BIOMATERIALS SYNTHESIS AND CHARACTERIZATION

Heat transfer properties and thermal cure of glass-ionomer dental cements

Lidia Gavic $^1\cdot$ Kristina Gorseta $^2\cdot$ Domagoj Glavina $^2\cdot$ Beata Czarnecka $^3\cdot$ John W. Nicholson 4

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Abstract Under clinical conditions, conventional glassionomer dental cements can be cured by application of heat from dental cure lamps, which causes acceleration in the setting. In order for this to be successful, such heat must be able to spread sufficiently through the cement to enhance cure, but not transmit heat so effectively that the underlying dental pulp of the tooth is damaged. The current study was aimed at measuring heat transfer properties of modern restorative glass-ionomers to determine the extent to which they meet these twin requirements. Three commercial glass ionomer cements (Ionofil Molar, Ketac Molar and EquiaTM Fill) were used in association with three different light emitting diode cure lamps designed for clinical use. In addition, for each cement, one set of specimens was allowed to cure without application of a lamp. Temperature changes were measured at three different depths (2, 3 and 4 mm) after cure

John W. Nicholson john.nicholson@bluefieldcentre.co.uk

- ¹ School of Dental Medicine, University of Split, Split, Croatia
- ² Department of Paediatric and Preventive Dentistry, School of Dental Medicine, University of Zagreb, Zagreb, Croatia
- ³ Department of Biomaterials and Experimental Dentistry, University of Medical Sciences, Poznan, Poland
- ⁴ Bluefield Centre for Biomaterials, Unit 34, 67-68 Hatton Garden, London EC1N 8JY, UK

times of 20, 40 and 60 s. The difference among the tested groups was evaluated by ANOVA (P < 0.05) and post hoc Newman-Keuls test. All brands of glass-ionomer showed a small inherent setting exotherm in the absence of heat irradiation, but much greater temperature increases when exposed to the cure lamp. However, temperature rises did not exceed 12.9 °C. Application of the cure lamp led to the establishment of a temperature gradient throughout each specimen. Differences were typically significant (P < 0.05) and did not reflect the nominal power of the lamps, because those lamps have variable cooling systems, and are designed to optimize light output, not heating effect. Because the thermal conductivity of glass-ionomers is low, temperature rises at 4 mm depths were much lower than at 2 mm. At no time did the temperature rise sufficiently to cause concern about potential damage to the pulp.

Graphical Abstract Temperature gradient through glass-ionomer (EquiaFil exposed to Bluephase Style lamp)





Glass ionomer cements are widely used in dentistry in the restoration of teeth damaged by caries [1]. They set by an acid–base reaction between special basic glasses, which are typically calcium or strontium alumina-silicates with additions of fluoride, and aqueous solutions of polymeric acids, such as polyacrylic acid or acrylic/maleic acid copolymer [1, 2]. Setting begins immediately the powdered glass and acid solution are mixed, and continues for some weeks after initial hardening in a complex process that includes ionic crosslinking of neutralized polymer molecules and formation of a poorly understood inorganic network [3]. Uses of glass-ionomers within dentistry include as liners and bases, full restorations, adhesives for orthodontic brackets, and fissure sealants [1].

Typically in clinical use, glass-ionomer cements are placed in a tooth and allowed to undergo their natural setting reaction in situ [1]. They may be protected from drying out by the application of either petroleum jelly or a varnish [4]. Such treatment has the effect of eliminating surface desiccation, which otherwise leads to the formation of numerous micro-cracks and the development of an undesirable chalky appearance [4].

The setting of glass-ionomers has been shown to be capable of acceleration, either by the application of ultrasound [5–8] or by the effects of heat from a dental curing lamp [9, 10]. The acceleration of setting has been found to improve early mechanical properties [7] and also marginal adaption [8], and suggests that the use of a dental cure lamp can be used to provide "command setting" for conventional glass-ionomers that set by neutralization [8].

The effectiveness of such treatment depends on the thermal properties of glass-ionomer cements. Specifically, the properties of thermal conductivity and thermal diffusivity are important if sufficient heat is to be transferred from the surface of the cement, where the cure light is applied, to the interior. Although glass-ionomers are generally claimed to have acceptable thermal properties, for example of thermal conductivity, for clinical application, there have been few studies of these properties. The most comprehensive of these, by Inoue et al. [11], determined the thermal diffusivity, the specific heat capacity and the thermal conductivity for five different commercial materials formulated at five different powder:liquid ratios. Values varied with brand of material and with powder:liquid ratios. The latter was found to influence the thermal properties linearly with high correlation coefficients (at least 0.97 in all cases). Increasing the powder: liquid ratio in the range 0.8 to 1.2 caused distinct increases in thermal diffusivity and thermal conductivity, but reductions in specific heat capacity. These effects relate

to the thermal properties of the glass powders in these cements, and showed that increases in the powder:liquid ratio reduce the temperature rise for a given input of heat, but increases the rate at which heat spreads through the material. These effects, particularly for thermal diffusivity, have been confirmed by other workers [12–14].

