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Laboratory evaluation of eight pit and fissure sealants

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Abstract

The penetration coefficients (PCs) and tensile bond strengths of eight pit and fissure sealant materials were determined. The PCs were tested at 22°C with five samples of each resin. Bond strengths were established in a tensile mode using an Instron testing machine with 10 specimens for each sealant. Despite considerable variation in the PC values, all sealants showed similar tensile bond strengths. Further, the filled sealants, which are very viscous, demonstrated similar bond strengths to the unfilled resins.*

Epidemiological studies carried out in the 1930s and 1940s showed that nearly 100 per cent of individuals living in western populations suffered from dental caries.[1,2] More recent studies indicate that industrialized nations have a decreasing caries incidence.[3] Despite a slight decline in the overall caries rate, tooth decay remains a major health problem in the United States and other industrialized countries.[3]

Longitudinal studies of pit and fissure caries have demonstrated the early onset and rapid rate at which occlusal decay occurs. Lewis and Hargreaves[4] found that 70% of the first permanent molars of subjects residing in a nonfluoridated area had pit and fissure decay within one year after eruption. This study demonstrated the need for early treatment of all pit and fissure areas. King, Shaw, and Murray[5] later found that 92% of first molars and 68% of second molars were decayed, missing, or filled by age 15; pits and fissures provide an excellent habitat for opportunistic cariogenic microorganisms.

The caries susceptibility of occlusal surfaces and the relative ineffectiveness of fluorides and mechanical plaque control to prevent decay have led to new preventive methods. The technological development of dental resin systems has permitted mechanical obturation of pits and fissures for caries prevention. Clinical trials have demonstrated effective long-term caries reductions with a number of different sealant products.[6,7]

The effectiveness of sealants depends on their ability to penetrate fissures before hardening, thus producing a mechanical barrier to caries. To produce bonding and retention, the sealant material must flow over the etched enamel surface and penetrate micropores in the etched surface. Because of this intimate relationship it was felt by some investigators that the penetrative ability of a sealant would affect its ability to bond to enamel.[8,9] Resin penetration, however, was found to be dependent on the underlying etch pattern, wetting ability of the enamel, and the materials' surface tension, viscosity, and rate of polymerization. [8,9]

The purpose of this in vitro investigation was to evaluate eight commercially available sealant materials. The parameters examined were the penetration coefficients (PCs) and tensile bond strengths. Scanning electron microscopy (SEM) was used to examine the fractured tensile bond strength specimens.

Methods and Materials

The eight sealants are commercially available Bis-GMA resins. Four of the sealants[a-d] are chemically activated two-component systems. Two of the sealants[e,f] are polymerized by ultraviolet light, and one of these[f] contains 64% lithium aluminum silicate filler particles. Two new visible light activated systems[g,h] also were examined. One of these materials[g] is filled, containing 64% silicate particles.[10]

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* Instron Corp.; Canton, Mass. 02021.

[a] Delton Pit and Fissure Sealant System, Johnson & Johnson; East Windsor, N.J. 08520.

[b] Delton Tinted Pit and Fissure Sealant System, Johnson & Johnson; East Windsor, N.J. 08520.

[c] Concise White Sealant System, 3M Company; St. Paul, Minn. 55101.

[d] Concise Enamel Bond System, 3M Company; St. Paul, Minn. 55101.

[e] Nuva Seal, L.D. Caulk Dental Products; Milford, Del. 19963.

[f] Nuva Cote, L.D. Caulk Dental Products; Milford, Del. 19963.

[g] Prisma Shield, L.D. Caulk Dental Products; Milford, Del. 19963.

[h] Visio Seal, Premier; Romano Dr.; Norristown, Pa. 19401.

