15 Jul 2004

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Recommended Citation

E. Twomey et al., "Investigation into the Ultrasonic Setting of Glass Ionomer Cements : Part II Setting Times and Compressive Strengths," *Journal of Materials Science*, vol. 39, no. 14, pp. 4631 - 4632, Springer, Jul 2004.
The definitive version is available at https://doi.org/10.1023/B:JMSC.0000034158.69184.84

Investigation into the ultrasonic setting of glass ionomer cements

Part II *Setting times and compressive strengths*

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Glass ionomer cements (GICs) are formed by the reaction of an ion leachable alumino-silicate glass with an aqueous solution of poly (alkenoic acid), PAA. Water is used as the reaction medium [1]. A two-stage setting reaction occurs, resulting in a cement consisting of residual glass particles embedded in a hydrogel polysalt matrix [2]. During the first stage the material is susceptible to water uptake and during the second it is susceptible to dehydration. For example, when GICs are stored in water after an initial set of 15 min, a surface softening occurs, which may be caused by an inhibition of the setting reaction in a superficial layer of the cement [3]. This short-term relationship with water restricts the potential of GICs for healthcare applications. It is for this reason that resin modified GICs (RMGICs) were developed. These materials, conventional GICs into which an organic, photo-polymerisable monomer has been incorporated [4], can be command set by the application of an intense light source. However, RMGICs have drawbacks related to the presence of both non-polymerised monomer and the resin itself [5, 6]. Rapid setting is important both for providing resistance to attack from moisture in the mouth and improving wear resistance, but an alternative method to light-curing which does not require the incorporation of additional chemicals would be beneficial.

The application of ultrasonic waves has long been used for setting cement in the building industry, and so studies were performed to utilise the same method for setting dental cements [7]. Most dental clinics have ultrasonic capability as it is used for de-scaling teeth. The effects of applying ultrasonic waves to the surface of the cement include a reduction in both porosity and mean glass particle size (due to breaking up of agglomerates), an improvement in the packing of the glass particles and a decrease in the mean molecular weight of the PAA [8]. Whilst it is not possible to further explain the curing mechanism at this stage, this work will determine the extent to which ultrasound affects the setting times and mechanical properties of a series of hand mixed and mechanically triturated GICs.

The following cement compositions were assessed:

- Ketac Molar Quick (ESPE, Germany). Batch #00150014.
- Fuji IX Fast (GC, Japan). Batch #0107165.
- Experimental GICs.

  These cements were based on SR1 glass (a strontium aluminosilicate glass) mixed with three different PAAs; PAA_{50}, PAA_{200} and PAA_{450} (Advanced Healthcare Ltd., Kent, UK). The molecular weights of the PAAs are included in Table I. A P:L mixing ratio of 8.1:1 was employed for the experimental GICs. Commercially a P:L ratio of greater than 10:1 is employed, but this is possible only with mechanical trituration.

The experimental GICs were hand mixed on a glass slab. Activation and mechanical mixing of the commercial GICs took place in accordance with the directions supplied by the manufacturers. The ultrasonic equipment employed was a Piezon® Master 400 dental descaler (EMS, Nyon, Switzerland), with a frequency of 25–30 kHz. The insert used (DS-003) was developed for de-scaling applications.

The setting times and compressive strengths of the cements were evaluated by standard methods [9]. However, the compressive samples prepared were reduced in size (3 mmϕ × 5 mm height) to more closely reflect the clinical situation. Samples were either left to set chemically or ultrasound was applied to one side of the sample. Ultrasonic exposure was for 45 s unless stated otherwise.

The working and setting times of Ketac Molar Quick (KMQ) were 100 and 200 seconds, respectively. The working and setting times of Fuji IX Fast (FIXF) were 75 and 180 s, respectively. With the application of ultrasound, the setting time for both cement systems was reduced and full command setting was achieved with 45 s of ultrasonic exposure (Table II).

