

Supplementary information

Sonochemistry and Sonocatalysis: Current Progress, Existing Limitations, and Future Opportunities in Green and Sustainable Chemistry

Quang Thang Trinh,^{1,*} Nicholas Golio,² Yuran Cheng,^{1,3} Haotian Cha,¹ Kin Un Tai,¹ Lingxi Ouyang,^{1,4} Jun Zhao,⁵ Tuan Sang Tran,¹ Tuan Khoa Nguyen,¹ Jun Zhang,^{1,6} Hongjie An,^{1,4} Zuojun Wei,³ Francois Jerome,² Prince Nana Amaniampong,^{2,*} Nam-Trung Nguyen^{1,*}.

¹*Queensland Micro and Nanotechnology Centre, Griffith University, 170 Kessel Road, Nathan, QLD 4111, Australia.*

²*CNRS, Institut de Chimie des Milieux et Matériaux de Poitiers (IC2MP), Université de Poitiers, Bat B1 (ENSI-Poitiers), 1 rue Marcel Doré, 86073 Poitiers, France.*

³*Key Laboratory of Biomass Chemical Engineering of the Ministry of Education, College of Chemical and Biological Engineering, Zhejiang University, Hangzhou, Zhejiang, 310058, P.R. China.*

⁴*School of Environment Science, Griffith University, 170 Kessel Road, Nathan, QLD 4111, Australia.*

⁵*Department of Biology, Hong Kong Baptist University, Hong Kong, China.*

⁶*School of Engineering and Built Environment, Griffith University, 170 Kessel Road, Nathan, QLD 4111, Australia.*

Corresponding authors:

q.trinh@griffith.edu.au (Q.T.T.)

prince.nana.amaniampong@univ-poitiers.fr (P.N.A.)

nam-trung.nguyen@griffith.edu.au (N.-T.N.)

1. A ‘sound’ story (History of sonochemistry)

Figure S1 briefly summarises the history of sonochemistry. Following the introduction of the concept of ultrasound in 1794, acoustic cavitation was the first observed phenomenon in water reported by Thornycroft and Barnaby in 1895.¹ 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 generation and implosion of large bubbles occurring during the movement of the blades. In 1917, Rayleigh explained this phenomenon using mathematical models of the formation, growth, and collapse of vapor bubbles in an incompressible fluid.² However, despite the early discovery of cavitation, its first useful application in chemistry was only observed in 1927 by Loomis, Wood, and Richards. Their application marked the first usage of the chemical effects of high-frequency ultrasound for accelerating reactions, facilitating particle aggregation, and even as a disinfectant.^{3, 4} Their research became the landmark in sonochemistry and strongly influenced the subsequent development of the field. In 1934, Frenzel and Shultes observed the first instance of “sonoluminescence” – the emission of light in liquid samples due to cavitation caused by ultrasonic irradiation.⁵ Several important developments in sonochemistry occurred subsequent to these discoveries, including the effects of ultrasound on electrochemistry in 1935⁶ and the application of ultrasound in organic chemistry in 1938.⁷ At this time, the applications of ultrasound in chemical and biological processes became more widespread leading to the seminal Richards’ review paper entitled “Supersonic phenomena” in 1939.⁸

In 1950, Weissler et al. studied the aqueous oxidation of potassium iodide under ultrasonic irradiation and found that the short-lived reactive species generated from cavitation were able to induce the secondary oxidation of iodide (I^-) to triiodide (I_3^-), which could be easily detected by spectrophotometry.⁹ This research laid the foundation for the development of chemical dosimetry method to evaluate the efficiency of the sonochemical Weissler reaction, which is still largely used in the present day. In 1951, Nolting et al. reported a modelling study on the dynamics of acoustic bubbles,¹⁰ contributing to understanding the thermodynamic properties of cavitation and predicting the extremely high temperature of 10,000 K inside cavitation bubbles. Due to this extremely high

temperature, sonochemistry is also called “hot-spot chemistry”. Those discoveries later led to the term “sonochemistry” coined by Weissler, a pioneering scientist in the field of ultrasound applications in chemistry, in his seminal paper.¹¹ The detection of highly active radicals (H and OH) formed during ultrasonic cavitation and the microjet effect produced upon bubble collapse were also reported in 1956¹² and 1961,¹³ respectively. However, despite this ground-breaking research, the field of sonochemistry was not widely recognised until the 1980s. According to a Scopus search, the term “sonochemistry” only appeared 8 times in research papers from 1953 to 1986.

