



An Evaluation of the Properties of Fluoride-Based and Amorphous Calcium Phosphate-Based Orthodontic Adhesives

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(Received: 14 July 2021

Revised: 1 September 2021

Accepted: 18 December 2021)

KEYWORDS

Fluoride-Based,
Amorphous

ABSTRACT:

During the course of this study, fluoride or amorphous calcium phosphate was used as orthodontic adhesives in order to reduce the adhesion of bacteria and the demineralization of enamel in vitro. ACP or fluoride adhesives were used for bracketing three buccolingual sections of forty human premolar teeth. A polarized light microscope and X-ray photoelectron spectrophotometry were used to study artificial lesions caused by pH cycling. Analyzing calcium and phosphorus content by XPS, Aegis Ortho was found to have the lowest content after 28 days. Quick Cure reduced lesion depth areas by 23.6% after 42 days, while Aegis Ortho reduced them by 20.3% ($P < 0.05$). Transbond XT was more resistant to *Streptococcus mutans* adhesion than Aegis Ortho or Quick Cure in the presence of 1% sucrose ($P = 0.05$). As compared with composites without fluoride or ACP, composites with fluoride or ACP reduce bacterial adhesion and lesion formation.

Introduction

During fixed orthodontic treatment, decalcification around orthodontic brackets is a common occurrence [1]. Approximately 2-96% of patients suffer from decalcification. Despite fluoride treatment [2], white spot lesions can develop within 4 weeks without fluoride application due to the prolonged accumulation of bacterial plaque [3]. Plaque-free environments enable these subsurface lesions to remineralize provided the surface layer remains intact [4]. A bacterial colony isolated from used brackets has been found to contain *Streptococcus mutans*, a known caries initiator [5]. The initial colonization of *S. orthodontists* occurred three days after the use of orthodontic bonding materials [6]. In order to prevent enamel decalcification, to prevent microbial growth, a microbial-resistant bonding material

was needed [7]. The reduction of white spots during orthodontic treatment can be achieved by including preventative agents in the dental bonding composites [8], but some of these efforts have adversely affected the composite's physical properties [9]. In spite of this, obtaining FDA approval can be difficult because these agents must be classified as drugs. As for Recaldent, a paste containing casein phosphopeptide-amorphous calcium phosphate, the FDA has approved its use.

The saliva only contains enough calcium and phosphate to maintain equilibrium or repair teeth slowly under normal conditions. During the formation of hydroxyapatite (HAP), ACP serves as a biocompatible intermediate [10]. Calcium and phosphate ions are released in a sustained manner when pH drops below [11]. Compared to conventional dental composites [12],



ACP composites have weaker mechanical properties. ACP-filled composites that are used to coat artificially produced lesions with caries-like characteristics recover 71 percent and 33 percent of their lost mineral content when microradiography is performed. There are various delivery systems that are being proposed for the application of ACP in dentistry, including topical and bleaching gels, toothpastes, mouthwashes, sugar-free gum, and mouthwashes. An orthodontic composite incorporating ACP is known as Aegis Ortho. To improve orthodontic treatment effectiveness, iatrogenic caries during orthodontic treatment should be reduced. In this study, we aim to accomplish the following objectives.

1. An in vitro study was conducted to examine the adhesion of *Streptococcus mutans* to three test composites, including one containing amorphous calcium phosphate (ACP).
2. Three composite test materials were evaluated in vitro using photoelectron spectroscopy (PLM) and XPS surface analysis (XPS).

