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Monomer Conversion in Dual-cured Core Buildup Materials

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Clinical Relevance

Some dual-cured core buildup materials demonstrate a very slow chemical initiation mechanism that leads to low conversion at areas distal to the activating light, providing a weak core structure during the early setting stages.

ABSTRACT

Objectives: To examine the impact of self-curing (SC) and light-curing (LC) mechanisms on the curing efficiency of dual-cured buildup restorative materials (DCB).

Methods: The DCB materials tested were: Clearfil DC Core Automix (CD), Cosmecore DC (CM), LuxaCore DC (LX) and MultiCore DC Flow (MC). The SC material Clearfil Core New Bond (CN) and the LC material Clearfil Photo Core (CP) were used as controls. Specimens (Ø=4 mm, d=1 mm, n=4) of DCB materials prepared without light-exposure were tested after 10-, 20-, 30- and 60-minutes of storage in dark and dry conditions (37°C), whereas DCB specimens light-cured immediately after mixing (20 seconds, 850mW/cm^2) served as a reference. The amount of remaining C=C bonds (%RDB) was measured on the bottom specimen surfaces using ATR-FTIR spectroscopy. One-way ANOVA and multiple comparison tests were used to evaluate the statistically significant differences (α=0.05).

Results: Polymerization mode had a strong effect on the polymerization efficacy of the DCB materials (*p*<0.0001). The control materials (CN, CP) performed better than the DCB materials (*p*<0.0001), except for LX in the LC mode. LX and MC showed better SC conversion during the initial 20-minute period. After 60 minutes, the %RDB values of CM were still above 60%, whereas in CD and LX, they reached the level of LC specimens.

Conclusions: The LC initiation mechanism of the DCB materials that were tested resulted in better C=C conversion when compared to their native SC mechanisms. The SC and LC control

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materials performed better than the DCB materials, with the exception of LX in the LC mode.

INTRODUCTION

Resin composite core buildup materials have been introduced for the restoration of teeth with compromised resistance and retention form prior to crown preparation. The adhesive properties of these materials with dental hard tissues, as mediated by the corresponding adhesive treatments, along with their improved strength, load transfer characteristics and durability, have established them as alternatives to previously applied restoratives, such as amalgam and glass ionomers.[1] Recently, dual-cured versions of resin composite buildup restoratives (DCB) that combine the advantages of light-curing (LC) and self-curing (SC) mechanisms have been marketed. It has been proposed that DCB overcomes the limitations of extended chairside time, reduced interlayer strength, increased interfacial porosity and depth of cure problems that can occur when the buildup proceeds in incremental layers with LC materials.[2] In addition, DCB provides extended working time and better handling than SC materials, along with a reduced extent of oxygen inhibition on surfaces exposed to air.

These materials have been designed to allow the clinician to build up extended foundation restorations quickly in a single, bulk layer. Activation of the fast-setting LC mechanism rapidly forms a rigid outline of the restoration, while the slow-setting SC mechanism initiates polymerization at regions distal to the activating light, where light attenuation is sufficiently strong to not achieve efficient curing. Furthermore, it has been claimed that the slower setting reaction rate of the SC mechanism produces less shrinkage stresses than the LC mechanism at critical areas, such as bulk interfaces with dental hard tissues and retentive elements (for example, prefabricated root canal posts).[3]

The incorporation of SC and LC activation mechanisms in the same material has failed to attain maximum curing in traditional DCB.[4] However, there is limited information on the curing efficiency of contemporary DCB that has reduced viscosity when compared with previous products.

The current study assessed the contribution of the SC and LC mechanisms on the curing efficiency of modern DCB materials. The testing hypotheses were the following:

1) No statistically significant differences exist in the curing efficiency among the DCB materials set only by the SC mechanism and their LC analogues.
2) No statistically significant differences exist in the curing efficiency among the DCB materials and the SC or LC control materials.
3) There are no statistically significant differences in the curing profile over time within and among the DCB material groups when cured only by the SC mechanism.

METHODS AND MATERIALS

The DCB materials tested are listed in Table 1. Micro-attenuated total reflectance Fourier transform infrared spectroscopy was used (micro-ATR FTIR) for curing efficiency measurements.

