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Dual and Self-curing Potential of Self-adhesive Resin Cements as Thin Films

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Clinical Relevance

Self-adhesive resin cements present slower polymerization and lower degree of conversion than conventional resin cements, in either the dual- or self-cure mode.

SUMMARY

In this study, the dual- and self-curing potential of self-adhesive resin cements (SARCs) as thin, clinically-relevant cement films was investigated. The SARCs tested were: BisCem (BSC; Bisco), Maxcem Elite (MXE; Kerr), RelyX Unicem clicker (UNI; 3M ESPE), seT capsule (SET; SDI), and SmartCem 2 (SC2; Dentsply Caulk). The conventional cement RelyX ARC (3M ESPE) was tested as a reference. The degree of conversion (DC) as a function of time was evaluated by real-time Fourier transform infrared spectroscopy with an attenuated total reflectance (ATR) device. The cements were either photoactivated for 40 seconds (dual-cure mode) or not photoactivated (self-cure mode). The cement film thickness was 50 ± 10 μm. The DC (%) was evaluated 1, 5, 10, 15, 20, 25, and 30 minutes after placing the cement on the ATR cell. Data for DC as a function of time were analyzed by two-way repeated measures analysis of variance (ANOVA). DC values at 30 minutes for the self- and dual-cure modes were submitted to one-way ANOVA. *Post hoc* comparisons were performed using the Student-Newman-Keuls test ($p<0.05$). The rate and the extent of conversion were lower for the SARCs compared with the conventional cement. Means ± standard deviations (SD) for the dual-cure mode at 30 minutes were: 75 ± 5 (ARC)^{a}, 73 ± 8 (SET)^{a}, 61 ± 4 (MXE)^{b}, 51 ± 9 (BSC)^{c}, 51 ± 4 (UNI)^{c}, and 48 ± 3 (SC2)^{c}, while in the self-cure mode means and SD were 62 ± 6 (ARC)^{a}, 54 ± 3 (MXE)^{b}, 40 ± 6 (SC2)^{c}, 35 ± 2 (UNI)^{c}, 35 ± 3 (SET)^{c}, and 11 ± 3 (BSC)^{d}. The DC for the dual-cure mode was generally

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higher than the self-cure, irrespective of the time. Discrepancies in DC between the dual- and self-cure modes from 11% to 79% were observed. In conclusion, SARCs may present slower rate of polymerization and lower final DC than conventional resin cements, in either the dual- or self-cure mode.

INTRODUCTION

Self-adhesive resin cements (SARCs) were introduced in the past few years in an endeavor to simplify the adhesive luting procedures by eliminating the etching, priming, and bonding steps. The use of SARCs should also prevent incomplete infiltration of dentin and reduce the occurrence of postoperative sensitivity. The adhesive properties of SARCs are attributed to acidic methacrylate monomers that simultaneously demineralize and infiltrate the tooth substrate, resulting in micromechanical retention. Secondary reactions have been suggested to provide additional chemical bonding to the hard dental tissues.[1-4]

The basic F-Al-Si–based inorganic fillers in self-adhesive cements are able to react with the monomers with acid functionalities.[1,5] The dominant setting reaction, however, occurs via free-radical polymerization, initiated either by light or a redox system that allows resin polymerization in an acidic environment. The time necessary to obtain proper polymerization of the cement is critical. A fast polymerization reaction may avoid water from the dentin to access through the unpolymerized cement film and interfere with the bonding performance or further polymerization of the luting agent. Immediate cement polymerization may also allow occlusal adjustments and loading without interfering with the cement layer.

Several studies have investigated the bonding effectiveness of SARCs, with results even comparable to conventional resin-based cements.[6-10] The curing mechanism of SARCs, on the other hand, has been evaluated rarely. Few studies have assessed the C=C degree of conversion (DC) of these cements.[11-14] A recent study has reported that the curing efficiency of SARCs is far from adequate.[14] However, most of those previous studies tested thick cement specimens, which do not reflect the actual clinical scenario. Thick specimens may not allow adequate light penetration within the bulk of the cement, interfering especially with the dual-curing mechanism.[15,16] Therefore, different outcomes could be observed for thin cement films.