Overall, results from these studies have shown that glass-ionomers possess thermal properties that are a good match for human dentine. For example, thermal diffusivity for glass-ionomers was found to range from 0.212×10^{-2} to 0.303×10^{-2} cm² s⁻¹ [11], compared to 0.258×10^{-2} cm² s⁻¹ for human dentine [15]. Similarly, specific heat capacity was found to range from 1.011 to 1.368 J K⁻¹ g⁻¹ for glass-ionomers [11], compared with 1.283 J K⁻¹ g⁻¹ for human dentine [15].

The current study was undertaken to determine in detail the heat transfer properties of glass ionomer dental cements when heated with dental cure lamps having different power outputs. Three different light emitting diode (LED) cure lamps were used, and the temperature rise generated at the lamp tip was determined over time for all three. These lamps were then applied to the cure of three different brands of glass-ionomer dental cement, and the temperature at three different depths within the material was determined for time intervals of up to 60 s. From this, an indication of the rate of heat transfer was obtained, and the extent of the internal temperature rise within the cements determined. Since heat has been identified heat as a primary cause of pulpal injury in clinical dentistry [16], with temperature rises of 16.6 °C capable of causing non-viability of 100 % of pulps tested [17], results from the present study are of use in assessing whether or not accelerating the cure of glass-ionomers with modern LED lamps is clinically acceptable.

2 Materials and methods

The LED cure lamps used in this study are described in Table 1. The output temperatures were measured with a digital thermocouple instrument TC 309 (Dostmann Electronic GMBH, Wertheim-Reicholzheim, Germany) placed at the tip of each light.

Three different restorative commercially available glass ionomers cements were used in this study, details of which are given in Table 2. The cements were supplied in capsulated form and were shade A2. The capsules were activated and mixed with a vibratory mixer (CapMix[®], 3 M ESPE, Seefeld, Germany) in accordance with the procedures supplied by each manufacturer. After mixing, cements were placed in Teflon moulds that included a cylindrical hole, 3 mm in diameter and 4 mm deep. Samples were covered with a celluloid matrix strip and pressed flat. Two series of experiments were then carried out. In one, the temperature was recorded at depths of 2, 3 and 4 mm at times of 20, 40 and 60 s from the end of mixing. In the other, one end of the specimen was exposed to one of the cure lamps for 60 s, and temperatures were also determined after 20, 40 and 60 s. In all experiments, the temperature was measured with a digital thermocouple TC 309 (Dostmann Electronic GMBH, Wertheim-Reicholzheim, Germany) and the tip of the thermocouple was placed in the centre of the Teflon moulds at the appropriate depths from the top of the specimen. Each test was repeated three times for each depth, and the values averaged to determine the mean temperature rise.

The data were analysed for significance using Analysis of Variance (ANOVA) and the Newman–Keuls test (P < 0.05), the analysis being carried out with the Statistica 7.0 software package.

3 Results

All samples of glass-ionomer showed a small setting exotherm over 60 s in the absence of heating from the cure lamp, confirming previous observations for materials of this type [18]. The highest temperature rise was 2.2 °C, recorded was for Equia Fil at a depth of 2 mm. In general, there were only very small differences in temperature at varying depths for specimens cured without exposure to a curing lamp, though again Equia Fil was an exception, since it showed a temperature rise of only 1.3 °C at a depth of 4 mm. The mean of the output temperatures in °C for the three LED curing lamps at different times are shown in the Fig. 1. The temperature rise at the lamp tip for the Bluephase G2 lamp was significantly greater than those of the Bluephase Style and Bluephase 16i lamps (P < 0.05). The temperature rises for the Bluephase 16i lamp and the Bluephase Style were not significantly different from each other.

The values of temperature at the three different depths for the three glass-ionomers at 20, 40 and 60 s are shown in Table 3. Where cure lamps were employed, temperature rises were greatest at the shallowest depth of cure, with values typically being significantly different from those at 4 mm (P < 0.05), though there were exceptions to this, e.g. for Ketac Molar at 20 s cure time, there was no significant difference between 3 and 4 mm depth. Values with a cure lamp were much higher than those in specimens allowed to cure without exposure to heat, and this was statistically significant (P < 0.05).

