



## The Shear Bond Strength of Acetone and Ethanol-based Bonding Agents to Bleached Teeth

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### Abstract

**Purpose:** The purpose of this in vitro study was to evaluate the: (1) shear bond strength (SBS) of acetone and ethanol-based bonding agents to composite resin 24 hours after being bleached with 35% hydrogen peroxide; and (2) interface morphology and mode of fracture (IMMF) between composite resin and enamel.

**Methods:** Forty extracted teeth were randomly assigned into 4 groups: (1) group 1: 35% H<sub>2</sub>O<sub>2</sub>+acetone; (2) group 2: H<sub>2</sub>O<sub>2</sub>+ethanol; (3) group 3: acetone; (4) group 4: ethanol. All teeth were acid etched, bonding agents were applied, and a composite resin stub was bonded and stored again in saline for 24 hours. SBS was determined, and scanning electron microscopy (SEM) was used for IMMF evaluation.

**Results:** SBS was significantly lower in bleached than unbleached teeth ( $P<.001$ ), but SBS did not differ between the acetone-based or ethanol-based adhesives ( $P=.55$ ) and bleaching did not interact with adhesive treatment ( $P=.39$ ). IMMF analysis of the unbleached teeth showed a continuous interface between the resin and enamel. By contrast, bleached teeth showed sparse contact between the resin and enamel and the resin was poorly infiltrated into the enamel surface.

**Conclusion:** Morphologic changes in human tooth enamel, 24 hours after bleaching, were associated with a reduction in the shear bond strength of adhesives. (Pediatr Dent 2006;28:531-536)

**KEYWORDS:** IN-OFFICE BLEACHING, BONDING AGENTS

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Bleaching is one of the most conservative techniques to improve the appearance of discolored teeth. In addition, it is an alternative to more invasive and costly procedures such as veneers and full coverage crowns. There are several methods for whitening teeth that involve either home or in-office procedures. Hydrogen and carbamide peroxides are the most widely used agents to achieve a white smile and studies have shown their safety, even when they are used in children.<sup>1</sup>

The mechanism of action of these peroxide agents is not entirely understood. It is speculated, however, that an oxidation process takes place that removes the stain by an oxygen-releasing and mechanical cleansing action.<sup>2,3</sup> The penetration of the bleach through enamel and dentin is

accomplished by the low molecular weight of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), and it can be quickened by application of heat. The H<sub>2</sub>O<sub>2</sub> moves through tooth structure and is able to denature proteins that increase tissue permeability and allows the movement of ions through the tooth.<sup>4</sup>

The degree of whitening is dependent on the concentration of peroxide and its application time.<sup>5</sup> The advantages of in-office bleaching procedures are that they produce results in one office visit and avoid the use of trays for long hours or overnight. With the in-office procedures, 35% H<sub>2</sub>O<sub>2</sub> is commonly used, as opposed to 10% carbamide peroxide used in trays. It has been found that 35% H<sub>2</sub>O<sub>2</sub> is stronger, more potent, and more acidic.<sup>5</sup> A commonly used in-office product, Opalescence Xtra (Ultradent, South Jordan, Utah) is a light-activated 35% H<sub>2</sub>O<sub>2</sub> gel. The manufacturer claims the carotene within the formula raises the gel's temperature when a visible light-curing unit is used and creates a more temperature controlled product.<sup>4</sup> Ideally, a bleaching product should be fast acting, selective, and harmless to tooth structure and soft tissue. Several studies, however, have shown that this type of treatment may alter the enamel and dentin morphology and cause reduction in bond strengths after bleaching.<sup>6-11</sup>

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Evaluation of enamel morphology using scanning electron microscopy (SEM) has shown alterations of enamel after bleaching and acid etching. Enamel was described as having increased porosity and increased formation of surface precipitate that may decrease the bond strength of composite to enamel after bleaching.<sup>4,7</sup>

Adhesive systems have been tailored and improved to allow bond strengths of about 20 MPa.<sup>12,13</sup> Currently, fourth through sixth generation adhesive systems are commonly used. The fourth and fifth generations require a pre-etching step. The newest generation (sixth generation) has been further simplified by eliminating the pre-etching step.<sup>14</sup>