Penetration Coefficient

The penetration rate of a liquid under its own capillary force into an open, horizontal capillary tube can be derived from Poiseuille's law.[9] The PC of a resin can be calculated from the surface tension of the resin (γ), the viscosity of the resin (η), and the contact angle of the resin on a capillary wall (θ). Determination of the parameters γ, η, and θ is difficult and requires sophisticated instrumentation. As the PC is equal to the square of the distance penetrated into a horizontal capillary tube of unit radius in unit time under capillary pressure, the PC of a sealant can be obtained from the slope of the straight line of the length of the liquid column squared (x^2) versus time (t).[9]

$$PC = \frac{\text{Slope}}{r}$$

(r = the radius of the capillary tube)

Penetration coefficients were obtained for each sealant using a method developed by Fan et al.[9] The two filled sealants could not be tested due to their extreme viscosity. Sections of thick-walled glass capillary tubes were cut into four-inch lengths. The diameters were determined with a traveling microscope[i] and the tubes cleaned overnight in concentrated nitric acid, washed and dried. An aluminum reservoir[j] was attached to the end of the tube and the entire apparatus was taped to graph paper on a horizontal surface.

The sealants were placed in the reservoir, the two component systems having been mixed according to manufacturers' instructions. The distance (x) penetrated in millimeters was measured and recorded directly on the graph paper at 5-second intervals (t). All the PC tests were conducted at room temperature (approximately 22°C). A straight line was produced by plotting time (t) in seconds on the horizontal axis and the values of distance squared (x^2) in centimeters on the vertical axis (Figure 1). The slope was calculated by regression analysis and the PC of each sealant was determined using a derivation of Poiseuille's law.[9] A computer-generated random table was used to assure a random testing sequence with five PC values being determined for each material.

Tensile Bond Strength

The test method used for determining tensile bond strength was developed by Kemper and Kilian[11] and modified by Retief and Mallory.[12] Extracted noncarious human permanent molars, which had been stored in 70% ethanol, were used. The crowns of the teeth were separated from the roots and shaped so that the mesial or distal surface could be oriented upwards in a specimen cup. Retention grooves were prepared on the occlusal and pulpal surface of each tooth. Epoxy resin was used to embed each crown in a tooth specimen cup with the mesial or distal surface projecting above the lip of the cup. Half the specimens were lathed on a machine to produce circular pegs 2 or 3 mm in diameter projecting from the tooth surface. The 3 mm pegs were used with the chemical and visible light activated systems. Due to the limited polymerization depth wth ultraviolet light systems (±2 mm), the 2 mm pegs were used for these materials. Just prior to testing, each specimen was wet polished using 320, 400, and 600 silicon carbide discs, respectively. A polishing block was used to ensure a planar enamel surface which would be perpendicular to the direction of the applied tensile forces during testing.

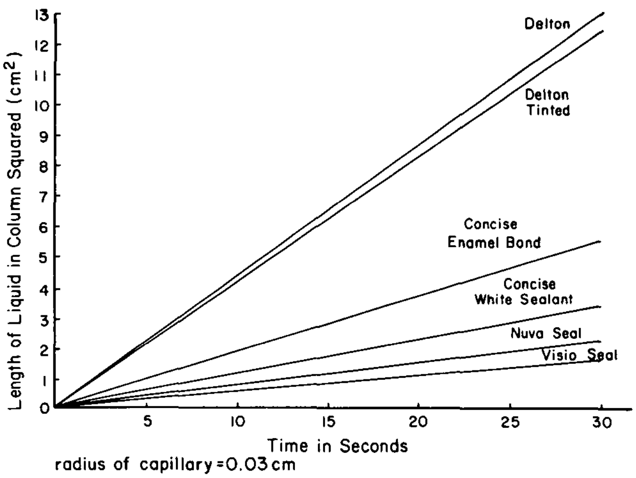

Figure 1. (left) The sealant penetration coefficients are represented graphically by an x^2 vs t plot.

