

Effects of cements and eugenol on properties of a visible light-cured composite

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Abstract

An in vitro study evaluated the effects of barrier materials on mechanical properties of a visible light-cured composite restorative. The composite was cured and aged over a widely used cavity liner (Dycal®), various cements (Fleck's, Durelon, Vitrabond, ZOE B & T) and free eugenol and tested at seven and 28 days. The properties of interest were tensile stress (strength) at rupture, compressive strain (deformation at rupture), and stress-strain ratio at rupture. The experiment indicated that underlying barrier materials exerted neither a beneficial nor an adverse effect upon the restorative's ability to withstand compressive strain and tensile stress ($P < 0.05$). (Pediatr Dent 15:104-7, 1993)

Introduction

In the clinical setting, it is difficult to determine whether a visible light-cured (VLC) restoration attains a sufficient level of polymerization. If the clinician attempts to polymerize too thick a layer or uses too short an exposure to the curing light, material in deep portions of the cavity may not polymerize completely. According to Phillips,¹ the presence of undercured resin in deep areas of the cavity might foster diffusion of substances potentially cytotoxic to tooth pulp.

Typically, clinicians cover the pulpal floors and walls of cavity preparations with a barrier material to protect vital pulp tissue from attack by chemical irritants. Barrier materials used for this purpose include varnishes; essential oil suspensions of calcium hydroxide; and powder-liquid mixtures of zinc oxide and orthophosphoric acid, zinc oxide and polyacrylic acid, and alumina-silicate glass and polyacrylic acid.

Civjan² used hardness measurements (Rockwell 15T) to monitor the interactions of varnishes, cavity liners, and cements with five chemically cured restoratives. The investigators concluded that polymerization of composites may be impaired by liners and bases, with eugenol containing the materials that exert the most deleterious effect.

Lingard³ observed that the essential oil constituents (eugenol and butyleneglycol disalicylate) of certain barrier materials adversely affected the color, topographical features, and hardness of two chemically cured composite restoratives.

Marshall et al.⁴ assessed the extent to which zinc oxide-eugenol, ortho-ethoxybenzoic acid (EBA), glass ionomer, and polycarboxylate cements affect the hardness of: a conventional, chemically cured resin; a chemically cured microfilled resin; and a light-cured resin. In a separate experiment, they examined the effect of a calcium hydroxide liner on the hardness of the light-cured microfilled restorative. The hardness of each of the three composites was impaired significantly by zinc oxide-eugenol, EBA, and glass ionomer cements. However, curing the VLC

microfilled material and its bonding agent over polycarboxylate cement or the calcium hydroxide liner did not elicit a significant reduction in hardness.

The apparent absence of data relating to the effects barrier materials may have on the compressive and tensile properties of a VLC microfilled composite suggested the appropriateness of the present study.

Methods and materials

The in vitro study was based on a two-factor design: barrier material (cavity liner, cement, or medicament) upon which a specific composite restorative was cured and aged (seven levels); and aging time after curing (two levels). The dependent variables were rupture properties (tensile stress, compressive strain, and stress-strain ratio) of a specific VLC microfilled composite (Silux Plus™ Universal, Dental Products Division, Minnesota Mining and Manufacturing Co., St. Paul, MN).

The barrier materials consisted of a calcium hydroxide liner (Dycal®, The L.D. Caulk Co., Division of Dentsply International, Inc., Milford, DE); zinc phosphate cement (Fleck's Cement, Mizzy, Inc., Clifton Forge, VA); zinc polycarboxylate cement (Durelon®, ESPE GmbH and Co., KG, D-8031, Seefeld/Overbay, Germany); glass ionomer cement (Vitrabond™, Dental Products Division, Minnesota Mining and Manufacturing Co., St. Paul, MN); zinc oxide-eugenol cement (ZOE B & T, The L.D. Caulk Co., Division of Dentsply International, Inc., Milford, DE); and reagent grade eugenol (Fisher Scientific Company, Chemical Manufacturing Division, Fair Lawn, NJ).

The components of the cavity liner and each cement were proportioned and mixed as specified by the respective manufacturers. Each mix was transferred to a separate sheet torn from a paper-mixing tablet. With a spatula, each mix was distributed evenly over a paper's 9.4 x 7.0 cm working surface. All liner- and cement-coated papers were set aside and left undisturbed under ambient temperature and relative humidity for 30 min. The hardened coatings

so formed were about 0.02 cm thick.

Additional mixing papers were coated with eugenol. About 5 cc eugenol was deposited on a paper and wiped over its entire working surface with a spatula. Excess liquid was removed by shaking the paper. Immediately after coating, these papers were used in the production of composite resin specimens.

Each individual mold for fabricating composite specimens was a polytetrafluoroethylene (Teflon®, E.I. Dupont, de Numours & Co., Wilmington, DE) disc with a concentric, face-to-face, cylindrical cavity. Nominal mold cavity dimensions were diameter = 0.6 cm and height = 0.3 cm. The placement of one planar face of any mold against a coated paper enabled the forming, curing, and aging of a composite resin specimen against a substrate of cavity liner, cement, or eugenol. In like fashion, the molds were used with uncoated papers to produce control specimens.

