

# Bonding and gap formation of glass-ionomer cement used in conjunction with composite resin

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Glass-ionomer cement has been suggested as liner in cavities restored with composite resin. The purpose of the present investigation was to measure 1) tensile bond strength between etched glass-ionomer cement and composite resin, and 2) gap formation as assessed by wall-to-wall polymerization contraction and by microleakage with a silver nitrate technique. The influence of the following variables was examined: type of glass-ionomer cement and composite resin, duration of acid etching, irradiation time of unfilled and composite resin, preparation of bevel, conditioning with polyacrylic acid, and storing time in water before gap measurement. Bond strengths varied from 0 MPa when etching was omitted to 3.9 MPa after etching. Glass-ionomer cement lining reduced wall-to-wall contraction and penetration of silver nitrate. A positive correlation was found between wall-to-wall contraction and silver nitrate penetration.

□ *Adhesive dentistry; dental materials; operative dentistry*

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Use of glass-ionomer cements has been increasing owing to several advantageous properties. These include the ability to bond to enamel and dentin (1-4), biocompatibility with pulpal tissue (5, 6), slow release of fluoride (7, 8), and a relatively low solubility in acidic solutions (9, 10). Glass-ionomer cements, nevertheless, also have less desirable properties, such as brittleness, lack of strength, and low resistance to wear (11). To compensate for these deficiencies, so-called silver-reinforced glass-ionomer cements have been developed. The compressive strengths of the two types of glass-ionomer cements have, however, been found to be of similar magnitudes (12, 13).

Composite resins are recognized as being superior to glass-ionomer cements from an aesthetic point of view. Marginal seal in composite resin fillings is obtained by acid etching of enamel walls. Establishing an effective marginal seal in class V cavities extending into dentin can, however, be very difficult. Several bonding agents have been developed for the purpose of promoting a chemical bond between composite resin and dentin. Glass-ionomer cements have also been suggested as cavity liners to ensure marginal

seal at cavity walls without enamel (3). This technique implies sealing cervical dentin with glass-ionomer cement and restoring the enamel structure with composite resin. In using this technique, the feasibility of creating a bond between glass-ionomer cement and composite resin is a prerequisite. It seems questionable, however, whether glass-ionomer cements, which undergo hardening shrinkage, are capable of averting marginal leakage and gap formation.

It was the purpose of the present investigation to measure the tensile bond strength between glass-ionomer cement and composite resin and the wall-to-wall polymerization contraction and microleakage associated with composite resin restorations having glass-ionomer cement as cavity liner.

## Materials and methods

The materials included in the investigation are listed in Table 1 and were treated in accordance with the manufacturers' instructions. The investigation was divided into two sections, in which bonding and gap formation were studied.

Table 1. List of materials used in the investigation

Name	Manufacturer
Etching Gel (31% H <sub>3</sub> PO <sub>4</sub> )	ESPE, Seefeld, FRG
Ketac-Bond	
Ketac-Silver	
Visio-Bond	
Visio-Molar, light paste	
Durelon, liquid	
Concise	3M, St. Paul, USA
Silux, light paste	

### *Bond strength between glass-ionomer cement and composite resin*

Powder and liquid of the glass-ionomer cement (Ketac-Bond or Ketac-Silver) were mixed at room temperature and filled into a brass mould (diameter, 12.0 mm; height, 1.5 mm). The cement was covered on both sides with transparent plastic film and glass slabs and was left to set for 10 min at 37°C and 100% relative humidity. Specimens were then separated from the mold and either not etched or acid etched for 15, 30, or 60 sec, respectively. The etching gel was removed by spraying with water for 30 sec, and the specimens were gently dried. A thin film of light-activated resin (Visio-Bond) was applied to the surface of glass-ionomer cement and irradiated for 10 sec with a Translux unit (Kulzer & Co., FRG). A cylindrical Teflon mold (diameter, 3.5 mm; height, 7.0 mm) was now attached to the specimen and filled to half its height with light-activated composite resin (Visio-Molar or Silux), which was polymerized for 20 or 40 sec. After 24 h at 37°C and 100% relative humidity a round bur, to be attached in the Instron universal testing machine, was held in place as earlier described (14) and embedded in the self-curing resin (Concise) used to fill the other half of the Teflon mold. The specimen was left to set for 5 min at room temperature and thereafter placed in water at 37°C. After 6 days in water the bond strengths were measured under tension at a cross-head speed of 0.5 mm/min. Each of the above-mentioned combinations was tested on six specimens.

