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The quartz crystal microbalance as a microviscometer for improved rehabilitation therapy of dysphagic patients

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Abstract— **The viscosity of non-solid foods, and the stability of their viscosity over time, temperature change and shearing is critical in managing dysphagia. The starch-based foodstuffs thickeners used in dysphagia therapy are highly non-Newtonian and their viscosity is dependent on shear rate, shear history, temperature and time. This inherently results in the subjective measurement of viscosity and hence the management of dysphagia to be error prone. Given the ageing population, this problem is likely to become more widespread. This paper identifies the need for an objective measurement device rather than the current subjective methods. The use of the quartz crystal microbalance (QCM) as a viscometer for the starch-based thickeners is reported.**

I. INTRODUCTION

Dysphagia is a common, but relatively short-lived sequelae of a cerebrovascular accident and can occur on a chronic basis in many neuro-degenerative diseases. Dysphagia that is present immediately following a stroke is found to resolve in half of all patients. The small group of patients for whom dysphagia persists for more than three weeks generally have a less favourable outcome [1]. The condition occurs in as many as 60% of stroke patients [2] and can lead to dehydration, malnutrition and, most notably, pulmonary aspiration [3], [4]. Aspiration aids the transport of pathogenic bacteria into the lungs and can lead to aspiration pneumonia [5], [6]. Numerous respiratory problems have been related to aspiration [7]. Dysphagic stroke patients have difficulty swallowing fluids and often have their fluids thickened with a starch-based thickener as part of prescribed therapy.

On a clinical basis the thickened fluid's viscosity is often judged subjectively and monitored using terms such as *syrup* or *honey*. It is vital that drinks of the correct viscosity are prepared consistently, as too low a viscosity may travel faster into the pharynx and be more likely to enter the airway before protective mechanisms of the swallow can be initiated. Too high a viscosity is rejected by patients and can lead to malnutrition and dehydration. For some patients a high viscosity and hence increased transit time increases the likelihood of aspiration. Miller and Watkin [8] and Goulding and Bakheit [9] illustrated that a high viscosity fluid can aggravate swallowing and may worsen dysphagia. Therefore it is clear that the viscosity must be precise, as too low viscosity causes harm to health and too high viscosity is rejected and in some cases also causes harm. These problems are exacerbated by the complexity of the viscosity of the starch fluids. We have recently illustrated that the starch-based foodstuffs thickeners used in dysphagia therapy are highly non-Newtonian and their viscosity is dependent on shear rate, shear history, and time [10].

It has been shown using an electromyograph (EMG), manometry and videofluoroscopy techniques [11]-[14] that fluids with increased viscosity have increased transit times during the swallow due to the increased oropharyngeal muscle activity. Higher viscosity fluids allow more time for the patient to trigger the swallowing reflex [15]. Therefore during a swallowing analysis, such as a videofluoroscopy, the correct viscosity of any liquids consumed by a patient can be determined. However, since it is impractical and expensive to use a viscometer or rheometer at the bedside, the fluids can only be judged subjectively by the carer or patient. The expensive instrumentation and skilled therapists using the videofluoroscopy or EMG are partly redundant since the correct viscosity determined by these techniques cannot be quantified to the desired precision at the bedside.

Viscosity measurements are generally taken using capillary viscometers, falling-ball viscometers, rotational viscometers or oscillatory rheometers. These often involve time consuming and expensive processes and are clearly not practical at the bedside. The QCM has been used to probe the viscous properties of fluids in many applications [16]-[23]. The QCM is a viable cheap and practical alternative to a benchtop viscometer and it has a less disturbed and more linear response when compared to conventional viscometers [24]. Recently we have illustrated the practicality of the QCM, in that it can be used numerous times with gentle cleaning when used with starch thickeners [25].

Initially quartz crystals were used as sensitive deposition monitors, after Sauerbrey [26] derived a relationship between the mass applied (foreign layers deposited on crystal) and the consequent change in resonant frequency.

$$\Delta f = \frac{-2f_0\Delta m}{A\sqrt{\rho_q \mu_q}} \tag{1}$$

where Δf is the change in frequency, f_0 is the fundamental resonant frequency of the crystal, Δm the mass change, A the piezoelectrically active area, ρ_q the density of quartz, and μ_q the shear modulus.

