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Sonochemistry: Science and Engineering

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View Author Information ▾✓ **Cite this:** *Ind. Eng. Chem. Res.* 1999, 38, 4, 1215–1249

Publication Date: March 9, 1999 ▾

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in the true sense of the term occurs when ultrasound induces “true” chemical effects on the reaction system, such as forming free radicals which accelerate the reaction. However, ultrasound may have other mechanical effects on the reaction, such as increasing the surface area between the reactants, accelerating dissolution, and/or renewing the surface of a solid reactant or catalyst. This comprehensive review summarizes several topics of study in the sonochemical literature, including bubble dynamics, factors affecting cavitation, the effects of ultrasound on a variety of chemical systems, modeling of kinetic and mass-transfer effects, the methods used to produce ultrasound, proposed cavitation reactors, and the problems of scaleup. The objective of this paper is to present a critical review of information available in the literature so as to facilitate and inspire future research in the field of sonochemistry.

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1. Introduction

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Ultrasound has proven to be a very useful tool in enhancing the reaction rates in a variety of reacting systems. It has successfully increased the conversion, improved the yield, changed the reaction pathway, and/or initiated the reaction in biological, chemical, and electrochemical systems. This nonclassical method of rate enhancement, a field termed *sonochemistry*, is becoming a widely used laboratory technique. However, its use in industry is limited because the process of producing ultrasound is very inefficient and burdened with high operating costs. It is starting to attract attention because the operating costs may be off-set by reducing or eliminating other process costs. The use of ultrasound may enable operation at milder operating conditions (e.g., lower temperatures and pressures) eliminate the need for extra costly solvents

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Clausthal Technical University (Clausthal-Zellerfeld) which uses a modular sonochemical reactor to produce up to 4 metric tons of Grignard reagent/year. They found ultrasound to increase the conversion by a factor of 5 and reduce the induction period from 24 h to 50 min (Ondrey et al., 1996).

With such high enhancements in rates, it is no wonder that the number of publications in the field of sonochemistry has increased significantly in the last 20 years. Much of the pioneering work in the field has been done by chemists and physicists who have found that the chemical, and some mechanical, effects of ultrasound are a result of the implosive collapse of cavitation bubbles. The interest of the chemical engineer in capturing and quantifying these beneficial effects is rising, but publications in such crucially applied areas as mass transfer, reaction kinetics, reaction modeling, and reactor design are sparse. The objective of this paper is to present a critical review of information available in the literature so as to facilitate and inspire future research in the field of sonochemistry.

2. History

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The interest in ultrasound and cavitation effects dates back over 100 years. The first report of cavitation was published in 1895 by Thornycroft and Barnaby when they noticed that the propeller of their submarine, the H.M.S. Daring, was pitted and eroded. Twenty-two years later, in 1917, Lord Rayleigh published the first mathematical model describing a cavitation event in an incompressible fluid. Ultrasound was not used to enhance reaction rates until 1927 when Loomis reported the first chemical (Richards and Loomis, 1927) and biological (Wood and Loomis, 1927) effects of ultrasound. Ten years later Brohult (1937) discovered that ultrasound led to the degradation of a biological polymer. Research in this field of ultrasonics was expanded to the degradation of synthetic polymers by Schmid and Rommel in 1939.

One of the most basic concepts of sonochemistry is that free radicals are formed as a result of the cavitation of microbubbles which are created during the rarefaction (or negative pressure) period of sound waves. In 1994, Harvey et al. introduced the concept of rectified diffusion (the growth of microbubbles due to unequal transfer of mass across the interface during bubble oscillation). The early 1950s brought about several new and exciting developments in the field of sonochemistry. Noltingk and Nenniras (1950) performed the first computer calculations

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of ultrasound in liquid systems was also increasing. Naude and Ellis (1961) hypothesized the existence of microjets formed during asymmetric cavitation. This concept is still alive today and is used to explain the pitting of solid surfaces and the overall particle size reduction in heterogeneous systems. In 1953 Weissler published the first observation of a decrease in the rate of a sonochemical reaction with an increase in the ambient reaction temperature, a phenomenon observed in several other systems to date. In 1964 Flynn coined the now commonly used terms “transient cavitation” and “stable cavitation”.

In the 1970s there was a lull in publications concerning sonochemistry; however, the field regained interest in the 1980s and the number of publications increased dramatically. Neppiras (1980) used the term “sonochemistry” for the first time in a review of acoustic cavitation. Makino et al. (1982) used spin trapping and ESR (electron spin resonance) measurements to verify the formation of $H^\bullet$ and $OH^\bullet$ during the sonolysis of water. The field of sonochemistry was becoming so popular that in 1986 the first international meeting devoted to it was held by the Royal Society of Chemistry Annual Congress at Warwick University in the U.K. Such meetings have since become an annual event. In 1987 the Royal Society of Chemistry founded a Sonochemistry Group based at the Harwell laboratory of U.K.'s Atomic Energy Authority. The objective of the Sonochemistry Group is to develop methods to facilitate the use of ultrasound in industry. In the late 1980s and early 1990s several manufacturers started designing and marketing equipment exclusively for sonochemical research. The first issue of “Ultrasonics Sonochemistry”, a new periodical dedicated to the applications of ultrasound in chemistry, was released in 1994.

3. General Introduction

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Ultrasound occurs at a frequency above 16 kHz, higher than the audible frequency of the human ear, and is typically associated with the frequency range of 20 kHz to 500 MHz. The frequency level is inversely proportional to the power output. Low-intensity, high-frequency ultrasound (in the megahertz range) does not alter the state of the medium through which it travels and is commonly used for nondestructive evaluation and medical diagnosis. However, high-intensity, low-frequency ultrasound does alter the state of the medium and is the type of ultrasound typically used for sonochemical applications. A current list of the general applications of

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atomization	cleaning
cell disruption	coal-oil mixtures
crystal growth	cutting
crystallization	degradation of powders
defoaming	dental descaling
degassing	drilling
depolymerization	echo-ranging
dispersion of solids	erosion
dissolution	fatigue testing
drying	flaw detection
emulsification	flow enhancement
extraction	imaging
filtration	medical inhalers
flotation	metal-grain refinement
homogenization	metal tube drawing
sonochemistry	nondestructive testing of metals
stimulus for chemical	physiotherapy

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There are several references available which provide a general overview of the field of sonochemistry and the types of chemical reactions which have been studied (see, e.g., Suslick, 1988, 1990b; Ley and Low, 1989; Mason, 1991; Mason and Lorimer, 1989). Some authors have compared the various effects of ultrasound to other types of chemistry, such as mechanochemistry (Boldyrev, 1995). However, as will be evident from this review, it will take the combined effort of scientists and engineers to thoroughly understand sonochemical reactions and develop rational design procedures for sonochemical reactors.

4. Theory

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The chemical and mechanical effects of ultrasound are caused by cavitation bubbles which are generated during the rarefaction, or negative pressure, period of sound waves. During the negative-pressure cycle, the liquid is pulled apart at sites containing some gaseous impurity (nucleation sites), forming a void. Nucleation sites are also known as “weak spots” in the fluid. Nucleation in the absence of ultrasound can be seen everyday when drinking a carbonated beverage. The bubbles of carbon dioxide form at scratches in the glass where gaseous impurities, such as air, are harbored and act as nucleation sites. When using ultrasound, the cavitation activity is directly proportional to the number density of particles present in the medium (Madanshetty and Apfel, 1991). Chemical effects due to ultrasound are not observed when there are no dissolved gases in the system, when the sound intensity is not greater than the cavitation threshold of the system (Fitzgerald et al., 1956), or when the reactant is not volatile enough to enter the cavitation bubble during its formation (Griffing, 1952).

The physical and chemical effects of ultrasound are a result of both stable and transient cavitation events, which are described in the following sections. Two competing theories exist to explain the chemical effects due to cavitation: the *hot-spot theory* and the *electrical theory*. The hot-spot theory postulates that when the bubbles cavitate, localized hot spots are formed

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4.1. Bubble Dynamics. The chemical effects of ultrasound have been attributed to the collapse of both stable and transient cavitation events. Stable cavities oscillate for several acoustic cycles before collapsing, or never collapse at all. Transient cavities, conversely, exist for only a few acoustic cycles. The following sections provide a brief explanation of bubble dynamics and its modeling as published in the literature. For a comprehensive review of acoustic cavitation and bubble dynamics, reference may be made to Neppiras (1980).

4.1.1. Stable Cavities. Stable cavities are bubbles which form and oscillate around a mean radius in a sound field and exist for many acoustic cycles. For this to occur their growth rate during the rarefaction must be equivalent to their rate of contraction during the compression phase. This specifies that rectified diffusion, or the unequal transfer of mass into the bubble during the acoustic wave cycle, is not occurring. The wall motion of a stable bubble in an acoustic field is described by the relation

$$R\ddot{R} + \frac{3}{2}\dot{R}^2 = \frac{1}{\rho}[p_L(R) - p_\infty(t)] \quad (1)$$

where p_∞ is the pressure in the liquid far from the bubble, ρ is the density of the fluid, $\dot{R}$ and $\ddot{R}$ represent respectively the first- and second-order time derivatives of the bubble radius, and $p_L(R)$ is the liquid pressure just outside the bubble wall, given by

$$p_L(R) = p_T(R) - \frac{4\mu\dot{R}}{R} - \frac{2\sigma}{R} \quad (2)$$

where both eqs 1 and 2 are based on, but not limited to, the assumptions that the liquid phase is incompressible ($\rho = \text{constant}$) and viscous forces are neglected. The pressure far from the

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where P_0 is the hydrostatic (ambient) pressure. The pressure of the total mass content in the bubble $p_T(R)$ at a given radius R is the sum of both the permanent-gas pressure, $p_g(R)$, and the vapor pressure, $p_v(R)$. When it is assumed that the bubble is filled with an ideal gas, contains no vapor, and behaves as an adiabatic system ($PV^\gamma = \text{constant}$), the following expression, known as the Rayleigh–Plesset equation, is obtained:

$$R\ddot{R} + \frac{3}{2}\dot{R}^2 = \frac{1}{\rho} \left[\left(P_0 + \frac{2\sigma}{R_0} \right) \left(\frac{R_0}{R} \right)^{3\gamma} - \frac{2\sigma}{R} - \frac{4\eta\dot{R}}{R} - P_\infty \right] \quad (4)$$

where R_0 is the bubble radius at equilibrium, γ is the specific heat ratio of the gas within the bubble, and ρ , σ , and η are the density, surface tension, and viscosity of the bulk fluid, respectively. Equation 4 can be used for the isothermal case by setting γ equal to 1.

Bubbles with an equilibrium radius R_0 in a liquid system with a fixed temperature T and negligible viscous forces will pulsate with a resonance frequency ω_r , as defined by

$$\omega_r^2 = \frac{1}{\rho R_0^2} \left[3\gamma \left(P_0 + \frac{2\sigma}{R_0} \right) - \frac{2\sigma}{R_0} \right] \quad (5)$$

The eigenfrequency ω_r' takes into account damping of the linear oscillations due to viscous forces and is defined by

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presence of KMnO_4 (Cum et al., 1988, 1990, 1992). Because many ultrasonic transducers are designed with a set frequency, operating under resonance conditions is achieved by changing the system parameters in order to alter the bubble resonant frequency to match that of the transducer. This can be done by varying the hydrostatic pressure (Cum et al., 1988) and the system temperature (Cum et al., 1990). Other factors which affect the resonating frequency of the bubble include the characteristics of the liquid, such as its density and surface tension. Conversely, varying the ultrasonic frequency in order to drive the bubble dynamics toward transient cavitation was also investigated (Cum et al., 1992).

4.1.2. Rectified Diffusion. Rectified diffusion is the event where cavitation bubbles grow more during expansion than they shrink during contraction due to the unequal diffusion of gases and vapor from the bulk liquid phase into the bubble. A large volume of literature is available on rectified diffusion (see, e.g., Crum, 1980, 1984; Crum and Hansen, 1982) and will not be covered here.

4.1.3. Transient Cavitation. A transient cavity is one which exists for only a few acoustic cycles. During its existence it grows several times larger than its initial size and, upon implosion, creates extreme temperatures and pressures within its cavity. The wall motion of a transient, gas-filled cavity is described by (Neppiras, 1980)

$$R\ddot{R} + \frac{3}{2}\dot{R}^2 = \frac{1}{\rho} \left[P \left(\frac{R_{\max}}{R} \right)^{3\gamma} - P_m \right] \quad (7)$$

where $R_{\max}$ is the maximum radius of the bubble obtained just before collapse, P is the gas pressure in the bubble at its maximum size, and P_m is the liquid pressure at transient collapse. Because the collapse of the bubble always occurs when the acoustic pressure is near its peak, P_m is similar in value to the sum of the maximum pressure amplitude and the hydrostatic pressure ($P_A + P_0$) in an infinite fluid where the bubble density is very small and can be approximated as P_0 , the hydrostatic pressure, in an intense acoustic field where the bubble density is high. The ratio of the minimum bubble radius to the maximum radius is given by

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pressure of the gas within the bubble (neglecting the effects of vapor) is equal to the saturation vapor pressure of the liquid surrounding the bubble ($P_g = P_s$). When it is assumed that the bubble is filled with an ideal gas and when the surface tension and viscosity of the fluid are neglected, the maximum temperature ($T_{\max}$) and pressure ($P_{\max}$) obtained within the transient collapsing bubble are given by

$$T_{\max} = T_0 \left[\frac{P_m(\gamma - 1)}{P} \right] \quad (9)$$

$$P_{\max} = P \left[\frac{P_m(\gamma - 1)}{P} \right]^{\gamma/(\gamma-1)} \quad (10)$$

If the cavity contains vapor as well as gas, the collapse will be cushioned, and the maximum pressure and temperature will decrease because some of the energy generated during collapse will go toward condensation of the vapor. The time for complete collapse (τ_m), developed under the same assumptions by Khoroshev in 1963, is given by

$$\tau_m \approx 0.915 R_{\max} \left(\frac{\rho}{P_m} \right)^{1/2} \left(1 + \frac{P}{P_m} \right) \quad (11)$$

4.1.4. Modeling. Models have been developed to explain the bubble dynamics and sonoluminescence for a single, stable cavitation bubble (Gaitan et al., 1992). In addition, experimental results have been compared with various simulations, including the Keller–Miksis radial equation with a linear polytropic exponent approximation (Keller and Miksis, 1980), the Keller–Miksis radial equation using a more exact formulation for the internal pressure (Prosperetti et al., 1986), and Flynn's formulation which includes thermal effects inside the bubble (Flynn, 1975).

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bubble theory should be reinterpreted, at least in modeling the local surroundings of the single bubble, to account for the bubble population and the effects of the bubbles on one another. This contradicts “classical” approaches which take the position that each cavitation event should be solved separately because the dynamics of the bubbles in a sound field are nonlinear (Alippi, 1992).

4.2. Factors Affecting Cavitation. The ambient conditions of the reaction system can greatly influence the intensity of cavitation, which directly affects the reaction rate and/or yield. These conditions include the reaction temperature, hydrostatic pressure, irradiation frequency, acoustic power, and ultrasonic intensity. Other factors which significantly affect the cavitation intensity are the presence and nature of dissolved gases, choice of solvent, sample preparation, and choice of buffer. Each of these factors is described in detail below.

4.2.1. Presence and Nature of Dissolved Gases. Dissolved gases act as nucleation sites for cavitation. As gases are removed from the reaction mixture because of the implosion of the cavitation bubbles, initiation of new cavitation events becomes increasingly difficult. Bubbling gases through the mixture facilitates the production of cavitation bubbles, but the type of gas used is important. As a general rule, a gas with a high specific heat ratio gives a greater cavitation effect than one with a low specific heat ratio. Because the collapse of the bubble occurs in such a small amount of time ($\sim 3.5 \mu\text{s}$ as estimated by Prasad Naidu et al., 1994), it can be assumed to occur adiabatically. Monatomic gases, such as argon and helium, convert more energy upon cavitation than diatomic gases, such as nitrogen and oxygen, because of the larger ratio of specific heats. Gases which are extremely soluble in the reaction mixture may reduce the cavitation effect because the bubbles formed may redissolve before collapse occurs. The bubbles which do not dissolve often become so large (because of facile penetration of gas into the bubble) that they float to the surface and explode. The thermal conductivity of the gas is also important because, although the collapse is modeled as adiabatic, there is a small amount of heat which is transferred to the bulk liquid mixture during collapse. As the thermal conductivity of the gas increases, the amount of heat loss due to thermal dissipation also increases.

The dependence of cavitation intensity on the characteristics of the dissolved gases was studied by Entezari et al. (1997), who investigated the effect of ultrasound on the rate of carbon

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When the formation of hydroxyl radicals plays an important role in the mechanism of the reaction, a finite amount of oxygen in combination with a monatomic gas may accelerate the rate over the use of a pure monatomic gas alone. The liberation of iodine from aqueous potassium iodide was investigated under several different atmospheres consisting of various ratios of oxygen to argon (Hart and Henglein, 1985). The reaction was carried out in the presence of ammonium molybdate and hydrogen peroxide (H_2O_2), which catalyzes the oxidation of the iodide ion to iodine. The reaction had a maximum rate in the presence of a 30% oxygen–70% argon atmosphere. Similar results were found for the ultrasonic degradation of phenol in an aqueous solution (Berlan et al., 1994). Although the ratio of specific heats (γ) is higher for argon than for oxygen, it was determined that the concentration of oxygen facilitated the formation of hydroxyl radicals, which consequently accelerated the reactions.

Using the same system as Hart and Henglein (1985), Prasad Naidu et al. (1994) rationalized the experimental data by accounting for the dissolved gases within their model (refer to the paper for detailed information). The nature and presence of the dissolved gas were considered using bubble dynamics equations to predict the amount of free radicals (in moles) generated during collapse in order to estimate a concentration of free radicals to use in their kinetic model. The authors started with the Rayleigh–Plesset equation (as given by eq 4 but altered slightly to account for the pressure of the vapor within the cavitation bubble). They assumed that the entire growth phase and the initial part of the collapse phase of the bubble were isothermal ($\gamma = 1$). They also assumed that the vapor in the cavity was equal to the saturation vapor pressure of the liquid (P_s) and initialized the system assuming that the bubble radius was equal to the equilibrium radius R_0 with a wall velocity of zero ($\dot{R} = 0$, indicating steady state).

The collapse phase was assumed to become adiabatic once the pressure of the gas within the cavity became equal to the saturation vapor pressure of the liquid ($P_g = P_s$). The parameter R_2 was defined as the radius of the bubble at which the transition from isothermal to adiabatic collapse occurs. However, the predictive ability of the model varied depending upon the type of dissolved gas. The model adequately explained the experimental data obtained under an oxygen atmosphere but deviated greatly from experiments conducted under a nitrogen atmosphere. One of the main reasons for this deviation may be because the authors assumed a constant number of bubbles produced within the sonicated mixture, regardless of gas content and KI concentration. However, as discussed previously in this section, the number of bubbles

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4.2.2. Ambient Temperature. Contrary to chemical reactions in general, an increase in the ambient reaction temperature results in an overall decrease in the sonochemical effect. The decrease is a result of a sequence of events. First, as the reaction temperature is raised, the equilibrium vapor pressure of the system is also increased. This leads to easier bubble formation (due to the decrease of the cavitation threshold); however, the cavitation bubbles which are formed contain more vapor. As discussed in section 4.2.1, vapor reduces the ultrasonic energy produced upon cavitation because it cushions the implosion in addition to using enthalpy generated in the implosion for the purposes of condensation. In general, the largest sonochemical effects are observed at lower temperatures when a majority of the bubble contents is gas.