### *Gap formation*

Human teeth that had been kept in 1% chloramine since extraction were ground flat on wet no. 1000 carborundum paper until the surface of enamel and dentin was plane. Cylindrical butt-joint cavities were then prepared (diameter, 4.0 mm; height, 1.5 mm), half of whose cavosurface margin was placed in enamel and half in dentin. In the main series mixed glass-ionomer cement was applied with a ball-pointed instrument to form a layer covering the dentin part of the cavity, in correspondence with the technique depicted in Fig. 1. The cement was chamfered to the dentin wall, and an attempt was made to let the cement end exactly at the cavosurface margin. The cement was allowed to set for 10 min at 37°C and 100% relative humidity. Cement and enamel were acid-etched for 60 sec and water-sprayed for 20 sec. After the cavity had been dried with compressed air, Visio-Bond was applied and irradiated for 10 sec. The cavity was then filled with composite resin, covered with transparent plastic film, and polymerized for 40 sec. In additional series, various combinations of treatments and materials were investigated: in one series the dentin was treated with Durelon liquid for 15 sec and water-sprayed for 30 sec before glass-ionomer cement was applied. In another series a 45° bevel 0.5–1.0 mm wide was prepared in the cavosurface enamel margin. In other

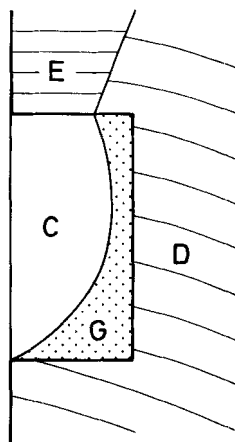


Fig. 1. The restorative technique. E = enamel; D = dentin; G = glass-ionomer cement; and C = composite resin.

Table 2. Materials and treatments used in measurements of wall-to-wall contraction and silver stain penetration

Code	Glass-ionomer cement	Composite resin	Time in water before polishing
A	Ketac-Bond	Visio-Molar	10 min
B*	Ketac-Bond	Visio-Molar	10 min
C†	Ketac-Bond	Visio-Molar	10 min
D‡	Ketac-Bond	Visio-Molar	10 min
E	Ketac-Silver	Visio-Molar	10 min
F	Ketac-Bond	Visio-Molar	24 h
G	Ketac-Bond	Silux	10 min
H	Ketac-Bond	Silux	24 h
J	Ketac-Bond	-	10 min
K	-	Visio-Molar	10 min
L	-	Silux	10 min

\* Resin irradiated 40 sec.

† Durelon pretreatment.

‡ Bevel along the enamel margin.

series the glass-ionomer cement lining was omitted, or the entire cavity was filled with glass-ionomer cement. Table 2 gives the combinations of treatments and materials investigated. After setting, the fillings were stored in tap water for 10 min or 24 h, and the tooth surfaces were then ground and polished gently. It was estimated that a few tenths of a millimeter of the surface were removed by this procedure. In the main series the polishing exposed the glass-ionomer cement at the dentin margin. Each of the above series consisted of six specimens.

#### *Wall-to-wall polymerization contraction*

In a light microscope with a measuring ocular (Leitz, Wetzlar, FRG;  $80 \times 0.8 \times 8$ ), the maximum width of contraction gaps was measured. The diameter of the cavity was also determined, in order that the wall-to-wall polymerization contraction could be calculated in per cent.

#### *Marginal leakage*

Marginal leakage was assessed by means of silver nitrate (15). In a series of preliminary experiments the tooth surfaces surrounding the restorations were sealed with a layer of nail polish. This method, however,

was rejected because no difference in extent of silver stain uptake could be detected between sealed and non-sealed specimens. It is considered impossible to apply the polish precisely around the restoration margins. Either a zone of non-sealed tooth surface remains around the restoration, making application of polish pointless, or varnish fills up the marginal gaps, preventing uptake of silver nitrate.