The temperature rises within the cement were typically much lower than those measured at the lamp tip. The highest temperature rise within a cement that was recorded was 12.9 °C, at a depth of 4 mm for Equia Fil at 20 s in association with the Bluephase Style lamp, yet its lamp tip showed a temperature rise of 30 °C. This shows that the cements acted as thermal insulators, and did not transmit heat easily to greater depths.

There was no correlation between the output temperature of the lamp and the measured temperatures within the cements. In general, greater internal temperatures were found for the two lower output lamps, though the differences were not always significant. Overall, the results show that LED cure lamps increase the temperature of glass-

Table 1 LED cure lamps		Manufacturer	Wavelength (nm)	Power-density (mW/cm ²)
	Bluephase Style	IvoclarVivadent, Lichtenstein	385-515	1100
	Bluephase 16i	IvoclarVivadent, Lichtenstein	430-490	1600
	Bluephase G2	IvoclarVivadent, Lichtenstein	385-515	1200

Table 2 List of glass-ionomer cements

Material	Manufacturer	Lot	Composition	Powder: Liquid ratio
Ketac Molar Quick Aplicap	3M ESPE, Seefeld, Germany	#493820	La-containing alumino-fluorosilicate glass, acrylic/maleiccopolymer tartaric acid, water	3.4:1
Ionofil Molar AC Quick	VOC, Cuxhaven, Germany	#1232230	Alumino-fluorosilicate glass, poly(acrylic acid), tartaric acid, water	3.7:1
EquiFil	GC Corp., Tokyo, Japan	#1208061		3.3:1



Fig. 1 Temperature at the tips of the LED lamps

ionomer cements, but that these cements have only limited capacity to transmit this heat into their interiors.

4 Discussion

Glass-ionomer cements set by a neutralization reaction, and reactions of this type are typically exothermic [19]. This is why there is a small temperature rises in all cements at all depths when they were allowed to set without the assistance of a cure lamp. This confirms previous observations [18].

The heat provided by the cure lamps caused consistently greater temperature rises inside all cements. In all three materials, the pattern was of the greatest increase occurring at the lowest depths, a result which demonstrates that these materials are reasonable insulators, and have low thermal conductivities. All three materials are formulated at similar powder:liquid ratios, which means that there is no significant difference between the set cements in terms of their thermal conductivity. The glass component has higher thermal conductivity than the matrix, and the fact that the proportion of glass is similar in all cements (Table 2) means that thermal conductivities of the three cements will be similar.

The highest temperature cure lamp (Bluephase G2) did not cause the greatest temperature measurements within the cements, not even at the lowest depth of 2 mm. The design of the lamps, including the addition of cooling systems, means that there is no correlation between the nominal power output of the lamp and irs corresponding heating effect. Nonetheless there is a distinct heating effect, as shown by the temperature rises in all cements at the smallest depths. The relative small temperature rises at greatest depths show that the cements transmit only a small fraction of the incident thermal energy. Such transmission eventually leads to increases in temperature of 5–6 °C at depths of 3–4 mm within the cement. This suggests that, under clinical conditions, cements in typical cavities may have their cure reaction affected at leats to a small extent throughout their bulk.

The temperature increases that we have measured at depths within the specimens is sufficient to cause an acceleration in setting rate of the cement. This acceleration is capable of improving the mechanical properties of the cement, making it stronger and less likely to fracture prematurely. Heat from cure lamps therefore seems to be advantageous for improving clinical performance.

Modern LED cure lamps give out less heat than the older type of quartz-tungsten-halogen lamps used for lightcuring dental materials [20]. This lower heat emission has been promoted as an advantage of the modern cure lamps, but clearly is not advantageous when they are applied to glass-ionomers, because it is the heat not the light output which causes the acceleration in curing. However, the latest LED lights have much higher power densities and hence higher thermal emissions [21, 22], and these are likely to be more staisfactory for enhancing the cure of conventional glass-ionomer cements.

There have been few previous studies on the thermal properties of glass-ionomers, but what information that is available is consistent with our obervations that these materials have low thermal conductivities at the powder:liquid ratios used for practical cements. An early study considered the property of thermal diffusivity, i.e. the measure of how quickly a material reacts to a change in temperature [14] and showed it to be low for some of the earliest commercial brands of glass-ionomer cement. This study also reported that glass-ionomers were good thermal insulators [14], a finding that has been confirmed in other studies concerned mainly with thermal diffusivity [12, 13, 23].

The effect of heat on the dental pulp is a topic of concern in practical conditions in the clinic. As mentioned earlier, a temperature rise of 16.6 °C is sufficient to cause pulp death in 100 % of cases [17]. A temperature rise of at least this value was found to be generated at the lamp tip of all of the cure lamps in this study. However, because all three glass-ionomers had poor thermal conductivity, in no case did the temperature rise deep within the specimen reach this value. The highest temperature rise recorded at a depth of 4 mm was for Equia Fil cured with the Bluephase G2 lamp, where the temperature rose by 6.2 °C, i.e. over 10 °C lower than that needed to cause damage to the pulp.

