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ORIGINAL ARTICLE

Evaluation of the mechanical properties of dental adhesives and glass-ionomer cements

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Abstract Adhesives and lining/base materials should relieve the stresses concentrated at the tooth/restoration interface. The study aimed at comparing the mechanical properties of eight adhesives and six glass-ionomer cements (GICs). The adhesives were applied on dentin disks, whereas 2 mm×3 mm×2 mm GICs specimens were prepared in a teflon mold. Vicker's hardness (VH), elastic modulus (*E*), creep (Cr) and elastic work (We/Wtot) were measured with a micro hardness indenter. One-way ANOVA and Tukey's test were used to compare the mechanical properties within each materials' type and among the materials' classes. Enamel and dentin were used as references. Significant differences were detected within each materials' type and among the materials' classes and enamel and dentin. GICs were superior to adhesives in VH and E and showed a VH similar to dentin. GICs presented mechanical properties more similar to enamel and dentin than adhesives.

Keywords Adhesives · Mechanical properties · Glass-ionomer cements · Elastic modulus · Hardness

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Introduction

The interface between a resin composite restoration and the tooth is subjected to stresses potentially leading to debonding and subsequent clinical failure of the restoration. The interfacial stress occurs even before the restored tooth is subjected to functional load, due to the polymerization shrinkage of the composite [1]. An adhesive or a lining/base material may act as an elastic intermediate layer between the tooth and the resin composite. These materials can resist polymerization shrinkage [2] and absorb the shock produced by occlusal loads [3]. Besides conventional adhesives, flowable composites [4] or glass-ionomer cements (GICs) [5] could also be used for this purpose. Nevertheless, the information on the interfacial stress between restoration and tooth, as well as on the materials which could relieve this stress, is mainly based on the results of laboratory studies, whereas a clinical validation is still lacking.

Due to the ability to bond directly to dental tissues that can be enhanced by a short polyalkenoic acid pretreatment for conditioning tooth surface [6], and thanks to the property of fluoride release [7], glass-ionomer cements have found several applications in the dental practice. Besides their use as base materials [5], they have been used for core build-up procedures [8] and for luting prosthetic restorations [9] and orthodontic bands [10]. They have also been proposed for the atraumatic restorative treatment (ART) technique [11], and as dental sealants [12, 13]. They are suitable for class III and V cavity restorations, whereas for class I or II restorations their use is advisable only in low stress bearing areas [14].

The limited use of conventional glass-ionomer cements in the posterior region could be ascribed to their poor mechanical properties, compared to those of resin compo-

sites [15, 16]. Later, resin-modified glass-ionomer cements were launched on the market. The latter showed improved mechanical properties, comparable to those of microfilled and packable composites [17], though poor wear rates.

Although the mechanical properties of conventional and resin-modified glass-ionomer cements have already been assessed and compared to those of resin composites, information regarding the mechanical behaviour of these materials in comparison with bonding agents is still lacking. In a literature review by Peumans et al. [18], glass-ionomer cements have been reported to provide a better clinical retention of non-carious cervical restorations as compared with conventional adhesives [18]. Conversely, laboratory studies reported lower bond strengths to tooth substrates for glass-ionomer cements compared to those of composite restorations bonded with dental adhesives [13, 19]. However, recent studies showed an improvement of the clinical performance of simplified adhesives [20, 21]. Besides the loss of retention, the marginal breakdown was also reported to be a common reason for restorations' failure [22]. The loss of marginal integrity could also be related to poor mechanical properties of the bonding and/or restorative materials. Thus, it could be of interest to assess the mechanical features of these materials.

Therefore, the aim of this laboratory study was to compare the mechanical properties of eight adhesives (five one-step self-etch, two two-step self-etch and one two-step etch-and-rinse) and six glass-ionomer cements (two conventional and four resin-modified) and to confront them with those of enamel and dentin. The tested null hypotheses were (1) that the mechanical properties of the materials within each class of materials are comparable and (2) that the mechanical properties of GICs and adhesives are similar to each other and also to those of enamel and dentin.

Materials and methods

Specimens preparation and measurement of the mechanical properties

Batch numbers, chemical compositions and modes of use of all the materials used in this study are reported in Table 1.

