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ORIGINAL ARTICLE
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Influence of drying time of adhesive systems on the bond strength between resin cement and feldspathic ceramic

Influência do tempo de secagem de sistemas adesivos na resistência de união entre cimento resinoso e cerâmica feldspática

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ABSTRACT

Objective: This study evaluated the effect of drying times of two total-etch & rinse adhesives on the resin bond strength to a feldsphatic ceramic, before and after aging. **Material and Methods:** Feldsphatic-ceramic CAD-CAM bars were cut into blocks (12×10×4 mm) with a cutting machine (N = 32). Impressions were made of each ceramic block with silicone putty material and the negative space was filled with a composite resin. The bonding ceramic surface was etched with hydrofluoric acid, silanized, and the adhesive system (SB- Single Bond 2, 3M-ESPE; or PB- Prime & Bond NT, Dentsply) was applied. The samples were dried at different times (5, 10 and 15 s) before the cementation. The resin and ceramic blocks were cemented by a dual cure resin cement. All samples were stored in distilled water at 37 °C for 24 h. For the μ-TBS test, the samples were sliced into microbars. Half of the bars of each block was tested after 24 h and, the other bars were submitted to thermocycling (12,000×) and water storage (150 d). For the 24 h groups, the longer drying time increased ($p < 0.05$) the bond strength of SB (water/alcohol adhesive), while reduced ($p < 0.05$) for the PB group (acetone based adhesive). **Results:** For the aged groups, the bond strength for the different drying times had no significant difference, for the

RESUMO

Objetivo: Este estudo se propôs avaliar o efeito de diferentes tempos de secagem de dois adesivos tipo 'total-etch & rinse' e ciclagem térmica na resistência adesiva entre cerâmica feldspatica e cimento resinoso. **Material e Métodos:** Trinta e dois blocos (12×10×4 mm) de cerâmica feldspática e respectivos blocos (32) de resina composta foram obtidos. A superfície de cimentação de cada bloco cerâmico foi condicionada por ácido fluorídrico (HF), silanizada (S), e recebeu a aplicação de um dos dois sistemas adesivos testados (SB - Single Bond 2, 3M-ESPE; ou PB – Prime & Bond NT, Dentsply). Então a superfície com o adesivo foi seca em diferentes tempos (5, 10 e 15s) antes da cimentação. Após, um cimento resinoso foi aplicado sobre a superfície de cimentação e o bloco correspondente de resina foi cimentado. Os palitos para microtração foram obtidos, sendo que metade deles foram imediatamente testados, enquanto os demais foram submetidos à ciclagem térmica e armazenagem (150 dias). Para os grupos sem envelhecimento, o tempo de secagem mais longo (15s) aumentou a resistência para o adesivo SB, enquanto que reduziu a resistência para o adesivo PB. **Resultados:** Para os grupos envelhecidos, as resistências adesivas dos diferentes tempos de secagem não foram diferentes estatisticamente, independente do adesivo. A falha predominante foi coesiva do cimento resinoso, seguido pela falha na interface cerâmica cimento. **Conclusão:** Concluiboth adhesives. **Conclusion:** Longer drying times increased the bond strength values of SB. Smaller drying times increased the bond strength values of PB. The aging protocol influenced the bond strength of SB groups.

se que tempos mais longos de secagem podem melhorar a resistência adesiva à cerâmica testada, usando o adesivo SB. Por outro lado, tempos mais curtos podem otimizar a adesão para o adesivo PB. O envelhecimento afetou a adesão somente nos grupos do adesivo SB.

KEYWORDS

Microtensile; Adhesion; Feldspar ceramic; Drying time; Adhesive system.

PALAVRAS-CHAVE

Microtração; Adesão; Cerâmica de feldspato; Tempo de secagem; Sistema adesivo.

INTRODUCTION

Acid-sensitive ceramics (glass-based) are well-known to be dependent on a properly bond interface between teeth and restoration [1,2]. As a result, the use of resin cement are the better option when compared to other cements such as zinc phosphate and glass ionomer [3]. In order to improve the bond strength between resin cement and glass-based ceramic, the ceramic surface must be etched with hydrofluoric acid (HF), silanated, followed by an adhesive system application [4,5].