In certain reaction systems, an optimum reaction temperature may lead to more favorable results. In such systems, an increase in temperature will increase the kinetic reaction to a point at which the cushioning effect of the vapor in the bubble begins to dominate the system. When this occurs, the rate of the reaction decreases upon further increase in ambient reaction temperature (see, e.g., Ibsi and Brown, 1967; Ley and Low, 1989). The rate may even reach a plateau with temperature and then decrease as the temperature increases, as observed by Sehgal and Wang (1981) when investigating the degradation of thymine. The observed temperature effect was dominated by the reaction kinetics in and around the cavitating bubble. They argued that because thymine is relatively nonvolatile, the degradation reaction was occurring in the gas-liquid film between the cavitating bubble and the bulk liquid mixture (which will be discussed in greater detail in section 8.1). As the reaction temperature increased, the rate of diffusion of thymine from the bulk liquid phase to the reaction zone was accelerated. However, increasing the temperature was also simultaneously decreasing the intensity of cavitation, thus reducing the amount of free radicals produced within the bubble. It was speculated that these free radicals were required for the degradation reaction to occur and that they diffuse from the vapor cavity to the gas-liquid film where reaction ensues. As the rates of the counterdiffusing reactants became comparable, a further increase in temperature had little or no effect on the reaction (i.e., the percent change in thymine concentration reached a plateau as a function of temperature). However, as the temperature continued to be increased, the declining production of free radicals began to have a negative effect on the rate of degradation.

4.2.3. Ambient Pressure. An increase in the ambient reaction pressure generally results in an

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pressure was decreased to 115 psig, the effects of ultrasound were significantly increased. It appeared that operating at pressures of 200 psig and above increased the cavitation threshold in the system to a level at which the cavitation bubbles could no longer be produced or were produced in such small quantities that they did not significantly effect the overall reaction. For any given system an optimum operating pressure will most likely exist (see, e.g., Berlan et al., 1994).

As discussed in section 4.1.1, changing the hydrostatic pressure can alter the resonance frequency (eq 5) and equilibrium radius (eq 4) of the bubble and drive the system toward resonance conditions. This approach was taken by Cum et al. (1988), who found that operating the system under resonance conditions increased the rate and yield of the reaction.

4.2.4.Choice of Solvent. Cavities are more readily formed when using a solvent with a high vapor pressure, low viscosity, and low surface tension. However, the intensity of cavitation is benefited by using solvents with opposing characteristics (i.e., low vapor pressure, high viscosity, and high surface tension). Lorimer and Mason (1987) investigated the effects of the natural cohesive forces of the solvent on cavitation and found the most intensive cavitation occurring in solvents with a higher viscosity. Other researchers found that cavitation was inhibited when using the extremely volatile solvent diethyl ether, which has a vapor pressure of ~0.73 atm at 25 °C (see, e.g., Fitzgerald et al., 1956; Luche, 1987). When choosing a solvent for a particular reaction system, the appropriate “family” of solvents to use is frequently dictated by the type of chemistry involved (i.e., due to temperature, solubility, and/or other issues). Once a family of solvents is identified, then the effects of the various solvents within that family on cavitation can be investigated.

4.2.5. Ultrasonic Frequency. The frequency of the ultrasound has a significant effect on the cavitation process because it alters the critical size of the cavitation bubble. At very high frequencies, the cavitation effect is reduced because either (1) the rarefaction cycle of the sound wave produces a negative pressure which is insufficient in its duration and/or intensity to initiate cavitation or (2) the compression cycle occurs faster than the time required for the microbubble to collapse. In the past, most sonochemical reactions were carried out at frequencies between 20 and 50 kHz. This is because the alteration of frequency has no apparent effect in several reactions, such as in the dissociation of carbon disulfide (Entezari et

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of argon than in the presence of air. This was expected as discussed in section 4.2.1. However, when operating at 900 kHz, the rate was 3.13 times greater in air. It seems likely, therefore, that although the intensity of cavitation was lower in the presence of air because of its lower polytropic ratio, the formation of hydroxyl radicals from the air–water mixture was accelerated because of the larger number of useful cavitational events at 900 kHz, and thus the rate of oxidation of iodide was increased. These findings agree with those of Petrier et al. (1992b), Entezari and Kruus (1996), and Hua and Hoffmann (1997). Petrier et al. (1992b) reasoned that because bubble lifetimes were shorter at higher frequencies (3×10^{-7} s at 514 kHz as compared to 3×10^{-5} s at 20 kHz), the OH radicals have an opportunity to escape the cavitation bubble before undergoing any reaction or recombination. A similar hypothesis was proposed by Mason et al. (1994), who speculated that the escape of the OH radicals to the bulk solution increases their availability for reaction.

Francony and Petrier (1996) found that the rate of sonochemical degradation of carbon tetrachloride was also enhanced when using a higher frequency (500 kHz as compared to 20 kHz). Both frequency levels had a constant power dissipation of 30 W and produced Cl^- and CO_2 as the primary products of degradation. However, the products were produced at a faster rate when operating at 500 kHz. When investigating the degradation of trichloroethylene at 20 and 520 kHz, Drijvers et al. (1996) concluded that the reaction was “energetically more efficient” at 520 kHz. As a side note, the same authors also investigated the effect of pH on the degradation kinetics and found the rate constant to increase as the buffer solution became more basic.

In summary, lower frequency ultrasound produces more violent cavitation, leading to higher localized temperatures and pressures at the cavitation site. However, higher frequencies may actually increase the number of free radicals in the system because, although cavitation is less violent, there are more cavitational events and thus more opportunities for free radicals to be produced (Crum, 1995). In addition, the shortened bubble lifetime may increase the amount of free radicals which are able to escape from the cavitation site to the bulk mixture, where they facilitate the bulk reaction. It is contended that the optimum frequency is system specific and depends on whether intense temperatures and pressures are required (thus enhanced by lower frequencies) or if the rate of single electron transfer is more important (enhanced by higher frequencies).

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The optimum power level is also dependent on the operating frequency (Whitlock and Harvey, 1997b). When investigating the rate of corrosion of 304L stainless steel, the authors found that curve maxima were different (i.e., different power optima) for different ultrasonic frequencies. No maximum was observed when operating at 20 kHz, as was also found by Hagenson et al. (1994) when researching the synthesis of dibenzyl sulfide.

4.3. Sonoluminescence. Sonoluminescence is the emission of light associated with cavitation. It was discovered in 1934 by Frenzel and Schultes when they observed a faint luminescence over a water bath when it was exposed to intense ultrasound. No general consensus has been reached as to what causes this light emission, although several hypotheses have been put forward (see, e.g., Lepoint-Mullie et al., 1996). A review of sonoluminescence was published in 1984 by Walton and Reynolds which includes several of the theories in the literature attempting to explain the origin of sonoluminescence. They believe that it is due primarily to the recombination of free radicals generated within cavitation bubbles during collapse. However, Suslick et al. (1990) favor an alternative theory that the light emission is caused by thermally created chemiluminescence. More work is needed before a general consensus can be reached.

The experimental work performed in the area of sonoluminescence has resulted in several interesting observations. For instance, in an air–water system the sonoluminescence intensity was the highest at lower temperatures and decreased exponentially with increased system temperature, up to a temperature of 90 °C (363 K), where it became undetectable (Chendke and Fogler, 1985). The sonoluminescence intensity is dependent on the solvent and was found to increase in the order $\text{MeOH} \sim \text{EtOH} < n\text{-PrOH} \sim t\text{-BuOH} < \text{C}_6\text{H}_6$ (Sehgal et al., 1977). It is also dependent on the dissolved gas in solution, as it decreases with increasing thermal conductivity of dissolved rare gas in the order $\text{Xe} > \text{Kr} > \text{Ar} > \text{Ne} > \text{He}$ (Hickling, 1963). A theoretical study of sonoluminescence was published in 1993 by Kamath et al.

4.4. Estimation of Ultrasonic Parameters. The following sections and Table 2 provide a summary of pertinent ultrasonic parameters which have been estimated by different authors through either experimental research or model simulations. While the actual values of the parameters are system dependent, the values in the table are provided as estimates of orders of magnitude for modeling studies.

Table 2. Estimates of Parameters Necessary for Modeling of Ultrasonic Systems

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		4×10^5 (L s)	organic so
			mixtures
			ultrasonic
			freq: 20 kl
$P_{\max}$	final pressure	78 atm	aqueous s
	(maximum produced		ultrasonic
	by collapsed bubble)		freq: 20 kl
R_0	initial cavity size	$2.0 \mu\text{m}$	aqueous s
			ultrasonic
			freq: 20 kl
τ_m	transient bubble	$3.5 \mu\text{s}$	aqueous s
	collapse time		ultrasonic
			freq: 20 kl
$T_{\max}$	final temperature	2064 K	aqueous s

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4.4.1. Ultrasonic Velocities in Pure Fluids and Mixtures. The ultrasonic velocity (C , the speed of propagation of an ultrasonic wave) in pure fluids can be determined as a function of bulk temperature T and carbon number C_n using

$$C = a + \frac{b}{C_n} - \left(d + \frac{e}{C_n}\right)T \quad (12)$$

The constants have been determined by Wang and Nur (1991) for n -alkanes and 1-alkenes and are given in Table 3 (units of $C [=]$ m/s and $T [=]$ °C). As shown by eq 12, the velocity increases linearly as the bulk temperature of the fluid decreases. In addition, the velocity increases as a function of carbon number, but the magnitude of the increase decreases with increasing carbon number. A velocity correlation and constants are also given in the reference for naphthenes as a function of temperature alone.

Table 3. Values Given for the Constant Terms in Equation 12

compound a b d e reference					
n -alkanes	1585.6	-2482.1	3.2594	7.4070	Wang and Nur
1-alkenes	1569.4	-2379.2	3.0804	8.8784	(1991)

The compressional velocity of ultrasound in a hydrocarbon mixture can be determined from the simple relationship (Wang and Nur, 1991)

$$C_{\text{mixture}} = \sum_{i=1}^n X_i C_i \quad (13)$$

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dissipated as heat, as shown by

$$P_{\text{diss}} = \left(\frac{dT}{dt} \right)_{t=0} m C_p \quad (14)$$

where m and C_p are the mass and heat capacity of the solvent, respectively, and $(dT/dt)_{t=0}$ is the initial slope of the temperature rise of the reaction mixture versus time of exposure to ultrasonic irradiation. The initial temperature rise of the system is independent of the initial bulk liquid temperature (below 40 °C), the height of the liquid in the vessel, and the horn height (see, e.g., Kimura et al., 1996; Ratoarinoro et al., 1995a). Many authors have not reported the acoustic power dissipated in their reaction systems, making it difficult for subsequent researchers to reproduce results or compare reaction conditions.

When Hagenson and Doraiswamy (1998) used eq 14 to estimate the power dissipated in their reaction system, they found it to be inadequate. It predicted that only 33% of the power delivered by the transducer was dissipated as heat, which would indicate that the other 67% of the power was lost in the transfer process or by other means. It was concluded that eq 14 needed to be modified to account for the heat absorbed by the reaction vessel as well as the solvent, as shown by

$$P_{\text{diss}} = \left(\frac{dT}{dt} \right)_{t=0} (m_{\text{solvent}} C_{p,\text{solvent}}) + \left(\frac{dT_v}{dt} \right)_{t=0} (A_{\text{ws}} x_w) \rho_{\text{vessel}} C_{p,\text{vessel}} \quad (15)$$

where T_v is the temperature of the inner vessel wall (refer to article for figure), A_{ws} is the area of the wetted surface of the vessel, x_w is the thickness of the inner wall, and m and C_p are the

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comparison of calorimetry and the Weissler reaction as measures of ultrasonic power (Kimura et al., 1996) showed that the two methods provided similar predictions. However, the efficiency of the ultrasonic device, as measured by calorimetrically estimated energy dissipation, may not directly translate into energy utilized for generating cavitation conditions. Thus, the observed relation by Kimura et al. (1996) needs reconfirmation with different reactions.

The losses in an ultrasonic system occur in several different ways. First, there is a loss of energy in the conversion of electrical energy to mechanical (sound) energy. There are also losses by heat production, the production of cavitation bubbles, and sonoluminescence. There are losses from attenuation of energy through the fluid, also called viscous dissipation. In addition, anything added to the reactor, such as baffles, cooling coils, impellers, and solid particles, will cause losses due to reflection, absorption, and sound re-emission. The walls of the vessel result in similar losses.

4.4.3. Ultrasonic Intensity. The maximum ultrasonic intensity ($I_{\max}$) is related to the pressure amplitude (P_A) by

$$I_{\max} = \frac{P_A^2}{2\rho C} \quad (16)$$

where ρ is the density of the liquid medium and C is the velocity of sound in that medium. The intensity (I) will decrease as the distance from the transmitting source (d_T) increases, as shown by

$$I = I_{\max} \exp(-2\alpha d_T) \quad (17)$$

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The acoustic intensity can also be quantitatively determined using a chemical dosimeter such as the decomposition ratio of 5,10,15,20-tetrakis(4-sulfotophenyl)porphyrin (H_2TPPS^{4-}) (Nomura et al., 1996).

5. Applications in Organic Synthesis

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A significant amount of work has been published concerning the sonochemical effect on various systems in organic synthesis. Reviews in this area include those by Mason (1996, 1997), Bremner (1994), Low (1995), Miethchen (1992), Einhorn et al. (1989), and Davidson et al. (1987). Several books have chapters devoted to organic synthesis (see, e.g., Suslick, 1988; Ley and Low, 1989; Mason, 1990a,b; Price, 1992). Table 4 contains a representative list of organic reactions studied under the influence of ultrasound. These reactions (homogeneous and heterogeneous) are classified by the following categories:

Table 4. Representative List of Organic Synthetic Reactions Studied under the Influence of Ultrasound Homogeneous Reactions

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	Solvent: toluene (benzene and methylene chloride are also investigated as solvents and give different results) Other Diels-Alder cyclizations are reported	Yield: 77.9%	Yield: 97.3%	
B	Oxidation of indane to indan-1-one Solvent: 60:40 (v/v) mixture of pH 8.0 THAM buffer and acetonitrile	Stirring: 500 rpm Temp: 25 °C Press: 760 torr Rxn time: 3 hrs Yield: 5.27% $k_{\text{obs}} = 5.12 \times 10^{-4} \text{ s}^{-1}$	Probe system Freq: 21.5 kHz Power*: 90 W Temp: 25 °C Press: 760 torr Rxn time: 3 hrs Yield: 13% $k_{\text{obs}} = 2.96 \times 10^{-4} \text{ s}^{-1}$	Cum <i>et al.</i> (1988)
B	Alkaline hydrolysis of nitrophenyl esters Compound 1: R = CH₃ 2: R = CH₂CH₃ 3: R = (CH₂)₃CH₃ 4: R = (CH₂)₄CH₃ Solvent: 60:40 (v/v) mixture of pH 8.0 THAM buffer and acetonitrile	Stirring: Conditions not given	Bath system Freq: 20 kHz Temp: 35 °C ~ 14.5% enhancement over stirring for all 4 compounds	Kristof <i>et al.</i> (1981)
B	Solvolysis of 2-chloro-2-methylpropane Solvent: 30% ethanol (v/v) aqueous solution	Temp: 0.5 °C Conversion: 85% $k_{\text{obs}} = 2.43 \times 10^{-4} \text{ s}^{-1}$	Bath system Freq: 80 kHz Power*: 100 W Temp: 0.5 °C Conversion: 85% $k_{\text{obs}} = 4.30 \times 10^{-4} \text{ s}^{-1}$	Lorimer and Mason (1990); Mason <i>et al.</i> (1985)
C	Kornblum-Russell alkylation of nitronate anions Ultrasound favored sequential electron transfer process over the polar (simultaneous bi-electronic) mechanism Ratio of 7:3e reached a maximum with ultrasound power except in the case of standing waves, where the ratio continued to increase	Stirring: Product: 3b	Probe system Power: range of 0 - 35000 V investigated Products: 7 and 3e in various ratios Effect of standing waves investigated and found to promote S _N 1 pathway	Dickens and Luche (1991)
D	Acid catalyzed ketalization of acetophenone Follows an ionic mechanism	Stirring Temp: 12 °C Rxn time: 30 min Yield: 48% Rxn time: 3.5 hrs Yield: 94%	Bath system Temp: 12 °C Rxn time: 30 min Yield: 48% Rxn time: 3.5 hrs Yield: 94%	Einhorn <i>et al.</i> (1990)

* Power rating quoted by the manufacturer; A = initiated, B = enhanced, C = changed the pathway, D = no effect

Table 4 (Continued)

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	Solvent: pentane			
B	Synthesis of 5,5-disubstituted hydantoin In one case, when R = <i>p</i>-ClC₆H₄COMe, conventional conditions did not yield the desired product (2), but the use of ultrasound led to an isolated yield of (2) of 96%.	Stirring Several runs studied (with different substrates) Run time: 4–26 hrs Temp: 58–70 °C Yield: 0–92%	Bath system Same set of reactions studied Run time: 3–4.5 hrs Temp: 45–50 °C Yield: 45–96%	Li et al. (1996)
B	Epoxydation of long-chain unsaturated fatty esters MCPBA = <i>m</i>-chloroperoxybenzoic acid Also experimented with reagent MMPP (magnesium mono-peroxyphthalate) in place of MCPBA Other examples of epoxidation reactions are given in article	Stirring Conditions not specified Run time: 2 hrs Yield: 48%	Probe system Freq: 20 kHz Temp: 20 °C Run time: 15 min Yield: 92%	Lie Ken Jie (1995)
	Use of ultrasound in the preparation of biologically active compounds			Low (1995)
B	Oxidation of arylalkanes Solvent: water Other examples of oxidation reactions are given in article	Stirring: 150 rpm Temp: 30–35 °C Run time: 4 hrs Yield: 12%	Bath system Freq: 23 kHz Power*: 120 W Temp: 30–35 °C Run time: 4 hrs Yield: 80%	Soudagar and Samant (1995a)
B	Grignard reagent formation (several examples are given and different factors are varied and investigated)			Tadumets et al. (1995)
B	tert-Butyloxycarbonylation of amines from their salts 16 BOC preparations reported	Stirring Run time: >24 hrs	Bath system Run time: 1.5–6 hrs Yield: >90% Solvent: Ethanol Also used methanol in some cases.	Einhorn et al. (1991)
B	Michael addition of nitroalkanes to monosubstituted α,β-unsaturated esters PTC: Aliquat® 336 Other examples of Michael addition reactions are given in article	Temp: 40 °C Run time: 2 days Yield: 85%	Bath system Freq: 60 kHz Power*: 80–160 W Temp: 25 °C Run time: 2 hrs Yield: 90%	Jouglet et al. (1991) Note: several other reactions are given in article

Table 4 (Continued)