Four groups of specimens (n=4 per group) were prepared by placing the mixed but unset pastes in plastic

Table 1: *Materials Tested*

Product	Code	Composition*	Curing Mode**	Manufacturer
Clearfil DC Core Automix (Lot #040AB)	CD	Resin: BisGMA/TEGDMA Filler: Silanated glass, silica (74%wt)	DC	Kuraray Medical Inc, Okayama, Japan
Cosmecore DC (Lot #043502)	CM	Resin: BisGMA/UEDMA/Diacrylate Filler: Ba-glass, silica (70%wt)	DC	Cosmedent Inc, DeltaMed GmbH, Friedberg, Germany
Multicore DC Flow (Lot #H01695)	MC	Resin: BisGMA/ TEGDMA/UDMA Filler: Ba-glass, BaAlFSiO4, silica, YF_3 (71%wt)	DC	Ivoclar-Vivadent, Schaan, Liechtenstein
LuxaCore DC (Lot #590488)	LX	Resin: BisGMA/UDMA/TEGDMA Filler: Silanated Ba-glass, silica (72%wt)	DC	DMG, Hamburg, Germany
Clearfil Core New Bond (Lot #02528B/02717A)	CN	Resin: BisGMA/TEGDMA, Filler: Silanated silica, silica (78%wt)	SC (control)	Kuraray Medical Inc, Okayama, Japan
Clearfil Photo Core (Lot #02266A)	CP	Resin: BisGMA/TEGDMA Filler: Silanated Ba-glass, silanated silica (83%wt)	LC (control)	Kuraray Medical Inc, Okayama, Japan

**According to the manufacturer's information*
***DC: Dual-cured, SC: Self-cured, LC: Light-cured.*
BisGMA: Bisphenolglycidyl dimethacrylate, TEGDMA: Triethyleneglycol dimethacrylate, UEDMA: Urethane dimethacrylate

Table 2: *Results of the %RDB Measurements of the Materials Tested*

Setting Conditions	CD	CM	LX	MC	CN (control SC)	CP (control LC)
10 minutes SC	97.52 (1.55)	92.46 (0.75)	56.11 (1.75)$^{b,\#}$	59.92 (0.43)	56.74 (0.28)$^{\#}$	
20 minutes SC	64.27 (1.50)	79.15 (1.14)	53.73 (1.32)^{b,c,1}	55.91 (0.26)^{e,1}	-	-
30 minutes SC	51.69 (0.97)^{a,2}	67.60 (1.46)	51.34 (1.70)^{c,d,2}	54.64 (0.65)^{e}	-	-
60 minutes SC	49.73 (1.32)^{a,3}	63.37 (0.95)	50.04 (1.05)^{d,3}	51.92 (1.05)^{3}	-	-
LC	50.57 (0.68)	51.53 (0.39)	48.17 (0.51)	56.03 (0.33)		43.04 (0.39)

**means and standard deviations in parentheses*

***Same superscripts indicate mean values with no statistically significant difference (p>0.05). Same letters: Within each material group; same numbers: among material groups at the same condition; same symbols: among DCB (10 minutes) and control materials.*

Table 3: *Ranking of the Statistically Significant Differences of %RDB Mean Values (left) and of the Percentage Mean Differences in %RDB Per Testing Period for the DCB Materials Set Only by the SC Mechanism (right)*

Storage Period	Ranking of %RDB Mean Values*	% Differences of %RDB Mean Values**
10 minutes	CD> CM > MC> LX	CD, CM: 64% more than LX, MC
20 minutes	CM > CD> MC, LX	CM: 36.5% more than LX, MC and CD
30 minutes	CM > MC> CD, LX	CM: 29% more than LX, MC and CD
60 minutes	CM >MC, LX, CD	CM: 25% more than LX, MC and CD

**p<0.001.*

***The differences were calculated based on the mean value of every group.*

ring molds (Ø=4 mm, d=1 mm) sandwiched between two transparent polystyrene strips covered with microscopic cover slips and pressed to remove any excess. In the first group, the specimens of non-irradiated DCB materials were tested after 10, 20 and 30 minutes and 1-hour storage in dark and dry conditions at 37°C; whereas in the second group, the specimens were light-cured for 20 seconds immediately using a quartz halogen bulb unit (Trilight, 3M ESPE, Seefeld, Germany) operated in standard irradiation mode (850 mW/cm^2 light intensity). The third and fourth groups were comprised of the LC and SC control specimens that were tested immediately after light curing (LC) or following 10-minute storage in dark, dry conditions at 37°C (SC).

