

An Overview of Ultrasound Irradiation as A Greener Approach in Chemistry for Rapid and Efficient Synthesis

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Abstract: *In chemical synthesis, ultrasound irradiation is important because it provides a more efficient and environmentally friendly method than conventional techniques, especially when considering sustainable development. Although it offers many benefits, such as improved reaction rates, lower energy consumption, and milder conditions, it also has drawbacks that must be addressed. By facilitating greener, more economical, and efficient processes, ultrasound provides a useful tool for promoting sustainable chemical synthesis. Although there are obstacles to overcome, especially in terms of scaling up and comprehending mechanisms, ultrasound offers substantial advantages in accomplishing sustainable development objectives. Ultrasound can be a potent technology for the future of chemical manufacturing if the benefits and drawbacks are carefully weighed and reaction conditions are optimized.*

Keywords: Ultrasound, Sonochemistry, Sonication, Green Chemistry

I. INTRODUCTION

1.1: The revolution of Ultrasound Irradiation

Acoustic cavitation, which Thornycroft and Barnaby reported in 1895 [1], was the first phenomenon seen in water following the introduction of the idea of ultrasound in 1794. They observed that the propeller of the torpedo boat destroyer HMS Daring was eroded and pitted by the so-called "cavitation events," which involved the formation and implosion of large bubbles during the blades' movement. In 1917, Rayleigh described this phenomenon using mathematical models of the formation, growth, and collapse of vapor bubbles in an incompressible fluid [2]. Although cavitation was discovered early on, its first practical application in chemistry was not recognized until 1927 by Loomis, Wood, and Richards. High-frequency ultrasound's chemical effects were applied for the first time to accelerate reactions, encourage particle aggregation, and even serve as a disinfectant [3, 4]. Their research became a landmark in sonochemistry and had a big influence on the field's subsequent expansion. In 1934, Frenzel and Schultes documented the first "sono-luminescence" observation. This phenomenon is the release of light from liquid samples due to cavitation caused by ultrasonic irradiation [5]. These discoveries resulted in several important developments in sonochemistry, including the application of ultrasound in organic chemistry in 1938 and its impact on electrochemistry in 1935 [6,7]. Richards' seminal review paper, "Supersonic phenomena," was published in 1939 as a result of the extensive use of ultrasonic technology in chemical and biological processes during this time [8].

1.2: Overview of Ultrasound Irradiation in research paper publications

In 1950, Weissler et al. studied the aqueous oxidation of potassium iodide under ultrasonic irradiation. They found that iodide (I-) could undergo secondary oxidation to triiodide (I₃⁻) due to the short-lived reactive species created by cavitation, which was easily detected by spectrophotometry.[9]. This research led to the development of the chemical dosimetry method, which is still widely used today to evaluate the efficacy of the sonochemical Weissler reaction.



Nolting et al. published a modeling study on the dynamics of acoustic bubbles in 1951 [10] in order to understand the thermodynamic properties of cavitation and predict the extraordinarily high temperature of 10,000 K inside cavitation bubbles.

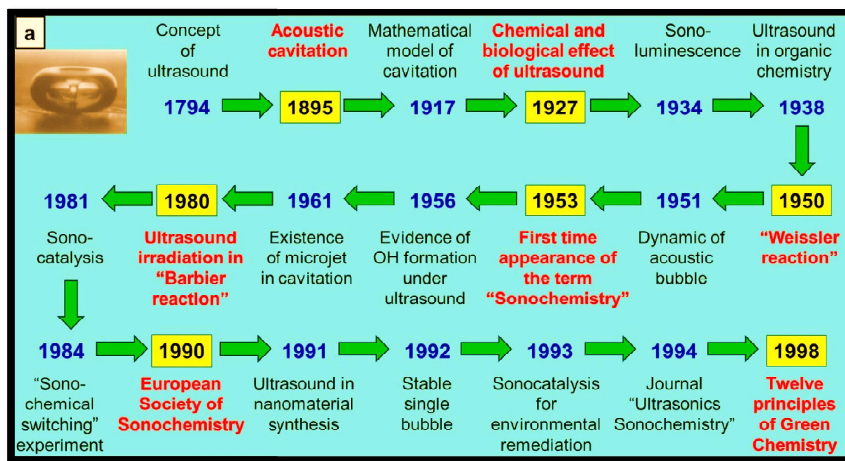


Figure 1: (a) The revolution of sonochemistry.

