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Mechanical properties of contemporary orthodontic adhesives used for lingual fixed retention

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Abstract

Objective: The aim of the present study was to test the mechanical properties of different adhesives used in orthodontics for fixed retainers and to investigate their possible interrelations.
Materials and methods: Specimens of six different adhesive resins were prepared: Transbond XT, Transbond LR and an experimental BPA-free orthodontic adhesive, as well as IPS Empress Direct (IPS-ED), ZNano and Accolade. The mechanical properties tested were Martens hardness (HM), indentation modulus (E_{IT}), the ratio of elastic to total work, commonly known as elastic index (η_{IT}) and Vickers hardness (HV). These properties were determined using instrumented indentation testing according to ISO 14577-2002. The results of the aforementioned properties were statistically compared with one-way ANOVA-test and Student-Newman-Keuls multiple comparison test at a=0.05, while possible correlations among the properties tested were analyzed by Pearson correlation.
Results: Significant differences were identified among all the materials tested for HM, with Transbond LR presenting the highest value. This resin presented the highest E_{IT} too. Significant E_{IT} differences were identified among the materials and only ZNano and IPS-ED showed no significant differences for this property. Transbond LR and ZNano showed higher HV values. ZNano demonstrated the highest elastic index. Pearson analysis showed a strong positive correlation between HM and E_{IT} (0.970), HM and HV (0.837), and E_{IT} and HV (0.695), while a weak negative correlation was found between E_{IT} and elastic index (-0.505).
Conclusions: The materials tested demonstrated significant differences in their mechanical properties, and thus differences in their clinical performance are anticipated.

Keywords: dental composite resin; fixed retainer; hardness; indentation modulus.

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Introduction

Adhesives are widely used in dentistry, and their mechanical properties have important implications in their clinical performance and longevity. Yet the requirements concerning the clinical properties are different for adhesives used to bond fixed orthodontic retainers than for adhesives used to bond brackets, or adhesives applied elsewhere in dentistry [8]. The stipulation to achieve life-long retention after orthodontic treatment makes high adhesive forces a prime goal for an adhesive for fixed retainers, especially given that detachment has been recognized as the main cause for retainer failure [6]. Accordingly, increased shear bond strength [21, 23] and wire pull out resistance [3] are required. Moreover, a greater surface area of the lingual retainer composite resin remains exposed to the oral cavity than in cases of bracket adhesives. Therefore, material hardness [19], wear resistance and water absorption [20, 24] are among the critical mechanical properties for the longevity of fixed retainers under intra-oral conditions. In addition, fixed retainer adhesives are more vulnerable to aging mechanisms as they remain in the oral cavity for longer periods than the bracket adhesives [1, 16].

The biomechanical response of a fixed retainer is very complicated to be precisely defined. However, the determination of the mechanical properties of its primary elements (i.e. adhesive cement and orthodontic wire) is a prerequisite for the clarification of its clinical performance. The traditional mechanical setups, such as tensile, bending, torque and others, require bulky specimens with specific dimensions, which are sometimes impossible to produce in the determined dimensions. Especially for photo-cured dental resins, a homogenized polymerization is hindered by the shallow polymerization depth. When compared to conventional mechanical testing, the development of instrumented indentation testing (IIT) can provide an array of mechanical properties (elastic modulus, creep, relaxation, different expression of hardness and others), according to ISO [4, 10, 13], based on the continuous recording of time, force and indentation depth with a Vickers, Berkovich or any other type of indenter that is in contact with the sample surface.

Although the Vickers hardness (HV) of orthodontic adhesives has been tested, there is no information for other mechanical properties and thus the present investigation aimed to clarify whether significant differences exist in the mechanical properties of different adhesives routinely used in orthodontics for fixed retainers and if a recommendation could be made based on these results. Moreover, the study aims to investigate possible interrelations among different mechanical properties. Given the diversity of monomers and filler types among orthodontic adhesives, the null hypothesis is that significant differences in mechanical properties are anticipated among different materials.

