

## EUROPEAN ROADMAP OF PROCESS INTENSIFICATION

### - TECHNOLOGY REPORT -

TECHNOLOGY: Sonochemical reactors (ultrasound and low-frequency sonics)

TECHNOLOGY CODE: 3.2.5

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# 1. Technology

## 1.1 Description of technology / working principle

*(Feel free to modify/extend the short technology description below)*

To bring about any permanent (lasting) change in the physicochemical properties and/or form of the material requires a certain amount of energy transfer (addition or removal) over and above a certain minimum level (activation energy). This restricts the form and type of energy which can be used to bring about this change.

Use of diffused sound energy and/or the use of the pressure and kinetic energy of the fluid by concentrating it and delivering it at the actual site of the physicochemical transformation, is one such emerging technology. This concentration of the energy is achieved through phenomena of cavitation, which when induced using the high frequency sound (with frequency of  $\geq 20$  kHz), is described as acoustic cavitation and reactors used for carrying out the given operation have been described as sonochemical reactors. Cavitation can be defined as the formation, growth and subsequent collapse of vapor and/or gas-vapor filled bubbles in the liquid. The energy which the cavity gains during its growth over terms of microseconds is released during its subsequent implosive collapse in a time-duration of few microseconds. This exceptionally fast energy dissipation results into local high temperatures ( $\sim 14000$  K) and high pressures ( $\sim 10000$  atm) as the heat and mass transport processes occur much slowly (order of milliseconds).

The use of these extreme local conditions to bring about the desired transformation is known as cavitationaly induced transformations. These extreme temperatures and pressure also generate certain high reactive radical species, which brings about the physicochemical change. The changes in the physical conditions such as dispersion, emulsification, atomization, grinding are brought about as a result of the extreme pressures and shock-waves generated during the collapse of the cavity.

The local conditions generated within and surrounding the cavity are decided by the speed of the cavity collapse; faster the collapse, more extreme are the conditions. This is thus decided by the dynamics of the cavity (radius-time history) and its relationship with the required energy levels to bring about the desired transformations.

## 1.2 Types and “versions”

*(Describe the most important forms/versions of technology under consideration, including their characteristic features, differences and similarities)*

Though cavitation as a phenomenon can be generated using a variety of means such as sound, fluid energy, laser and high energy particle beams, the first two are the only two means of generation which are industrially relevant.

The use of high frequency sound where the operational frequency is beyond the audible range i.e. starting from 16 kHz reaching up to the diagnostic range of ultrasound of about 2 MHz.

The three basic variables which are responsible in changing the dynamics of the cavity and thus the sonochemical transformational conditions are as follows:

- a. Frequency of irradiation
- b. Amplitude and
- c. Geometric configuration of the reactor.

The vibrational frequency in the ultrasound range is generated using either by piezoelectric transducers vibrating at their resonant frequency or using magneto-restrictive transducers. The sound wave consists of compression and rarefaction cycles and the operating frequency decides the duration of this cycle. Higher frequency reduces these cycle duration and hence does not allow the cavity to grow to a larger size; still the compression is faster and hence the collapse occurs more violently. Similarly the maximum amplitude of the vibration is decided by the tolerable mechanical integrity of the vibrating surface. Higher amplitude allows the cavity to grow to a higher size (lower negative

pressures during the rarefaction cycle) and hence the subsequent collapse is with higher compression ratio and hence more severe.

The geometric configuration parameters include number and location of the transducers delivering the required power and the resultant sound pressure fields generated in the entire liquid continuum affecting the dynamics of the individual cavity differently, depending on its relative location. This finally decides the overall performance of the sonochemical reactors. The different reactor configurations have now been discussed with some comments on the scale up aspects later.

Sonochemical reactors differ mainly in terms of the number and placement of transducers (geometrical parameters) and frequency and intensity of irradiation (operating parameters). The various lab scale/pilot plant scale reactors which have been used for the studies for different applications of cavitation process have now been discussed with relative merits and demerits.

Ultrasonic horns are the most commonly used reactor designs amongst the sonochemical reactions. These are typically immersion type of transducers and very high intensities (pressures of the order of few thousands atmosphere) are observed very near to the horn. The intensity decreases exponentially as one moves away from horn and vanishes at a distance of as low as 2 to 5 cm depending on the maximum power input to the equipment and also on the operating frequency (Chivate and Pandit, 1995). Ultrasonic horn systems can work effectively if operated in geometry where most of the working liquid is constrained within the longitudinal high-intensity region or where the liquid is stirred vigorously in addition to the ultrasonic irradiation. Horst et al. (1996) have reported a novel modification in terms of using high intensity ultrasound from a concentrator horn. It has been shown that the concept of a conical funnel fits the demands for nearly perfect radiation effectiveness and a good reaction management. The design used by Dahlem et al. (1998,1999) also needs a special mention here. Telsonic horn, which has radial vibrations as against conventional longitudinal vibrations for the immersion system, gives dual advantages of higher irradiating surface (lower intensity of irradiation resulting in better yields) coupled with good distribution of the energy in the radial direction. Moreover, even if the horn is radially vibrating, local measurements just below the horn (Dahlem et al., 1999) have also indicated high cavitation activity, which will be again more beneficial in enhancing the global cavitation effects. The scale up prospects of horn type systems, are very poor as it cannot effectively transmit the acoustic energy into large process fluid volume. Also, they suffer from erosion and particle shedding at the delivery tip surface due to high surface intensity ( $W/m^2$ ), they may also be subjected to cavitation blocking (acoustic decoupling), and the large transducer displacement (amplitudes) increases stress on the material of construction, resulting in the possibility of stress induced failure. Thus, ultrasonic horn type systems are generally recommended for laboratory scale investigations for obtaining the scale up and other design parameters.

