Research Article

The effect of curing mode on extent of polymerization and microhardness of dual-cured, self-adhesive resin cements

Milena Cadenaro, DDS, PhD, **Chiara Ottavia Navarra**, DDS, PhD, **Francesca Antoniolli**, Eng, PhD, **Annalisa Mazzoni**, DDS, PhD, **Roberto Di Lenarda**, DDS, **Frederick Allen Rueggeberg**, DMD, MS & **Lorenzo Breschi**, DDS, PhD

Abstract: Purpose: To compare the effect of curing mode (self- or light-cure) on the extent of polymerization (%EPl as measured using differential scanning calorimetry, (DSC) and microhardness of two dual-cured, self-adhesive resin cements, using a conventional, dual-cured resin cement as control. **Methods:** Small amounts of the commercial self-adhesive cements Maxcem and RelyX Unicem or Panavia F2.0 dual-cure resin based cement used as control were polymerized within the DSC chamber at 35°C under a nitrogen atmosphere. 10 specimens were light-cured immediately (20 seconds, 600 mW/cm^2) and left undisturbed for 2 hours and 10 additional specimens were left to self-cure in the dark for 2 hours. Following DSC treatment, microhardness of the specimens was measured (Vickers). For each test parameter, data were analyzed with a two-way ANOVA and the Tukey *post hoc* test. **Results:** %EP and microhardness of all cements were higher when the light-cure mode of dual-activation was used ($P < 0.05$) instead of only self-curing. No significant difference in %EP was found between either self-adhesive cement or the control using either the light- or self-curing modes. In the light-activated mode, the conventional, dual-cure control cement demonstrated lower microhardness than the self-adhesive cements ($P < 0.05$). (*Am J Dent* 2010;23:14-18).

Clinical significance: Dual-cured, self adhesive resin cements showed an extent of polymerization comparable to the conventional, dual-cured resin cement tested.

✉: Prof. Lorenzo Breschi, Department of Biomedicine, Unit of Dental Sciences and Biomaterials, University of Trieste, Piazza Ospedale 1, I-34129 Trieste, Italy. E-✉: lbreschi@units.it

Introduction

Because of their enhanced mechanical properties, good esthetic qualities, and ease of handling, resin cements are widely used to lute fiber posts and composite or ceramic indirect restorations.[1] Because of the tremendous reduction in polymerization light reaching the resin cement, the so called "dual-cure" cements were developed.[2] Besides being able to photo-polymerize, these materials are characterized by their ability to undergo a totally dark, self-curing reaction in the absence of light.[3]

Recently, self-adhesive dual-cure cements were introduced in order to simplify the luting procedure. Besides the photo- and self-activating components, these materials contain acidic monomers and priming agents that cause tooth demineralization and hybridize the dentin surface simultaneously, without additional surface pre-treatment. Thus, etching, priming, bonding, and cementing are combined into one single product and one clinical step.

The acidic monomers in self-adhesive cements inhibit the amine co-initiator,[4] which in turn adversely affects polymerization of self-cured and dual-cured materials. Aromatic sulfinic acid sodium salts are sometimes added to avoid this inhibitory effect.[4,5] On a different topic, the adverse effect of hydrophilic monomers on polymerization kinetics has been advocated recently as one of the main reasons for the intrinsic instability of these simplified self-adhesive systems, in which highly acidic and hydrophilic monomers are usually blended in large quantities.

Using differential scanning calorimetry,[6,7] microhardness testing[6] and micro-Raman spectroscopy,[8] it was found that these simplified adhesives (one-step self-etch systems) exhibit lower degrees of conversion than do conventional adhesives (three- and two-step etch-and-rinse and two-step self-etch systems). It was speculated that the quality of the bonds created by these self-etch adhesives would degrade faster, since the presence of greater amounts of uncured monomer expedites water sorption and compromises hybrid and adhesive layer longevity.[9] Moreover, the low degree of conversion compromises mechanical properties.[10] With respect to this knowledge base, it may be speculated that self-adhesive cements would also demonstrate lower degrees of conversion than conventional resin-based cements.[11,12]