Materials and methods

In this study, six different adhesive resins were tested. Their commercial names, the monomer type, filler type and content are presented in Table 1. Disk-shaped specimens (diameter 15 mm, height 2 mm, n=6) were prepared by dispensing each material into an opaque rubber cylindrical mold placed between transparent matrix strips and microscopic glass slides, to remove excess and avoid oxygen inhibition. The materials were polymerized for 40 s employing an LED light curing unit (Radii plus, SDI, Bayswater, Victoria, Australia) operated at a standard high irradiance mode (1500 mW/cm^2). The tip of the curing unit was moved across the entire surface to obtain a homogeneous exposure. The directly irradiated surfaces were then ground up to 4000 grit size SiC water coolant papers in a grinding/polishing unit (DAP-V, Struers, Ballerup, Denmark) to remove the resin-rich layer.

Then the mechanical properties were determined using IT according to ISO 14577-2002. Force to indentation depth curves were monitored during testing using a load of 9.8 N in a universal hardness testing machine ZHU0.2/Z2.5, with an accuracy of ±5% of the indicated load; the standard resolution of depth measurement system was 0.02 μm. The mechanical properties tested were Martens hardness (HM), indentation modulus (E_{IT}), the ratio of elastic to total work (η_{IT}) and HV. The results were statistically analyzed by one-way ANOVA test and the significant differences among groups were identified by post hoc Student-Newman-Keuls multiple comparison test at a=0.05. Finally, possible correlations among all properties tested were analyzed using Pearson correlation.

Results

Figure 1 illustrates representative force-indentation depth curves from all materials included in this study. The decreased indentation depth denotes lower hardness while a more vertical unloading curve denotes increased indentation modulus. All the numerical data along with the results of statistical analysis are presented in Figure 2 in descending order from left to right. Significant differences were identified among all materials tested for HM

Table 1: List of the evaluated orthodontic adhesives and dental restorative composites (manufacturer, chemical composition of matrix and filler as well as filler content by weight and code).

Product	Composition[a]	Manufacturer	Code
Transbond-LR (orthodontic adhesive)	BisGMA, TEGDMA, silanated quartz (75–85% wt)	3M ESPE, St. Paul, MN, USA	TBLR
Experimental BPA-free (orthodontic adhesive)	PCDMA, TEGDMA, UEDMA silanated glass (70 wt%)	Danville Materials S. Ramon, CA, USA	BPA-FR
Transbond-XT (orthodontic adhesive)	BisEMA, BisGMA, silanated quartz, silanated silica (70–80% wt)	3M ESPE, St. Paul, MN, USA	TBXT
Accolade (microhybrid flowable restorative)	BisGMA, TEGDMA Ba-glass, amorphous silica (65% wt)	Danville Materials S. Ramon, CA, USA	ACC
IPS Empress Direct Dentin (nanohybrid restorative)	UEDMA, TCDDMA, BisGMA, Ba-Al-fluorosilicate (0.7 μm), ytterbium trifluoride (100 nm), prepolymers (1–10 μm) (80% wt total)	Ivoclar Vivadent Schaan, FL	IPS-ED
ZNano (nanoparticle restorative)	BisGMA, TEGDMA nanodispersed zirconia/silica (80 nm, 73% wt)	Danville Materials S. Ramon, CA, USA	ZNano

[a]BisGMA, Bisphenol A-glycidyl dimethacrylated; BisEMA, ethoxylated bisphenol A- dimethacrylate; UEDMA, urethane dimethacrylate; TCDDMA, tricyclodocane; TEGDMA, triethylene glycol dimethacrylate; PCDMA, phenyl-carbamoyloxypropane dimethacrylate.