Forty sound human third molars were collected for the preparation of the adhesives specimens. Any residual soft tissue was removed from the tooth surface with a hand scaler. The teeth were disinfected in 2.5% sodium hypochlorite for 2 min and stored in distilled water at 4°C until they were used for the study. The superficial enamel was removed from the occlusal aspect of the teeth by grinding with a wet 220-grit silicon carbide paper disk. The crowns were sectioned perpendicular to the long axis in order to obtain one 1-mm-thick slice from each tooth (Isomet; Buehler, Lake Bluff, IL, USA). The peripheral enamel was removed with wet 220-grit silicon carbide paper in order to obtain disk-shaped specimens consisting only of dentin. A clinically relevant smear layer was created by grinding the occlusal aspect of each dentin disk with 600-grit silicon carbide paper under water cooling. Five teeth were assigned to each tested adhesive resulting in five bonded dentin disks. For the adhesives based on the etch-and-rinse technique (Admira Bond and Solobond Plus; VOCO), dentin was previously etched with 35% phosphoric acid gel (Vococid; VOCO, Batch # 560819) for 15 s, abundantly rinsed with deionised water and air dried. All the tested adhesives were applied on the dentin disks and light-cured through a Mylar stripe according to their respective modes of use as reported in Table 1.

In order to obtain the GICs specimens, a 2 mm×3 mm×2 mm teflon mold with opened upper and lower surfaces was positioned on a glass plate with a polyacetate sheet interposed. The materials were introduced into the mold and the upper surface was covered with a polyacetate sheet. A second glass plate was compressed on the upper surface of the mold in order to obtain specimens with flat surfaces. The specimens of the resin-modified GICs were light-cured in a top-to-bottom direction and then left undisturbed for 15 min in the mold prior to be stored. The conventional GICs were simply left undisturbed for 15 min in the mold prior to storage. Five specimens were prepared with each tested GIC.

The same LED curing unit (Elipar Freelight 2; 3M ESPE) was used throughout the study. The spectral distributions and the irradiance of the curing unit were determined by means of a calibrated fibre optic spectrally resolving radiometer equipped with an integrating sphere (S2000; Ocean Optics, Dunedin, FL, USA). The total irradiance was obtained by the integrate calculus of the irradiance as a function of the wavelength over the entire wavelength range, divided by the effective area of the curing unit tip. The diameter of the tip was measured with a digital micrometre and the effective area was defined as the area of the tip without cladding. The total irradiance of the curing unit was 1,226 mW/cm^2.

All the specimens were stored for 24 h prior to testing of mechanical properties. The GICs specimens were stored in deionised water at 37°C, whereas the adhesives were kept in an environment 100% saturated with humidity at 37°C. Vicker's hardness, modulus of elasticity, elastic indentation work and creep of the tested materials were assessed. The measurements were performed by means of a micro hardness indenter (Fischerscope H100C; Fischer, Sindelfingen, Germany). The test procedure was carried out force controlled. A load application time of 50 s was set and subdivided as follows: the force increased at a constant speed from 0.4 to 30 mN in 20 s, the maximal force of

Table 1 Batch numbers, chemical compositions and modes of use of the materials used in the study