Recently the QCM has been used to probe the viscous properties of liquids. Kanazawa and Gordon [27] established a relationship between Δf and kinematic viscosity based on a physical model which couples the shear wave in the

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quartz to the damped shear wave in the fluid:

$$\Delta f = -{f_0}^{3/2}\sqrt{\frac{\eta_l \rho_l}{\pi \mu_q \rho_q}} \tag{2}$$

where Δf is the fundamental frequency (of the dry crystal), η_l and ρ_l are the absolute viscosity and density of the liquid respectively.

Ward and Buttry [28] indicated that the current QCM formats are not adequate for commercial use. Our work establishes a novel fluid application technique and aims to examine some of the practical aspects of using the QCM [25]. It is our aim to increase its potential as a commercial medical viscometer, not only specifically for dysphagia therapy but as a general medical aid.

II. EXPERIMENTAL

The aim of this work was to examine the QCM response for the starch thickeners used in dysphagia therapy and to determine any problems associated with measurement. We have previously only observed changes in resonant frequency. This work includes changes in Q. Ideally this work will highlight issues regarding bringing the QCM out of the laboratory and into hospitals as a reliable medical instrument. The experiments used a starch (modified maize) thickener that is popular in health care, both in its current form and post gelatinisation[1]. For each sample application a measurement of the change in Q (ΔQ) and resonant frequency (Δf) was recorded for both the fundamental and third harmonic.

A. Solution preparation

Each solution was made by adding 0.5, 1, 1.5, 2, 2.5 or 3 grammes ($\pm$0.0005g) of a commercial thickener based solely on modified maize starch, to 50ml ($\pm$0.08ml) of deionised water. Throughout this paper, 'concentration' refers to the number of grammes in 50ml of water. All solutions were made by mixing the powdered starch into the water for 30 s at an approximate shear rate of 50 s^{-1} at room temperature (24-24.5°C). The solutions were allowed to stand for 5 min before being stirred at a similar shear rate for 30 s. The sample was allowed to stand for a further 2 min before being stirred again and applied to the QCM. For the gelatinisation experiments the samples were made in a similar way, after being heated to 90°C for 10 min.

Leaving a starch-based fluid to stand allows the formation of a gel network [29], [30], therefore the gel was agitated to breakdown the structure and return the fluid to a sol by gentle swirling before each droplet application.

B. Sample application

Quartz crystals of resonant frequency 10 MHz (supplied by C-MAC, Somerset, UK), were driven and monitored by a Hewlett-Packard 8753E network analyser. The crystals degree of polish was characterised by C-MAC by the amount of material removed. The crystals used in these experiments were polished to the degree of 2μm removed. A more rigorous characterisation for the surface roughness was obtained by the use of an Atomic Force Microscope (AFM) in the Physics department at Lancaster and was found to be 27$\pm$5nm RMS. The application of the droplet to the QCM surface can be seen in figure 1. Approximately 0.5mm^3 of the sample was applied to one surface of the crystal using a pipette. This produced a sessile droplet covering the entire electrode but not extending to the edges where shorting of the electrodes could occur through conductance of the sample. The same side of the crystal was used throughout the experiments. This technique is unique to Lancaster and allows only small amounts of sample to be used [31].

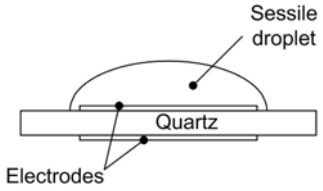

Fig. 1. Schematic of sample application to QCM.

C. Cleaning procedure and measurements

The resonant frequency for the fundamental and third harmonic was recorded before application of the sample (unloaded data). Δf was recorded once the sample was applied for both harmonics (loaded data). In addition the Q was recorded before and during droplet application for both harmonics. Cleaning of the crystal was performed by holding the QCM in a stream of 60-70^{o}C tap water for 30 s. Excess water was shaken off and the crystal was placed in a stream of hot air ($\approx$60^{o}C) for 30 s to speed up evaporation. The crystal was connected to the network analyser and allowed to equilibrate for 10 min before the next sample was applied. We have previously verified the efficacy of this technique [25].