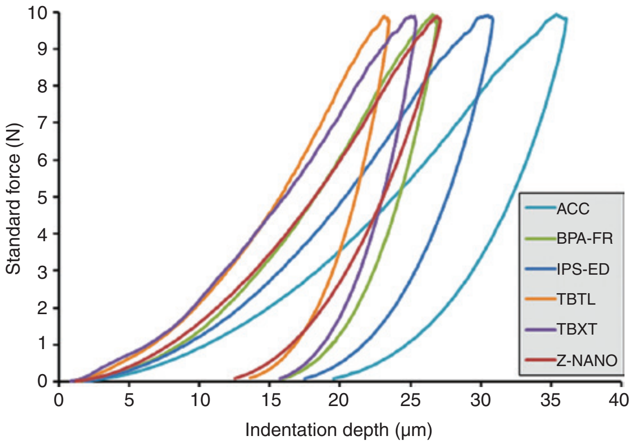

Figure 1: Representative force-indentation depth curves for all the materials tested.

(Figure 2A), while only ZNano and IPS Empress Direct (IPS-ED) showed no significant differences for E_{IT} (Figure 2B). ZNano demonstrated the highest elastic index followed by Accolade (ACC) (Figure 2C). No significant differences were identified for IPS-ED, experimental BPA-free orthodontic adhesive (BPA-FR) and Transbond-LR (TBLR), while Transbond-XT (TBXT) showed the lowest elastic index. TBLR and ZNano showed higher HV values with significant differences compared to TBXT. BPA-FR and IPS-ED showed lower HV values but without significant differences between each other, while ACC had the lowest HV (Figure 2D).

Table 2 presents Pearson correlation coefficients r and p values among the properties tested. A strong positive correlation was identified between HM and E_{IT}, HM and HV and E_{IT} and HV, while a weak negative correlation was found between E_{IT} and elastic index (Figure 3).

Discussion

Based on the statistically significant differences in mechanical properties among the materials tested, the null hypothesis must be accepted. Three of the products tested (TBLR, TBXT, BPA-FR) showed positive results as orthodontic adhesives. TBLR has been developed specifically for bonding lingual retainers and is used extensively in clinical practices and recent experimental studies, commonly as the control adhesive [16, 19, 21, 23]. TBXT is an orthodontic bonding adhesive resin used mostly for

