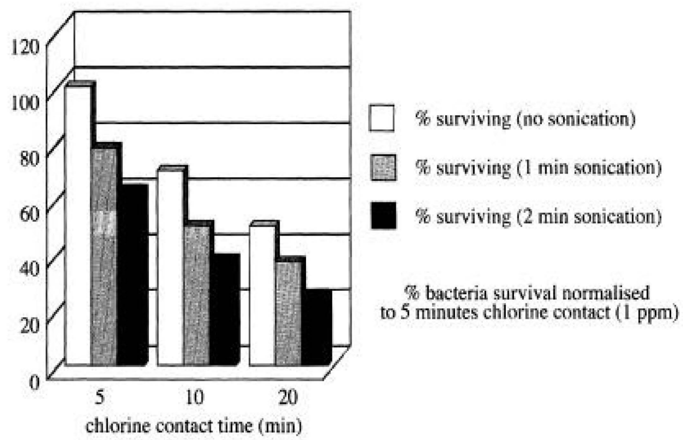

Fig. 3: The effect of short bursts of ultrasound (20 kHz) and chlorine on the dechlorination of river water [25].

crevices that are not easily reached by conventional cleaning methods. For this reason, such cleaning is used for a range of items from large crates used for food packaging and transportation, to delicate surgical implements such as endoscopes [21].

Power ultrasound is currently under investigation for use in the microbial inactivation [23]. The results obtained by flow cytometry indicated that the amounts of DNA or RNA formed in yeast cells decreased with an increase in ultrasonic irradiation time without any threshold [24]. Increasing the ultrasonic power in the range of 1.7-12.4 W enhances the inactivation of Gram-negative bacterium and Gram-positive bacterium. The examinations of bacterium showed lethal damages due to the interaction of bacterial cells with the cavitational bubbles [25]. Current trends are towards the reduction in quantity of the biocide used in sterilization. Power ultrasound affords the opportunity of increasing the efficiency of a biocide such as chlorine Fig. 3 [26].

Microstructure changes and micro-damage behavior of some probiotic bacteria (LAB) under the impact of ultrasound shock was studied by F. Tabatabaie and A. Mortazavi. Three kinds of micro damages are usually produced by ultrasound, micro-cracks, micro-voids and ruptures. Studies by Transmission Electron Microscope (TEM) showed that ultrasound could increase the cell wall permeability of the cells, which is important in the release of enzymes such as ß-galactosidas reduction of the coagulation time. The survival of LAB was very low in very long exposures of ultrasound [27].

One further application of sonochemistry for environmental use is in the treatment of sewage sludge. Ultrasonic treatment at a frequency of 31 kHz and high acoustic intensities at half technical scale shortens the residence time of sludge to eight days with a biogas production 2.2 times that of a control fermenter. Once optimized, it is likely that this type of process will also be adopted on an industrial scale. Anaerobic fermentation is the frequently applied process for stabilization of sewage sludge and provides mass reduction, methane production and improved dewatering properties. Ultrasonic irradiation has improved the process of anaerobic digestion, solubilization, cell disruption of microorganisms, disintegration of bio-solids, the reduction of flock size and reduction in weight and volume of sludge. Thus ultrasound treatment has the potential to improve the anaerobic digestion process and enhance the recovery of valuables [28].

The positive chemical and mechanical effects generated by irradiating a liquid medium with power ultrasound is largely exploited in lab-scale for extraction, synthetic application and persistent organic pollutants (POPs) degradation. Among the common chemical reactions of industrial interest the transesterification of vegetal oils for biodiesel production is probably the main priority, given the demand for a rational use of biomass under environmentally friendly conditions. Compared to typical batch ultrasound reactors, flow reactors stand out for their greater efficiency and flexibility as well as lower energy consumption. With developed ultrasonic flow reactor biodiesel can be produced by transesterification of soybean oil. The results show considerable reduction in energy consumption, by using two-step procedure: first a conventional heating under mechanical stirring followed by ultrasound irradiation at the same temperature [29].