Methodology

A total of forty large, noncarious, extracted human premolars were obtained and kept at 4°C in 0.1% thymol until needed. In addition to removing soft tissues and calculus from the teeth, dental scalers are used to remove the remaining hard tissues. The root portions of a crown were removed by separating a disk from the crown. Deionized water was used to rinse and air dry the tooth crowns after fluoride-free prophylaxis. XPS experiments were conducted on ten teeth and PLM experiments on 30 teeth, including those used in pilot PLM experiments. A diamond wafering blade was used to section each crown buccolingually into three parts. The teeth were fixed in epoxy in standard cylinders before being divided into three segments. Diamond blades were used to section the specimen with extreme caution. A cylinder of red wax was used to arrange the specimens for PLM slices in order to facilitate trim and polishing the slices more precisely. A single tooth was composed of two lateral segments, each 3 mm wide, which allowed orthodontic brackets to be placed. Nonbracketed, nonadhesive, and pH-cycled control specimens of enamel with a width of 1-2 mm were reserved for the 1-2 mm wide central segments. Random number tables were used to assign the samples to the control and treatment groups. This project is exempt research that does not involve human subjects,

according to the university's Office for the Protection of Human Subjects.

In the test, three adhesives were used:

1. The Transbond XT composite does not contain fluoride or ACP. On the occlusal and apical surfaces of each bracket, 1mm of acid-resistant varnish (nail polish) was applied, with windows exposed on both sides.
2. This composite contains fluoride and is made by Reliance Orthodontics Products in Itasca, Illinois.
3. It has 38% ACP fillers and is a light cure composite product made by Bosworth Co. in Skokie, IL.

The Brackets Synergy bicuspid brackets were placed using a template on lateral teeth. Each bracket was coated with acid-resistant varnish (nail polish) within 1 mm of the occlusal and apical surfaces, with windows exposed above and below. As part of the XPS study, this step was not performed. 37% phosphoric acid gel was used to etch the bracket area. Based on the study group, premolar brackets were bonded with the orthodontic adhesive specified by the manufacturer. Rocky Mountain Orthodontics in Denver, CO, cured all flash using a Pac-Dent LED light at 450 nm.

An artificial saliva solution was applied to the tooth sections for 12 hours before a demineralizing solution was applied. A solution containing 20 mmol/L sodium bicarbonate, 3 mmol/L bicarbonate, and 1 mmol/L calcium chloride was prepared from artificial saliva, whereas a solution containing 2.2 mmol/L calcium, 2.2 mmol/L potassium, and 50 mmol/L acetic acid was prepared with demineralization solution. The pH/mV meter was used to measure the solutions Two times daily, alternating between artificial saliva and demineralizing solutions, one hour of demineralizing solution was applied to the tooth sections at room temperature. Through constant circulation of solutions, subsurface lesions similar to those caused by caries were created on a stir plate. The XPS group cycled the tooth segments for 28 days. A 28-day pH cycle was conducted on pilot samples prior to PLM evaluation. Since the samples were sectioned, one set of lesions was too small to quantify [14]. The samples were then cycled for 14 more days in order to collect 42 days of cycling in total. We removed brackets from all segments, and then used a Silverstone Taylor Series 1000 Deluxe hard tissue microtome to



make buccolingual sections of 140 to 160 μ m in thickness from each segment.

X-Ray Photoelectron Spectrophotometry (XPS)

In order to analyze the specimens for XPS, Kratos Analytical Ltd., Manchester, England, used an Axis165 spectrometer. In order to perform this experiment, monochromatic Al K-alpha X-rays (1486.6 eV) were used as follows: The dimensions of the slot aperture are 700 x 300 μ m, the filament neutralization current is 1.7 A, the charge balance is 2.6 V, the filament bias is 1.3, and the C 1s peak is calibrated at 285.0 eV. In this study, binding energies between 0 and 1,000 eV were scanned. Using Kratos Vision computer programs, the elemental compositions of the surface were calculated. An electron orbit analysis of calcium 2p, phosphorus 2s, oxygen 1s, fluorine 1s, silicon 2p, and carbon 1s was carried out. On day 1 and day 28 of the study, we analyzed four central segments. At days 1 and 28 of the testing protocol, each of the four groups examined four lateral tooth segments. Day 1 of this study, exposed enamel from the top of the tooth section and from the bottom was analyzed. Then removed the brackets from the composite groups samples on day 28, returned them to the XPS instrument, and achieved another reading of top, middle, and bottom sites. The interobserver variability was determined by repeating the samples. In total, 119 sites were tested in both the experimental and pilot trials of XPS [15].