The main components in most dental resin adhesives are hydrophobic adhesive monomers, hydrophilic monomers (to improve the wettability of hydrophobic monomers on the demineralized matrix), organic solvents such as acetone, ethanol or water, initiators such as canforoquinone and fillers particles [6-10]. Among the composition, the solvent plays an important role, by transporting the monomers into the treated surface (enamel and/or dentin). After the application, the adhesive layer should receive a gently air to evaporate the solvents (following the manufacturer's instructions for each type of adhesive). The objective in 'drying' the adhesive agent is to avoid the deleterious effect caused by the amount of residual solvent that can inhibit the resin polymerization and have a significant negative effect on the mechanical properties of the adhesives [6,11].

Different studies showed that adhesives with acetone as a solvent are more sensitive to volatilization when compared to ethanol adhesive based solvent [6,11,12]. Acetone is more volatile because it has a boiling point of 25ºC of 200 mmHg compared to 54.1 mmHg of ethanol [9,13]. Thus, it is important to evaluate the protocols of different solvents to increase volatilization and perhaps improve the bond effectiveness of these materials.

Resin adhesive systems are used during many restorative treatments, however are concerning about the effect of during bonding procedures. As a result, this study aimed to evaluate the effect of different drying times of two adhesive systems composed by different organic solvents on the bond strength between feldspathic ceramic and resin cement, after aging. The tested hypotheses were: (1) different drying times will not influence the bond strength values; (2) aging protocol will decrease the bond strength values, independent the adhesive system or drying time.

MATERIALS AND METHODS

The material, brand names, main compositions and manufacturer of the products used in the current study are presented in Table 1.

Specimen Preparation and Bonding Procedures

Sixteen blocks (12 × 10 × 4 mm) of feldspathic ceramic were obtained from

Table 1 - Brand name, manufacturer and composition of the materials used in the study

Material	Manufacturer	Composition
Vita block Mark II	Vita, Bad Säckingen, Germany	Mixture of feldspathic crystalline particles embedded in a glassy matrix
Ceramic conditioner (hydrofluoric acid at 10%)	Dentsply	HF 10%
Monobond S (silane)	Ivoclar Vivadent	Alcohol solution of silane metacrylate
Single Bond 2 (adhesive system)	3M ESPE	Water, ethanol,HEMA, Bis-GMA, dimethacrylates,initiator, metacrylate functional copolymer of polyacrylic acids and silica nanofillers
Prime & Bond NT (adhesive system)	Dentsply	Di- and Trimethacrylate resins, PENTA: dipentaerythritol penta acrylate monophosphate, Nanofillers-Amorphous Silicon Dioxide, Photoinitiators, Stabilizers, Cetylamine hydrofluoride, Acetone
Variolink II (resin cement)	Ivoclar Vivadent	The monomer matrix: Bis-GMA: bisphenol A diglycidyl methacrylate, Urethane, Dimethacrylate, Triethylene glycol dimethacrylate The inorganic fillers: Barium glass, Ytterbium trifluoride, Ba-Al-fluorosilicate glass, Spheroid mixed oxide.
Filtek z350 (resin-matrix composite)	3M ESPE	BIS-GMA: bisphenol A diglycidyl methacrylate, BIS-EMA: ethoxylated bisphenol A glycol, dimethacrylate, UDMA: urethane dimethacrylate or 1,6-di(methacryloyloxyethylcarbamoyl)-3,30,5-trimethylhexaan, TEGDMA: triethylene glycol dimethacrylate

VitaMark II blocks (Vita Zahnfabrik, Bad Sackingen, Germany). Impressions were made of each ceramic block with addition silicon putty material (Aquasil, Dentsply, York, PN, USA). The negative space obtained in the impression material received two millimeters increments of resin-matrix composite, which were photo activated during 40 s each (Radii Cal, SDI, Australia), producing resin-matrix composite blocks identically to ceramic blocks. The ceramic blocks were ultrasonically cleaned during 5 min in distillated water and randomly divided according the adhesive system and the drying time (Table 2).

