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Josep Rodríguez-Sendra, Noé Jiménez, Rubén Pico, Joan Faus, and Francisco Camarena

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Calcium sulfate setting monitoring with ultrasonic backscattering analysis

Josep Rodríguez-Sendra
i3m, Universitat Politécnica de València, Valencia, 46022, SPAIN; jorodsen@upv.es

Noé Jiménez
Consejo Superior de Investigaciones Científicas, Valencia, SPAIN; noe.jimenez@csic.es

Rubén Pico
Universitat Politècnica de València, Gandia, Valencia, SPAIN, rpico@fis.upv.es

Joan Faus
Instituto Valenciano de Investigaciones Odontológicas, Gandia, SPAIN; joan.faus@hotmail.com

Francisco Camarena
Fisica Aplicada, i3M, Universitat Politècnica de València, Valencia, 46022, SPAIN; fracafe@fis.upv.es

Ultrasonic backscattering techniques are used to monitor the setting process of calcium sulfate. A 3.5 MHz frequency pulse-echo system is used to obtain the acoustic properties of a sample of calcium sulfate. The temperature of the sample and the rf-backscattering signal are measured every two seconds during the whole setting process (53 min; 1590 rf-signals). Apparent integrated backscatter (AIB) and speed of sound (SOS) are calculated from each measured rf-signal. A positive correlation is observed between SOS and AIB during the setting process, identifying three states of the sample: liquid, transient and solid. Sound velocity changes from 1480 m/s to 2700 m/s, meanwhile AIB changes between -15 dB and 0 dB. In the transient phase, the two parameters are sensible to the fast change in the mechanical properties of the medium. A smooth and continuous evolution of both parameters is observed as a consequence of the gradual change in the mechanical properties of the calcium sulfate during the setting in the middle region. The presented results show that the state of a sample in a setting process can be determined by performing backscattering analysis, which could have application in monitoring changes in guided bone regeneration processes for dental implant procedures.

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I. Introduction

The loss of teeth affects the quality and volume of the maxillary or mandibular bone [1]. Guided bone regeneration (GBR) is a technique that allows the recovering of bone volume, but takes between 6 and 9 months during which several postoperative complications may appear, such as soft tissue growth, failure of osseointegration or tissue infection [2]. Ultrasound is a non-ionizing and non-invasive technique able to monitor the process of guided bone regeneration.

During the last two decades, different techniques have been proposed to relate the bone physical properties with the parameters of the propagation of the ultrasonic waves travelling through it. The speed of sound (SOS) is linked with the elastic parameters of the bone, and the ultrasonic backscatter energy can be linked to the structure and composition of the bone tissue, such as volume fraction or mineral density [3], which allows quantitative ultrasound techniques for their characterization.

Many ultrasonic backscatter techniques are based on the use of the apparent backscatter transfer function (ABTF) [4]. It represents the backscattered power from the sample corrected for the frequency response of the measurement system. A parameter is obtained from ABTF, the apparent integrated backscatter (AIB), which consists of an average frequency measurement of the backscattering power in a portion of the ultrasonic backscattering signal [5]. AIB is determined in a logarithmic scale and is referenced with a measurement on a metallic surface [5]. During the process of bone regeneration mechanical properties of the material are modified. In this work we propose a proof of concept to monitor the setting of a bone graft substitute using ultrasonic backscattering methods. The acoustic properties of the material have been obtained as a function of time using a pulse-echo technique. A phantom of calcium sulphate has been used, since this material has already been used for GBR [6]. The change of state from a viscous fluid to a porous solid simulates the GBR in a short duration that can be studied under laboratory conditions (less than one hour). We report that the ultrasonic backscatter energy is correlated to the physical properties of the tissue. We propose the use of a single sensor improving the accessibility of medical instrumentation for tissue characterization.

II. Methodology

II.1. Phantom preparation

A type IV calcium sulfate (Ventura Pinkmod) has been used as a synthetic bone substitute to perform the experiments. In particular, the material in the powder state is calcium sulphate hemihydrate in α format. When the powder is mixed with water, dihydrate calcium sulphate is created in an exothermic reaction [6]. During the process of setting a phase change is produced from viscous liquid to porous solid. The total drying time is around 20 minutes. To study the repeatability, 4 experiments were performed with samples consisting of 23 ml of water and 50 g of calcium sulphate. The powder was dissolved in water at 20ºC and mixed to obtain homogenous samples. The samples were measured in a cylindrical plastic container, with a diameter of 36 mm obtaining samples of 18 mm in thickness.