Material	Type	Composition	Mode of use
Admira Bond (VOCO) Batch # 580606	Two-steps etch-and-rinse adhesive	Ormocers, Bis-GMA, HEMA, BHT, acetone, organic acids	Apply on dentin 30 s. Air dry. Light-cure 20 s
Futurabond NR (VOCO) Batch # 610643	One-step self-etch adhesive	*Liquid A*: Polyfunctional adhesive monomers (methacroyl-phosphorus-acid-ester, methacroyl-carbon-acid-ester), dimethacrylates, functionalised SiO_2-nano-particles, Initiators *Liquid B*: ethanol, water, hydrophilic adhesive monomers, fluorides	Mix Liquid A and Liquid B 5 s. Apply mixture on dentin and massage 20 s. Air dry 5 s. Light-cure 20 s
Solobond Plus (VOCO) Batch # 591648	Three-steps etch-and-rinse adhesive	*Primer*: water, acetone, maleic acid, acid-functionalised methacrylates, fluorides *Adhesive*: acetone, dimethacrylate, hydroxymethacrylate	Apply Primer 30 s. Apply Adhesive 15 s. Air dry. Light-cure 20 s
Hybrid Bond (Sun Medical) Batch # LS2	One-step self-etch adhesive	*Hybrid base*: methyl methacrylate, 4-methacryloxyethyiltrimellitic acid anhydride, tris(2-hydroxyethyl)-isocyanurat-triacylate, HEMA, acetone, water *Hybrid Brush*: Sodium *p*-toluenesulfinate, aromatic amine	Dispense few drops of hybrid base in the plastic dispensing dish. Stir with hybrid brush for few seconds. Apply on dentin 20 s. Air blow 5-10 s. Light-cure 20 s
Clearfil SE Bond (Kuraray) Batch # 41471	Two-steps self-etch adhesive	*Primer*: HEMA, MDP, Hydrophilic aliphatic dimethacrylate, DL-camphorquinone, water, accelerators, dyes and others *Bond*: HEMA, Bis-GMA, MDP, Hydrophobic aliphatic dimethacrylate, colloidal silica, DL-camphorquinone, initiators, accelerators and others	Apply the Primer on dentin. Leave in place 20 s. Air dry. Apply the Bond. Make the Bond film uniform with a gentle air blow. Light-cure 20 s
Clearfil S^3 Bond (Kuraray) Batch # 41117	One-step self-etch adhesive	2 HEMA, ethanol, bis-GMA, MDP, Colloidal silica, DL-camphorquinone, water, initiators, accelerators and others	Apply on dentin. Leave in place 20 s. Air dry more than 5 s. Light-cure 20 s
Clearfil Protect Bond (Kuraray) Batch # 41130	Two-steps self-etch adhesive	*Primer*: HEMA, MDP, MDPB, Hydrophilic aliphatic dimethacrylate, Water, Initiators, Accelerators, Dyes and others *Bond*: HEMA, sodium fluoride, Bis-GMA, MDP, hydrophobic aliphatic dimethacrylate, colloidal silica, DL-camphorquinone, initiators, accelerators and others	Apply the Primer on dentin. Leave in place 20 s. Air dry. Apply the Bond. Make the Bond film uniform with a gentle air blow. Light-cure 20 s
Experimental i Bond (Heraeus Kulzer) Batch # VP130706 Ep1	One-step self-etch adhesive	Acetone, water, glutaraldehyde, 4-META	Apply on dentin 30 s. Air dry. Light-cure 20 s
Fuji Fil LC (Shade A3) (GC) Batch # 0512161	Resin-modified GIC	*Paste A*: aluminosilicate glass, HEMA, urethanedimethacrylate *Paste B*: distilled water, polyacrylic acid, urethanedimethacrylate, silicone dioxide	Mix Paste A and Paste B 15 s. Dispense in the mold. Light-cure 20 s

Table 1 (continued)

Material	Type	Composition	Mode of use
Fuji II LC improved (Shade A3) (GC) Batch # 05101112	Resin-modified GIC	*Powder*: aluminosilicate glass	Mix powder with liquid no more than 20–25 s
		Liquid: polyacrylic acid, HEMA, proprietary ingredient, 2,2,4, trimethyl hexamethylene dicarbonate	Light-cure 20 s
Fuji IX (GC) Batch # 0512081	Conventional GIC	*Powder*: polyacrylic acid, aluminosilicate glass	Mix powder with liquid 15–20 s
		Liquid: polyacrylic acid, proprietary ingredient	Dispense in the mold
Photac Fil (Shade A3) (3M ESPE) Batch # 238979	Resin-modified GIC	HEMA, polyalkenoic acid, fluoroaluminosilicate glass	Dispense directly in the mold. Light-cure 20 s
Vitremer (Shade A3) (3M ESPE)Batch # 200512272	Resin-modified GIC	Modified polyalkenoic acid, fluoroaluminosilicate glass	Mix powder with liquid. Dispense in the mold. Light-cure 20 s
Ionofil Molar (VOCO) Batch # 580342	Conventional GIC	Polyacrylic acid, fluoride silicate glass	Mix powder with liquid. Dispense in the mold

HEMA 2-hydroxyethyl methacrylate, *MDP* 10-methacryloyloxydecyl dihydrogen phosphate, *Bis-GMA* Bisphenol A diglycidylmethacrylate, *MDPB* 12-methacryloyoxydodecylpyridinium bromide, *4-META* 4-methacryloxyethyltrimellitic anhydride, *BHT* butylated hydroxy toluene

30 mN was kept constant for 5 s, then the force decreased at a constant speed from 30 to 0.4 mN in 20 s and the minimal force of 0.4 mN was kept constant for 5 s. The load and the penetration depth of the indenter (Vicker's pyramid: diamond right pyramid with an angle α=136° between the opposite faces at the vertex) were continuously measured during the load–unload cycle.