After being polished and dried, the teeth were immersed in 50% w/w aqueous silver nitrate solution for 2 h in the dark. The restorations were then rinsed in distilled water for 60 sec and immersed in rapid photo-developer solution (Kodak Dektol, Kodak A/S, Albertslund, Denmark) for 3 h, exposed to laboratory light. The teeth were rinsed in water and sectioned through the middle of the restorations in an occlusoapical direction with a diamond disc. The enamel/composite resin, the composite resin/glass-ionomer cement, and the glass-ionomer cement/dentin interfaces of each restoration were examined in a stereomicroscope with a measuring ocular (Leitz, Wetzlar, FRG;  $18 \times 1$ ), and the length (in mm) of silver stain penetration along the three interfaces was measured. From the two halves of each restoration a mean value was calculated for each interface.

MPa

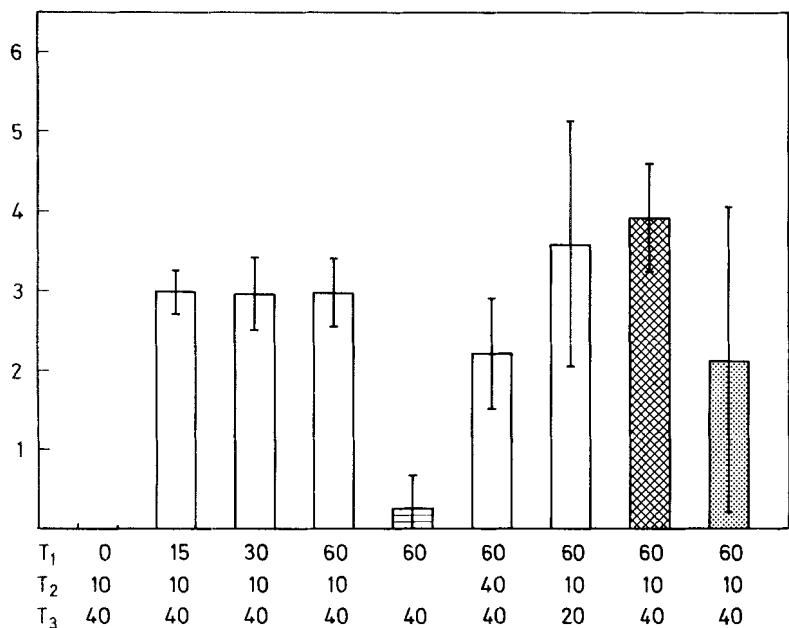


Fig. 2. Mean bond strengths and standard deviations (vertical lines). Open columns: Ketac-Bond, Visio-Bond, and Visio-Molar; striped column: Ketac-Bond, and Visio-Molar; cross-hatched column: Ketac-Bond, Visio-Bond, and Silux; and dotted column: Ketac-Silver, Visio-Bond, and Visio-Molar. T<sub>1</sub> = etching time (sec); T<sub>2</sub> = irradiation time of resin (sec), and T<sub>3</sub> = irradiation time of composite resin (sec).

## Results

The results are presented in Fig. 2 and in Table 3. The statistical treatment involved analysis of variance and Student's *t* test (16).

### Bond strengths

Mean values and standard deviations of the bond strengths are shown in Fig. 2. Acid etching was necessary to obtain any bond between composite resin and glass-ionomer

Table 3. Wall-to-wall contraction and silver stain penetration

Code	Wall-to-wall polymerization contraction (%)	Length of silver stain uptake at the interface indicated (mm)	
		Enamel/composite resin	Dentin/glass-ionomer cement
A	0.082 ± 0.025	0.44 ± 0.66	1.09 ± 0.43
B	0.134 ± 0.032	0.17 ± 0.14	1.12 ± 0.28
C	0.136 ± 0.039	0.45 ± 0.60	1.36 ± 0.30
D	0.068 ± 0.013	0.17 ± 0.30	1.04 ± 0.44
E	0.141 ± 0.038	0.95 ± 0.30	0.88 ± 0.45
F	0.178 ± 0.055	0.75 ± 0.70	1.52 ± 0.84
G	0.235 ± 0.048	0.02 ± 0.02	1.22 ± 0.23
H	0.103 ± 0.021	0.09 ± 0.14	1.53 ± 0.42
J	0.313 ± 0.085	0.90 ± 0.51*	1.20 ± 0.43
K	0.350 ± 0.088	0.58 ± 0.70	3.19 ± 1.98†
L	0.417 ± 0.087	0.00 ± 0.00	1.91 ± 0.67†

\* Enamel/glass-ionomer cement interface.