III. RESULTS

A. First Harmonic

Figure 2(a) illustrates the change in Q at the fundamental frequency when the crystal is loaded by varying concentrations of starch thickeners. The error bars represent one standard deviation from the mean. Although there does seem to be a small linear decrease in ΔQ as concentration increases, it is clear from the error bars alone that the samples do not significantly differ and a Analysis of Variance (ANOVA) test is not necessary. The gelatinised starch results (Figure 5(a)) however suggest that there may be a general linear decrease in ΔQ. Figure 3(a) shows how the QCM fundamental frequency changes with increasing starch concentration. Since the error bars overlap less than figure 2(a), it is necessary to perform an ANOVA multiple range test

[1]Gelatinisation is the process that happens to starch fluids when heated. The heat causes the hydrogen bonds to break and the starch granules to swell. When the kinetic energy of the water molecules becomes sufficient to overcome the attraction between the hydrogen-bonded starch molecules within the granule, water molecules can penetrate the starch granules causing the granules to swell.

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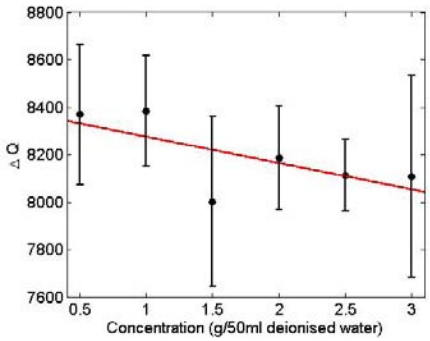
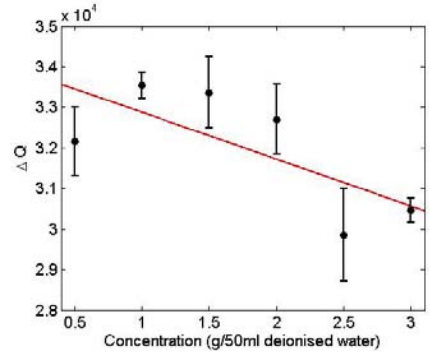
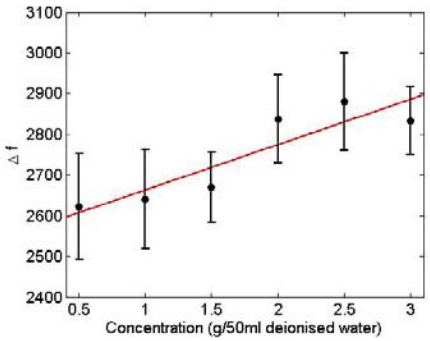


(a) Fundamental frequency

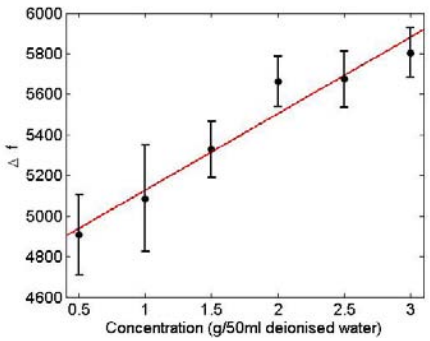


(b) Third Harmonic

Fig. 2. Graph illustrating the change in Q of an unloaded oscillating crystal when it is loaded with varying concentrations of starch solutions for the first and third harmonic. Linear models are fitted using least squares fit method.



(a) Fundamental frequency



(b) Third harmonic

Fig. 3. Graph illustrating the change in resonant frequency of an unloaded oscillating crystal when it is loaded with varying concentrations of starch solutions for the first and third harmonic.