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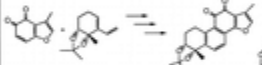
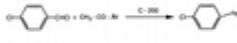
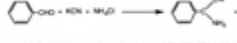
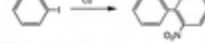
	Solvent: hexane Other examples of reactions involving inorganic solids in organic solutions given in article			
B, D	Acylon condensation and cyclization of carboxylic esters (15 examples given in article)			Faddl <i>et al.</i> (1990)
B	The α -alkylation of 5-hydroxy chromones  RX = benzyl bromide Solvent: <i>N</i> -methyl pyrrolidone (NMP) Other examples are given in article.	Stirring Temp: 65 °C Rxn time: 105 min Yield: 48%	Probe system Temp: 65 °C Rxn time: 60 min Yield: 79%	Mason <i>et al.</i> (1990c)
B	Diels-Alder cycloaddition of α -quinone  Ultrasound increased yield of cycloadducts and improved regioselectivity. Other examples are given in article.	Refluxing with benzene Yield cycloadducts: 15% (A:B) = (1:1)	System not specified Yield cycloadducts: 76% (A:B) = (5:1)	Lee and Snyder (1999)
B	Synthesis of chalcones by Claisen-Schmidt condensation  Solvent: ethanol (99%) Catalyst: activated barium hydroxide C-200	Stirring Temp: 25 °C Rxn time: 60 min Yield: 5% Catalyst wt: 1.0 g	Bath system Temp: 25 °C Rxn time: 10 min Yield: 76% Catalyst wt: 0.1 g	Fuentes <i>et al.</i> (1987)
B	Strecker synthesis of α -aminonitriles using an alumina support  Support: Al_2O_3 (improves the selectivity of PhCH(CN)NH_2) Solvent: acetonitrile Other examples of Strecker synthesis are given in article	Stirring Temp: 50 °C Rxn time: 24 hrs Yield: 64%	Bath system Temp: 45 kHz Power*: 100 W Temp: 50 °C Rxn time: 24 hrs Yield: 90%	Hanafusa <i>et al.</i> (1987), see also Mondor <i>et al.</i> (1986)
B	Preparation of tricyclohexylborane by hydroboration 	Stirring Temp: 25 °C Rxn time: 24 hrs	Bath system Temp: 50 kHz Power*: 150 W Temp: 25 °C Rxn time: 1 hr	Lindley and Mason (1987b) Note: several other reactions are given in article
B	Ullmann coupling of 2-iodonitrobenzene  Mole ratio of Cu / substrate = 4 Copper powder preincubated for 15 minutes	Stirring Temp: 63 ± 1 °C Rxn time: 2 hrs Yield < 1.5%	Microtip probe system Temp: 20 kHz Power*: 135 W Temp: 63 ± 1 °C Rxn time: 2 hrs Yield: 70.4%	Lindley <i>et al.</i> (1987a, 1986)
B	Platinum catalyzed hydrosilation of alkenes $\text{H}_2\text{SiCl}_2 + n\text{-C}_6\text{H}_{13}\text{CH=CH}_2 \xrightarrow{\text{Pt} / \text{C}} n\text{-C}_6\text{H}_{13}\text{CH}_2\text{CH}_2\text{SiCl}_2$ Other examples of hydrosilation reactions are reported	No reactions are given for direct comparison. Wagner and Strohmer (1993) have performed these types of reactions at 45-115 psi and 100-300 °C.	Bath system Temp: ~ 30 °C Pressure: atmospheric Rxn time: 1 hr	Han and Boudjouk (1983)

Table 4 (Continued)

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	 Various solvents and types of zinc powders were tested Other examples of Reformatsky reaction are given in article	Yield: 50%	Temp: 25 - 30 °C Rxn time: 30 min Yield: 98%	Boudjouk and Han (1984)
B	Selective reduction of α , β -unsaturated carbonyl compounds in the presence of Zn-NiCl ₂ Solvent: Ethanol and water Nine other examples are given in the article.	Stirring Rxn time: 48 hrs Conv: 62%	System conditions not specified Rxn time: 2.5 hrs Conv: 97%	Petrier and Luche (1987a), see also Petrier and Luche (1987b)
B	Barbier reaction: retention of optical activity from S(+) 2-octyl halides Solvent: tetrahydrofuran (THF) Other examples are given in the article with Cl or I as the halide	Stirring Temp: 0 °C Rxn time: 7 hrs Yield: 50% Configuration: R(+) %e.e.: 6	Probe system Temp: 0 °C Rxn time: 30 min Yield: 59% Configuration: R(+) %e.e.: 10	de Souza-Barboza <i>et al.</i> (1987), see also de Souza-Barboza <i>et al.</i> (1988), Luche <i>et al.</i> (1987a)
B	Aromatic carboxylic acid ester saponification Increased the yield or obtained similar yields to stirring in shorter reaction times.	Reflux Rxn time: 90 min	Probe system Freq: 20 kHz Rxn time: 10 min	Moon <i>et al.</i> (1979), Moon (1987)
C	Change in pathway from the Friedel-Crafts reactions to nucleophilic substitution KCN / Al ₂ O ₃ Mechanical agitation KCN / Al ₂ O ₃)))	Stirring Temp: 50 °C Product: o- and p- benzyltoluene Yield: 75%	Bath system Freq: 45 kHz Power*: 200 W Product: benzyl cyanide Yield: 71%	Ando <i>et al.</i> (1984a)

* Power rating quoted by the manufacturer; A = initiated, B = enhanced, C = changed the pathway, D = no effect

- A. Ultrasound initiates the reaction.
- B. Ultrasound accelerates the rate of the reaction.
- C. Ultrasound changes the reaction pathway.
- D. Ultrasound has little or no effect on the reaction.

As is evident when the examples given in the table are reviewed, reactions following an ionic mechanism are often unaffected, or are only slightly affected, by ultrasound. In some cases ultrasound actually changes the pathway of the reaction by favoring free-radical or SET (single electron transfer) mechanisms in reactions which follow either an ionic or free-radical pathway, depending on the reaction parameters. Sonochemical activation and/or enhancement of

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some of the bond cleavages observed as a result of sonication, the role of transient high pressure, electron exchange, and stereochemical effects must be investigated in greater detail (Luche, 1996). The importance of electron exchange was recently demonstrated by Takizawa et al. (1996) using the hydroxylation of phenolic compounds.

Ultrasound has been found to be very useful in the degradation of contaminants in water (Hoffmann et al., 1996), such as the chlorofluorocarbons CFC 11 and CFC 13 (Cheung and Kurup, 1994); HCFC-225ca, HCFC-225cb, and HFC-134a (Hirai et al., 1996); the pesticide parathion (Kotronarou et al., 1992); sodium hypochlorite (Mason et al., 1996a); pentachlorophenate (Petrier et al., 1992a); and *p*-nitrophenol (Kotronarou et al., 1991). Sonication also led to the complete destruction of aqueous solutions of chlorinated hydrocarbons such as methylene chloride, carbon tetrachloride, trichloroethylene, and α -1,1,1-trichloroethane (Cheung et al., 1991; Toy et al., 1990).

5.1. Homogeneous Systems. 5.1.1. Aqueous Systems. The use of ultrasound in aqueous solutions leads to the sonolysis of the components of the solution, such as $\text{H}_2\text{O} \rightarrow \text{OH}^\bullet + \text{H}^\bullet$ (see, e.g., Henglein, 1985, 1987). The free radicals in these solutions were detected by spin trapping and ESR measurements (Riesz et al., 1990; Krishna et al., 1987; Makino et al., 1982, 1983; Henglein and Kormann, 1985), a terephthalate dosimeter (see, e.g., Fang et al., 1996), and a Fricke solution dosimeter (see, e.g., Jana and Chatterjee, 1995). In addition, the mechanism of radical formation was investigated using isotopic techniques (see, e.g., Fischer et al., 1986). Sonolysis of aqueous mixtures will be discussed in greater detail in section 8.

5.1.2. Organic Systems. As in the case of aqueous systems, the enhancing effects of ultrasound in organic systems are not directly related to thermal effects but are instead a result of acceleration of the single-electron-transfer (SET) process. The SET step is required as an initial step in several reactions, such as cycloadditions involving carbodienes and heterodienes (Nebois et al., 1996). In the cases where the reaction mechanism did not seem to require an SET step, ultrasound was found to have little or no effect on the reaction. It has also been found that adjusting the system to obtain standing waves has the favorable result of increasing a sequential electron-transfer process and promoting it over a bielectronic mechanism (Dickens and Luche, 1991).

The degradation of CCl₄ was investigated in a variety of organic alcohols at two different

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5.2. Heterogeneous Systems. 5.2.1. Liquid–Liquid Systems. Ultrasound forms very fine emulsions in systems with two immiscible liquids, which is very beneficial when working with phase-transfer-catalyzed or biphasic systems (as will be discussed in sections 7.1 and 7.2). When very fine emulsions are formed, the surface area available for reaction between the two phases is significantly increased, thus increasing the rate of the reaction. This aspect of ultrasound has also been used for coal, oil, and water mixtures to increase the efficiency of combustion, as well as decrease the amount of pollutants produced during the combustion process (Dooher et al., 1980).

5.2.2. Liquid–Solid Systems. The most pertinent effects of ultrasound on liquid–solid systems are mechanical and are attributed to symmetric and asymmetric cavitation. When a bubble is able to collapse symmetrically, localized areas of high temperatures and pressures are generated in the fluid. In addition, shock waves are produced which have the potential of creating microscopic turbulence within interfacial films surrounding nearby solid particles, also referred to as microstreaming (Elder, 1959). This phenomena increases the transfer of mass across the film, thus increasing the intrinsic mass-transfer coefficient, as well as possibly thinning the film. Hagenson and Doraiswamy (1998) obtained evidence of a 2-fold increase in the intrinsic mass-transfer coefficient when modeling experimental data obtained for the synthesis of dibenzyl sulfide in the presence and absence ultrasound.

The shock waves produced by cavitation bubbles increase the momentum of solid powders in solution, causing them to collide with great force. When the solid is inorganic, the particles are fractured upon collision, leading to an overall decrease in the average particle size. For example, the particle size of inorganic solid KOH was reduced from its initial size of 240 μm to 15–20 μm within 5 min of sonication (Ratoarinoro et al., 1992). Hagenson and Doraiswamy (1998) confirmed this trend when working with sodium sulfide (Na_2S). The particle size was reduced from an average initial size of 34 μm to 15–20 μm within 15 min, as shown in Figure 1. Figure 2 shows the histogram of the particle distribution, which is highly skewed to the right at lower sonication times but becomes narrower as the exposure time is increased. The minimum particle size was 10 μm because it was the pore size of the filter paper used to capture the solids for analysis, although the filtrate appeared to be clear and free of particles.

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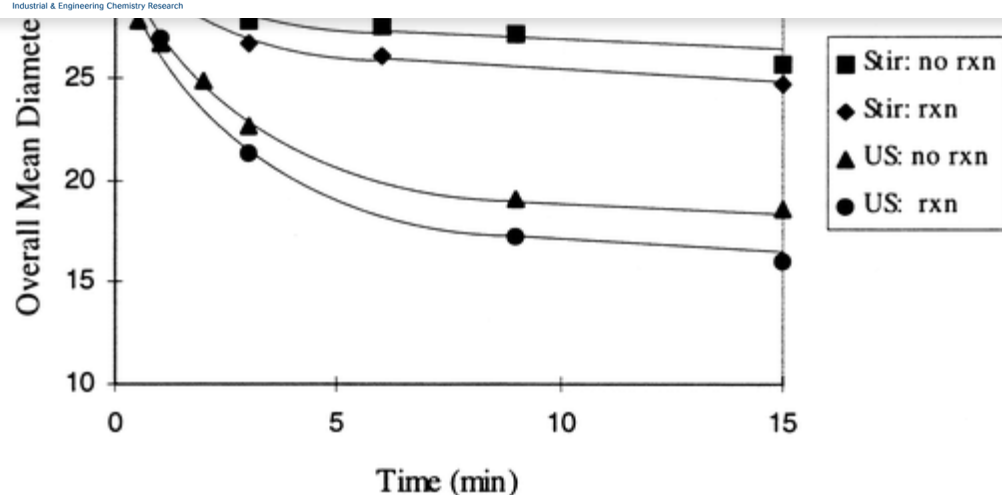


Figure 1 Particle degradation of Na_2S in the presence and absence of ultrasound with and without reaction (Hagenson and Doraiswamy, 1998).

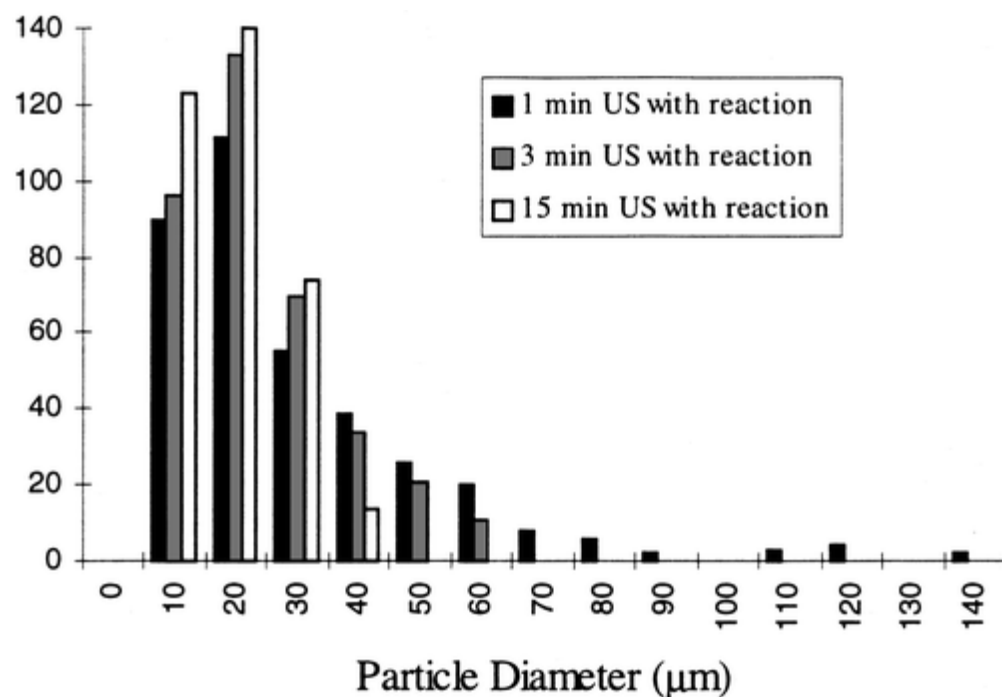


Figure 2 Histogram of average diameter data for samples of Na_2S in the presence of ultrasound (Hagenson and Doraiswamy, 1998).

The effect of ultrasound on the particle size reduction of the inorganic particles TeSe and MoO_3

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momentum of the particle becomes too small to create the impact required to cause particle fragmentation.

When the bubble is collapsing near a solid surface which is several orders of magnitude larger than the cavitating bubble [i.e., a surface greater than 200 μm in diameter when operating at 20 kHz as estimated by Suslick (1990a)], symmetric cavitation is hindered and collapse occurs asymmetrically (Neppiras, 1980). As the bubble collapses, microjets of solvent are formed perpendicular to the solid surface. These microjets have an estimated speed of 100 m/s [aqueous solution; Suslick et al. (1990)] and lead to pitting and erosion of the surface, in addition to the well-known cleaning effects associated with ultrasound. This behavior leads to enhancement in some heterogeneous reactions. For example, ultrasound facilitates the synthesis of silanes or organosilicon halides using magnesium hydride particles in a liquid medium by removing surface-inhibiting halides from the magnesium surfaces, thus increasing their reactivity (Klein et al., 1995).

There is a limit to the impurities ultrasound can remove, as found by Hagenson and Doraiswamy (1998). When the solid-phase reaction of benzyl chloride and sodium sulfide was investigated, ultrasound was not successful in removing the product layer, NaCl, from the surface of the unreacted solid, Na_2S . This product layer was responsible for limiting the rate of the reaction. Unfortunately, there are not many published papers on the effects of ultrasound on inorganic impurities and surface layers.

Metal Powders. Metal powders behave differently from inorganic particles when exposed to sonication. While inorganic particles are degraded in the presence of ultrasound, metallic particles decrease in size and then tend to agglomerate with prolonged sonication (see, e.g., Doktycz and Suslick, 1990). Using a scanning electron microscope, Doktycz and Suslick (1990) studied the agglomeration of several different metals which were $\sim 10\ \mu\text{m}$ in diameter. The only metal they tested which did not fuse was tungsten, which has a melting point of 3410 $^\circ\text{C}$. From this they concluded that the maximum collision temperature reached during sonication is less than 3410 $^\circ\text{C}$. The surfaces of the particles also became very smooth over time (see also, Suslick and Casadonte, 1987). From an independent experiment, they estimated the impact velocities of these small metal particles as 100–500 m/s. Suslick's hypothesis that the metallic particles were fusing as a result of localized heating at the collision site has been discredited by

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during bubble collapse, forming $H^\bullet$ or $R^\bullet$.

Ultrasound can be used to activate extremely reactive metals such as lithium, magnesium, zinc, and aluminum, which are commonly not used as catalysts because they are prone to deactivation due to the formation of coatings on their surfaces in the presence of water or oxygen. Ultrasound was shown to remove these coatings, making the metals viable candidates for catalysis. For example, ultrasound increased the catalytic activity of Ni powder by more than 10^5 (Suslick and Casadonte, 1987). This dramatic increase was attributed to the reduction of the surface oxide layer covering the particles, as well as changes in surface morphology and the sweeping action of ultrasound. Zinc was successfully activated for the promotion of the Simmons–Smith cyclopropanation of olefins (Repic and Vogt, 1982). Several reactions carried out in the presence of lithium wire were also accelerated using ultrasound, such as the coupling of heteroaryl halides to form isomeric bipyridines (Osborne et al., 1989) and the coupling of organic halides (Han and Boudjouk, 1981). Luche (1994) investigated possible mechanisms of activation for various metal surfaces in terms of the mechanical properties of the passivating surface layer and the passivated metal.

Ultrasound also activates transition-metal salts, enabling the reaction to be carried out at much milder conditions. For example, traditional conditions used to synthesize the carbonyl anions (required to produce $V(CO)_6^-$) are 160 °C and 200 atm of CO, plus the addition of the catalyst $Fe(CO)_5$. However, with exposure to ultrasound, the same yield of $V(CO)_6^-$ was obtained at 10 °C and 4.4 atm of CO without the use of the catalyst (Suslick and Johnson, 1984).

Solid Catalysts. When the solid present in the medium acts as a catalyst, ultrasound can significantly influence the chemistry occurring within the system. The intensity of ultrasound can alter the stereoselectivity of a particular reaction, as observed in the case of the cyclization of the tetracyclic 19-iodotabersonine to the vindolinie epimers (Luche et al., 1990). It was determined that ultrasound influenced the desorption of the radical anion from the metal surface. When the intensity of the ultrasound was low, as in the case of the ultrasonic bath system, the desorption of the anion was slower and the stereoselectivity of the product was controlled by the absorption of the substrate. However, when the sound intensity was high, as in the case of the probe system, the desorption process was accelerated and the cyclization process occurred in the liquid phase, leading to lower stereoselectivity.

