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Ultrasound assisted liquid–liquid extraction in microchannels—A direct contact method

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ABSTRACT

A new method to apply ultrasound to a microchannel for liquid–liquid extraction was explored. The microchannel tubes are subjected to the ultrasound by direct contact with the transducer without the presence of a liquid medium. The design was constructed with the objectives of reproducibility, proper control of the ultrasound parameters and visibility of the behaviour of the two phase flow under the influence of ultrasound throughout the length of the channel. Two mechanisms of emulsion formation were observed. The effectiveness of the system under the influence of various operating and design parameters was quantified by calculating the yields of the two phase hydrolysis reaction of *p*-nitrophenyl acetate. The behaviour under various frequencies and amplitude was explored. At a frequency of 20.3 kHz, amplitude of 840 mV and flow rate of 0.1 ml/min the highest increase in yield was observed, which was almost 2.5 times that of the silent condition. A comparison was also made against silent batch and flow conditions to determine the actual effectiveness of the system. To obtain an identical yield of 75% the required residence time could be reduced by a factor of 20 in the sonicated flow condition compared to the silent batch condition.

1. Introduction

Liquid–liquid extraction is a common and important unit operation in any chemical industry. There have been many innovative technologies developed to carry out this process in the most efficient manner. A recent development is the use of microreactors. There is an increasing interest in microreactors because of their advantages [1–4] of large interfacial area per unit volume, shorter diffusion lengths, increased mixing effects by the formation of internal circulations and enhanced heat transfer. When two immiscible liquids are admitted into a microchannel, different flow patterns are generated depending on the fluid properties and operating conditions [5]. In certain situations the advantage of the increased interfacial area and mixing effects may not be sufficient enough to balance the short residence time. Many methods have been developed to increase the mixing effects in a microreactor which are broadly classified as active and passive ones [6]. Ultrasound is an example of the active approach, providing a non-contact method of mixing [7] within the very small structures of the microreactor. The advantages of using ultrasound in chemical synthesis and in microstructured devices for multiphase flow have been reported in literature [8–13].

Ultrasounds are sounds waves with frequencies larger than 20 kHz. When these sound waves are applied to a liquid medium cavities are formed which in turn cause many chemical and physical effects such as acoustic streaming, jet formation and local hot spot generation [13]. In this research, the physical effects are pursued, mainly the improved mixing by the vibration of the bubbles created by cavitation and the shockwaves created by implosion of the cavitation bubbles [14]. When two immiscible phases are subjected to ultrasound an emulsion of the two phases is created [10,11,15]. Hubner et al. report the formation of vibrating bubble clusters in the dispersed phase which disturb the interface by creating jets of the dispersed phase to move into the continuous phase, thus causing an emulsion formation [10]. This experiment was done in a glass capillary placed on a sonotrode, wherein the continuous phase was the aqueous phase and the organic phase was dispersed. In this paper it is further explored whether this mechanism persists in the reverse flow condition of the [10,11] aqueous phase being the dispersed phase and the organic phase being the continuous one by using a hydrophobic plastic tubing.

There are various designs available for coupling microstructured devices with ultrasound. The common approach is the utilization of the ultrasound bath [8,9] to achieve good transfer of

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Nomenclature

Latin

a	Interfacial area per unit volume m^2/m^3
C	Interfacial area per unit volume mol/m^3
C_{aq}	Reactants' concentration in the aqueous phase mol/m^3
C_{org}	Reactants' concentration in the organic phase mol/m^3
K_l	Mass transfer coefficient m/s

Greek

α	Fraction of the reactant consumed –
τ	Residence time s

the ultrasound vibrations. In ultrasound baths the area of maximum intensity is determined by various techniques like hydrophones and calorimetric tests. Knowing this area the microstructure is then suspended in these defined locations and the ultrasound is applied. There are several disadvantages associated with the ultrasound bath concept. Firstly, there is always loss of ultrasound intensity in the liquid transfer medium (i.e. the water bath), for which Hübner et al. [10] made use of pressure to minimize the cavitational activity in the transfer medium. Secondly, as each ultrasound bath behaves differently with respect to the regions of high ultrasound intensity, this also creates difficulties for reproducibility and comparability between different reactors reported in literature. Thirdly, since all these reactors have the microstructure suspended in the liquid medium, it is difficult to observe what exactly happens to the two phase immiscible systems when ultrasound is applied. Finally, the