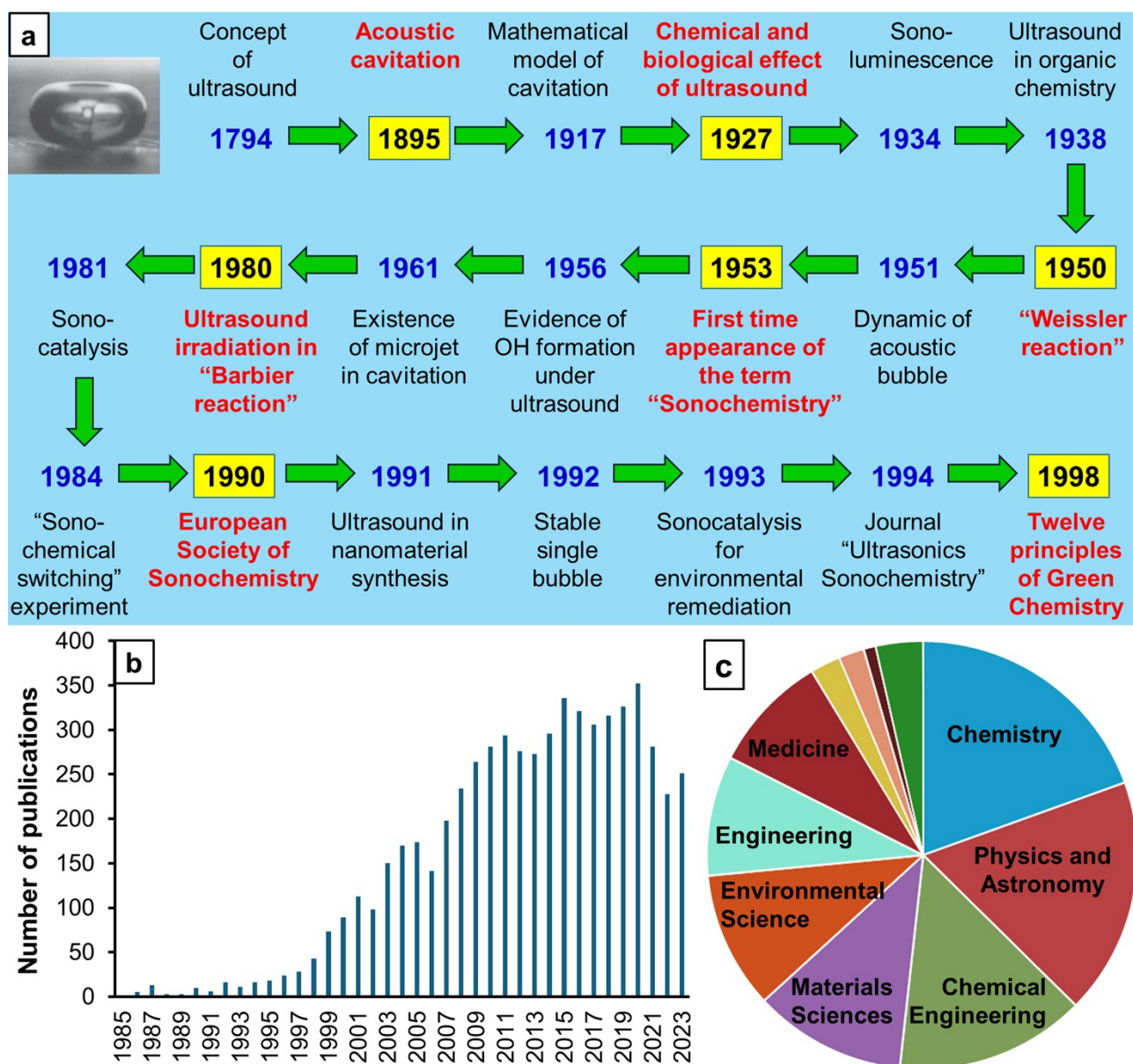


Figure S1. (a) The revolution of sonochemistry. Inserted image shows a microjet bubble produced during sono-chemical cavitation. Some important landmarks in the history of sonochemistry are highlighted in red. (b) Number of publications with the keyword “sonochemistry” in different years, from Scopus search in September 2024. (c) Subject areas of research with the keywords “sonochemistry”, from Scopus search in September 2024.