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5.3. Changes in material structure. Ultrasound has not been found to alter the crystalline structure of solids already present in the system (see, e.g., Ando et al., 1985; Thompson and Doraiswamy, 1998). However, it can alter the formation of crystals in its presence. Domingos et al. (1997) found a 25% increase in the mean crystallite size of a boehmite phase (aluminum monohydroxide) by sonicating it during aging. In addition, the BET surface area and the pore volume were decreased by 40%.

Ultrasound can also change the long-range ordering of certain materials. Diodati et al. (1997) synthesized an amorphous phase of palladium by sonicating a mixture of palladium acetylacetonate and toluene. The disordered phase of palladium had the same X-ray diffraction spectra as crystalline Pd, indicating that it was indeed a disordered phase of Pd and not a Pd-based compound such as PdH or PdO. The phase became increasingly disordered as the concentration of palladium acetylacetonate increased. However, it appeared to be independent of acoustic power.

Ultrasound changed the normal bonding of water contained in an ammonium alum crystal lattice (Homer et al., 1995). Water molecules moved through the lattice when under the influence of sonication, as determined using NMR, and returned to their equilibrium sites when the ultrasound was removed. This mobility during sonication actually modified the normal melting point of the crystals of ammonium and potassium alums. It was speculated that ultrasound may also alter the normal melting points of other crystals which contain water within their crystalline lattice.

During investigations focused on the dispersion of metal powders in the presence of ultrasound, the ease of dispersion of the powder in a particular solvent increased as the lattice energy of the metal decreased (Luche, 1987). For example, lithium could not be dispersed in toluene because the lattice energy of the metal was greater than the energy liberated by the cavitating solvent.

5.4. Modeling. Modeling the effects of ultrasound in heterogeneous systems tends to be extremely complicated. Several authors have tackled segments of the problem. The absorption of ultrasound in liquids containing clusters of molecules was modeled by Lewis et al. (1991). The propagation of ultrasonic waves in suspensions and emulsions was modeled by Ahuja (1972, 1973) and Allegra et al. (1972), while Ahuja and Hendee (1978) looked at the effects of particle shape and orientation. Edwards and Jarzynski (1983) investigated the scattering effects

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6. Other Applications

6.1. Organometallic Chemistry. Table 5 lists a number of organometallic reactions that have been investigated under the influence of ultrasound [refer to the reviews by Luche (1987) and Suslick (1986) and the books by Price (1992), Mason (1990a), Ley and Low (1989), and Suslick (1988) for more information on organometallic chemistry].

Table 5. Organometallic Reactions

reaction type	effect of ultrasound	reference
reduction of hexacyanoferrate(III) by thiosulfate ions mediated by $\text{RuO}_2 \cdot x\text{H}_2\text{O}$		activation
hydrostannation of several different substrates with Ph_3SnH		rate enhancement
synthesis of triorganylboranes		increase in yield
formation of iron–chromium catalyst ($\text{Fe}_2\text{O}_3\text{--Cr}_2\text{O}_3$)		obtained in higher yields
synthesis of π -allyltricarbonyliron complexes		
ligand substitution of metal carbonyls		rates were increased
formation of lithium organometallic reagents		greatly increased
oxidation of aerated aqueous Fe^{2+} solutions in the presence of aliphatic alcohols		oxidation rate

6.2. Biotechnology. The use of ultrasound in the field of biotechnology has been investigated extensively (e.g., Sinisterra, 1992; Price, 1992). One of the most well-known uses of ultrasound in biotechnology is for microbial cell disruption (see, e.g., Suslick, 1988). When cellular material is placed in an ultrasonic field, the shock waves produced by surrounding cavitation events (as discussed in section 5.2.2) are capable of causing mechanical damage to the surrounding cellular material. These effects are not described in detail in this paper. For more information

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synthesis step in the production of α -, β -, and cyclic spaglumic acids (Veera Reddy and Ravindranath, 1992b).

6.3. Polymerization. Reviews and books available in the literature concerning the use of ultrasound in polymerization processes include those by Price (1992, 1996), Urban and Salazar-Rojas (1988), Lorimer and Mason (1987), and Mason (1990a,b). Ultrasound accelerates the rates of free-radical polymerization and copolymerization and, in some cases, eliminates the need for an initiator. It also accelerates emulsion polymerization because of its ability to create fine emulsions (refer to section 5.2.1). Suspension polymerization was enhanced because ultrasound prevented agglomeration of monomer droplets (Hatate et al., 1981). The mechanism of polymerization in the presence of ultrasound is very complex. In addition to combination of radicals with the primary polymer chain, existing molecules are fractured within the cavities and may combine with other molecules (see, e.g., Price et al., 1990).

Koda et al. (1996) investigated the copolymerization of sodium styrenesulfonate and vinylpyrrolidone in an aqueous system at three different frequencies (20, 40, and 540 kHz) and various acoustic intensities. The rate of polymerization increased with acoustic intensity at 20 and 40 kHz but was independent of intensity at 540 kHz. The highest conversions were obtained at 40 kHz, when the reaction rate was approximately twice as fast as the rate at 20 kHz. They used the kinetic model of Kruus and Patrabody (1985) to explain their experimental data.

Using a high-power ultrasonic probe system, the polymerization of methyl methacrylate was carried out without the use of an initiator (Kruus and Patrabody, 1985), producing a polymer with an average molecular weight of 400 000. The rate of polymerization was proportional to the square root of intensity. A reaction mechanism was speculated which appeared to explain the experimental observations and produce rate constants of polymerization comparable to literature values.

The polymerization of poly(vinylpyrrolidone) (PVP) was initiated from the sonication of vinylpyrrolidone monomer at frequencies of 20 and 40 kHz, without the need for a catalyst (Koda et al., 1995). Polymerization did not occur at 540 kHz. At the lower frequencies the yield of PVP increased linearly with sonication time. However, polymerization was terminated immediately when irradiation was stopped. Thus, sonication was required for the initiation and maintenance of the polymerization reaction.

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traditional methods. The degradation rate of the polymer was related to the power and sonication time (see also, Portenlanger and Heusinger, 1994) and then used to predict the mass percent of the polymer chain.

6.4. Medicinal Uses. A number of publications and patents are available concerning the use of ultrasound for medicinal purposes. As discussed previously, ultrasound promotes the production of free radicals in a given solvent. Similarly, continuous 1 MHz, 1 W/cm² sonication enhances the hydroxyl radical production of two anticancer drugs, adriamycin and mitomycin C (Tata et al., 1996). It was speculated that the enhancement of these redox cycling drugs occurs according to Fenton's pathway. Exposure to ultrasound for 30 min did not alter the chemical structure of the drugs.

Mitragotri et al. (1996) found that using low-frequency (20 kHz) ultrasound greatly enhances transdermal transport of several drugs when compared to using therapeutic levels of ultrasound (1–3 MHz). The use of ultrasound for the purpose of transdermal transport is known as sonophoresis. The authors have found enhanced transport of several permeants in vivo (human cadaver epidermis), including insulin (Mitragotri et al., 1998), estradiol, salicylic acid, corticosterone, and sucrose. The use of ultrasound for transdermal delivery of encapsulated drugs was patented by Mitragotri et al. (1998). A review of sonophoresis and explanations for the observed variations in transdermal enhancement from drug to drug can be found in work by Migragotri et al. (1997). To date, the use of ultrasound for sonophoresis does not appear to lead to long-term loss of the barrier properties of the skin.

Ultrasound was used in a patented process in which pharmaceuticals were delivered to the body from performed porous polymeric microparticles (Supersaxo and Kou, 1995). These microparticles release low levels of a particular drug for prolonged periods of time, providing up to 30 days of controlled release. The release rate could be increased by up to 3 orders of magnitude upon exposure to ultrasound. In the absence of irradiation, the release rates returned to the original, presonicated levels. It was the authors' hope that ultrasound could be used to deliver high doses of drugs, when needed, while continually maintaining the lower dosages in its absence.

Kost (1993) investigated the use of ultrasound to enhance the delivery of clinically useful drugs from polymeric supports. He found that peptides, proteins, and other drugs could be delivered at

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7.1. Phase-Transfer Catalysis. Several authors have combined the use of ultrasound and phase-transfer catalysts (PTC) (see, e.g., Goldberg, 1992). These two enhancement techniques complement each other greatly when they are used for polyphase reactions because ultrasound has the ability to form fine emulsions between liquid-liquid phases, thereby increasing the interfacial area available for reaction. A PTC bridges the barrier between the aqueous and organic phases, facilitating reaction between them. Hagenson et al. (1994) studied these combined enhancement techniques for the synthesis of dibenzyl sulfide from benzyl chloride and sodium sulfide. They found that after approximately 4 min of reaction, the conversion of benzyl chloride was enhanced by a factor of 1.5 in the presence of ultrasound alone, 5.9 in the presence 0.4% w/v of the PTC tetrabutylammonium bromide (TBAB), and 6.5 in the presence of both PTC and ultrasound, as shown in Figure 3. As is evident for this particular reaction, the use of ultrasound alone was not adequate to obtain a high yield of dibenzyl sulfide. The catalyst was required to assist with the ion-exchange reaction between the sparingly soluble Na_2S and the organic liquid reactant benzyl chloride. However, ultrasound enhances the reaction even further through mechanical effects such as particle size degradation and mass-transfer facilitation through the solid-liquid film (Hagenson and Doraiswamy, 1998).

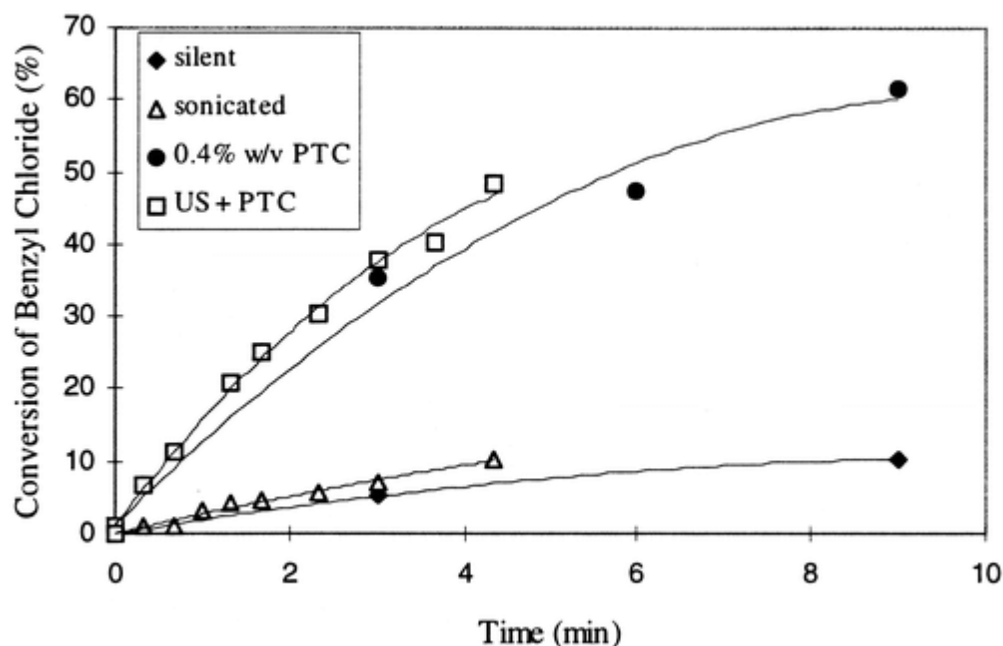


Figure 3. Conversion of benzyl chloride as a function of time for the reaction of benzyl chloride with sodium sulfide in the presence of ultrasound and phase-transfer catalyst (PTC).

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provided in their paper, as shown in Figure 4. When ultrasound is used in combination with the PTC, the reaction rate is initially much greater than that when ultrasound is used alone. However, with continued reaction time, the conversion in the case of ultrasound alone matches the conversion of ultrasound + PTC. Thus, when use of a variety of enhancement techniques is attempted, preliminary rate data may give a good indication of whether several techniques are required. One technique alone may be sufficient and may give comparable conversions without the costly separation processes required with the addition of a separate catalyst.

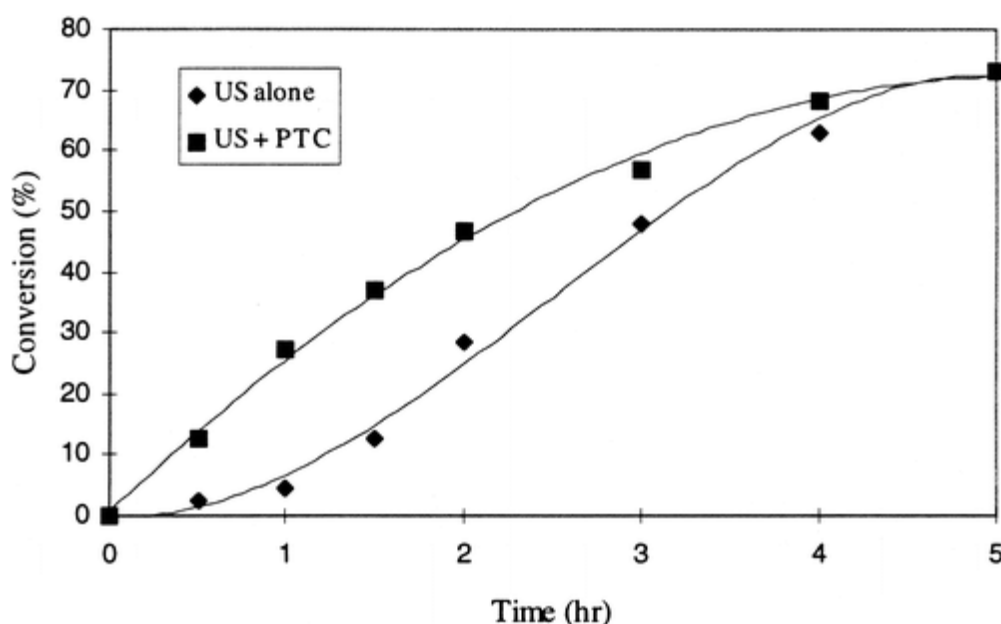


Figure 4 Use of ultrasound and the combination of PTC and ultrasound on the synthesis of Tos_3TACN (Madison et al., 1994).

Table 6 provides examples of other reactions which investigated the combination of the enhancement techniques of ultrasound and phase-transfer catalysis.

Table 6. Reactions Which Have Been Investigated Using PTC, Ultrasound, and/or Combinations of the Two Enhancement Techniques

reaction typeeffect of ultrasoundreference
enhancement of the Cannizzaro reaction

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dialkylation and cyclodialkylation of ethyl cyanoacetate
o-alkylation of 5-hydroxychromone derivatives with alkyl bromides
synthesis of RCN compounds from RBr and KCN supported on alumina
synthesis of silicon-containing aziridines from dichlorocarbamates and vinyl- or allylsilanes v

7.2. Biphasing. Biphasing is the addition of an immiscible phase to the traditional reaction mixture in order to increase the yield of the desired product by shifting the thermodynamic equilibrium in the direction of the products. The ability of ultrasound to create fine emulsions is very beneficial to biphasic systems. For example, it was used successfully to enhance the oxidation of secondary alcohols with sodium bromate in an aqueous- CCl_4 biphasic system mediated by ruthenium tetroxide (Mills and Holland, 1995). For each of the four alcohols investigated, the reaction time was decreased from several hours to several minutes, and the yield of ketone in each case was 100% when ultrasound was used in conjunction with supplementary stirring. The significant increase in reaction kinetics due to ultrasound was attributed primarily to the increased interfacial area. In a separate paper, the production of chloropropionamide in the biphasic system of water and 3-chloropropionitrile in the presence of ultrasound was investigated (Farhat and Berchiesi, 1992). Ultrasound did not appear to affect the Cl-C bond.

7.3. Surfactants. The addition of surfactants to ultrasonic systems reduces the surface tension of the liquid, thus reducing the cavitation threshold and facilitating the generation of bubbles. Some surfactants inhibit the production of free radicals when compared to the sonolysis of nonsurfactant solutes (Alegria et al., 1989). This effect was evident by the decrease in the formation of H_2O_2 during the sonolysis of aqueous solutions. Three surfactants were examined: sodium dodecyl sulfate, dodecyltrimethylammonium bromide, and *n*-octyl β -D-glucopyranoside (OGP). All three appeared to localize at the gas-liquid interface of the cavitation bubble.

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the mechanical effects of ultrasound, such as particle degradation, surface cleaning, and increase mass transport.

8. Kinetic Analysis

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8.1. Reaction Zones. EPR and spin trapping studies of volatile and nonvolatile solutes have provided evidence of three regions of sonochemical activity (i.e., three reaction zones) in sonicated systems (Riesz et al., 1990). The three reaction zones, shown in Figure 5, are as follows:

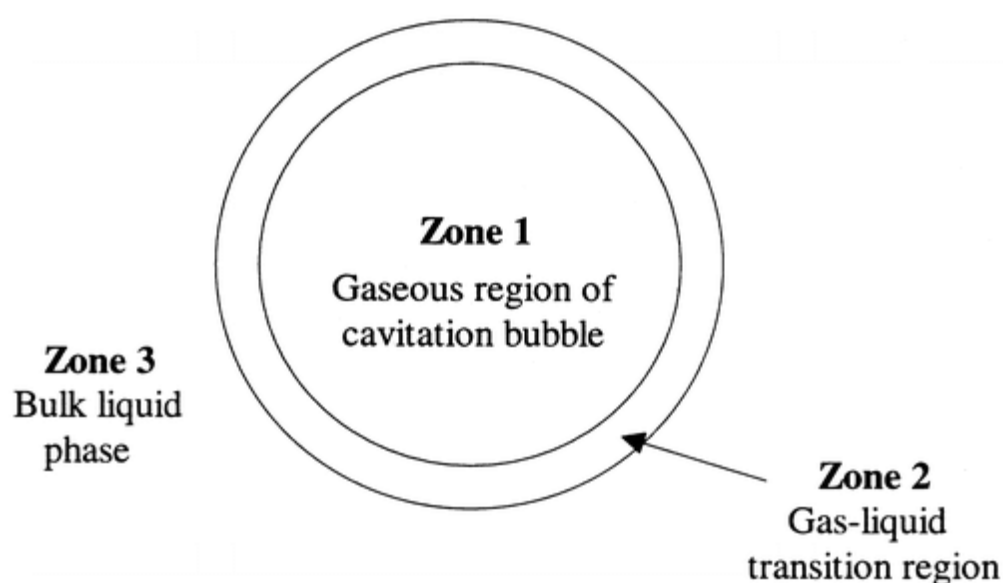


Figure 5 Three reaction zones of sonochemical reactions.

Zone 1. The gaseous region of the cavitation bubble containing both permanent gas and vaporized reaction mixture.

Zone 2. The gas–liquid transition region containing less volatile reaction components and surfactant (if present in the reaction system).

Zone 3. The bulk liquid phase.

The magnitude of reaction occurring in each zone depends on the ultrasonic conditions (such

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radicals ($\text{O}_2^{\cdot-}$) were produced during the sonolysis of argon-saturated aqueous solutions (Kondo et al., 1996).

