Acta Odontologica Scandinavica. 2014; 72: 543–548

informa
healthcare

ORIGINAL ARTICLE

Influence of ceramic thickness and type on micromechanical properties of light-cured adhesive bonding agents

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Abstract

Objective. The aim of this study was to evaluate the micromechanical properties of different adhesive bonding agents when polymerized through ceramics. ***Materials and methods.*** Sixty sound extracted human third molars were selected and the crowns were sectioned perpendicular to the long axis in order to obtain dentin slices to be bonded with one of the following adhesives: Syntac/Heliobond (Ivoclar-Vivadent) or Adper-Scotchbond-1XT (3M-ESPE). The adhesives were cured by using a LED-unit (Bluephase®, Ivoclar Vivadent) with three different curing times (10 s, 20 s and 30 s) under two ceramics (IPS-e.max-Press, Ivoclar-Vivadent; IPS-Empress®CAD, Ivoclar-Vivadent) of different thicknesses (0 mm, 0.75 mm, 2 mm). Thirty groups were included, each containing 60 measurements. Micromechanical properties (Hardness, HV; indentation modulus, E; and creep, Cr) of the adhesives were measured with an automatic microhardness indenter (Fisherscope H100C, Germany). Data were statistically analyzed by using one-way ANOVA and Tukey's post-hoc test, as well as a multivariate analysis to test the influence of the study parameters (SPSS 18.0). ***Results.*** Significant differences were observed between the micromechanical properties of the adhesives ($p < 0.05$). The ceramic type showed the highest effect on HV (Partial-eta squared (η^2) = 0.109) of the tested adhesives, while E ($\eta^2 = 0.275$) and Cr ($\eta^2 = 0.194$) were stronger influenced by the adhesive type. Ceramic thickness showed no effect on the E and Cr of the adhesives. ***Conclusions.*** The adhesive bonding agents used in this study performed well by curing through different thicknesses of ceramics. The micromechanical properties of the adhesives were determined by the adhesive type and were less influenced by ceramic type and curing time.

Key Words: *adhesive bonding agents, ceramic veneers, hardness, indentation modulus, micromechanical properties*

Introduction

Increasing patient demand for esthetic tooth-colored restorations caused development of the new esthetic materials and adhesives with higher mechanical and esthetic properties [1,2]. Improvements in dental ceramic systems have made indirect restorative systems, especially such as porcelain laminate veneers (PLVs), a favorable and popular clinical treatment option [1,3]. Despite improvements in the adhesive systems, the bonded interface between the tooth surface and the restoration may remain questionable [4].

Long-term clinical success of a ceramic restoration mainly depended on the durability and stability of the adhesion complex formed between the four different components of the tooth surface, the bonding agent, the resin cement and the ceramic surface [3,4]. The interfacial adhesion structure at the tooth/ceramic interfaces is composed of both the adhesive bonding agent and the resin cement [5]. Therefore, besides resin cements, the adhesive bonding agents also play an important role for the longevity of a ceramic restoration.

The interface between a ceramic restoration and tooth is exposed to stresses due to polymerization shrinkage and/or functional occlusal loads [6]. Moreover, the distribution of the interface stress is not uniform [7,8]. Therefore, the interfacial adhesion structure should form an elastic intermediate layer able to absorb the interfacial stresses between two different structures of tooth and porcelain [6,9]. Besides resin cements, adhesive bonding agents could also be used for this purpose.

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(Received 19 September 2013; accepted 8 December 2013)

ISSN 0001-6357 print/ISSN 1502-3850 online
DOI: 10.3109/00016357.2013.876661

The success of a porcelain veneer restoration is mainly based on a high bond strength between the ceramic and the dental hard tissues [1,2]. In order to obtain high bond strength, an optimal curing of the adhesives is required [10]. Furthermore, the degree of a polymerization of the resin cement and bonding agent affects the mechanical properties of the adhesives and, thus, the survival of the restoration [11].