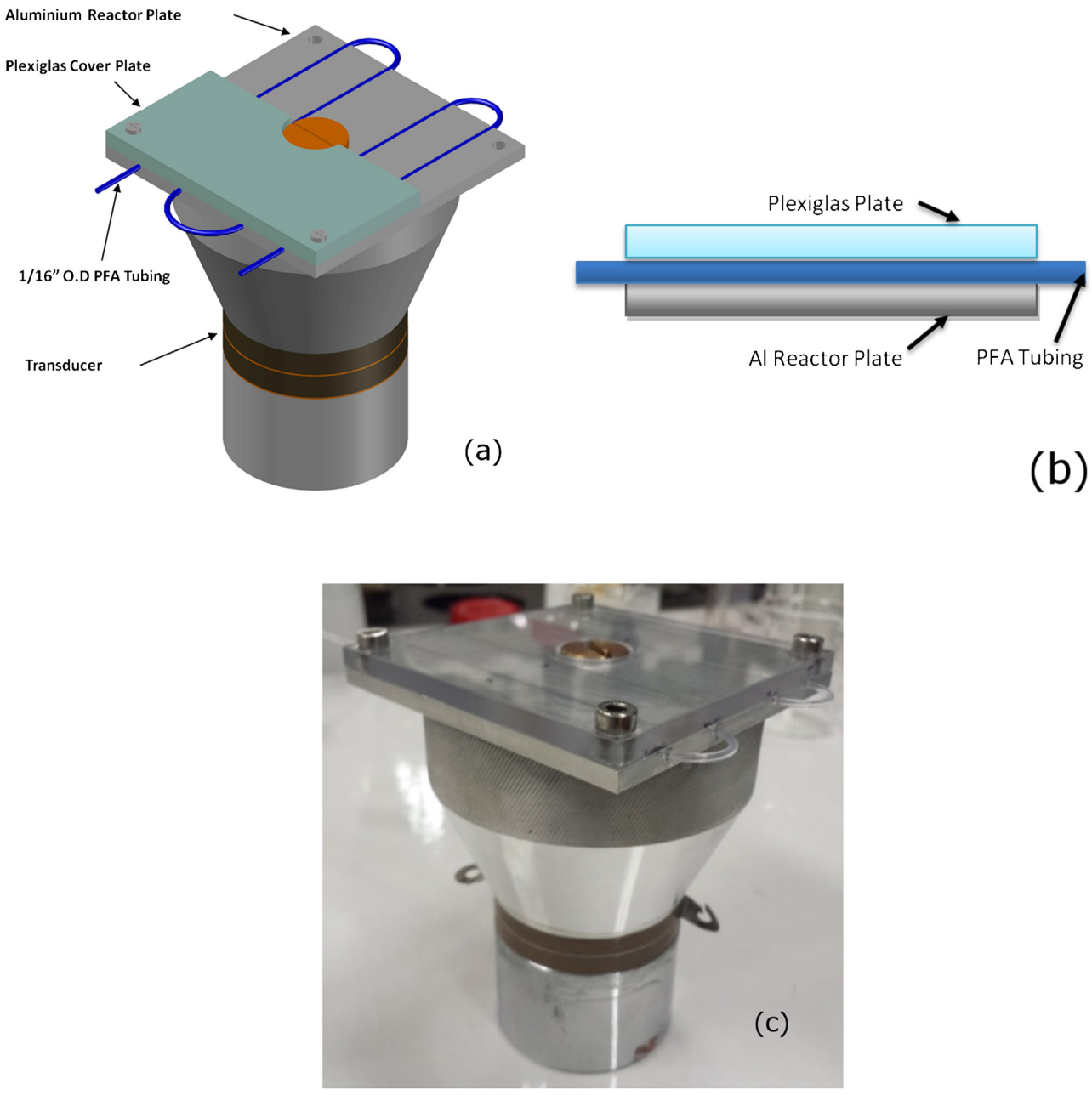

Fig. 1. (a) Schematic representation of the reactor, (b) side section of single channel, (c) complete reactor.