Our results thus confirm previous findings [18, 22, 23] that glass-ionomer cements are capable of protecting the

	Ketac Mola	r			Ionofil Mola	ar			Equia Fil			
Time (depth)	None	BG2	B16i	BS	None	BG2	B16i	BS	None	BG2	B16i	BS
20 s												
(2 mm)	1.1(0.0)	3.6 (1.3)	3.2 (0.8)	5.1 (0.3)	1.3(0.0)	3.1 (0.6)	3.4 (0.7)	3.5(0.9)	1.4(0.4)	4.1 (1.7)	7.9 (0.1)	6.3(0.3)
(3 mm)	1.3(0.0)	3.2 (1.0)	3.0 (1.7)	2.3 (0.7)	1.1 (0.0)	2.5 (0.5)	1.7 (0.2)	2.0(0.3)	1.7 (0.0)	2.8 (0.3)	3.8 (0.2)	4.3 (1.3)
(4 mm)	1.2(0.0)	2.1 (0.4)	1.4 (0.3)	2.0 (0.6)	1.1 (0.0)	2.3 (0.4)	1.6(0.4)	1.3(0.3)	1.2 (0.0)	2.4 (0.1)	2.2 (0.6)	1.6(1.0)
40 s												
(2 mm)	1.2(0.0)	6.3 (1.6)	4.6 (1.6)	8.0 (0.6)	1.4 (0.0)	4.8 (0.3)	5.7 (1.8)	5.6 (0.8)	1.7 (0.6)	8.1 (2.2)	9.8 (1.0)	10.4 (2.3)
(3 mm)	1.3(0.0)	5.8 (1.0)	5.4 (2.2)	4.4 (2.0)	1.1 (0.0)	4.7 (0.9)	3.5 (0.5)	4.3 (1.0)	1.7 (0.0)	5.2 (0.6)	6.9 (0.2)	6.0(1.1)
(4 mm)	1.2(0.0)	4.1 (0.7)	2.4 (0.6)	3.9 (1.0)	1.2 (0.0)	4.3 (0.8)	2.9 (0.7)	2.6(0.1)	1.3(0.0)	4.3 (0.9)	3.9 (0.6)	3.1 (1.0)
60 s												
(2 mm)	1.4(0.0)	8.4 (1.9)	6.4 (1.7)	11.0 (0.6)	1.5(0.1)	6.6(0.4)	7.5 (2.0)	7.5 (0.7)	2.2 (0.8)	10.4 (2.5)	12.4 (0.8)	12.9 (2.5)
(3 mm)	1.4(0.0)	7.8 (1.9)	7.2 (2.4)	5.8 (2.4)	1.2 (0.0)	6.5 (1.1)	4.6 (0.9)	5.7 (1.3)	1.8(0.0)	6.9 (0.8)	9.0 (1.4)	7.4 (1.1)
(4 mm)	1.3(0.0)	5.8 (0.9)	3.2 (0.8)	4.8 (1.3)	1.2 (0.0)	5.8 (1.0)	3.8 (1.1)	3.8(0.3)	1.3(0.0)	6.2 (1.0)	6.1 (0.6)	4.7 (1.1)

pulp for thermal damage. Despite this, they are able to transmit some heat into their bulk, and as such are capable of being heat-cured in the dental clinic, and this improving the rate at which they develop their optimum physical properties. However, accelerating the setting reaction should be avoided when the cements are used as cavity liners in layers less than 2 mm thick. Under these circumstances thermo-curing should also be restricted to times no longer than 20 s.

5 Conclusions

We have shown that three brands of modern commercial glass-ionomer cement studied showed a small setting exotherm in the absence of heat irradiation. Much greater temperature increases were observed when these cements were exposed to one of three dental cure lamps, though temperature rises did not exceed 12.9 °C in any case. Heat from the cure lamp led to the development of a temperature gradient throughout each specimen, with gradients being similar in all cases. Differences were typically significant (P < 0.05) and did not reflect the nominal power of the lamps. This is because the lamps have cooling systems that vary in their effectiveness, and also because they are designed to optimize light output, not heat emission. The thermal conductivity of all three glass-ionomers was shown to be low, which resulted in temperature rises at 4 mm depths being much lower than at 2 mm. The heat transmission through the cement was insufficient to cause potential damage to the pulp in all cases, suggesting that heat-curing of glass-ionomer cements with lamps of this type is a safe clinical procedure.

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