The purpose of this study was to investigate the polymerization of five commercial SARCs as thin films, in either dual- or self-cure modes, compared with a conventional dual-cure resin cement. The hypotheses tested were:

1) For each material, the DC as a function of time would be higher for the dual- compared with the self-cure mode;
2) The DC at 30 minutes would be lower for the SARCs compared with the conventional resin cement, regardless of the curing mode.

MATERIALS AND METHODS

Five dual-cure SARCs were tested: BisCem (BSC; Bisco), Maxcem Elite (MXE; Kerr), RelyX Unicem clicker (UNI; 3M ESPE), seT capsule (SET; SDI), and SmartCem 2 (SC2; Dentsply Caulk). The conventional dual-cure resin cement RelyX ARC (3M ESPE) was tested as a reference. Composition of the materials is shown in Table 1.

The DC of the resin cements as a function of time was evaluated using real-time Fourier transform mid-infrared spectroscopy (Prestige21; Shimadzu, Tokyo, Japan) with an attenuated total reflectance device (ATR) composed of a horizontal multiple-reflection ZnSe crystal with 45° mirror angle (Pike Technologies; Madison, WI, USA). Equal volumes of base and catalyst pastes were mixed for 10 seconds; the capsules of SET were mixed for 10 seconds using the Ultramat S mixer (SDI). After manipulation, the cement was placed on the ATR cell and covered with a 1-mm thick glass slide to avoid oxygen inhibition and to obtain a thin, clinically relevant cement film. Pressure was exerted to maintain the specimen in intimate contact with the crystal. The diameter of the specimens was restricted to match the diameter of the light guide (10 mm).

A preliminary reading for the unpolymerized material (monomer) was taken under the following conditions: 24 scans co-addition, 4 cm^{-1} resolution, Happ-Genzel apodization, and 2.8 mm/s mirror speed. After the monomer reading, the resin cement was immediately light-activated (dual-cure mode) for 40 seconds using a quartz-tungsten-halogen curing unit (XL3000; 3M ESPE) with 500 mW/cm^2 irradiance. A support was coupled to the spectrometer to hold the curing unit and standardize a 5-mm distance between the fiber tip and cement film. In the self-cure mode, no light-activation procedure was carried out. For both curing modes, the material was shielded from the ambient light throughout the experiment.

Table 1: *Materials Used In the Study*

Material	Manufacturer	Description*	Lot Code	Main Components*
BisCem	Bisco, Schaumburg, IL, USA	Self-adhesive resin cement	0800010947	Bis-GMA, dimethacrylates, phosphate acidic monomer, glass particles
Maxcem Elite	Kerr, Orange, CA, USA	Self-etch, self-adhesive resin cement	2994811	TEGDMA, inert mineral fillers, ytterbium fluoride, activators, stabilizers
RelyX Unicem	3M ESPE, St Paul, MN, USA	Self-adhesive resin cement	331271	TEGDMA, substituted dimethacrylate, methacrylated phosphoric acid esters, calcium hydroxide, sodium persulfate, sodium p-toluene sulfinate, glass powder, silica
seT	SDI, Bayswater, Victoria, Australia	Self-etch, self-adhesive resin cement	S0810282	Acidic monomer, UDMA, F-Al-Si glass particles
SmartCem2	Dentsply Caulk, Milford, DE, USA	Self-adhesive resin cement	0901212	UDMA, urethane modified Bis-GMA, TEGDMA, dimethacrylate resins, DPEPAP, Ba-B-F-Al-Si glass particles, amorphous silica
RelyX ARC	3M ESPE, St Paul, MN, USA	Adhesive resin cement	FW8HU	TEGDMA, Bis-GMA, functionalized dimethacrylate polymer, ceramic, silica

** As provided by the manufacturers.*
Abbreviations: Bis-GMA, bisphenol-A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; DPEPAP. dipentaerythritol penta-acrylate phosphate.