Sonochemistry is sometimes referred to as "hot-spot chemistry" due to this extremely high temperature. These results were later used by Weissler, a pioneering researcher in the field of ultrasound applications in chemistry, to create the term "sonochemistry" in his seminal work.[11]. Furthermore, reports of the detection of highly active radicals (H and OH) produced during ultrasonic cavitation and the microjet effect produced upon bubble collapse were published in 1956 [12] and 1961 [13], respectively. Even with this ground-breaking research, sonochemistry did not become widely recognized until the 1980s. Only eight times did the term "sonochemistry" appear in research papers between 1953 and 1986, according to a Scopus search. When the term "sonochemistry" reappeared in Neppiras' review of acoustic cavitation in 1980, sonochemistry was resurrected as a distinct field. The cost of commercializing ultrasonic equipment was subsequently lowered by notable developments in the development of piezoelectric materials and transducers in the 1980s [15]. The ultrasonic bath, ultrasonic horn, and ultrasonic probes with variable power and frequency were among the many ultrasonic devices that became accessible. Between 1980 and 1995, the term "sonochemistry" was used more than 100 times in published papers, suggesting that the availability of this technology led to an increase in research on the application of ultrasound in chemical and biological processes. Several significant discoveries were made during this period:

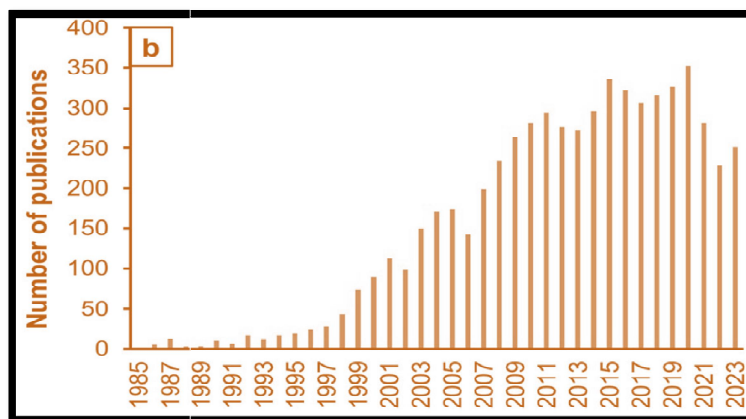


Figure 1. (b) Summary of research papers published on "sonochemistry"



(i) The highly successful "Barbier reaction"—an organometallic reaction between a carbonyl group and an alkyl halide under ultrasonic irradiation—was reported by Luche et al. in 1980; [16] (ii) Ando's "sonochemical switching" in organic synthesis in 1984 [18], where the reaction pathway changed from electrophilic to nucleophilic for the reaction between benzyl bromide and toluene; and (iii) Suslick et al. used the term "sonocatalysis" in 1981 [17] to describe olefin isomerization catalyzed by iron carbonyl catalysts, which was greatly enhanced under high frequency ultrasonic conditions.

1.3: Diversity in the research publication

These achievements, along with the work of prominent scientists of the time like Timothy Mason at Coventry University, Jean-Louis Luche at Joseph Fourier University, and Kenneth Suslick at the University of Illinois at Urbana-Champaign, laid the groundwork for the development of modern sonochemistry. Consequently, the Elsevier sonochemistry journal *Ultrasonics Sonochemistry* was founded in 1994 [20] and the European Society of Sonochemistry was founded in 1990 [19]. An important achievement during this period was the 1992 success of Gaitan et al. [21] in stabilizing a single bubble under ultrasonic irradiation and analyzing sonoluminescence during its expansion and contraction. This discovery established the fundamental framework for characterizing acoustic bubbles, which is still in use today. Ultrasound's application quickly expanded beyond organic synthesis to include biological engineering, environmental remediation, medical therapy, and the synthesis of nanomaterials (Figure 1c). Paul Anastas and John Warner published a paper titled "Twelve Principles of Green Chemistry" in 1998 that outlined a set of rules that "reduces or eliminates the use or generation of hazardous substances in the design, manufacture, and applications of chemical products." [22]. Sonochemistry is widely regarded as a "green" process because of these concepts. Recent developments in the field of biomass conversion and polymer degradation show how sonocatalysis could be very beneficial for a circular economy and sustainable, eco-friendly chemistry. [23–31].

