

A Simple Acoustic Technique To Assess The Setting Time Of Antibiotic Loaded Calcium Sulphate

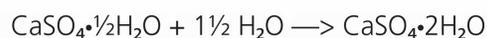
P. A. Laycock¹, M. J. Brayford¹, J. J Cooper¹

¹ Product Development, Biocomposites Ltd, Keele, Staffordshire, UK

Presented at eCM XII: Implant Infection, 22nd - 24th June, 2011, Congress Center, Davos, Switzerland

Introduction

Calcium sulphate hemi-hydrate when mixed with water results in the formation of a solid precipitated calcium sulphate dihydrate (gypsum):



Many factors affect the timing of this reaction¹, hence the need to quantify set times.

Current methods for determining the set times of calcium based bone cements include visual examination, ultrasound² or Vicat³ and Gillmore⁴ needle apparatus, however Vicat and Gillmore needle methods can be 'arbitrary' as they depend upon needle diameter and weight used and both give differing results. This work developed a new simple acoustic technique to measure the setting times of a calcium sulphate bone cement loaded with different antibiotic mixes.

Methods

A 100% pure, synthetic, calcium sulphate (Stimulan®, Biocomposites Ltd) in the form of a 'rapid cure' 10cc kit was mixed with 6ml of 4 different antibiotic mixing solutions. The solutions used were Tobramycin 80mg/2ml (Hospira, Inc.), Gentamicin 80mg/2ml (Hospira, Inc.), Tobramycin 80mg/2ml with 0.5g Vancomycin Hydrochloride (Hospira, Inc.) and Gentamicin 80mg/2ml with 0.5g Vancomycin Hydrochloride. A digital stopwatch was started as soon as the mixing solution was introduced into the Stimulan powder, which was then mixed for 30 seconds, rolled into a ball and allowed to cure undisturbed. Each mix was repeated for 3 experiments.

A new method was developed to quantify the setting times specifically of small volumes of synthetic bone cements. The method adopts an acoustic technique. A microphone connected to the sound card of a PC is used to pick up the sound the paste makes when dropped from a fixed height (30mm) onto a thin metal plate (Figure 5). As the paste hardens the frequency rises and the sound changes from a dull 'thud' to a higher pitch note until it reaches a similar frequency obtained with a fully set paste (24 hrs). The software used to analyse the frequencies was TrueRTA™ (True Audio) and the paste was said to be set when the 2 and 5 KHz frequency bars achieved a level of >5 dBu amplitude (Figures 1-4).