TABLE I

MULTIPLE RANGE TEST TO DETERMINE WHICH MEANS SIGNIFICANTLY DIFFER FOR Δf FOR THE FUNDAMENTAL AND THIRD HARMONIC FREQUENCY. MEANS FOLLOWED BY THE SAME LETTER DO NOT SIGNIFICANTLY DIFFER FROM ONE ANOTHER (P=0.05).

	Mean (Hz)	
Concentration	First harmonic	Third harmonic
0.5	2621 a	4908 a
1.0	2641 a	5087 b
1.5	2669 b	5327 c
2.0	2837 c	5664 d
2.5	2880 c	5675 d
3.0	2833 c	5804 d

since the ANOVA test indicated significant differences. Table I indicates which samples are significantly different using the this test. The gelatinisation results shown in figure 4(a) for the first harmonic are less precise and ANOVA indicates that non of the measurements significantly differ from another.

B. Third Harmonic

Figure 2(b) illustrates the change in Q at the third harmonic when the crystal is loaded by varying concentrations of starch thickener. The response appears to be non linear yet more variable than the first harmonic equivalent. The gelatinisation results follow a similar pattern (figure 5(b)). This suggests that Q is less linear and appropriate as a measurement at harmonics above the first.

Figure 3(b) illustrates that the third harmonic Δf is more sensitive to changes in concentration and gives a more linear response than the fundamental frequency. Table I indicates which samples are significantly different using the ANOVA multiple range test and indicates that more of the samples are significantly different than the fundamental frequency equivalent. The gelatinisation multiple range test results indicate that only the 0.5g sample is significantly different and the remaining samples are not.

Figure 6 shows the QCM's viscosity response and illustrates that although there is a consistent linear relationship between concentration and viscosity, it is far from the actual viscosity values for the starch fluids.

IV. DISCUSSION

Figures 2(a), 3(a), 2(b) and 3(b) indicate that there is a small measurable response when the QCM is used to detect the viscosity of starch samples. The figures also show that the third harmonic is more sensitive to changes in the starch concentration. Figure 6 suggests that the QCM response is too small and wholly inaccurate when compared with actual viscosity values of the starch solutions. This is surprising since our prior-art has demonstrated good correspondence with the viscosity of continuous phase fluids [21], [22].

A possible explanation for this is that starch is insoluble in water at room temperature. Starch granules are semi-

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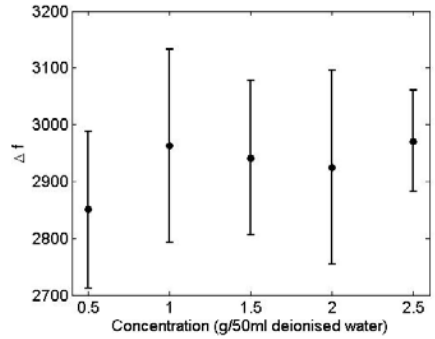
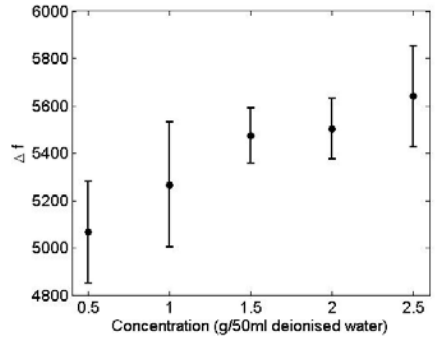
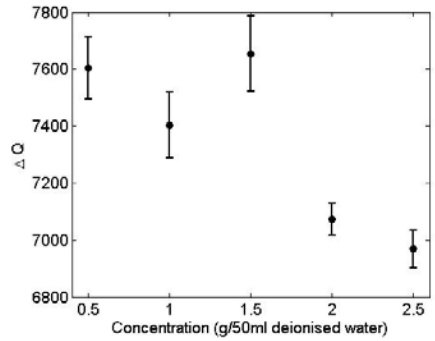


(a) Fundamental frequency

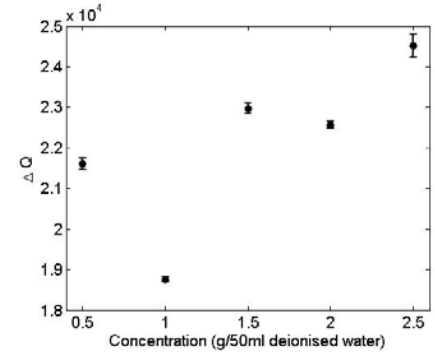


(b) Third Harmonic

Fig. 4. Graph illustrating the change in frequency of an unloaded oscillating crystal when it is loaded with varying concentrations of pre-gelatinised starch solutions for the first and third harmonic.