The process of bonding between enamel and resin involves etching to demineralize the tooth and remove the smear layer. Then, a primer dissolved in acetone or ethanol is applied to enhance wetting and penetration of resin.<sup>13</sup> The resin monomer then penetrates the porosities created by etching and the polymerization of the adhesive monomer that permeates into the subsurface forms the major mechanism of dental adhesion.<sup>15</sup>

It has been speculated that there is a reduction in bond strength caused by morphologic changes and presence of residual oxygen created by the bleaching. These changes may interfere with resin infiltration or inhibition of resin polymerization.<sup>2,7,16-18</sup> Based on these findings, some studies suggest that waiting a few weeks before bonding may avoid the adverse effect that bleaching has on enamel.<sup>18-20</sup> It has also been suggested that application of an alcohol-based bonding agent may decrease the inhibitory effects of bleaching on bond strength.<sup>5,17</sup> The authors suggested that alcohol-based agents resulted in better bond strength than when using an acetone-based bonding agent. They speculated that it is because alcohol reacted with water and oxygen, hence reversing the adverse effects.

The purpose of this *in vitro* study was to evaluate the:

1. shear bond strength (SBS) of acetone and ethanol-based bonding agents to composite resin 24 hours after being bleached with 35% hydrogen peroxide; and
2. interface morphology and mode of fracture (IMMF) between composite resin and enamel.

## Methods

Forty recently (up to 3 months) extracted noncarious human molars and premolars were used. Immediately after extraction, teeth were stored in saline solution (Baxter 0.9%, NaCl irrigation solution, USP, pH 5.5) at room temperature. All of the soft tissue was removed and teeth were cleaned with a nonfluoridated slurry pumice and a rubber cup before being stored in 0.1% thymol at 4°C.

Thirty-two teeth were used for bond strength analysis, and 8 teeth were examined for the resin-enamel IMMF utilizing the SEM.

### Shear bond strength

Teeth were randomly assigned to be treated in 1 of 4 groups, with 8 teeth in each group:

1. Group 1: Bleached/acetone-based adhesive;
2. Group 2: Bleached/ethanol-based adhesive;
3. Group 3: Unbleached/acetone-based adhesive; and
4. Group 4: Unbleached/ethanol-based adhesive.

Teeth were sectioned at the cemento-enamel junction using the Isomet 1000 low-speed saw and a diamond blade (Buehler, Lake Bluff, Ill), then mounted horizontally in a specimen-holding ring with acrylic, exposing only the buccal surface. The buccal surfaces were flattened with a 600-grit SiC paper under running water, polished with nonfluoridated pumice, and rinsed with water.

Teeth in groups 1 and 2 were bleached with a visible, light-activated 35% (H<sub>2</sub>O<sub>2</sub>) gel (Opalescence Xtra, Ultra-dent, South Jordan, Utah), as directed by the manufacturer. Using a syringe, about a 1-mm thick layer of gel was applied to the buccal surface and light activated with a light emitting diode (LED) curing light (Dentsply, Milford, Del) for 10 seconds and left to stand for 15 minutes. The gel was then removed, rinsed with water, and air dried. The procedure was repeated as it would be done in a clinical setting.

The teeth were stored in 0.9% NaCl irrigation solution for 24 hours in a humidifier at 37°C. After storage, all teeth were acid-etched with 37% phosphoric acid (Super Etch, Southern Dental Industries, Victoria, Australia) for 20 seconds, rinsed with water for 20 seconds, and air dried. Bonding with the adhesive agents, Bond 1 and Bond 1 c&b (Pentron Clinical Technologies, Wallingford, CT) was administered for each group by applying 2 consecutive coats. Next, they were dried with a gentle stream of air using an air syringe to evaporate the excess solvent and then light cured.

The specimen ring was placed in a Bencor Multi-T instrument system (Danville Engineering, Inc., Danville, CA). A nozzle was screwed on the coupler to be able to glide up and down with an active rod. The nozzle was positioned over the flattened buccal surface of the tooth in the specimen ring. Then a commercial product composite (TPH—Dentsply International, Ontario, Canada) was placed in the standard 3.5-mm nozzle and any excess overflowing was removed. The composite was light cured at a 45° angle through the nozzle windows and then the active rod was lifted vertically to leave the bonded stub of composite on the tooth's buccal surface.