Estimates of the relative sizes of the reaction zones and the effective temperature in each zone have been made by Suslick and Hammerton (1986) and are described in the following section. Reactions with nonvolatile solutes occur primarily in the interfacial (gas-liquid transition) region or in the bulk liquid phase (Sehgal et al., 1982). As discussed in section 4.2.2, Sehgal and Wang (1981) speculated that the degradation of thymine, a compound of low volatility, occurs in the interfacial region. These findings were confirmed by Kondo et al. (1988a), who found thymine radicals in both the bulk solution and the interfacial region of the cavitation bubble. It was speculated that, at low concentrations of nonvolatile solute, the reactions involved primarily secondary radical reaction, i.e., scavenging of $\text{H}^{\cdot}$ and $\text{OH}^{\cdot}$ radicals, while at high concentration of solute, pyrolysis of the solute occurs (Kondo et al., 1989a). However, both mechanisms may be occurring simultaneously at all concentrations of solute (Misik and Riesz, 1996b). Other investigations of the sonolysis of aqueous solutions of nonvolatile solutes include acetate (Gutierrez et al., 1986); dipeptides, propionate, and sugars (Kondo et al., 1989a); nucleosides (Kondo et al., 1988b); nucleotides (Kondo et al., 1989a); and polymers (Henglein and Gutierrez, 1988). The sonolysis of organic liquids reported in the literature include dimethylformamide, methylformamide, dimethylacetamide, toluene, *n*-alcohols, *n*-alkanes, cyclohexane, dioxane, and tetrahydrofuran (Misik and Riesz, 1996b). The identified radicals were formed by either pyrolysis or H abstraction. If the reactant was not volatile enough to enter the interfacial region (zone 2), the reaction was not chemically enhanced with the use of ultrasound (Ando et al., 1996; Griffing, 1952).

Bulk liquid-phase (zone 3) reactions involve radicals which escape the implosion. The kinetics are similar to that of radiation chemistry (Fuchs and Heusinger, 1995; Kondo et al., 1988a).

The sonolysis of water under atmospheres consisting of various ratios of argon and acetylene was investigated by Hart et al. (1990). They found a great proportion of the products formed consisted of two to eight carbon atoms. The products were similar to products observed in the pyrolysis or combustion of acetylene, with the interesting finding that the products with an even number of carbons were more plentiful than those with an odd number. The authors concluded that the formation of products occurred in a single step, by a single cavitation event, and not in

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the degradation of trichloroethylene. They proposed that the reaction intermediates were formed during a single cavitational event because a free-radical trap in the bulk solution had no effect on the degradation rate. Aqueous mixtures of methanol produced primarily $\text{CH}_3\cdot$ and some $\cdot\text{CH}_2\text{OH}$ radicals (Riesz et al., 1990).

8.2. Kinetic Modeling. When studying the primary radical formation of *n*-alcohols, Misik and Riesz (1996a) found the logarithm of the rate of radical formation $k_{\text{r.f.}}$ to decrease linearly with the vapor pressure of the alcohol. They speculated that the relationship could be described as

$$\ln k_{\text{r.f.}} = \ln A - \frac{E_a}{R_g T_0 P_A (\gamma - 1)} P_v \quad (19)$$

However, they did not validate the equation with experimental data. Suslick and Hammerton (1986) had earlier found the same linear relationship while studying ligand substitution of metal carbonyls. To investigate the relationship further, they performed experiments where they measured the rate constant as a function of reactant vapor pressure, while holding the total vapor pressure of the system constant. They achieved this by varying the reactant concentration, ambient reaction temperature, and solvent mixture. For two different reaction systems, they found the observed rate constant to increase linearly with reactant vapor pressure and to have a nonzero intercept. They concluded that the linear dependence represented reaction occurring within the vapor phase of the cavitational event (zone 1 in Figure 5) and the nonzero intercept gave a quantitative value of the reaction occurring in the liquid phase, which they assumed to be the gas-liquid film surrounding the bubble (zone 2) (see also Suslick, 1990a). These findings were then used to estimate the size of the bubble cavity relative to that of the gas-liquid film surrounding the bubble (2.0×10^4 :1 before collapse). In addition, the effective temperatures were estimated to be $\sim 5200 \pm 650$ and ~ 1900 K for zones 1 and 2, respectively (see also Suslick et al., 1986). The liquid reaction zone was estimated to extend ~ 200 nm from the bubble surface and had a lifetime of < 2 μs . The effective temperature estimate can be compared with the estimate of ~ 2000 – 4000 K (not zone specific) made by

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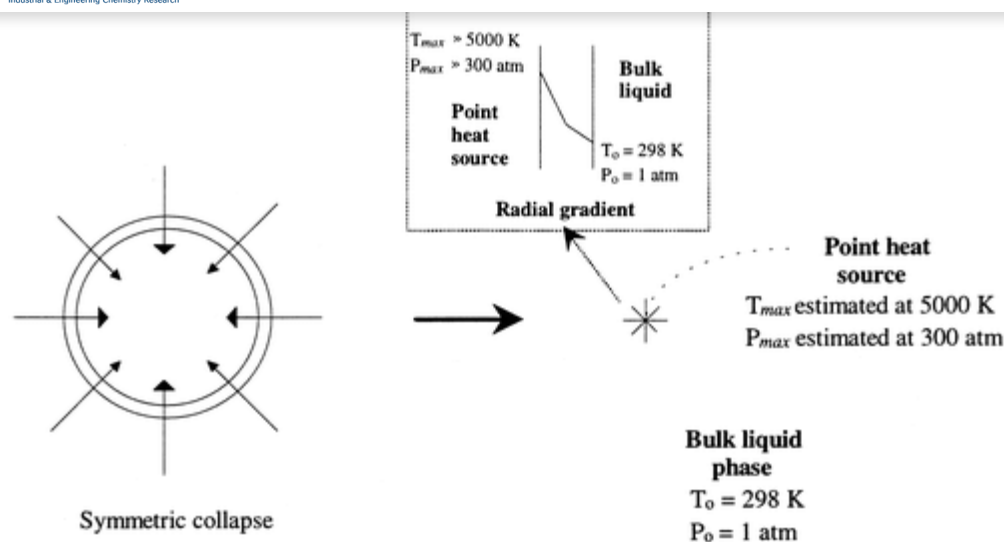


Figure 6 Symmetric collapse resulting in a localized hot spot.

The kinetic approach discussed above, and shown by eq 19, was expanded by Lorimer et al. (1991), who developed a kinetic model accounting for reaction in both the presence and absence of ultrasound. As a first step they defined three rate constants: k_{non} representing the rate constant in the absence of ultrasound, k_{US} representing the rate constant obtained in the presence of ultrasound, and k_{BUB} representing the rate constant associated with cavitation collapse. They also found that

$$k_{US} \neq k_{non} + k_{BUB} \quad (20)$$

or, in other words, the reaction rate constant in the presence of ultrasound cannot be accounted for by simply adding the rate constants in the absence of ultrasound and within the bubble region. The properties are not additive because the reaction within the cavitation region occurs at much higher temperatures and pressures than the reaction in the bulk liquid. In addition, although the reaction in the bulk is continuously occurring over time, the reaction in the bubble is only occurring for a fraction of the cycle time of the acoustic wave. Thus, two more

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$$k_{\text{non}} + F_v(k_{\text{BUB}} - k_{\text{non}}) \quad (21)$$

$$k_{\text{BUB}} = (1 - F_v)k_{\text{non}} + F_v k_{\text{et}} \quad (22)$$

where k_{et} is the rate constant of the reaction occurring at the elevated temperatures within the cavitation bubble. Substituting k_{BUB} from eq 22 into eq 21 results in

$$k_{\text{US}} = k_{\text{non}} + F_t F_v (k_{\text{et}} - k_{\text{non}}) \quad (23)$$

which, upon rearrangement, gives

$$\left(\frac{k_{\text{US}}}{k_{\text{non}}} - 1 \right) = F_t F_v \left(\frac{k_{\text{et}}}{k_{\text{non}}} - 1 \right) \quad (24)$$

Since the reaction within the cavitation bubble occurs at temperatures estimated at over 1000 times the average bulk temperature, it is reasonable to assume that $k_{\text{et}} \gg k_{\text{non}}$ (i.e., $k_{\text{et}}/k_{\text{non}} \gg 1$). It is also reasonable to assume that the reaction occurring in the cavitation bubble follows Arrhenius behavior, i.e.

$$k_{\text{et}} = A \exp\left(\frac{E_a}{R_g T_{\text{max}}}\right) \quad (25)$$

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$$T_{\max} = T_0 \left[\frac{m^{\gamma-1}}{P} \right] \quad (9)$$

Substituting eqs 9 and 25 in eq 24, and rearranging results in

$$\ln \left(\frac{k_{\text{US}}}{k_{\text{non}}} - 1 \right) = \ln F_t F_v + \frac{E_a}{R_g T_0} - \frac{E_a}{R_g P_m (\gamma - 1)} \frac{P_v}{T_0} \quad (26)$$

where P_m is the acoustic pressure at the initiation of collapse, T_0 is the ambient reaction temperature, and P_v is the vapor pressure of the reaction mixture.

Lorimer et al. (1991) found this method of analysis very promising when explaining kinetic data obtained from the solvolysis of 2-chloro-2-methylpropane in 30% (w/w) and 50% (w/w) ethanol–water solutions. They also speculated that eq 26 could be used to describe systems with nonaqueous solvents by making the modification that $k_{\text{US}} \gg k_{\text{non}}$ (i.e., $k_{\text{US}}/k_{\text{non}} \gg 1$).

Lorimer et al. (1991) did not account for possible ultrasonic effects on the activation energy and Arrhenius parameters (i.e., $E_{\text{et}} \neq E_{\text{non}}$ and $A_{\text{et}} \neq A_{\text{non}}$). Such effects occurred in reaction systems investigated by Tatsumoto and Fujii (1987), Mills et al. (1995), and Whillock and Harvey (1997a). The reason these parameters change is most likely due to the increased number of collisions of the chemical species of the reaction system, although more research is needed to elucidate these changes fully.

9. Mass-Transfer Studies

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9.1. Film Transfer. Hagenson and Doraiswamy (1998) found ultrasound to significantly increase the intrinsic mass-transfer coefficient and the effective diffusivity in a solid–liquid system: synthesis of dibenzyl sulfide from sparingly soluble sodium sulfide and benzyl chloride in the

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$$\frac{v_{A0}}{3k_l}x_A + \frac{v_{A0}}{6D_e}[1 - 3(1 - x_A)^{2/3} + 2(1 - x_A)] + \frac{R_{A0}}{k_s}[1 - (1 - x_A)^{1/3}] = \frac{vC_B M_A}{\rho_A}t \quad (27)$$

which is based on the conversion of the solid reactant A. The first term in eq 27 accounts for the diffusional resistance of the liquid reactant through the solid–liquid interfacial film, the second for the diffusional resistance of the liquid reactant through the product layer formed on the surface of the unreacted solid during the course of the reaction, and the third for the reaction occurring at the sharp interface between the product layer and unreacted solid. Because the reaction under investigation was only controlled by one of these resistances at any one time, Hagenson and Doraiswamy could extract the values of the intrinsic mass-transfer coefficient (k_l) and the effective diffusivity (D_e) from the experimental data obtained in the absence and presence of sonication. The results indicated that the intrinsic mass-transfer coefficient and the effective diffusivity were increased 2 and 3.3 times, respectively, in the presence of ultrasound as compared to stirring alone.

Ultrasound enhanced a Michael addition reaction over the use of very fast mechanical agitation (Ratoarinoro et al., 1995b). The rate of mass transfer (r_{mt}) was calculated from

$$r_{mt} = k_l a \frac{C_{A0}}{V} \quad (28)$$

where the intrinsic mass-transfer coefficient (k_l) of hydroxide ions through the solid KOH–liquid toluene film was determined using the empirical power correlation given by

$$k_l = D \left[\frac{g}{d_p} \right]^{1/4} \left(\frac{\mu}{\mu_0} \right)^{1/3}$$

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by Hagenson and Doraiswamy (1998) to compare the empirical result with their experimentally obtained value, they found eq 29 to grossly overestimate (by over 2 orders of magnitude) the value of the intrinsic mass-transfer coefficient in the presence of ultrasound. The use of this correlation as a predictive tool for k_l does not appear to be valid in the case of sonicated reactions. However, this was not critical to Ratoarinoro et al.'s conclusions concerning the enhancement of the Michael addition reaction because the reaction was kinetically controlled.

When investigating a diffusion-limited reaction, Worsley and Mills (1996) found ultrasound to increase the rate of the reaction by a factor proportional to D/δ , where D is the diffusivity and δ is the diffusion layer thickness. They concluded that ultrasound decreases the diffusion layer thickness. However, the effects on the diffusivity were not considered or investigated.

Using a three-phase sparger reactor, the effects of ultrasound were investigated for a solid–liquid and a gas–liquid reaction (Jadhav and Pangarkar, 1989). The authors concluded that ultrasound had a greater effect on the solid–liquid reaction but that the enhancement in the mass-transfer coefficient was not very significant. However, these authors used an available correlation to determine the surface area of the particles in their system and did not account for changes over time, which can be quite significant (Hagenson and Doraiswamy, 1998).

Ultrasound was shown to increase the mass transfer in a concurrent liquid–liquid extraction process and in a two-phase, three-component spray tower process. The effects of ultrasound on the liquid–liquid extraction process were investigated using a horizontal settling column with the transducer directly attached to the column (Woodle and Vilbrandt, 1960). The liquid mixture was operated plug-flow over the length of the transducer, as shown in Figure 7. The stage efficiency increased as the residence time of the liquids increased (i.e., for a power input of 50 W, the stage efficiency increased from 19% with 5.9 s of contact time to 57% with 18 s of contact time). The beneficial effects of ultrasound were attributed to the different mixing patterns of the liquid phases, as illustrated in Figure 8. Circulation currents and eddies were formed within each phase, reducing concentration gradients and removing stagnant pockets of liquid at the interfacial boundary of the two phases.

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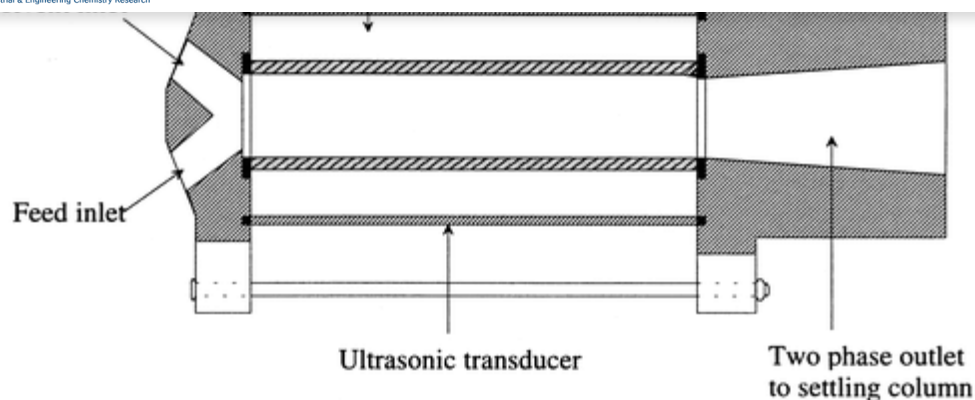
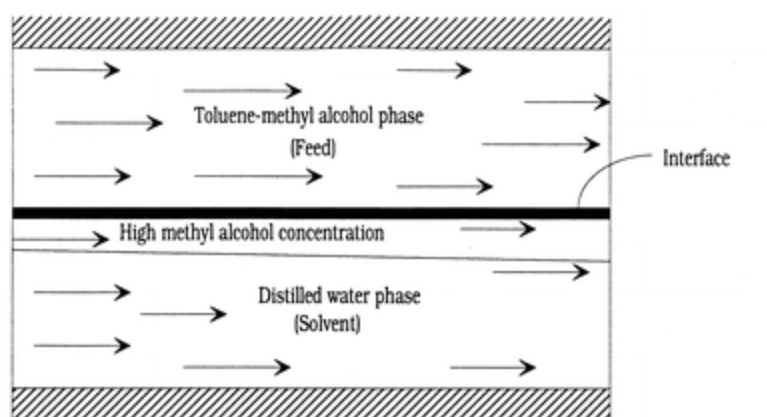
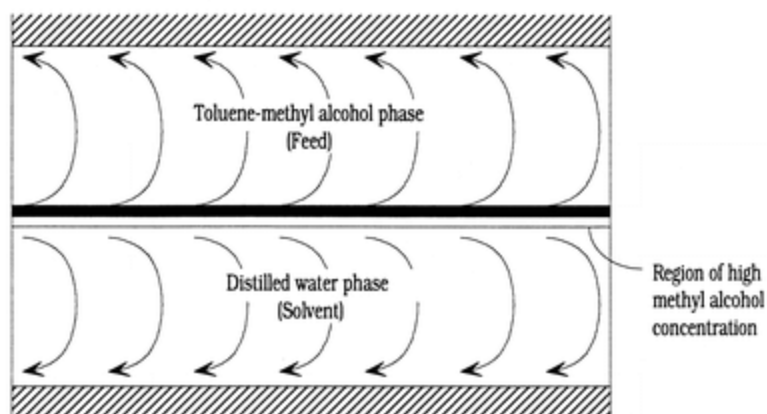


Figure 7 Ultrasonic horizontal settling column (Woodle and Vilbrandt, 1960).



(a) Extraction with no ultrasound



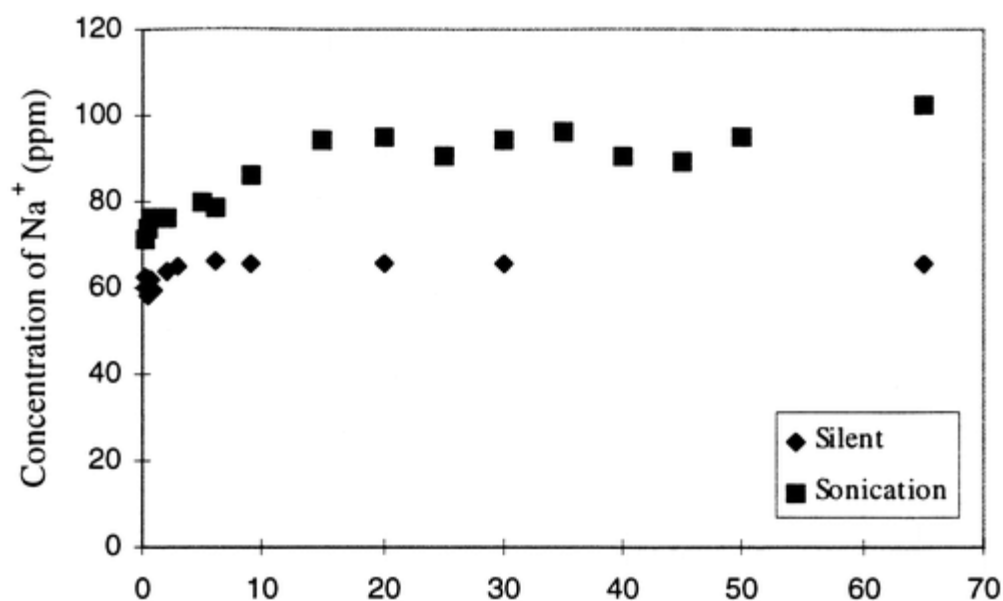
(b) Extraction upon exposure to ultrasound

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Similarly, the rate of reactive dissolution of *p*-chloranil increased in the presence of ultrasound (Booth et al., 1997). The diffusion layer thickness of the solid–liquid film decreased as the ultrasonic intensity increased and as the sample was moved closer to the tip of the horn. Using atomic force microscopy, heavy pitting of the solid surface caused by sonication was observed. On the basis of these observations, the authors suggested that transient ultrasonic cavitation played an important role in the dissolution process.