Another configuration which has been commonly used is ultrasonic bath, where the bottom of the reactor is irradiated with a single or multiple transducers. In this case, the active zone is restricted to a vertical plane just above the transducers with maximum intensity at the center of transducer. Thus, the area of irradiating surface should be increased (maximum possible) so as to get better distribution/dissipation of energy in the reactor and maintaining it just above the required cavitation threshold. This has a twin advantage in terms of the decreased ultrasonic intensity (defined as power dissipation per unit area of the irradiating surface), which will increase the magnitude of the pressure pulse generated at the end of the cavitation events (Gogate and Pandit, 2000). Dahlem et al. (1998,1999) have shown better local ultrasonic intensities and iodine liberation rates for the radially vibrating horn (1000 W dissipated through an area of 365  $cm^2$ ) as compared to conventional horn (longitudinal vibrations, 300 W dissipated through an area of 0.8  $cm^2$ ). Ultrasonic bath systems are much more effective as compared to the ultrasonic horn systems in terms of the uniformity of energy distribution and large scale applicability.

To increase the active zones existing in the reactor, one can easily modify the position of the transducers (if multiple transducers have been used which is likely to be the

case at large scale operation due to the fact that it is quite difficult to successfully operate single transducer with very high power and frequency due to limitations over the material of construction for the transducers) so that the wave patterns generated by the individual transducers will overlap, also resulting into uniform and increased cavitation activity. In the case of multiple transducer reactors (Figure 1), arrangements such as triangular pitch in the case of ultrasonic bath (Saudagar and Samant, 1995; Dahlem et al., 1999), tubular reactors with two ends either irradiated with transducers or one end with transducer and other with a reflector (Gonze et al., 1998), parallel plate reactors with each plate irradiated with either same or different frequencies (Thoma et al., 1997; Gogate et al., 2001) and transducers each on sides of hexagon (Romdhane et al., 1995; Gogate et al., 2003) can be constructed. It is of utmost importance to have uniform distribution of the ultrasonic activity in order to get increased cavitation effects and using multiple transducers is the only possible way of an effective scale up for large scale operation.