(a) Fundamental frequency



(b) Third harmonic

Fig. 5. Graph illustrating the change in Q of an unloaded oscillating crystal when it is loaded with varying concentrations of pre-gelatinised starch solutions for the first and third harmonic.

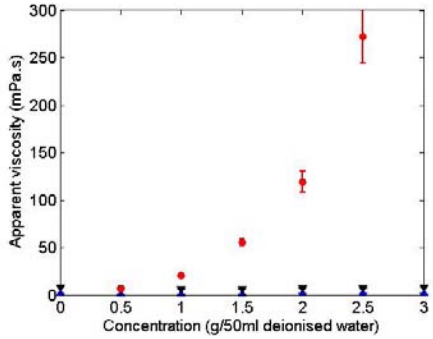


Fig. 6. Graph illustrating the QCM viscosity response, obtained using the equation derived by Kanazawa and Gordon [27]. The triangles represent the fundamental frequency, the inverted triangles represent the third harmonic and the circles are the actual viscosity values for the starch fluids at 20°C and a shear rate of 30^{-1}.

crystalline particles composed of a mixture of two polysaccharides, amylose and amylopectin. These two polysaccharides associate via hydrogen bonds either directly or through hydrate bridges, forming semi-crystalline micelles that exhibit birefringence and consequently yield amorphous areas [32]. These semi-crystalline regions are insoluble and are composed mainly of short chains of branched amylopectin arranged in a three-dimensional lattice through a double-helix formation [33]. The amorphous regions are composed of amylose molecules in a single-stranded helix formation. These regions undergo water imbibition [33]. Therefore, a starch gel can be regarded as a composite material, in which the continuous phase of the amylose gel matrix has swollen gelatinized granules made up of amylopectin distributed within it [34], [35]. The rheological properties of the continuous phase and the deformability of the dispersed phase, and the interactions between these two phases are associated with the mechanical properties of starch gels [34].

The QCM is possibly only responding to the continuous phase and is unaffected by the starch grains in the dispersed phase. Therefore since maize has a large constituent of insoluble amylopectin, the increasing starch concentration is having a minimal effect on the QCM response. Gelatinised starch should then however cause a vast increase in accuracy and precision. Given the gelatinised starch results, this is not the case. The gelatinised starch results actually appear less precise and more erroreous than the non-gelatinised equivalent.

V. CONCLUSIONS AND FUTURE WORKS

A. Conclusions

We have highlighted the need and possible solution for a much reputed problem within dysphagia therapy. Given the ageing population, this problem is likely to become more evident and a solution is sorely needed. This work currently suggests that the viscosity of normal maize starch solutions is not able to be accurately quantified using the QCM. Further experiments have shown that gelatinisation will not improve the QCM response. The results suggest a trend, that a high

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fundamental frequency QCM (≈15-20MHz) driven at the third or fifth harmonic may have sufficient sensitivity to accurately characterise the viscosity of starch fluids used in dysphagia therapy using Δf. The converse is true for Q. ΔQ becomes less consistent and accurate at higher harmonics. Use of an accurate microviscometer is likely to improve therapy as the inevitable ambiguity of subjective analysis can be avoided.

B. Future Works

There is an obvious need for a cheap practical viscometer throughout the health care profession, ranging from dysphagia therapy where the fluids to be consumed are measured to the measurement of bodily fluids such as various blood analyses. Such complex non-Newtonian fluids require a sensitive device to determine viscosity. A high frequency QCM driven at a high harmonic may meet the criteria and enable a portable microviscometer to be used throughout the healthcare profession.

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