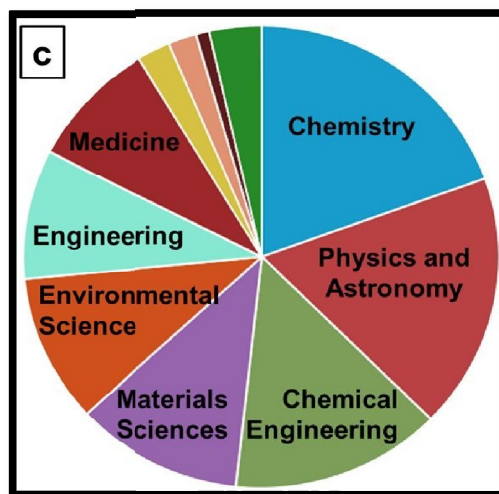


Figure 1. (c) Summary of research papers published on "sonochemistry" in different fields.

II. APPLICATION AREAS OF SONOCHEMISTRY

Cleaning treatments, organic sonochemistry, food and dairy sonoprocessing, environmental remediation, biomedical applications, and the synthesis of nanomaterials are just a few of the many industries that use sonochemistry (Figure S2a). These applications make use of both the chemical and physical effects of sonochemistry. Low frequency ultrasound (LFUS) is widely used to create physical effects in food processing and cleaning. Although these processes are sometimes called "false sonochemistry applications," [32] they rely on some of the chemical effects of cavitation events, which are essential to sonochemistry. Liquid jet and shockwave effects cause structural changes in cleaning



applications, such as material fragmentation, ductile material deformation, and surface contamination removal.³⁴ Sonochemical cleaning is more effective than conventional methods like water washing, mechanical abrasion, UV treatments, and aqueous chemical disinfection. Ultrasound provides two main advantages that lead to this enhanced efficacy: The contaminated particles' surface adhesion is lessened by (i) increased mass transport and (ii) localized mechanical shear force at the material's treated surface [35, 36]. Optimizing critical operational parameters, including ultrasonic power, frequency, and ultrasonic irradiation temperature, is necessary to achieve optimal performance. Recently, mathematical models have been proposed to predict the translation of cleaning efficiency on an industrial scale. [37, 39] Industrial ultrasound-assisted cleaning is still a significant challenge that will require additional technological advancement before it can be made economically viable, despite the promising results at the lab level.

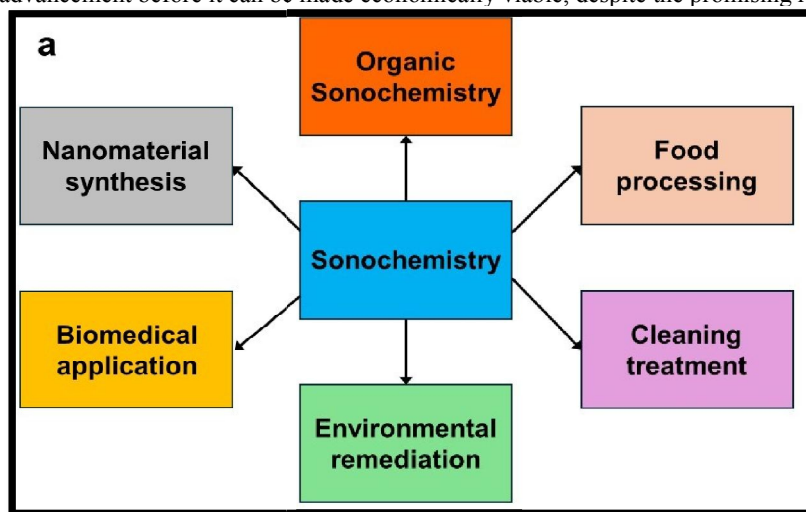


Figure 2. (a) General application areas of sonochemistry.

