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In vitro investigation of the tensile bond strengths of a chemically initiated and a visible light-initiated sealant with SEM observations

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Abstract

Two pit and fissure sealants, Delton and Helioseal, were evaluated in this study.

Tensile bond strengths of these 2 resins were evaluated to determine their retention in occlusal fissures in permanent and primary teeth in vitro using an Instron testing unit. The results showed significant differences among types of teeth and types of materials, with the Delton having the highest value for both types of teeth. The Delton sealant was retained on primary teeth almost as well as on permanent teeth.

Scanning electron microscopy was used to evaluate the failure site of the sealants. A difference was noted among mode of failure of both materials.

Many researchers have developed and used tensile-bonding tests to evaluate adhesive-enamel bond strengths.[1] A new method was used in the current study by utilizing the occlusal surfaces to simulate the in vivo environment instead of using approximal surfaces of the permanent molars (Wright and Retief 1984) or labial surfaces of maxillary central incisors (Retief and Mallory 1981).

Much research has been reported in Ripa's review (1979) including reports that sealant retention in primary teeth appears to be comparable to that obtained in permanent teeth. In the present study, permanent as well as primary teeth were evaluated.

The objective of this study was to conduct an in vitro evaluation of 2 fissure sealants, chemically polymerized Delton[a] (Bis-GMA resin) and visible-light sealant Helioseal[b] (urethane dimethacrylate resin), by measuring tensile bond strength. Scanning electron microscopy (SEM) was used to examine the modes of sealant failure in fractured tensile bond specimens.

Methods and Materials

A 2 × 2 factorial treatment design with completely randomized experimental design (CRD) was used to evaluate 2 fissure sealants. The sealants' tensile bond strengths were determined by using the Instron testing machine and examining the modes of sealant failure in fractured specimens by using SEM.

Twenty extracted noncarious human permanent maxillary first premolars and 20 noncarious primary maxillary first molars were used in this study. The age range of the subjects was 8–14 years. The teeth were embedded in cold-cure acrylic in a plastic tube with the crown projecting above the lip of the tube. The occlusal surface of the specimen was cleaned thoroughly with fluoride-free prophylaxis paste and a prophylaxis brush using a slow-speed handpiece. The occlusal surfaces of the permanent teeth and the primary teeth were conditioned for 1 min and 2 min, respectively, with the etching agent supplied by the manufacturers (37% phosphoric acid), rinsed thoroughly with water for 30 sec and dried with oil-free air for 30 sec. Orthodontic ligature wire (0.014) was used to hold orthodontic mesh[c] of 4 mm diameter. The mesh had been adapted to the occlusal surface of the tooth before conditioning the enamel. The resin systems were used according to the manufacturer's instructions and applied to the conditioned enamel surface. One dropful was used for Delton, one-half dropful on the conditioned enamel, and one-half above the mesh. The wire and the mesh were held in position to allow polymerization. The specimens then were stored in distilled water for 24 hr for more

[a] Delton—Johnson & Johnson Dental Products Co; East Windsor, NJ.
[b] Helioseal, Isosit—Vivadent, Inc; Tonawanda, NY.
[1] Low et al. 1975; Kemper and Kilian 1977; Retief and Mallory 1981; Wright and Retief 1984.

[c] General Purpose Mesh—TP Laboratories.

TABLE 1. Tensile Bond Strengths for 2 Fissure Sealants as Affected by Tooth Material

Resin System	Number of Specimens	Mean Bond Strength MN/m^2		Standard Error (SE) MN/m^2	Coefficient of Variation %
Delton on permanent teeth	10	9.345	±	0.227	7.687
Helioseal on permanent teeth	10	6.074		0.242	12.634
Delton on primary teeth	10	7.206		0.329	14.465
Helioseal on primary teeth	10	4.746	±	0.171	11.418

polymerization. Two drops were used for Helioseal, 1 on the conditioned enamel and 1 on the top of the mesh. The Heliomat Model A Light system 120 V-60 Hz 2 AMPS Light was used in the current study. The light lamp was activated for 20 sec to polymerize the resin. An additional activation of 5 sec was used to insure complete polymerization. The specimens also were stored in distilled water for 24 hr.