Using a sapphire anvil, the bottom surface of each specimen was pressed against the diamond reflective crystal of a horizontal ATR unit (Golden Gate Mk II, Specac, Smyrna, GA, USA) placed in the sample compartment of an FTIR spectrometer (Spectrum GX, Perkin-Elmer, Beaconsfield, Bacon, UK). Spectra were acquired under the following conditions: 4000-600 cm^{-1} wavenumber range, 4 cm^{-1} resolution, 30 scans coaddition, 2 mm diameter diamond crystal of a single-reflection (45° angle), ZnSe lenses and 1.5 µm depth of analysis at 1000 cm^{-1}. For the SC and DCB materials, the uncured reference was prepared as follows: the thin edge (30 µm) of a metallic spacer was placed in a vertical direction against the ATR crystal, thus dividing the sampling surface into two equal parts, each which received one of the two resin components. Therefore, the spectra that was recorded corresponded to the average of the two pastes at a 1:1 ratio, as indicated by the manufacturers, without mixing the two resin components and activating the SC mechanism. For the LC material, the spectra of non-irradiated paste were recorded as the uncured reference. The amount of remaining C=C bonds (% RDB) at the bottom surfaces was measured relative to the unset reference materials based on the two-band technique. The peak of the C=C stretching IR vibrations at 1638 cm^{-1} was chosen as an analytical band, whereas the peak of the aromatic C=C stretching IR vibrations at 1605 cm^{-1}, which does not change during polymerization, was chosen as the reference band. The net peak absorbance height ratios of the analytical-to-reference bands were calculated and normalized to the respective unset material ratios in order to quantify the %RDB.

One-way Analysis of Variance (ANOVA) for repeated measurements was employed for comparisons among the SC and LC mechanisms of the same DCB material. One-way ANOVA and comparisons of means with the control (Dunnett's test) was applied to evaluate the curing efficiency of the DCB materials and their LC analogues vs the SC and LC controls, respectively. One-way ANOVA for repeated measurements was used to examine whether the curing profile among and within the DCB materials tested was different when cured only by the SC mechanism. The Tukey-Kramer procedure was applied for multiple-comparison testing. Statistical analysis was performed utilizing the JMP statistical analysis program (JMP, Version 5.0.1a, SAS Institute, Cary, NC, USA). For all comparisons, a 95% confidence level was chosen (α=0.05).

RESULTS

The %RDB values of the materials tested are summarized in Table 2. Comparisons between the SC and LC modes within each material group showed a statistically significant effect of the LC mode in the polymerization efficacy of the DCB materials ($p<0.0001$).

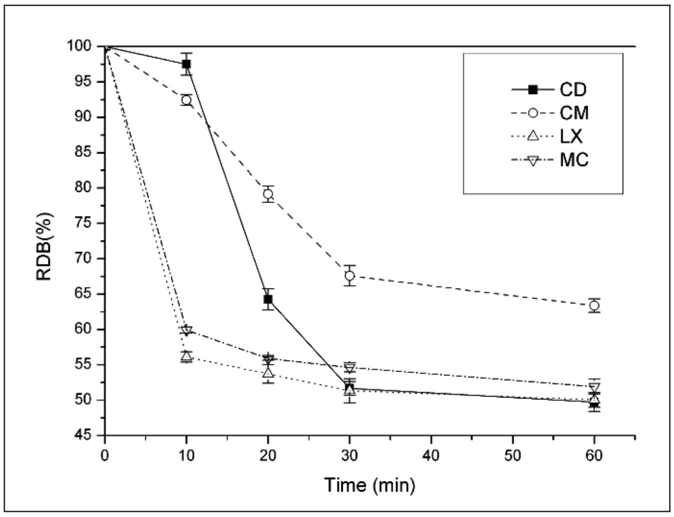

Figure 1. *Plot of the %RDB of the DCB materials tested under the SC curing mode as a function of storing time at 37°C.*

Comparisons between the SC and LC modes within each material group revealed significant differences in the time required for the SC mechanism to reach the corresponding %RDB values obtained after LC.

