Early Hardness of Self-Adhesive Resin Cements Cured under Indirect Resin Composite Restorations

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ABSTRACT

Purpose: To determine the influence of curing mode on the surface hardness of seven resin cements used to lute indirect composite restorations.

Materials and Methods: Seven commercial dual-curing resin cements were tested: two were total-etch (RelyX ARC [3M ESPE, St. Paul, MN, USA] and Variolink II [Ivoclar Vivadent, Schaan, Liechtenstein]); one was self-etch (Multilink Automix [Ivoclar Vivadent]), and four were self-adhesive (RelyX Unicem [3M ESPE], Maxcem Elite [Kerr Corp., Orange, CA, USA], SmartCem2 [Dentsply, Detrey, GmbH, Konstanz, Germany], and G-Cem [GC CORPORATION, Itabashi-Ku, Tokyo, Japan]). Three specimens (0.5×6.5mm) of each material were prepared for each of three experimental groups: Group 1 (cements allowed to self cure); Group 2 (cements light-cured for 40 seconds); and Group 3 (cements light-cured for 80 seconds). All specimens were cured through a 4-mm-thick composite cylinder (Filtek Z250-A3). Surface microhardness numbers were determined at 20 min after preparation. Results were analyzed by two-way analysis of variance and Student–Newman–Keuls tests ($p < 0.05$).

Results: Superficial hardness was significantly influenced by the resin cement tested ($p < 0.0001$), the curing mode ($p < 0.0001$), and their interaction ($p < 0.0001$). RelyX ARC exhibited the highest mean microhardness values regardless of the curing mode. Light-curing significantly increased the microhardness of all resin cements studied, and these values increased even further with a doubling of irradiation time. Self-adhesive cements exhibited different behavior according to the curing mode. RelyX Unicem was highly sensitive to light irradiation, showing the lowest mean values in the self-curing mode. After light irradiation for 40 or 80 seconds, Maxcem Elite exhibited the lowest mean hardness values of all the resin cements tested.

Conclusion: The microhardness of resin cements is highly dependent on the brand. Dual-curing resin cements should always be light irradiated for longer periods than that recommended by manufacturers.

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CLINICAL SIGNIFICANCE

Dual-curing resin cements should always be light-cured for longer irradiation times, as light irradiation for 80 seconds yields the highest microhardness values in comparison with self-curing or light irradiation for 40 seconds. However, some self-adhesive resin cements exhibit low microhardness values when used to cement 4-mm-thick indirect composite restorations regardless of the curing mode applied.

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INTRODUCTION

There has been a marked increase in the development of new resin cements because of their widespread use to lute indirect esthetic restorations, including polymeric or ceramic materials and fiber posts. Resin cements achieve a better marginal seal and possess superior physical and mechanical properties, retentive capabilities, and esthetics in comparison with conventional cements such as zinc phosphate, zinc polycarboxylate, or glass-ionomer cements.[1–4]

Resin cements can be classified according to their mechanism of interaction with the smear layer and by their curing mode. With regard to the former, resin cements can require application of an etch-and-rinse adhesive system or a self-etching primer.[5–7] More recently, a new subgroup was introduced into the self-etching category (i.e., self-adhesive resin cements) which are applied to enamel and dentin without previous application of an adhesive system.[5–7] Use of these self-adhesive cements has become increasingly common, and new products are constantly being launched. Their clinical success is based on their ability to adequately bond to different restorative substrates[3] and on their reduced technique and operator sensitivity.[5,8,9] Resin cements can also be classified as chemically, light-, or dual-cured. Chemically cured resin cements set uniformly in the absence of light, but the clinician is unable to control the setting and working time.[10,11] In contrast, light-cured resin composite cements are easy to use, but the thickness of the indirect restorative material may attenuate the amount of light reaching the cementing material, potentially compromising the photoactivation.[10,11]

Dual-cured materials were developed to capitalize on the most desirable properties of chemically and light-cured resin cements. Dual-cured resin cements possess a chemical curing system that can achieve complete polymerization in dark localizations[10] alongside a light-curing mechanism that allows for an extended working time and a rapid initial hardening of the resin cement to stabilize the restoration.[12] Hence, indirect resin composite restorations with a thickness > 3 mm should be luted with dual-cure resin cements,[13] as the restorative material significantly reduces the amount of light reaching the bottom of the restoration.[14–17] These cements possess complementary and independent chemical and light activation mechanisms,[18] and the former would be largely responsible for the curing at sites not reached by the light exposure. The chemical curing component in some dual-cure resin composites has been described as slower, less effective,[3,12,13,19,20] or virtually ineffective.[10,15,21,22]