A two-increment technique was used to fill the molds. The uncured volume of each composite increment was about 0.09 cm³. In all cases, the first increment was cured before the second increment was placed. Curing was accomplished by a 40-sec exposure of each uncured increment to visible light emitted by an artificial source (Visilux II, Dental Products Division, Minnesota Mining and Manufacturing Co., St. Paul, MN)

Eighty-four specimens were produced—12 test pieces for each of six experimental groups (one liner group, four cement groups, and one eugenol group) and one control group.

The cured specimens and their respective underlying coated or uncoated papers were transferred to an environmental chamber maintained at 37°C ± 2°C and 100% relative humidity. After the seventh day, one-half of the specimens belonging to each experimental group and the control group were taken from the chamber and separated from their molds and underlying papers. The specimens retained in the chamber were allowed to age an additional 21 days.

The apparent tensile stress at rupture of seven- and 28-day-old composite resin specimens was determined by the diametral compression method of Civjan.⁵

Crosshead and chart speeds of the testing machine (Instron® Universal Testing Machine, Instron Corp., Canton, MA) were 0.05 cm/min and 2.5 cm/min, respectively. A machine-generated graphic record of time versus load for each diametral compression test made possible the measurement of vertical diametric strain at rupture for each composite specimen.

For example, at a chart speed of 2.5 cm/min, the chart travels 0.25 cm in 0.1 min. Concurrently, the crosshead moving at a rate of 0.05 cm/min descends a vertical distance (distance = rate x elapsed time) of 0.005 cm.

Given, for instance, a total chart travel time of 0.9 min at rupture of a specimen, the crosshead would have traveled a vertical distance of 0.045 cm.

Accordingly, the specimen's vertical diameter would have experienced a compression of 0.045 cm. Dividing the

change in diameter (0.045 cm) by the specimen's original diameter and multiplying the resultant quotient by 100 yields vertical, diametric compressive strain in percent.

The availability of data pertaining to stress and strain made possible the calculation of the ratio of stress to strain at specimen rupture.

For each of the aforementioned properties, assessment of the effects of material; aging time; and material x aging time was accomplished by two-way analysis of variance (CLR ANOVA®, Claris Research, Inc., Houston, TX). Multiple comparisons of mean values were performed using Tukey's test of honestly significant difference.

Results

Findings from measurement of the VLC composites properties are summarized in Tables 1, 2, and 3.

Tensile stress at rupture

The highest mean tensile strength, 36 MPa (one Mega Pascal = 145 pounds per square inch), was exhibited by 28-day-old specimens cured and aged over eugenol (Table 1). Seven-day-old specimens cured and aged in contact with calcium hydroxide or eugenol showed the least strength (25 MPa). Variances between the mean of the calcium hydroxide seven-day subgroup and the means of the zinc polycarboxylate and eugenol 28-day subgroups were significant ($P < 0.05$). Similarly, variances between the mean of the eugenol seven-day subgroup and the means of the polycarboxylate and eugenol 28-day subgroups were too large to have arisen solely by chance.

Whereas all mean tensile strength values affiliated with the five cement-groups and the eugenol-group (Table 2) were statistically equivalent to that of the control group, the variance between the means of the calcium hydroxide liner and zinc polycarboxylate cement groups was significant ($P < 0.05$).

Vertical, diametric compressive strain

The eugenol and control 28-day subgroups yielded the highest and lowest mean values for strain at tensile rupture, respectively (Table 1). The relatively low mean strain values for the control 28-day subgroup (4.70% ± 0.58%) and calcium hydroxide liner 28-day subgroup (4.74% ± 0.71%) differed significantly from the means of the zinc polycarboxylate cement 28-day subgroup (6.51% ± 0.75%) and the eugenol 28-day subgroup (6.81% ± 0.38%).

ANOVA indicated that the effects of barrier material ($P = 0.0601$) and aging time ($P = 0.5953$) on strain at rupture were not significant.

Findings from Tukey's test on the effects of barrier material and aging time on strain at rupture are presented in Tables 2 and 3, respectively.

Ratio of stress to strain at tensile rupture

The highest mean stress-strain ratio (571 MPa) was exhibited by seven-day-old specimens cured and aged

over zinc polycarboxylate cement (Table 1). Seven-day-old specimens cured and aged in contact with the calcium hydroxide liner yielded the lowest mean stress-strain ratio (448 MPa).

ANOVA showed that the effects of barrier material ($P = 0.6128$) and barrier material x aging time ($P = 0.0927$) on stress-strain ratio at tensile rupture were not significant. The effect of specimen aging time, however, was significant ($P = 0.0062$).

Post hoc analyses of the effects of barrier material x specimen age, barrier material, and specimen aging time on stress-strain ratio at tensile rupture are presented in Tables 1, 2, and 3, respectively.

Discussion

The principal mode of clinical fracture of dental restorations is tensile shear, so tensile strength is believed to be more important than compressive strength.⁶

Considering the foregoing premise, we wanted to determine the extent to which barrier materials affect the tensile strength of a widely used composite restorative.