The DC was evaluated in the absorbance mode 1, 5, 10, 15, 20, 25, and 30 minutes after placing the resin cement on the ATR cell. Calculation was carried out using a baseline technique,[17] considering the intensity of C=C stretching vibration (peak height) at 1635 cm^{-1} and as an internal standard, using the symmetric ring stretching at 1608 cm^{-1}. The percent of DC at each time point was calculated as previously described.[16,18] For calculating the DC in MXE, SET, and SC2, the absorption peak of the C=O ester groups (at 1716 cm^{-1}) was used as internal reference,[14] as no appreciable aromatic peaks were identified, as shown in Figure 1. The cement thickness was measured after polymerization using a digital caliper accurate to 0.001 mm; the specimens were eliminated where the cement thickness was in excess of 50 ± 10 μm. The resulting thickness was relevant to a cement film in clinical luting procedures.

For each curing mode, five specimens per material were tested under controlled temperature (25°C ± 2°C) and humidity (45% ± 5%) conditions. Data from each material were analyzed separately by two-way repeated measures analysis of variance (ANOVA) (curing mode vs time) followed by Student-Newman-Keuls as a *post hoc* test ($p<0.05$). Additionally, DC values at 30 minutes, for the self- and dual-cure modes, were separately submitted to one-way ANOVA and Student-Newman-Keuls test ($p<0.05$).

RESULTS

Results for DC for all cements are shown in Table 2. The factor “curing mode” was significant for all materials ($p<0.001$), except for SC2 ($p=0.07$). The factor “time” was significant for all materials ($p<0.001$). Similarly, the interaction “curing mode × time” was significant for all materials ($p\leq0.011$).

In the dual-cure mode, a significant increase in DC was detected up to 10 minutes for BSC, UNI and SET, 20 minutes for MXC, and 25 minutes for SC2. The conventional material showed significant increase in conversion only up to 5 minutes after photoactivation. In the self-cure mode, the DC increased significantly up to 10 minutes for SC2, 15 minutes for UNI, 20 minutes for BSC and MXC, and 25 minutes for SET. For the conventional resin cement, significant increase in conversion was detected up to 15 minutes of polymerization. Irrespective of the time, all cements tested except SC2 showed DC for the dual-cure mode significantly higher than for the self-cure mode. For SC2, the DC