Deficient polymerization of the resin cement negatively affects its physical and mechanical properties (e.g., hardness, modulus, flexural strength, occlusal wear, and water sorption and solubility) and reduces its bond strength to dental structures.[3,10,13,15,19] In the clinical setting, the mechanical properties of dual-cured cements must be sufficient to withstand occlusal and masticatory forces immediately after cementation.[14,23]

Self-adhesive resin cements are all dual-cured, but some manufacturers offer (in their instructions) the possibility of their application in self-cure mode. Various studies have reported low bond strength values for one of these resin cements, RelyX Unicem (3M ESPE), when tested in the self-cure mode.[3,24–26] However, new self-adhesive resin cements are constantly being introduced, and there is no information available on the effectiveness of their self- and light-curing components. Moreover, their complex and proprietary chemistry is reported to produce differences in their behavior.[8,27–30]

The degree of conversion of resin cements can be assessed by indirect methods, and microhardness testing is a widely accepted approach.[10,16,27] The objective of this study was to compare early microhardness, as a measure of the degree of conversion, between self-adhesive and conventional resin cements when self-cured or light irradiated for 40 or 80 seconds through an indirect resin composite restoration. The null hypothesis tested was that conventional and self-adhesive resin cements exhibit similar microhardness values regardless of the curing mode applied.

MATERIALS AND METHODS

Seven commercial dual-cured resin cements were tested in the present study: two were total-etch (RelyX ARC [3M ESPE, St. Paul, MN, USA] and Variolink II [Ivoclar Vivadent, Schaan, Liechtenstein]), one was self-etch (Multilink Automix [Ivoclar Vivadent, Schaan, Liechtenstein]), and four were self-adhesive (RelyX Unicem [3M ESPE, St. Paul, MN, USA], Maxcem Elite [Kerr Corp., Orange, CA, USA], SmartCem2 [Dentsply, Detrey, GmbH, Konstanz, Germany], and G-Cem [GC CORPORATION, Itabashi-Ku, Tokyo, Japan]). The composition of these resin cements is listed in Table 1.

A single operator prepared the specimens and performed the measurements. Resin cements were mixed and dispensed according to the manufacturers' instructions. The cements were inserted into a metallic mold (0.5-mm thick and 6.5 mm in diameter) placed on a microscope slide. A Mylar strip was inserted between the mold and the slide to facilitate separation of the specimens. The resin cements were covered with a second Mylar strip after insertion into the mold, and pressed with a transparent glass slide to remove excess cement. All specimens were covered with a resin composite cylinder (4-mm thick and 6.5 mm in diameter) (Filtek Z250 A3, 3M ESPE, St. Paul, MN, USA) to simulate the clinical situation in which an indirect resin composite is luted in a proximal box.

Three specimens of each resin cement were polymerized through the composite cylinder and tested for each of the three different scenarios:

1. Group 1: Cements were allowed to self-cure.
2. Group 2: Cements were light-cured for 40 seconds with a Bluephase LED unit (Ivoclar Vivadent, Schaan, Liechtenstein).
3. Group 3: Cements were light-cured for 80 seconds with the same curing unit.

In Groups 2 and 3, the Bluephase LED unit was applied at high intensity (1,200 mW/cm^2) and the tip was placed in contact with the resin composite cylinder.

All specimens were removed from the molds at 20 min after preparation, and microhardness measurements were performed by means of a Vickers digital microhardness tester (Buehler 2101, Lake Bluff, IL, USA). Ten indentations were recorded on each specimen, applying a load of 100 g for 30 seconds.

The independent variables were the resin cement tested and the curing mode. Their influence on microhardness values was analyzed by two-way analysis of variance. Post

TABLE 1. COMPOSITION OF THE RESIN LUTING AGENTS STUDIED.