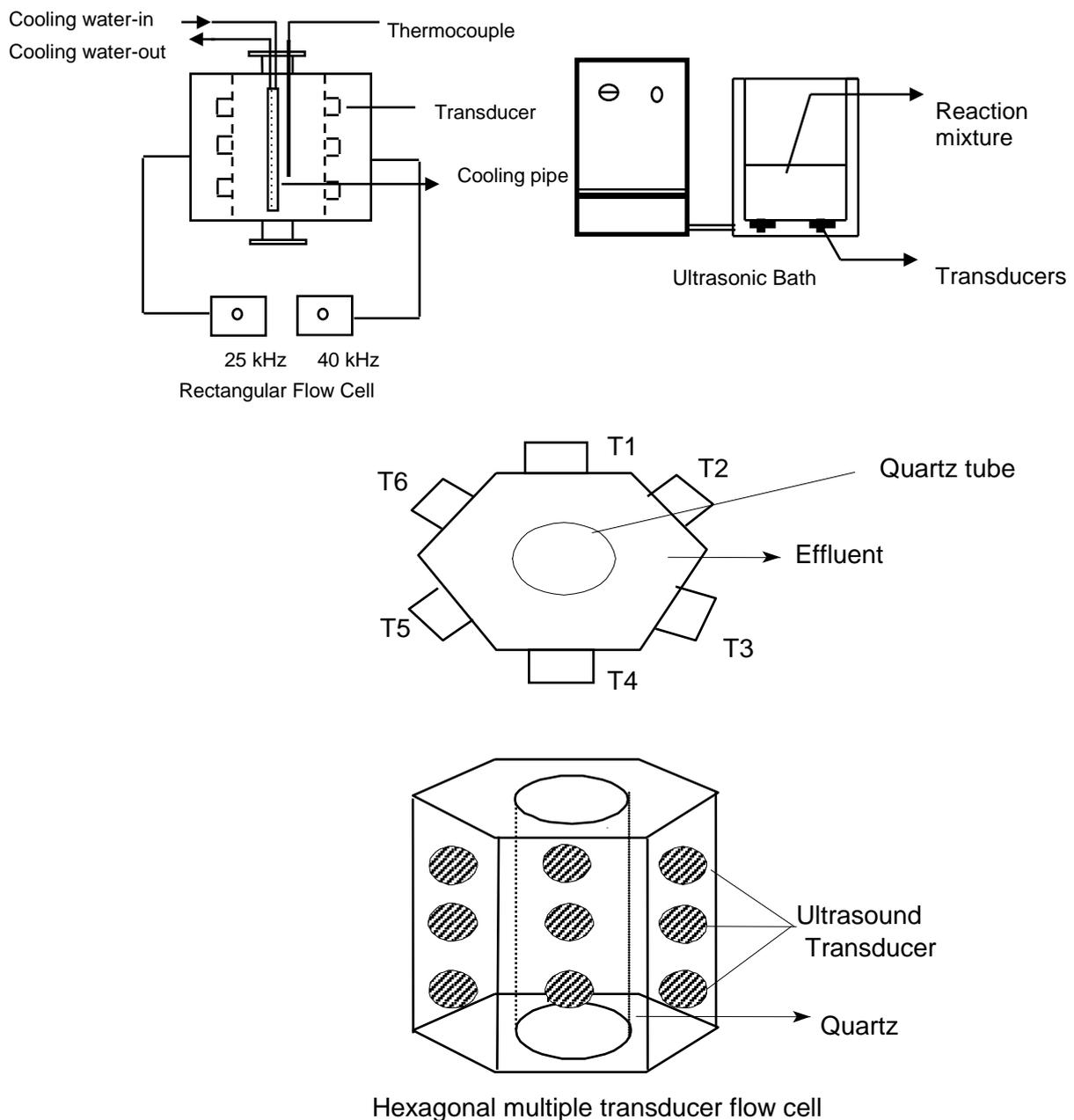


Figure 1: Multiple transducer based sonochemical reactors

Direct bonding of the transducer to the surface of the vessel has made it possible to increase the number of transducers attached to the vessel depending on the required power input and size of the vessel or arrange these vessels or modules in sequence to generate a full scale operating systems. Improvements in the bonding method, and a move to use multiple transducers with lower individual outputs, have enabled the design of systems with large numbers of transducers to give an acoustic pattern that is uniform and noncoherent above the cavitation threshold throughout the reactor working volume. The use of low-output transducers gives the additional advantage of avoiding the phenomenon of cavitation blocking (acoustic decoupling), which arises where power densities close to the delivery point are very high. In addition, these multi-transducer units very effectively concentrate ultrasonic intensity towards the central axis of the cylinder and away from the vessel walls, thus reducing problems of wall erosion and particle shedding. The vessel can be operated in batch mode or, for larger-scale work, in continuous mode whereby units can be combined in a modular fashion for “scale-out” and increased residence time. In summary, a plurality of low electrical and acoustic power ( $1-3\text{W}/\text{cm}^2$ ) transducers produces 25-150 W/L, but ideally 40-80 W/L. The power can be applied continuously or in a pulsed mode.

### 1.3 Potency for Process Intensification: possible benefits

*(In Table 1 describe the most important documented and expected benefits offered by the technology under consideration, focusing primarily on energy; CO<sub>2</sub> emission and costs, providing quantitative data, wherever possible. Add other benefits, if needed).*

Table 1: Documented and expected benefits resulting from technology application

Benefit	Magnitude	Remarks
Energy Savings	10 % to order of magnitude	When energy delivery is required on micro-scale and only locally, the specified energy savings are possible. As a general alternative for chemical or physical transformations, sonochemical reactors are not energy efficient on a global basis. Overall energy efficiencies are only in the range of 2 to 40%.
Less CO <sub>2</sub> emission	No data	Since it is proportional to the use of energy (thermal or electric), similar remarks as given in the criteria of energy savings are applicable. If electricity is generated using non-conventional resources such as wind, solar or nuclear, the CO <sub>2</sub> emissions will be reduced as separate heating energy is not required. The sonochemical transformations are known to show maximum intensification at room temperatures.
Cost Savings	No direct industrial data as a result of the observed competitive edge of the adaptation of technology. In some specific cases 80 to 90% reduction in the processing costs have been observed.	The cost savings result out of the following advantages: <ol style="list-style-type: none"> <li>1. Faster processes leading to several fold reduction in the batch times</li> <li>2. Smaller equipment volume</li> <li>3. Higher product yield and selectivity</li> <li>4. Self cleaning system</li> <li>5. Reduction in the catalyst deactivation rates</li> </ol>
Increased yield/selectivity	10 to 400%	As a result of the extremely short time scales (micro to nano) of severe conditions, side reactions are restricted.
Increased Safety	Yes particularly due to the requirement of less severe overall operating conditions. Also in high energy material crystallization	Smaller equipment size, safer operating conditions (no heating) and lower inventory of toxic materials

Defouling	No data available	Self cleaning system due to cavitation surface cleaning
Reaction medium (solvent)/ catalyst savings	More than 100%	No catalyst requirement in some cases, Prevention of catalyst deactivation due to surface cleaning, increased reactant transport due to shock waves and liquid jets reducing the diffusional resistances
Better Quality product	No data available on an industrial scale of operation due to competitive edge and secrecy issues	Due to increased selectivity and removal of trace impurities, tremendous benefits to the chemical processing industries especially in the pharmaceuticals sector

## 1.4 Stage of development

Even though there is a good theoretical understanding of the parameters, responsible for controlling the dynamics of the cavity and hence its resultant effects, technologically it has not been achieved.

The scale up of sonochemical reactors is carried out again on the basis of gross parameters such as power per unit volume, same operating frequency etc. which are inadequate and do not result into the same level of intensification observed on bench scale.

The scale up criteria is still not well established though general guidelines are available and hence it is still necessary to optimize the parameters on a large scale experimentally, which is very expensive.