In 1980, the term “sonochemistry” re-emerged in the review on acoustic cavitation by Neppiras,¹⁴ marking the rebirth of sonochemistry as a distinct discipline. In the 1980s, great successes in the development of piezoelectric materials and transducers consequently made the commercialisation of ultrasonic equipment more affordable.¹⁵ Different types of ultrasound apparatus, such as the ultrasound bath, the ultrasound horn and ultrasound probes with controllable power and frequencies became available. The availability of this technology boosted the number of studies on the application of ultrasound in chemical and biological processes, as was evidenced by the appearance of the term “sonochemistry” more than 100 times in published papers between 1980 and 1995 (Figure S1b). Some landmark discoveries in this period are (i) the highly effective “Barbier reaction”, which is the organometallic reaction between a carbonyl group and an alkyl halide under ultrasonic irradiation, reported by Luche et al. in 1980;¹⁶ (ii) the use of the term “sonocatalysis” by Suslick et al. in 1981¹⁷ relating to olefin isomerization catalysed by iron carbonyl catalysts, which was drastically enhanced under high frequency ultrasonic conditions; and (iii) the Ando’s “sonochemical switching” in organic synthesis in 1984,¹⁸ where ultrasound changed the reaction pathway from electrophilic to nucleophilic for the reaction between benzyl bromide and toluene. These achievements and the works from prominent scientists of this period (for example, 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 foundation for the development of modern sonochemistry, resulting in the establishment of the European Society of Sonochemistry in 1990¹⁹ and the Elsevier journal dedicated to sonochemistry, *Ultrasonics Sonochemistry*, in 1994.²⁰ One important achievement in this period is the success of Gaitan et al. ²¹ in stabilising a single bubble under ultrasonic irradiation and studying sonoluminescence during its expansion and contraction in 1992. This discovery established the fundamental framework to characterise acoustic bubbles, which is still being used at present. The application of ultrasound was quickly expanded beyond organic synthesis and found great successes in other areas, such as nanomaterials synthesis, environmental remediation, medical therapy, and biological engineering (Figure S1c). In 1998, Paul Anastas and John Warner published a paper entitled “Twelve principles of Green Chemistry”, outlining the set of principles that “*reduces or*

eliminates the use or generation of hazardous substances in the design, manufacture and applications of chemical products".²² Based on these principles, sonochemistry is widely accepted as a "green" process. For example, recent developments in the field of sonocatalysis for biomass conversion and polymer degradation proved that it could make a prominent contribution towards sustainable and eco-friendly chemistry and a circular economy.²³⁻³¹

2. Application areas of Sonochemistry

Sonochemistry is used in many diverse areas of applications, including cleaning treatments, food and dairy sonoprocessing, organic sonochemistry, environmental remediation, biomedical applications, and nanomaterials synthesis (Figure S2a). These applications leverage both the physical and chemical effects of sonochemistry. Physical effects induced primary from low frequency ultrasound (LFUS) are widely used in cleaning and food processing. Although these processes are sometimes categorised as "*false sonochemistry applications*",³² they are driven by cavitation events which are at the core of sonochemistry and utilise some of their chemical effects³³. In cleaning applications, liquid jet and shockwave effects cause structural changes, such as material fragmentation, ductile material deformation, and the removal of surface contamination.³⁴ Sonochemical cleaning is more efficient than conventional methods, such as water washing, mechanical abrasion, UV treatments, and aqueous chemical disinfection. This enhanced efficiency comes from two main advantages supplied by ultrasound: (i) improved mass transport and (ii) localised mechanical shear force at the treated surface of the material, which both help to loosen the adhesion between the contaminated particles on the surface.^{35, 36} Important operational parameters need to be optimised to obtain the best performance, including ultrasonic power, frequency, and temperature of the ultrasonic irradiation. Recently, mathematical models have been proposed to predict how the cleaning efficiency will be translated on an industrial scale.³⁷⁻³⁹ Despite the promising results achieved at the laboratory scale, the application of ultrasound-assisted cleaning at an industrial scale still remains a significant challenge, requiring more technological development before it becomes economically feasible.