The specimens were again stored in 0.9% NaCl irrigation solution for 24 hours in a humidifier at 37°C prior to the SBS testing. The Bencor Multi-T system was used to measure the SBS of the various adhesive systems. A notched flat guillotine blade was attached to the coupler in a horizontal position. The guillotine was able to glide up and down and engage to the composite stub on the bonded surface passively between the enamel-resin interface. The SBS was computed as force per unit area.

$$\text{SBS (MPa)} = \frac{\text{Force (N)}}{\pi (3.5\text{mm}/2)^2}$$

All samples were evaluated under a high-magnification microscope with a grid using Adobe Photoshop Elements

2.0 (Adobe Systems Incorporated, San Jose, CA) to determine how much resin was left on the tooth after shearing (0%-100%). All samples were then categorized and grouped as: (1) cohesive failure; (2) adhesive failure; or (3) dentin failure.

The teeth were evaluated by SEM to see the interface between enamel and composite resin. The teeth were prepared as described by Titley (1991). To evaluate the enamel-resin attachment in cross-section, the occlusal one third of the crown was cut using the Isomet 1000 low-speed saw to reach the center of the resin cylinder and then finished flat with a 600-grit silicon carbide paper. The specimens were: (1) washed with distilled water; (2) dried; (3) etched for 5 seconds with 0.1 N HCl; (4) rinsed with distilled water; and (5) dried. The teeth were then mounted on SEM studs and surface-coated with gold.

All SBS data and IMMF percentages were transferred to SPSS for Windows (SPSS Inc, Chicago, Ill). The effects of the 4 treatment conditions were summarized as means and standard deviations and compared in a 2-way analysis of variance (ANOVA) for independent groups, with 2 levels of material (acetone and ethanol) and 2 levels of preparation (bleach and not). Significance of each main effect and their interaction was set at 0.05.

## Results

The 2-way ANOVA used to test the null hypothesis for this 2x2 study design showed that there was no difference between acetone-based or alcohol-based bonding agents in the bleached and unbleached teeth ( $P > .05$ ).

Figure 1 shows the SBS for the 4 groups: (1) group 1 =  $7.9 \pm 2.7$ ; (2) group 2 =  $8.9 \pm 2.0$ ; (3) group 3 =  $18.8 \pm 1.4$ ; and (4) group 4 =  $18.6 \pm 1.2$ . No statistically significant difference was found between the acetone-based and ethanol-based adhesives ( $F[1,29] = 0.37$ ;  $P = .55$ ), nor was there an interaction between the 2 adhesives and bleaching. On the other hand, a statistically significant reduction in SBS was found when teeth were bleached ( $F[1,29] = 248.5$ ;  $P < .001$ ).

Further analyses addressed the question of any difference between groups in cohesive, adhesive, or other type of failure after shearing. Most failures involved were a combination of cohesive and adhesive failures. In Group 4, however, dentinal failure was noted on 4 samples, suggesting that the bond between enamel and adhesive was stronger than the cohesive strength of dentin. Two-way ANOVA showed no difference in the percentage of cohesive failure by group. The following percent of cohesive failure was found: (1) group 1 =  $48.2 \pm 42.3$ ; (2) group 2 =  $20.3 \pm 30.1$ ; (3) group 3 =  $38.5 \pm 24.1$ ; and (4) group 4 =  $36.9 \pm 30.6$  (Figure 2). The failure mode was mostly adhesive in all groups, and there was no statistical difference among the 4 groups.

The SEM analysis of the interface between enamel and resin penetration and structures are illustrated in Figure 3. At high magnification, the resin penetration into the enamel can be seen in all groups. For both bleached groups, the resin penetration into the enamel was: (1) sparse; (2)

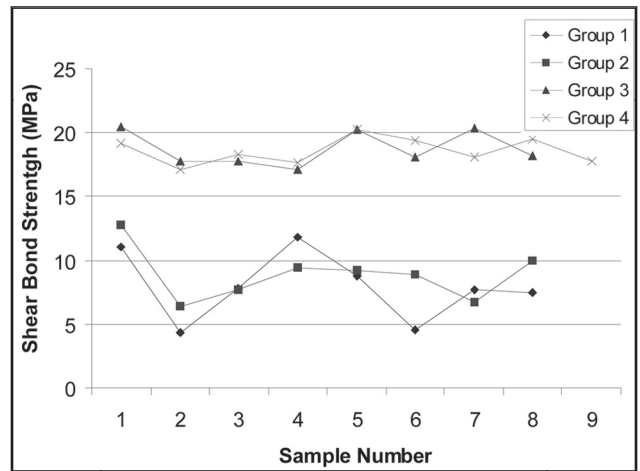


Figure 1. Shear bond strength for groups of teeth bonded with acetone- and ethanol-based adhesive with and without prior treatment with bleach.