We used the incremental placement and curing technique to increase the probability of maximizing the quality of the restorative's cure in the presence of substances that are known to impair the surface hardness of composites.²⁻⁴

Interestingly, the mean stress at tensile rupture for each of seven specimen groups and 14 subgroups fell within a previously established range of tensile strength values (25–40 MPa) for microfilled composites.⁷

For the most part, materials that exhibit inordinately high levels of plastic deformation when subjected to masticatory forces are not suitable for use as dental restoratives. Similarly, barrier materials that compromise the ability of BIS-GMA resin to resist deformation are not suitable for use with a dental composite restorative.

Heretofore, it has been assumed that a composite restorative-barrier material interaction that softens the contact surface of the restorative¹⁻³ will impair the ability of the restorative to resist compressive and tensile deformation.

To challenge this assumption, we measured strain and calculated stress-strain ratio.

The stress-strain ratios here are simply ratios of stress at tensile rupture to vertical diametric strain, as determined by diametral compression of small cylindrical

Table 1. Effect of barrier material x specimen age on properties of a VLC composite

Material	Age Days	Tensile Strength MPa	Compressive Strain %	Stress-Strain Ratio MPa
CHL	7	25 ± 3*	5.68 ± 1.18	488 ± 54
EUG	7	25 8*	4.92 1.28	499 89
ZPC	7	26 3	4.93 0.70	531 97
GIC	7	28 4	6.37 1.05	454 66
Control	7	28 3	5.88 0.95	481 39
ZOE	7	29 3	5.75 0.49	514 76
PCC	7	29 2	5.28 0.88	571 99
CHL	28	26 5	4.74 0.74*	560 86
Control	28	26 1	4.70 0.58*	559 76
ZOE	28	27 7	5.33 1.06	515 67
ZPC	28	29 4	5.35 0.63	546 50
GIC	28	34 6	6.15 1.45	569 71
PCC	28	35 4†	6.51 0.75†	538 30
EUG	28	36 ± 6†	6.81 ± 0.38†	523 ± 87

CHL = calcium hydroxide liner; EUG = eugenol; ZPC = zinc phosphate cement; GIC = glass ionomer cement; Control = no barrier material; ZOE = zinc oxide-eugenol cement; and PCC = polycarboxylate cement.

* Mean value significantly lower than † mean value ($P < 0.05$).

| = all mean values in column are statistically equivalent ($P > 0.05$).

Table 2. Effect of barrier material on properties of a VLC composite

Material	Tensile Strength MPa	Compressive Strain %	Stress-Strain Ratio MPa
CHL	26 ± 4*	5.21 ± 1.08	504 ± 86
Control	27 2	5.29 0.98	520 68
ZPC	27 4	5.14 0.07	538 71
ZOE	28 5	5.54 0.85	515 65
EUG	30 9	5.87 1.33	511 69
GIC	31 6	6.26 1.27	512 85
PCC	32 ± 4†	5.89 ± 1.02	555 ± 74

CHL = calcium hydroxide liner; Control = no barrier material; ZPC = zinc phosphate cement; ZOE = zinc oxide-eugenol cement; EUG = eugenol; GIC = glass ionomer cement; and PCC = polycarboxylate cement.

* Mean value significantly lower than † mean value ($P < 0.05$).

| = all mean values in column are statistically equivalent ($P > 0.05$).

specimens. Although such ratios enable assessment of the VLC composite's capacity to resist deformation, they are not purported to be measures of modulus of elasticity or modulus of rigidity.

The present data do not refute that composite restorative-barrier material contact-surface softens as reported by others. They do suggest, however, that the magnitudes of suspected, untoward interactions occurring at the barrier material-composite interface may be too small to cause deterioration of the composite's bulk properties.

The extent to which a composite restorative must be softened to detect adverse changes in tensile strength, strain, and stress-strain ratio is unknown and remains to be studied.

The results of this study may not apply to long-term use in vivo.

Additionally, minor differences in resin-filler ratio, compositional and morphologic features of the filler component, and practices pertaining to compounding and manufacture may exert profound effects on the mechanical properties of BIS-GMA composite restoratives.

Accordingly, the data that characterize the behavior of the restorative used in the present study should not be used to predict the performance potentials of seemingly similar VLC microfilled composites.

Conclusion

A popular calcium hydroxide liner; zinc phosphate cement; zinc polycarboxylate cement; glass ionomer cement; zinc oxide-eugenol cement; and free eugenol did

Table 3. Effect of specimen aging time on properties of a VLC composite

Specimen Age Days	Tensile Strength MPa	Compressive Strain %	Stress-Strain Ratio MPa
7	27 ± 5*	5.55 ± 1.08	500 ± 78*
28	31 ± 6†	5.65 ± 1.16	544 ± 66†

* Mean value significantly lower than †mean value ($P < 0.05$).

| = mean values in column are statistically equivalent ($P > 0.05$).

not affect significantly ($P > 0.05$) the tensile and compressive properties of a specific VLC, microfilled composite restorative.

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