Ultrasonic irradiation has also been used for chemical remediation of water. The mode of sonochemical degradation of organic compounds in aqueous solution depends upon their physical and chemical properties [30-32]. Various aromatic compounds, i.e. nitrobenzene, aniline, phenol, benzoic acid, salicylic acid, 2-chlorophenol, 4-chlorophenol, styrene, chlorobenzene, toluene, ethylbenzene and *n*-propylbenzene were decomposed under same conditions of ultrasonic irradiation. The compounds showing hydrophobicity had significant accumulation at the gas-liquid interface of the bubbles which is the main factor for the sonochemical degradation of aromatic compounds [33]. The sonolytic degradation of the textile dye *martius yellow* in water revealed that pyrolysis does not play a significant role in its degradation. An OH° radical induced reaction is the main degradation pathway of the dye-taking place at the bubble/solution interface [34]. Similarly in another experiment with dilute perfluorochemicals (PFCs) concentrations, adsorption to the bubble-water interface is ultrasonically enhanced due to high-velocity radial bubble oscillations [35]. The cavitation bubble can function in two ways. In the case of volatile chemicals that enter the bubble and destruction occurs through the extreme conditions generated on collapse. In the case of chemicals remaining in the aqueous phase the bubble acts as a source of radicals (H°, HO° and HOO°) which enter the bulk solution and react with pollutants. This is neatly illustrated in a comparative study of the decomposition of phenol to carboxylic acids and carbon tetrachloride to CO_2 and $C1^-$ in water saturated with oxygen at different frequencies Table 1 [36].

Table 1: Comparison between H_20_2 production and the rates of phenol and carbon tetrachloride disappearance at different frequencies (pM min-l) [33]

Frequency (kHz)	20	200	500	800
H_2O_2 formation	0.7	5.0	2.1	1.4
Phenol degradation	0.5	4.9	1.9	1.0
CCl_4 degradation	19	33	37	50

An important process in tanning of pelt/hide prior to tanning is degreasing. Presence of natural fat at the interior of skin/hide makes degreasing process quite difficult. Usually, organic solvent or detergent based degreasing processes are used which lead to environmental problems. The use of power ultrasound in aqueous degreasing process has been investigated with different degreasing systems. Glutaraldehyde pre-tanning has also been employed for carrying out the degreasing process at ultrasonic bath temperature. The results show that there is a significant increase in the degreasing efficiency due to the application of ultrasound. About twofold increase in fat removal has been observed. Comparing the degreasing efficiencies of the solvent with aqueous based ultrasonic processes, about 80% of the solvent degreasing efficiency could be obtained for aqueous degreasing process [37].

Industrial Processing

Mixing and Emulsification: One of the earliest devices that were developed for this purpose was the so-called liquid whistle and this continues to be used widely. Typical examples of the use of such whistles include the preparation of emulsion bases for soups, sauces or gravies that consist of a premix of water, milk powder, edible oil and fat together with flour or starch as thickening agent. After passing through the homogenizer, a fine particle-size emulsion is generated with a smooth texture. Another example is the production of ketchup as a smooth product with increased thickness and improved taste compared with conventional mixers as a result of the complete dispersion of any clumps of tomato pulp. In the textile industry, poor quality dying of fabrics usually can be attributed to the mechanical mixing used. Agglomerates of non-dispersed dye can produce a speckled appearance and uneven mixing may change the shade. By passing the mixture through an ultrasonic homogenizer before use can solve these problems [38].

Extraction and Impregnation: Solvent extraction of organic compounds contained within the body of plants arid seeds is significantly improved by the use of power ultrasound. The benefits of sonochemically forced impregnation of porous materials can be found in a wide variety of technologies.One of these is in the preparation of catalysts of the type often termed 'egg shell' where a catalytically active material is supported on the outside of an inert support material [39]. To enhance the dying of leather the same process of sonochemically forced impregnation can be used. In the presence of 20 kHz ultrasound (approximately 5 W cm-2) the dye impregnation is substantially increased [40].