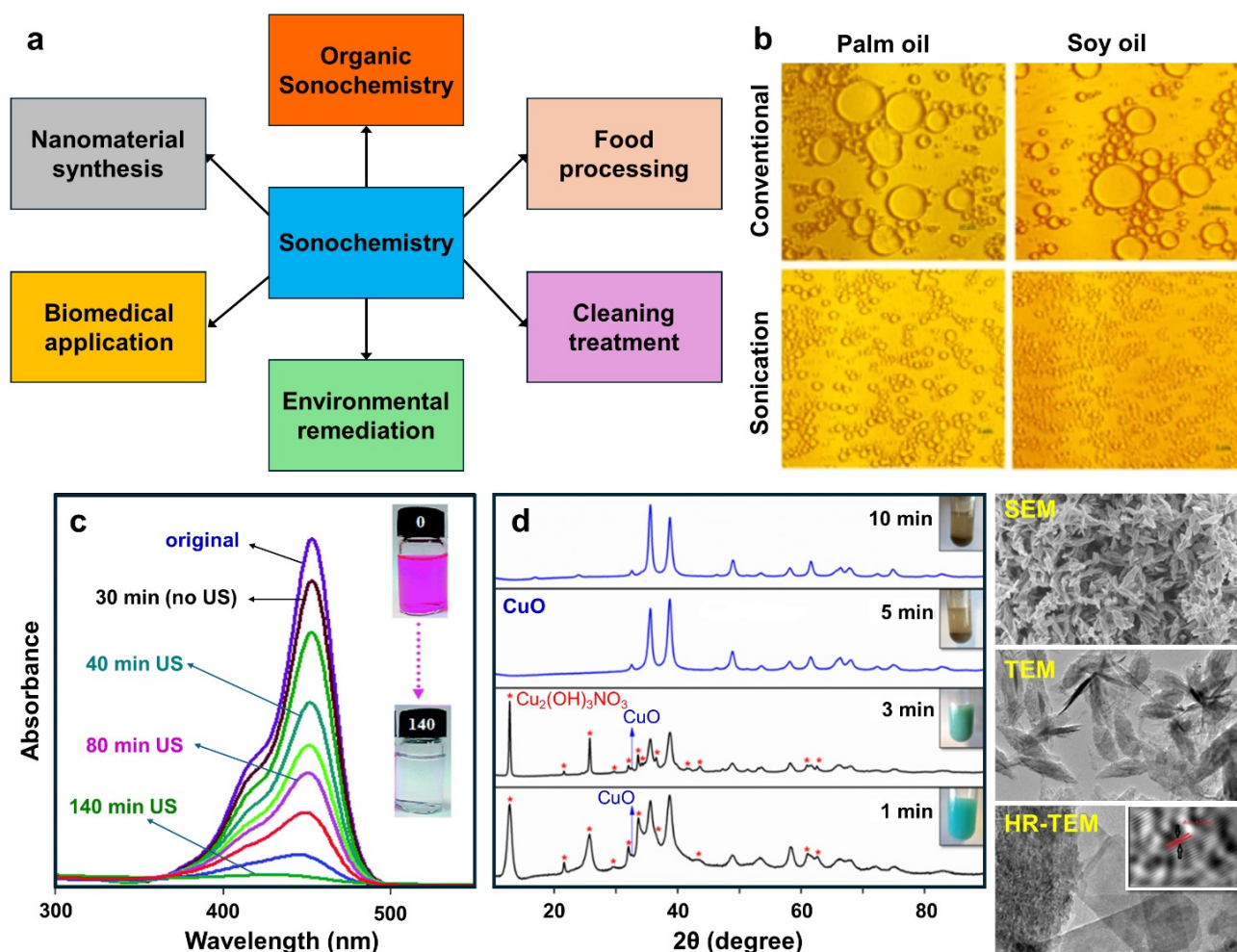


Figure S2. (a) General application areas of sonochemistry. (b) Optical microscopy images of oil-in-water emulsions obtained via sonication and conventional methods, reproduced with permission from Taha et al.⁴⁰ Copyright 2018, Elsevier. (c) The sonodegradation process of Rhodamin B (RhB), reproduced from an open access publication.⁴¹ (d) XRD patterns showing the structural changes in CuO sonocatalysts at different ultrasonic irradiation times, reproduced with permission from Amaniampong et al.²³ Copyright 2018, Royal Society of Chemistry.

The strong physical effects generated from cavitation induced by LFUS have also been used in many food processes and in the dairy industry,⁴². The main driving force for ultrasound-induced extraction in food applications is the generation of micro-jets during asymmetrical cavitation near the solvent and cell tissue interface, disrupting the cell walls and enhancing the transport of solutes into solvent.⁴³ This phenomenon facilitates the extraction of specific compounds from natural products with higher yields and shorter times at ambient temperatures, avoiding the use of a large amount of solvent and heating in conventional methods. Some successful applications of sono-extraction have been reported for the collection of polyphenols from an orange peel⁴⁴ and from purple corn pericarp,⁴⁵ β -carotene from fresh carrots,⁴⁶ pectin from grapefruit⁴⁷ and from a pomegranate peel,⁴⁸ C-phycocyanin from *Spirulina platensis*,⁴⁹ caffeine from green coffee beans,⁵⁰ and other high value-

added products, such as antioxidant compounds, anthocyanins, chlorophyll, and flavonoids.⁵¹⁻⁵⁴ In emulsification and de-emulsification processes, high shear forces, intense shock waves and micro-jets generated by cavitation at the boundary between two immiscible liquid phases promote the disruption of droplets into the dispersion medium, facilitating the formation of stable 10-100 nm nanoemulsion droplets. Figure S2b shows an example of oil-in-water nanoemulsions produced using ultrasound-assisted processes. Ultrasound significantly improves the quality and homogeneity of the nanoemulsions over the conventional method.