The ceramic surface was etched with 10% hydrofluoric acid gel (HF) for 60 s (Porcelain conditioner, Dentsply, Rio de Janeiro, RJ, Brazil), rinsed with water (60 s) and air dried during 60 s using compressed air. The silane coupling agent (Monobond S, Ivoclar Vivadent, Schaan, Liechtenstein) was applied with a microbrush and, after 60 s, dried for 5 s using compressed air. One standardized layer of the respective

Table 2 - Experimental design of the study

Adhesive system	Drying time (seconds)	Condition
Single Bond 2	5	Non-aging
		Aging
	10	Non-aging
		Aging
	15	Non-aging
		Aging
Prime & Bond NT	5	Non-aging
		Aging
	10	Non-aging
		Aging
	15	Non-aging
		Aging

resin adhesive system, either Single Bond 2 (SB) or Prime & Bond NT (PB), was applied during 5 s with microbrush and allowed to evaporate in accordance with the specific time of each group (5, 10 and 15 s) and photo activated for 20 s (Radii Cal, SDI). After that, the resin-matrix composite block was cemented on the ceramic block with a dual cure resin cement (Variolink II, Ivoclar Vivadent) using a vertical load of 750 g. The excess of cement was removed and each side of the ceramic/composite resin block was photo activated for 40 s (Radii Cal, SDI).

Cutting Procedure

The ceramic/composite resin blocks were sectioned in the x- and y-axes using a diamond disc at low speed under water-coolant on a sectioning machine (Isomet® 1000, Buehler). The resulting beams had a cross-sectional bonded area of 1 mm2 and non-trimmed interface. The beams obtained from each ceramic/composite resin block were randomly divided into two testing conditions. The non-aged specimens were submitted to the microtensile bond strength test (μ-TBS) after 24 h, while the aged specimens were submitted to aging before the test.

Aging Method

Half of the beams of each block were submitted to 12,000 × thermal cycles (5ºC to 55ºC, baths of 30 s and transfer time of 2 s) (for approximately 8.6 days) and stored in distilled water at 37 ºC during 150 days before mechanical test.

Microtensile Bond Strength Test (μ-TBS)

For the μ-TBS, the beams were attached to a metallic device with cyanoacrylate gel glue (Suprabond gel; Suprabond; Brazil), without touching the adhesive interface, and the μ-TBS was carried out in a universal testing machine at crosshead speed of 0.5 mm/min (Emic DL-2000, Emic, São José dos Pinhais, Brazil).

Failure mode evaluation

The specimens were analyzed under optical microscopy (Mitutoyo Microscope MFA – Series 5051H; Kyoto, Japan) at 50-100 × magnification. Some specimens of each group were selected to obtain scanning electron microscope (SEM) images (FEI Company, Inspect S50, USA). The failure types were classified as follows: adhesive between ceramic and cement (Adhes Cc); adhesive between cement and composite resin (Adhes Rc); cohesive of ceramic (Cohes C); cohesive of cement (Cohes c); cohesive of composite resin (Cohes R).

Data analysis

The block was considered as the experimental unit (the data was an average of the microbeams per block in each condition). The bond strength results of non-aging groups and aging groups were separately evaluated by One-way ANOVA and Tukey test ($\alpha = 0.05$). To compare the effect of the aging (bond durability) for each drying time of the adhesive (pair-comparison), the t-Student test ($\alpha = 0.05$) was applied. Specimens, which failed before the test (pre-test failures) and with cohesive failures, were not included on the statistical analysis.

RESULTS

The failure mode of the beams, including pre-test and post-test, are present in table 3. The pre-test failures were predominantly adhesive between resin cement and ceramic, while post-test failures were predominantly cohesive in the resin cement (Figure 1).

Table 4 shows the mean and standard deviations of the μ-TBS for the aged and non-aged groups of SB and PB adhesive systems, as well as the respective statistical analysis. Considering the immediate non-aged condition, higher bond strength was observed with longer drying time for the Single Bond adhesive (water/alcohol adhesive). Lower values of bond strength was associated to longer drying time for PB adhesive (acetone-based adhesive). For the aged condition, the bond strengths for different drying times were not statistically different, irrespective of the adhesive type.