Several *in vitro* studies quantified a considerable light attenuation by polymerizing the resin cements through ceramics [1,12,13]. However, the information on the micromechanical properties of the adhesive bonding agents which are involved in the adhesion complex of PLVs, when cured especially under the ceramics, is limited in the dental literature. Therefore, the aim of this study was to evaluate the micromechanical properties of the adhesive bonding agents for cementation of the PLVs when polymerized through ceramics by assessing the influence of parameters like adhesive type, curing time, ceramic type and ceramic thickness.

Materials and methods

A filled (Adper-Scottchbond-1XT; 3M ESPE, Seefeld, Germany) and an unfilled (Heliobond/Syntac; Ivoclar Vivadent, Schaan, Liechtenstein) light-cured adhesive bonding agent were selected for this study. The descriptions of all the materials included in this study are summarized in Table I.

Sixty sound freshly extracted human third molars were collected. Any residual soft tissue was removed from the tooth surface with a hand scaler. The teeth were disinfected in 10% formol solution for 14 days and stored in distilled water at room temperature until they were used for the study. The superficial enamel was removed from the occlusal aspect of the teeth by grinding with a wet 120-grit silicon carbide paper disk (Leco® VP 100, Leco Instrumente GmbH, Mönchengladbach, Germany). The crowns were sectioned perpendicular to the long axis in order to obtain three 1-mm-thick slices from each tooth (IsoMet® Low Speed Saw, Buehler®, Lake Bluff, IL, USA). The peripheral enamel was removed with wet 120-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. Since the adhesives are based on the etch-and-rinse technique, the dentin was previously etched with 37% phosphoric acid gel (Total Etch; Ivoclar Vivadent) for 15 s, abundantly rinsed with deionized water and air-dried.

A lithium disilicate glass-ceramic (IPS e.max Press; Ivoclar Vivadent) and a leucite reinforced glass-ceramic (IPS Empress®CAD; Ivoclar Vivadent) were selected for this study. For the IPS e.max Press ceramic wax patterns of 0.75 mm and 2 mm in thickness and 10 mm in diameter were prepared, invested in Starvest®-SOFT-3 investment (Weber Dental, Stuttgart, Germany) and burnout in a furnace (Type CL-V2; Heraus Kulzer, Hanau, Germany) at the temperatures of 800°C for 60 min, 600°C for 30 min and 850°C for 60 min, respectively. The investment and an ingot of IPS e.max Press were then transferred to the furnace (EP 500; IPS Empress, Ivoclar Vivadent) and automatically pressed with program 16 (930°C, 60 min). Similar discs were prepared from the IPS Empress®CAD ceramic blocs by cutting with a low speed saw (Isomet® Low Speed Saw, Buehler®). All ceramic discs were then ground with silicon carbide paper of grit 600.

Bonding agents were dropped onto the dentin disks as two coats according to the manufacturers' instructions. As control groups, the bonding agents were cured directly without using any ceramic for three

Table I. Materials used in this study.

Brand name	Manufacturer	Composition	Remarks	LOT number
Adper Scotchbond 1-XT Adhesive	3M ESPE, Seefeld, Germany	Dimethacrylates, HEMA, polyalkenoid acid copolymer, 5 nm silane treated colloidal silica, ethanol, water, photoinitiator	Two-step etch&rinse light-curing adhesive	9UY
Syntac-Heliobond	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, TEGDMA, catalysts, stabilizers	Two-step etch&rinse light-curing adhesive	L24292
IPS e.max Press	Ivoclar Vivadent, Schaan, Liechtenstein	SiO_2, Li_2O K_2O, P_2O_5 ZrO_2, ZnO other oxides color oxides	Lithium disilicate glass-ceramic	M13076
IPS Empress®CAD	Ivoclar Vivadent, Schaan, Liechtenstein	SiO_2 Al_2O_3 K_2O, Na_2O other oxides pigments	Leucite reinforced glass-ceramic	M02654