The Universal Hardness is defined as the test force divided by the apparent area of the indentation at maximal force. From a multiplicity of measurements stored in a database supplied by the manufacturer, a conversion factor between Universal Hardness and Vicker's hardness (VH) was calculated and implemented into the software, so that the measurements were expressed in Vicker's hardness units.

The indentation modulus was calculated from the slope of the tangent of the indentation curve at maximal force and is comparable with the modulus of elasticity of the material (E).

The total mechanical work (Wtot) was measured during the indentation procedure according to the formula $W = \int F dh$ (F=load; h=indentation depth). The plastic deformation work (Wp) and the work of the elastic reverse deformation (We), which are the two components of the mechanical work, were also measured. The elastic indentation work (We/Wtot) was calculated as the percentage of Wtot represented by We.

By measuring the variation of the indentation depth occurring when the maximal force was kept constant, a relative change of the indentation depth was calculated. This value represented the creep of the material. The creep (Cr) is defined as the ratio between the change in indentation depth measured during the 5 s in that the force of 30 mN was maintained constant and the indentation depth measured at the maximal force of 30 mN.

The above-mentioned mechanical properties had also been measured in enamel and dentin in a preliminary study of the authors, starting from the cuspal tip and performing measurements from enamel to dentin with a distance of 100 μm between each measurement point (Fig. 1 A–D). The resulting mean values of the mechanical properties of enamel and dentin were also compared to those of the two tested materials' classes.

Statistical analysis

The normal distribution of the data and the homogeneity of variances were verified with the Kolmogorov–Smirnov test and the Levene's test, respectively. The mechanical properties were then compared within each materials' class (adhesives and GICs) using the one-way analysis of variance. The data of each mechanical property of all the materials belonging to the same class were pooled together, and a comparison among the average values of the mechanical properties of the two materials' types and those

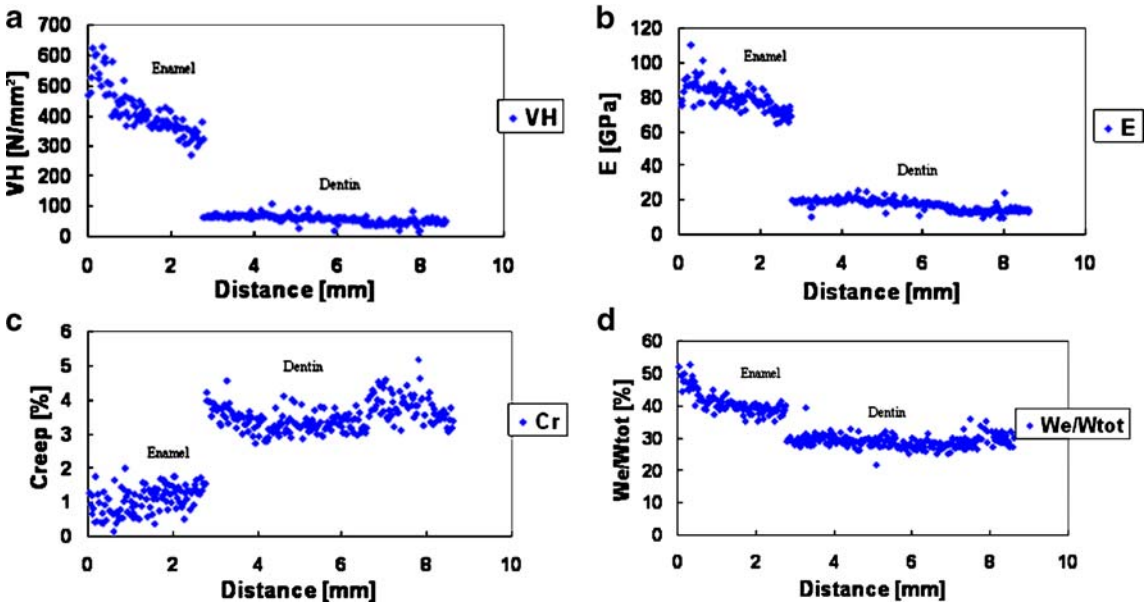

Fig. 1 The graphs represent the variation of **a** the Vicker's hardness (*VH*), **b** elastic modulus (*E*), **c** creep (*Cr*) and **d** elastic indentation work (*We/Wtot*) measured in enamel and dentin as function of the distance from the cuspal tip

of enamel and dentin was performed by means of the one-way analysis of variance. The Tukey HSD test was used for post-hoc multiple comparisons. In all the analyses, the level of significance was set at $p<0.05$. The calculations were handled with SPSS 14.0 software for Windows (SPSS Inc.; Chicago, IL, USA).