A variety of methods have been employed to determine the extent of resin monomer polymerization. Differential scanning calorimetry (DSC) is a direct method that analyzes the extent of polymerization based on the assumption that heat generated during resin polymerization (*i.e.* the exothermic heat of polymerization) is proportional to the amount of reacted monomer.[13-15] Ep represents how thorough the reaction process is,[16] *i.e.* the end of the curing reaction, while it does not correspond to the complete conversion of the C=C bonds.[17] In other words,the extent of polymerization represents how far the reaction can occur, relative to the tested resin blend.[16,17] Microhardness has been shown to be a simple and reliable indicator of double bond conversion,[18] and has been used as an indirect measurement of the extent of polymerization.[19,20]

This study compared the effect of utilizing either the light- or self-curing modes of dual-cure adhesive cements on the extent of polymerization and the microhardness, compared to a conventional, dual-cure cement (control). The research hypotheses tested for each cementing system were that (1) the extent of polymerization and microhardness when light-activating the dual-cured, self-adhesive cements was greater than when letting

Table 1. Composition of the tested cements (supplied by manufacturer).

Maxcem	Glycerol phosphate dimethacrylate (GPDM) co-monomers (mono-, di- and tri-functional methacrylate monomers, water, acetone and ethanol, camphorquinone-based photo-initiator system, proprietary redox initiators *Fillers:* barium glass fillers, fumed silica, sodium hexafluorosilicate (66 wt%)	***Self-adhesive one-step*** 2 paste/ "auto-mix" dual barrel syringe
RelyX Unicem	Methacrylated phosphoric ester, dimethacrylate, pigment, substituted pyrimidine, peroxy compound, sodium persulfate, acetate, initiator,stabilizer *Fillers:* glass powder, fumed silica, calcium hydroxide, (72 wt%)	***Self-adhesive one-step*** Capsule/mechanically mixed
Panavia F2.0	*ED Primer II A&B:* HEMA, 10-MDP, 5-NMSA, water, accelerator, sodium benzene sulphinate	
	Cement only: 10-MDP, 5-NMSA, BPEDMA, BPO, dimethacrylates, dibenzoyl peroxide, sodium aromatic sulfinate, N,N-diethanol p-toluidine, photoinitiators	***Dual-cure*** 2 paste/ hand-mixed
	Fillers: barium glass, silanized silica, sodium fluoride (70.8 wt%)	

the cements self-cure only, and that (2) the extent of polymerization and microhardness of the self-adhesive cements were lower than those of the conventional, dual-cure material (control), regardless of mode of polymerization activation.

Materials and Methods

Two commercial self-adhesive cements were tested: Maxcem[a] and RelyX Unicem.[b] Additionally, a conventional, dual-cured resin cement, Panavia F2.0[c] was used as a control. Cement compositions are reported in Table 1.

Measuring the extent of polymerization - A "heat flux" Q10 differential scanning calorimeter[d] (DSC) was used at a constant temperature (35°C), and in a nitrogen atmosphere to avoid oxygen inhibition. Two aluminum pans (diameter = 6.5, 1.2 mm thick) were placed on the sample platforms of the instrument. The first pan was filled with 10 mg of cement material as weighed by an analytical balance (A&D HR Series,[e] resolution: 0.1 mg ~ 0.01 mg), previously mixed and prepared following the manufacturer instructions. The second pan was left empty, acting as a thermal reference. An aluminum cover with an 8 mm diameter quartz window was employed to allow light to pass through and to permit specimen photo-curing inside the DSC chamber. During photo-polymerization, the halogen curing-unit tip (Elipar 2500[b]) with an output intensity of 600 mW/cm^2 was positioned 5 mm from the target pan using a custom made support, which allowed full irradiation of the adhesive-containing pan. The output of the curing unit was verified before testing with a radiometer.[a]

Polymerization of the dual-curable cements was performed under two different conditions:

1. The specimen was immediately irradiated for 20 seconds, after which the DSC was maintained isothermally for 2 hours.
2. The specimen was not exposed to the curing light, and the DSC was maintained isothermally for 2 hours to detect heat derived only from the self-curing chemical reaction.