According to manufacturers' information.

different times of 10, 20 and 30 s with a LED-unit (Bluephase®, Ivoclar Vivadent, 1200 mW/cm^2). The rest of the specimens were cured under the ceramics of two different thicknesses (0.75 mm and 2 mm) of the selected ceramics for three different curing times (10, 20 and 30 s) with the same LED-unit. The combination of all parameters gives a total of 30 groups ($n = 6$). In order to avoid oxygen-inhibition during polymerization, mylar strips were positioned over the bonding agents before the curing procedure. The curing unit was directly centered on the sample surface to maintain the maximum energy of light onto the surface of measurement. The specimens were stored after curing for 24 h at 37°C by maintaining moisture conditions with distilled water.

Vickers hardness (HV), indentation modulus (E) and creep (Cr) were superficially measured by using an automatic micro-hardness indenter (Fischerscope H100C, Fischer, Sindelfingen, Germany). For each specimen, 10 indentation points were selected on the top surface of the cured bonding layer and, thus, 60 measurements were made per group. The test procedure was carried out force-controlled. The test load increased and decreased with constant speed between 0.4–30 mN. The load and the penetration depth of the indenter were continuously measured during the load–unload-hysteresis.

The Universal hardness was defined as the test force divided by the apparent area of the indentation under the applied test force. From a multiplicity of measurements stored in a database supplied by the manufacturer, a conversion factor between Universal hardness and HV was calculated and implemented into the software so that the measurement results were indicated in the more familiar Vickers hardness units. The indentation modulus was calculated from the slope of the tangent of indentation depth-curve at maximum force and is comparable with the E. By measuring the change in indentation depth for 5 s with a constant test force of 30 mN, a relative change in the indentation depth was calculated. This was a value for the creep of the materials.

Results were statistically analyzed using one-way ANOVA and Tukey HSD post-hoc test (PASW Statistics for Windows, version 18.0; SPSS Inc., Chicago, IL) ($\alpha = 0.05$), as well as a multivariate analysis (general linear model) to test the influence of parameters like adhesive type, curing time, ceramic type and ceramic thickness on the micromechanical properties of adhesives.

Results

As a function of tested parameters, which are adhesive type, ceramic thickness and type, and curing time, the descriptive statistics of the micromechanical properties of Adper Scothbond 1-XT (AS) and Heliobond (HB) adhesive bonding agents are shown in Tables II and III, respectively. The statistical analysis revealed significant differences in the mechanical properties among the tested adhesives (Tables II and III) ($p < 0.05$).

For AS bonding agent, light curing for 10 s generally exhibited lower HV and E and higher Cr values than the groups cured for 20 and 30 s (Table II). The highest E values were observed when this adhesive was cured under e.max Press ceramics 0.75 and 2 mm in thicknesses for 20 s. AS exhibited the highest HV

Table II. Indentation modulus (E), Vickers hardness (HV) and creep (Cr) values of the Adper-Scotchbond-1XT adhesive bonding agent.