A specimen alignment attachment was used to align the specimen tubes in the jaws of the Instron testing machine so that the force applied on the specimen was perpendicular to the floor. A crosshead speed of 0.5 mm/min with a cell load of 2500 kg was used and specimens were stressed to failure in the tensile mode. The force required to break the bond was recorded in kg and the tensile bond strength calculated by using the following equation:

$$\text{Tensile Bond Strength} = \frac{\text{Load (kg)}}{\text{Surface Area (mm)}^2}$$

The data were converted to and expressed in Mega Newtons/meter^2 (MN/m^2) or Mega pascals (MPa) by multiplying the bond strengths by 9.807. Ten values were obtained for each material and for each type of teeth. The data were analyzed for differences using a 2-way ANOVA at the 0.01 level of significance.

Scanning Electron Microscopy

After determining the peak force required to break the bond, the crowns of the teeth were separated from the roots and split into labio-lingual halves. This was accomplished by using an electrical saw and water. The cutting disk was 300 μ in thickness. The teeth then were decalcified by immersing them in 70% nitric acid for 30 sec, rinsed and dried. All SEM specimens were mounted on aluminum stubs and coated with gold palladium by using vacuum evaporation at 200–300 A°. The specimens were examined with the SEM Cambridge Stereoscan S4-10 to establish the mode and site of failure.

TABLE 2. Analysis of Variance (ANOVA)

Source of Variability	df	SS	MS	F	PR > F
Treatment	3	113.806	37.9356		
P	1	30.045	30.045	48.350*	0.01
M	1	82.113	82.113	132.141*	0.01
P×M	1	1.647	1.647	2.651	0.112
Error	36	22.370	0.621		
Total	39	136.177374			

* Significantly different at level $P < 0.01$; P = Teeth (permanent and primary teeth); M = Materials (Delton and Helioseal fissure sealants); P×M = Interaction between teeth and materials.

Results

Tensile Bond Strength

The mean ± standard error (± SE) of the tensile bond strengths of the 2 materials used in this study are presented in Table 1. Statistical analysis of the data was accomplished by using 2-way ANOVA, accepting an alpha level of 0.01 of significance (Table 2). There were significant statistical differences among the materials—8.276 MN/m^2 for Delton fissure sealant and 5.410 MN/m^2 for Helioseal fissure sealant at level $P < 0.01$ (Table 3). In addition, Delton showed high tensile bond strength in both primary and permanent teeth, while Helioseal showed low tensile bond strength in both types of teeth. There were significant statistical differences among the teeth at level $P < 0.01$. The permanent teeth showed high tensile bond strength with both materials compared with the primary teeth (Table 3). It was interesting to note that the mean tensile bond strength of Delton in the primary teeth was higher than that of Helioseal in the permanent teeth (Table 1). Statistical analysis showed there were no interactions by teeth and materials at the 0.01 level (Table 2).

TABLE 3. Means of Tensile Bond Strengths Among Teeth and Materials

	P_1 MN/m^2	P_2 MN/m^2	
M_1 MN/m^2	9.345	7.206	8.276^a
M_2 MN/m^2	6.074	4.746	5.510^b
	7.709^c	5.976^d	$\bar{x} = 6.843$

Average coefficient of variation (CV) = 11.519%; Mean square of the error (MSE) = 0.621; ^{a,b} = Means of the materials in the same column are significantly different at level $P < 0.01$; ^{c,d} = Means of the teeth in the same row are significantly different at level $P < 0.01$; P_1 = Permanent teeth; P_2 = Primary teeth; M_1 = Delton fissure sealant; M_2 = Helioseal fissure sealant.

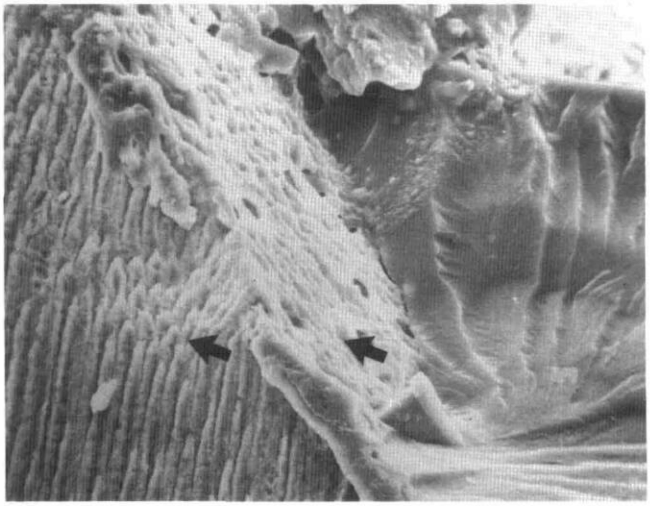

FIG 1. Failure of tensile bond at the resin in the interface and often involved small area of enamel (SEM 600×). E = Enamel, D = Delton fissure sealant.

