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Curing efficiency of four self-etching, self-adhesive resin cements

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ABSTRACT

Objectives. To evaluate the degree of cure (%DC) of four self-etching, self-adhesive resin cements, and one conventional resin cement, in their self- and dual-curing mode.

Methods. The self-etching, self-adhesive resin cements studied were RelyX™ Unicem (3M™ ESPE™ AG), Maxcem™ (Kerr Corporation), Biscem™ (Bisco, Inc.) and Multilink® Sprint (Ivoclar Vivadent® AG) and the classic resin cement was Multilink® Automix (Ivoclar Vivadent® AG). Twelve specimens of each material (1.8 mm × 4 mm × 4 mm) were prepared in room temperature (23 ± 1) °C following the manufacturers' instructions. Six of them were treated as dual-cured, thus irradiated for 20 s with a halogen light curing unit and left undisturbed for 5 min. The other six were treated as self-cured and were not irradiated, but left in dark and dry conditions for 10 min. The assessment of the %DC was made using micro-ATR FTIR spectrometry.

Results. The %DC in their self-curing mode was very low (10.82–24.93%), with Multilink Sprint exhibiting the highest values among the five. In the dual-curing mode the values obtained were also low (26.40–41.52%), with the exception of Multilink Automix (61.36%). Maxcem was found to have the lowest DC.

Significance. The low %DC found raises questions as to whether these materials can be successfully used in clinical applications, where light attenuation takes place. Increased irradiation times could potentially lead to higher %DC, in applications where light is not completely blocked by the overlying restoration.

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1. Introduction

Resin cements have an increasing application in the cementation of fixed prostheses [1], since they exhibit enhanced mechanical, physical and adhesive properties, compared to conventional luting agents [2]. Further, they provide adequate stability [3,4] and increased fracture resistance of overlying all-ceramic restorations [5,6], together with an optimal esthetic

result. However, the fact that they are technique-sensitive materials [7] complicates clinical procedures and makes the cementation time-consuming and susceptible to manipulation errors.

In an attempt to simplify procedures, a new group of resin cements, the self-etching, self-adhesive resin cements, have been introduced. According to their manufacturers, these products are self-adhesive, including acidic and hydrophilic

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monomers in their composition, which simultaneously demineralize and infiltrate enamel and dentin, resulting in strong bonding. Therefore, they require no conditioning or priming pretreatments of tooth substrate [8–11]. Furthermore, in the case of one of those materials (RelyX™ Unicem), the phosphate groups of the functionalized monomers included in the monomer mixture are claimed to react with the hydroxyapatite of the tooth substrate, resulting in additional retention through chemical bonding. Indeed, a chemical interaction between the cement and the Ca⁺ of hydroxyapatite has been reported [12]. Moreover, these acidic monomers are claimed to interact chemically with the basic inorganic fillers of the material, leading to an additional acid–base setting reaction, apart from the free radical polymerization of the material [10]. Information for this type of chemical reactions is not provided for the other materials of this group, which are currently available on the market.

The literature regarding self-etching, self-adhesive cements, concerns mainly RelyX™ Unicem, since it is the first material of this group which has been launched on the market. The majority of the studies conducted, concern the bonding effectiveness of RelyX™ Unicem, which has been found to have low demineralization capacity, since it seems to interact only superficially with dentin, failing the formation of a hybrid layer or resin tags [13–15]. Nevertheless, its bond strength to dentin has been found to be comparable to other widely used resin systems [13,16–18]. When bonding to enamel [13,17–21] and to root dentin [22–25], however, RelyX™ Unicem has been found to be less effective. Maxcem™, another material of this group, has been found to have a relatively poor bonding ability, irrespective of the tooth substrate [26,27]. No literature is available regarding other materials of this group.

The literature concerning other properties of self-etching, self-adhesive resin cements, such as the curing efficiency of these materials [28,29], is limited. It has been reported that low degrees of conversion (%DC) result in inferior clinical performance [30], in terms of ultimate hardness [31], fracture toughness [32], wear resistance [33], elastic modulus [34], solubility and hydrolytic degradation [35,36], as well as biocompatibility [37,38].