Several vendors and reactor configurations are available, though there are no "universal-multipurpose" sonochemical reactor configurations which can be recommended. The reactor configuration i.e. frequency, power (amplitude) intensity in  $W/m^2$  and the geometry are extremely chemical system specific.

If the job of sonochemical reactor is the destruction of the chemicals and biological products i.e. their oxidation, pyrolysis, decolorization, microbial disinfection etc. or physical processes such as emulsification, crystallization etc., then the gross scale up criteria have been found to be adequate; however for organic synthesis, considerable more work, which is system specific is to be carried out or required for this technology to get accepted in specialty chemical industry.

## 2. Applications

### 2.1 Existing technology (currently used)

*(Describe technology (-ies) that are conventionally used to perform the same or similar operations as the PI-technology under consideration)*

Conventionally multiphase reactors mainly mechanically agitated contactors are used to carry out chemical and physical processing applications such as chemical synthesis, crystallization, extraction etc. where it is expected that the sonochemical reactors can be used with great success. Heating/cooling jackets and/or coils are provided depending on the process conditions and various heat transfer media such as steam or mineral oils are applied. Continuous processes are usually carried out in stirred-tank reactors in-series or in tubular flow reactors.

### 2.2 Known commercial applications

*(Is the technology broadly applied on commercial scale? In which process industry sectors is the technology most often applied: large volume chemicals – specialty chemicals & pharma – consumer products – ingredients based on agro feedstocks? What is the estimated number of existing applications? In Table 2 provide the most prominent examples of realized applications and provide their short characteristics)*

Use of sonochemical reactors as a process intensification technology has been languishing to the confines of laboratory scale application and only recently pilot or industrial scale applications are being attempted at. Also due to secrecy issues, actual plant data is not readily available. Table 2 presents some of the gross data with which either our group was associated in the development process or is available in the literature.

Table 2. Industrial-scale applications of the Technology (existing and under realization)

Sector	Company - Process/Product name/type	Short characteristic of application	Product on capacity/ Plant size	Year of application	Reported effects
Biotechnology	PLAFAR, Romania	Extraction of different herbs using ethyl alcohol	1000 L operating scale	1995	Significant intensification (6 h processing time as against few days for conventional process) in the extraction processes
Pharma	Dr. Reddy's Laboratory, India	Crystallization and Synthesis	< 100 tons/year	2004	Better crystal size distribution and intensification of synthesis process
Pharma	CIPLA, India	Crystallization and Synthesis	< 100 tons/year	2005	Better crystal size distribution and intensification of synthesis process
Pharma	Accentus, UK (supplier)	Intensification and improvement in the crystallization operations	Many case studies available	Many case studies available	Better crystal size distribution and also lower processing times. Combination of ultrasonic flow cells can be used based on the scale of operation

### 2.3 Known demonstration projects

*(Are there any demonstration projects known related to the technology under consideration? In which process industry sectors are those projects carried out: large volume chemicals – specialty chemicals & pharma – consumer products – ingredients based on agro feedstocks? In Table 3 provide the short characteristics of those projects.)*

Table 3. Demonstration projects related to the technology (existing and under realization)

Sector	Who is carrying out the project	Short characteristic of application investigated, including product name/type	Aimed year of application	Reported effects
Specialty chemicals	Technische Universität Clausthal, Germany	Synthesis of 4 tons per annum of Grignard solution	1996	Up to 4 times enhancement in the rate constant for synthesis of Grignard solution
Biotechnology	UICT, Mumbai	Recovery of lactose from waste of dairy industries	2006	Lactose can be effectively recovered from the wastewater of the processing industries and with order of magnitude increase in the rates of crystallization

Wastewater treatment/ New energy sources	TUHH, Germany	Use of ultrasonic reactors for intensification of biogas generation	2006	Significantly accelerated biosolids degradation with less digested sludge being produced and increased biogas production being attained. A full-scale ultrasound reactor system was developed for continuous operation under real life conditions on sewage treatment plants (STP).
Specialty chemicals	UICT, Mumbai	Improvement in crystallization of High energy materials (e.g. CL20)	2007	Better crystal size distribution as well as lower mean size of the high energy material (CL20) can be achieved in lower processing times along with an increase in the final mass yield, compared to the conventional antisolvent based precipitation process. Inherently safe process even to handle high energy materials
Specialty chemicals	UICT, Mumbai	Size reduction of rubber latex particles for preparation of nano-suspensions	2007	High intensity cavitation is indeed able to produce nano-suspensions from rubber latex solutions in the range of 6 to 10 % solid concentration

## 2.4 Potential applications discussed in literature

*(Provide a short review, including, wherever possible, the types/examples of products that can be manufactured with this technology)*

The main application of sonochemical reactors is in intensification of chemical synthesis processes. The different ways in which cavitation can be used in the chemical processing applications are:

- a. Reaction time reduction
- b. Increase in the reaction yield
- c. Use of less forcing conditions (temperature and pressure) as compared to the conventional routes
- d. Reduction in the induction period of the desired reaction
- e. Possible switching of the reaction pathways resulting in increased selectivity
- f. Increasing the effectiveness of the catalyst used in the reaction
- g. Initiation of the chemical reaction due to generation of highly reactive free radicals

In general, sonochemical reactors have been reported to affect different types of chemical which include Oxidation, Hydrolysis, Nucleophilic reactions, Radical initiated reactions, Polymerization/Depolymerization reactions, Organometallic synthesis, Thermolytic decomposition, Substitution/Addition/Condensation reactions and Phase transfer catalyst based reactions. Sonochemical reactors also can intensify processes for synthesis of Nano particles/Nano spheres, porous and non porous particles.