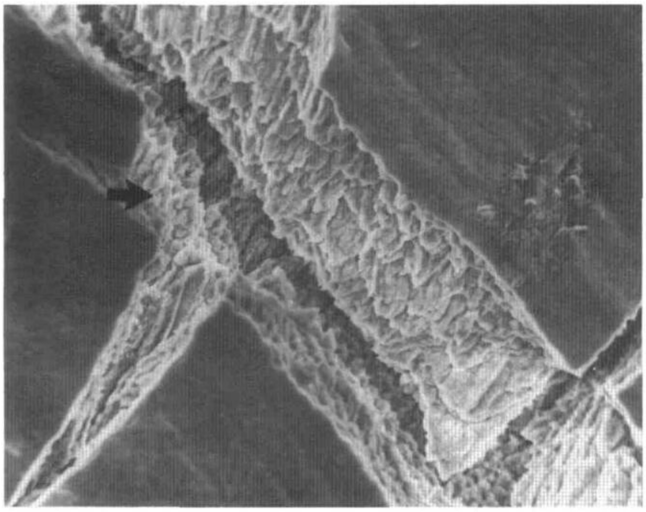

FIG 2. Failure of tensile bond specimens often involve large areas of enamel (SEM 1200×). E = Enamel.

Scanning Electron Microscopy

Examination of fractured test specimens for Delton by SEM revealed that the majority of the test specimens failed partly within the resin at the interface and in the enamel (Fig 1). A few specimens showed small fractures within the enamel involving a few prisms (Fig 1) or large fractures (Fig 2). The majority of the Helioseal specimens failed within the resin (Fig 3) while a few specimens fractured within the tags (Fig 4). A high magnification revealed that fracture of the sealant material often was associated with air bubbles (Fig 5). It was shown that trapped air in the fissures prevented further penetration of a sealant after equilibrium was reached. This resulted in incomplete penetration even for the Helioseal fissure sealant with low viscosity. It was found that sealant rarely penetrated to the base of the fissure regardless of the material and the teeth. Even when the sealants did penetrate to the bottom of the fissure, they did not appear to produce resin/enamel bonding. This indicated that the fissure base did not etch. However, the sealants remained in intimate contact with the tooth (Fig 6).

Discussion

Tensile Bond Strength

Determination of the tensile bond strengths revealed higher values for Delton in both permanent and primary teeth, compared to Helioseal. It has been reported by many researchers that primary teeth have a comparable rate of retention to permanent teeth (Ripa 1979). High tensile bond strength for Delton was reported by Retief and Mallory (1981). Previous studies revealed higher tensile bond strength values compared to the current study (Wright and Retief 1984; Thomson et al. 1981). Variations between this and previous studies demonstrated the dependence of bond strengths on the specific test system. Differences in the testing method, enamel structure, material handling, and storage time of bonded units prior to evaluation all may affect bond strength. In the present study a new methodology was used. The occlusal surfaces of the permanent and primary teeth were used to simulate conditions during clinical use of the material. A lower average of coefficient of variation was recorded, 11.519%. This finding indicated that the numbers of variable factors in such failure of tensile bond strengths are limited. This observation also revealed that the test method can be considered to be sufficiently sensitive to detect minor changes in the tensile bond strengths with reliability.