Therefore, the purpose of this study was to evaluate the %DC of four self-etching, self-adhesive resin cements, and one conventional resin cement, in their self- and dual-curing mode. The testing hypothesis was that there are no significant differences in the %DC, among the materials tested.

2. Materials and methods

Four self-etching, self-adhesive resin cements and one conventional resin cement were used in the present study. The materials, their batch numbers and their composition, are listed in Table 1.

In order to evaluate the %DC using both chemical and light activation modes, six specimens (1.8 mm × 4 mm × 4 mm) of each material were prepared. Plastic molds were placed on glass microscope slides, covered with transparent celluloid matrix and overfilled with resin cement, following manufacturers' instructions. The overfilled molds were covered with transparent celluloid matrix and a glass microscope slide, and pressed with finger pressure to remove the excess. Finally, each specimen was irradiated for 20 s, in accordance with the manufacturers' instructions. The light curing device used was a halogen curing light (Elipar™ Trilight, 3M ESPE) in a standard irradiation mode, emitting light intensity 850 mW cm⁻². Then, the specimens were left undisturbed for 5 min, for the completion of the polymerization reaction. The whole procedure took place at room temperature (23 ± 1) °C.

For the assessment of the chemical activation mode in the %DC, six specimens of each material were prepared. Specimen preparation was made as above, except that they were not light-cured; instead they were placed in dark and dry conditions for 10 min, a clinically relevant time which corresponds to the setting times given by the manufacturers, and for the majority of the materials is almost twice the setting time proposed.

The method used for the assessment of the %DC was the micro-attenuated total reflectance Fourier transform infrared spectrometry (micro-ATR FTIR), which is a well established technique in the relevant literature [39,40,15,41]. An FTIR spectrometer was used (Spectrum GX PerkinElmer Corp., Bea-

Table 1 – The materials used in the study and their composition according to their manufacturers.

Resin cement	Batch number	Type	Composition (wt%)
RelyX Unicem™ (3M ESPE AG); code: RXU	#280614	Self-etching, self-adhesive dual-cured resin cement	Monomer: methacrylated phosphoric acid esters (15–25), TEGMA (10–20) fillers (72)
Maxcem™ (Kerr Corporation); code: MXC	#457614	Self-etching, self-adhesive dual-cured resin cement	Monomer: GPDM (20–35), comonomers fillers (67)
Bisцем™ (Bisco, Inc.); code: BCM	#0700002465	Self-etching, self-adhesive dual-cured resin cement	Monomer: bis(hydroxyethyl methacrylate) phosphate (10–30), TEGMA (10–30) fillers (40–70)
Multilink Sprint® (Ivoclar Vivadent® AC) ^a ; code: MLS	#J11950	Self-etching, self-adhesive dual-cured resin cement	Monomer: dimethacrylate, methacrylated phosphoric acid ester (5.0) fillers (74.1)
Multilink Automix® (Ivoclar Vivadent® AG); code: MLA	#J19499	Dual-cured resin cement	Monomer: dimethacrylates (22–26), HEMA (6–7) fillers (40)

^a Multilink Sprint discontinued (Fall 2007).

consfield, UK), equipped with a horizontal single-reflection ATR attachment (Golden Gate Mk II, Specac, Smyrna, GA, USA) operated under the following conditions: 4000–500 cm^{-1} range, 4 cm^{-1} resolution, 20 scans coaddition, 2 mm diameter diamond ATR crystal, ZnSe lenses, depth of analysis $\sim 2 \mu\text{m}$ at 1000 cm^{-1} .

The specimens were placed one at a time in the sample holder of the device and spectra were recorded. A small amount of uncured resin cement from each material was also scanned and its spectrum was used as unpolymerized reference. The %DC was calculated by the two frequency technique, using the absorption peak of C=C groups at 1638 cm^{-1} (analytical frequency), and the absorption peak of the aromatic C...C groups at 1608 cm^{-1} (reference frequency), according to the equation:

$$\%DC = 100[1 - (Aa_{(C=C)}Ab_{(C...C)} / Ab_{(C=C)}Aa_{(C...C)})]$$

where Aa and Ab the net peak absorption areas after and before polymerization at the specific frequencies, respectively.