Apart from this important application in chemical processing, sonochemical reactors can be used effectively for the destruction of the contaminants in water because of the localized high concentrations of the oxidizing species such as hydroxyl radicals and hydrogen peroxide, higher magnitudes of localized temperatures and pressures and the

formation of the transient supercritical water. The variety of chemicals that have been degraded using sonochemical reactors though in different equipments and on a wide range of operating scales are p-Nitrophenol, Rhodamine B, 1,1,1 trichloroethane, parathion, pentachlorophenate, phenol, CFC 11 and CFC 113, o-dichlorobenzene and dichloromethane, potassium iodide, sodium cyanide, carbon tetrachloride among many others.

Sonochemical reactors can also be used effectively for the rupture of cells with energy requirements as less as just 5 to 10% of the total energy consumed by the conventional methods. The intensity of the cavitation phenomena can also be controlled so as to control the mechanism of the rupture of the cells to selectively release the intracellular enzymes or enzymes present in the cell wall. Lower intensity application of cavitation helps in retaining the activity of the leached out enzymes as well as it also reduces the cost of operation.

Apart from these broad-spectrum applications, sonochemical reactors have also been found to be beneficial in Crystallization operations (for controlling the crystal size distribution and also reduction in total operational time), atomization (for controlling the droplet size distribution at minimum energy requirements), Azeotropic distillation (for breakage of the azeotrope), intensification of solid-liquid extraction or photochemical reactions as well as in petroleum industry for refining fossil fuels, determination of composition of coal extracts, extraction of coal tars etc. and textile industry for enhancing the efficacy of dyeing technique.

### 3. What are the development and application issues?

#### 3.1 Technology development issues

*(In Table 4 list and characterize the essential development issues, both technical and non-technical, of the technology under consideration. Pay also attention to “boundary” issues, such as instrumentation and control equipment, models, etc.) Also, provide your opinion on how and by whom these issues should be addressed)*

Table 4. Technology development issues

Issue	Description	How and by whom should be addressed?
Engineering Design and Scale-up issues	<p>Commercial systems with size ranging from few liters to few thousands of liters are available. The delivered power range of 50 W to 8000 W either through single or multiple transducers are also available readily. However, the scale-up criteria used in all this development are only gross i.e. frequency, power per unit volume <math>W/m^3</math> and intensity of irradiation <math>W/m^2</math> and are not physicochemical transformation specific. The relationship between the desired sound pressure field and its effect on the cavity dynamics and its specific physicochemical effect is still evolving. It is still very system specific and only general scale up guidelines, are available, which are only qualitative in nature.</p> <p>There still exists a problem of material erosion contaminating the reactor contents especially at higher levels of power delivery. The development of new hard materials and/or focusing of the sonic energy away from the vibrating surface are some of the issues, which need to be sorted out.</p>	R&D projects carried out at the Universities in collaboration with equipment manufactures and producers of large size sonochemical reactors.

Modeling and Scale-up Methodologies	<p>The modeling of sound pressure fields in multi-transducer, multi-frequency systems having complex geometrical configurations i.e. cylinder, hexagon, pentagon etc. is the need of the hour.</p> <p>The second stage of modeling is to understand the effect of such a pressure field on the cavity dynamics and predict the local conditions generated in terms of temperatures and pressures.</p> <p>The third stage of modeling efforts are chemistry oriented where the generation of the likely chemical species depending on the local conditions predicted in stage 2 and relating this rate to the desired physicochemical transformation.</p> <p>Though, researchers have individually worked on each of the above aspects, a comprehensive modeling strategy is just evolving, which also needs to be validated using experiments.</p>	<p>The solution to this issue is only through the collaborative efforts of physicists, chemists, chemical engineers and the equipment manufactures. This can be done through “mission oriented projects” involving all the above mentioned departments of the University simultaneously</p>
Control Systems	<p>The control of the sonochemical transformation requires the manipulation of the sound pressure field by controlling the energy delivered by the individual transducer to the system in the case of multi-transducer system, which is currently being done based on the feedback pressure signal.</p> <p>In the case of single transducer system, a considerable spatial non-uniformity exists and hence the flow of the reactants through this spatially uneven pressure field needs to be controlled and monitored.</p> <p>A control strategy involving mean and fluctuating pressure measurements (use of hydrophones) and flow measuring devices (LDA, HWA etc.) needs to be evolved.</p>	<p>R&amp;D projects carried at the universities, in collaboration with equipment manufacturers; However, the technology is not matured enough to devote resources for this activity at this stage.</p>

### 3.2 Challenges in developing processes based on the technology

*(In Table 5 list and characterize the essential challenges, both technical and non-technical, in developing commercial processes based on the technology under consideration. Also, provide your opinion on how and by whom these challenges should be addressed)*