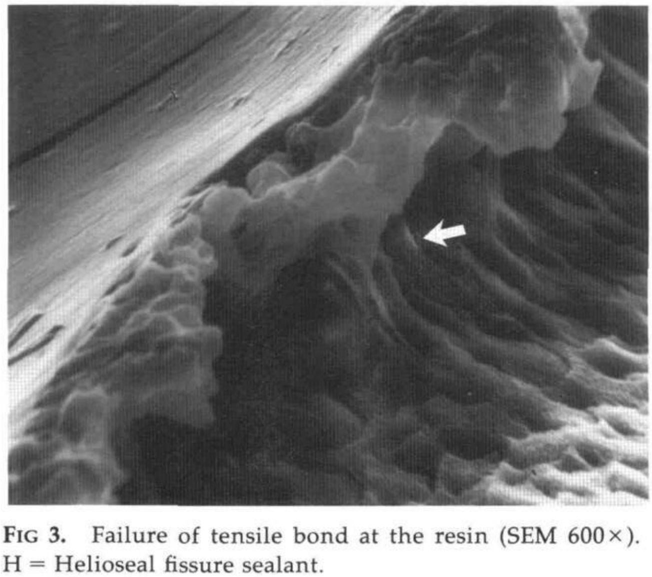

FIG 3. Failure of tensile bond at the resin (SEM 600×). H = Helioseal fissure sealant.

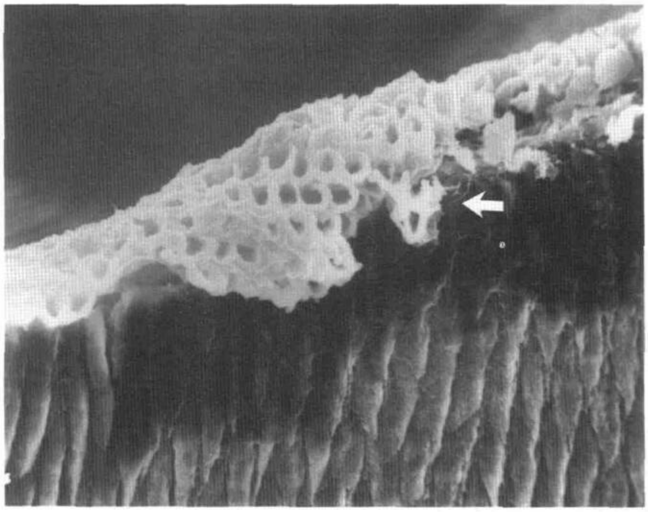

FIG 4. Failure of tensile bond at the resin within the tags (SEM 2400×). H = Helioseal fissure sealant, T = Tag.

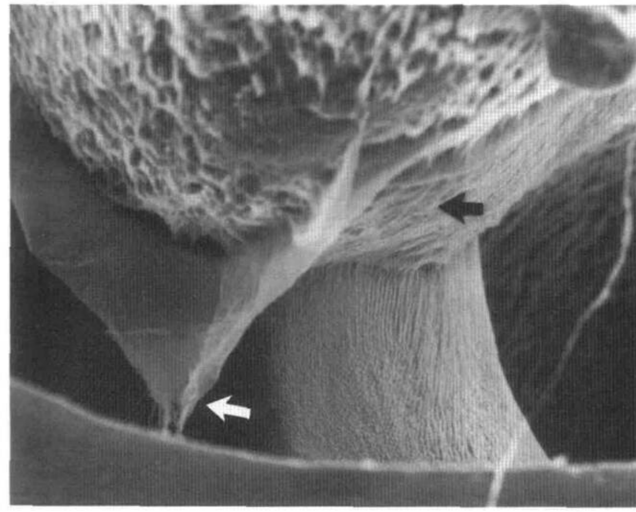

FIG 6. Rough surface in an adhesive aspect of interface and smooth surface at the base indicated that etching is confined to the cuspal slopes leaving the fissure base unetched (SEM 2400×). S = Sealant, E = Enamel.