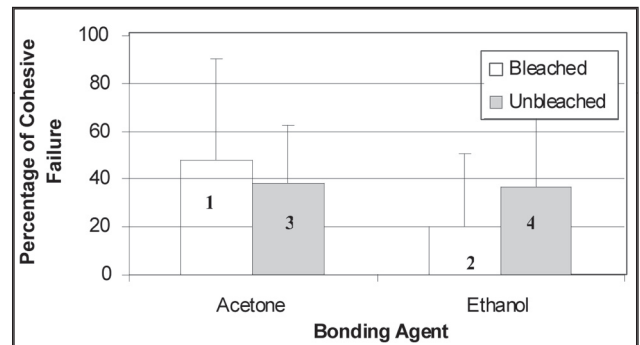


Figure 2. Mean proportion of cohesive failures for groups of teeth bonded with acetone- and ethanol-based adhesives with and without treatment with bleach.

poorly defined; and (3) adapted only poorly into the enamel surface. On the other hand, for both unbleached groups, the resin: (2) was contiguous; and (2) penetrated well into the enamel surface. In addition, the enamel surface in the bleached groups appeared porous, with a coating on the surface. By contrast, unbleached samples were: (1) less porous; and (2) without coating.

## Discussion

Many studies have shown reduced bond strength of composite resin to enamel after bleaching. This reduction in bond strength has been attributed to morphologic changes in the enamel and to the interfering effect of residual oxygen on resin infiltration and polymerization.<sup>2,7,16-18</sup> Consistent with a temporary effect, bond strength recovers a few weeks after bleaching.<sup>18-20</sup> By the hydroxyl theory, it has been suggested that the application of an ethanol-based bonding agent may reverse the inhibitory effect of bleaching on bond strength by scavenging residual oxygen.<sup>5,17</sup> In this study, an ethanol-based bonding agent performed no better than an acetone-based agent 24 hours after bleaching with 35% H<sub>2</sub>O<sub>2</sub> when the enamel was thought to be at its weakest point. Furthermore, SEM analysis showed

similar problems in the interface between the enamel and either resin after bleaching. Results fail to support the hydroxyl theory of shear bond strength loss following this bleaching procedure.

These findings differ from those of Sung et al<sup>5</sup> and Kalili et al,<sup>17</sup> who reported that utilizing an ethanol-based adhesive agent improved the bond strength of bleached teeth. Unlike the present study, they used a weaker bleach (10% carbamide peroxide) and waited a longer time (5 days) before bonding the composite resin. Thus, it is possible that waiting longer and/or generating fewer radicals may have provided better results.

Composite resin bonds to tooth enamel mechanically, as resin penetrates and polymerizes into irregularities in enamel surface. While resin penetration is normally enhanced by etching, Josey et al<sup>3</sup> and Titley et al<sup>7</sup> reported that etching after bleaching may lead to over-etched enamel with a loss of prism boundaries that may adversely affect the retentive qualities of composite resin to enamel. In their preparation, resin tags appeared: (1) short; (2) sparse; (3) structurally incomplete; and (4) free of resin in some areas of enamel. Current data are consistent with this structural alteration, and additional research may provide a more likely explanation of lowered SBS.

Furthermore, the SEM analysis revealed differences in the interface morphology between composite resin and enamel on bleached and unbleached teeth, regardless of the adhesive agent type used. Therefore, the null hypothesis that there is no difference in the interface morphology between composite resin and enamel using acetone- or ethanol-based adhesives on bleached or unbleached teeth cannot be rejected. The difference was noted as a result of the bleaching agent, not the type of adhesive agent used. In the bleached groups, the resin and enamel interface was sparse with poor contact and the resin was poorly infiltrated into the enamel surface. In the unbleached groups, however, the resin and enamel interface was continuous and closely contacting and the resin was well infiltrated into the enamel surface. Titley<sup>7</sup> had similar findings in his study using 35% H<sub>2</sub>O<sub>2</sub> and bonding composite resin to bovine enamel.