Table 5. Challenges in developing processes based on the technology

Challenge	Description	How and by whom should the challenge be addressed?
Achieving spatial uniformity of cavitation activity in the entire volume of the reactor	<p>The relationship between the locations of multiple transducers, their interaction with each other needs to be established through mathematical models and validated with experiments. There exists a limitation in terms of the maximum power which can be delivered by a single transducer and hence multiple transducer systems are essential for large scale systems</p>	<p>R&amp;D projects carried at the universities, in collaboration with equipment manufacturers</p>

Development of new materials for the vibrating surfaces and focusing the sound energy away from the surface	The techniques developed in the area of medical application such as Lithotripsy and HIFU (high frequency focused ultrasound) need to be adapted here in sonochemical reactors to protect the vibrating surface from cavitation damage. New hard materials with the desirable ductility need to be experimented with.	R&D projects carried at the universities, in collaboration with equipment manufacturers
Safety issues	There still is a problem of having sonochemical reactors working in a flame-proof environment needed especially in the organic synthesis area involving flammable solvents. Though attempts have been made using inert gases covering the electrical connections to the transducers, these are not fool-proof. Safe electrical connections need to be developed for large scale equipments. Some sonochemical equipments also generate a lot of noise and hence may require sound-proof housing, which in a continuous plant is tedious to achieve.	R&D projects carried at the universities, in collaboration with equipment manufacturers
Control	Remote control instrumentation system for large scale plant in assessing and controlling the spatial uniformity of cavitation activity are required. Control logic, on-line control strategies are to be evolved.	R&D projects carried at the universities, in collaboration with equipment manufacturers

## 4. Where can information be found?

### 4.1 Key publications

*(Provide the list of key publications in Table 6)*

Table 6. Key publications on the technology

Publication	Publication type (research paper/review/book/report)	Remarks
Weissler A, Sonochemistry: The production of chemical changes with sound waves. J Acoust Soc Am 25: 651-657 (1953) Weissler A, Formation of hydrogen peroxide by ultrasonic waves: Free radicals. J Am Chem Soc 81: 1077-1081 (1959)	Research papers	Earliest papers to confirm beneficial use of sonochemical reactors in radical production and hence chemical processing applications
Lindley, J., Mason, T.J., Use of ultrasound in chemical synthesis. Chem. Soc. Rev. 16, 275 (1987)	Review	Mainly deals with ultrasound enhanced chemical synthesis processes
Mason, T.J., Practical Sonochemistry: users guide in chemistry and chemical engineering, Ellis Horwood series in Organic chemistry. Chichester, UK (1992)	Book	
Mason, T.J., Ultrasound in synthetic organic chemistry. Chem. Soc. Rev. 26, 443, (1997)	Review	Mainly deals with ultrasound enhanced organic chemistry processes
Luche, J.L., Synthetic Organic Sonochemistry, New York, Plenum Press (1998).	Book	

Mason T.J., Lorimer J.P., Sonochemistry: Theory, applications and uses of ultrasound in chemistry. Ellis Horwood Ltd. Chichester UK (1998)	Book	
Thompson, L.H., Doraiswamy, L.K., Sonochemistry: Science and Engineering. Ind. Eng. Chem. Res. 38, 1215 (1999)	Review	Overviews chemical processing applications
Keil, F.J. and Swamy, K.M., Reactors for sonochemical engineering-present status. Rev. Chem. Eng. 15, 85-155 (1999)	Review	Engineering aspects of sonochemical reactors
Shah, Y.T., Pandit, A.B., Moholkar, V.S., Cavitation Reaction Engineering., Plenum Publishers NY USA (1999)	Book	
Adewuyi, Y.G., Sonochemistry: Environmental Science and Engineering applications. Ind. Eng. Chem. Res. 40, 4681 (2001)	Review	Deals with applications in wastewater treatment
Gogate, P.R., Shirgaonkar, I.Z., Sivakumar, M., Senthikumar, P., Vichare, N.P., Pandit, A.B., Cavitation reactors: Efficiency analysis using a model reaction AIChE J., 47, 2326 (2001)	Research paper	Compares different forms of cavitation reactors in terms of energy efficiency and cavitation effects
Vinatoru, M., An overview of the ultrasonically assisted extraction of bioactive principles from herbs, Ultrason. Sonochem. 8, 303 (2001)	Review	Overviews applications in intensification of extraction
Gogate, P.R., Tatake, P.A., Kanthale, P.M., Pandit, A.B., Mapping of sonochemical reactors: Review, Analysis and experimental verification. AIChE J., 48, 1542 (2002)	Review	Overviews literature related to mapping of sonochemical reactors for identifying the distribution of cavitation activity
Gogate, P.R., Pandit, A.B., A review of imperative technologies for Waste water treatment II: Hybrid methods, Adv. Env. Res., 8(3-4), 553-597 (2004)	Review	Deals with combination of sonochemical reactors with other advanced oxidation processes and chemical treatment with a special emphasis on water treatment
Gogate, P.R., Pandit, A.B. Sonophotocatalytic oxidation based reactors for wastewater treatment: A critical review, AIChE J., 50, 1051 (2004)	Review	Overviews combination of sonochemical reactors with photocatalytic reactors with a special emphasis on wastewater treatment
Margulis, M.A., Sonochemistry as a new promising area of high energy chemistry, High Energy Chem., 38, 135 (2004)	Review	Overviews the development of knowledge base of sonochemistry as high energy chemistry
Gedanken, A., Using sonochemistry for the fabrication of nanomaterials, Ultrason. Sonchem., 11, 47 (2004)	Review	Overviews application of sonochemistry for synthesis of nanomaterials
Ruecroft, G., Hipkiss, D., Ly, T., Maxted, N., Cains, P. W., Sonocrystallization: The Use of Ultrasound for Improved Industrial Crystallization, Org. Proc. Res. Dev., 9, 923-932 (2005)	Review	Overviews applications in intensification/ improvement of crystallization operations
Rosenthal, I., Sostaric, J.Z., Riesz, P., Enlightening sonochemistry, Res. Chem. Intermediates, 30, 685 (2004)	Review	Reviews research on the excitation of solutes by sonoluminescence, the combined effects of ultrasound and light on liquid systems and the effect of ultrasound on photocatalytic reactions.

