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System modelling and device development for passive acoustic monitoring of a particulate-liquid process

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ABSTRACT

This paper presents the development of a passive ultrasonic monitoring system for the detection of acoustic emission (AE) created by chemical particles striking the inner wall of a reactor vessel. The finite element (FE) code PZFlex was used to analyze the complex interactions between chemical particles and the vessel wall. A 4-layer 2D model was developed comprising a liquid load medium and a glass-oil-glass combination corresponding to the jacketed vessel reactor. The model has been experimentally validated with excellent correlation achieved. The excitation function was derived from Hertz's theory and used as the model stimulus corresponding to particles striking the inner glass wall. Analysis of the FE simulations provided the transducer specifications for a passive ultrasonic monitoring system. The system comprises two transducers with complementary characteristics: narrow bandwidth/high sensitivity; wideband/low sensitivity. Importantly, the sensitivity of the resonant transducer provides discrimination of particle concentration. Moreover, the broader bandwidth of the off-resonant device demonstrates potential for *in situ* estimation of particle size. The performance afforded by this approach has considerable potential for real-time process monitoring in the chemicals and pharmaceutical industries.

1. Introduction

Acoustic monitoring techniques offer a significant advantage over optical techniques such as near infrared and Raman spectrometries for process monitoring in that they can be applied to samples that are optically opaque without the need for any sample preparation. In particular, the ability of acoustic waves to penetrate optically opaque media such as stainless steel enables acoustic techniques to be configured to operate in a non-invasive mode of operation, *e.g.* by sensor attachment or location of a microphone close to the outer vessel wall, without the need for incorporation of a window in the vessel. While there are only a few reports of the use of non-invasive active acoustic techniques for *in situ* process monitoring [1], passive acoustics has been used more widely particularly for the non-invasive monitoring of particulate processes. Passive acoustic monitoring techniques use the acoustic emission (AE) generated by collisions of particles primarily with the inner surface of vessel or pipe walls [2], although collisions with any internal structures or between particles may also contribute, to determine information related to the status of the process. Measurement of AE has been used successfully to monitor, for example, high-shear granulation [3–6], powder blending [7], drying [8] and various fluidized bed processes [9–15], heterogeneous reactions [16–20], and the transport of powders [21,22], tablets [23] and slurries [24].

A number of experimental and theoretical studies have been published to understand how different factors affect the AE signal generated. In a series of papers, Leach et al. [25–30] investigated AE generated by collisions between spherical, cylindrical and irregular-shaped particles in a rotating vessel. A condenser microphone was used to detect the AE generated by the collisions, with the frequency of emission inversely related to particle size. The same relationship was also observed for the collision of two steel balls [31], and for collisions between glass spheres [32,33] and sediment gravel in water [34]. In addition, it was determined that the amplitude of AE increased with the number of colliding spheres, but that the frequency of AE was unaffected [32]. AE generated by the impact of objects with surfaces has been investigated using a microphone or a transducer attached to the surface of the impacted

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Table 1
Particle information used in both the experimental and simulation investigations.

Mnemonic	Experimental distribution (μm)	Simulated (μm)
Size α	$0 < x < 251$	200
Size β	$251 < x < 500$	400
Size γ	$500 < x < 853$	600

structure. The amplitude of AE arising from collisions between a steel plate and a ball bearing increased as the number of impacting objects was increased, but as for collisions between spheres, the frequency of AE did not change [35]. During the pneumatic conveyance of coal, the maximum frequency of the vibrational modes of the structure excited on impact was found to decrease with an increase in particle size [36,37]. In studies of the mixing of dry powders [7,38] and particles in a liquid [17], the AE signal increased with an increase in the mass and size of particles, while the portion of AE at lower frequencies increased with particle size.

In previous experimental work [16,17], AE generated by the impact of particles in a fluid with the internal wall of a 1 L jacketed glass reactor was studied. More recently, mathematical models were derived to describe the AE generated by particle impacts with the vessel wall [39–41]. The vessel wall was modelled as a single layer circular plate and analytical expressions were derived to describe the impact of particles with the plate. While it was possible to obtain particle size [39,40] and particle concentration [41] information, it becomes increasingly difficult to derive mathematical expressions, which can be solved analytically, describing the propagation of acoustic waves for more complex geometries and material properties. In such cases, it has been shown that a numerical method like finite element (FE) modelling can be used [42,43]. Therefore, in this work, the generation of AE from the impact of particles with a reactor vessel wall for the experimental set-up described in references [16,17] was investigated using FE modelling. A FE model of the experimental set-up comprising a liquid load medium and the glass-oil-glass combination corresponding to the reactor vessel wall structure was developed using PZFlex (Weidlinger Associates Inc., New York, USA). The FE model was used to investigate particle size and concentration characteristics through analysis of the frequency spectra associated with the generated AE from the particle–wall collisions. The results of the investigation have been used to define the ultrasonic transducer system specification for a new passive acoustic monitoring approach, through which a pair of ultrasonic transducers have been designed and fabricated. Interestingly, the complementary characteristics of these transducers can provide additional particulate information from the heterogeneous system under investigation.