Resin cement	Composition
RelyX ARC 3 M ESPE, St. Paul, MN, USA Shade: A3 Batch n°: FAGH	Paste A: BisGMA, TEGDMA, silane treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, 4-(Dimethylamino)-Benzeneethanol. Paste B: Silane treated ceramic, TEGDMA, BisGMA, silane treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide. (72% in wt).
Variolink II Ivoclar Vivadent, Schaan, Liechtenstein Shade: A3 Batch n°: Base: J19730 Catalyst: J19103	Base: BisGMA, TEGDMA, UDMA, fillers, Ytterbium trifluoride, stabilizers, pigments. Catalyst: BisGMA, TEGDMA, UDMA, fillers, ytterbium trifluoride, stabilizers, pigments, benzoyl peroxide. (Base: 26.3% in wt; Catalyst low viscosity: 27.9% in wt).
Multilink Automix Ivoclar Vivadent, Schaan, Liechtenstein Shade: A3 Batch n°: H23051	Base and Catalyst: Dimethacrylates and HEMA, Barium glass fillers, Ytterbium trifluoride, Silicon dioxide fillers, catalysts and stabilizers, pigments. (Base: 30.5% in wt; Catalyst: 30.2% in wt).
RelyX Unicem 3M ESPE, St. Paul, MN, USA Shade: A3 Batch n°: 326313	Powder: Glass fillers, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator. Liquid: Methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator. (70% in wt).
Maxcem Elite Kerr Corp., Orange, CA, USA Shade: A3 Batch n°: 2986507	HEMA, MEHQ, CHPO, TiO_2, Uncured Methacrylate Ester Monomers, pigments.
SmartCem2 Dentsply , Detrey, GmbH, Konstanz, Germany Shade: MED Batch n°: 0809251	UDMA, did and tri- methacrylate resins, phosphoric acid modified acrylate resin, barium boron fluoroaluminiumsilicate glass, organic peroxide initiator, CQ, photoinitiator, phosphene oxide photoinitiator, accelerators, butylated hydroxyl toluene, UV stabilizer, TiO_2, iron oxide, hydrophobic amorphous silicon dioxide.
G-Cem GC CORPORATION, Itabashi-Ku, Tokyo, Japan Shade: AO3 Batch n°: 0707041	4-META, UDMA, alumino-silicate glass, pigment, dimethacrylate, distilled water, phosphoric ester monomer, initiator.

BisGMA: Bisphenol A Diglydicyl Methacrylate; TEGDMA: Triethylene Glycol Dimethacrylate; UDMA: Urethane Dimethacrylate; HEMA: 2-Hydroxyethyl Methacrylate; MEHQ: 4 Methoxyphenol; CHPO: Cumene HydroPerOxide; TiO_2: Titanium Dioxide; CQ: camphorquinone; 4-META: 4-methacryloyloxyethyl trimellitate anhydride.

hoc comparisons were performed using the Student–Newman–Keuls test. All statistical testing was performed at a preset alpha of 0.05 using SPSS 16.0 for Windows software (SPSS Inc., Chicago, IL, USA).

RESULTS

Table 2 shows the mean microhardness values for the seven resin cements as a function of the curing mode. Superficial hardness was significantly influenced by the resin cement tested ($p < 0.0001$), the curing mode ($p < 0.0001$), and their interaction ($p < 0.0001$).

All resin cements tested exhibited significantly different mean microhardness values, and their ranking changed according to the curing

TABLE 2. MEAN VICKERS MICROHARDNESS VALUES OF THE RESIN CEMENTS ACCORDING TO THE CURING MODE (WITH STANDARD DEVIATIONS).

	Type of cement	Self-cured	Light-cured 40 seconds	Light-cured 80 seconds
RelyX ARC (3M ESPE)	Total-etch	17.59 (3.52) a5	29.65 (2.94) b6	33.13 (2.64) c5
Variolink II (Ivoclar Vivadent)	Total-etch	7.82 (0.93) a3	9.34 (1.95) b2	18.15 (2.35) c3
Multilink Automix (Ivoclar Vivadent)	Self-etch	5.79 (1.48) a2	16.75 (0.78) b5	19.37 (1.15) c4
RelyX Unicem (3M ESPE)	Self-adhesive	1.57 (0.47) a1	11.78 (2.20) b3	17.95 (2.07) c3
Maxcem Elite (Kerr)	Self-adhesive	6.26 (0.59) a2	6.92 (0.57) b1	9.21 (1.22) c1
SmartCem2 (Dentsply)	Self-adhesive	5.80 (1.05) a2	10.16 (0.52) b2	12.62 (1.34) c2
G-Cem (GC)	Self-adhesive	9.53 (0.45) a4	13.05 (0.77) b4	18.07 (1.01) c3