† Dentin/composite resin interface.

cement ( $P < 0.005$ ), but the duration of the etching seemed insignificant ( $P > 0.05$ ). Application of resin was indispensable ( $P < 0.001$ ), but irradiation for 40 sec did not yield higher but rather lower bond strengths ( $P \approx 0.05$ ). Ketac-Silver gave rise to the same bond strengths as Ketac-Bond ( $P > 0.05$ ). Halving the irradiation time of Visio-Molar did not have any statistically significant influence ( $P > 0.05$ ).

When the values obtained with Visio-Molar on Ketac-Bond were pooled, a bond strength of  $2.96 \pm 0.89$  was obtained. In comparison, Silux on Ketac-Bond gave a bond strength value that was not significantly higher ( $P > 0.1$ ).

#### *Marginal gaps*

Table 3 shows the wall-to-wall polymerization contraction and the length of silver stain penetration.

#### *Wall-to-wall polymerization contraction*

An analysis of variance showed that the differences in wall-to-wall contraction were statistically significant ( $P < 0.0005$ ). Student's *t* tests showed the following with regard to materials and treatments: The restorations without a glass-ionomer cement lining had larger wall-to-wall contraction than those with a lining (K versus A, L versus G;  $P < 0.001$ ). When polishing was postponed for 24 h, the wall-to-wall contraction of Visio-Molar was increased ( $P < 0.005$ ; A versus F), whereas that of Silux was decreased ( $P < 0.0005$ ; G versus H). Polishing after 10 min meant a lower wall-to-wall contraction for Visio-Molar than for Silux ( $P < 0.001$ ; A versus G). After 24 h, however, the wall-to-wall contraction of Silux was statistically lower than that of Visio-Molar ( $P < 0.01$ ; F versus H). Pretreatment with Durelon increased the contraction ( $P < 0.01$ ; A versus C), which was also the case with prolonged irradiation of resin ( $P < 0.01$ ; A versus B). Preparation of a bevel did not decrease the wall-to-wall contraction with statistical significance ( $P > 0.05$ ; A versus D). The contraction obtained with Ketac-Silver was higher than with Ketac-Bond ( $P < 0.01$ ; A versus E).

Restorations made only of glass-ionomer cement resulted in a wall-to-wall contraction similar in magnitude to that obtained with the restorative resins ( $P > 0.05$ ; J versus K/L).

The location of the marginal gaps was found to vary with the technique. In the enamel area only seven cases of gap formation were observed: one specimen belonging to group F and the specimens belonging to group J showed gaps. In the first case the gap was located between the enamel and the restorative resin; in the other cases it was located cohesively within the glass-ionomer cement. In the dentin area a gap was observed in all cases. In groups K and L the gaps were located between restorative resin and dentin. In group J, gaps were also observed within the glass-ionomer cement near the cavity wall. In the other groups the location of the gaps was less well definable, possibly because a film of unfilled resin was often observed between glass-ionomer cement and dentin, even though the unfilled resin was applied *after* the glass-ionomer cement. Accordingly, gaps were located between the various possible combinations of materials, except between unfilled and filled resin. Occasionally, gaps were observed within the glass-ionomer cement. No pattern in the location of the gaps could be established.

#### *Silver stain penetration*

No penetration of silver stain was recorded at the resin/glass-ionomer cement interface, and the corresponding column was thus omitted from Table 3. In contrast, penetration of silver stain was found between enamel and composite resin and between dentin and glass-ionomer cement. The depth of penetration varied considerably; in the enamel area it was between 0 and 0.95 mm and in the dentin area between 0.88 and 3.19 mm. The length of silver stain penetration at each of the two interfaces was by analyses of variance found to differ with statistical significance ( $P < 0.0005$ ). When K and L were left out of the analyses of the dentin/glass-ionomer cement interface results, the differences were no longer of

statistical significance. Thus, restorations consisting of composite resin only showed a higher extent of silver stain penetration along the dentin margin than restorations with a glass-ionomer lining.