For the calculation of the %DC in MXC, the absorption peak of the C=O ester groups was used as reference, no aromatic peaks were identified. In this case, the %DC was calculated as follows:

$$\%DC = 100[1 - (Aa_{(C=O)}Ab_{(C=C)} / Ab_{(C=C)}Aa_{(C=O)})]$$

2.1. Statistical analysis

Descriptive statistics and means of the measurements with 95%-CI were used to illustrate the results. Balanced analysis of variance (ANOVA) was used for testing the group effect. For the comparison of the %DC between self-cured and dual-cured groups per material, multiple comparison of pairwise differences was done. The multiple testing problem was controlled with the method of BONFERRONI-HOLM. All computations were performed with the statistical software SAS system version 9.1.

3. Results

The mean and standard deviation values of the %DC in the self-curing mode are presented in Table 2. MLS exhibited the highest %DC, statistically significantly different from all ($p < 0.05$). No statistically significant differences in %DC were found between RXU, MXC, BCM and MLA ($p > 0.05$).

The results of the %DC in the dual-curing mode are demonstrated in Table 3. MLA exhibited again the highest %DC

Table 2 – Results of the %DC in the self-curing mode. Same letters indicate mean values with no statistically significant differences.

Group	%DC self-curing mode (mean \pm SD)
RXU	11.05 \pm 4.16 a
MXC	14.32 \pm 4.95 a
BCM	10.82 \pm 5.51 a
MLS	24.93 \pm 5.59 b
MLA	14.47 \pm 6.92 a

Table 3 – Results of the %DC in the dual-curing mode. Same letters indicate mean values with no statistically significant differences.

Group	%DC dual-curing mode (mean \pm SD)
RXU	37.27 \pm 5.01 a
MXC	26.40 \pm 4.19 b
BCM	41.52 \pm 15.56 a
MLS	40.39 \pm 9.03 a
MLA	61.36 \pm 6.23 c

($p < 0.05$) of all the materials tested. MXC exhibited the lowest %DC, significantly lower than the values of RXU, BCM and MLS ($p < 0.05$). No statistically significant difference was found among RXU, BCM and MLS ($p > 0.05$).

Statistical analysis has shown a highly significant difference in the %DC ($p < 0.0001$), between the two curing modes. Significant differences ($p < 0.05$) were found in the %DC between the self-cured and the dual-cured groups of MXC ($p: 0.0026$), BCM ($p: 0.0034$) and MLS ($p: 0.0071$), whereas the difference was highly significant ($p < 0.0001$) in the case of RXU and MLA. As shown in Fig. 1, in every case, the %DC was higher for the dual-cured groups.

4. Discussion

According to the results of the present study, the testing hypothesis that there are no significant differences in the %DC among the materials tested, should be rejected.

The %DC of the materials tested in the self-, as well as in their dual-curing mode, was much lower than the one expected, according to the literature. Indeed, the %DC of dual-cured luting agents has been reported to range from 59.3% to 75.0% in the self-curing mode and from 66.6% to 81.4% in their dual-curing mode of polymerization [41].

All self-etching, self-adhesive cements tested contain acidic monomers in their composition. Acidic monomers have been shown to negatively affect the %DC of dual-cured materials, especially in their self- but also in their dual-curing mode of polymerization, since they seem to interact chemically with the amine initiator, dual-cured resins contain. To overcome this incompatibility, proprietary activator/initiator systems should be included in their composition, like sodium aryl sulfate or aryl-borate salts [42]. The use of this different type of initiation system, however, might result in a different polymerization behavior, which may involve low initial %DC values [29].

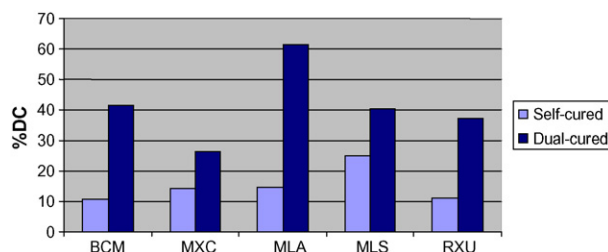


Fig. 1 – Comparison of the %DC between self-cured and dual-cured groups per material.

