Original Article

Examination of Six Orthodontic Adhesives with Electron Microscopy, Hardness Tester and Energy Dispersive X-ray Microanalyzer

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ABSTRACT

Objective: To examine the ultrastructure of six light-cure orthodontic adhesives with scanning electron microscope (SEM) and transmission electron microscope (TEM), microhardness tester, and energy dispersive X-ray microanalyzer (EDX).

Materials and Methods: The orthodontic adhesives evaluated were Transbond XT, Light Bond, BeautyOrtho Bond, Kurasper F, Heliosit Orthodontic, and Salivatect. Specimens of each adhesive were carefully prepared for observation under SEM and TEM. Furthermore, the Vickers hardness was tested, and the adhesives were evaluated with EDX.

Results: SEM and TEM images illustrated great diversity of the adhesives ultrastructure. The Vickers hardness test showed significant differences among all the adhesives (except Transbond XT and Salivatect). Although some similar elements were detected with EDX, the concentration was different in each adhesive.

Conclusion: Orthodontic brackets can be bonded to the enamel surface with the adhesives available on the market. However, orthodontists might achieve better results identifying their properties and compositions.

KEY WORDS: Adhesives; SEM; TEM; EDX

INTRODUCTION

The direct bonding of orthodontic brackets with composite resin, as described in 1965 by Newman,[1] has been considered the most popular method and the clinical standard for attaching orthodontic brackets to teeth.[2] This technique resulted in advances to the clinical treatment, including greater comfort for the patient, easier oral hygiene, enhanced esthetics, and reduced chair time.[3] Therefore, the bonding of orthodontic brackets has became an essential procedure to accomplish the clinical treatment, and researchers have worked hard to achieve the best qualities of bonding agents to maintain a sound unblemished enamel surface after removing orthodontic brackets.[4–7]

Recently, the bond strength of orthodontic brackets has been studied by numerous authors around the world.[8–15] In agreement with Eliades,[16] the education curricula for orthodontic residents requires them to build a solid background in materials science, which allows them to make informed decisions on new materials and techniques, as opposed to selecting them based solely on advertisement brochures. In response to that suggestion, new studies have been carried out to evaluate the influence of filler level on the bond strength of orthodontic adhesives.[17,18] The fillers are added to the polymeric part of the adhesives to increase strengthen, increase stiffness, reduce dimensional changes, and improve handling.[18] On the other hand, the use of scanning electron microscope (SEM)[19] and sophisticated equipment, such as the focused ion beam system, have been successfully used to study the effects of enamel conditioners.[20]

However, to our knowledge, there are no studies of orthodontics adhesives using electron microscopy, hardness test, and energy dispersive X-ray microanalysis (EDX). Hence, in an effort to provide some complementary information for the study of orthodontic adhesives, the objective of this study was to examine

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Accepted: September 2007. Submitted: July 2007.

the ultrastructure of 6 light-cure orthodontic adhesives with SEM, transmission electron microscope (TEM), microhardness tester, and EDX.

MATERIALS AND METHODS

Adhesives

Five orthodontic adhesive pastes (Transbond XT, 3M Unitek, Monrovia, Calif; Light Bond, Reliance Orthodontic Products, Itasca, Ill; BeautyOrtho Bond. Shofu, Kyoto, Japan; Kurasper F, Kuraray Medical, Tokyo, Japan; Heliosit Orthodontic. Ivoclar Vivadent AG, Schaan, Liechtenstein) and a flowable orthodontic resin (Salivatect, Shofu) were studied.

SEM

A total of 30 square blocks were made by injecting the resin into a metal mold (4 × 4 mm); immediately after, the surface was covered with a microslide glass and light cured for 60 seconds (BlueLEX, Yoshida Dental, Tokyo, Japan). The borders of each block were rounded with a cutter, and the samples were mounted in acrylic resin. The surfaces of the adhesives were gently polished with sandpaper sheets (1000, 2000, 4000 grit) and the fillers were naked to observe under SEM. Subsequently, a grinder polisher (Minimet 1000, Buehler, Lake Bluff, IL) was used adding 6 μ and 0.025 μ diamond pastes for 10 minutes (Metadi II, Diamond Polishing Compound, Buehler, Lake Bluff, IL). The surfaces were slightly etched with solution of 0.8% (wt/vol), H_3PO_4 for 10 seconds to obtain a clearer image during observation. After that, the specimens were ultrasonically cleansed for 5 minutes, placed on aluminum stubs with conductive tape, coated with osmium for 10 seconds (HPC-1S, Vacuum Device, Ibaragi, Japan), and observed under SEM (S-4500, Hitachi, Tokyo, Japan), with back-scattered electron signal.