The enamel surfaces were conditioned for 60 seconds with the etching agent supplied by the manufacturers, rinsed thoroughly with water, and dried with oil-free air. Two tooth cups were mounted in a bonding alignment block to ensure that the prepared enamel surfaces were aligned parallel to each other. The resin systems were used according to the manufacturers' instructions and applied to the conditioned enamel surface of the lower peg. The upper tooth cup was lowered so the conditioned enamel surface contacted the peg and any excess material was wiped away gently with a cotton pledget. A one-pound load was placed on the specimen and polymerization was allowed to progress for 15 minutes. Visible light materials were polymerized by directing the light from three different positions for 1 minute each. The ultraviolet light systems were given 2-minute exposures from three different directions. Excessive light exposures were used to ensure complete polymerization. The bonded unit was removed from the mounting block and immersed in water at 37°C for 24 hours to allow for further polymerization.

A specimen alignment block was used to mount the bonded specimen cups in the jaws of an Instron[k] testing machine. A cross head speed of 0.02 in./min. was used and the specimens were stressed to failure in the tensile mode. The force required to break the bond was recorded in pounds and the tensile bond strength calculated and

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[i] Gaertner Scientific Corp.; Chicago, Ill. 60611.

[j] Pure Aluminum Heavy Duty Foil, S.S. Kresge Co.; Troy, Mich. 48084.

[k] Instron Corp.; Canton, Mass. 02021.

expressed in MN/m^2. Ten values were obtained for each material with the sequence for testing being derived from a computer-generated random table.

Results

The means (±SD) of the penetration coefficients of the eight materials are presented in Table 1. A one-way analysis of variance was used to analyze the data accepting a $p < 0.05$ level of significance. The Delton materials had significantly higher PC values compared to the other products, while Nuva Seal and Visio Seal had the lowest PC values (Figure 1). The Concise materials were in the middle range and were significantly different from each other.

The means (±SD) of the tensile bond strengths are given in Table 2. There were no significant differences among the tensile bond strengths of any of the eight materials at $p < 0.05$ using a one-way analysis of variance.

Examination of the fractured tensile bond specimens with SEM revealed that failure occurred at the enamel/resin interface, in the resin and in the enamel. None of the specimens examined had fractures which were exclusively interfacial between the resin and enamel (Figure 2). Enamel fractures were seen in a large number of specimens with wide variation in the extent of fracture. In many specimens there were only small fractures within the enamel involving a few prisms (Figure 3). In other specimens large fractures occurred in the enamel extending to the dentin-enamel junction or even into the dentin. Fracture of the sealant material often was associated with small air bubbles trapped in the sealant. The fracture front sometimes appeared to occur along a line of bubbles. Failure within the material or cohesive fracture produced linear wave-like patterns in the sealant which radiated from the area of material failure (Figure 4). Cohesive failure in the filled sealants produced rougher fractured surfaces.

Discussion

The PCs of pit and fissure sealants obtained in this study were generally consistent with those reported previously.[9,10] The Delton materials were found to have a slightly increased PC (±12 cm/s) compared to values in the literature (7-9 cm/s).[12,13] The different sealants tested showed considerable variation in PC values ranging from the free-flowing Delton (12.7 cm/s) to the rather viscous Visio Seal (1.6 cm/s). However, all unfilled sealants tested had PCs above the 1.3 cm/s value which O'Brien et al.[13] indicated could fill 93% of a test fissure. Prisma Shield and Nuva Cote have filler particles making them quite viscous compared to any of the unfilled sealants.

Table 1. Penetration Coefficients of Six Sealant Materials,

Resin System	*Number of Specimens*		*Mean PC cm/sec*		*± SD cm/sec*	*Coefficient of Variation %*
Delton	5	*	12.7	±	1.1	8.4
Delton Tinted	5	*	12.2	±	0.5	3.7
Concise Enamel Bond	5		5.2	±	0.5	9.9
Concise White Sealant	5		3.4	±	0.2	6.9
Nuva Seal	5	**	2.2	±	0.3	15.5
Visio Seal	5	**	1.6	±	0.2	10.5

Means grouped consecutively by a similar number of asterisks (* or **) are not significantly different at $p < 0.05$.