Among the materials studied, only RXU is known to contain sodium persulfate, whereas no information is available for MXC, BCM and MLS. Therefore, the low %DC found in the present study, could be attributed either to the presence of a different initiation system, which modifies the polymerization behavior, or to the absence of such, in which case incompatibility of the acidic monomers and the amine initiator occurs [26,43].

In agreement with the data reported in the literature [28,29,44], the %DC the materials exhibited in the present study, was much lower in the self-curing mode than in the dual-curing mode. The extent, to which the mode of activation affects the %DC, is related to the initiation system each material contains, which could favor chemical or light activation. This was more obvious in the case of RXU and MLA, which showed a highly significant difference when the two ways of activation were compared.

It should be mentioned that there is no literature regarding the %DC of the four new materials examined in the present study, with the exception of RXU. Kumbuloglu et al. [28] and Tezvergil-Mutluay et al. [29] measured the %DC of RXU in its self-curing mode, and they found it to be 26% and 28.7%, respectively. These findings do not agree with the findings of the present study (11.05%), however, the difference may be attributed to the increased time the material was allowed to set in the first two studies, which was 15 min, in comparison to the time of 10 min in the present study. Further, the %DC of RXU in the dual-curing mode, as measured in the above studies, was found to be 58% and 54.9%, respectively, values which do not agree with the %DC obtained in the present study (37.27%). This may be attributed to the increased irradiation time of 40 s they used, compared to the 20 s used in the present study. It can, therefore, be concluded that the irradiation time proposed by the manufacturers is inadequate. This might be the reason for the low %DC in their dual-curing mode, which all the materials tested exhibited [45].

It should be noted that all specimens were prepared at room temperature (23 ± 1) °C instead of body temperature (37 ± 1) °C. Since higher temperature increases the mobility of the molecules during chemical reactions, the %DC of the materials tested might be higher during clinical application. Increased setting time might have also resulted in higher %DC. According to Santos et al. [46] the Knoop hardness, which provides an indirect indication of the %DC, of three dual-cured resin cements at 15, 30, 60 min and 24 h after mixing, increased significantly after 24 h. Therefore, it could be assumed that the setting time of 10 min used in the present study corresponds to the initial setting phase. However, the materials tested are luting agents placed in thin layers between two rigid components (crown material and dentin). Initial setting is of paramount importance for the integrity of the restoration. The period of 10 min is considered to be an adequate and clinically relevant setting time period for a luting agent to obtain a great percentage of the optimal setting characteristics. Otherwise, intraoral functional loading may be detrimental to a slow setting material integrity. Moreover, for materials demonstrating these setting reactions (acid–base and free-radical) as the self-etching, self-adhesive cements, hardness measurements cannot be used as a reliable method of assessment of post-curing conversion since the values recorded are implicated

from the slow neutralization rate of the acid–base reaction.

To date, a minimum acceptable %DC has yet to be established. However, taking into consideration the values suggested by Harashima et al. [41], a comparison of the %DC found in present study with the expected values, could be made.

When it comes to the %DC the materials exhibited without light activation, it is quite obvious that the values obtained were very low, far from the expected level. This finding suggests that, all the cements tested may demonstrate inadequate early strength in applications where light is not likely to reach the cement, such as cementation of endodontic posts, metal–ceramic or all-ceramic opaque restorations or even inlays of increased thickness. In the dual-curing mode of polymerization and when light attenuation does not occur, MLA appears to have %DC which is closer to the expected values after 20 s of irradiation, whereas RXU, MXC, BCM and MLS fail to reach the expected level using this time. An increase in the irradiation time may probably result in higher %DC.

It should be noted that all the materials tested are used for the cementation of fixed prostheses so, during their clinical applications, there will always be an overlying restoration which would lead to light attenuation of some degree. Therefore, questions may rise as to whether this performance would be deteriorated during their clinical use, especially at the early setting stages after bonding. Further studies are necessary in order to provide more information on this issue.

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