TEM

The specimens were prepared by placing the adhesives directly into a silicon rubber plate for embedding; their surfaces were covered with a microslide glass and light cured for 60 seconds. The specimens were cut with a diamond knife (Diatome 45°, Biel, Switzerland) positioned in an ultra-microtome (MT2-B, Ivan Sorvall, Newtown, Conn). After which, ultra-thin sections of 80 nm in thickness were obtained. The sections were placed on fine grid-meshes (F-200, Nisshin EM, Tokyo, Japan) and observed under TEM (H-7100, Hitachi).

Microhardness test

Thirty discs were made with a plastic mold of 5.5 mm in diameter × 2 mm in height. The resin was placed into the mold; both surfaces were covered with slide glasses and light cured for 60 seconds. Vickers hardness of the adhesives was evaluated with a microhardness tester (Shimadzu HMV2, Newage Testing Instruments, Southampton, PA). The load was applied to the adhesive discs at 2.942 *N* for 10 seconds, and the scores were recorded in hardness Vickers (HV). The test was performed 50 times for every adhesive and the procedure was divided into 10 times for each resin disc. Descriptive statistics, including the mean and standard deviation, were calculated, and Scheffè's post hoc multiple comparison test (one-way analysis of variance) with significance predetermined at $P < .05$ was carried out.

EDX

Resins blocks of 4 × 4 mm were prepared as described before, and the specimens were placed on carbon stubs. The samples were coated with osmium for 5 seconds. The X-ray microanalysis of the adhesives was performed with Super Xerophy (S-817XI, Horiba Stec, Kyoto, Japan), attached to SEM S-4500. The information was obtained after 300 seconds of measurement.

RESULTS

SEM

The observation with back-scattered electron signal provided an adequate contrast between resin matrix and fillers (Figure 1). The shapes and sizes of the filler particles were different among the adhesives. The biggest particles were observed in Transbond XT followed by BeautyOrtho Bond and Kurasper F, these adhesives presented great variety of filler particle sizes. Light Bond showed slightly more homogeneous sizes of filler particles. Although Heliosit Orthodontic seemed to be an unfilled adhesive, few and small particles were observed. Salivatect (flowable resin) presented homogeneous, smaller, filler-particle sizes than the other adhesives; however, few slightly larger filler particles were also observed.

TEM

Images of the adhesives under TEM (excluding Light Bond) are presented in Figure 2. Great diversity of the resin matrix and filler particles was observed among the five adhesives. The resin matrix of BeautyOrtho Bond and Salivatect contains several microfillers surrounding the macrofillers, whereas few microfillers were found in Transbond XT and Kurasper F. Heliosit Orthodontic gave the impression of being composed of microfiller grouped in some areas and dispersed in the resin matrix. The sectioning with ultramicrotome and diamond knife resulted in complica-