Table 2. Tensile Bond Strengths of Eight Sealant Materials

Resin System	*Number of Specimens*	*Mean Bond Strength MN/m^2*		*± SD MN/m^2*	*Coefficient of Variation %*
Delton	10	26.6	±	8.9	33.4
Delton Tinted	10	27.5	±	3.8	13.7
Concise Enamel Bond	10	27.5	±	6.8	24.8
Concise White Sealant	10	27.6	±	5.0	18.1
Nuva Cote	10	26.1	±	6.0	23.1
Nuva Seal	10	22.4	±	8.5	37.8
Prisma Shield	10	30.4	±	7.3	23.9
Visio Seal	10	29.8	±	8.5	28.7

There were no significant differences in bond strengths among materials at $p < 0.05$.

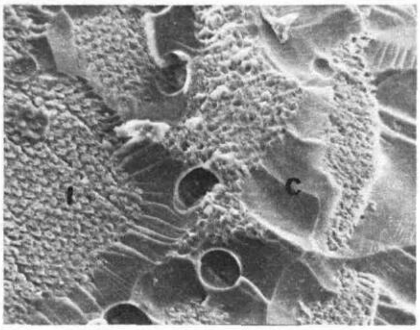
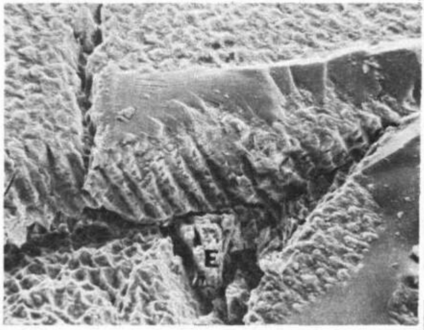


Figure 2. (right) The complex sealant fracture patterns involving interfacial (I) and cohesive (C) failure (500x).

Figure 3. Failure in the tensile bond specimens often involved small areas of enamel (E) (SEM 800x).



Comparison of the tinted sealants to their nontinted counterparts revealed the affects on the PC by different tinting agents. Delton Tinted Sealants tinting agent, annatto vegetable dye, produces the yellow orange color of the material. Examination of the two Delton materials showed their PC values were not significantly different from each; this indicates that addition of the annatto dye does not alter the PC of the material significantly. Concise White Sealant is tinted with titanium dioxide, which produced a significant PC reduction when compared to Concise Enamel Bond. This PC reduction associated with the addition of titanium dioxide corroborates the work of Retief and Mallory.[12]

The significant differences seen from one sealant product to the next result largely from variations in the diluent monomers which have been added to the Bis-GMA resins.[10] Diluents such as methyl methacrylate, triethylene glycol methacrylate, and bisphenol A dimethacrylate are used alone or in combination.[10] These monomers are added to a mixture of the relatively viscous Bis-GMA resins to enhance their handling and application for sealants.[10] In addition to diluent composition and concentration, the addition of titanium dioxide as a tinting agent may affect the viscosity and PC of sealants. Other sealant components which might influence a sealant's PC are filler particles, storage stabilizers, and plasticizers.[10]

Determination of the tensile bond strengths revealed higher values (22-30 MN/m^2) for the eight materials compared to earlier studies. Low et al.[14] concluded that a tensile bond strength range of 2.4-3.4 MN/m^2 could be considered adequate for pit and fissure sealant retention. The high tensile bond strengths may reflect the ideal conditions under which the sealants were evaluated. The enamel was polished on 600 grit silicon carbide discs while acid etching and sealant placement were accomplished without risk of moisture or oil contamination. Wide variations between this and previous studies demonstrate the dependence of bond strengths on the specific test system. Differences in the testing method, enamel structure, material handling, and storage time of the bonded units prior to evaluation may all affect bond strength. In this study enamel was obtained from the approximal surfaces of permanent molars. Previous studies often utilized maxillary central incisors because of the flat labial surfaces.[12]