Adper-Scotchbond-1XT	Ceramic thickness (mm)	Curing time (s)	E (Mean ± SD)	HV (Mean ± SD)	Cr (Mean ± SD)
Reference	0	10	4.0 ± 0.8^{b}	20.0 ± 5.2^{cde}	6.2 ± 0.9^{fg}
		20	4.7 ± 1.1^{de}	28.0 ± 10^{gh}	4.9 ± 0.9^{ab}
		30	4.8 ± 0.9^{de}	27.5 ± 11.7^{gh}	5.2 ± 1.1^{abcd}
IPS Empress®CAD	0.75	10	2.1 ± 0.8^{a}	8.1 ± 15.1^{a}	6.5 ± 1.4^{g}
		20	4.8 ± 0.7^{def}	15.4 ± 1.8^{bc}	6.1 ± 0.5^{fg}
		30	4.3 ± 1.0^{bcd}	18.0 ± 2.8^{cd}	6.3 ± 0.6^{fg}
	2	10	4.3 ± 1.0^{bcd}	11.1 ± 3.4^{ab}	5.4 ± 0.7^{bcde}
		20	4.0 ± 0.9^{bc}	22.8 ± 9.2^{defg}	5.5 ± 0.9^{cde}
		30	4.6 ± 1.6^{cde}	19.8 ± 10.5^{cde}	5.8 ± 0.9^{ef}
IPS e.max Press	0.75	10	4.5 ± 0.9^{bcde}	26.4 ± 17^{fgh}	5.6 ± 0.9^{de}
		20	5.4 ± 1.3^{fg}	31.5 ± 9.9^{h}	4.8 ± 0.7^{ab}
		30	5.1 ± 0.7^{efg}	24.5 ± 5.8^{efg}	5.5 ± 0.7^{cde}
	2	10	4.2 ± 0.7^{bcd}	21.3 ± 7.6^{def}	5.3 ± 1.0^{abcde}
		20	5.5 ± 1.1^{g}	26.8 ± 7.4^{fgh}	5.0 ± 0.8^{ab}
		30	4.7 ± 0.9^{de}	27.4 ± 6.6^{gh}	4.8 ± 0.8^{a}

Superscript letters show statistically homogeneous sub-groups ($p > 0.05$).

Table III. Indentation modulus (E), Vickers hardness (HV) and creep (Cr) values of the Syntac-Heliobond adhesive bonding agent.

Syntac-Heliobond	Ceramic thickness (mm)	Curing time (s)	E (Mean ± SD)	HV (Mean ± SD)	Cr (Mean ± SD)
Reference	0	10	3.9 ± 0.1^{fg}	20.1 ± 1.7^{defg}	4.8 ± 0.3^{bcd}
		20	3.5 ± 0.4^{cde}	24.0 ± 4.1^{h}	4.2 ± 0.5^{a}
		30	4.0 ± 0.3^{g}	22.2 ± 3.3^{gh}	4.5 ± 0.2^{b}
IPS Empress®CAD	0.75	10	3.5 ± 0.5^{cde}	17.7 ± 3.4^{cd}	4.8 ± 0.5^{cd}
		20	3.7 ± 0.4^{ef}	19.1 ± 3.6^{de}	4.7 ± 0.6^{bcd}
		30	4.0 ± 0.3^{g}	21.5 ± 3.9^{fg}	4.7 ± 0.4^{bcd}
	2	10	3.2 ± 0.3^{abc}	13.8 ± 2.0^{a}	5.4 ± 0.2^{ef}
		20	3.1 ± 0.6^{ab}	15.2 ± 1.6^{ab}	5.2 ± 0.3^{e}
		30	3.6 ± 0.3^{def}	18.1 ± 3.0^{cd}	4.9 ± 0.5^{d}
IPS e.max Press	0.75	10	3.0 ± 0.7^{a}	17.8 ± 2.0^{cd}	4.8 ± 0.3^{cd}
		20	3.7 ± 0.2^{fg}	19.5 ± 5.6^{def}	4.8 ± 0.5^{bcd}
		30	3.4 ± 0.6^{c}	21.3 ± 8.6^{efg}	4.6 ± 0.8^{bc}
	2	10	3.1 ± 0.3^{ab}	13.6 ± 2.6^{a}	5.6 ± 0.3^{f}
		20	3.4 ± 0.4^{cd}	16.5 ± 1.4^{bc}	5.2 ± 0.2^{e}
		30	3.3 ± 0.4^{bc}	20.0 ± 4.4^{defg}	4.7 ± 0.4^{bcd}

Superscript letters show statistically homogeneous sub-groups ($p > 0.05$).

when cured under 0.75 in thickness of e.max Press ceramic. The lowest Cr values were obtained when AS was cured for 20 s through 0.75 mm and for 30 s through 2 mm e.max ceramics (Table II).