Toukoniitty, B., Mikkola, J.-P., Murzin, D.Y. Salmi, T. Utilization of electromagnetic and acoustic irradiation in enhancing heterogeneous catalytic reactions, Applied Cat. A. Gen. 279, 1 (2005)	Review	Overviews combinative use of microwave irradiation and ultrasounds along with details about individual operations
Gogate, P.R., Pandit, A.B., Sonochemical Reactors: Scale up aspects, Ultrason. Sonochem., 11, 105-117 (2004)	Review	Reviews bubble dynamics studies for prediction of cavitation intensity and also concentrates on the scale up aspects
Cravatto, G., Cintas, P., Power ultrasound in organic synthesis: Moving cavitation chemistry from academia to innovative and large-scale applications, Chem. Soc. Rev., 35, 180 (2006)	Review	Recent review covering organic synthesis applications and also focusing on pathway for large scale applications

#### 4.2 Relevant patents and patent holders

*(Provide the list of relevant patents in Table 7. Under "remarks" provide, where applicable, the names/types of products targeted by the given patent.)*

Table 7. Relevant patents

Patent	Patent holder	Remarks, including names/types of products targeted by the patent
Corrosion Resistant Ultrasonic Horn (US Patent 6652992)	Gunnerman, R.W.	Ultrasonic horns of titanium are made corrosion resistant in aqueous media by the providing the horns with a silver end surface, either as a portion of the end surface or as the entire end surface.
Organism inactivation method and system (US Patent application 2006/0086604)	Puskas, W.L.	Use of multiple frequency irradiation for microbial disinfection and water treatment
High power ultrasonic reactor for sonochemical applications (US 7157058)	Evgeny Marhasin, Marina Grintzova Vicktor Pekker Yuri Melnik	Sonochemical reactor based on magneto-restrictive transducers emitting uniform ultrasonic field
Production of crystals in a fluidized bed with ultrasonic vibrations (US 3510266)	Midler, M. and Clark N.J.	Uses a combination of conventional fluidized bed crystallizer and ultrasonic irradiation where cavitation is used for breakage of the over size crystals, which in turn serve as seeds
Ultrasonic Flow Cell (WO0035579 B1)	Perkins J.P. AEA Technologies	Modular flow cell of 5 L capacity with multiple transducers; for large scale operation combination of flow cells can be used

#### 4.3 Institutes/companies working on the technology

*(Provide the list of most important research centers and companies in Table 8)*

Table 8. Institutes and companies working on the technology

Institute/Company	Country	Remarks
Texas Tech University (Dominick J. Casadonte Jr.)	USA	Chemical processing applications including wastewater treatment, synthesis
Institute of Chemical Technology, University of Mumbai ( A.B. Pandit, P.R. Gogate)	India	Theoretical analysis and applications including chemical synthesis, nanotechnology, leaching and wastewater treatment
Indian Institute of Technology, Guwahati (V.S. Moholkar)	India	Theoretical analysis of cavitation phenomena and wastewater treatment applications
University of Abertay Dundee (D.H. Bremner)	Scotland, UK	Experimental studies related wastewater treatment applications; a novel combination with advanced Fenton oxidation for process intensification
University of California (A. Szeri)	USA	Theoretical aspects of bubble dynamics in cavitation phenomena, applications to transport processes in bubbles, ultrasound in medicine, sonoluminescence
University of Illinois (Kenneth S. Suslick)	USA	Chemical Effects of Ultrasound, Physical and Mechanistic Sonochemistry and Sonoluminescence and Biomedical Applications of Sonochemistry
Bharatidasan University (M. Sivakumar)	India	Nano material synthesis using cavitation
University of Melbourne (Franz Grieser and M. Ashokkumar)	Australia	Sonochemistry applications in food industry, polymer synthesis; Sonoluminescence; Combined sonochemistry and photochemistry for environmental applications
National Institute of Advanced Industrial Science and Technology (Y. Iida, K. Yasui)	Japan	Fundamental research into bubble dynamics, mapping, environmental applications, material synthesis
University of Bath (Gareth Price)	UK	Applications to Polymer chemistry
Universite de Savoie (Christian Petrier)	France	Applications to environmental chemistry
Laboratoire de Génie Chimique (Anne Marie Wilhelm, H. Delmas)	France	Applications to multiphase reactions/reactors
Universität Göttingen (R. Mettin)	Germany	Theoretical research related to bubble dynamics, bubble activity and cavitation structures
Jinan University (W. Guo)	China	Sonochemical synthesis of nanomaterials