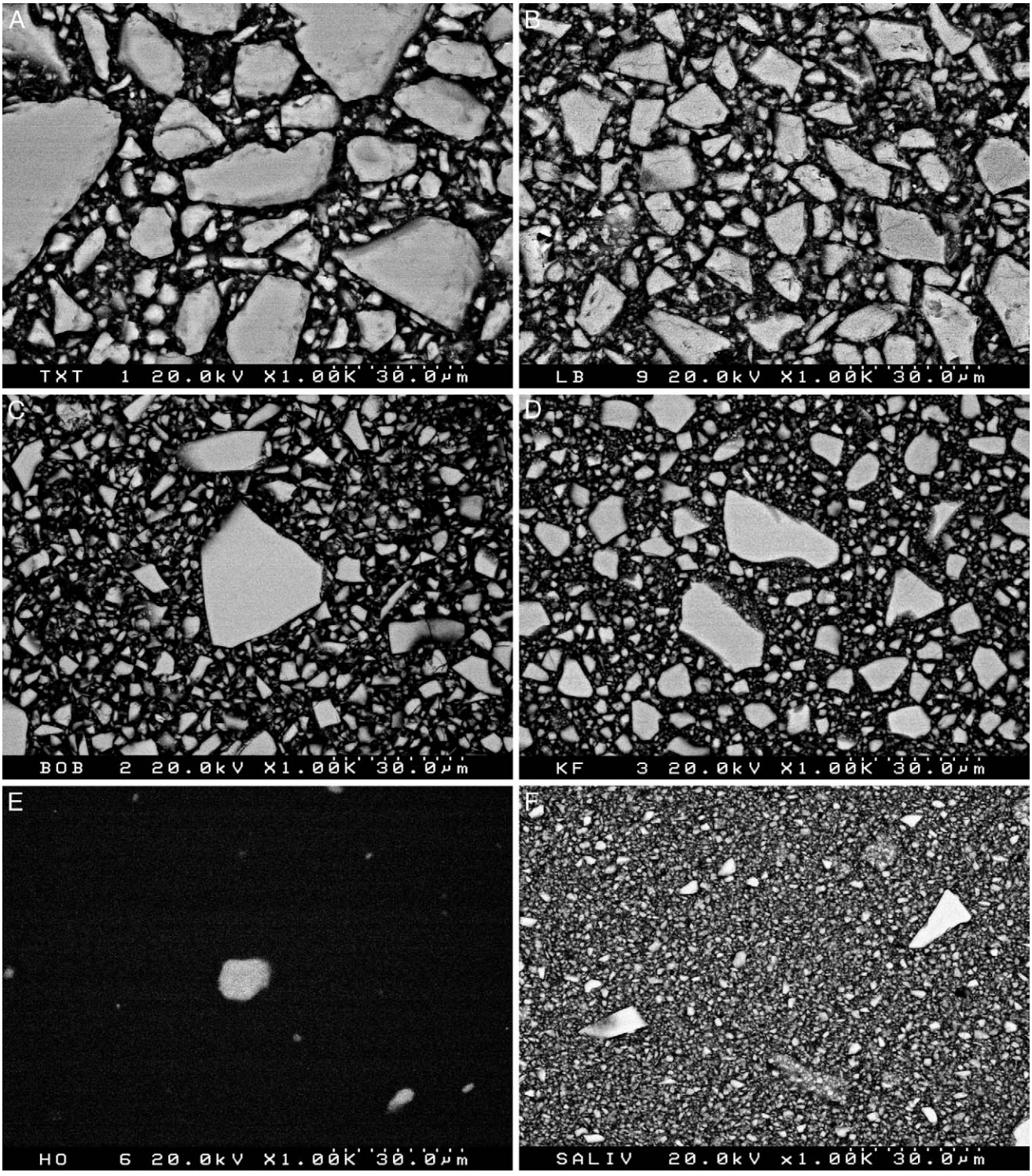

Figure 1. Scanning electron microscope back-scattered images of the orthodontics adhesives. (A) Transbond XT. (B) Light Bond. (C) BeautyOrtho Bond. (D) Kurasper F. (E) Heliosit Orthodontic. (F) Salivatect. (original magnification ×1,000.)

tions because of the hardness of the adhesives, and Light Bond could not be sectioned with this method. In addition, some cracks in the filler were produced by diamond knife, and these artifacts were confirmed when the ultra-thin sections were observed under SEM (Figure 3). Nonetheless, five adhesives were successfully observed, and the side effects described previously might be reduced or otherwise avoided using equipments for specimen preparation, such as focused ion beam.[20]