Results

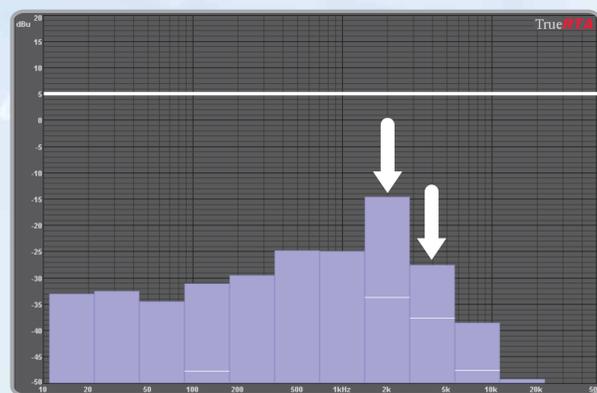


Figure 1. Frequency profile of a paste prior to setting

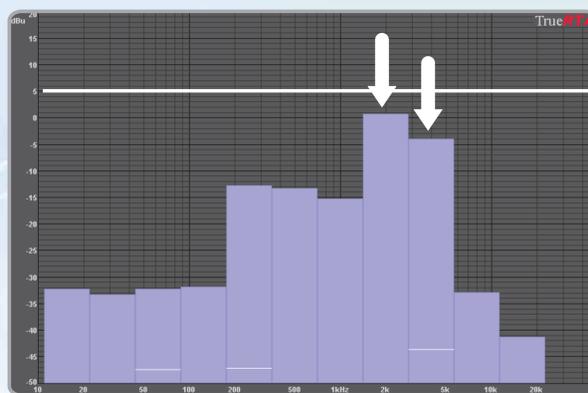


Figure 2. Frequency profile of a paste nearly set

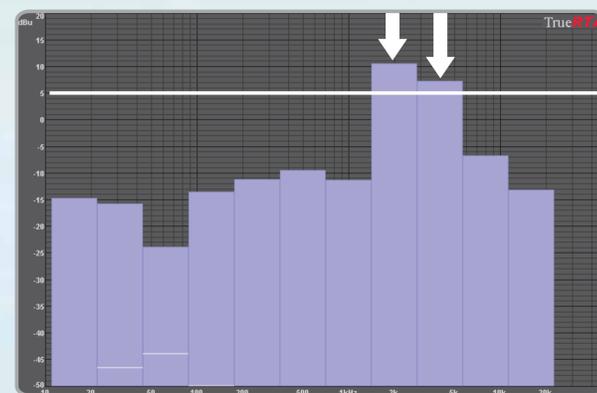


Figure 3. Frequency profile of a paste immediately on setting

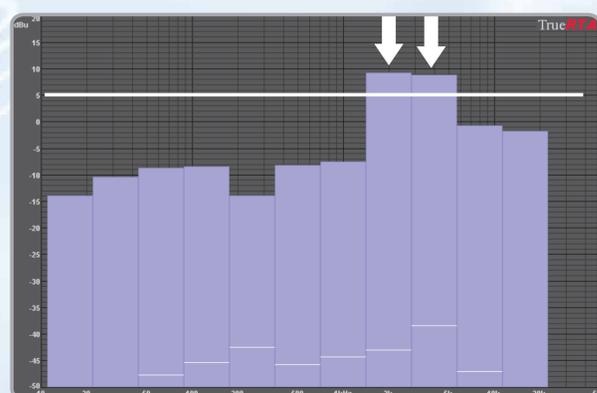


Figure 4. Frequency profile of a paste 24 hours after setting



Figure 5. Apparatus used containing microphone, plate and sample drop height guide

| Stimulan + | S1 | S2 | S3 |
|------------|-------|------|-------|
| Tobramycin | 10:25 | 9:58 | 10:09 |
| Gentamicin | 4:04 | 3:47 | 3:50 |
| Tob/Vanc | 9:09 | 8:45 | 9:01 |
| Gent/Vanc | 3:39 | 3:45 | 3:38 |

Table 1. Setting times (Minutes:Seconds) of Stimulan mixes (n=3)

Discussion & Conclusions

A new simple acoustic test has been developed and used to assess the effect of different antibiotic additions on the setting time of a 'rapid cure' calcium sulphate bone cement (Stimulan). This technique gives an 'absolute' result as the frequency is comparable to a fully set cement. The test gives reproducible and consistent results and can be used with very small quantities of cement (consistent results have been achieved with 0.5cc up to 10cc). Further investigations could be undertaken to correlate this method to other techniques such as the Vicat and Gillmore needles.

References

- [1] Singh N. B. and Middendorf B., Progress in Crystal Growth and Characterization of Materials, Volume 53, Issue 1, March 2007, Pages 57-77, Calcium sulphate hemihydrate hydration leading to gypsum crystallization.
- [2] Carlson J., et al. Biomaterials, Volume 24, Issue 1, January 2003, Pages 71-77, An ultrasonic pulse-echo technique for monitoring the setting of CaSO₄-based bone cement.
- [3] Huang Y., et al. Key Engineering Materials, Volumes 192-195, 2001, Pages 853-862, Study on the Applied Properties of Tobramycin-Loaded Calcium Phosphate Cement.
- [4] Winkler M. M., et al. J Prosthet Dent, Volume 79, Issue 5, May 1998, Pages 532-6, Comparison of four techniques for monitoring the setting kinetics of gypsum.

This article may include the use of products and/or techniques that go beyond the current clearance/approval granted by the relevant regulatory authority.