When investigating the dissolution of sodium sulfide in acetonitrile, Thompson and Doraiswamy (1998) found that ultrasound increased the driving force for mass transfer, as well as simultaneously increasing the intrinsic mass-transfer coefficient and the interfacial area. The enhancements of the latter two factors are well-known effects of ultrasound, but the supersaturation of a sparingly soluble solid solute in a solvent was not previously reported. The results of the dissolution studies are shown in Figure 9 and are plotted as average values of three to six replications which have an overall pooled standard deviation (s_p) of 5.13 (based on 101 data points). The concentration of sodium available in the bulk liquid solution reached a maximum of 1.4 times the equilibrium saturation concentration using stirring alone. The supersaturation level appears to cycle, reaching a minimum approximately every 20 min. This cycling may be the result of random fluctuations in the average or may be due to competing dissolution and precipitation processes.



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These results are very exciting because, as shown by eq 30,

$$-\frac{dC_A}{dt} = k_{sl}a(C_A^* - C_A) \quad (30)$$

it is possible to increase the rate of mass transfer in the presence of ultrasound by increasing all of the factors influencing the rate, as summarized in Figure 10. This phenomenon has great potential in processes where the reaction occurs in the bulk liquid phase or where the solubility of the solid is the limiting factor.

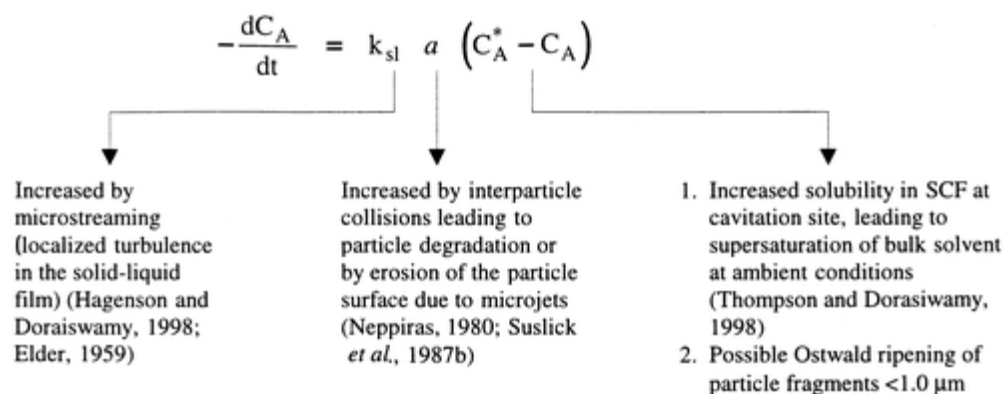


Figure 10 Ultrasonic enhancement of the rate of dissolution.

10. Methods of Producing Cavitation

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Cavitation can be generated within a fluid using transducers (devices which convert one form of energy to another). Gas-driven transducers, such as dog whistles, use high-velocity gas flow to generate ultrasound. Liquid-driven transducers, such as submarine propellers, force liquid across a vibrating plate or through an orifice, creating a cavitation zone. Electromechanical transducers, the most commonly used transducers in sonochemical research, convert electrical energy to sound energy.

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produce higher intensities in reaction mixtures as compared to ultrasonic cleaning baths. Cavitation generated in situ can reach intensities comparable to direct sonication, as found by Pandit and Joshi (1993), who hydrodynamically induced cavitation with a throttling valve to increase the hydrolysis of fatty oils. The yields and reaction conditions obtained using a hydrodynamic system were similar to those of a probe system.

Cavitation can also be induced in situ using a focused electromagnetic acoustic transducer (EMAT) which produces a high-intensity lithotripter shock wave in the fluid concerned (Carnell et al., 1995). Once the shock wave is induced, cavitational bubbles are formed in the negative pressure region of the wave, causing rupturing of the fluid. Secondary cavitational transients, created by the collapse of the primary bubbles, may also occur.

10.1. Piezoelectric vs Magnetostrictive Transducers. The two main types of electrochemical transducers used in industrial applications are piezoelectric and magnetostrictive. Piezoelectric transducers are constructed using a piezoelectric material, such as quartz, which expands and contracts in an alternating *electric* field, thus producing sound waves from the electric signal. Magnetostrictive transducers are constructed from materials, such as nickel alloys, which expand and contract in an alternating *magnetic* field. Each transducer has its own advantages and disadvantages, as outlined in Table 7 (Hunicke, 1990).

Table 7. Comparison of Piezoelectric and Magnetostrictive Transducers (Hunicke, 1990)

piezoelectric transducersmagnetostrictive transducers
relatively inexpensive
relatively small and light
damaged at temperatures >~150 °C
will age considerably, i.e., have a reduced power output, with continuous operation at high tem
may be damaged by large impact

Several methods exist to characterize the sound field of different acoustic reactors, including the chemiluminescence of luminol, the use of thermistor probes (Martin and Law, 1980, 1983) and thermoelectric probes (Romdhane et al., 1997), and the use of aluminum foil. A comprehensive review concerning characterization techniques was recently published by Hodnett and Zeqiri (1997). The PPIMP, developed by Soudagar and Samant (1995b), has the shape of a traditional ultrasonic probe and is fitted with a sandwich-type PZT piezoelectric crystal. It was designed with the intention of characterizing the sound field of ultrasonic cleaners and was shown to be an effective characterization technique.

Using an electrochemical method, Trabelsi et al. (1996) determined the active regions within their high-frequency ultrasonic reactor using an electrode and monitoring the localized current of the diffusion-controlled reduction of potassium ferricyanide. The regions with the highest current readings, leading to the highest Sherwood numbers, were taken as the most active regions in the reactor. The Sherwood number was determined by

$$Sh = k_d d_e / D_{ox} \quad (31)$$

where the mass-transfer coefficient was directly proportional to the reduction current, d_e was the electrode diameter, and $D_{ox} = 0.9 \times 10^{-9} \text{ m}^2/\text{s}$. Experiments using this technique of characterization gave conflicting results as compared to the active regions determined via calorimetry. Similar results were observed by Renaudin et al. (1994) when comparing two other methods of sound field characterization: the chemiluminescence of luminol and the use of a thermocouple probe. They found that the regions of highest acoustic intensity as determined with the thermocouple did not correspond to the regions giving the highest luminous intensity. The authors speculated that a phenomenon referred to as the “geyser” effect was occurring in the system (refer to the original reference for further explanation).

12. Ultrasonic System Types

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would require additional mechanical agitation. In addition, the bath walls would be exposed to the reaction mixture and/or irradiation, making them susceptible to corrosion or erosion.

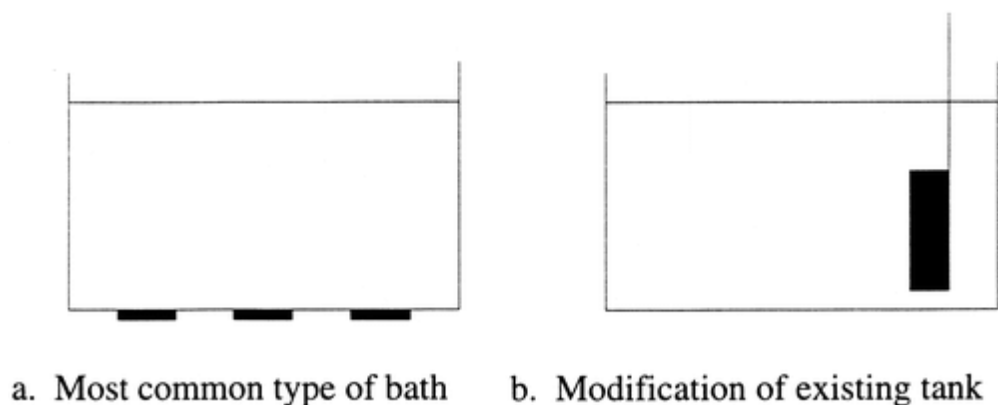


Figure 11 Ultrasonic bath systems with (a) mounted transducers and (b) submersed transducers.

When indirect sonication is used, the ultrasonic power which reaches the reaction vessel is relatively low as compared to other ultrasonic systems, such as a probe. In addition, obtaining reproducible results may be difficult because the amount of power reaching the reaction mixture is highly dependent upon the placement of the sample in the bath. The results can also vary with time as the bath warms during operation (Lickiss and McGrath, 1996). Because every bath has different characteristics, it is important to determine the optimum conditions for each bath and to place the reaction vessel in the same location for each experiment. In addition, it is important to use the same type of reaction vessel for each reaction because the shape of the bottom of the reaction vessel significantly influences the wave pattern, even when placed in the same position in the bath (Mason et al., 1994; Pugin, 1987; Weber and Chon, 1967).

Another disadvantage to using a bath system is that the coupling fluid surrounding the reaction vessel(s) will eventually increase in temperature, making the maintenance of isothermal conditions difficult. Cooling coils can be placed within the bath, but they will have an effect on the sound field and may reduce the amount of power reaching the vessel.

12.2. Probe (Horn) Systems. Probe systems, also called horn systems, are being more frequently used for sonochemical research in the laboratory. This may be because

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transducer construction, material considerations, methods of measuring input amplitude, and health and safety aspects.

Several authors have carried out experiments to determine the sound field characteristics in a probe system. The localized areas of ultrasonic intensity in a fluid is highly dependent on the power delivered to the transducer. Contamine et al. (1994) observed that when the power delivered to the system is low (i.e., 8 W), the distribution of ultrasonic intensity is characteristic of a standing wave in the axial direction. However, as the power delivered to the system is increased, the wave pattern dissipates and the intensity becomes higher near the probe tip and decreases axially. In the radial direction, they found that at low powers (i.e., 8 W) the intensity is slightly higher at the center of the reactor but is comparable over the cross section of the reactor. However, as the power delivered increases, the ultrasonic intensity increases at the center of the reactor and dissipates in the radial direction. At an input power of 200 W, the active region in the radial direction is equal to that of the horn (the remaining radial direction had negligible activity). A minimum liquid height of 1 cm must be maintained in the reaction vessel, below which the transducer does not function properly (Ratoarinoro et al., 1995a).

Aluminum foil was used to characterize the sound field of a probe system, providing the data required to develop an empirical equation (eq 32)

$$P_1 = 9.82 \times 10^{-4} s^{-1.5} \quad (32)$$

which can be used to estimate the order of magnitude of the pressure intensity (P_1), in atmospheres, as a function of distance (s), in meters, of the aluminum foil from the probe tip (Chivate and Pandit, 1995). The pressure intensities were found to be on the order of several thousands of atmospheres very near the probe tip and to decrease exponentially with distance from the tip of the horn.

12.3. Planar Transducers. This type of setup is typically made in the laboratory and consists of a planar transducer (Figure 12) connected to a vessel which contains either the reaction mixture

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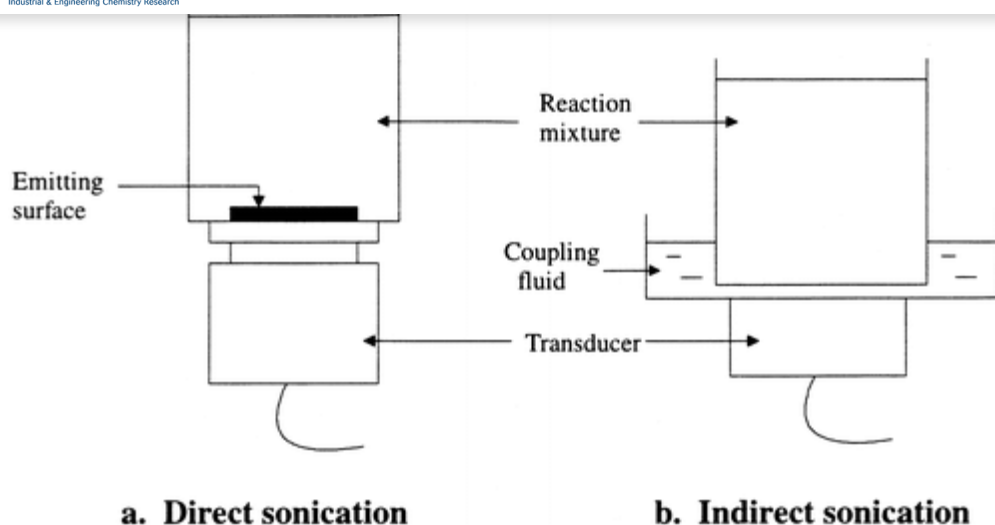


Figure 12 Planar transducer systems.

The active regions of a high-frequency (500 kHz) planar transducer system, similar to Figure 12a, were investigated by Renaudin et al. (1994). A thermocouple was used to probe the regions in the radial direction, based on the assumption that the measured temperature was directly proportional to the cavitation intensity. The ultrasonic intensity was found to be higher at the center of the vessel, directly above the emitting surface, and to decrease in the radial direction. Similar behavior was observed when Contamine et al. (1994) investigated the value of the local mass-transfer coefficient at different axial and radial positions in the reaction vessel. The axial variations in the local mass-transfer coefficient were characteristic of a standing wave. Radially, the local mass-transfer coefficient was the highest in the center of the reactor, above the emitting surface, and decreased as it moved out toward the edges. The value of the coefficient was also dependent upon the height of the liquid in the reaction vessel. This behavior was modeled by Aersten et al. in 1967 and was experimentally verified with data obtained from the liberation of Cl from CCl_4 . The equipment used was similar to that of the planar transducer system shown in Figure 12b. Starting from the basic principles describing the pressure variations of a propagating sound wave, one can write

$$p_x = P_A e^{-\alpha x} e^{i(\omega t - kh)} \quad (33)$$

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amplitude at $x = n\lambda/2$, to give

$$P_{(x=n\lambda/2)} = P_A \left| \frac{[(1 - \sigma)^2(1 - \cos 2kh)^2 + (1 + \sigma)^2 \sin^2 2kh]^{1/2}}{1 + \sigma^2 + 2\sigma \cos 2kh} \right| \quad (34)$$

where σ is the reflection coefficient at the solid–liquid interface (the contact area between the transducer and the reaction cell). Assuming 100% reflection ($\sigma = 1$), eq 34 becomes undefined at values of h where

$$h = \frac{(2n + 1)\lambda}{4}, \quad n = 0, 1, 2, \dots \quad (35)$$

This indicates that the pressure amplitude is undefined at these points, indicating maximum pressure intensity. When the liquid height in the cell is

$$h = \frac{(n + 1)\lambda}{2}, \quad n = 0, 1, 2, \dots \quad (36)$$

the pressure amplitude given by eq 34 is zero, indicating a pressure intensity of zero. As predicted with the model, a maximum in Cl_2 yield was observed when the liquid height above the transducer was equal to points given by eq 35. No observable Cl_2 was produced when the

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emitter types investigated. The yields obtained were 98% for the horn, 97% for the crucible, 91% for the cup-horn, and 77% for the bath.

13. Sonochemical Reactors with Electromechanical Transducers

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Incorporating ultrasonic technology into current reactor design is becoming increasingly important in today's industries. Currently Germany's Clausthal Technical University, with the help of several companies including Hoechst, is operating a modular sonochemical reactor which produces up to 4 metric tons of Grignard reagent/year (Ondrey et al., 1996). In France, the Electricite de France is funding the piloting of an ultrasonic electrolytic reactor to be used for the indirect oxidation of cyclohexanol to cyclohexone (Ondrey et al., 1996). (It is the hope of the present authors that this technology will be accepted and encouraged within the United States as a viable enhancement technique for several types of fine chemical processes.) Other papers in the literature concerning the scale-up of sonochemical reactions include those published by Moholkar et al. (1996), Berlan and Mason (1992), Grinthal and Ondrey (1992), Martin and Ward (1992), Mason (1990b), and Hunicke (1990).

13.1. Batch and/or Continuous-Flow Reactors. 13.1.1. Mounted Transducers. Berger's **Sonochemical Reactor**. The ultrasonic reactor design developed by Berger et al. (1996) contains 6–8 transducers built into the wall of a continuously stirred tank reactor and 3–5 transducers built into the bottom of the vessel, as shown in Figure 13. The reactor is equipped with an impeller for mechanical agitation, an external jacket for isothermal control, and reaction ports which allow for operation in the batch, semibatch, or continuous mode. The transducers may be operated independently of one another and are designed with caps protecting them from atmospheric disturbances. This reactor configuration was developed with the hope of remedying the problems of other types of sonochemical reactors, such as the efficiency and reproducibility problems typically associated with ultrasonic baths and the small active cavitation region associated with ultrasonic probes which deliver power to a narrow band of liquid directly under the probe tip. This reactor also allows for sonication of a solid–liquid system, which is a problem with vibrating plate systems which only allow for liquid–liquid systems.

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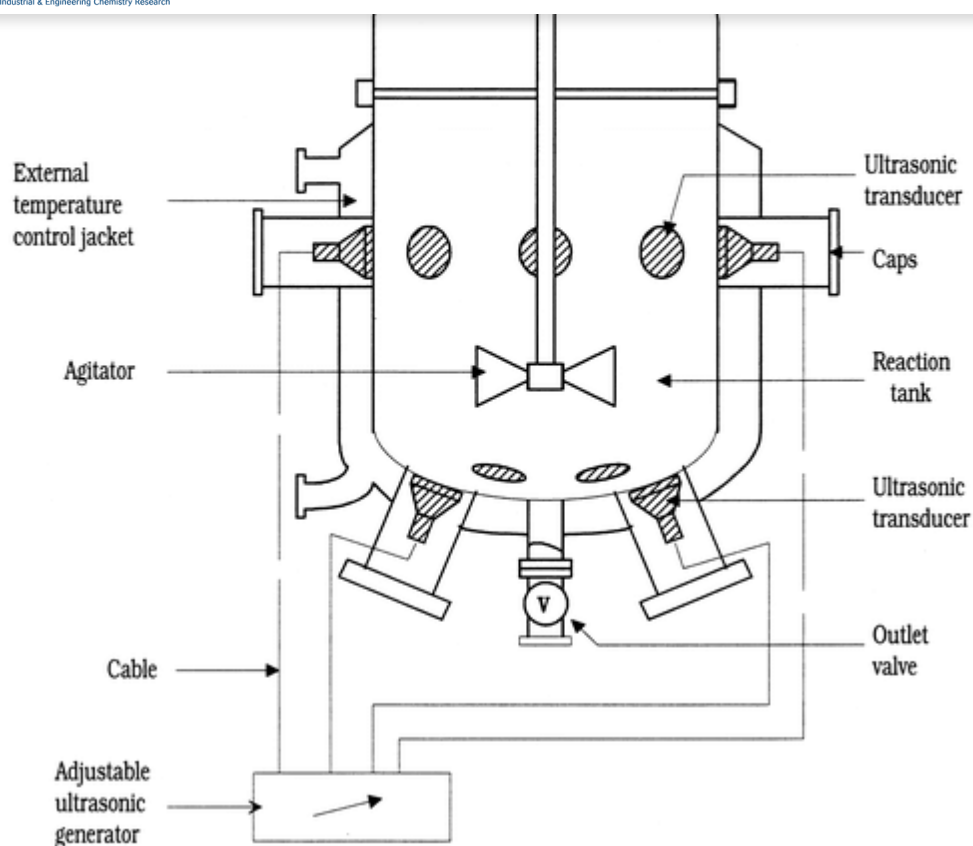
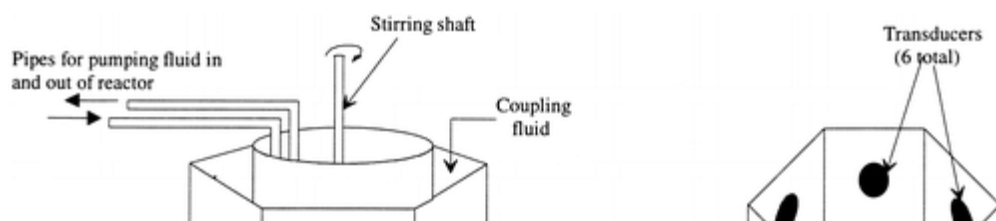


Figure 13 Sonochemical stirred-tank reactor (Berger et al., 1996).