MC reached the native LC values at 20 minutes, CD at 30 minutes and LX at 60 minutes, whereas CM failed to reach the corresponding LC values. At 10-minutes of storage, no statistically significant difference was found between the SC control (CN) and the SC initiated polymerization for LX. However, MC, CM and CD demonstrated significantly higher %RDB than the SC control ($p<0.0001$).

Table 3 summarizes the ranking of the statistically significant differences and the percentage mean differences in %RDB per testing period when the DCB materials were set only by the SC mechanism. At 10-minutes of storage, CD and CM showed ~64% more %RDB than LX and MC ($p<0.0001$). Nevertheless, at the 20-minute, 30-minute and 60-minute intervals, only CM showed the highest values, ranging from 36.5% (20 minutes) to 25% (60 minutes). At 20-minute intervals, CD performed better than CM, but it was still inferior to LX and MC, while at 30-minutes, CD and LX performed equally better than MC ($p<0.0001$).

The curing profiles of the DCB materials initiated by the SC mode showed distinct differences (Figure 1). CM and DC presented a gradual reduction rate in %RDB, with statistically significant differences among all time intervals ($p<0.0001$), except DC after 30 minutes, which reached equilibrium. MC and LX were characterized by a great reduction in the %RDB during the 10-minute measurements. MC showed statistically significant differences between 10-, 20- and 60-minute measurements, whereas the statistically significant differences in LX were limited to between 10- and 60-minute measurements.

DISCUSSION

According to the results of the current study: a) light curing significantly enhanced the polymerization efficacy of all DCB materials tested; b) the control materials performed better than the DCB materials in both the SC and LC modes, with the exception of LX in the SC mode, which performed equally to the SC control material (CN) and c) the DCB materials exhibited different polymerization behavior when polymerized in the SC mode, with CM exhibiting the highest unsaturation at any time interval examined. Therefore, all three testing hypotheses should be rejected for all the DCB materials (except for LX, which performed equal to the SC control material).

A significant impact of the SC mechanism on the setting profile of the DCB materials has been documented in the current study, which agrees with previous findings;[5-6] however, the expression of the SC mechanism varied. LX and MC showed a more efficient SC mechanism during the early setting stages (even 20 minutes after mixing) than CD and CM. The reduced contribution of the SC mechanism to the early setting stages of the DCB materials is profound, since these materials are designed to allow for an extended working time. Within a reasonable time after mixing, though, it is expected that these materials will adequately set without light exposure. Based on the manufacturers' information, a 5-7 minute intraoral self-curing time is required for adequate setting. Nevertheless, two products (CD and CM) showed a very high unsaturation (>90% RDB) in the SC mode, even after 10 minutes of storage at 37°C, without evidence of macroscopic setting. Consequently, care should be exercised when using these materials for bulk buildups, as they may not guarantee the safe application of crown preparation procedures in the same appointment. In CD, the manufacturer advocates a layering buildup, which is quite controversial for a dual-cured material. Therefore, it can be concluded that, in CD and CM, light-curing is the predominant character, which has been previously reported for CD.[5,7-8]

The results of the current study confirmed previous findings that the polymerization behavior of DCB is strongly material-dependent.[4] Since no quantitative monomer composition is given for the materials tested, no conclusions can be drawn on the monomer contribution to the curing efficiency. From the data available, it is clear that they are either based on BisGMA/TEGDMA comonomers (CD, CN, CP) or on the same comonomers plus UEDMA for partial substitution of BisGMA (MC, LX), as the latter has been shown to improve C=C conversion.[9-10] In all the DCB products tested, LC provided better results than 10 minutes of SC, which confirms the documented advantages of light curing, at least during the initial setting stages.[4] An interesting finding was that the control LC material (CP) demonstrated a significantly lower amount of %RDB than the corresponding dual-cured material (CD) when tested in the LC mode, despite both containing the same monomers and CD incorporating a slow setting SC mechanism. A possible explanation is a reduced camporoquinone content in the latter to minimize sensitivity to the ambient light.

Regarding the efficiency of the SC mode, LX and MC showed equal performance with the control SC material (CN), which implies a strong self-curing character in these materials even at 10-minute measurements. It seems that a determinant factor of the conversion rate and the final conversion values of the DCB materials, when cured in the SC mode, is the type and concentration of the peroxide initiator, the most common being dibenzyl peroxide.