Use of Silux with a glass-ionomer cement liner resulted in less penetration at the enamel/composite resin interface than Visio-Molar when polishing was postponed for 24 h ( $P < 0.05$ ; F versus H). In restorations consisting of Silux only penetration was also less than in restorations of glass-ionomer cement only ( $P < 0.002$ ; J versus L). Pretreatment with Durelon, preparation of a bevel, or prolonged irradiation of resin did not reduce the depth of silver stain penetration at the enamel margin. Ketac-Silver and Ketac-Bond resulted in the same penetration depths ( $P > 0.05$ ; A versus E).

## Discussion

### *Bond strength*

The results have shown that composite resin was not able to bond, through a resin layer, to non-etched surfaces of glass-ionomer cement. Causton et al. (17) found that etching with 30% phosphoric acid produced deep etch patterns in the cement surface, facilitating creation of a mechanical bond. In agreement with this, we found higher bond strengths after etching with phosphoric acid. The time used to etch the enamel was 60 sec. No difference in bond strength was measured after etching the glass-ionomer cement for 15, 30, or 60 sec. Thus, in the subsequent experiments an etching time of 60 sec was chosen for practical reasons. In agreement with earlier measurements (17) we found that an intermediate layer of resin was necessary as a 'wetting agent' under the rather stiff composite resins.

It has previously been found (17) that the mechanically mixed silver-'reinforced' glass-ionomer cement (Ketac-Silver) was after etching less porous than the hand-mixed cement (Ketac-Bond). This may have influenced the highly varying and slightly lower bond strengths obtained with Ketac-Silver.

The lower porosity of Ketac-Silver may

also account for the fact that the fractures were adhesive, while the fractures of Ketac-Bond, the more porous material, were cohesive within the cement. Thus, when Ketac-Bond was acid-etched and coated with resin, the bond strengths were higher than the bulk strength of the cement itself.

With regard to the composite resins, irradiation for 20 sec sufficed to polymerize Visio-Molar fully, giving as high a bond strength as obtained with 40 sec of irradiation.

### *Wall-to-wall polymerization contraction*

The finding that a glass-ionomer lining beneath the restorative resins reduced the wall-to-wall contraction of the resins may be explained by the adhesion between glass-ionomer and tooth and between restorative resin and etched glass-ionomer. It is in agreement with earlier findings that adhesive layers between composite resin and dentin cavity walls reduce the gap formation (18).

The gaps obtained with Silux in cavities lined with Ketac-Bond were smaller after 24 h than after 10 min. A trend in the opposite direction was observed with Visio-Molar in similar cavities. This disparity in behavior between Silux and Visio-Molar is presumably due to differences in hygroscopic expansion between the two materials. Silux has a relatively large hygroscopic expansion and will therefore show a tendency to close marginal gaps (18). Visio-Molar, however, has a hygroscopic expansion that is virtually nil and is unable to reduce the size of marginal gaps (19). The reason that the gaps increased with Visio-Molar may be that the polymerization contraction continued beyond 10 min after irradiation.

Pretreating the dentin with Durelon liquid, a solution of polyacrylic acid, did not have a reducing effect on wall-to-wall contraction. This finding contrasts with that of Powis et al. (1), who measured increased bond strength between dentin and glass-ionomer after pretreating the dentin with polyacrylic acid. An explanation may be found in possible differences in the thickness of the dentinal smear layer. When the smear layer is thick, pretreatment with polyacrylic acid

will reduce the thickness of the smear layer without affecting the structure of underlying dentin. A better bonding may be the result. When the smear layer is thin, an acidic pretreatment will remove not only the smear layer but also calcium from underlying dentin. Since glass-ionomer cements bond by attachment to calcium in the dentinal surface, a reduction in bond strength may be the result.