⁴⁰ Nanoemulsions have broad applications in food, cosmetic, and pharmaceutical industries due to their high bioavailability, low turbidity, and low polydispersity and have been efficiently produced from the ultrasonic emulsification and de-emulsification.⁵⁵⁻⁶¹ However, technical challenges in scaling up the processes are currently hindering their application at an industrial scale.

The application of ultrasound in cleaning, extraction, and emulsion described above predominantly uses the physical effects of cavitation and does not take advantage of its full chemical potential. The chemical effects of sonochemistry are much more significant in other applications, including in organic synthesis (called organic sonochemistry), water remediation, nanomaterials synthesis, and in medical applications. There are many recent examples of ultrasound's success in producing chemicals with higher yield and selectivity than conventional synthesis schemes in the literature.⁶²⁻⁶⁴ In polymer synthesis, which is a specific area of organic synthesis, sonochemistry contributed to facilitating polymerization with a higher yield and superior quality.⁶⁵ The frequency range generally applied to organic sonochemistry is from 20-100 kHz. For example, Jayarajan et al. observed that the selective substitution reaction at the meta position of the arenes ring could not occur without sonication, however, in an ultrasonic bath with a frequency of 37 kHz (Figure S3a) they were able to achieve a yield of 98%.⁶⁶ The use of higher frequency ultrasound is not as common in organic sonochemistry, but it usually results in unprecedentedly high activity.^{24, 30, 67} Both the chemical and physical effects of sonochemistry play an important role in facilitating organic chemistry reactions. Physical effects enhance mass transfer, while chemical effects accelerate the activity and selectivity of the reactions.