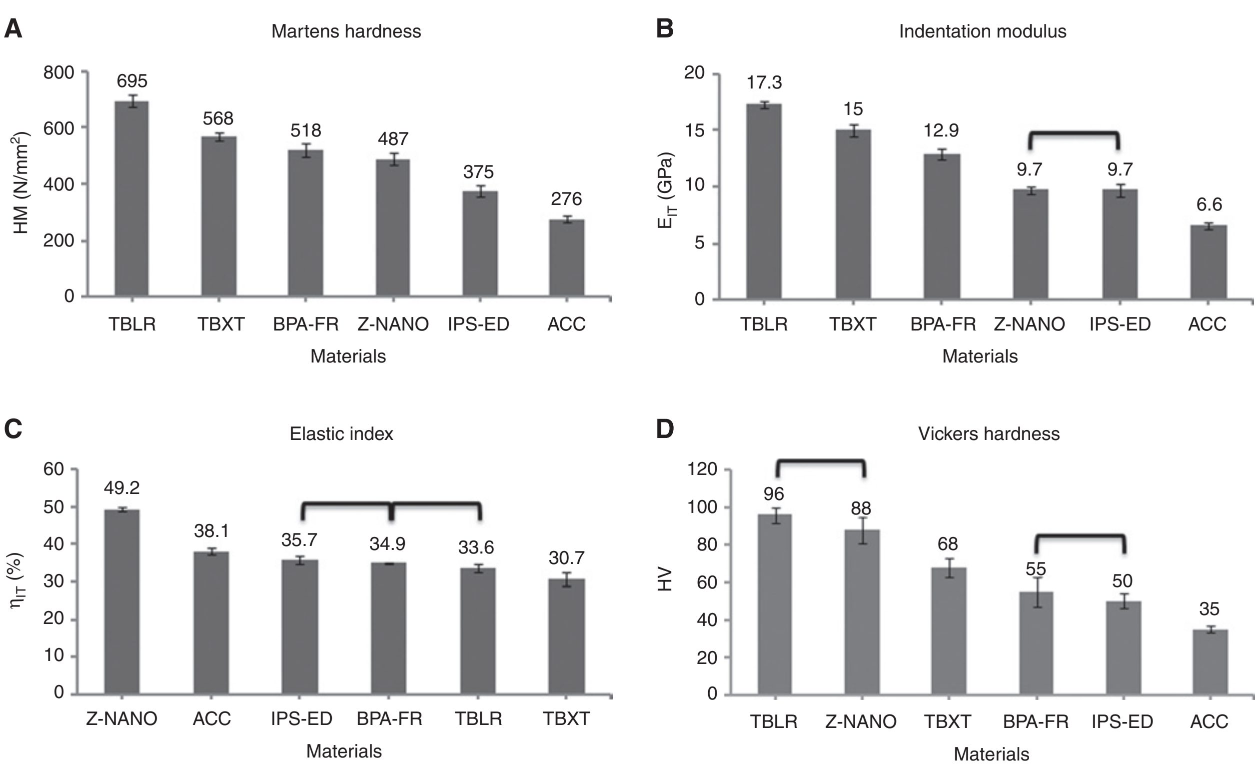

Figure 2: The results of the mechanical properties tested in descending order.
Lines above columns connect mean values without statistical significant differences (p>0.05). (A) Martens hardness, (B) indentation modulus, (C) elastic index and (D) Vickers hardness.

Table 2: Correlation coefficients r and p values of mechanical properties tested after Pearson correlation.

Property	Indentation modulus	Elastic index	Vickers hardness (HV)
Martens hardness (HM) (N/mm^2)	r=0.970 p<0.001	NC	r=0.837 p<0.001
Indentation modulus (E_{IT}) (GPa)		r=-0.505 p<0.050	r=0.695 p<0.001
Elastic index (%)			NC

NC, No correlation (p>0.05).

brackets but has been occasionally used for the construction of fixed retainers in the laboratory setting [5, 22, 23]. The rest of the three products were tested for comparison purposes. ACC is a flowable composite, IPS-ED is a nanohybrid and ZNano a nanoparticle restorative composite. Although these materials have no indication for orthodontic bonding, restorative dental resin has been used in the past for the preparation of lingual retainers in a clinical or laboratory setting [2, 23]. Recently, some flowable composites, originally created for restorative dentistry, have been used for the construction of fixed retainers [7, 9].

Fabrication of specimens for testing of adhesives utilized in lingual fixed retainer bonding should differ from their conventional bonding brackets counterparts to account for the differences in structure and design. In the former, the objective is two-fold as, along with the bonding of the construction (wire-adhesive) to the enamel, the adhesive must be of significant thickness to provide the required wear resistance. This necessity arises considering the exposure of the retainer to the masticatory forces, necessitating thus, a material of greater thickness as opposed to conventional bracket bonding, where only a margin of the adhesive on the