Hexagonal Bath System. This type of sonochemical reactor provides indirect sonication to the reaction vessel, as shown in Figure 14, and the design is similar to that of an ultrasonic bath. The outer hull of the reactor is hexagonal in shape and is fitted with transducers mounted in the center of each side. The hexagonal shape is used to facilitate the attachment of the transducers, as it is very difficult to mount transducers on a rounded surface. Each transducer delivers 100 W of power, which is focused toward the center of the bath. The reactor is simply a conventional type of mixed reactor which can be operated in the batch or continuous mode.

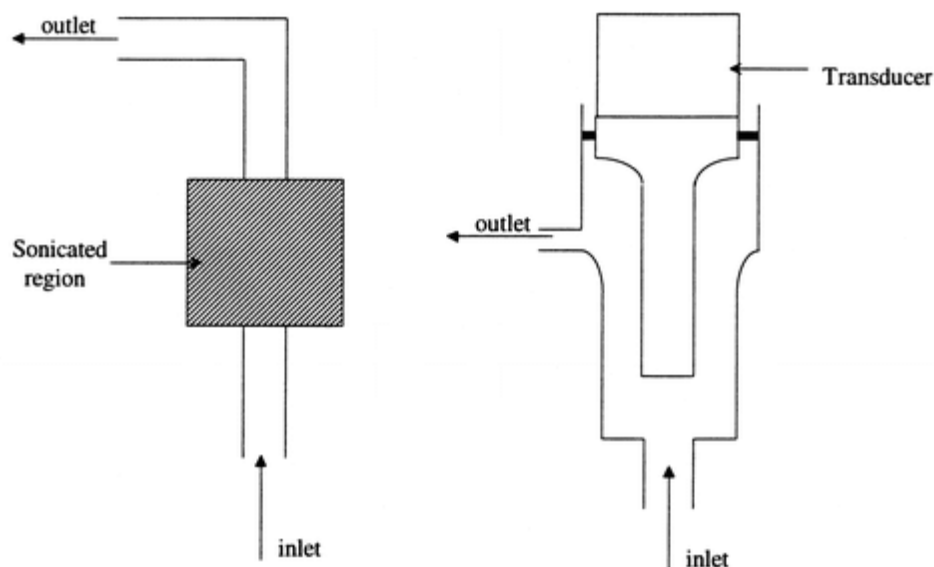


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aromatic carboxylic esters in the presence of ultrasound (Steinmetz and Matosky, 1993). While not many specifics are given, the reaction appears to be carried out in a continuous, pressurized reactor (although batch and semibatch reactions are also reported) in the presence of ultrasound. The operating conditions for the ultrasound consist of a frequency in the range of 15–100 kHz and an acoustic intensity in the range of 1–20 kW/cm² (presumably delivered intensity).

13.2. Batch Reactors with an External Flow Loop. 13.2.1. Probe Systems. Conventional batch reactors were modified by attaching an external flow loop which provides sonication to a small volume reaction mixture within the loop. This sonication can be provided using several methods, two of which are shown in Figure 15. This type of reactor has several advantages, one of the most important being that the sound fields created in small liquid volumes using probe(s) and mounted transducers have been well-characterized in the literature. In addition, the operator has control of the residence time of the mixture through the active sonication region. The external flow loop is usually modular, facilitating maintenance of equipment.



a. Mounted transducer

b. Probe system

Figure 15 Flow loops used to sonicate external streams of batch and continuous-flow reactors.

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of the connecting stream between the sonicated zone and the main reaction vessel. If, on the other hand, the species created in the sonicated zone is an intermediate required for a series reaction (i.e., if the reaction is $A \rightarrow B \rightarrow C$ and B is created in the presence of sonication), the overall reaction can be greatly enhanced using an external flow loop.

A process using a flow-through cell with an ultrasonic amplifying horn, similar to the one shown in Figure 15b, was patented for the production of aluminum compounds of the form $Al_2(OH)_6 \cdot aX_a$ (where X is Cl^- , Br^- , F^- , I^- , SO_4^{2-} , or NO_3^-) (Joshi and Parekh, 1993). The slurry consisted of 10–50 wt % of alumina in an aqueous solution where additional mechanical agitation was used (when necessary) in conjunction with the sonication. They reported using a typical frequency of 20 kHz but did not give the intensities at which they were operating. Horst et al. (1996) recently developed a concentrator horn which sonicates the fluid in a conical funnel, appearing to provide very high radiation effectiveness.

13.2.2. Mounted Transducers. Harwell Sonochemical Reactor. The design for Harwell's sonochemical reactor (Anonymous, 1990) was a collaborative effort of 19 different companies, including British Petroleum, Sandoz, Rhone-Poulenc, and ABM Chemicals. As shown in Figure 16, it is a 20 L batch reactor, fitted with an external flow loop, which sonicates a small volume of the reaction mixture and returns it to the main vessel. The sonoreactor module contains three mounted transducers which are evenly spaced around the 13 cm diameter piping containing a buffer fluid (Mason and Berlan, 1992). The transducers are not brought directly into contact with the reactive stream to avoid corrosion and to facilitate equipment maintenance. The heat exchanger is placed before the sonication zone presumably to reduce the ambient temperature of the reaction mixture in order to heighten the sonochemical effect (as discussed in section 4.2.2).

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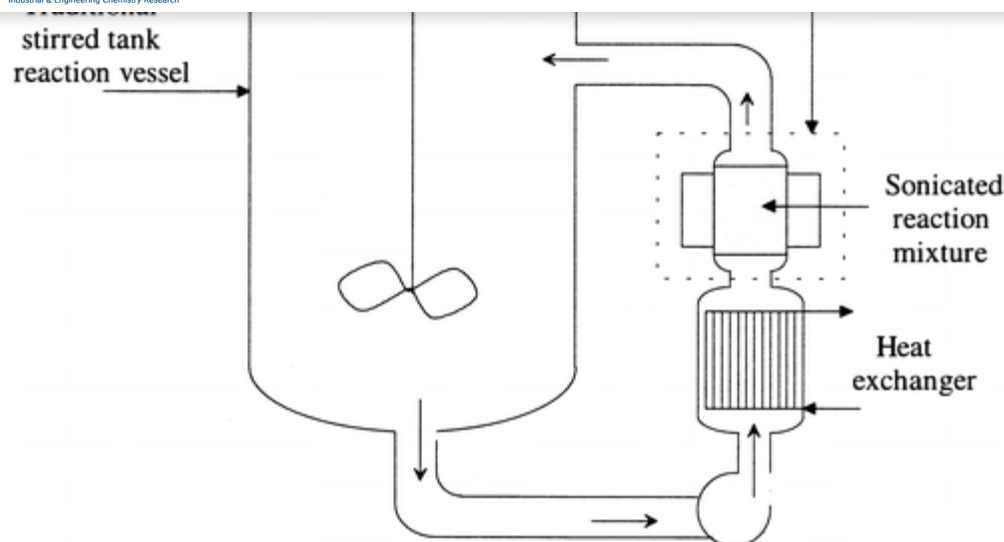


Figure 16 Harwell sonochemical reactor.

13.3. Tubular Reactors. Tubular sonochemical reactor configurations provide either direct or indirect sonication to the process stream. Several designs exist, some of which may be used to provide sonication to an external flow loop in a mixed reactor. Examples of tubular reactors are described in the following sections.

13.3.1. Probe Systems.**Branson Sonochemical Reactor.** This sonochemical reactor, manufactured by Branson ultrasonics, consists of modular units which may be combined in series, as shown in Figure 17. Each unit consists of two ultrasonic horns in contact with a coupling fluid, which is used to reduce erosion and pitting of the probe tip. Using indirect sonication also prevents contamination of the process stream with fragments of the probe tip which may be incurred during the erosion process.

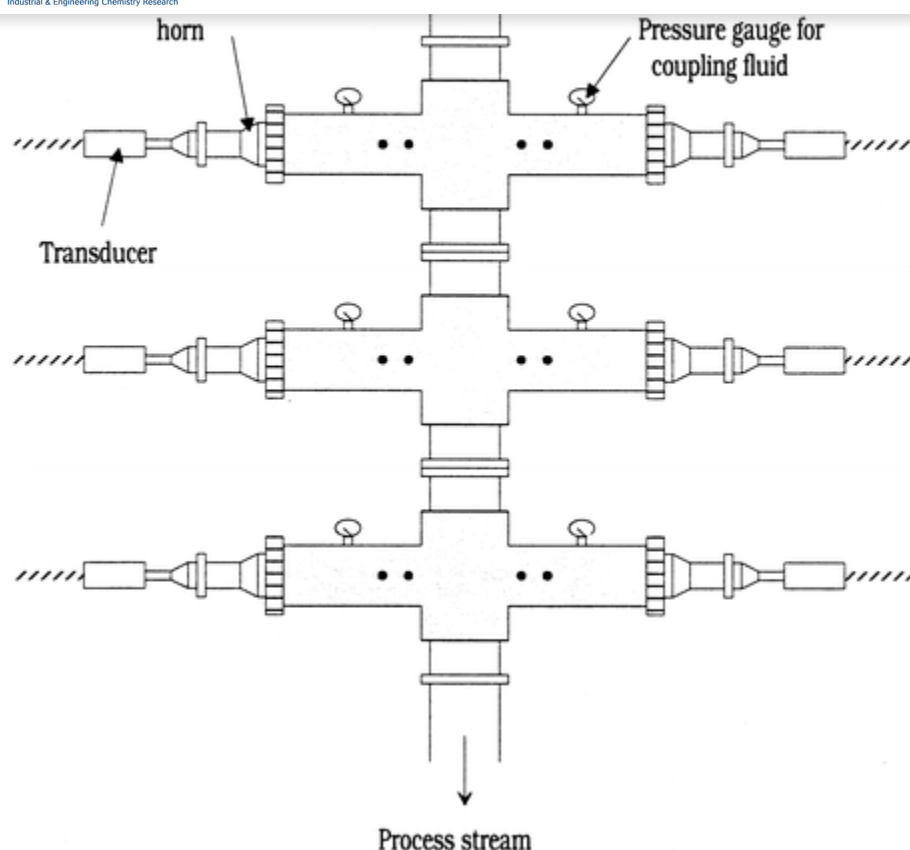


Figure 17 Branson sonochemical reactor (tubular configuration).

Ragaini's Triphase Catalyst Reactor. Two types of ultrasonic reactors were patented by Ragaini (1992) which were designed specifically to enhance polyphase reactions. The first reactor (shown in Figure 18) has the ability to emulsify the inlet liquid streams before they come into contact with a triphase catalyst (i.e., a phase-transfer catalyst bound on a polymeric support) contained in a fixed catalyst bed. The compartment has screens on either end to prevent the loss of the catalyst. Other options for this system include a gas inlet port (not shown) in which a gaseous reactant may be fed into the tank for mixing before contact with the catalyst. This type of reactor configuration greatly facilitates the reaction between the multiple-phase reactants because the ultrasonic probe increases the interfacial area between the reactants before they come into contact with the catalyst bed. The author also states that it has great advantages over typical liquid–liquid–solid slurry reactors because the catalyst does not have to be separated from the reaction mixture at the end of each cycle, enabling an easier and more cost-

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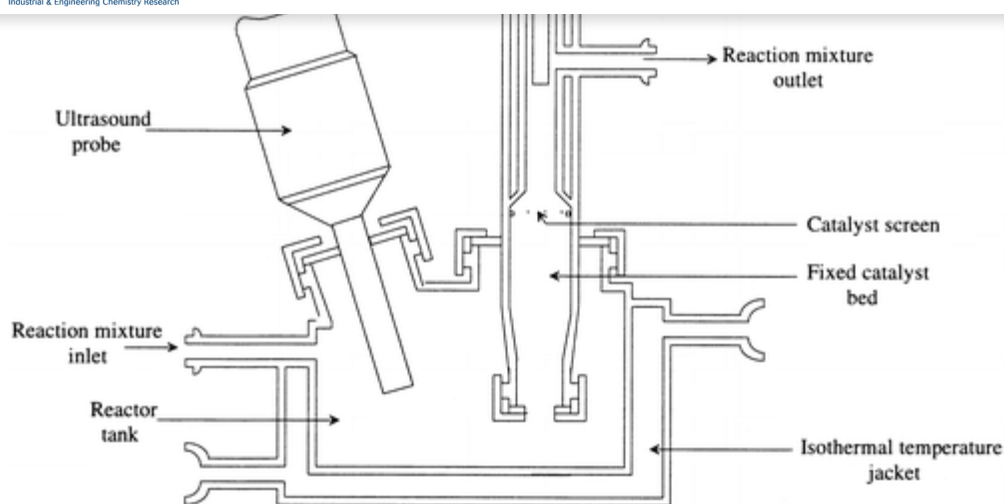
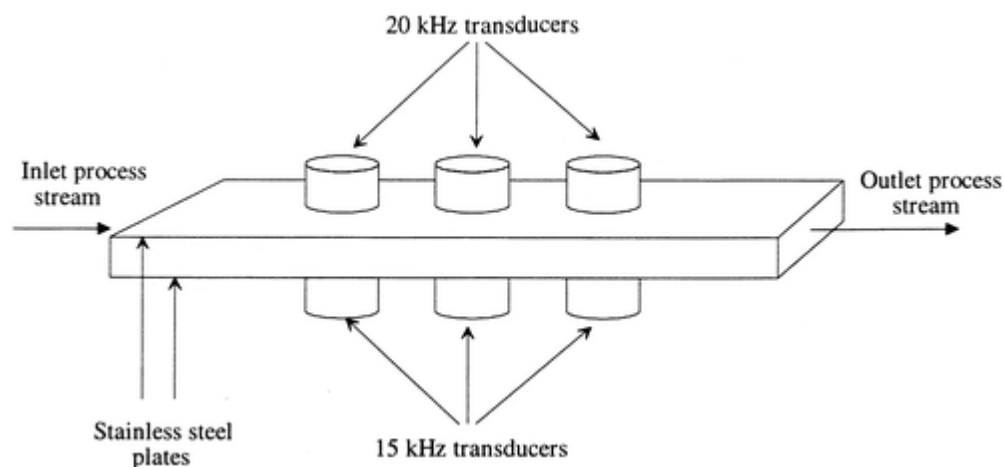


Figure 18 Ultrasonic reactor designed to emulsify immiscible liquid streams within the reaction chamber and then send the emulsified mixture through a fixed-bed triphase catalyst chamber (Ragaini, 1992).

13.3.2. Mounted Transducers. Lewis Nearfield Acoustic Processor (NAP). The Lewis NAP, also termed the Reverberatory Ultrasonic Mixing (RUM) System in some papers, is designed with transducers mounted, facing one another, on opposite sides of stainless steel plates (refer to Figure 19). The transducers operate at different frequencies (15 and 20 kHz) in opposing directions in order to generate an acoustic intensity within the process stream which is greater than the sum of the single plate intensities.



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designed (as shown in Figure 21). Each transducer should be mounted to focus the acoustic energy toward the center of the reactor.

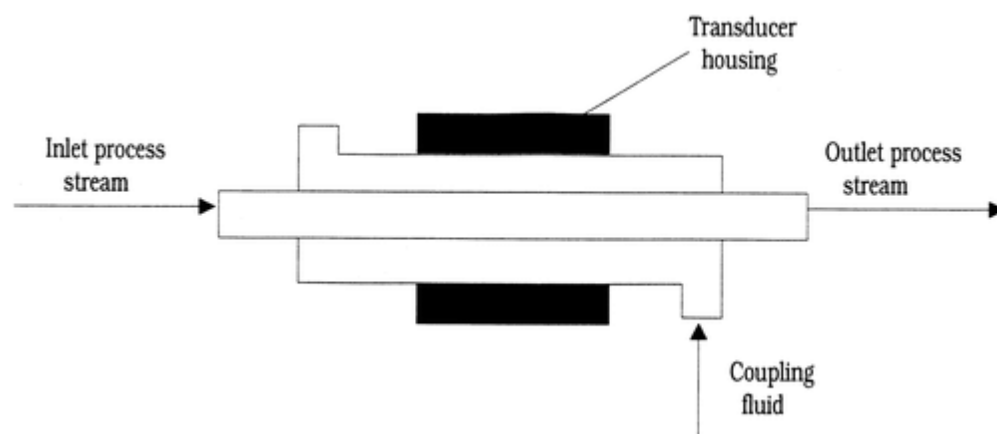


Figure 20 Cylindrical pipe reactor.

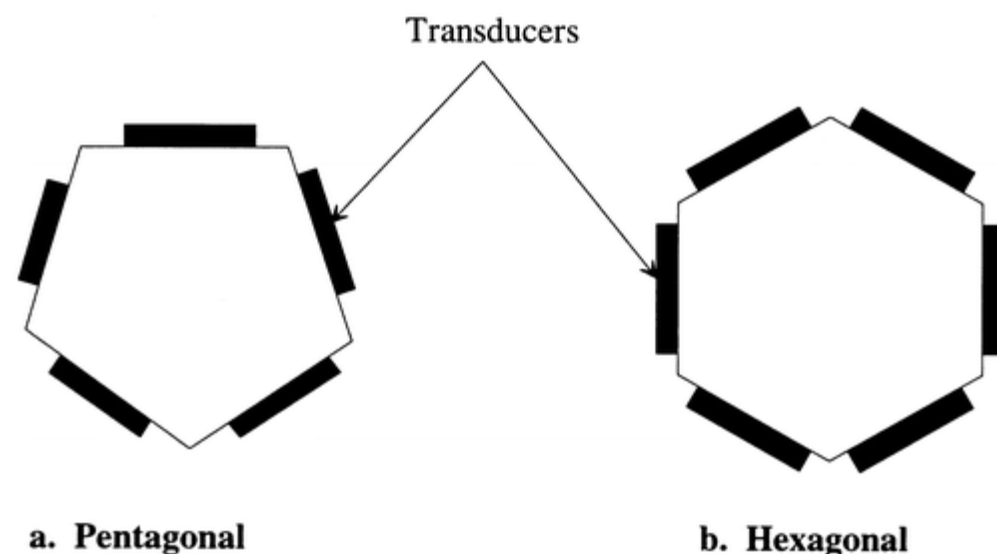


Figure 21 Cross sections of two types of tube reactors.