Sonochemistry Research Center, Coventry Center (Tim Mason)	UK	Chemical Synthesis, Environmental protection, Food Technology, Reactor design and scale up, Combination with Electrochemistry
Bar-Ilan University (A. Gedanken)	Israel	Applications of sonochemistry and sonoelectrochemistry for nano material synthesis
Shiga University (T. Ando, T. Kimura)	Japan	Investigation into chemical aspects of ultrasound assisted chemical reactions
Nagoya University (S. Koda)	Japan	Investigation into chemical aspects of ultrasound assisted chemical reactions
California Institute of Technology (M. Hoffmann)	USA	Fundamentals and applications of ultrasonic irradiation for water and wastewater treatment
Ohio State university (L. Weavers)	USA	Application to wastewater treatment and membrane defouling
North Carolina State University (Y. Adewuyi)	USA	Application of ultrasonic irradiation and its combination with AOP for wastewater treatment
Cornel University (W. Sasche)	USA	Fundamental understanding of ultrasound propagation, development and characterization of ultrasonic transducers
Universidad de Extremadura (P. Cintas)	Spain	Chemical processing using ultrasound and its combination with microwave irradiations
University of Milan (V. Ragaini, C. Bianchi)	Italy	Synthesis of catalysts, polymerization reactions and use of sonochemical reactors with combination of photocatalytic oxidation
University of Turin (Giancarlo Cravotto)	Italy	Intensification of synthesis and combination with microwave irradiations
TUHH (Uwe Neis)	Germany	Use of ultrasonic reactors for intensification of sludge digestion, biomass generation and wastewater treatment

## 5. Stakeholders

### 5.1 Suppliers and developers

*(Provide the list of key suppliers/developers in Table 9)*

Table 9. Supplier and developers

Institute/Company	Country	Remarks
Sonics and Materials Inc.	USA	Reactors for chemical processing, atomization, ultrasonic welding, cutting etc.

Hielscher Ultrasonics	Germany, USA	Suppliers for laboratory and industrial scale reactors; Typically low frequency high power dissipation reactors for homogenizing, emulsifying, disintegration or de-agglomeration, dispersion etc. on production level
Branson Ultrasonics	USA	Reactors based on high-frequency ultrasonic energy applied to biological and chemical processing for disruption of cellular structures, homogenization, Emulsification, dispersion etc.
Dakshin	India	Tailor made sonochemical reactors specialty in multiple frequency multiple transducer reactors (laboratory to pilot scale capacity)
Prosonix	UK	Specialized in intensification and improvement of crystallization operations, also in liquid atomization
Active Ultrasonics	Switzerland	Suppliers of single or multi frequency sonochemical reactors (ultra and mega range) with a wide range of power dissipation suitable for laboratory to industrial scale applications
Advanced Sonic Processing Systems	USA	Suppliers of single or multi frequency sonochemical reactors, vibrating trays etc and reactors based on the use of magneto-restrictive transducers which facilitate continuous operation

## 5.2 End users

*(Describe the existing and potential end-users, other than those already listed in Table 2)*

**Chemical Processing Industries:** For intensification of chemical processing applications by way of either using sonochemical reactors in a supplementary role (e.g. increased rates of mass transfers and/or mixing) or by complete replacement of the existing technologies. It also finds an application in fine particle dispersion e.g. for preparation of nano-suspensions

**Food Processing Industries:** For homogenization operation for making uniform mixtures of liquids or liquid suspensions and also for emulsification operations involved in processing foods.

**Biotechnology:** For disruption of cells for release of intracellular products and also cell lysing for opening biological tissues and cells to extract enzymes and DNA, prepare vaccines.

**Environmental Protection:** For wastewater and water treatment mostly in combination with the existing Advanced Oxidation Processes or biological oxidation

## 6. Expert's brief final judgment on the technology

*(maximum 5 sentences)*

Sonochemical reactors have come of age, if one is looking for non-specific conversions i.e. mineralization of pollutants, disinfection of water etc. However, in terms of the cost of the treatment, it can be used only where conventional treatment methods either are not effective or very time consuming. They also have a role, in a supplementary mode for process intensification. The physical applications of sonochemical reactors, such as sonocrystallization, emulsification are however regularly practiced in the industry and growing significantly in number. Larger and larger systems are now commercially available (up to 8 kW of power delivery through a single transducer).

The chemical synthesis applications are however very few and are still used in a supplementary mode and not as a replacement, possibly due to incomplete understanding of the reaction path-ways and spatial non-uniformities associated with the cavitation activity in the whole of the reactor content. For such applications, technology remains in early stage due to the problems associated with a reliable scale-up and the material of construction of the vibrating (transducer) surface.

Considerable R & D effort in a collaborative manner is required involving physicists, chemists, material scientists and chemical engineers for the next five years for a reliable scale up of sonochemical transformations.

The ability of such sonochemical reactors to deliver energy only at the location of transformation has a considerable potential savings of the overall energy requirement. This has been well established on a Laboratory scale and shows an excellent long term potential on a small and medium scale, especially in a specialty chemical industry.