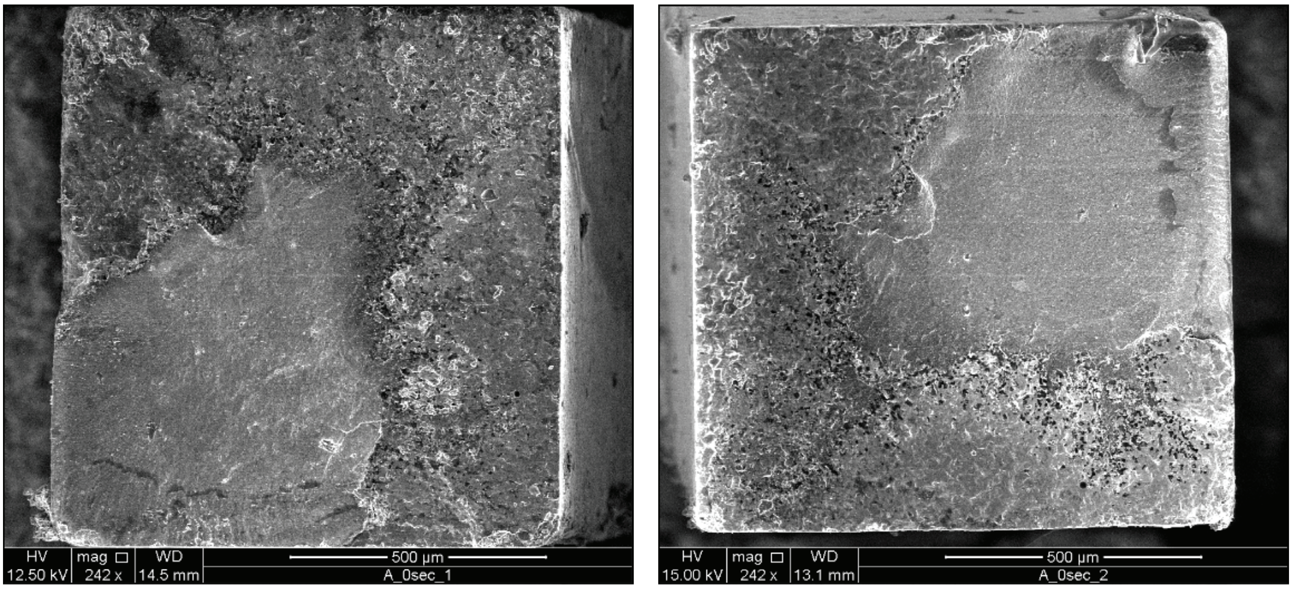

Figure 1 - Representative SEM micrograph of the ceramic and composite resin surfaces after the test. The failure mode was classified as cohesive failure of the resin cement, since it is possible to observe resin cement in both half's of the specimen.

Table 3 - Rate of failure modes of each group

Adhesive system	Aging	Drying time (s)	Mode of failure*					Pre-test failure	Specimens tested
			Adhes Cc	Adhes Rc	Cohes c	Cohes C	Cohes R		
Single Bond 2	No	5	21%	3%	69%	0	3%	16 (19%)	64 (81%)
		10	25%	3%	89%	0	0	11 (13%)	69 (87%)
		15	7%	4%	69%	0	3%	10 (12%)	70 (88%)
Single Bond 2	Yes	5	4%	4%	70%	0	0	19 (22%)	61 (78%)
		10	23%	0	77%	0	0	26 (28%)	54 (72%)
		15	23%	0	77%	0	0	13 (14%)	67 (86%)
Prime Bond NP	No	5	6%	0	75%	0%	0	18 (21%)	62 (79%)
		10	8%	0	84%	0%	0	28 (29%)	52 (71%)
		15	13%	4%	70%	0%	0	35 (37%)	45 (63%)
Prime Bond NP	Yes	5	0	0	100%	0	0	6 (6.5%)	74 (93%)
		10	10%	0	90%	0	0	31 (34%)	49 (66%)
		15	0	0	67%	0	0	33 (36%)	47 (64%)

Adhes Cc: adhesive between ceramic and resin cement; Adhes Rc: adhesive between resin composite and resin cement; Cohes C: cohesive failure of ceramic; Cohes c: cohesive failure of resin cement; Cohes R: cohesive failure of resin cement.