The reason why preparation of a bevel did not reduce the wall-to-wall contraction in the present investigation may be that only the enamel half of the cavity margin was beveled. Withdrawal from the gingival margin could therefore take place with the same ease, irrespective of the presence of an enamel bevel. The increase in wall-to-wall contraction found with beveled cavities, although not statistically significant, may be related to the earlier finding that the contraction of a restorative resin increases with increasing diameter of the cavity (18).

Ketac-Silver was less effective than Ketac-Bond in reducing the width of marginal gaps, possibly because Ketac-Silver yields a smaller bond to dentin than does Ketac-Bond (20).

Prolonged irradiation of the intermediary

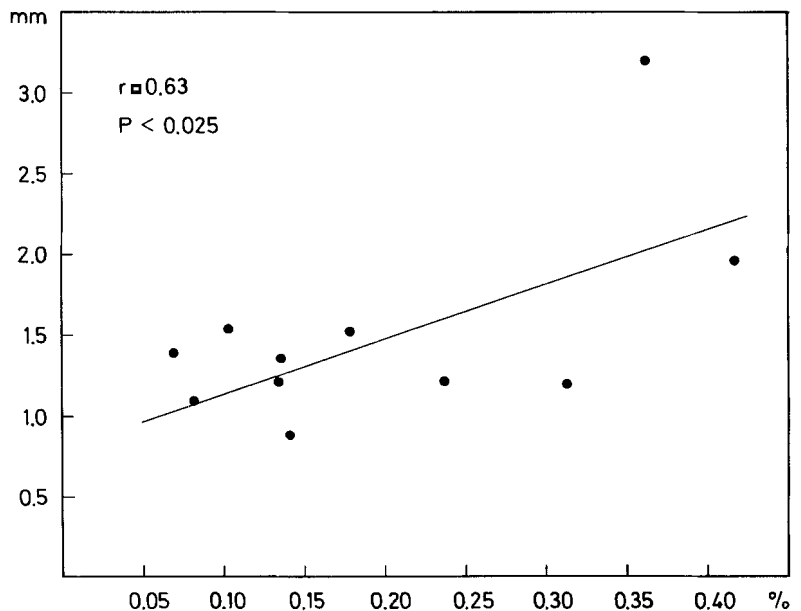
resin resulted in larger wall-to-wall polymerization contraction. It is conceivable that irradiation generates heat in such quantities that the glass-ionomer cement becomes less effectively bonded to the dentin. The finding that the bond strength between glass-ionomer cement and composite resin decreased when the intermediary resin was illuminated for a prolonged period may also have played a role.

The observation that unfilled resin in quite a few cases was located between glass-ionomer cement and dentin is in agreement with the findings of Crim & Shay (20). Obviously, a debonding of the glass-ionomer cement takes place in these cases, enabling penetration of the unfilled resin. The debonding may be due to contraction of the glass-ionomer cement or to penetration of the applied phosphoric acid along the dentin/glass-ionomer interface. Another possibility is that unfilled resin has penetrated through cracks created in the glass-ionomer cement during the etching (21).

#### Silver stain penetration

Application of a glass-ionomer liner meant less silver stain penetration at the dentin

Fig. 3. Correlation between wall-to-wall polymerization contraction in percentage (abscissa) and length of silver stain penetration in millimeters (ordinate) at the dentin/restorative interface.



margin. Despite the ability of glass-ionomer cement to bond to the hard tissues of teeth, microleakage always occurred to a greater or lesser extent, dependent on the technique. The finding that glass-ionomer cement cannot totally avert gap formation and microleakage is in agreement with earlier investigations (20, 22, 23).

In the present study gap formation was assessed by two methods: measurement of wall-to-wall contraction and silver stain penetration. The relationship between the two methods was analyzed by regression analysis of the data presented in Table 3. No correlation was present between the contraction and penetration of silver stain in the enamel area, but in the dentinal area a significant positive correlation could be established, as shown in Fig. 3. The coefficient of correlation was  $r = 0.63$ , which was different from zero at  $P < 0.0025$ . Consequently, both methods are applicable, but owing to the smaller coefficients of variation, the wall-to-wall contraction has the greater discriminating power.

On the basis of the present investigation we conclude that gap formation may be reduced but not eliminated by use of a glass-ionomer liner under composite resin.

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