Results

Tables 2 and 3 report the descriptive statistics of the mechanical properties of adhesives and GICs respectively. Fig. 2 reports the comparison of the mechanical properties of the materials between the two tested materials' classes and enamel and dentin.

The statistical analysis revealed significant differences in the mechanical properties among the tested adhesives (Table 2). Admira Bond and Clearfil Protect Bond showed comparable VH values ($p>0.05$), which were significantly lower as compared to those of the other tested adhesives ($p<0.05$). No significant differences in VH were detected among Futurabond NR, Solobond Plus, Clearfil SE Bond, Clearfil S^3 Bond and experimental i Bond ($p>0.05$). Hybrid Bond exhibited the highest VH value (25.6±5.6 N/mm^2), which was comparable to those of Solobond Plus, Clearfil S^3 Bond and experimental i Bond ($p>0.05$).

Hybrid Bond showed the highest *E* (5.3±1.0 GPa), though comparable to that of experimental i Bond ($p>0.05$). On the contrary, the lowest *E* value (3.6±0.7 GPa) was shown by Clearfil Protect Bond and it was statistically similar to the *E* of Futurabond NR ($p>0.05$) and significantly lower to the E of all the other tested materials ($p<0.05$). No statistically significant differences in *E* were observed among Admira Bond, Futurabond NR, Clearfil SE Bond and Clearfil S^3 Bond ($p>0.05$). The *E* of Solobond Plus was significantly higher than the *E* of Futurabond NR ($p<0.05$) but similar to those of Admira Bond, Clearfil SE Bond, Clearfil S^3 Bond and experimental i Bond ($p>0.05$).

Concerning the creep, Solobond Plus exhibited a significantly lower value compared to the other tested materials ($p<0.05$). The Cr of Futurabond NR, Hybrid Bond and Clearfil S^3 Bond were statistically similar, as well as the Cr of Admira Bond, Clearfil SE Bond, Clearfil Protect Bond and of experimental i Bond ($p>0.05$). Moreover, Clearfil S^3 Bond showed a comparable creep to those of Clearfil SE Bond and experimental i Bond ($p>0.05$). Futurabond NR and Solobond Plus showed significantly higher values of elastic indentation work than the other tested adhesives ($p<0.05$). The We/Wtot of Hybrid Bond, Clearfil SE Bond, Clearfil S^3 Bond, Clearfil Protect Bond and experimental i Bond resulted statistically comparable ($p>0.05$). Admira Bond presented the lowest elastic indentation work, though similar to those of Hybrid Bond, Clearfil Protect Bond and experimental i Bond.

The statistical analysis showed significant differences in the mechanical properties among the tested GICs ($p<0.05$; Table 3). With respect of the VH, Photac Fil showed the lowest VH, which was comparable to those of Fuji Fil LC and Vitremer, but significantly lower than those of the other three tested GICs ($p<0.05$). The VH of Fuji Fil LC, Vitremer, and Ionofil Molar were statistically similar and significantly lower than those of Fuji II LC and Fuji IX ($p<0.05$), which were significantly higher in comparison to all the other tested GICs ($p<0.05$). The E of Fuji IX was

Table 2 Means (M) and standard deviations (SD) of Vicker's hardness (VH), elastic modulus (*E*), creep (Cr) and elastic indentation work (We/Wtot) of the tested adhesives