Ultrasound also resulted in improvements in compressive strength (Table III), with a relative increase of

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TABLE I Molar mass details of the PAAs

PAA	M_w	M_n	P.D.
PAA_{50}	51,900	21,900	2.4
PAA_{200}	210,300	111,300	1.9
PAA_{450}	435,700	208,500	2.1

TABLE II The influence of ultrasound upon the setting time of GICs

Ultrasonic exposure (s)	KMQ Setting time (s)	FIXF Setting time (s)
0	200	180
15	115	50
30	75	30
45	Command set	Command set
60	Command set	Command set

TABLE III The influence of ultrasound upon the compressive strength of GICs. Standard deviations are included in brackets

		Compressive strength (MPa)	
Cement	Mode of set	1 day	7 days
KMQ	Chemical	137 (*13*)	171 (*8*)
	Ultrasonic	154 (*9*)	196 (*10*)
FIXF	Chemical	130 (*7*)	156 (*12*)
	Ultrasonic	153 (*12*)	175 (*10*)

TABLE IV The effect of 45 s of ultrasound upon the setting time of the experimental GICs

	PAA_{50}	PAA_{200}	PAA_{450}
Chemical set	275s	240s	220s
Ultrasonic set	Command set	Command set	Command set

12 and 18% (1 day) and 15 and 12% (7 days), being observed for KMQ and FIXF, respectively.

The handling properties and compressive strengths of the experimental GICs are included in Tables IV and V. All the experimental GICs could be command set with exposure to 45 s of ultrasound.

Increasing PAA molecular weight from PAA_{50} to PAA_{200} increased strength, but a further increase to PAA_{450} reduced strength. The PAA_{200} cements showed a percentage increase in strength of 13% at 1 day and 24% at 7 days, compared with the chemically set samples.

This work shows that ultrasound has a beneficial effect on the setting times and mechanical properties of GICs. Compressive strengths of GICs are known to increase with maturation time but the influence of ultrasonic setting has not previously been analysed. The commercial materials both showed increases in compressive strength with ultrasonic setting, but the efficacy of the technique depended upon the composition of the material. Of the experimental cements, $SR1PAA_{200}$ gave the best results for 1 and 7 day samples. These results showed that there is an optimum PAA molecular weight for these materials. The strengths of the ultrasonically set materials after 1 day are close to the values for the 7 days chemically set samples, reinforcing the theory that ultrasound accelerates the setting reaction.

TABLE V Influence of mode of set and maturation time on the compressive strength of GICs. Standard deviations are included in brackets

		Compressive strength (MPa)	
Cement	Mode of setting	1 day	7 days
$SR1/PAA_{50}$	Chemical	207 (*13*)	226 (*6*)
	Ultrasonic	225 (*7*)	245 (*10*)
$SR1/PAA_{200}$	Chemical	256 (*9*)	272 (*9*)
	Ultrasonic	288 (*11*)	336 (*8*)
$SR1/PAA_{450}$	Chemical	185 (*9*)	214 (*8*)
	Ultrasonic	205 (*7*)	231 (*5*)

The authors have shown earlier that the breaking up of agglomerates of glass particles and densification of the cement by ultrasound are influential factors in the ultrasonic setting process [8]. This work does not attempt to explain the curing mechanism further, but it does show experimentally, the improvements in setting times and mechanical properties that result from such a technique.

Acknowledgment

The present study was supported by the BRITE-EURAM Program from the 5th Framework of R&D of the European Union (UltraSet, G5RD CT2001 00475).

References

1. B. E. KENT and A. D. WILSON, *Br. Dent. J.* **135** (1973) 322.
2. S. CRISP and A. D. WILSON, *J. Dent. Res.* **53** (1974) 1408.
3. R. J. G. DE MOOR and R. M. H. VERBEECK, *Biomaterials* **19** (1998) 2269.
4. S. B. MITRA, European Patent Application No. 0323 120 (1989) Vol. A2.
5. J. W. NICHOLSON and H. M. ANSTICE, *J. Mater. Sci: Mater. Med.* **3** (1992) 447.
6. M. A. CATTANI-LORENTE, V. DUPUIS, J. PAYAN, F. MOYA and J. M. MEYER, *Dental Mater.* **15/1** (1999) 71.
7. M. R. TOWLER, A. J. BUSHBY, R. W. BILLINGTON and R. G. HILL, *Biomaterials.* **22/11** (2001) 1401.
8. M. R. TOWLER, C. M. CROWLEY and R. G. HILL, *J. Mater. Sci.: Lett.* **22/7** (2003) 539.
9. ISO 9917(E) International Standard. (1991) pp. 1.

Received 25 February
and accepted 18 March 2004