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An activated fluid stream – New techniques for cold water cleaning

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ABSTRACT

Electrochemical, acoustic and imaging techniques are used to characterise surface cleaning with particular emphasis on the understanding of the key phenomena relevant to surface cleaning. A range of novel techniques designed to enhance and monitor the effective cleaning of a solid/liquid interface is presented. Among the techniques presented, mass transfer of material to a sensor embedded in a surface is demonstrated to be useful in the further exploration of ultrasonic cleaning of high aspect ratio micropores. In addition the effect of micropore size on the cleaning efficacy is demonstrated. The design and performance of a new cleaning system reliant on the activation of bubbles within a free flowing stream is presented. This device utilised acoustic activation of bubbles within the stream and at a variety of substrates. Finally, a controlled bubble swarm is generated in the stream using electrolysis, and its effect on both acoustic output and cleaning performance are compared to the case when no bubbles are added. This will demonstrate the active role that the electrochemically generated bubble swarm can have in extending the spatial zone over which cleaning is achieved.

1. Introduction

The cleaning of a material or an interface is at the centre of many processes which are important to health or to the production of high value commodities. This cleaning process should be fast, efficient (in terms of consumables and energy) and cause the least possible damage to the substrate while removing the target contaminate from the surface in question. While there are undoubtedly many possible methods to achieve these goals, in a variety of processes ultrasonic cleaning of an interface has been found to be useful [1–3]. In this technology the interaction of sound with materials and bubbles (which generates unusual physical and chemical conditions [4–8] within a fluid) has led to a rich set of exploited technologies and fascinating technical challenges. At the heart of ultrasonic cleaning is the interaction between sound and gas bubbles [9,10]. However, this interaction is complex [11] and the environments within which they occur can have intricate geometries [12]. Amongst the technological approaches used, the cleaning bath is perhaps the most well-known although other systems (for example 'megasonic fields' which have been explored with electrochemical probes [13,14]) are noteworthy. While the immersion of an object in a cleaning bath is undoubtedly effective in many examples, this approach has limitations [15]. For example the presence of areas of the bath which are active (so called 'hot spots') and areas which are inactive ('cold spots') could result in uneven treatment of a sample [16]. In order to characterise this spatial variation, cleaning activity can be mapped through the use of electrochemical [17–20], imaging [21–23] and acoustic measurements [24,25]. In addition local activity has also been correlated with cell death [26]. However, the location of cavitation hot spots will also depend on the immersion of an object into the bath which will also perturb the system [15,27]. Further restrictions are encountered through the spatial requirement for immersion of the sample within the bath. Clearly these limitations are associated with the 'bath' itself and could be avoided if this immersion approach was not employed. For example a liquid stream directed at the surface to be cleaned could be envisaged [15,28]. Here the cleaning action of bubbles excited with a suitable ultrasonic field should be generated at the end of a fluid stream. In addition low flow rates of fluid within this approach are useful in releasing the contaminant from the surface and avoiding re-deposition at another location (a further possible limitation in bath geometries). The low velocity stream approach has many advantages; however, two basic criteria are necessary for this strategy to be successful. First, the sound field must be sufficient to generate bubble activity at the solid/liquid interface of the material to be cleaned. Second, a suitable bubble population must also be present. This population can then be driven by the sound field deployed and act on the contaminant at the interface in question (through suitable oscillation

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[29–31] and shear forces [32] for example [30,31]). These two requirements are by no means trivial to create within a flowing stream [33,34]. However, such an approach has been adopted in an ultrasonically activated stream (UAS). In this case the device, which has been constructed to fulfil the requirements outlined above, can operate in an aqueous environment under ambient conditions without the need to add chemical additives to the media. While this simple approach has many advantages, there may be other circumstances when the naturally occurring bubble population is limiting. Under these circumstances an approach with an introduced appropriate bubble population (or bubble swarms) may aid the cleaning of an interface. One strategy for the generation of such conditions is the use of electrochemically generated bubble swarms. This approach is highlighted here, with the effect of the bubble population on the pressure field within the fluid stream and the cleaning of a fluorescent material from a large surface area structure reported.

2. Experimental

Micropores and large extended surfaces were chosen in this study for a number of reasons. First, micropores represent an occluded geometry where conventional fluid flow is particularly ineffective. In this environment the ability of acoustically excited gas bubbles is highlighted through the rapid decontamination of the pore in question. Second, the pore is well suited for the employment of electrochemical sensing approaches which enable some degree of quantification of the process. Third, the appropriate use of transparent media for the micropore electrode substrate allows for high-speed visualisation to be performed simultaneously with the electrochemical experiments in efforts to investigate the mechanistic details of the cleaning process. Lastly, extended surfaces (e.g. the fluorescent loaded tiles) are also pertinent as they illustrate the effect of the bubble population on surface cleaning over a large spatial domain.