Adhesives	VH (N/mm^2)		E (GPa)		Cr (%)		We/Wtot (%)	
	M	SD	M	SD	M	SD	M	SD
Admira Bond	18.7^{A}	4.1	4.5^{BCD}	0.9	5.9^{D}	0.7	35.8^{A}	3.2
Futurabond NR	22.8^{B}	2.7	4.0^{AB}	1.0	5.0^{B}	0.3	43.7^{C}	5.8
Solobond Plus	24.2^{BC}	4.8	4.6^{CD}	1.0	4.5^{A}	0.5	44.0^{C}	5.4
Hybrid Bond	25.6^{C}	5.6	5.3^{E}	1.0	5.2^{B}	0.8	37.5^{AB}	5.3
Clearfil SE Bond	22.1^{B}	6.1	4.3^{BC}	1.0	5.6^{CD}	0.5	39.2^{B}	4.2
Clearfil S^3 Bond	23.9^{BC}	5.9	4.4^{BC}	1.0	5.4^{BC}	1.1	40.1^{B}	7.8
Clearfil Protect Bond	17.2^{A}	2.8	3.6^{A}	0.7	5.8^{D}	0.8	37.4^{AB}	4.8
Experimental i Bond	23.4^{BC}	5.6	5.0^{DE}	1.4	5.6^{CD}	1.0	37.4^{AB}	5.2

Different superscript letters indicate statistically significant differences (Tukey HSD test, $p<0.05$)

significantly higher than those of the other GICs ($p<0.05$), followed by the E of Fuji II LC. Ionofil Molar, Fuji Fil LC and Photac Fil showed statistically homogeneous *E* values ($p>0.05$). The *E* of Vitremer was lower than those of all the other tested GICs, and the differences were significant when the latter was compared to Ionofil Molar, Fuji II LC and Fuji IX ($p<0.05$). Significant differences in the creep were detected only between Fuji IX and, respectively, Vitremer and Fuji Fil LC. The former presented a significantly lower creep ($p<0.05$). The We/Wtot of Fuji IX was significantly lower than that of Fuji II LC and that of Vitremer ($p<0.05$). The latter showed a We/Wtot significantly higher than the other GICs ($p<0.05$).

Finally, when the mechanical properties of the two classes of materials and those of enamel and dentin were compared, statistically significant differences in the *E*, creep and elastic indentation work were detected among adhesives, GICs, dentin and enamel (Fig. 2 A–C). Enamel presented the highest *E* value (Fig. 2A), followed by dentin, GICs and adhesives ($p<0.05$). The creep (Fig. 2B) of enamel was the lowest, followed by dentin, GICs and adhesives ($p<0.05$). Enamel showed also the highest We/Wtot (Fig. 2C), followed by adhesives, GICs and dentin ($p<0.05$). Regarding the VH (Fig. 2D), no significant difference was found between dentin and GICs ($p>0.05$) and both showed significantly higher VH values than the adhesives and significantly lower when compared to enamel ($p<0.05$).

Discussion

The tested adhesives and glass-ionomer cements differed in their mechanical properties both within each materials' class and between the two materials' types. Moreover, the mechanical properties of the two materials' classes differed from those of enamel and dentin. Thus, both null hypotheses were rejected.

Eight adhesives were tested for their mechanical properties in the present investigation. When the study was performed, seven out of eight bonding systems were already on the market, whereas i Bond was still at the experimental stage, but, currently, it was also available. The

Table 3 Means (M) and standard deviations (SD) of Vicker's hardness (VH), elastic modulus (E), creep (Cr) and elastic indentation work (We/Wtot) of the tested GICs

GICs	VH (N/mm^2)		E (GPa)		Cr (%)		We/Wtot (%)	
	M	SD	M	SD	M	SD	M	SD
Fuji Fil LC	48.7^{AB}	13.9	11.3^{AB}	2.2	5.0^{B}	0.9	33.9^{AB}	3.6
Fuji II LC	69.2^{C}	12.6	14.7^{C}	2.2	4.6^{AB}	0.5	35.8^{B}	3.7
Fuji IX	67.9^{C}	11.3	17.2^{D}	3.9	4.4^{A}	0.5	32.4^{A}	4.5
Photac Fil	46.2^{A}	9.0	10.6^{AB}	2.0	4.8^{AB}	0.9	34.3^{AB}	4.2
Vitremer	51.4^{AB}	15.7	9.8^{A}	2.4	4.9^{B}	0.7	39.0^{C}	4.5
Ionofil Molar	57.4^{B}	15.2	12.3^{B}	2.1	4.6^{AB}	0.7	34.8^{AB}	3.3

Different superscript letters indicate statistically significant differences (Tukey HSD test, $p<0.05$)