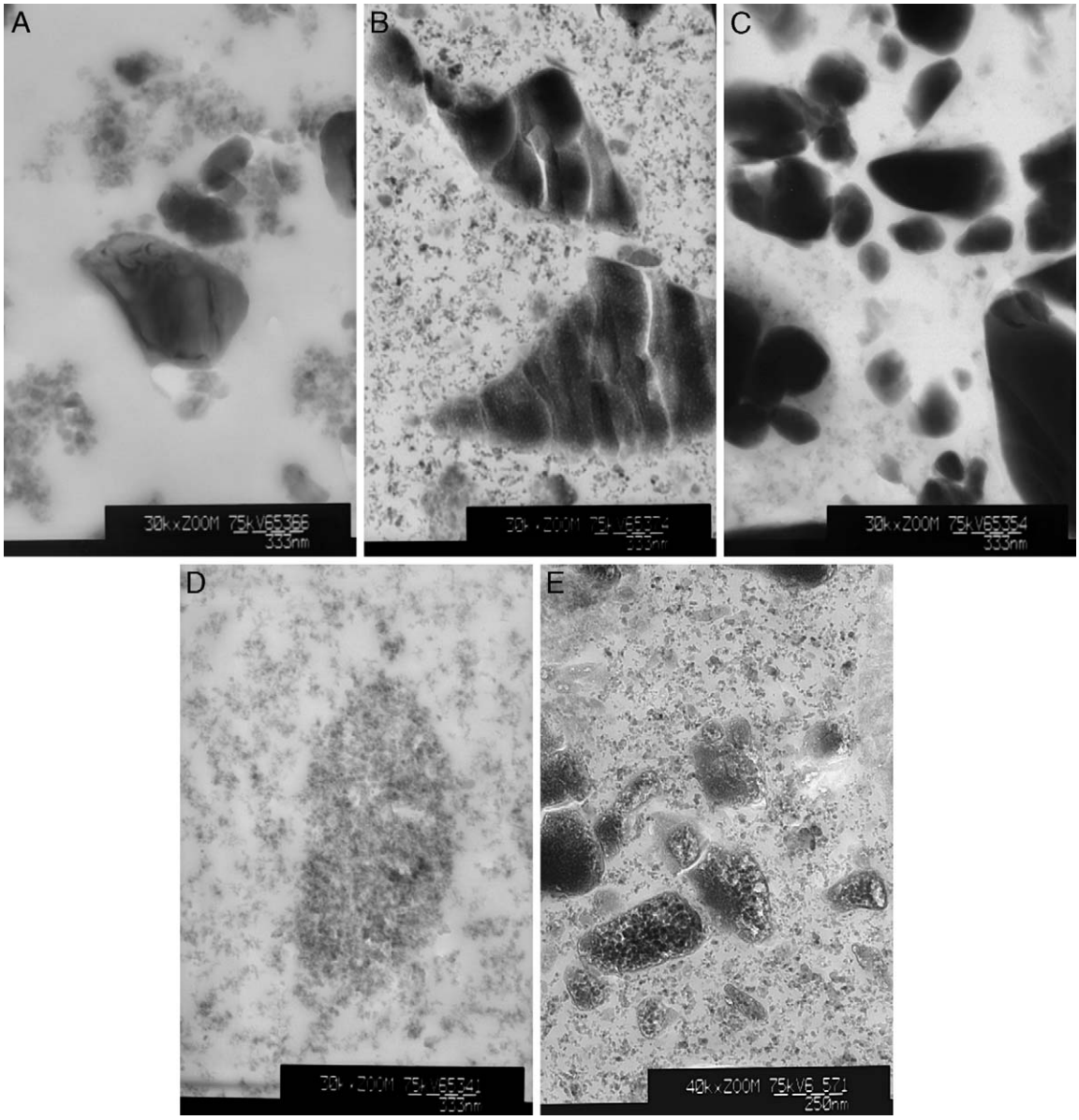

Figure 2. Appearance of the adhesives under transmission electron microscope. (A) Transbond XT. (B) BeautyOrtho Bond. (C) Kurasper F. (D) Heliosit Orthodontic (original magnification ×30,000). (E) Salivatect. (Magnification was increased to observe the smaller filler particles of this flowable adhesive, original ×40,000.)

Microhardness Test

Vickers hardness of the adhesives tested are shown in Table 1. There were great differences of microhardness among the adhesives, and the results were statistically significant in all comparisons except between Transbond XT and Salivatect. Light Bond showed the highest mean value (96.5 ± 9.5 HV), whereas Heliosit Orthodontic presented the lowest score (21.7 ± 1.3 HV).