A second DSC analysis was performed in order to evaluate the amount of monomer that did not react, but that still potentially could. For this analysis, the same specimens in Groups 1 and 2 were irradiated with an additional 2 minutes of light exposure. A third irradiation (2 minutes) showed that no supplementary reaction occurred either in light-cured and self-cured specimens, thus the heat obtained from this last irradiation represented the heat generated only by the light unit and was subtracted from the previous DSC curves,[6] while the heat of reaction obtained from the first 2-minute exposure could be considered as the maximum reaction attainable for each cement.

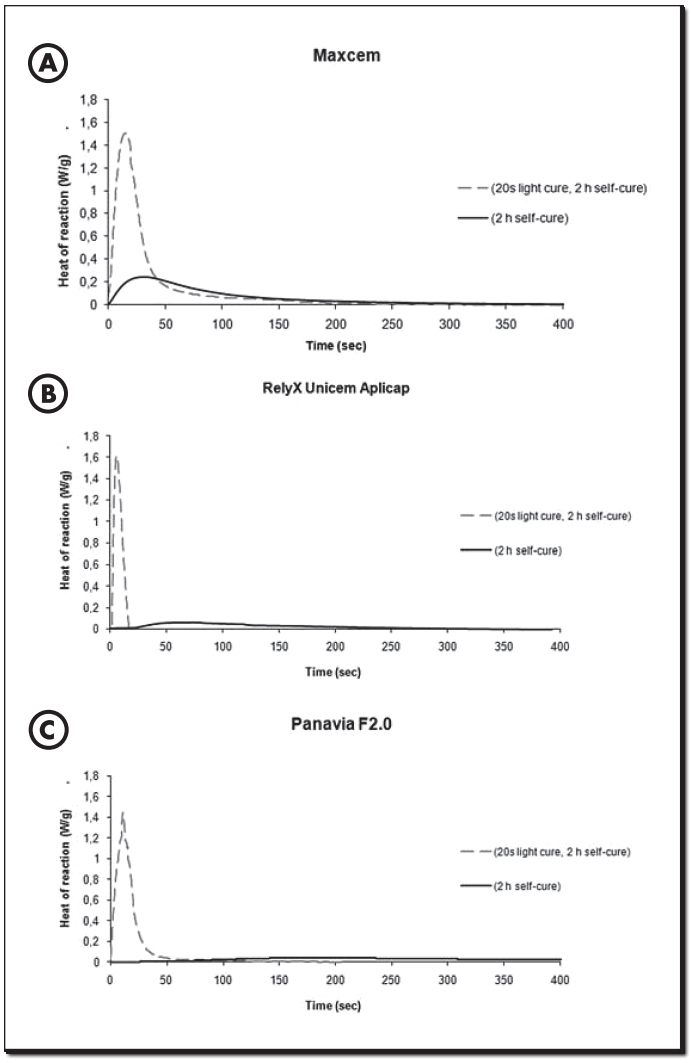

Figure. **A-C**. Representative DSC curves of the cements tested in different curing modes. A narrow and high peak was obtained with light-cure mode, while self-cured specimens showed a smaller and broader peak related to the slower speed of reaction.

The exothermic heat of reaction due to cement polymerization was evaluated as the integral of heat flux detected from the DSC over the time of observation, as described from the following equation:

$$H_p = \int_0^t W(t)dt \qquad (1)$$

Where: H_p = exothermic heat from the polymerization reaction; W(t) = heat flux detected from the DSC; t = time; dt = time differential.

In such a way, the extent of polymerization (Ep) after light-cure or self-cure was evaluated as the relative percentage of the

Table 2. Extent of polymerization (%) (mean ± sd) for the tested materials in the two different curing modes. N = 10/group.

Cement	Initial 20 seconds light 2-hour self-cure	2-hour self-cure
Maxcem	54.6 ± 1.9[a]	40.5 ± 3.8[b]
RelyX Unicem	54.4 ± 1.4[a]	31.7 ± 1.4[c]
Panavia F2.0	48.6 ± 1.1[a]	31.4 ± 1.8[c]

Values having same superscript letter are not significantly different.

maximum reaction attainable for each cement according to the following equation:

$$E_p = \frac{H_{1p}}{H_{1p} + H_{2p}} \cdot 100 \qquad (2)$$

where H_{1p} represents the exothermic heat detected in the first calorimetric analysis and H_{2p} represents the value obtained in the second DSC analysis.