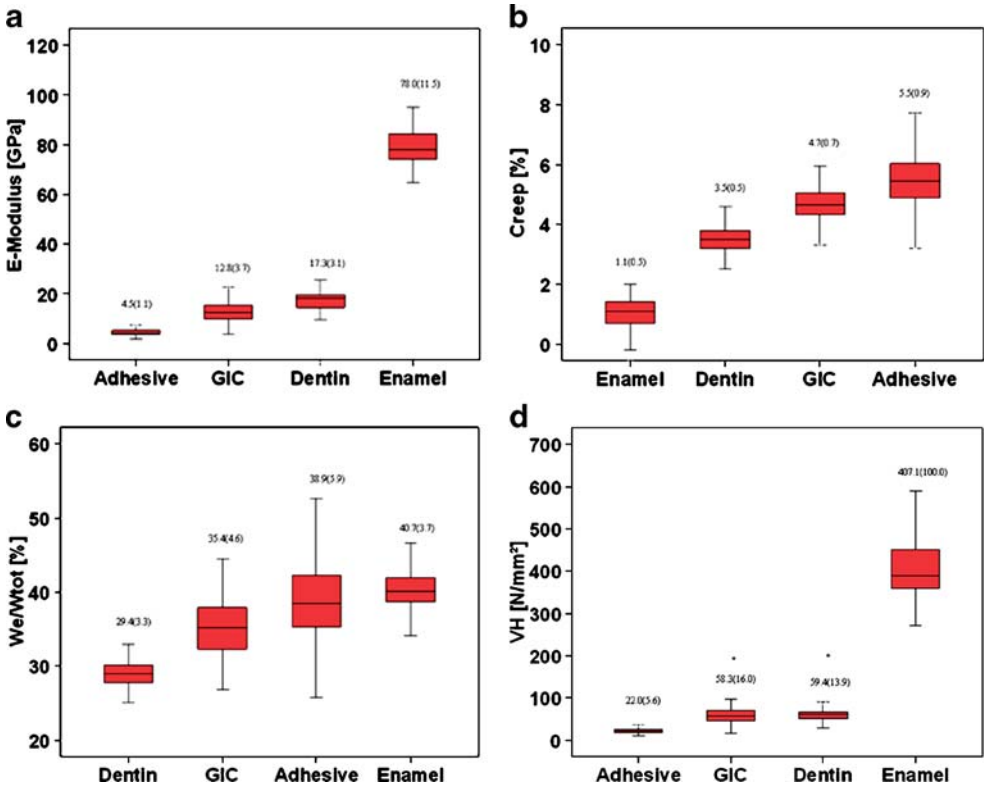

Fig. 2 The graphs represent the comparison among the mechanical properties of the two tested materials' classes and of enamel and dentin. Means (SD) are reported. The *asterisks* indicate statistically similar groups (Tukey HSD test, $p>0.05$)

adhesives differed significantly in all the tested mechanical properties, regardless of the adhesive class.

The Vicker's hardness of Admira Bond and Clearfil Protect Bond was significantly lower compared to those of the other adhesives. Admira Bond is a two-step etch-and-rinse adhesive based on the organically modified ceramic ("ormocer") technology, which combines an inorganic backbone based on silicon dioxide with polymerizable organic units, in order to form three-dimensional compound polymers [23, 24]. This ormocer-based adhesive was developed to be used in combination with the ormocer-based composite Admira. Previous studies reported that the mechanical properties of an ormocer-based composite were comparable to those of other restorative materials [25, 26]. The present investigation showed that the tested ormocer-based adhesive had lower mechanical properties than other tested adhesives. In fact, among the tested adhesives, Admira Bond presented also the lowest We/Wtot value and the highest Cr (which indicates an increase of indentation depth under maximal load). Nevertheless, the absence of filler particles in this ormocer-based adhesive could have contributed to the lower mechanical properties. However, as the restorative system Admira/Admira Bond showed a clinical performance comparable to that of conventional bis-GMA based materials [27], it may be speculated that the mechanical behaviour of the whole ormocer-based restorative system compensates for the slightly lower mechanical properties of the adhesive. Nevertheless, the correlation between the mechanical properties of a material and its clinical performance is still not clear. Clearfil Protect Bond is a two-steps self-etch adhesive, which has antibacterial properties due to the presence of the monomer 12-methacryloyoxydodecylpyridinium bromide (MDPB) in the primer solution [28] and of sodium fluoride in the bond. Interestingly, with the exception of the Cr, Clearfil Protect Bond showed significantly lower mechanical properties than Clearfil SE Bond, which has an analogous chemical composition but does not contain MDPB. Thus, it could be hypothesised that the addition of the antimicrobial, which, on the other side, has been reported not to impair the microtensile bond strength [29], could have lowered the mechanical properties. Although it does not contain fillers, Hybrid Bond showed the highest E and VH among the tested adhesives. This adhesive contains an aromatic amine in the dispensing brush, which additionally activates the chemical polymerization, making this adhesive based on a dual-curing mechanism, which could be responsible for a better curing. The results of this study highlighted that the in vitro mechanical behaviour of dental adhesives does not necessarily reflect their clinical performance. In fact, according to Peumans et al. [18], three-step etch-and-rinse adhesives and two-step self-etch adhesives showed the best clinical performance. On the contrary, less favourable clinical performance was observed for two-step etch-and-rinse adhesives and one-step self-etch adhesives [18],