EDX

The composition of the adhesives, including the elements percentage, is presented in Table 2. Similar elements, such as carbon, oxygen, and silicon were detected in all adhesives. The filler content showed interesting differences of elements and concentrations. Although the adhesives contained silicon, the concentration was different in every case; Light Bond presented the highest amount followed by Transbond XT. In addition, the filler content of BeautyOrtho Bond and Salivatect included sodium, strontium, and aluminum. Kurasper F was the only adhesive that contained barium.

DISCUSSION

Advances in orthodontic materials have affected orthodontic practice, most prominently in mechanother-

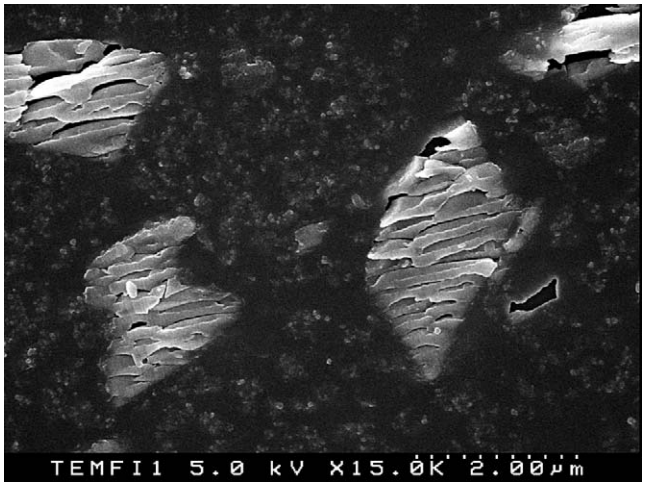

Figure 3. Ultra-thin section of an adhesive viewed under scanning electron microscope. (original magnification ×2,000.) The specimen preparation for transmission electron microscope (TEM) with ultramicrotome and diamond knife produced some damage of the fillers, and cracks are observed in the image. Nonetheless, 5 adhesives were successfully observed with TEM.

Table 1. Hardness of the adhesives

Adhesive	Mean	SD	Scheffèr Test*
Transbond XT	59.1	3.0	A
Light Bond	96.5	9.5	B
BeautyOrtho Bond	66.2	5.1	C
Kurasper F	79.3	6.3	D
Heliosit Orthodontic	21.7	1.3	E
Salivatect	56.1	9.7	A

* Adhesives with different letters are significantly different from each other.

apy and biomechanics research. The search for efficient materials and convenient techniques to improve and reduce treatment time has made significant progress.[21] As an example, the bond strength of orthodontic adhesives has been widely tested, and it continues been studied.[8–15] Nonetheless, the composition and properties of orthodontic adhesives should be also studied in detail. Composite resin content fillers are added to improve strengthen, increase stiffness, reduce dimensional changes, and improve handling. Presently, most composites are filled with silicate particles based on oxides of barium, strontium, zinc, aluminum, or zirconium. In despite of the great variety, there is no superiority of any specific filler because every type of filler offers advantages and disadvantages. However, the best mechanical properties could be achieved by incorporating high concentrations of filler particles of various sizes into the resin.[18]

When the adhesive were observed under SEM and TEM, an interesting diversity of ultrastructure was shown (Figures 1 and 2). Unfortunately, Light Bond could not be sectioned for TEM observation with the method described due to the significantly highest Vickers hardness. In addition, the composition of this adhesive exhibited the highest concentration of silicon and the lowest amount of carbon. These findings suggest that Light Bond presented greater filler content than the other five adhesives. Moreover, it has been reported that there is a linear relationship between composite wear and filler particle content and that increased filler levels are accompanied by greater wear resistance.[17]

Faltermeier et al[18] found that the filler level influences the bond strength of orthodontic brackets, because higher filled adhesives seem to provide greater bond strength than do lower filled or unfilled resins. On the other hand, Heliosit Orthodontic gave the impression of being an unfilled adhesive, and it showed the significantly lowest Vickers hardness. The lesser filler content of Heliosit Orthodontic illustrated in SEM and TEM micrographs agreed with the low concentration of silicon and the highest amount of carbon. In this context, clinicians should consider that polymerization shrinkage increases as the filler content decreases and this may cause formation of microleakage-promoting microgaps between the adhesive and the enamel surface, which might initiate the undesirable effect of white spot lesions.[22]

The mechanical properties in composite resins with high concentrations of filler particles of various sizes have shown better results.[18] Light Bond showed the highest filler content, whereas SEM illustrated a greater variety of filler particle sizes in Transbond XT followed by BeautyOrtho Bond and Kurasper F. The significant differences of Vickers hardness amongst the adhesives warrant further research to study the long-term effects of the adhesive hardness during orthodontic treatment.