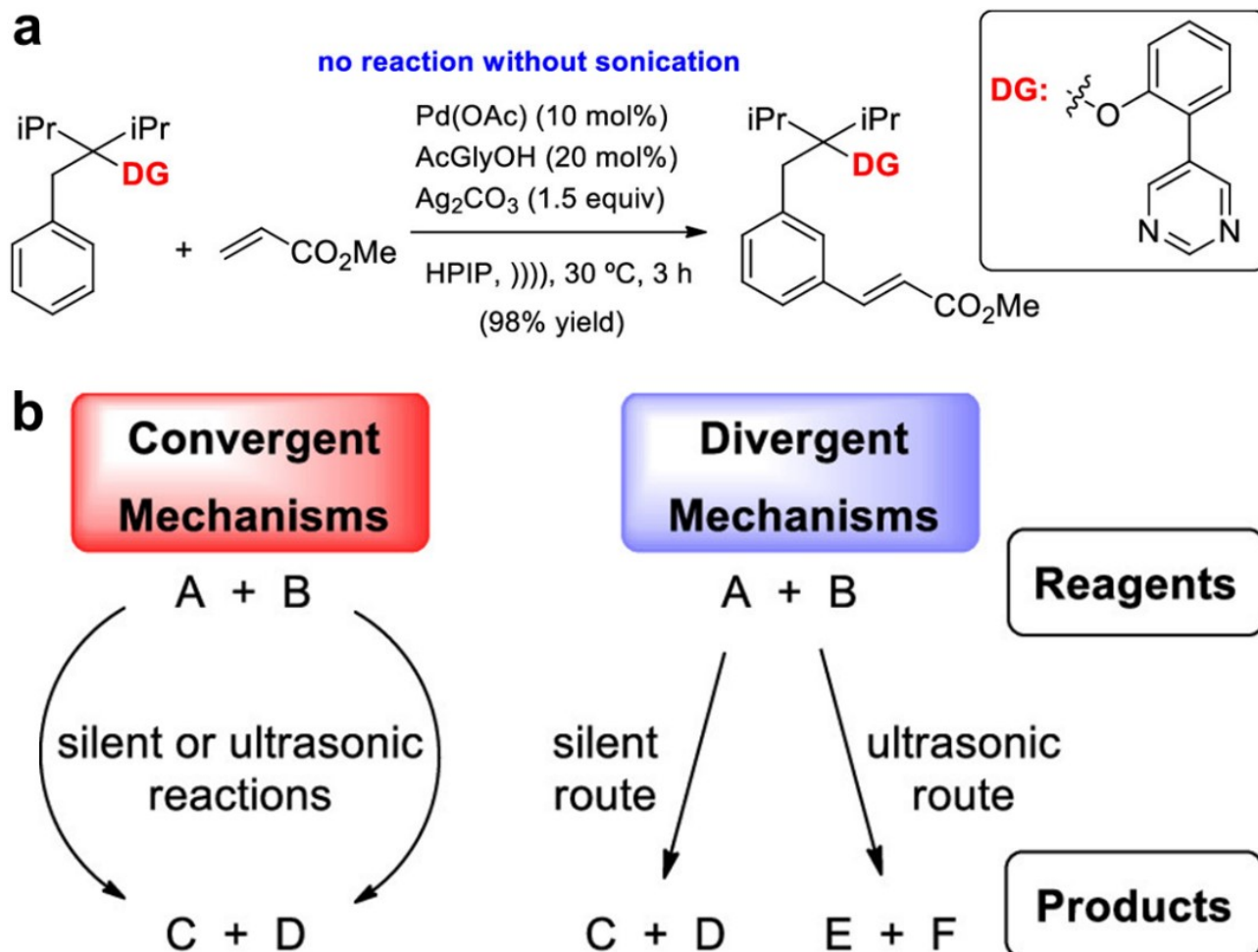


Figure S3. (a) Direct and selective meta-C–H substitution of arenes mediated by ultrasound, reproduced with permission from Jayarajan et al.⁶⁶ Copyright 2020, John Wiley and Sons. (b) Classification of organic sonochemical reactions from a mechanistic perspective, reproduced from an open access publication.⁶⁸