For each row, different letters indicate a significant influence of the curing mode ($p < 0.05$).
For each column, different numbers indicate significantly different mean microhardness values among the resin cements for each curing mode ($p < 0.05$).

mode applied. The only exception was the total-etch resin cement, RelyX ARC, which showed the highest mean hardness values regardless of the curing mode.

The self-adhesive resin cements, except G-Cem, exhibited significantly lower mean microhardness values versus RelyX ARC and Variolink II when allowed to self-cure. Multilink Automix showed lower mean hardness values in comparison with these total-etch cements and G-Cem but similar values to those achieved with Maxcem Elite and SmartCem2. RelyX Unicem showed the lowest hardness values when the self-curing mode was applied.

This ranking was different when the cements were light-cured for 40 seconds. RelyX ARC remained the hardest cement, followed by Multilink Automix and G-Cem, with significant differences among them. RelyX Unicem exhibited intermediate mean values, which were higher than those for SmartCem2, Variolink II, or Maxcem Elite. Maxcem Elite was significantly softer with this curing mode.

Doubling of the light-curing time significantly influenced the hardness of these cements, but RelyX ARC was again the hardest cement and Maxcem Elite the least hard one. Multilink Automix showed significantly lower mean hardness values in comparison with RelyX ARC but higher values than found for Variolink II, G-Cem, and RelyX Unicem, which did not significantly differ among them. The self-adhesive resin cement SmartCem2 exhibited significantly lower mean hardness values versus all cements with the exception of Maxcem Elite.

Light irradiation for 40 seconds significantly increased the microhardness of all tested resin cements, and they were significantly harder when the light exposure time was doubled. However, resin cements exhibited different behaviors according to the curing mode. Thus, light curing for 40 seconds produced a marked increase in hardness for RelyX ARC, Multilink Automix, SmartCem2, G-Cem, and, especially, RelyX Unicem. All tested resin cements showed significantly higher mean hardness values after 80 seconds than after 40 seconds, but this increase was more marked for Variolink II and less so for RelyX ARC, Multilink Automix, RelyX Unicem, and G-Cem. Maxcem Elite showed increased hardness values after light-curing for 40 and 80 seconds but remained the least hard cement with this curing mode.

DISCUSSION

Indirect resin composite restorations are increasingly preferred to direct resin composite restorations, as the negative effects of polymerization shrinkage are restricted to the luting material space and superior physical properties and anatomical shape can be obtained.[31] Dual-cured cements are indicated for the luting of indirect composite restorations, as the light exposure is attenuated by the presence of the composite restoration.[16,20] Self-adhesive resin cements are an attractive option for this purpose, as they are all dual-cured and easy to use, with no need for pretreatment of dental tissues.

In the present study, the self-curing capacity of self-adhesive and conventional resin cements was determined by simulating a clinical situation in which a 4-mm-thick composite inlay/onlay is luted in a proximal box without light curing. Microhardness results evidenced that this capacity was product-dependent, with no difference as a function of the conventional or self-adhesive nature of the resin cement.

Under these experimental conditions, the total-etch resin cement RelyX ARC achieved the highest mean hardness values in comparison with the other cements under study. The other total-etch cement, Variolink II, was significantly softer than RelyX ARC. These results are in agreement with the observation by Kumbuloglu and colleagues[32] of a higher degree of conversion at 15 min after mixing for RelyX ARC (61%) than for Variolink II, which has been reported to have a poor self-curing mechanism[33]. Lee and colleagues[11] also found a much slower curing speed for Variolink II than for RelyX ARC, and much longer was taken to reach the peak polymerization shrinkage rate.

The self-etch resin cement, Multilink Automix, showed mean microhardness values that were significantly lower than those found for RelyX ARC, Variolink II, and G-Cem and similar to those observed for the self-adhesive resin cements Maxcem Elite and SmartCem2. Therefore, the effectiveness of their self-curable component does not appear very high. It should be kept in mind that Multilink is described by the manufacturer as a self-curing luting material with a light-curing option.