2. Experimental pilot study

The experimental set-up that was modelled in the present study is described in detail in references [16,17], with the main findings summarized here. The experimental apparatus employed consisted of: a 1 L glass reactor (VWR International, Dorset, UK) with an oil jacket, which was connected to a heater–chiller unit for temperature regulation; a glass stirrer rod and paddle connected to an overhead stirrer motor; a Nano30 AE sensor (Physical Acoustics Limited, Cambridge, UK) attached to the outer wall of the reactor vessel; and a PC for data acquisition and processing.

Broadband AE signals were collected of itaconic acid particles (Sigma Aldrich, Dorset, UK) mixing in 500 mL of toluene (Bamford Laboratories, Rochdale, UK) at 20 °C. To generate different particle size ranges, the itaconic acid was sieved into three fractions (see Table 1). To investigate the effects of particle size and concentration on the broadband AE signals, each particle size range of itaconic acid was added stepwise, up to 200 g, to toluene.

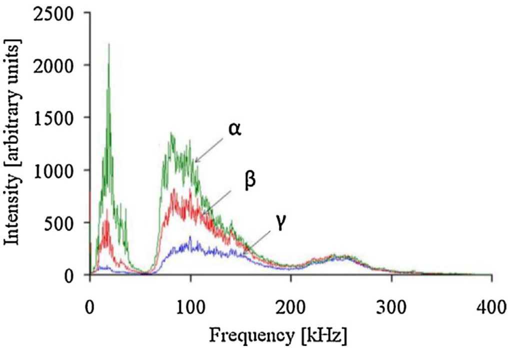

Fig. 1. Measured AE power spectra of 40 g dm^{-3} of itaconic acid in toluene with particle sizes α, β and γ.

Examples of AE spectra of different particle sizes, α, β and γ, of 40 g dm^{-3} of itaconic acid in toluene are illustrated in Fig. 1. The principal frequencies of interest lay in the 0–350 kHz range, with components above 200 kHz demonstrating less sensitivity to changes in particle sizes to those at lower frequencies.

A variety of data analysis methods were employed to determine both particle size and concentration information. Analyzing the signal area under each AE spectrum, as illustrated in Fig. 1, demonstrated the best opportunity for particle concentration and sizing information. Particle size information was extracted by considering the ratio of signal area between 55 and 200 kHz, where there is clear discrimination between the different particle size ranges, to the overall signal area between 55 and 500 kHz. Full details of the results obtained are given in reference [17].

To characterize the frequency response of different components of the experimental equipment (the jacketed reactor vessel, the Nano30 (Physical Acoustics) transducer and the preamplifier), the inner face of the vessel was excited using a 'pencil lead break' (Hsu-Nielson source) [44] to simulate impulse excitation of the system. Fig. 2 shows the measured frequency spectrum of the system impulse response. On comparing the experimental AE spectra (Fig. 1) with this measured impulse response, the similarity is clearly evident. Therefore, it can be concluded that the acoustic AE signals observed are modified by the frequency transfer function of the transducer, and by the filtering effects of the reactor vessel material and electronic devices.

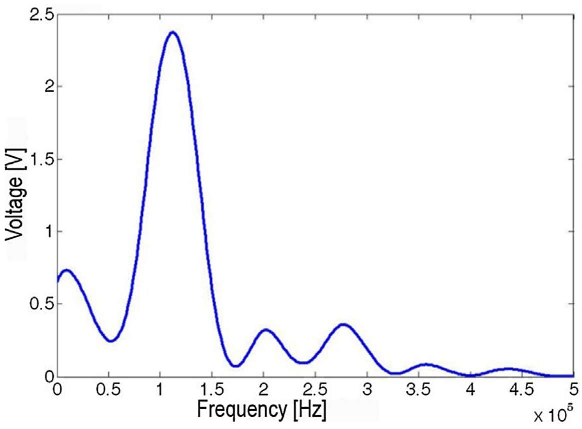

Fig. 2. Experimentally measured spectral profile of the impulse response of the reactor, transducer and preamplifier.