Polarized Light Microscopy

To quantify demineralized lesions, a dual-stage polarized light microscope was used (Olympus Model BH-2, Dualmont Corporation, Minneapolis, MN). Each of the four test groups contained eight tooth segments (three composite groups and no composite control group). In each section of a tooth segment, 140 to 160 μ m of deionized water solution were used to wet it. Photographs were taken under maximum illumination at 13.2 times magnification of areas of demineralization centered within the field of view. Using the Image Pro Plus software, a digital template of 0.5 mm width was used to measure the depth area of each section of the lesion. We averaged buccolingual lesion depths for each sample to determine the overall mean depth.

Bacterial Adhesion Testing

Custom moulds were used to form composite disks (9 mm by 3 mm) using each adhesive, Light curing was

applied to both sides for 20 seconds each. The oxidative layers were removed with diamond 320 grit paper, and debris was removed with sonication. To remove bacteria from each composite, 60 samples were made and sanitized under UV light overnight. A combination of brain-heart infusion broth with and without 1% sucrose was used as an inoculant for *Streptococcus mutans* to be inoculated with under 48 hours at 37°C on stainless steel orthodontic ligature wires suspended on brain-heart infusion broth. In order to remove bacteria that had not adhered to the composite disks, five dips in PBS were conducted following the incubation period. The attached *S. mutans* were then dislodged by sonication in 3 mL of 1 N NaOH for 6 minutes. An assessment of the absorbance of *S. mutans* suspensions was performed using a 550 nanometer wavelength. By using BHI broth alone, we controlled a composite disk.

Data Analysis

Statistical analysis was performed using SPSS software. The XPS methodology paragraph above included Kruskal-Wallis and Mann-Whitney tests for analyzing all ions. Moreover, we used PLM for determining P 0.05 differences in lesion size. The t-test and one-way ANOVA were used to compare the results of the bacterial adhesion test with and without sucrose (P 0.05). Small sample sizes necessitated nonparametric evaluations for the subgroups.

Results

XPS Determinations

The ACP orthodontic adhesive was not homogeneous in three composite samples analyzed using XPS. There was no change in carbon values in the control group without pH cycling. Results from various analyses did not differ significantly depending on where the tooth surface was located. While ACP orthodontic adhesive was significantly degraded after pH cycling, its performance after pH cycling was not affected. As well as the chemical composition of the samples, the carbon values also changed significantly. There is a significant difference in pH sensitivity among the ACP orthodontic adhesives studied. The XPS elemental mass concentrations of Ca, P, O, F, Si, and C in the samples before and after orthodontic adhesive application (T1) were not significantly different by 0.05 ($P < 0.05$). Using the Kruskal Wallis Test, Transbond XT, Quick Cure, and



Aegis Ortho significantly differed in their mass elemental concentrations for Ca, P, and C. However, the mass elemental concentrations for Transbond XT and Quick Cure did not differ significantly. There was a significant difference between the results obtained with Transbond XT and those obtained with Aegis Ortho with respect to Ca ($P < 0.007$), P ($P < 0.001$), Si ($P < 0.038$), and C ($P < 0.004$). Calcium levels decreased more rapidly in Aegis Ortho between T1, 26.98%, and T2, 16.46%, compared to Transbond XT. Aegis Ortho, which has a lower phosphorus content than Transbond XT, has a higher carbon and silica content. An alpha coefficient of reliability of 0.994 was found after analyzing 12 sets of lesion images after one week interval, indicating a high level of reliability. Detailed information about lesion depth and lesion area is presented. There was a significant difference between the groups according to Kruskal-Wallis testing ($P = 0.02$). In comparison to the control group, Quick Cure showed a reduction of 23.6% in lesion depth area. In comparison with the control group, Aegis Ortho had a reduction of 20.3% in lesion depth area, whereas Transbond XT had a 3.2% increase. Polished light micrographs representative of the techniques.