Table III reports the descriptive statistics of the micromechanical properties of HB which presented lower values than AS. Curing for 30 s of both reference and 0.75 mm IPS-CAD groups exhibited the highest E scores for HB bonding agent. Reference groups of 20 and 30 s curing times also showed higher HV and lower Cr values than other groups (Table III).

Table IV presents the level of the effect of different ceramic types, ceramic thicknesses and curing times on the micromechanical properties of the adhesive bonding agents by showing the partial eta-squared values (η^2). The adhesive type exhibited the highest effect on the micromechanical properties of the adhesive bonding agents (η^2-E = 0.275, η^2-HV = 0.046, η^2-Cr = 0.194) followed by curing time (η^2-E = 0.106, η^2-HV = 0.095, η^2-Cr = 0.073) and ceramic type (η^2-E = 0.037, η^2-HV = 0.109, η^2-Cr = 0.058) ($p < 0.05$). Ceramic thickness showed a very low effect on HV (η^2-HV = 0.006) and no effect on E and Cr.

Discussion

The present study analyzed the micromechanical properties of two different adhesive bonding agents for the cementation of porcelain laminate veneer restorations to evaluate whether these adhesives perform well when polymerized under ceramics by considering the effect of adhesive type, curing time, ceramic type and ceramic thickness. All these effects were expressed in terms of E, HV, and Cr measured on thin adhesive films.

Clinically, PLVs are cured finally when the veneers are completely positioned after the application of both the resin cement and the adhesive bonding agent to the adhesion surfaces [14]. As the ceramic absorbs a certain percentage of the emitted light, an adequate curing of the adhesive systems under ceramic restorations is essential for the success of restorations [14,15]. Therefore, as well as for the resin cements, whether there is sufficient curing for the adhesive bonding agents is an important issue.

Table IV. Effects of study parameters on the micromechanical properties of the adhesive bonding agents.

	Eta-squared values		
Parameters	E	HV	Cr
Adhesive	0.275	0.046	0.194
Time	0.106	0.095	0.073
Ceramic	0.037	0.109	0.058
Thickness	—*	0.006	—*
Ceramic & Adhesive	0.093	0.091	0.057
Ceramic & Thickness	0.006	0.008	0.006
Ceramic & Time	0.026	0.007	0.009
Adhesive & Thickness	0.018	0.018	0.086
Adhesive & Time	0.060	0.021	0.012
Thickness & Time	0.043	0.004	0.007

*Statistically no significant effect ($p > 0.05$).
The higher the eta-squared value, the stronger the effect of study parameters on the measured micromechanical properties.

The mechanical properties of a material can be a factor of influence when the material is in clinical service [16]. Surface hardness is a parameter frequently used to evaluate material surface resistance to plastic deformation by indentation [17]. The Vickers hardness (HV) value in this study is the result of the described measurement procedure by applying a load of 0.4–30 mN and measuring simultaneously the indentation depth. The indentation modulus was calculated from the slope of the tangent of indentation depth-curve at maximum force. When a material is subjected to a constant load (in this study 30 mN for 5 s), it generally expresses a time-dependent increase in strain. This phenomenon is known as creep (Cr), a term which is used to describe the tendency of a solid material to slowly deform permanently to relieve stresses. Therefore, an ideal adhesive should exhibit increased HV and E values as well as decreased Cr values [1].

The adhesives may wear at the tooth–restoration interfaces resulting in marginal gap formation due to the accumulation of interfacial stresses [18]. Wear resistance, as well as bonding effectiveness, durability and longevity of the bonding agents depends on their mixture of ingredients [18,19]. An unbalanced composition of ingredients may lead to reduced physical and mechanical characteristics of the dental adhesives [19]. Therefore, the chemical composition of an adhesive bonding agent can influence the clinical outcome of the restoration.