The microhardness values of self-adhesive resin cements differed according to the brand. G-Cem obtained the highest values, intermediate between those of RelyX ARC and Variolink II. The other three cements, SmartCem2, Maxcem Elite, and RelyX Unicem, attained very low values. Kumbuloglu and colleagues[32] and Vrochari and colleagues[34] also reported a poor curing of RelyX Unicem (26 and 11%, respectively) when tested in self-curing mode. Although there is little information on the other self-adhesive cements, it is possible that the amount of chemical-curing initiator is inadequate.

A deficient polymerization affects the mechanical properties of the cements[1,22] and may lead to adverse consequences, including reduced marginal adaptation and increased wear.[35] Moreover, it may impair the bond strength of the interface between dental tissues and indirect restorative material.[35] Previous studies have reported low bond strength results when RelyX Unicem was applied without light irradiation.[25,26] Moreover, the curing mechanism of RelyX Unicem is not only dependent on light exposure but also on the presence of tooth structure,[18] which was absent in the present study. RelyX Unicem setting involves an additional acid-base reaction between the phosphorylated methacryalte radicals and the hydroxyapatite responsible for pH neutralization.[18,24]

Light irradiation for 40 seconds resulted in a significant increase in the microhardness of all resin cements evaluated, in agreement with other studies.[10,16,20] The presence of the indirect resin composite reduces but does not completely block the light exposure,[14] and photoinitiators were reported to be sufficiently sensitive to light exposure.[23] According to Moraes and colleagues,[36] only a minimum energy dose is required to excite the photoinitiator and produce an adequate number of free radicals for polymerization. As the degree of polymerization is dependent on the energy supplied during light curing, our use of a curing unit with high light irradiance might also have compensated for the light attenuation due to the resin composite restoration.[36]

After light-curing, the microhardness was also product-dependent, with statistically significant differences among the resin cements. RelyX ARC was again the hardest resin cement. We highlight that Multilink Automix and RelyX Unicem were especially sensitive to light irradiation and achieved a much higher microhardness than in the self-curing mode, in agreement with a recent study.[34] Variolink II specimens did not exhibit such a marked increase in microhardness, as also observed with Maxcem Elite. A low degree of curing (26%) was also reported for Maxcem, the previous version of this cement.[34] The hardness of G-Cem significantly increased, and it continued to be the hardest cement in the self-adhesive group.

The application of a longer light-exposure time (80 seconds) significantly increased the hardness values of RelyX ARC, Multilink Automix, RelyX Unicem, and G-Cem resin cements, with a specially marked increase for Variolink II specimens, which changed the ranking of this cement from the least hard to the hardest. Accordingly, a longer exposure time should always be applied when this cement is used to lute thick indirect composite restorations. SmartCem2 and Maxcem Elite yielded the lowest hardness mean values and were not markedly affected by the increased exposure time. Therefore, neither of these cements appears to be suitable for the clinical situation simulated in this study.

In the present study, microhardness was assessed at 20 min after preparation of the resin cement specimens, as the period immediately after cementation is critical for resin cements, and an effective degree of cure is required to resist occlusal adjustment, polishing procedures, and subsequent occlusal masticatory forces.[23] Moreover, it has been reported that the chemical reaction between the base and catalyst pastes takes place during the first 10 minutes, hence the final microhardness may not be very different from the values measured at 20 min.[23,33] However, further studies are required to confirm this finding for self-adhesive resin cements, as they also possess an acid-base polymerization mechanism.

CONCLUSIONS

Our results confirm that the microhardness of conventional and self-adhesive resin cements is highly dependent on the brand. The differences observed among self-adhesive cements are probably related to their complex chemistry. As few and disperse data are available on the behavior of these new self-adhesive resin cements, further studies are needed to evaluate other mechanical and adhesive properties. With regard to the effect of the curing mode, significantly lower microhardness values were obtained for all resin cements tested when light-curing was not applied. Doubling the light-irradiation time significantly increased mean microhardness values. Hence, dual-curing resin cements should always be photoactivated for longer periods than those recommended by the manufacturers when the light is attenuated by a thick indirect resin composite restoration.

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