Micropores where generated using an electrochemical etching approach [35]. Micropore experiments where performed as reported elsewhere [12] using a 23 kHz piston like emitter (or ultrasonic horn) immersed in an electrochemical cell and set 5 mm away from the surface of the electrode body containing the micropore. In these experiments a 0.1 M $Sr(NO_3)_2$ (Sigma–Aldrich, 99+%), 5 mM $K_3[Fe(CN)_6]$ (Alfa Aesar,99.97%) and F54 (dstl) emulsion was used. The contaminant was a polymer thickened methyl salicylate matrix (dstl).

A brief experimental protocol and description of the construction and operation of the electrochemically enhanced UAS (or e^2UAS) device is given here. The device is based on a rho-c matched cone or horn (matched to the acoustic impedance of water) attached to an ultrasonic transducer. Complete UAS devices (or StarStream systems) can be obtained from Ultrawave Ltd (F0030001). Note, the UAS concept (including the use of electrochemically generated bubbles swarms to enhance cleaning) was detailed in 2011 [28].

Bubble swarms, generated through controlled electrolysis within the UAS structure, were produced from 100 μm diameter Pt microwires (Advent research Materials) inserted so that they bisected the flow of liquid. These wires were used to electrolyse water by applying 24 Vdc for a variety of time periods (5–30 ms) in a controlled manner with respect to the sound field (which was also operated in a pulsed mode). The timing control of both the sound field and the electrochemical bubble swarm was achieved using a microprocessor interfaced to a PC through a RS232 connection and software written in-house (Visual Basic 6). The ultrasonic signal was generated by a gated (by the microprocessor) TTi2512a function generator (530 mV peak-to-peak amplitude) and an E&I power amp (240L). The solution consisted of 0.1 M Na_2SO_4 (Fisher lab reagent) and 2 mM sodium dodecyl sulphonate (Sigma 98%+) prepared under aerobic conditions. In the UAS experiments reported here a closed loop flow system was used where 1.5 dm^3 of the electrolyte media was pumped (up to 3 dm^3 min^{-1}) around the system using a small pump (Totton pumps NDP 14/2). Pressure measurements where made using a pressure sensor calibrated to a Bruel & Kjaer 8103 hydrophone in the frequency range used placed ~1 cm from the nozzle of the horn/cone structure of the UAS device. The data was recorded using a Handyscope HS3 (Tiepie Engineering) USB oscilloscope using an average of 32 pulses in each case. Images of the fluorescent particulate tracer (Wash & Glow UV Germ Fluid, Glowtec) were taken in the dark and illuminated with a UV lamp. Domestic ceramic tiles (~10 cm square, 5 mm thick) were used to assess the spatial extent of cleaning using the activated stream technology. These ceramic tiles were loaded with several (~3) drops, spread by a gloved hand and allowed to dry for 15 min before use. Each tile was treated with a UAS or e^2UAS for a period of 10–15 s. All chemicals were used as supplied.

3. Results and discussion

While many protocols and systems have been deployed to investigate the cleaning ability of ultrasonic fields, electrochemical technology has a number of major advantages [17,32,36,37,14]. Electrodes may be embedded into the substrate in question and in turn recessed to achieve a more complex representative substrate onto which cleaning experiments can be performed. Such an approach has been used to demonstrate the ability of ultrasound to remove an electrochemically inert matrix from a micropore structure [12]. Offin et al. showed that bulk fluid flow was ineffective in comparison to bubble activity captured in the micropore. Here we extend this approach and explore surface cleaning in an immersed electrode/sound source arrangement. However, the dimensions of the micropores deployed are further reduced while increasing the recess depth employed. This has the effect of increasing the aspect ratio of the pore and hence enables the exploration of the ability of activated gas bubbles to remove material from these structures. Fig. 1(a) and (b) shows two schematic representations of the micropore and acoustically excited bubbles that result in pore cleaning. Note that bubbles, which in the absence of a suitable sound field are inactive, can if excited have oscillating ripples (Faraday waves) induced on their surfaces and this has been shown to generate local shear [31,32] and convection (microstreaming) [38–40]. Such acoustically excited bubbles enter the outer layers of the inert liquid and start to remove material [12]. Fig. 1(a) shows a representation of a micropore filled with an electrochemically inert matrix (here tMS, coloured in blue in Fig. 1, ■). In this case the electrochemically active molecule present in the bulk (here 'A' or $[Fe(CN)_6]^{3-}$) of the fluid is unable to reach the electrode surface. Under these conditions, even though the electrode was held under mass transfer limiting conditions for the redox probe employed, the electrode is unable to reduce compound 'A' and no current will be observed ($i = 0$). However, the presence of oscillating gas bubbles, driven by the acoustic field employed, results in penetration of the outer tMS layer and removal of material (as indicated by the arrows) from the micropore. This has been confirmed by high-speed imaging of micropore structures in combination to the electrochemical measurements [12]. Fig. 1(b) shows a schematic representation of the pore after the active gas bubbles have removed the inert tMS from the pore. Under these circumstances the electrode is now able to electrochemically reduce compound 'A' to 'A^-' under mass transfer limiting conditions. Hence an electrochemical current will only be observed as the tMS is removed from the system allowing