Tensile strengths of sealants, in a study by Dennison and Powers (1979), were greater in magnitude than tensile bond strengths determined in this study. The results of this investigation indicate the tensile strengths of Delton to be higher than the tensile bond strength itself. This finding was supported by the SEM analysis where the fractured tensile bond specimens appeared to fail at the resin/enamel interface and in the enamel. Helioseal specimens failed within the resin. A few specimens failed within the tags. This result may be due to low tensile bond strength and low diametral tensile strength. There were statistical differences between tensile bond strengths of

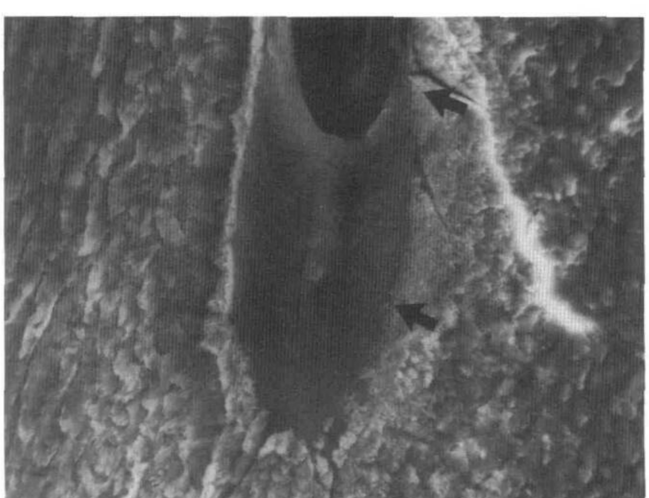

FIG 5. Failure of tensile bond specimens often associated with an air bubble, an intimate contact of the resin to the base of the fissure even with no etch (SEM 1300×). E = Enamel, S = Sealant.

the 2 materials in both permanent and primary teeth with Delton having the highest value despite the fact that Helioseal has a low viscosity. This result is in agreement with previous studies which showed that tensile bond strength is not affected by low viscosity resin (Retief and Woods 1981). Asmussen (1977) found that with different viscosities there are no significant differences in tag lengths or penetration times. Complete penetration was obtained for monomers with viscosities below a certain value, and the depth of penetration decreased only slightly with viscosity above this critical value. However, these results and the present results are in contrast with the report of Dogon (1975) which implied the need for low viscosity resins to enhance resin penetration.

There were significant differences between the 2 types of teeth with the permanent teeth having the highest values. The mean of tensile bond strength of Delton in primary teeth was 7.206 MN/m^2 while Helioseal was 4.746 which indicated significant differences between the 2 materials in primary teeth. This finding implied that Delton was retained on primary teeth almost as well as on permanent teeth. This result is in agreement with the findings of a clinical study reported by Ripa and Cole (1970). It should be possible, by starting at an early age and making several applications, to protect the occlusal surface of primary teeth until their natural exfoliation time. Low tensile bond strength on primary teeth compared to permanent teeth may be due to the presence of the prismless layer (Gwinnett 1966, 1973), or the presence of exogenous organic material on the outer surface of primary teeth which limited the porosity and the penetrability of the enamel (Silverstone et al. 1975).

Scanning Electron Microscopy

The mode of fracture of Delton fissure sealant observed in this study may be due to the high diametral strength of the material compared to the bond strength (Dennison and Powers 1979). Fracture of the enamel could be due to the use of nonvital teeth (Wright and Retief 1984). The mode of fracture of Helioseal specimens could be explained by the low diametral tensile strength and tensile bond strength of the material. For both materials fine filamentous tags remained in the enamel even after interfacial fracture; thus, pure enamel/resin separation rarely occurred.

This finding is in agreement with the report by Retief (1974) in determining areas of failure. The presence of air bubbles at the fractured line was observed. Stress concentrations could arise at these sites and be propagated along these voids during tensile failure. This observation has been reported previously by other authors.[2]

Despite the type of the materials and their physical properties, it was shown that trapped air in the fissures prevented further penetration of the sealant. However, the entrapped air may dissipate as a result of increased pressure by the resin inflow (Asmussen 1977). It appears in Fig 6 that rough surfaces occurred on the slopes of the fissure while smooth surfaces occurred on the base of the fissure. These findings coincide with those of Silverstone (1974) who reported that the bottom of the fissure is most probably coated with organic material resistant to the conditioning acid. However, retention of sealant in the fissure region is not impaired in any way since numerous tags were found on the sides of the fissure and along the cuspal slopes (Fig 6).

Conclusions

1. Delton has a high tensile bond strength. It is a better retained material than Helioseal under conditions of this study. Delton can also be used for primary teeth.
2. Air bubbles trapped within the resin must be prevented when applying either material.

[2] Retief and Mallory 1981; Wright and Retief 1984; Retief 1974.

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