Experimental Report

Proposal: **6-04-271** **Council:** 4/2012

Title: Dental Cements and Inelastic Neutron Scattering: Meeting the Challenges of Today's Health Concerns

This proposal is a new proposal

Researh Area: Soft condensed matter

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Samples: Commercial dental cement mixed with a polymeric acidic aqueous solution
Commercial dental cement mixed with a polymeric acidic aqueous solution containing resin
Commercial dental polymeric acidic aqueous solution containing resin
Polymeric modified commercial dental cement mixed with water
Commercial dental polymeric acidic aqueous solution

Instrument	Req. Days	All. Days	From	To
IN10	10	10	26/11/2012	06/12/2012

Abstract:

In typical odontological studies water sorption in the restoration material is determined by weighting a freshly mixed cement at regular
intervals. In the case of resin-modified cement, such process was proven to be rapid; and over the first 8 h, absorption was shown to
follow Fick’s law. These findings were attributed to conformational changes in hydrophilic segments of the polymer on absorption of
aqueous sodium chloride. It is considered that in this scenario the molecules form more compact coils than in the presence of pure
water. To improve such evaluation process, to compare different dental cements and, at the same time, to develop new materials to
be used in dental treatment, insight on parameters such as consistency, working and setting times, as well as the chemical reaction
dynamics are important. Such factors are usually hard to determine accurately and non-destructively. Two techniques,

Dental Cements and Inelastic Neutron Scattering: Meeting the Challenges of Today's Health Concerns

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The understanding of the setting reaction of glass ionomer cements (GIC), information on proton interaction and mobility within the pore structure is still limited. In order to optimize GIC, a thorough understanding of their structure and hydration process is required. One of the most promising techniques to achieve this aim is neutron scattering. The use of quasi-elastic (QENS) gives a unique perception of the molecular motions within matter. In addition, QENS allows the assessment of the dynamics and associated geometric of motion of protons to be investigated on very short time scales (1,2). Here two samples were analyzed, Aqua and Poly. From our results obtained using IN10, a better understanding of the proton dynamics of these dental GICs was achieved (2). As the cement pastes hydrates as a function of time, the viscosity of the paste increases with time together with the degree of structural order due to the development of hydrogen bonds. In order to access the evolution of the microstructure of GIC during the hydration process, the elastic intensity of the signal detected using IN10 with the Doppler off was followed for the first 24 hours and at the 5th and 23rd day after the start of the process. Following this approach the immobile hydrogen index (IHI), defined as the ratio between the elastic intensity that evolves with setting of the GIC (at each time point) by the total elastic intensity, which is constant over time, was related to the cement setting.

At the time-scale covered by IN10, the mobility of the slower and longer polymeric chains was assessed using IN10. From the data we observed that the polymerization rate is faster for the less viscous cement (Aqua) during the first 24 hours of hydration, but almost the same at the 5th day (Figure 1). Proton mobility was also assessed as a function of temperature by following the elastic signal of the dental cements from 2K until body temperature (Figure 2). No real

difference (within the experimental error) in the number of mobile protons could be identified after 5 days of the start of the mixture. However the activation temperature for each sample was proven to be different. This result can be connected to the different porosity observed in each sample. The data are still under analysis.

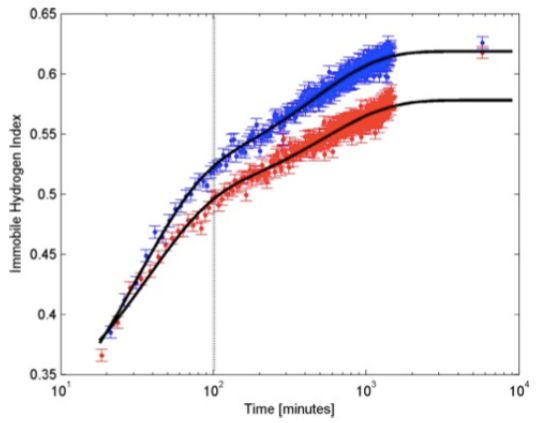

Fig1. The time evolution of the immobile hydrogen index (IHI) for the IN10 experiment on the ns time-scale. The AQUA sample is in blue and the POLY sample is in red. The measured IHI is fitted to an exponential growth model (black line). The vertical dotted line indicates the threshold between two distinct parts of the setting reaction.

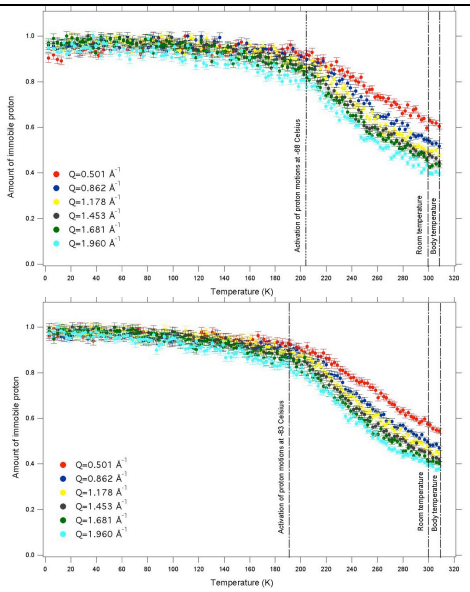

Fig2. The temperature evolution of the immobile hydrogen for the IN10 experiment on the ns time-scale. Noe that the motions are activated at a higher temperature for the AQUA (upper figure).

[1] J. Jacobsen, M.S. Rodrigues, M.T.F. Telling, A.L.Beraldo, S.F. Santos, L.P. Aldridge and H.N. Bordallo (2013) Nano-scale hydrogen-bond network improves the durability of greener cements. Sci. Rep. **3**, 2667.

[2] J. Jacobsen, Master Thesis, University of Copenhagen 2013.