Two different adhesive bonding agents were employed in this study. They were a filled adhesive (Adper Scothbond 1-XT) and an unfilled adhesive (Heliobond). In the present study, the filled adhesive exhibited higher E and HV values than the unfilled adhesive, whereas unfilled adhesive showed lower Cr values than the filled adhesive. The filled adhesive contains 10% by weight of 5 nm-diameter spherical silica particles, which could be responsible for higher mechanical properties. However, the relevance of the strengthening effect of the filler in adhesives has been reported to be controversial, especially because only small concentrations of fillers are added to their composition [20].

The partial eta-squared is defined as the proportion of total variation of the dependent variable attributable to a factor, excluding other factors from the total non-error variation. It represents an index of strength of association between an experimental factor and the dependent variable and ranges normally between 0–1 [21,22]. In the present study, the adhesive type showed the highest influence on the investigated properties, while there was no effect of ceramic thickness on E and Cr according to the partial eta-squared values derived from multivariate analysis (Table IV). Furthermore, ceramic thickness showed a considerably low effect on HV of the bonding agents when compared with the other factors. This result shows that adhesives could be polymerized sufficiently even under the thick (2 mm for this study) ceramic restorations. However, the depth of polymerization may vary depending on the type of the selected adhesive. In addition, when the ceramic thickness is considered with the other factors, especially such as time, it shows a slight effect on the micromechanical properties of the adhesives. Therefore, it can be considered to increase the curing time under the thick ceramic restorations.

The effect of ceramics on the mechanical properties of resin cements has been investigated in many *in vitro* [23–25] studies. Mechanical properties of dental adhesives have also been examined in various studies [18,26–28] with different methods. Doucet et al. [29] studied on the adhesion strength between a dental ceramic and an adhesive by using the Vickers indenter test and found a significant relation between the Vickers hardness and the adhesion strength. However, until now, no effort has been made on assessing the micromechanical properties of adhesive bonding agents when cured, especially under the dental ceramics according to the authors knowledge.

The results of the current study showed that the type of ceramics influences the micromechanical properties of the underlying adhesive bonding agent (Table IV). The two ceramic types used in this study have different microstructures. In the lithium disilicate glass ceramic, the main crystalline phase consists of elongated lithium disilicate ($Li_2Si_2O_5$) crystals building a scaffold of many small interlocking needle-like crystals that are randomly oriented [2,30]. A second crystalline phase, consisting of a lithium orthophosphate (Li_3PO_4) of a much lower volume, was also reported [31]. On the other hand, the microstructure of the leucite-reinforced glass-ceramic is less dense and is characterized by single crystal formation of leucite crystals, $KAlSi_2O_6$, without interlocking between crystals [2,31].

The present study demonstrates that adhesive type, curing time and ceramic type influences the micromechanical properties of the selected adhesives. Therefore, these factors can play a role in the clinical success of the ceramic restorations. However, the present study is limited by including two ceramics, two adhesives and just three exposure times. Furthermore, no long-term measurement was performed by assessing the behaviour of the tested materials after aging. Therefore, further research is needed to investigate other possible factors affecting the mechanical behavior of dental adhesives.

Conclusions

Within the limitations of this study, the following conclusions can be addressed:

(1) The filled bonding agent exhibits higher micromechanical properties than the unfilled adhesive bonding agent.

(2) Adhesive type, curing time and ceramic type have a significant effect on the micromechanical properties of adhesive bonding agents.
(3) Ceramic thickness up to 2 mm is not an effective factor on the micromechanical properties of the adhesive bonding agents.

Acknowledgments

This investigation was conducted in the Dental School of the Ludwig-Maximilians University, which is a collaborating research laboratory located in the Continental European Division of the International Association for Dental Research area, by winning of the IADR.CED Visiting Scholar Stipend.

***Declaration of interest*:** The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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