The powerful physical effects of cavitation have also been used by the dairy industry and many food processing operations [42]. The main mechanism behind ultrasound-induced extraction in food applications is the formation of micro-jets during asymmetrical cavitation near the solvent and cell tissue interface, which breaks down the cell walls and enhances the transport of solutes into solvent [43]. As opposed to conventional methods, which require heating or a large amount of solvent, this phenomenon allows specific compounds to be extracted from natural products more rapidly and with higher yields at room temperature. Sono-extraction has been successfully used to extract polyphenols from orange peels [44] and purple corn pericarp [45], β -carotene from fresh carrots [46], pectin from grapefruit [47] and pomegranate peels [48], C-phycocyanin from *Spirulina platensis* [49], caffeine from green coffee beans [50], and other high-value products like antioxidants, anthocyanins, chlorophyll, and flavonoids [51–54]. During the emulsification and de-emulsification processes, high shear forces, strong shock waves, and micro-jets created by cavitation at the interface between two immiscible liquid phases promote the disruption of droplets into the dispersion medium, making it simpler to form stable 10-100 nm nanoemulsion droplets.

Ultrasound significantly improves the quality and homogeneity of the nanoemulsions when compared to the conventional method. Forty The food, cosmetic, and pharmaceutical industries have made extensive use of nanoemulsions due to their high bioavailability, low turbidity, and low polydispersity. Additionally, ultrasonic emulsification and de-emulsification are efficient methods for producing them [55–61]. In the aforementioned cleaning, extraction, and emulsion processes, ultrasound is mainly employed for the physical effects of cavitation; its full chemical potential is not fully utilized. The chemical effects of sonochemistry are far more significant in other applications, such as organic synthesis (also called organic sonochemistry), water remediation, the synthesis of nanomaterials, and medical applications.

Many recent examples of ultrasound's capacity to generate chemicals with higher yield and selectivity than conventional synthesis schemes can be found in the literature [62–64]. In the field of polymer synthesis, which is a



subset of organic synthesis, sonochemistry assisted in enabling polymerization with a higher yield and better quality [65]. Frequencies between 20 and 100 kHz are commonly used in organic sonochemistry. Higher frequency ultrasound usually results in previously unseen activity [24, 30, 67]. Organic chemistry reactions are greatly aided by the physical and chemical effects of sonochemistry. Physical effects enhance mass transfer, while chemical effects increase the activity and selectivity of the reactions. Luche et al. [69] classified organic sonochemistry into three classes based on which effect predominated in the early stages of the field: class 1 was driven by free radicals generated by cavitation in homogeneous reactions, class 2 was driven by mechanical effects in heterogeneous media, and class 3 combined the traits of classes 1 and 2 with a critical step involving the transfer of a single electron. "False sonochemistry" and "true sonochemistry," respectively, were assigned to classes 2 and 3. However, organic sonochemical reactions in modern sonochemistry can only be classified as convergent or divergent from a mechanistic standpoint (Fig. b).⁶⁸

Organic sonochemistry adheres to the Apfel rules, which require that the acoustic parameters and calibration of ultrasonic devices be appropriately designed to ensure the accuracy and repeatability of experimental results [70, 68]. New technological developments are combined with other organic sonochemical techniques and piezo-redox chemistry. [71–75]. Flow-chemistry^{76–83} and automation chemistry^{84–89} demonstrate the high potential impact of organic sonochemistry in both synthetic and nonsynthetic applications under environmentally friendly conditions. The chemical effects of sonochemistry are even more apparent in environmental remediation applications because cavitation generates highly reactive oxygen species (ROS) such as $\cdot\text{OH}$, $\cdot\text{O}_2$, and $\cdot\text{OOH}$. Sonochemical environmental remediation is therefore classified as an advanced oxidation process (AOP) [90,91]. The removal of organic and inorganic pollutants from soil [94–96], sludge [97–99], sediment [94], and wastewater treatment [91–93] is a common application of environmental sonochemical remediation. It is considered a "green process" because it uses few chemicals and operates in ambient conditions. Volatile organic pollutants can be pyrolyzed in cavitation bubbles due to their extremely reactive environment. Moreover, oxidative degradation of these organic compounds can occur at the bubble/liquid boundary, where radicals are formed simultaneously with the formation of the cavitation bubble, or in the bulk solution, where active radicals are released after bubble collapse.