II.2. Ultrasonic measurement

An Olympus V382 transducer with a center frequency of 3.5 MHz and a bandwidth of 2.34 MHz (-6 dB) has been used. A pulser receiver (Panametrics 5072-PR) has been used to emit and receive pulses. The received signal has been digitized with a red-pitaya (STEMlab 125-14 board, Red Pitaya, Solkan,Slovenia) at a sampling frequency of 125 MHz. For each experiment there are 1600 acquisitions, one every 2 seconds. The total duration of each experiment was 53 minutes, enough to record the whole cement setting process.

II.3. Acoustic parameters

The speed of sound (SOS) has been calculated as twice the thickness of the sample divided the time of flight between the first two echoes from the bottom of the sample. ABTF is obtained from:

$$ABTF = 10\log_{10}\left(\frac{P_s(f)}{P_{ref}(f)}\right) \quad (1)$$

where $P_s(f)$ is the power of the backscattering signal as a function of frequency, and $P_{ref}(f)$ is the frequency-dependent power of the first echo from a reference reflector, e.g. steel plate [4]. On the other hand, the AIB is obtained as the frequency-averaged ABTF as

$$AIB = \frac{1}{\Delta f}\int_{f_1}^{f_2} ABTF\,(f)df \quad (2)$$

where $\Delta f = f_2 - f_1$, and f_1 and f_2 are given by the 6 dB bandwidth of the transducer. The corresponding values for the effective limiting frequencies are $f_1 = 3.4\,MHz$ and $f_2 = 4.6\,MHz$.

III. Results and discussion

Figure 1 (a) shows the experimental results: A-line signals are presented in a logarithmic scale normalized to the maximum value as a function of time. The vertical axis represents the time of the experiment during the setting process (in minutes), the horizontal axis represents the time of the ultrasonic signal (in microseconds). From t = 0 to t = 2 all the ultrasonic signals have a value of 0 dB corresponding to the emission pulse. There are three regions in the figure corresponding to the three states of the material, the upper part corresponds to the sample in viscous fluid state (from T = 0 min to T = 17 min), the lower part corresponds to the sample in solid porous state (from T = 30 min to T = 53 min). The range from T = 18 min to 30 min corresponds to a transition state from viscous liquid to porous solid. Between t = 10 µs and t = 25 µs, the echo corresponding to the background of the sample is observed and around t = 30 µs the repetition of the background echo can be seen.

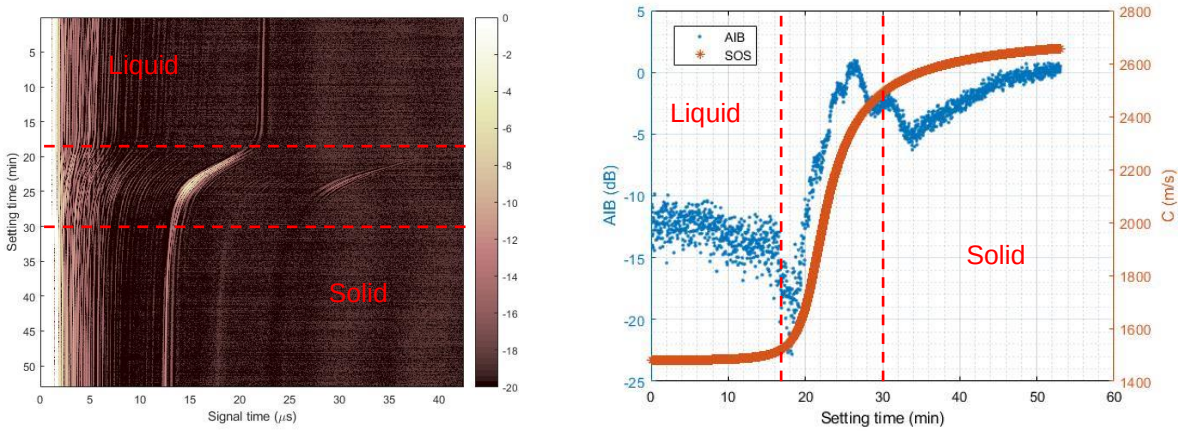

Figure 1 (a) Representation of all the experimental signals (RAW data) as a function of the setting time. Colormap in normalized dB scale. The red dashed lines mark the different states of the sample. (b) Evolution of the speed of sound (SOS), (orange dots) and apparent integrated backscatter energy (AIB) (blue dots) as a function of the setting time.