Ten specimens (N=10) were prepared for each luting agent for each group.

Microhardness testing - Vickers microhardness measurements (VMHT) were obtained using a 50g load applied for 15 seconds.[f] New cement specimens (N=10 for each luting agent for each group) were prepared and polymerized as described in the above DSC analysis section. Following that treatment, the microhardness of the exposed surface was taken at three randomized locations. These values were averaged, reporting a single Vickers hardness for each specimen.

Statistical analyses - A two-way ANOVA was used to evaluate the DSC and microhardness data. The two variables were resin cement and mode of cure. Differences between groups were identified using Tukey's multiple comparison test at $\alpha = 0.05$. Correlations among microhardness and the extent of polymerization (Ep) were analyzed using the Pearson product moment correlation test at $\alpha = 0.05$.

Results

Differential scanning calorimetry - The figure shows the DSC curves of the two modes of cure (photo- and self-cure) of the three tested cements. For all materials light-cure produced an initial high and narrow peak rapidly decreasing after light exposure, while in the self-cured specimens the peak was lower and broader.

Extent of polymerization of all cements was higher when both the photo-activated and self-activated modes were used ($P < 0.05$; Table 2) in comparison to the self-cure polymerization alone. No difference was found among the three cements polymerized when using the light-activation mode. RelyX Unicem and Panavia F2.0 polymerized using only the self-cure mode showed lower values than Maxcem ($P < 0.05$; Table 2).

Microhardness evaluation - Microhardness values of all cements were higher when the light activation of the dual-cured material was utilized ($P < 0.05$; Table 3). Panavia F2.0 showed the lowest values in the light-activation mode, while RelyX Unicem showed the lowest microhardness in the self-cure mode ($P < 0.05$; Table 3).

Pearson product moment correlation test showed a significant ($P < 0.05$) correlation between extent of polymerization and microhardness for all cements for each curing condition.

Table 3. Vickers microhardness (mean ± sd) for each tested material in the two different curing modes. N = 10/group.

Cement	Initial 20 seconds light 2-hour self-cure	2-hour self-cure
Maxcem	43 ± 6[a]	30 ± 5[c]
RelyX Unicem	44 ± 5[a]	26 ± 3[c]
Panavia F2.0	37 ± 4[b]	31 ± 3[c]

Values having same superscript letter are not significantly different.

Discussion

The first hypothesis stating that extent of polymerization of all tested cements would be higher when the dual-cure systems were initially light-activated compared to self-cure mode was accepted. These observations were in accordance with previous data[11,12,21,22] showing that self-cure polymerization leads to lower degrees of conversion values than does utilizing the light-curing mode. As the tested cements are marketed for cementation of fixed prostheses, inlays, or endodontic posts (*i.e.* clinical conditions in which light activation is dramatically attenuated or even absent), the results of this study suggest that all these cements may be inadequately polymerized if no light activation can be performed.

The current study is the first to report on the extent of polymerization of Maxcem. On the other hand, previous studies tested the degree of polymerization of RelyX Unicem and Panavia F2.0.[11,12] When testing these products in self-cure mode, lower degrees of polymerization were found than those obtained in the current study. However, the duration of time the materials were allowed to self-cure in the present study was longer (2 hours) than the 15 minutes used in the other studies.[11,12] Conversely, those same studies described higher polymerization values of RelyX Unicem and Panavia F2.0 when polymerized in the light-cure mode than those obtained in the present investigation. This finding may be explained by the reduced irradiation time (20 seconds) used in the current study (a value that was in accordance with manufacturers' instructions) compared to the 40-second duration used by the previous investigations above.