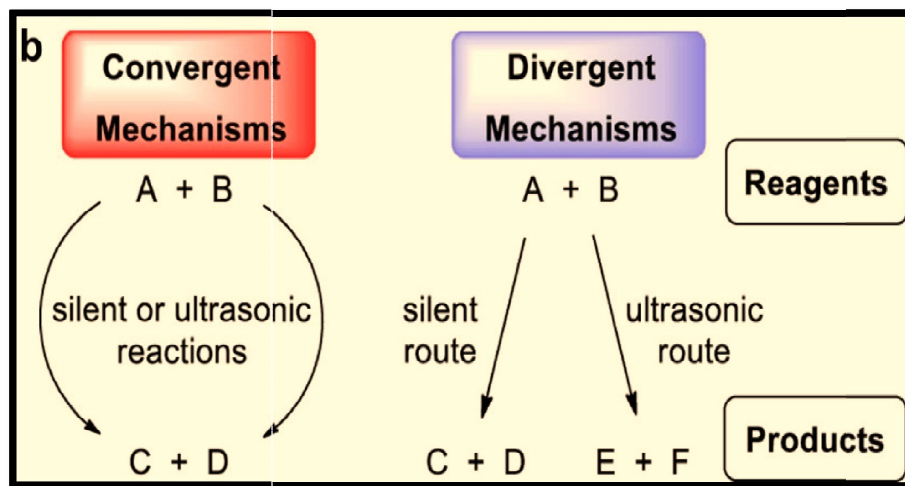


Figure 3. (b) Classification of organic sonochemical reactions from a mechanistic perspective, reproduced from an open access publication.⁶⁸

Sonochemical remediation has also successfully eliminated a wide range of hazardous pollutants, including polycyclic aromatic hydrocarbons, drugs, pesticides, fungicides, dyes, and pigments [90–92, 100–102]. For instance, Andani et al. reported 96% efficiency for Rhodamine B degradation using sonotreatment in an ultrasonic bath with a frequency of 37 kHz. This efficiency was twelve times greater than that of a traditional method without the extra benefits of sonochemistry. For sonochemical treatment to be effective, the frequency, ultrasonic wave power, and irradiation duration are crucial. While lower frequency ultrasound (20–40 kHz) is commonly used for soil and sludge remediation, higher frequency ultrasound (200–600 kHz) is typically used for wastewater treatment due to the need for a high ROS



density [94, 103]. It is challenging to rapidly quench active oxygen species in the solution using sonochemical treatment because there are only 10% of active radicals in the bulk solution. Therefore, sonochemistry can enhance wastewater treatment [105] when combined with other AOP processes such as coagulation, ozonation, and Fenton oxidation [104, 100]. Because sono-elimination of hazardous compounds may not always result in their complete degradation to H₂O and CO₂, it is crucial to closely monitor the types of by-products that are produced to ensure that there is no secondary environmental toxicity [106,107]. Sonochemical treatments are currently developed at the laboratory scale for a simulated wastewater composition because it is not feasible to use them for environmental remediation on a commercial scale [108]. On a larger scale, efforts are being made to enhance the technology and practicality of sonotreatment techniques. These efforts include designing more effective reactors, optimizing energy use, and developing continuous processes [106].

Additionally, sonochemistry shows promise for biomedical applications in the eradication of diseases and the treatment of cancer. In this case, sonochemistry can be used to improve the efficacy of current cancer treatment methods, which are often expensive, time-consuming, and incapable of precisely targeting cancer cells [109]. Exogenous medical microbubbles created during sonochemical cavitation facilitate the delivery of cancer-treating drugs to affected cells by keeping the plasma concentration within the therapeutic range [110–112]. Due to its high precision, high specificity, and non-invasiveness, sonochemistry has been used as a treatment for brain tumors [107], xenograft tumors [113], breast cancer cells [114], melanoma cancer cells [115], and head and neck cancer cells [116]. Because sonochemistry can stabilize nanoemulsions, it is also used in many drug delivery applications, including topical, ocular, oral, and intravenous methods [60, 112, 117, 118]. High frequencies (500 kHz–1 MHz) are commonly used in these applications. Drug delivery with ultrasound assistance has been shown to be substantially more effective [119]. Due to its potential for treating cancer, sonodynamic therapy—which combines sonochemistry and nanostructured catalysts—has received a lot of attention recently [120–122]. The presence of nanostructured catalysts, which accelerate and enhance the production of highly active oxidative agents that can specifically kill cancer cells without causing side effects, demonstrates the potential of sonochemistry in cancer treatment [115, 123]. Sonochemistry is especially useful for the synthesis of nanomaterials, including metal oxides, nanoparticles, core-shell structures, metal alloys, and two-dimensional materials.