The evolution of the SOS with the setting time is shown in Fig. 1 (b). The initial value measured for the SOS corresponds to c_1 = 1477±50 m/s close to the value of liquid water at same temperature (1495 m/s). When the material is solidified, the SOS reaches a stable value close to c_2 = 2731 m/s. As expected, it can be observed that the change of the state of the material corresponds to an increase of the SOS. This particular smooth transition was measured in all samples and can be described by three different phases: the liquid state, the transition and the solid state.

In addition to SOS, energetic parameters can also provide information about the change of phase of calcium sulphate. In particular, we show in Fig. 1 (b) the AIB as a function of the setting time. When the sample is in a viscous-liquid state, from t = 0 min to about t_1= 17 min, the mean value of AIB is low and constant, taking a value of about −15 dB. However, the AIB estimations present high dispersion due to the low amplitude of the backscatter signals. The weak scattering and the weak echo reflection are caused by the strong absorption and high homogeneity of the sample in its initial viscous-liquid phase. When the exothermic chemical reaction starts, AIB initially decrease. AIB takes a value down to -20 dB at 20 min.
However, as the reaction continues (after minute 17) the scattering energy increases: the intrinsic absorption of the consolidated synthetic bone substitute is lower compared to its viscous-fluid phase. Thus, AIB progressively increases and its dispersion is reduced. During this time the transition of state in the setting process is produced; the mechanical and acoustic features of the sample are drastically modified and the microstructure of the porous solid material starts to consolidate, leading to an increase of the amplitude of the backscatter signal. The consolidated material presents less absorption. However, as the porous solid consolidates its inner microstructure the backscatter energy increases. After some small oscillations during the transition the AIB ends with a value of about -5 dB at 25 min. Finally, after 30 min, the sample is transformed to solid state and the AIB is smoothly increased from -5 dB to a plateau reaching a maximum value 0 dB at 50 min. Therefore, while the dispersion of the AIB is low and it reaches a stable value, the amount of the energy of the ultrasonic pulse that reaches the end of the sample is reduced due to scattering processes. This is caused by the final properties of the bone graft substitute that present high micro-heterogeneity and porosity. After this time, the setting process is completed, and the material is mechanically and acoustically stable.

	Liquid	**Solid**
SOS (m/s)	1480 ± 50	2600 ± 50
AIB (dB)	-13 ± 2	-2 ± 2

Table 1 The acoustic values with the ranges of AIB and SOS for the liquid and solid states

IV. Conclusion

The change of state of a bone graft substitute (calcium sulphate) has been monitored and characterized by ultrasonic backscattering analysis. Speed of sound (SOS) and apparent integrated backscatter energy (AIB) were measured during the setting process of the sample using a pulse-echo technique. The evolution of SOS shows a smooth transition between two limiting values, corresponding to the viscous-fluid and porous solid state of the calcium sulphate samples. The evolution of AIB is shown to be correlated with SOS. The apparent integrated backscatter energy is proven to be an appropriate parameter to describe the transition from a viscous-liquid state, with values around -15 dB, to a complex porous solid, with values around 0 dB. This relation confirms that the backscattering analysis with ultrasound is a powerful tool to monitor complex materials with time-varying mechanical properties using a single transducer and pulse-echo techniques. This study represents a contribution to the use of ultrasound for the monitoring of GBR, which is necessary for the successful placement of dental implants. A synthetic bone substitute has been used as a bone phantom because its mechanical properties change during the setting process from a viscous liquid to a rigid porous material, as, under reasonable simplifications, occurs in GBR applications. However, many practical questions remain open and should be explored. These include the effect of the varying coupling of the transducer and the tissue between acquisitions, the mechanical/acoustical behavior of real tissue during bone regeneration, the scattering properties of these unconsolidated and consolidated tissues, patient variability, or the impact of the intrinsic properties of real bones during regeneration (porosity, tortuosity, viscosity, ...) in the estimated parameters. These issues should be explored in future studies. Finally, it is worth to mention that one advantage of backscattering analysis with respect to speed of sound is that using backscattering techniques there is no need to know the thickness of the sample.

Note that when using a single transducer, the estimation of the thickness of the sample is critical to accurately estimate SOS and, therefore, scattering techniques might be preferred. This is a significant benefit to envisaged implant applications in-vivo where the GBR can be monitored.

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