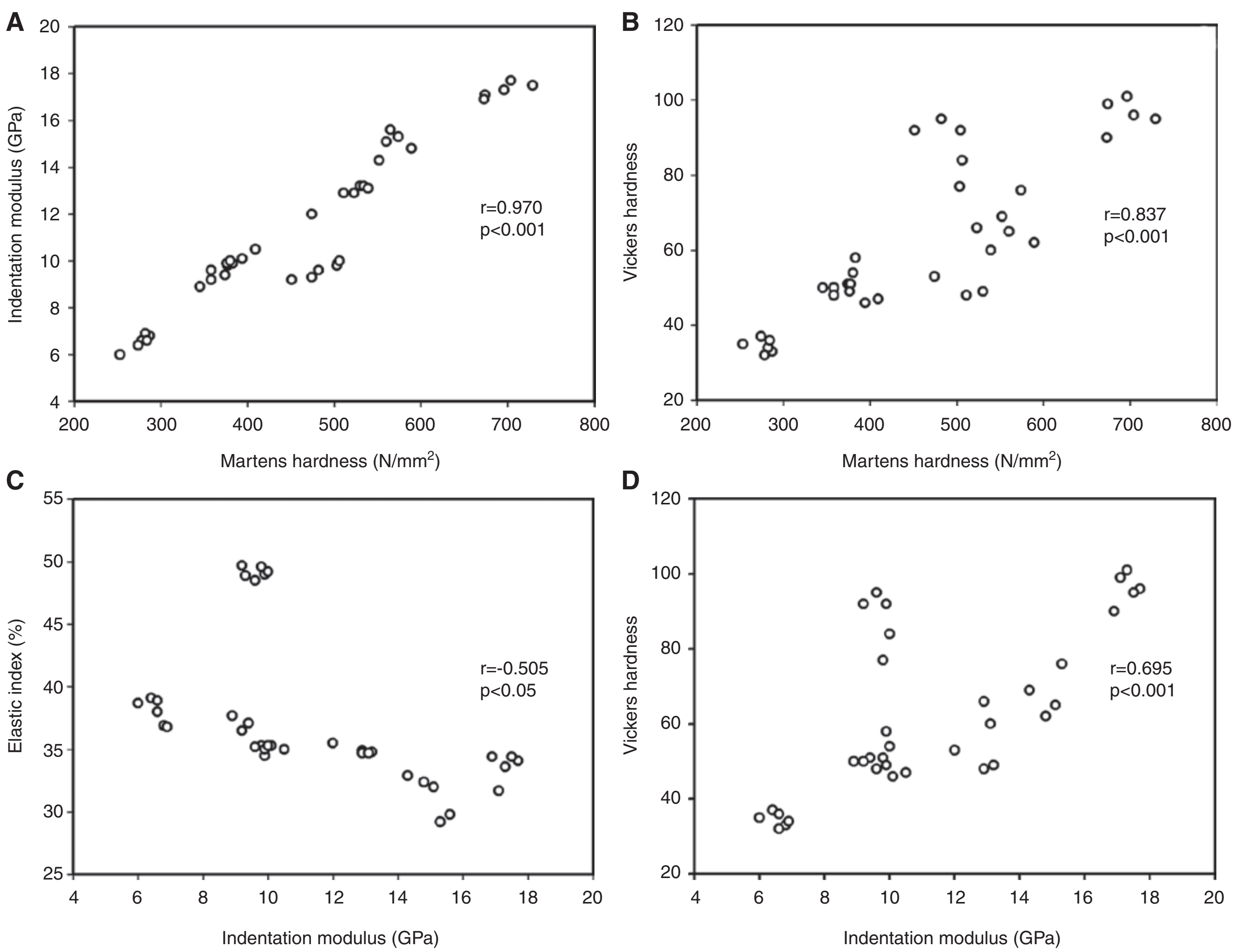

Figure 3: Graphical presentation of correlations between the properties tested with r and p values after Pearson analysis.
(A) Martens hardness vs. indentation modulus, (B) Martens vs. Vickers hardness, (C) indentation modulus vs. elastic index and (D) indentation modulus vs. Vickers hardness.

periphery of the bonded bracket is exposed to the oral cavity.

Two different expressions of Hardness were included in this study. HM is derived automatically by IIT employing force, indentation depth and impression surface, while HV is traditionally measured based on the diagonal of the resulted impression on the material surface. These two different expressions of the same property were selected for the following reasons: HM is derived by a fully automated procedure overcoming parameters affecting the accuracy of HV (i.e. resolution of optical system, operator's perception, rebound of material around the indentation and others [17], however there is no data for HM in dental literature and thus HV was selected for comparisons with previous studies.

TBLR showed the highest HV values, which were higher than the values presented in dental literature [16, 19]. TBXT showed identical HV values as those provided by Iijima et al. [12] and close to those provided by Uysal et al. [20]. Similarly IPS-ED demonstrated similar values to the ones given by Bauer & Illie [1]. The differences in HV values might be appended to the different loading conditions (2.94 N compared to 9.8 N) and the possible implication of indentation size effect (ISE), a complication where the hardness testing tends to over- or underestimate Vickers values [17] below a threshold, which is dependent of the material type. Indentation modulus of ZNano was found to be close to the value given by the manufacturer (9 GPa) [11], and IPS-ED to 7.1 given by Bauer & Illie [1]. The total work of indentation is divided into elastic and plastic parts, but as the absolute values are load-dependent the elastic index is defined as the elastic to total work ratio, which is independent of the load applied [15]. The increase of elastic index occurs at the expense of plastic energy and thus a higher elastic index indicates a less ductile material. Based on the results of this study, we found that the restorative ZNano is the less ductile material and TBXT the most ductile one. TBLR was found to be less ductile than TBXT.