In the early days of sonochemistry, Luche et al.⁶⁹ classified organic sonochemistry into three classes based which effect was dominant: class 1 was driven by the free radicals generated by cavitation in homogeneous reactions, class 2 was driven by mechanical effects in heterogeneous media, and class 3 combined the features both classes 1 and 2, while also involving the transfer of a single electron in a key step. The classes 1 and 3 were considered as “*true sonochemistry*”, while class 2 was classified as “*false sonochemistry*”. However, modern sonochemistry only categorises organic sonochemical reactions into two categories, convergent and divergent, according to a mechanistic point of view (Fig. S3b).⁶⁸ Organic sonochemistry follows the Apfel rules, which state that the acoustic parameters and calibration of ultrasonic devices need to be designed appropriately to ensure the accuracy and reproducibility of experimental results.^{68, 70} The integration of recent technological innovations with organic sonochemical techniques, such as piezo-redox chemistry,⁷¹⁻⁷⁵

flow-chemistry⁷⁶⁻⁸³ and automation chemistry⁸⁴⁻⁸⁹ demonstrates the high potential impact of organic sonochemistry in synthetic and nonsynthetic applications under eco-friendly conditions.

The chemical effects of sonochemistry are even more pronounced in environmental remediation applications, owing to the highly reactive oxygen species (ROS) generated by cavitation (e.g., $\bullet\text{OH}$, $\bullet\text{O}_2$, and $\bullet\text{OOH}$). Thus, environmental remediation via sonochemistry is classified as an advanced oxidation process (AOP).^{90, 91} Because of its use of relatively few chemicals and operation under ambient conditions, environmental sonochemical remediation is considered to be a “green process” and is extensively applied to remove both inorganic or organic pollutants in wastewater treatment⁹¹⁻⁹³ and in soil,⁹⁴⁻⁹⁶ sludge⁹⁷⁻⁹⁹ and sediment remediation.⁹⁴ Volatile organic pollutants can be pyrolyzed inside cavitation bubbles due to the extremely reactive environment present inside the bubble. In addition, 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.

Sonochemical remediation has also been successfully used to eliminate a wide range of hazardous pollutants, including polycyclic aromatic hydrocarbons, pharmaceuticals, pesticides, fungicides, dyes, and pigments, with high efficiency.^{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 (Fig. S2c).⁴¹ This efficiency was 12-fold higher than that of a conventional process without the added benefits of sonochemistry. Important parameters for efficient sonochemical treatment are the frequency, the power of the ultrasonic waves, and the irradiation time. Ultrasound with higher frequencies (200 to 600 kHz) are typically applied for wastewater treatment due to the need for a high density of ROS, whereas lower frequencies (20-40 kHz) are often used in sludge and soil remediation.^{94, 103} One challenge of sonochemical treatment is the fast quenching of active oxygen species in the bulk solution (only 10% of active radicals are present in the bulk solution). Therefore, sonochemistry has the potential to be combined with another AOP processes, such as Fenton oxidation,¹⁰⁴ ozonation^{94, 100} and coagulation, to enhance the wastewater treatment.¹⁰⁵ We note that sono-elimination of hazardous compounds might not always result in their complete degradation to

H₂O and CO₂, and therefore it is important to carefully track the identity of by-products formed in order to ensure that there is no secondary environmental toxicity.^{106, 107} To date, most sonochemical treatments for environmental remediation are developed at laboratory scale for a simulated wastewater composition, and is not yet feasible for commercial scale.¹⁰⁸ Efforts are being made in this field to enhance the technology and economic feasibility of sonotreatment processes at a larger scale. These efforts include designing more effective reactors, developing continuous processes, and optimising their energy consumption.¹⁰⁶