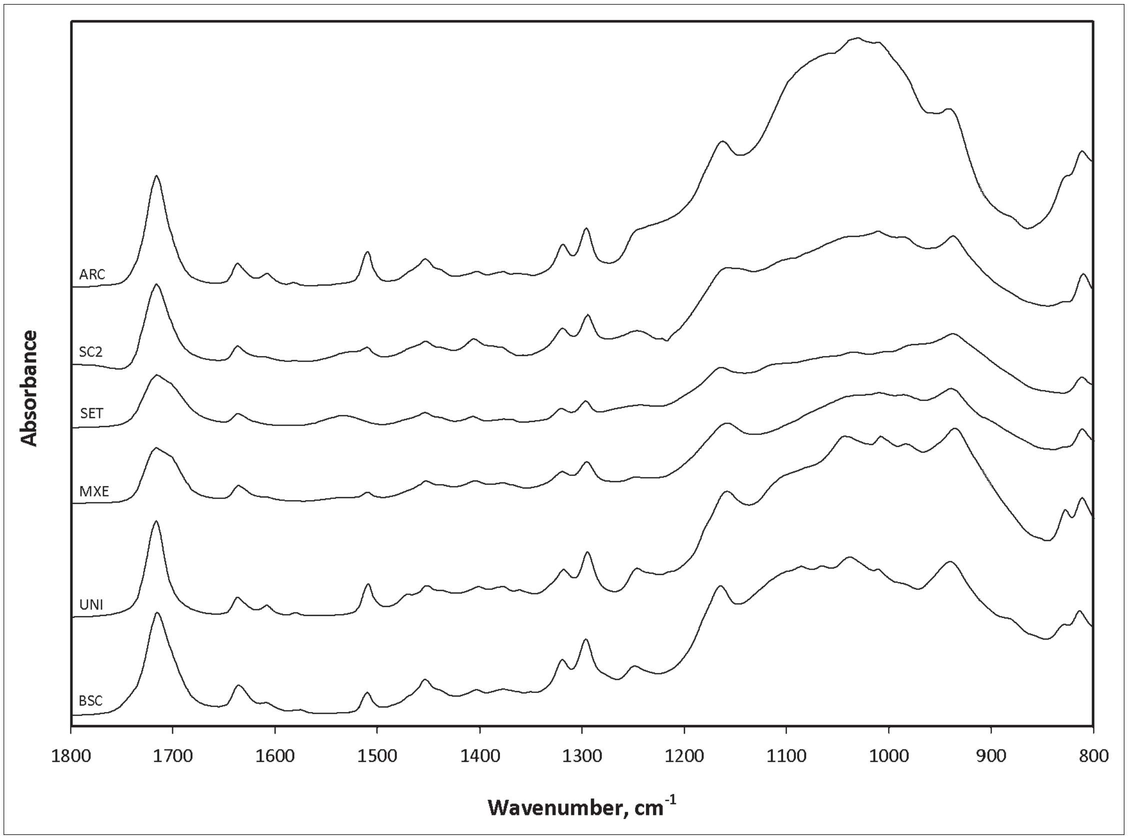

Figure 1. *Mid-infrared spectra of the unpolymerized resin cements tested in the study (24 scans co-addition, 4 cm^{-1} resolution, 800-1800 cm^{-1} frequency range): BisCem (BSC), RelyX Unicem (UNI), Maxcem Elite (MXE), seT (SET), SmartCem2 (SC2), and RelyX ARC (ARC). The spectra were shifted for clarity. Note the absence of appreciable aromatic peaks (at 1608 cm^{-1}) in MXE, SET, and SC2.*

for the self-cure mode was similar to the dual-cure mode for the times between 5 and 25 minutes of polymerization.

The conventional resin cement achieved, in the dual-cure mode, 97% of its maximum extent of conversion at 10 minutes, which is an approximate time indirect restorations might be placed under occlusal loads after the luting procedure. The SARCs, when dual-cured, presented varied extents of maximum conversion at 10 minutes: 95% (SET), 92.5% (UNI), 88% (BSC), 85% (MXC), and 67% (SC2). In the self-cure mode, the conventional material achieved, at 10 minutes, 74% of its maximum extent of conversion. The SARCs showed again varied values: 70.4% (SC2), 66% (MXC), 56% (UNI), 24% (SET), and 12% (BSC).

Results for DC at 30 minutes for all materials and both curing modes are shown in Figure 2. Significant differences between groups were detected for both the self- and dual-cure modes ($p<0.001$). In the dual-cure mode, the DC for the conventional resin cement was significantly higher than for all SARCs except SET. MXC presented intermediate results, while BSC, UNI, and SC2 showed the lowest values. In the self-cure mode, the DC for the conventional resin cement was significantly higher than for all SARCs tested. MXC showed significantly higher DC than UNI, SC2, and SET, which presented similar results. BSC showed significantly lower DC compared with all materials. At 30 minutes of polymerization, as shown in Table 2, the DC for the dual-cure mode was significantly higher than for the self-cure mode for all resin cements.

DISCUSSION

The DC for the dual-cure mode for all resin cements was significantly higher than for the self-cure mode, generally irrespective of the time. Therefore, the first hypothesis is accepted. This result is in agreement with previous studies that showed the conversion of dual-cure materials being remarkably dependent on exposure to light.[15,16,19] In addition, it was clear that the SARCs presented lower extent of C=C conversion, as well as slower rate of polymerization, than the conventional resin cement, irre-

Table 2: *Means (Standard Deviations) for Degree of Conversion (%) as a Function of Time**