The peroxide/tertiary amine system has long been used in self-curing dental resin composites. However, with the advent of adhesive systems incorporating bonding resins with acidic monomers, an incompatibility of SC materials with these adhesives has been identified.[11-12] The mechanism involves protonization and deactivation of the reducing amine by the ionized fraction of acidic monomers of the unset superficial layer of these adhesives due to oxygen inhibition of their setting reaction.[13] Although the most common LC, SC and DC activation mechanisms contain amine reducing components, the prolonged setting reaction of SC, especially of DC materials, provides adequate time for diffusion of the unset acidic monomers into the composite mass adjacent to the cavity walls and, consequently, to an extended amine neutralization. This reaction inhibits proper polymerization of the composite restorative at the composite/adhesive interface and leads to low interfacial bond strength.[14] The slow setting reaction of all the materials tested, except CP, may be implicated in such a problem. The use of dual-cured adhesives, amine-free LC, SC or DC materials,[14] or the use of a LC non-acidic bonding resin[15] as an intermediate layer between the main restorative material and the adhesive, have been proposed as solutions to this problem. Unfortunately, no information is given for the catalysts of the materials tested, and only one manufacturer mentions the compatibility issue of DCB with the adhesives.

The delay of the DCB materials reaching the curing efficiency of their LC analogues (from 20 minutes to 1 hour) when initiated only by the SC mechanism, indicates development of less polymerization shrinkage and lower contraction stress rates. It has been well established that the higher the curing efficiency, the greater the final shrinkage and the elastic modulus of a composite.[16] In cases of fast reaction rates, the polymerizing matrix reaches the critical conversion for gelation within a short time, rapidly increasing composite stiffness and buildup of interfacial stress,[17] which could lead to gap formation and detachment of the restoration. It has been proposed that, if a higher proportion of the total volumetric contraction occurs while the composite remains in a non-rigid state, a smaller fraction of the shrinkage will effectively be responsible for stress development.[18] To comply with this stress buffering mechanism, most modern DCB materials are designed with a more flowable consistency, compared to older versions, which resembled the stiff consistency of composite restoratives. Moreover, this consistency makes auto-mixing of the two components easier, without pore inclusion. Nevertheless, the relationship between curing efficiency and contraction stress seems to be more complex, since there is a threshold in the curing efficiency above which a non-significant increase in conversion corresponds to a significantly higher stress level.[16] For commercially available DCB materials, no correlation has been reported between %RDB, linear polymerization shrinkage and polymerization stress.[4]

It is not possible to clearly distinguish which part of the material is polymerized through LC or SC initiation. The initial light exposure will cause a rapid increase in the resin conversion, up to a depth dependent on the optical properties of the material, resulting in a very viscous gel. This rapid increase in viscosity hinders the migration of active radical components that would be responsible for further chemically-induced polymerization.[19] From that depth and beyond, only the SC mechanism initiates polymerization. In some products, the use of dual-cured adhesives has been advised to improve SC-induced bulk conversion. This may enhance conversion adjacent to the area treated with the adhesives but not into bulk, due to the limited diffusion of the unset dual-cured adhesive into the much more viscous uncured composite structure.

Although variations in the composition of DCB materials lead to different properties,[6] it is beyond any doubt that high C=C conversion is required, since it has been associated with improved performance in a wide spectrum of mechanical properties, chemical stability and biocompatibility.[9,20-22]

According to the results of the current study, for some DCB materials, the clinician should consider placing the core material in a first appointment and making the final preparation (trimming, contouring, polishing, etc) in a second appointment, after 24 hours. This way, the risks of failure from the adverse effects of mechanical vibrations and stresses associated with the relevant preparation procedures on a core material with inadequate bulk conversion are diminished.

Moreover, additional information regarding the curing performance of DCB materials under the SC mode should be provided by the manufacturers, since the setting time given implies solidification of the material, which, for dental dimethacrylates, usually occurs at a very low C=C conversion value.[23]

CONCLUSIONS

Based on the results of the current study, the following conclusions can be drawn:

a) The light-curing initiation mechanism of the dual-cured core buildup materials tested showed better C=C conversion when compared to their native self-curing mechanisms. It took more than 30 minutes for the majority of the dual-cured core buildup materials to reach the level of light-curing when initiated only by their self-curing mechanism.

b) The self-curing and light-curing control materials performed better than the DCB materials, with the exception of LX in the LC mode.

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