possibly due to their higher technique sensitivity. Nevertheless, in terms of mechanical properties, this trend was not confirmed. However, it should be considered that the review of Peumans et al. [18] did not include the results of more recent clinical studies [20, 21], which showed an improved clinical performance of new simplified adhesives, like those tested in this study.

Since the mixing procedure does not affect the microhardness and the modulus of elasticity of GICs [17], cements that are available both in capsules and in hand-mixing formulation, the latter was used in this investigation. It was previously reported that the hardness and the modulus of elasticity of the examined RMGICs did not decrease in the subsurface layers of the materials, where the curing light might penetrate with lower intensity [17]. The latter finding indicated that the chemical hardening based on the glass-ionomer reaction still plays an important role for these materials [17], as well as for conventional GICs. Despite their excellent clinical performance [18], resin-modified GICs did not seem to benefit from the addition of methacrylate-based monomers in their chemical composition, if compared to conventional GICs, as far as micromechanical properties are concerned. As a matter of fact, the tested resin-modified GICs did not show consistently superior values of each measured mechanical property than the conventional ones. Nevertheless, in terms of macromechanical properties, such as flexural strength, diametral tensile strength and compressive strength, the resin-modified GICs have been reported to be superior to conventional GICs [17], thus suggesting that the light-cured methacrylate-based polymers improve the first phase of the polymerization of these materials, making them less susceptible to the formation of cracks due to dehydration.

In order to compare adhesives and GICs in terms of mechanical properties, all the measurements performed within each materials' class were pooled and statistically analysed. This way, it was possible to evaluate to what extent each property varied within the materials' classes. Moreover, as the materials involved in a restorative system should ideally have a mechanical behaviour as similar as possible to that of the adjacent tooth structures, the mechanical properties of enamel and dentin were used as references. The GICs had a higher mean E and VH values than adhesives, conversely the mean We/Wtot was lower. The measured E and VH values of the GICs spread within a range which was close to that of the values measured in dentin, whereas adhesive showed values more than 50% lower. When compared to enamel, GICs presented average E and VH almost seven times lower and the discrepancies were much wider for adhesives. The Cr of the adhesives was higher than those of GICs and enamel and dentin, thus suggesting that adhesives presented a lower stability under load. As far as the elastic indentation work is concerned, the adhesives and the GICs presented values, which are intermediate between those of enamel and dentin. The latter finding indicates that under load, both materials' classes showed a higher plastic deformation when compared to enamel, but presented a more elastic behaviour than dentin. These differences could be significant if considering that during functional load the stresses tend to concentrate at the interfaces between structures with different mechanical behaviour, possibly contributing to the loss of integrity of the enamel margins of restorations. However, the GICs establish a chemical bond with calcium ions of hydroxyapatite [6], which could contribute to stabilise the interface between these materials and the tooth structures.

It might be questioned that the direct comparison among the mechanical properties measured in thin adhesives' layers applied on dentin and in 2-mm-thick GICs' specimens could have been inappropriate, since completely different types of specimens have been used for the two materials' classes. Nevertheless, under the loading conditions applied in the present study, the depth of penetration of the micro indenter was limited to a few micrometres under the specimen's surface. Thus, the specimen's thickness, as well as the substrate on which the tested materials have been applied, did not play any role on the outcome of the measurements.

It is still not proved whether the mechanical properties of adhesives and GICs correlate with their clinical performance. However, since fracture and marginal defects have been reported to be principal reasons for restorations' failure [22], the investigation of properties which could explain the behaviour of a material under load could be also of clinical relevance.

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