Martin Walter Push–Pull System. The Martin Walter push–pull system was developed in Straubenhardt, Germany, and is shown in Figure 22. The ends of the titanium bar are attached to opposing piezoelectric transducers. The length of the bar is equal to a multiple of the half-wavelengths of ultrasound produced. The two transducers are connected with electrical

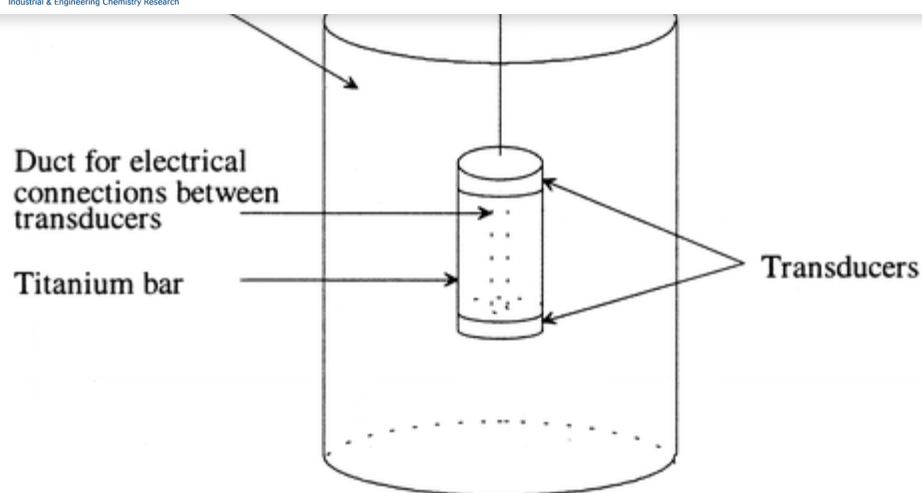
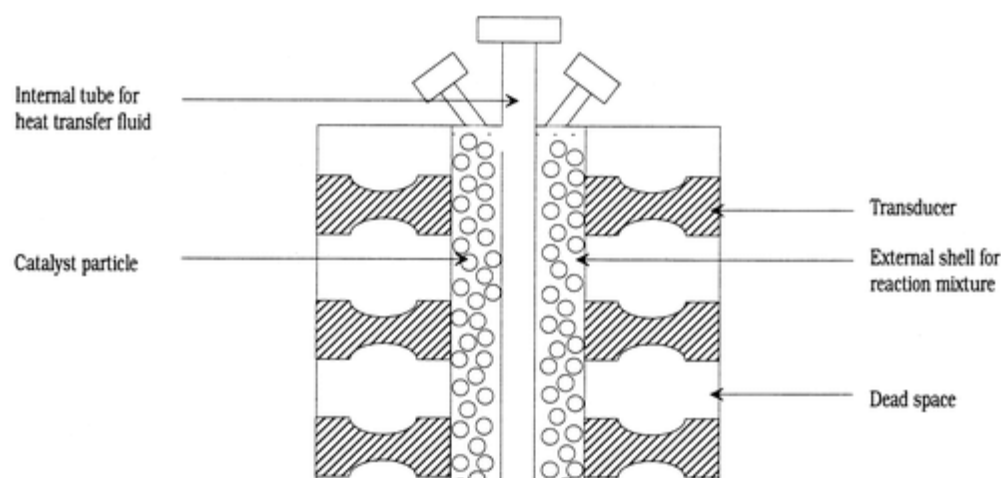


Figure 22 Martin Walter push-pull system.

Cylindrical Reactor with Core Cooling. The cylindrical reactor with core cooling, shown in Figure 23, was the second reactor configuration proposed by Ragaini (1992). The transducers are mounted over the length of the catalyst bed in order to sonicate the immiscible reactant streams while they are in contact with the catalyst. The advantages of the first reactor configuration (shown in Figure 18) over this one are as follows: it may be less expensive because it consists of one ultrasonic probe, not multiple transducers; the probe tip can be easily replaced if it becomes pitted, which is not the case when the transducers are part of the wall of the catalyst bed; and the triphase catalyst is not exposed to ultrasound, which may otherwise lead to polymer degradation and reduce the life and effectiveness of the triphase catalyst.

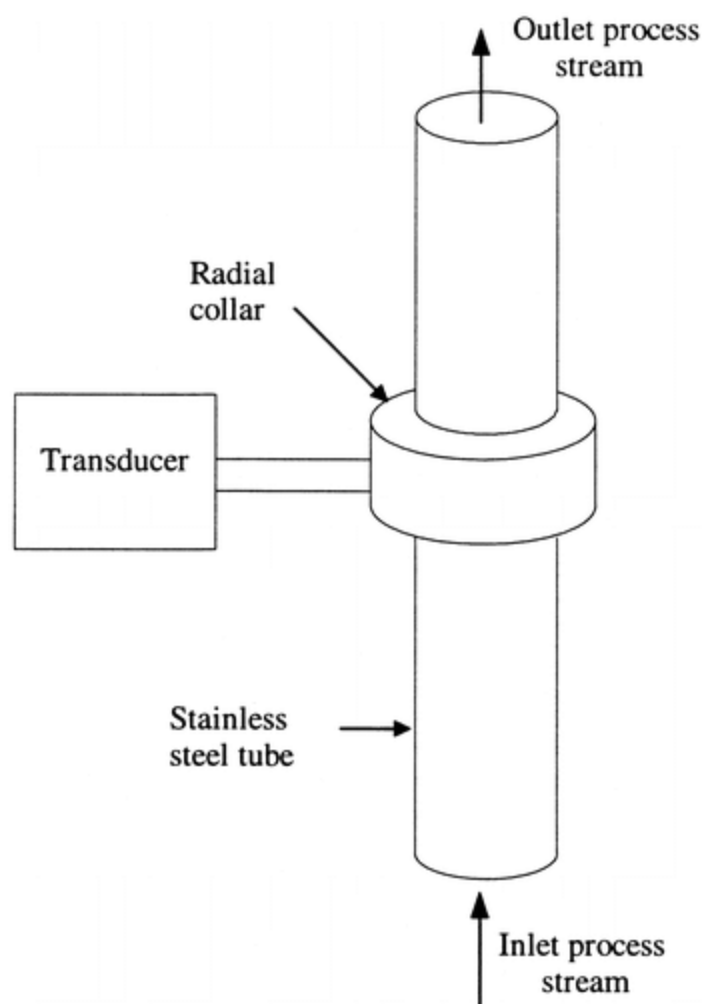


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Although this reactor was designed for use with triphase catalysts, any type of reaction system can be used. The internal tube provides core cooling to aid in maintaining isothermal conditions.

13.3.3. Miscellaneous. Sodeva Sonotube. This ultrasonic system was designed by Sodeva, a company based in France. The radial collar is attached to a transducer and acts as a cylindrical resonator (refer to Figure 24). Using a length of 1.2 m and an internal tube diameter of 42 mm, the unit can be operated at 2 kW with 80% efficiency (Mason, 1992b). The maximum ultrasonic power obtained is located at half-wavelength distances along the process pipe. The ends of the pipe are null points, making it possible to retrofit the device to existing pipe work.



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generated by the blade is dependent upon the flow rate of the fluid, which must be adjusted to a rate high enough to create cavitation in the fluid. Liquid whistles are very useful and inexpensive methods of producing fine emulsions in immiscible liquid streams and have been used successfully in food technology (Mason et al., 1996b). The apparatus may be installed on-line using an existing process stream and is capable of processing large volumes. Solid-liquid systems can also be used, but the solid particles may cause rapid erosion of the blade. In addition, the intensity of cavitation may not be high enough for some applications (i.e., chemical reactions requiring high intensities to obtain desired effects).

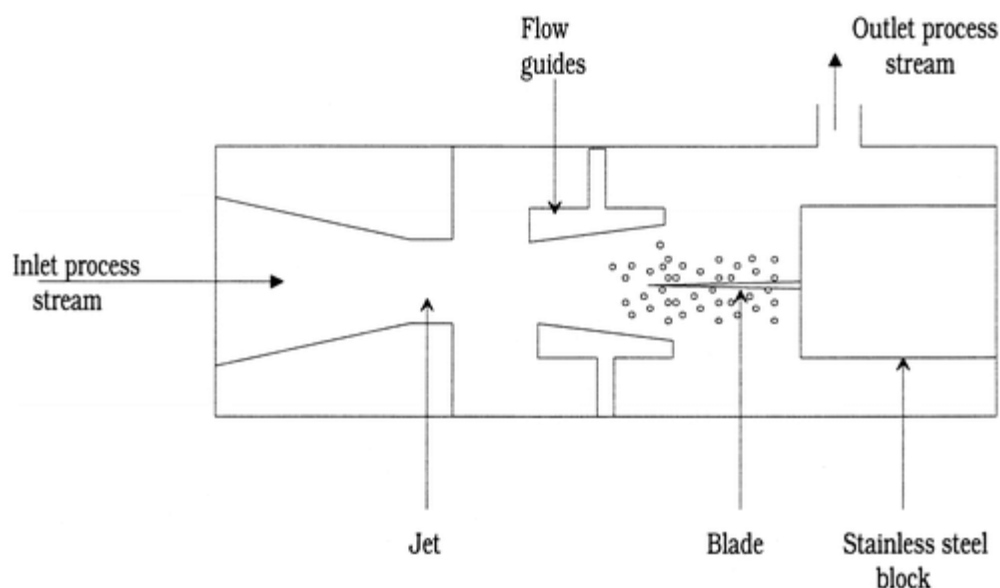
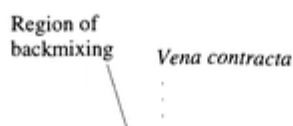


Figure 25 Liquid whistle.

14.2. Hydrodynamic Cavitation Reactor. Cavitation can also be generated in situ by forcing the fluid through an orifice, as shown in Figure 26, resulting in a pressure drop in the fluid. When the pressure falls below that of the vapor pressure of the fluid stream, cavitation sites are created. The magnitude of the pressure drop is dependent upon the flow rate of the fluid and the size of the orifice. When gases are dissolved in the fluid, cavitation may occur at pressures higher than the vapor pressure of the fluid (Moholkar and Pandit, 1997; Pandit and Moholkar, 1996).



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parameter termed the cavitation number, C_N , which is defined as

$$C_N = \frac{P_2 - P_v}{\frac{\rho v^2}{2}} \quad (37)$$

where P_2 is the pressure in the fluid after it passes through the orifice, P_v and ρ are the vapor pressure and density of the fluid, respectively, and v is the velocity of the fluid at the orifice. The parameter C_N is independent of the liquid velocity in the pipe but increases linearly with the ratio of orifice diameter to pipe diameter (Yan et al., 1988). It has been shown that cavitation inception occurs when C_N is in the range of 1.5–2.5.

15. Scale-Up Considerations

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When considering whether to scale-up a reaction which is accelerated by ultrasound, there are several factors to consider. First, it is important to know what role ultrasound is playing in the enhancement. Are the effects truly chemical (i.e., is the enhancement due to the formation of free radicals) or are they primarily physical? If they are physical, what effects are most important to the enhancement of the reaction? If particle degradation is the only critical role ultrasound is playing, a sonochemical reactor may not be necessary. Instead, the solids can be sonicated before they are placed within a conventional reactor. However, if the other physical effects of ultrasound are important, such as the enhanced rate of mass transfer and/or surface renewal, then sonication will be required over the course of the reaction.

In some cases, the use of ultrasound may generate a reaction intermediate which catalyzes the reaction, as in the case of the Diels–Alder cycloaddition between cyclopentadiene and methyl vinyl ketone (Reisse et al., 1996). However, if this is the only role ultrasound is playing, it may be more cost-effective to physically add the necessary intermediate rather than using ultrasound to generate it.

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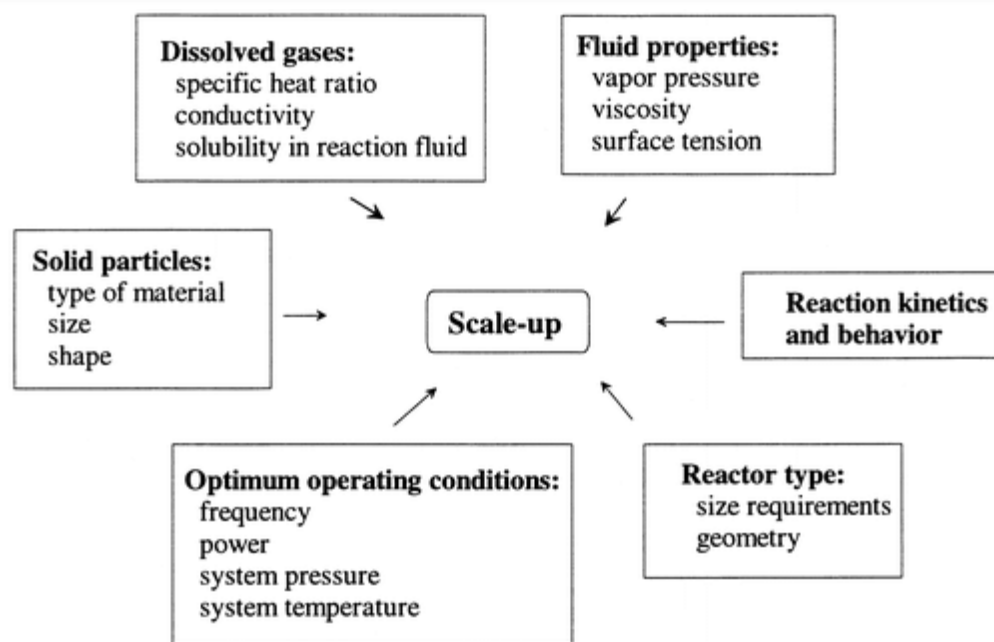


Figure 27 Scale-up considerations.

In addition to knowing the characteristics of the reaction mixture and the kinetics of the reaction, one must also have knowledge of the optimum system and ultrasonic conditions, such as the ambient reaction temperature, pressure, frequency, dissipated power, ultrasonic field, and their interactions. Addition of equipment within a reactor (i.e., baffles, stirrers, and cooling coils) affects the distribution of ultrasonic energy because of wave reflection. All scale-up considerations discussed thus far are summarized in Figure 27.

16. Conclusions

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The advances in the field of ultrasound in the last 20 years have been plentiful, but there is still a lot of new frontier to be covered. Researchers have found that ultrasound chemically enhances reactions which depend on a SET process as a key step. Reaction systems which follow an ionic mechanism are enhanced by the mechanical effects of ultrasound. These enhancements are a result of increases in the intrinsic mass-transfer coefficient, increases in surface area resulting from particle degradation, and, in some cases, increases in the driving force for dissolution. In some reaction systems, ultrasound changes the reaction pathway from ionic to one which

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energy and frequency factor of various reaction systems. However, the actual mechanisms behind these enhancements have not been discerned. In addition, as is evident when reading sections 13–15, the amount of available engineering data in the areas of ultrasonic reactor design and scale-up are lacking. It will take the combined work of scientists from all fields to resolve the role of ultrasound in reacting systems and to make it a viable rate enhancement technique for commercial industrial processes. Nomenclature

a = interfacial area of solid particles

A = Arrhenius parameter

A_p = area of the probe tip

A_{ws} = area of the wetted surface

C = velocity of sound in the liquid medium

C_A^* = saturation concentration

C_A = concentration of solid reactant in solution

C_{A0} = initial solid reactant concentration

C_B = concentration of the liquid-phase reactant

C_N = dimensionless cavitation number

C_n = carbon number

$C_{p,solvent}$ = heat capacity of the solvent

$C_{p,vessel}$ = heat capacity of the reactor vessel

D = diffusivity of reactant through solvent

d_e = diameter of the electrode

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E_a = activation energy

F_c = shape factor

F_t = cycle time at which bubble phase reaction occurs

F_v = volume of the reaction mixture occupied by bubbles

h = height of the liquid above the transducer

I = acoustic intensity

I_{diss} = intensity of ultrasound at the emitting surface

I_{max} = maximum acoustic intensity

k = wavenumber ($=2\pi/\lambda$)

k_{BUB} = rate constant associated with bubble collapse

k_d = mass-transfer coefficient

k_{et} = rate constant of reaction occurring within the bubble

k_l = intrinsic solid-liquid film transfer coefficient

k_{non} = rate constant in the absence of ultrasound

$k_{\text{r.f.}}$ = rate constant of radical formation

k_s = rate constant of surface reaction

k_{sl} = solid-liquid mass-transfer coefficient for dissolution

k_{US} = rate constant in the presence of ultrasound

m_{solvent} = mass of the solvent

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P_A = pressure amplitude delivered by the transducer

P_{diss} = actual power dissipated in the reaction mixture

$p_g(R)$ = pressure of permanent gas within the bubble

P_I = pressure intensity

$p_L(R)$ = liquid pressure just outside the bubble wall

P_m = liquid pressure at transient collapse

P_{max} = maximum pressure developed at the moment of bubble collapse

P_0 = hydrostatic pressure

P_s = saturation vapor pressure of the liquid

$p_T(R)$ = pressure of total mass content within the bubble

P_v = vapor pressure of the liquid

$p_v(R)$ = pressure of vapor within the bubble

p_x = pressure amplitude and distance x from the transducer

R = radius of bubble

$\dot{R}$ = first derivative of the bubble radius with respect to time

$\ddot{R}$ = second derivative of the bubble radius with respect to time

R_{A0} = radius of the solid reactant

R_g = universal gas constant

R_{max} = maximum bubble radius

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t = time

T = temperature

T_0 = ambient reaction temperature

$T_{\max}$ = maximum temperature developed at the moment of bubble collapse

T_v = temperature of the inner vessel wall

v = wave velocity

V = volume of the liquid medium

v_{mj} = microjet velocity

x = distance from the transducer

x_A = conversion of solid reactant

X_i = volume fraction

x_w = thickness of the inner wall of the reactor vessel

Greek Letters

α = attenuation coefficient of the medium

δ = diffusion layer thickness

λ = wavelength

γ = polytropic ratio of specific heats of the bubble mixture

μ = viscosity of the bulk liquid medium

ν = frequency ($=2\pi/\omega$)

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σ_r = reflection coefficient at the liquid–solid interface

τ_m = time for complete transient collapse

ω = angular frequency

ω_r = resonance frequency

ω'_r = resonance eigenfrequency

u = velocity of the fluid at the orifice

Abbreviations and Acronyms

freq. = ultrasonic frequency

PTC = phase-transfer catalyst

rxn = reaction

SET = single electron transfer

Sh = dimensionless Sherwood number

TBAB = tetrabutylammonium bromide

w/v = mass of solid per unit volume of organic phase (g/mL)

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