

## $^1\text{H}$ - $^{19}\text{F}$ Stray-field Magnetic Resonance Imaging Studies of a Commercial Glass Ionomer Cement

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**Abstract:** Glass-Ionomer Cements (GICs) are biomaterials used in restorative dentistry. They are particularly important because of their anticariogenic properties, which are obtained/OR which are observed when fluorine components are used in the glass composition, and are due to fluoride leaching from the cement matrix. These dental materials are particularly beneficial to patients with risk of recurrent caries because they release fluoride in the presence of acids produced by cariogenic bacteria. Although it is known that when immersed in fluoride solutions GICs absorb fluorine ions, leaching and absorption processes are still not well understood. Spatial information about the distribution of fluorine ions in GIC samples will contribute to the understanding of the leaching-absorption processes. The commercial product (FUJI II, GC) is provided as a two-phase component: a liquid that contains mainly polyacrylic acid and water, and an aluminosilicate-based glass that includes Sr, P and F. When mixed, these components undergo an acid-base reaction with the subsequent formation of a GIC. Aspects relevant to the GIC performance were investigated by  $^1\text{H}$  &  $^{19}\text{F}$  Stray-Field MRI. 1D MRI was used to follow the GIC setting reaction over 65 h, in order to obtain the spatially resolved kinetics and evidence for the involvement of fluorine in the cure reaction, as compared with hydrogen;  $^1\text{H}$  and  $^{19}\text{F}$  2D images of the GIC were also acquired, which exhibited different spatial distributions.

Glass Ionomer Cements (GICs) are biomaterials used in restorative dentistry.<sup>1,2</sup> They are particularly important because of their anticariogenic properties, which are observed when fluorine components are used in the glass composition<sup>3,4</sup> and are due to fluoride leaching from the cement matrix. Although it is known that when immersed in fluoride solutions GICs absorb fluorine ions<sup>3</sup>, leaching and absorption processes are still not well understood.

Spatial information about the distribution of fluorine ions in GIC samples will contribute to the understanding of the leaching-absorption processes. Stray-field (STRAFI) magnetic resonance imaging (MRI) is a technique which enables simultaneous  $^1\text{H}$  and  $^{19}\text{F}$  image acquisition on solid samples.<sup>5,6</sup> In this study, STRAFI MRI is used to obtain  $^1\text{H}$  and  $^{19}\text{F}$

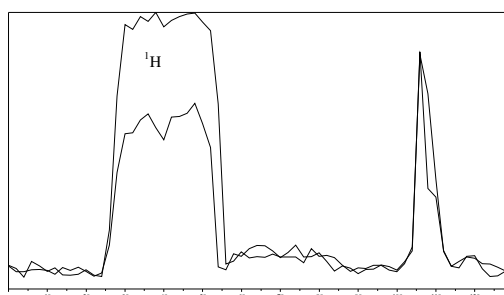
images of a commercially available GIC in order to monitor the curing reaction through the spatially-resolved kinetics, and to map  $^{19}\text{F}$  distribution.

A commercially available GIC, Fuji II (F2) (GC Corporation, lot. 050391) was used. A BRUKER MSL 300P NMR A commercially available GIC, Fuji II (F2) (GC Corporation, lot. 050391) was used. A BRUKER MSL 300P NMR spectrometer with a 7 T magnet, equipped with a dedicated STRAFI probe head (BRUKER, Germany), was used to run MRI experiments at 2.9 T and 3.1 T in a 37.5 T/m static magnetic field gradient. was used to run MRI experiments at 2.9 T and 3.1 T in a 37.5 T/m static magnetic field gradient. 1D  $^1\text{H}$  and  $^{19}\text{F}$  images (profiles) of an F2 cylindrical sample (4 mm height and 6 mm diameter) were acquired, periodically, over 65 h. The

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intensity variation of 3 slices from each one of the  $^1\text{H}$  and  $^{19}\text{F}$  profiles was monitored as a function of time. The figure presents the first and the last  $^1\text{H}$  and  $^{19}\text{F}$  profiles recorded during the setting reaction.



**Figure 1.**  $^1\text{H}$  and  $^{19}\text{F}$  profiles of the first (more intense) and final (less intense) images. Slices T (Top), M (Middle) and B (Bottom) are indicated. A plastic disc signal was used as an intensity reference. Double exponential functions ( $y = y_0 + A_1\exp(-x/t_1) + A_2\exp(-x/t_2)$ ) were used to fit the magnetisation intensity variation of the six slices as a function of time.  $t_1$  and  $t_2$  values for  $^1\text{H}$  data are shown in Table 1.

In the case of  $^{19}\text{F}$  slices, no intensity variations with time were observed. Best fit results were obtained with a double-exponential function, showing the existence of chemical reactions with very different rates ( $t_2 \geq 44 \times t_1$ ), during the GIC setting period.  $^{19}\text{F}$  1D profiles did not show significant variations during the curing reaction. However, changes should be more difficult to observe due to the lower signal to noise ratio of the  $^{19}\text{F}$  profiles when compared with the  $^1\text{H}$  profiles. With the images obtained, the 1D distribution of  $^1\text{H}$  and  $^{19}\text{F}$  was observed, separately, in the same experiment.

Table 1.  $t_1$  and  $t_2$  time constants of the double-exponential function used to fit  $^1\text{H}$  magnetisation decays, obtained from the different slices, with setting time.

Slice	$t_1$ (min)	$t_2$ (h)
T	25 ( $\pm 1\%$ )	28 ( $\pm 2\%$ )
M	36 ( $\pm 2\%$ )	27 ( $\pm 1\%$ )
B	25 ( $\pm 1\%$ )	19 ( $\pm 1\%$ )

$^1\text{H}$  spatial resolved kinetics of the GIC setting reaction was obtained. Moreover,  $^{19}\text{F}$  profiles were recorded showing a homogenous distribution in the sample. 2D  $^1\text{H}$  and  $^{19}\text{F}$  images showed a surface layer of 0.45 mm where the setting reactions are faster.

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#### References

1. A. Wilson, J. McLean, *Glass-Ionomer Cements*, 1<sup>st</sup> ed., Quintessence (1988).
2. A. Wilson, J. Nicholson, *Acid Base Cements – Their biomedical and industrial applications*, 1<sup>st</sup> ed. Cambridge University Press: Cambridge (1993).
3. C. Francci, T.G. Deaton, R.R. Arnold, E.J. Swift Jr., J. Perdigão, J.W. Bawden, *Journal of Dental Research* **78** (1999) 1647.
4. M. Braden, R.L. Clarke, J. Nicholson, S. Parker, *Polymeric Dental Materials*, 1<sup>st</sup> ed., Springer: Berlin (1997).
5. E.W. Randall, A.A. Samoilenko, T. Nunes, *J. Magn. Reson.* **A117** (1995) 317.
6. C.H. Lloyd, S.N. Scrimgeour, G. Hunter, J.A. Chudek, D.M. Lane, P.J. McDonald, *Journal of Materials Science: Materials in Medicine* **10** (1999) 369.