As expected, HM and HV showed a strong correlation as it is actually a different expression of the same property. Both HM and HV illustrated a strong correlation with E_{IT}, a finding which is in accordance to previous research correlating Knoop and Brinnel hardness to elastic modulus of resin composites [14, 18]. Elastic index showed a weak negative correlation to E_{IT} denoting that the increase in modulus is achieved at an expense of ductility of the material. This might be explained by the fact that the increase in modulus is mainly achieved with an increase in filler loading, which decreases the capacity of the matrix for plastic deformation. However, composites need a high percentage of inorganic-filler particles to withstand high mechanical stresses [10].

Mechanical properties tested also have clinical implications. Hardness is a factor of prime importance considering the abrasion that can be caused by mastication. Hence materials with higher hardness, and thus high wear resistance are preferred for the construction of the retainers. Different commercial brands of resin composites have different hardness levels, but diluting the composite before bonding may further decrease this property [3, 19]. TBLR is used specifically for bonding lingual retainers and the higher hardness is required because, in several cases, fixed retainers remain bonded for even more than 10 years [6]. Moreover, in the specific application of lingual fixed retainer bonding, the adhesive is exposed to the oral environment for a substantially larger time, i.e. more than a decade, than the conventional orthodontic application of bonding brackets to enamel, which may exceed 10 years. Concomitantly, the required integrity of the lingual retainer does not only take into account the survival of the bond to enamel, but also includes a property, which defines the resistance of the composite to wear caused due to the masticatory forces during contact of the opposing dentition with the retainer, or stresses developed from the contact of the bolus of food to the lingual fixed retainer construction. Thus wear resistance, which is not a property of importance when bonding brackets owing to the lack of exposure of the adhesive to the oral environment, is of fundamental significance in lingual fixed retainer bonding.

Moreover, the elastic properties of the fixed retainer elements must be measured if its performance is to be fully described. Elastic modulus is associated with the dimensional properties of lingual retainers as materials with higher modulus (higher rigidity) can provide equal resistance to exerted stresses with smaller cross sections, making the acceptance from the patient easier. TBLR presents high modulus, which means higher resistance in elastic deformation under the same stresses compared with the other composites. If the modulus is reduced, then the elastic deformation of the structure would perhaps predispose for detachment from the enamel because the composite, or fracture of the wire-adhesive interface.

Conclusions

The orthodontic adhesives tested demonstrated significant differences in their mechanical properties.

Strong correlations were identified between the hardness and elastic modulus.

The orthodontic adhesive for fixed retainers presented the highest hardness and resistance in elastic deformation between the evaluated composites.

More research should examine properties of the materials used for lingual fixed retainer bonding, using clinically important and application-related properties, which has applications for this specific construction (lingual fixed retention), such as wear resistance.

References

[1] Bauer H, Ilie N. Effects of aging and irradiation time on the properties of a highly translucent resin-based composite. Dent Mater J 2013; 32: 592–599.

[2] Bearn DR. Bonded orthodontic retainers: a review. Am J Orthod Dentofacial Orthop 1995; 108: 207–213.

[3] Bearn DR, McCabe JF, Gordon PH, Aird JC. Bonded orthodontic retainers: the wire-composite interface. Am J Orthod Dentofacial Orthop 1997; 111: 67–74.

[4] Chudoba T. Measurement of Hardness and Young's Modulus by Nanoindentation. In: Cabaleiro A, De Hosson JT, Lockwood DJ, editors. Nanostructured Coatings. New York: Springer Science 2006: 216–260.