The benefit of using ultrasound in plant extraction has already been demonstrated for a number of compounds of interest in pharmacology and food industries. Example of the benefits include the extraction of tea solids from dried leaves with water using ultrasound giving an improvement of almost 20% in yield at 60°C, approaching the efficiency of thermal extraction at 100°C. The culinary herb rosemary *Rosmarinus Officinalis* is known to contain materials with antioxidant properties. The most active antioxidants Carnosic acid and rosmarinic acid are reported to be isolated from the herb. By using butanone, ethyl acetate and ethanol as solvents, sonication improved the yields of carnosic acid for all three solvents and reduced the extraction times. Sonication also shortened the solvent effect of ethanol which being a poor solvent under conventional conditions, gave same level of extraction efficiency to the other two when sonicated. The extraction of dried herb with ethanol proved to be more efficient than that of fresh material [41].

It is important to identify organic compounds from plant extracts because they can be used as an excellent source of phytotherapics. The typical extraction procedures for the separation of organic compounds of medicinal plants are maceration, percolation and Soxhlet extraction. These techniques usually involve long extraction times, thereby decreasing efficiencies. Moreover, many natural products are thermally unstable and may degrade during conventional processes where the matrix is kept in a boiling solvent, such as Soxhlet extraction [42,43]. Many reports on the beneficial effects of medicinal plants ultrasound extraction have been published [44-46] and the main reported improvements have been found to be enhanced efficiency and shortening of extraction time [41,42,46]. A most likely mechanism for ultrasonic extraction is an enhanced mass transfer, the improved penetration of the solvent into the vegetal due to cell disruption and capillary effects [47,48]. The influences of several experimental parameters on the ultrasonic extraction of *Hibiscus tiliaceus L.* flowers were investigated like, extraction time, solvent polarity, sample amount, solvent volume and sample particle size. It was concluded that the most significant variables were extraction time and solvent polarity. The optimized process employed 5g of ground flowers, 150ml of methanol and 140min of extraction. The extracts were separated using preparative silica columns and the resulting fractions were analyzed by GC/MS. Some saturated hydrocarbons, fatty acids, fatty acid methyl esters, phytosterols and vitamin E were identified in the plant extracts [49]. Another study has been carried out for the effect of high-power ultrasound on olive paste, on laboratory thermo-mixing operations for virgin olive oil extraction. Virgin olive oil is obtained from the olive fruits through the use of physical procedures [50]. Among the process steps, olive paste malaxation is most significant. In order to obtain high oil quality and optimal process yields, kneading time and paste temperature are regulated. High-power ultrasound positively effects the malaxation step by providing a quick heating of olive paste, improving extractability and modulation of olive oil composition without alteration. The application of this technology in malaxation step of olive oil process requires the conversion to industrial plant [51].

Leaching and Bioleaching: The presence of iron compounds in silica sand is unaffordable in making of optical fibers, glass, ceramics and refractory materials. Iron can best be removed from the silica sand in aqueous oxalic acid under ultrasound irradiation. The method induces a remarkable acceleration for the iron leaching process, the leach acid concentration reduces dramatically and the removal efficiency increases when compared with conventional stirring method. Sonobleaching of silica sand may therefore be considered as a competing technology for industry [52].

The difficulties associated with the recovery of metals from ores, such as shales and schists, using conventional techniques are expensive with high-energy consumption and environmental pollution from these technologies. Bioleaching is a new and more economical technique for mining industries. Although the bioleaching process is very slow but with the application of ultrasonics it is proving to be of considerable interest for rapidly intensifying the performance of live bio-organisms and catalysts. Controlled sonication can have beneficial effects on leachability of metals without damaging cell walls. Ultrasonic treatment on the bioleaching of metals from black shale using an indigenous strain of *Aspergillus niger* enhances the production of organic acids which increases the rate of metal leaching and contributes to the efficient recovery of metals [53].