Table 4 - Means (standard deviation) and P-values (α = 0.05) for microtensile bond strength (μ-TBS) data (MPa)

Single Bond 2 (ethanol/water based adhesive)			
Drying times	Non-aging*	Aging*	T-Student test**
5	18.0 (6.9) [b]	16.7 (6.9) [a]	P = 0.14 ns
10	20.8 (8) [b]	17.5 (5.0) [a]	P = 0.004
15	24.5 (10.7) [a]	16.9 (5.0) [a]	P < 0.001
	p < 0.001	p = 0.89	
Prime & Bond NT (acetone based adhesive)			
Drying times	Non-aging*	Aging*	T-Student test**
5	33.5 (12.7) [a]	22.9 (7.6) [a]	p < 0.001
10	19.2 (5.6) [b]	21.6 (9.5) [ab]	p = 0.094 ns
15	20.7 (7.0) [b]	17.3 (6.1) [b]	p = 0.007
	p < 0.001	p = 0.02	

*ANOVA and Tukey tests: similar letters indicating similar statistically bond strength results after Tukey test (columns).
** p<0.05 means difference statistically for the non aged and aged groups, keeping the same 'drying time' (row).
ns = no significance.

DISCUSSION

The results of this study showed that different drying times influence the bond strength results of the adhesive systems when considering the immediate test condition, rejecting the first hypothesis. The acetone-based adhesive (PB) exposed to air drying for 5 s and the water/alcohol-based adhesive (SB) exposed to 15 s of air drying, presented higher values of bond strength compared to the other groups. On the other hand, the different drying times did not present significant differences in bond strength after aging, which can mean its irrelevant impact for adhesion when the ceramic-adhesive-cement interfaces are exposed to the storage and thermocycling condition. The use of adhesive system for the cementation of glass ceramics could be an option to ceramic surface treatment [4,5]. Adhesive systems may contain ethanol or water/acetone as solvents in their composition [6-10], and should be applied according to its composition.

The water/ethanol solvents present a low vapor pressure and high ebullition temperature values [14], which translates into a lower evaporation pattern in a clinical setting [15]. Studies showed that the longer time allows water/ethanol solvent evaporation, and may decrease the distance between the monomers [16], increase the degree of conversion [17] and results in a better bond strength. This could explain the higher values obtained by SB after 15 s of air-drying in comparison with the other groups. In addition, the acetone solvent has high values of vapor pressure and low ebullition temperature values [14], showing a higher pattern of evaporation in comparison with water-ethanol solvent [17,18]. These properties are used to guarantee a high conversion degree and a stable pattern of union with the dentin [17].

Prime & Bond NT contain a high rate of acetone in its composition (almost 80%) and consequently a low rate of monomer [19]. The longer the evaporation period before photo activation, the more time acetone has to infiltrate and damage the union between silane and resin cement. The higher number of pre-test failures observed after high evaporation periods (Table 3) could be an indicative of this behavior.

The t-Student test showed that the aging protocol influenced the bonding, decreasing the values of most groups (Table 4), which lead to accept the second hypothesis of the study. These results are in accordance with other authors, showing that independent of the ceramic surface treatment, the ceramic/cement interface did not present a stable union after aging [18,20,21]. These results also reinforce the necessity of more researches to improve the quality of this adhesive interface. Moreover, Passos et al. [22] showed that the ceramic bonding protocol with adhesive application is questionable, since it appears do not improve the bond strength between resin cement and etchedsilanated ceramic. One limitation of the present study is the mechanical test used to evaluate the bond strength. Many studies mentioned the benefits of the microtensile bond strength test, in comparison with other tests [20,21,23]. On another hand, this test is very sensitive, generating great number of pre-test failures, especially in specimens with low adhesive strength.

Based on the results, it was concluded that: (1) The aging protocol negatively affected the bond strength between resin cement and glass ceramic, independent of the solvent of the adhesive; (2) longer drying times are beneficial to the bond strength values between resin cement and glass ceramic, when a water/alcohol based adhesive is applied; (3) shorter drying times are beneficial to the bond strength values between resin cement and glass ceramic, when a acetone based adhesive is applied.

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