What is more, the enamel surface in the SEM analysis appears different in the bleached groups than the unbleached groups, regardless of the adhesive agent used. The enamel in the bleached teeth appears to have a coating of precipitation on the surface and appears more porous. In the unbleached teeth, however, a lesser degree of porosity was noted and the coating was not present. Similar findings were seen in Titley's<sup>6</sup> and Yurdukoru's<sup>4</sup> studies. It was noted in Titley's

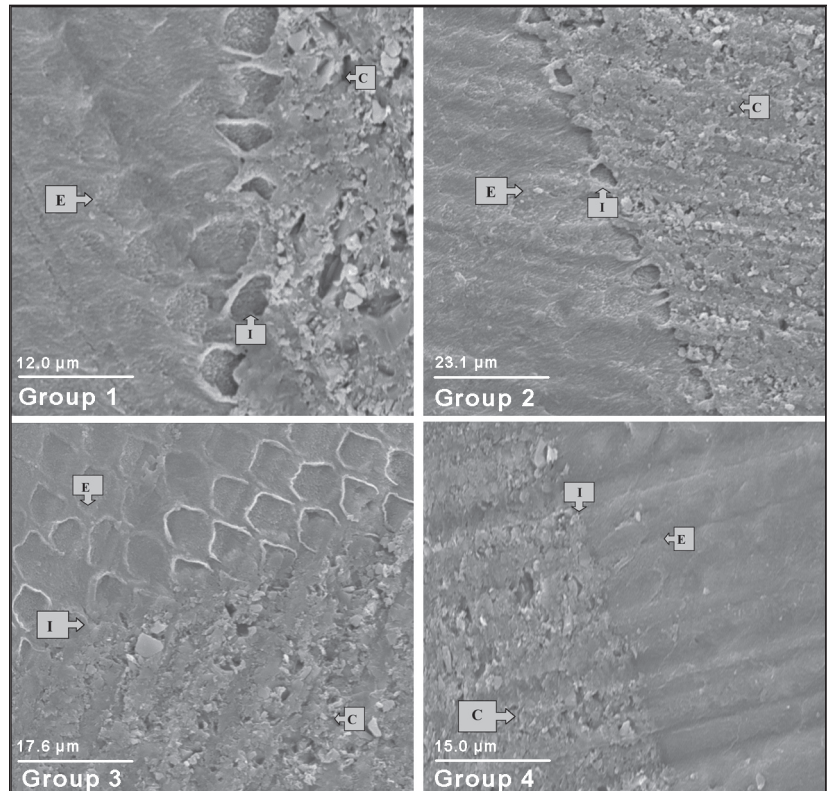


Figure 3. Scanning electron micrographs showing the resin/enamel interface for the four treatment groups. E=enamel, C=composite resin, and I=resin/enamel interface. In groups 1 and 2, the bleached groups, the resin was sparse and not well penetrated into the enamel surface. In groups 3 and 4, the unbleached groups, the resin was contiguous, in close contact, and penetrated into the enamel surface.

study<sup>6</sup> that when a bleached enamel surface was etched with 37% phosphoric acid, the enamel surface under SEM appeared to have increased porosity and formation of surface precipitation compared to the unbleached control. This acid was utilized in the present study to bond composite resin, and similar findings on enamel were observed. The coating appears to prevent proper adhesion and wetting of resin and affects the retentive qualities of the composite resin to enamel. This may be the reason the shear bond strength of the bleached teeth were significantly reduced when compared to the unbleached teeth.

Fracture sites after SBS analysis were evaluated, and most fractures were a combination of cohesive and adhesive failures. Once the failure starts, which is difficult to determine at the initial site, it will propagate in any which way. Interestingly, it was noted in the unbleached+ethanol group that fractures occurred in dentin. The enamel/resin interface remained intact and apparently was stronger than the dentin's cohesive strength. This suggests that ethanol-based adhesives are stronger than acetone-based adhesives.

There were some limitations in this study. First, the history of the extracted teeth used in this study is unknown. For example, it is not known whether they had been bleached in the past or if whitening dentifrices were routinely used. Each condition could have an impact on

the bond strength of composite resin. On the other hand, teeth were assigned to condition at random, virtually ensuring that history effects were evenly distributed across treatment condition.

Second, results of this in vitro study cannot readily be generalized to a clinical situation. Also, only one type of bonding agent of each type was used, limiting generalization only to current materials. What is more, the teeth were stored in saline solution, absent conditions that characterize the oral cavity. From a clinical perspective, the effects of bleaching may be less severe in vivo if remineralization, tooth-brushing, and salivary contact act to renew the original structure after bleaching.