Material	Curing Mode	1 min	5 min	10 min	15 min	20 min	25 min	30 min
BisCem	Dual-cure	39.3 (8.6)^{C,a}	42.0 (9.6)^{BC,a}	44.8 (9.6)^{ABC,a}	46.6 (9.1)^{AB,a}	48.8 (9.2)^{A,a}	49.3 (7.4)^{A,a}	51.0 (9.2)^{A,a}
	Self-cure	2.0 (0.8)^{E,b}	4.5 (2.2)^{D,b}	6.2 (1.8)^{C,b}	7.0 (1.9)^{BC,b}	9.0 (3.8)^{AB,b}	10.2 (3.7)^{A,b}	10.5 (2.9)^{A,b}
Maxcem Elite	Dual-cure	34.3 (5.4)^{E,a}	46.7 (8.8)^{D,a}	51.2 (6.6)^{C,a}	54.2 (6.7)^{BC,a}	57.6 (4.7)^{AB,a}	58.4 (5.2)^{AB,a}	60.5 (3.6)^{A,a}
	Self-cure	6.3 (4.6)^{D,b}	29.6 (4.2)^{C,b}	40.2 (4.5)^{B,b}	44.7 (5.7)^{B,b}	49.9 (2.9)^{A,b}	52.5 (4.2)^{A,b}	53.6 (2.7)^{A,b}
RelyX Unicem	Dual-cure	41.1 (4.2)^{C,a}	44.4 (3.7)^{B,a}	46.5 (3.9)^{AB,a}	49.1 (7.5)^{A,a}	50.1 (6.6)^{A,a}	50.3 (4.0)^{A,a}	50.8 (3.6)^{A,a}
	Self-cure	4.8 (3.9)^{D,b}	20.7 (4.7)^{C,b}	28.4 (3.7)^{B,b}	31.5 (2.4)^{AB,b}	33.0 (2.5)^{A,b}	33.7 (2.4)^{A,b}	34.6 (2.1)^{A,b}
seT	Dual-cure	62.2 (5.8)^{C,a}	68.3 (6.3)^{B,a}	69.5 (6.0)^{AB,a}	71.4 (5.4)^{AB,a}	71.6 (7.6)^{AB,a}	72.3 (6.6)^{AB,a}	73.1 (7.7)^{A,a}
	Self-cure	0.7 (0.4)^{E,b}	3.0 (2.0)^{E,b}	17.8 (6.3)^{D,b}	23.8 (5.8)^{C,b}	28.3 (4.7)^{B,b}	33.0 (3.0)^{A,b}	34.7 (3.2)^{A,b}
SmartCem2	Dual-cure	21.9 (6.1)^{D,a}	29.5 (7.3)^{C,a}	31.8 (5.6)^{C,a}	37.8 (7.7)^{B,a}	40.3 (6.3)^{B,a}	43.5 (6.0)^{AB,a}	47.7 (3.0)^{A,a}
	Self-cure	2.9 (1.1)^{C,b}	21.5 (6.9)^{B,a}	33.6 (9.2)^{A,a}	35.1 (9.7)^{A,a}	37.2 (8.4)^{A,a}	39.6 (7.4)^{A,a}	39.9 (5.7)^{A,b}
RelyX ARC	Dual-cure	68.5 (7.7)^{B,a}	71.9 (7.8)^{AB,a}	72.7 (6.3)^{AB,a}	74.0 (2.8)^{A,a}	74.4 (3.1)^{A,a}	74.6 (5.0)^{A,a}	75.1 (5.3)^{A,a}
	Self-cure	14.5 (3.1)^{D,b}	49.5 (2.1)^{C,b}	55.4 (4.5)^{B,b}	58.9 (5.5)^{AB,b}	61.1 (5.0)^{A,b}	61.5 (6.1)^{A,b}	62.4 (5.8)^{A,b}

** For each material, significant differences are indicated by distinct uppercase letters in a same line and distinct lowercase letters in a same column.*

spective of the curing mode. Thus, the second hypothesis is also accepted.