The extent to which monomer interacts to form polymers is expected to affect the properties of dental resins. Several studies report a direct correlation between the degree of monomer conversion and the resulting mechanical properties of dental restorative resins.[19,20,23]

Similarly, in the current research, materials showed lower microhardness values when left to self-cure without light activation. This finding has also been reported by others.[24] Microhardness data are comparable only within the same resin system,[25] as they are not linearly correlated to the degree of cure if compared across different materials. Thus, the data derived from the present study were useful in comparing the polymerization achieved with different curing modes in a particular cement.[26] The results supported the hypothesis that the light-activation mode of all tested dual-cured resin cements improved their microhardness by increasing the extent of polymerization compared to using only the self-cure polymerization mode.

In addition, extent of polymerization of the self-adhesive cements was comparable to that of the conventional, dual-cure

material, both when using light-activation and when relying totally on the self-cure mode. Thus, the second tested hypothesis was rejected. Deactivation of the tertiary amine used in the chemically-cured resins by acidic resin monomers accounts for the reported incompatibility between acidic monomers and chemically-cured materials. Addition of aromatic sulfinic acid sodium salts, however, may counteract this negative effect and allow an adequate degree of conversion of self-cured materials, even in the presence of acidic monomers.[5] Among the tested materials, RelyX Unicem contains sodium persulfate, which may explain the comparable results with the non-acidic, dual-curing resin cement control (Panavia F2.0), whereas no information is provided by the manufacturer for Maxcem related to presence of these salts in its formulation.

Data from the current work were not in accordance with previous studies[11,12] that revealed lower degrees of conversion for RelyX Unicem than for Panavia F2.0 using Fourier transformed infrared spectroscopy. It should be stressed that the current work utilized DSC instead. The previous studies cited looked only at reaction of the monomer C=C units, whereas DSC is insensitive to the type of functional group undergoing the reaction process. Thus, any other types of simultaneous, secondary reactions, such as a glass-ionomer one, would be detected by the DSC, but unless investigators specifically monitored that reaction during IR analysis, it would be missed. Indeed, an additional acid-base setting reaction has been reported for RelyX Unicem, since its acidic monomers are claimed to chemically interact with the basic inorganic fillers of the cement.[27] Similar information is not provided for Maxcem.

Previous observations reported that Maxcem is characterized by a poor bonding ability to both enamel and dentin, independently[28,29] and that the product has a limited potential for removal or modification of the smear layer and for resin infiltration into the underlying dentin.[28] On a similar note, RelyX Unicem has been shown to produce an inadequate hybrid layer[30-32] and a low bond strength to enamel,[30,33,34] while its bond strength to dentin seems to be comparable to other resin cements.[33,34] These findings suggest that the etching potential of self-adhesive cements may be insufficient.[28] Low bond strength may also depend on the incorporation of acidic monomers in these self-adhesive cements, which may have interfered with formation of an optimal cross-linked polymer network. Thus, even though the overall extent of polymerization might have been high, the fragility of the polymer network formed would have conferred low mechanical properties and inadequate bond strength results. Under this perspective, use of the extent of polymerization as a predictor of the properties of resin cements, and thus of its potential for clinical success, may be not totally unequivocal.

Further studies are needed to evaluate if the extent of polymerization, as measured using DSC, of the recently introduced self-adhesive cements may affect their bonding ability and influence their long-term stability in the oral environment.

a. Kerr, Orange, CA, USA.
b. 3M ESPE, St. Paul, MN, USA.
c. Kuraray Medical, Tokyo, Japan.
d. TA Instruments, New Castle, DE, USA.
e. A&D, Tokyo, Japan.
f. Leica Microsystems, Milan, Italy.

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Dr. Cadenaro is Associate Professor, Dr. Navarra is Post-doctoral Researh Fellow, Dr. Antoniolli is Post-doctoral Research Fellow, Dr. Mazzoni is Post-doctoral Fellow, Department of SAU & FAL, University of Bologna, Bologna, Italy. Dr. Lenarda is Full Professor, Department of Biomedicine, Unit of Dental Sciences and Biomaterials, University of Trieste, Trieste, Italy. Dr. Rueggeberg is Full Professor, Department of Oral Rehabilitation, Medical College of Georgia, Augusta, GA, USA. Dr. Breschi is Associate Professor, Department of Biomedicine, Unit of Dental Science and Biomaterials, University of Trieste, Trieste, Italy.

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