Table 2. Energy dispersive X-ray microanalysis

	Carbon	Oxygen	Sodium	Aluminum	Silicon	Strontium	Barium	Total
Transbond XT	57.58	25.53			16.89			100%
Light Bond	38.16	35.33			26.50			99.99%
BeautyOrtho Bond	48.41	29.55	0.50	8.23	4.46	8.85		100%
Kurasper F	48.35	30.96			11.37		9.32	100%
Heliosit Orthodontic	64.65	28.59			6.76			100%
Salivatect	45.53	29.80	0.49	8.62	5.45	10.11		100%

Table 3. Concentration of fluoride in the adhesives

	Fluorine
Transbond XT	0.47
Light Bond	0.44
BeautyOrtho Bond	1.72
Kurasper F	0.00
Heliosit Orthodontic	0.29
Salivatect	3.12

The content of orthodontic adhesives is complex, and EDX provided general information of the elemental composition. In this connection, Transbond XT contained lower concentrations of silicon than Light Bond but higher concentrations than Kurasper F and much higher concentrations than Heliosit Orthodontic, BeautyOrtho Bond, and Salivatect. In contrast, deferent elements were detected in BeautyOrtho Bond and Salivatect because they are filled with surface prereacted glass ionomer (S-PRG) filler particles.[23] The content of S-PRG filler seemed to include strontium, aluminum, and silicon. Although the composition of BeautyOrtho Bond and Salivatect was similar, they differed on their appearance and filler particles sizes.

Despite all the advances in orthodontic material and treatment mechanics, demineralization around orthodontic brackets still remains a major problem for orthodontic patients.[24] The action of fluoride has demonstrated caries prevention,[25] and orthodontic adhesives have been formulated to release fluoride.[10,26] Hence, the concentration of fluoride was measured (Table 3). The highest amount was detected in Salivatect, followed by BeautyOrtho Bond; these findings could be explained because of S-PRG filler can release and recharge fluoride ions.[10,23]

In orthodontic practice, adhesive pastes are routinely used for the direct bonding of brackets, lingual buttons, and occasionally molar tubes. In contrast, the flowable adhesives could be prescribed for the indirect bonding of brackets and the placement of lingual bond retainers.[27] The smaller filler particles observed in Salivatect might be correlated to its flowable characteristic; nevertheless, the composition of this adhesive (filler content and level) was similar to that found in BeautyOrtho Bond, and its Vickers hardness was significantly higher than that of Heliosit Orthodontic and slightly lower than that of Transbond XT. The highest concentration of fluoride found in the composition of Salivatect could be a promising feature to prevent the formation of white spot lesions; however, further clinical evaluations are needed. On the other hand, the fluoride-releasing and rechargeable adhesives might be contraindicated for patients with fluorosis; in such patients additional bonding agents for atypical enamel surfaces could be recommended.[27]

The view of the orthodontic adhesives to the naked eye may expose differences in color, and their clinical use could show differences of handling; nonetheless, their ultrastucture, microhardness, and composition, as evaluated in this study, showed great differences. The analysis of orthodontic adhesives involves every stage of the treatment, because the brackets are bonded until the appliance is removed and the enamel is cleaned. In this light, some properties of adhesives require clinical studies during the active treatment, such as fluoride-releasing and recharging, filler level and filler content, hardness, amount of adhesive remnant, and ease of removal after debonding. Likewise, the action of the enamel conditioners is an important factor that should be considered.

CONCLUSIONS

- Although the bonding procedure has been improved with the development of new adhesives, further research is required to find the best qualities of the bonding agents.
- The diversity among orthodontic adhesives available in the market compels clinicians to analyze their characteristics to achieve a better bonding method.

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