All SARCs needed more time to achieve their maximum conversion than did the conventional material, irrespective of the curing mode. A similar result was reported by Tezvergil-Mutluay et al.,[13] with a conventional resin cement developing higher and faster DC beneath simulated indirect restorations for both light- and self-cure modes. Although the dominant setting mechanism for both self-adhesive and conventional cements is reliant on free-radical polymerization, SARCs have monomers with acidic functionalities. Previous studies reported that mixtures of acidic functional monomers with unmodified (di)methacrylates may exert a detrimental effect on both the rate and extent of copolymerization.[1,20,21] This event is explained by a deactivating effect of the acidic groups on free-radicals. Radicals terminated by an acid group are stable and hence less reactive than free-radicals derived from unmodified monomers, reducing the readiness with which the polymerization takes place.[20]

The effects of slower polymerization and lower final DC were more pronounced when the SARCs were left to polymerize in the self-cure mode. Dual-cured agents have a limit to the amount of self-polymerization promoters that can be added to the material in order to not impair their working time. As a result, depending on the formulation, the self-curing mechanism is restricted to some extent. Some SARCs showed large discrepancy (up to 79%) between the DC values for the dual-mode compared with the self-cure mode. Lower DC values may have an effect on both their strength of bonding to tooth structure[6,22] and to the amount of unreacted, leachable monomers within the material. It has been shown that resin cements may be less cytotoxic when dual-cured compared to self-cured alone.[23]

Differences in the rate and extent of self- and light-cure mechanisms might be related to different composition/amount of polymerization promoters.