It can be concluded from this study that there is a reduction in SBS and morphologic changes in enamel after 24 hours of utilizing the in-office visible-light activated 35% H<sub>2</sub>O<sub>2</sub> Opalescence Xtra. Furthermore, after 24 hours, the use of an alcohol-based adhesive agent did not appear to improve or reverse the adverse effects of H<sub>2</sub>O<sub>2</sub> on enamel to enhance composite resin bonding, casting doubt on the oxygen radical theory of bond strength loss. Enamel surface changes may have caused the weakening of bond strength. Future areas to investigate include studying the effects of 35% H<sub>2</sub>O<sub>2</sub> over 2 to 3 weeks after bleaching and, to see if bond strength returns, and at what pace.

Another area to investigate further is the effects of over-the-counter whitening toothpastes or whitening strips and their effect on SBS of composite resin. Finally, it is necessary to investigate the effects of bleaching on sixth and seventh generations of bonding agents. Based on this study findings and previous studies, the authors would recommend avoiding elective composite resin restorations (either ethanol- or acetone-based) for at least 24 hours after bleaching. The bonding agent types used becomes less critical, since enamel's adverse effects after bleaching are reversed with increased time.

## Conclusions

Based on this study's results, the following conclusions can be made:

1. Bleaching with 35% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) reduces the bond strength of both acetone- and ethanol-based composite resin adhesives.
2. Bleaching with 35% H<sub>2</sub>O<sub>2</sub> changes the surface morphology of enamel, and these changes were similar when using the acetone- and ethanol-based adhesives. Morphological changes may underlie loss of shear bond strength (SBS).
3. It appears that the bond between enamel and adhesive was stronger than the cohesive strength of dentin when using an ethanol-based adhesive on unbleached teeth.
4. This study supports the hypotheses that there is reduced SBS and morphologic changes in enamel as a result of bleaching with 35% H<sub>2</sub>O<sub>2</sub> after 24 hours, regardless of the type of adhesive used.

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## Abstract of the Scientific Literature



### Non-nutritive Sucking Patterns Between Ages 1 to 8

Sucking behaviors are normal in infants and young children. Prolonged duration of these behaviors, however, may have consequences for developing orofacial structures and occlusion. The purpose of this prospective study was to determine the: (1) duration of non-nutritive sucking (NNS) behaviors of children between ages 1 to 8; and (2) effect of prolonged habits on selected occlusal characteristics in the late primary dentition (age 5).

Data were collected from 797 children who were followed from birth. Data included: (1) completed periodic questionnaires by parents; (2) study models obtained for 372 children between the ages of 4 to 5 years; and (3) assessments for posterior crossbite, anterior open bite, and overjet. Subjects were classified according to the duration and type of habit. Children with NNS behaviors of less than 12 months were subclassified according to the duration of breast-feeding. Changes in the incidence and effect of the habits were compared over time. Significant decreases in the incidence of pacifier habits (40% to 1%) and digit habits (31% to 12%) between ages 1 and 5 and ages 1 and 4, respectively, were observed. Between ages 4 and 7, the decrease in the incidence continued at a slower rate. Prolonged NNS habits caused significant changes in the occlusal characteristics in the late primary dentition. The effects of pacifier habits were different from those of digit sucking. Researchers concluded that: (1) There were no significant differences in the prevalence of digit and pacifier habits between boys and girls between 1 and 8 years of age. (2) Children who had pacifier or digit habits lasting less than 12 months did not have significantly different occlusal characteristics than children who were breast-fed for 6 to 12 months. (3) Prolonged habits (older than age 4) have detrimental effects on the occlusion in the late primary dentition. (4) When changes in the prevalence of pacifier and digit habits were compared, more children with digit habits had difficulty in stopping the habits after 4 years of age.

**Comments:** It may be useful to attempt to substitute the digit habit with a pacifier habit as soon as possible. The transverse occlusal relationship should be evaluated in the primary dentition in children with prolonged habits, particularly in pacifier-sucking children, to intercept developing crossbites and functional shifts. If there are interfering contacts of the primary canines, the parents should be instructed to reduce pacifier-sucking time and appropriate treatment may be needed. **RKY**

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