Sonochemistry also shows promising biomedical applications in eliminating diseases as well in drug delivery for cancer treatment. In this context, sonochemistry can be applied to improve the efficiency of current cancer treatment methods, which are often expensive, time consuming, and lack selectively in targeting cancer cells.¹⁰⁹ Exogenous medical microbubbles generated during sonochemical cavitation facilitate the delivery of cancer treating drugs to affected cells by stabilising the plasma concentration within the therapeutic range.¹¹⁰⁻¹¹² Due to its high accuracy, high specificity, and non-invasive nature, sonochemistry has been used as a therapy for curing brain tumours,¹⁰⁷ xenograft tumours,¹¹³ breast cancer cells,¹¹⁴ melanoma cancer cells,¹¹⁵ and head and neck cancer cells.¹¹⁶ With its ability to stabilise nanoemulsions, sonochemistry is also used in a wide range of drug delivery applications, including topical, ocular, oral, and intravenous methods.^{60, 112, 117, 118} In these applications, high frequencies are usually applied (500kHz-1MHz). Much higher efficiency has been reported for ultrasound-assisted drug deliveries.¹¹⁹ Recently, a combination of sonochemistry and nanostructured catalysts, called sonodynamic therapy, has received great interest for its effectiveness in treating cancer.¹²⁰⁻¹²² The presence of nanostructured catalysts accelerates and enhances the formation of highly active oxidative agents, which can selectively destroy cancer cells without inducing side effects,^{115, 123} demonstrating the potential of using sonochemistry in cancer treatments.

One particularly useful application of sonochemistry is nanomaterials synthesis (including metal oxides, nanoparticles, core-shell structures, metal alloys, and 2D materials),¹²⁴⁻¹³⁰ which benefits from the corporation of both the physical and chemical effects of sonochemical cavitation events.

Nanomaterials play an important role in biomedicine, catalysis, environmental sciences and energy storage.¹³¹⁻¹³⁵ The exposed facet,^{136, 137} particle size,^{138, 139} and morphology¹⁴⁰ of nanomaterials are crucial to their properties. Controlling these structural features with conventional methods is challenging at nanoscale and usually requires rigorous conditions (high temperature), hazardous reagents, solvents, and surfactants, as well as long preparation times. Advantages of sonochemical synthesis are the short synthesis time, use of ambient conditions (room temperature) and environmentally friendly reactants, and a lack of template or surfactant, making it a “green chemical synthesis” of nanomaterials. One example of sonochemical synthesis is presented in Fig. S2d for the preparation of CuO nanoleaves.²³ The nanostructured CuO material was obtained after 5 minutes of sonication synthesis time at 25 °C, resulting in a highly crystalline and uniform 2D morphological structure. By contrast, conventional methods that utilise surfactants and high calcination temperatures (>400 °C) in a time-consuming and energy expensive synthesis result in a much lower quality material. The catalyst prepared via sonochemistry also exhibited much higher stability in the conversion of glycerol than the catalyst prepared through conventional methods.²³

Physical effects from cavitation events, such as microstreaming, high-speed microjets, and high-intensity shockwaves, contribute to enhanced heat and mass transfer during the synthesis of nanomaterials, resulting in fine control over the material’s morphology. In addition, due to an increase in collision probability caused by transient cavitation and the fast-cooling rate of sonochemical processes ($\sim 10^{10}$ K/s), the growth of large particles is inhibited, resulting in the formation of small and highly uniform particle sizes with large specific surface area and high porosity. Chemical effects of sonochemistry also help to control the structure of the synthesised nanomaterials. Primary sonochemistry activates the initial reagents as they are incorporated inside a cavitation bubble leading to the production of precursors for nanoparticle nucleation. Secondary sonochemistry releases a large quantity of active radicals into the bulk solution upon cavitation bubble implosion. These radicals attach to the nuclei of the nanoparticles, altering the surface energy and serving as structural template to control the exposed facet and morphology during the growth of nanomaterials. Key parameters that need to be carefully controlled in order to achieve the desired properties of the synthesised

materials are the ultrasonic frequency, power, sonication time, and ultrasonic activation mode. Usually, low frequency ultrasound (20-100 kHz) with a high intensity is employed in nanomaterials synthesis. Many materials, including metals, metal oxides, sulphides, alloys, composites, and amorphous materials, have been synthesised using this technique. This illustrates the power and great potential of sonochemistry as a process intensification technique in materials science. Although this approach is still not ready for large scale, it is expected to play an increasingly significant role in accelerating the production of next generation engineered materials.

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