Discussion

To determine whether orthodontic adhesives with ACP and fluoride-releasing orthodontic bonding systems altered the orthodontic gum surface, several systems released fluoride in vitro. Additionally, bacterial adhesion was determined between the bonding adhesives and composites created on the occlusal and gingival surfaces. During T1 and T2 (day 28), the levels of silica and carbon increased because the orthodontic adhesive contained high concentrations. When teeth samples were cycled in pH of 4.4, calcium and phosphorus levels decreased due to ions dissolving from the teeth samples. In comparison with Aegis Ortho, Transbond XT decreased calcium and phosphorus levels by a smaller amount. When analyzed by XPS, in response to the acid challenge, Aegis Ortho released calcium and phosphorus ions, which no longer remained on the surface of the degraded resin. In vitro induced enamel lesions of the Control group and Transbond XT group were shallower than those of the Quick Cure and Aegis Ortho with ACP groups. Due to the fact that quick cure releases up to 0.90

grams of F per square inch per day, we shouldn't be surprised by this result. The rate of 0.65-1.30 grams per cubic centimeter per day has been proposed by Rawls as sufficient to prevent the onset of caries in the vicinity of resin-based dental materials in sound enamel. There is evidence from various studies that fluoride inhibits glycolysis in *S. mutans*, as well as improving the resistance of enamel to acid dissolution. It is possible that calcium phosphate in amorphous form could shift the equilibrium of enamel strengthening in favor of calcium phosphate. This study found that when Aegis Ortho with ACP was combined with Quick Cure, there was greater demineralization. The systematic review considered an in vivo study with a caries-inhibiting effect greater than 50% to be significant. Mitchell, Turner, Trimpeneers, and Dermout tested a fluoride-releasing composite that resulted in an overall preventive fraction of 20%. According to the study, fluoride-releasing bonding materials are not significant contributors to prevention of demineralization.

In the presence of sucrose, *S. mutans* produces sticky glucan polymers, which adhere to composite disks. It was not examined whether sucrose-induced adherence was observed in *S. mutans*. Consequently, bacteria and sucrose solution adhered less strongly to each other. As compared to Transbond XT, fluoride-releasing composites demonstrated 37.7% reduction in bacterial adhesion, while ACP composites demonstrated 41.8% reduction in *S. mutans* adherence. did not detect *S. mutans* in the biofilms grown on their fluoride-releasing materials. In their study, the researchers found that these materials inhibited bacterial plaque metabolism. Bacterial adhesion to composites containing ACP has been studied relatively little in the past. There is a possibility that the reduction in *S. mutans* adhesion is caused by inhibition of the enzyme glucosyltransferase, which synthesizes adherent glucans. This area needs further research. This study did not determine how much calcium is released by the Aegis composite disks. Further research is required.

Conventional orthodontic composites differed significantly in mechanical properties from ACP-containing orthodontic resins. There is a need for more research on developing a preventative and mechanically efficient product.

**Table 1:** pH cycling after 42 days with PLM lesion measurements.

Treatment	Affected area	Depth as a percentage	Deepest point	Deepest point
Control	8.94	156	129	185
Intransbond XT	9.19	162	128	194
Cure in a flash	7.06	118	86	146
AEGI Orthodontics	7.33	124	80	166

Conclusions

When ACP was used in orthodontic adhesives, bacteria adhesion and lesion depth formation were reduced. However, the effects were better than those from orthodontic adhesive with fluoride, but not as good as those from orthodontic adhesive without fluoride. ACP release rates and composites made with ACP are worthy of further study, according to this study, as well as its safety/efficacy and the potential for clinical application in the future. Decalcification of enamel has not been completely prevented in any study so far in the literature. There is still no substitute for good oral hygiene and a healthy diet. Developing a long-term prevention agent for orthodontic patients with poor oral hygiene compliance is important.

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