[5] Cooke ME, Sherriff M. Debonding force and deformation of two multi-stranded lingual retainer wires bonded to incisor enamel: an in vitro study. Eur J Orthod. 2010; 32: 741–746.

[6] Dietrich P, Patcas R, Pandis N, Eliades T. Long-term follow-up of maxillary fixed retention: survival rate and periodontal health. Eur J Orthod 2015; 37: 37–42.

[7] Elaut J, Asscherickx K, Vande Vannet B, Wehrbein H. Flowable composites for bonding lingual retainers. J Clin Orthod 2002; 36: 597–598.

[8] Eliades T. Dental Materials in Orthodontics, Ch. 28. In: Graber LW, Vanarsdall RL Jr., Vig KWL, editors. Orthodontics: Current Principles and Techniques. 5th ed. Philadelphia: Elsevier Mosby 2012: 1023–1038.

[9] Geserick M, Wichelhaus A. A color-reactivated flowable composite for bonding lingual retainers. J Clin Orthod 2004; 38: 165–166.

[10] Hirayama S, Iwai H, Tanimoto Y. Mechanical evaluation of five flowable resin composites by the dynamic micro-indentation method. J Dent Biomech 2014; 5: 1–8.

[11] http://danvillematerials.com/UploadedFiles/Account_2/MediaFiles/ZNano%20Product%20Sheet.pdf) Accessed June 0, 2015.

[12] Iijima M, Muguruma T, Brantley WA, Yuasa T, Uechi J, Mizoguchi I. Effect of mechanical properties of fillers on the grindability of composite resin adhesives. Am J Orthod Dentofacial Orthop 2010; 138: 420–426.

[13] ISO 14577-1. Metallic materials-Instrumented indentation test for hardness and materials parameter – Part 1: test method. Geneva: International Organization for Standardization 2002.

[14] Li J, Li H, Fok AS, Watts DC. Multiple correlations of material parameters of light-cured dental composites. Dent Mater 2009; 25: 829–836.

[15] Mencik J. Determination of mechanical properties by instrumented indentation. Meccanica 2007; 42: 19–29.

[16] Ramoglu SI, Usumez S, Buyukyilmaz T. Accelerated aging effects on surface hardness and roughness of lingual retainer adhesives. Angle Orthod 2008; 78: 140–144.

[17] Shahdad SA, McCabe JF, Bull S, Rusby S, Wassell RW. Hardness measured with traditional Vickers and Martens hardness methods. Dent Mater 2007; 23: 1079–1085.

[18] Thomaides S, Kakaboura A, Mueller W, Zinelis S. Mechanical properties of contemporary composite resins and their interrelations. Dent Mater 2013; 29: e132–141.

[19] Uşümez S, Büyükyilmaz T, Karaman AI. Effects of fast halogen and plasma arc curing lights on the surface hardness of orthodontic adhesives for lingual retainers. Am J Orthod Dentofacial Orthop 2003; 123: 641–648.

[20] Uysal T, Basciftci FA, Sener Y, Botsali MS, Demir A. Conventional and high intensity halogen light effects on water sorption and microhardness of orthodontic adhesives. Angle Orthod 2008; 78: 134–139.

[21] Uysal T, Ulker M, Akdogan G, Ramoglu SI, Yilmaz E. Bond strength of amorphous calcium phosphate-containing orthodontic composite used as a lingual retainer adhesive. Angle Orthod 2009; 79: 117–121.

[22] Uysal T, Ulker M, Baysal A, Usumez S. Different lingual retainer composites and the microleakage between enamel-composite and wire-composite interfaces. Angle Orthod 2008; 78: 941–946.

[23] Veli I, Akin M, Kucukyilmaz E, Uysal T. Shear bond strength of a self-adhering flowable composite when used for lingual retainer bonding. J Orofac Orthop 2014; 75: 374–383.

[24] Zhao J, Platt JA, Xie D. Characterization of a novel light-cured star-shape poly(acrylic acid)-composed glass-ionomer cement: fluoride release, water sorption, shrinkage, and hygroscopic expansion. Eur J Oral Sci 2009; 117: 755–765.