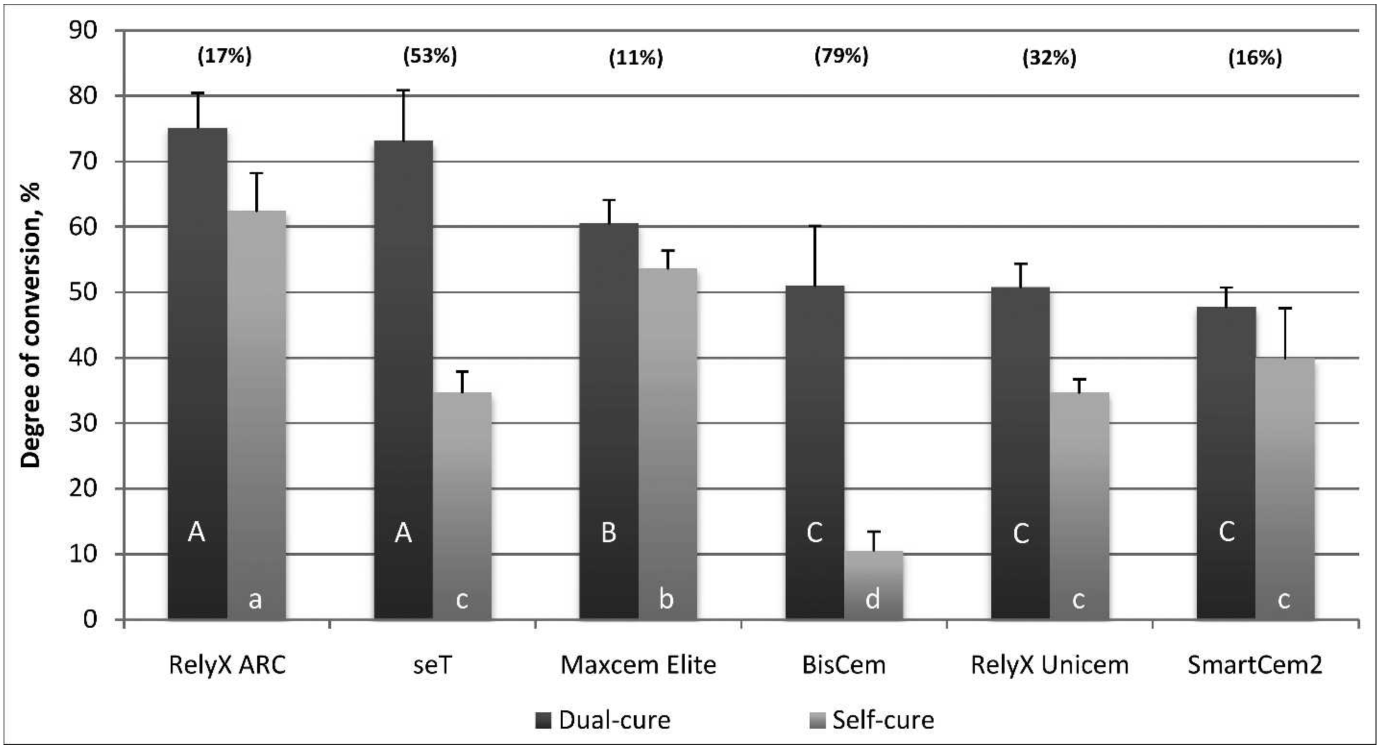

Figure 2. *Results for DC at 30 minutes for all materials. Distinct uppercase letters indicate significant differences in the dual-cure mode. Distinct lowercase letters indicate significant differences in the self-cure mode. For all materials, the DC for the dual-cure mode was significantly higher than for the self-cure mode. The percentage decrease in DC for the self- compared with the dual-cure mode for each material is shown in parentheses.*

Unlike conventional cements, SARCs need self/light-initiators that permit polymerization under acidic conditions. The acid medium may convert the conventional amine co-initiator to a protonated form, deactivating the initiating system, especially when the polymerization occurs slowly (as in the case of the self-cure alone). Another problem with the conventional benzoyl peroxide/amine system is the storage instability of benzoyl peroxide in the presence of acidic species. Therefore, a self-cure initiating system comprising a nonamine reducing agent and an acid-stable oxidizing agent is highly desirable.[24] Consequently, proper systems were included in the formulations of some materials. MXC, for instance, has an amine-free redox initiator system,[25] while UNI presents sodium sulfinate salts, which may aid in preventing chemical incompatibility between acidic monomers and self-curing components.[19] However, the present investigation showed, for some materials, high discrepancy between the dual- and self-curing potentials.

Another important component of SARCs is water. Water is formed during the neutralization of the reaction between the acidic monomers and the basic inorganic fillers, this process being necessary to increase the pH of the material. The water generated is yet expected to be reused for ionization of the acidic monomers, ultimately resulting in a switch to a hydrophobic cement matrix.[26] It is uncertain, though, whether all water is consumed. Any free, unused water may interfere with the polymerization reaction. This effect may also be related to the results of the present study. Any interference with the process of water generation/reuse may affect the properties of SARCs. This could also explain previous findings showing that even low-grade mixing errors may be detrimental to the properties of SARCs.[27]

Although the focus of the present investigation is related to chemical aspects, the outcomes presented here have clinical implications. Firstly, the DC values for SARCs observed in the present study are generally higher than the values reported in previous studies,[13,14] which have ranged, for instance, between 26.4% and 41.5% in the dual-cure mode, and between 10.8% and 24.9% in the self-cure mode.[14] These values could be considered very low, but the samples used in their study are thicker compared to the thin films tested here. Secondly, the SARCs presented a wide range of polymerization outcomes according to the curing mode. Differences in the dual- and self-curing potential for all materials are shown in Figure 2. Some materials, like SET and BSC, presented very high discrepancy of results, and might not be suited for luting procedures with restricted exposure to light.

Thirdly, and probably most importantly, is that the clinician might have in mind the potential impacts of low DC values in the clinical situation. These may include low bond strengths to the dental structures, high solubility in oral fluids, wear at the tooth/restoration interface, and increased cytotoxic reaction. For instance, limited to null adhesion to dentin has been reported using BSC,[28] which showed very low DC for the self-cure mode in the present study. It has to be acknowledged, however, that the conditions of the present study do not take into account the effect the increased body temperature may have on the redox reaction. Notwithstanding, all resin cements were photoactivated in an ideal scenario, with direct exposure to light. Under clinical conditions, the presence of overlaying indirect restoratives may attenuate the irradiance level reaching the cement layer,[15] interfering with the dual-cure mechanism and rendering the material more dependent on the self-curing.

CONCLUSIONS

For all resin cements tested, the degree of conversion for the dual-cure mode was significantly higher than for the self-cure mode, generally irrespective of the time. Self-adhesive resin cements presented slower rate of polymerization and lower final degree of conversion than the conventional resin cement, in either the dual- or self-cure mode.

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