Filtration: The application of ultrasound enables the filtration system to operate more efficiently and for much longer periods without maintenance, through two specific effects. Sonication will cause an agglomeration of the fine particles and will supply sufficient vibrational energy to the system to keep the particles partly suspended and therefore leave more free 'channels' for solvent elution. Studies of acoustic filtration and separation processes continue to be an important area of applied acoustics research [54]. By applying ultrasound, permeation in microfiltration hollow fiber membrane process can be studied by means of reflection technique. Violent collapse of cavity bubbles increases the membrane permeability module in the ultrasonic bath [55]. Ultrasonic field has also been applied in the treatment of oil emulsification wastewater by ZrO_2 ceramic membrane. The permeate flux and rejection ratio for membrane process improves by applying ultrasonic field. Different methods with and without sonication have been applied for membrane flux recovery. It is found that the ultrasonic power increases the flux recovery ratio. In addition, by using chemical agents along with ultrasonic irradiation results in the highest cleaning efficiency and the shorter cleaning time [56].

Food Preservation: Ultrasound has successfully been used by the food industry for the measurement of thickness of pipes, chocolate layers, fat, lean tissues in meat, canned liquids and shell eggs, detection of contaminants such as pieces of metal, glass or wood in foods, measurement of flow rates through pipes, determination of food composition and measurement of particle size distribution in dispersed systems. However, further research is required before ultrasound becomes an alternative method of food preservation. The future of ultrasound in the food industry for bactericidal purposes lie in thermosonication, manosonication and manothermosonication, as they are more energy-efficient and result in the reduction of microbial and enzyme activity when compared to conventional heat treatment. Ultrasound needs to be assessed and food manufacturers must decide whether the ultimate benefits outweigh the cost of converting and maintaining the processing equipment [57].

Therapeutic Ultrasound: One medical application is for the destruction of blood clots, where a miniaturized ultrasonic device (1MHz) is attached to the end of a catheter so that it can be inserted into the blood vessel. The device is brought close to the clot, at which point a fibrolytic enzyme is released. When the transducer is activated, the acoustic energy accelerates the enzymatic dissolution of the clot [38]. Ultrasound could increase cytotoxicity of Adriamycin on cancer cell line as a result of increased intracellular accumulation ascribed to cavitation. The results suggested that hydroxyl radical play the leading role in synergism between ultrasound and Adriamycin [58].

A system has been developed (ExAblate 2000) by combining focused ultrasound technology with the properties of magnetic resonance imaging which enables precise targeting within tissues. In addition, temperature sensitive (magnetic resonance) MR sequences provide real time feedback of focal rises in temperature to ensure safe delivery of an effective thermal dose. Although studies have been carried out in many different areas including breast, brain and liver tumors and uterine fibroids [59]. G.A. Husseini, *et al.* present an artificial neural network (ANN) model that attempts to predict the dynamic release of doxorubicin (Dox) drug from P105 micelles (drug carrier) under different ultrasonic power densities at 20 kHz. As artificial neural networks ANN are applied as controllers in many industrial applications, the goal is to optimize the ultrasound application to achieve a target drug release at the tumor site. This has been done by controlling power density and ultrasound duration via an ANN-based model predictive control. The parameters of the controller are then tuned to achieve satisfactory reference signal tracking in terms of smoothness and response speed [60].

CONCLUSION

Ultrasound technology is applied as a recent and very environment-friendly process in an increasing number of applications and processes of the chemical industry. Its remarkable application options are in pharmacy, chemistry, biotechnology and environmental engineering. The applications make use of the different effects of ultrasound for the processing of gaseous, liquid and solid media. Moreover, ultrasound has a generally accelerating and favorable impact on heterogeneous reactions. Cavitation, which is the main sono-physical effect, enhances mass transfer and mechanical damage at solid-liquid interfaces. It brings about localised extremes of temperature and pressure and transient electrical effects. Under these conditions the chemical processes manifest themselves in increased reaction rates, easier initiation, less extreme process conditions and changes in reaction selectivity.

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