Diametral tensile strengths of sealants reported by Dennison and Powers[15] were similar in magnitude to the tensile bond strengths determined in this study. The diametral tensile test essentially evaluates the force necessary to create cohesive fracture of the material in a tensile mode. It reflects the material's inherent strength. Comparison of diametral strengths and tensile bond strengths from the literature showed that the diametral strength was several times greater than the bond strength to etched enamel.[12,15] This implies that the material's strength is significantly greater than the enamel/resin bond strength. Bond strength values determined in the current study, however, were similar in magnitude (22-30 MN/m^2) to those reported for diametral tensile strengths (20-33 MN/m^2).[15] Thus, our results indicate that the resin/enamel bond was approaching or reaching the strength of the resin itself. This finding was corroborated by SEM analysis where none of the fractured tensile bond specimens failed exclusively at the resin/enamel interface. Areas of failure within the material were seen in every specimen examined.

Prisma Shield, a filled sealant, produced the highest tensile bond strength despite being one of the more viscous materials. Nuva Cote, which is also a filled resin, had a bond strength comparable to the other sealants. This supports the finding that filler particles will not prevent the intimate contact between the monomer resin and etched enamel which results in bonding.[16] Retief and

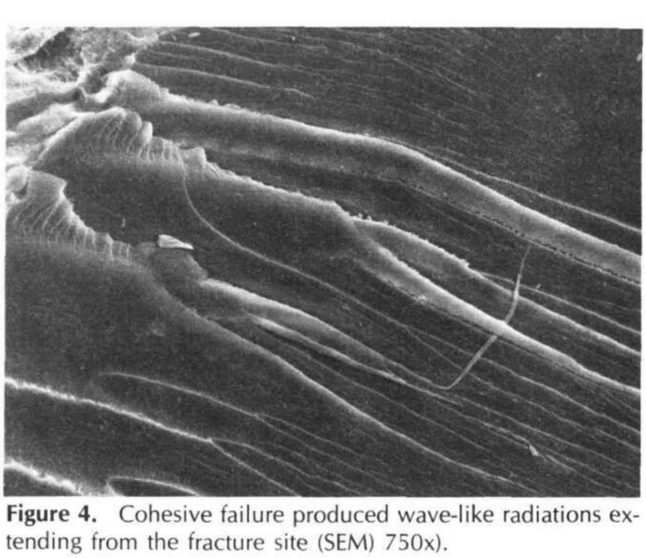


Figure 4. Cohesive failure produced wave-like radiations extending from the fracture site (SEM) 750x).

Woods[17] demonstrated the ability of filled composites to produce adequate bond strengths without an intermediate low viscosity bonding resin. The presence of filler particles thus does not appear to hinder or enhance the formation of a strong resin/enamel bond in filled sealants.

SEM evaluation of the fractured tensile bond specimens confirms previous reports that failure may occur in the resin, enamel, at the resin/enamel interface, or in any combination thereof.[18,19] All specimens examined displayed at least interfacial and cohesive resin failure with many having enamel fractures. The fractured tensile bond strength specimens from this laboratory study demonstrated similar modes of failure as have been implicated in sealant loss clinically.[20] The cohesive wave-like fractures appeared quite similar to the patterns which occur clinically.[20] The extensive enamel fracturing that occurred in some specimens may be a result of the high tensile bond strengths and also may be related to the degree of enamel manipulation and desiccation. Large enamel fractures have not been described with the loss of sealants clinically. Cohesive sealant failure in vitro thus appears to reflect the modes of sealant failure in vivo.

This study indicates that the PC (flow properties) of a pit and fissure sealant may be of minimal concern clinically. Thus, when selecting a sealant for clinical application, the practitioner should use criteria such as ease of application, tinted versus clear, and filled versus unfilled. Variation in the flow properties of different commercial sealants appears to be an insignificant factor in achieving an adequate enamel/resin bond.

Conclusion

Although the PCs of eight sealant materials varied significantly, there were no significant differences in the resins' tensile bond strengths to etched enamel. Similar tensile bond strengths were obtained regardless of the materials' PC value or the presence of filler particles. All commercial sealants evaluated had flow properties adequate for producing a strong enamel/resin bond.

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