The physical and chemical effects of sonochemical cavitation events are exploited in this process [124–130]. Nanomaterials play a major role in biomedicine, energy storage, environmental sciences, and catalysis [131–135]. Important features of nanomaterials include their exposed facet [136,137], particle size [138,139], and morphology [140]. Using conventional techniques, which usually require lengthy preparation times, dangerous reagents, solvents, and surfactants, as well as harsh conditions (high temperature), it is challenging to control these structural features at the nanoscale. Due to its short synthesis time, use of ambient conditions (room temperature), environmentally friendly reactants, and lack of template or surfactant, sonochemical synthesis has the advantage of being a "green chemical synthesis" of nanomaterials. Examples of physical effects from cavitation events that improve heat and mass transfer during the synthesis of nanomaterials and provide fine control over the material's morphology include microstreaming, high-speed microjets, and high-intensity shockwaves. Additionally, the inhibition of large particle growth due to the increased collision probability caused by transient cavitation and the fast-cooling rate of sonochemical processes (~10¹⁰ K/s) leads to the formation of small and highly uniform particle sizes with large specific surface area and high porosity. Sonochemistry's chemical effects can also regulate the structure of synthesized nanomaterials.

Primary sonochemistry activates the initial reagents as they are incorporated into a cavitation bubble, creating precursors for the nucleation of nanoparticles. Secondary sonochemistry releases a large number of active radicals into the bulk solution when cavitation bubbles burst. These radicals alter the surface energy and serve as a structural template to control the exposed facet and morphology as the nanomaterials expand by attaching themselves to the nuclei of the nanoparticles. To achieve the desired properties of the synthesized materials, it is essential to precisely control the ultrasonic frequency, power, sonication time, and ultrasonic activation mode. Nanomaterials are usually synthesized using high-intensity low frequency ultrasound (20–100 kHz). Many different materials, such as metals, metal oxides, sulphides, alloys, composites, and amorphous materials, have been created using this technique. This illustrates the potency and great potential of sonochemistry as a process intensification technique in materials science.



Although this approach isn't yet ready for general application, it is expected to play an increasingly significant role in accelerating the development of next-generation engineered materials.

III. CONCLUSION

In contemporary chemical science, ultrasound irradiation has become a potent, adaptable, and eco-friendly instrument that provides a more environmentally friendly substitute for traditional energy-intensive techniques. Under milder conditions, the special phenomenon of acoustic cavitation produced by ultrasonic waves allows for higher selectivity, better yields, faster reaction rates, and shorter reaction times. These benefits align ultrasound-assisted chemistry with the core ideas of green chemistry by greatly reducing waste, using less energy, and implementing safer operating procedures. Ultrasound irradiation's transformative potential is highlighted by its broad applicability in a variety of fields, including organic synthesis, catalysis, polymer chemistry, nanomaterial fabrication, and environmental remediation. Sonochemistry encourages sustainable process development without sacrificing efficiency or scalability by facilitating solvent-free or aqueous-phase reactions and enabling effective catalyst activation. Its compatibility with other enabling technologies, such as flow chemistry, biocatalysis, and microwave irradiation, also creates new opportunities for sustainable chemical synthesis innovation. Despite its many advantages, there are still issues with reactor design, scale-up, and energy optimization that need more research. The gap between laboratory-scale success and industrial implementation is anticipated to be closed by ongoing developments in ultrasonic equipment, mechanistic knowledge, and process modeling. All things considered, ultrasound irradiation is a promising and quickly developing green technology with enormous potential to transform chemical research and industrial processes in the direction of a more sustainable and environmentally friendly future.

IV. ACKNOWLEDGMENT

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