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Effect of conditioning agents combined with two adhesive resin cements on Micro-Tensile Bond Strength to polymeric CAD/CAM materials

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ABSTRACT

Purpose: To test the μ-tensile bond strength (μTBS) between resin composite cements and non-/conditioned CAD/CAM polymers.

Methods: PMMA (artBlock Temp) and exp. resin composite CAD/CAM blocks were fabricated, polished and air-abraded. The specimens were conditioned: i. “Monobond Plus/Heliobond”, ii. “visio.link”, iii. “Ambarino P60”, iv. “exp. VP connect”, v. non-conditioned as control group (CG) and luted with a self-adhesive resin composite cement Clearfil SA Cement or an adhesive resin composite cement Variolink II. The blocks were cut into 10 specimens and stored for 24 h at 37 °C water. Half of specimens were thermal cycled (5000 ×, 5 °C/55 °C). μTBS with failure types were assessed. Data was analysed using 4-way ANOVA, Kruskal-Wallis, Mann-Whitney U-, and Chi^2-test ($\alpha = 0.05$).

Results: The highest influence on μTBS was exerted by conditioning method (partial eta squared $\eta_P^2 = 0.715$, $p < 0.001$), followed by aging level ($\eta_P^2 = 0.260$, $p < 0.001$), cement ($\eta_P^2 = 0.196$, $p < 0.001$) and substrate material ($\eta_P^2 = 0.135$, $p < 0.001$). Visio.link showed the highest μTBS, regardless of the substrate material and aging level. Adhesive resin composite cement groups in combination with visio.link and Ambarino P60 showed no impact of aging. Remaining groups presented a negative impact of aging on μTBS. No impact of cements was observed for PMMA without/conditioning using visio.link and composite conditioned using VP connect. Specimens luted using self-adhesive resin composite cement showed higher μTBS than specimens luted using adhesive resin composite cement. Visio.link and VP connect combined with self-adhesive resin composite cement showed higher μTBS on PMMA than on exp. resin composite. No impact of substrate was found for non-aged Monobond Plus/Heliobond in combination with self-adhesive resin composite cement. The remaining groups showed higher μTBS values on experimental resin composite substrate.

Conclusion: For long-term bonding of CAD/CAM polymers an adequate pretreatment method is necessary.

1. Introduction

Due to an increasing demand for esthetic properties, tooth colored restoration materials have gained great relevance [1]. All ceramic has been preferred over decades. Polymeric materials are alternatives to ceramic materials. They are esthetic and provide sufficient mechanical properties [2,3]. With today's computer-aided design/computer-aided manufacturing (CAD/CAM) technologies dental restorations can be milled out of high-density polymer blocks. This can be done chairside or labside in the dental laboratory [4].

Generally, a reliable cementation of restorations is crucial for the clinical long-term success [5]. According to the composition of the adhesive system and the conditioning of the restoration and tooth surfaces, bonding emerges by mechanical and/or chemical reaction [6]. Due to the high grade of conversion less reactive C=C bindings are available [7]. Therefore, novel polymeric CAD/CAM materials require further conditioning to establish bond to resin composite luting cements [7–10].

Resin composite cements can be distinguished into – multi-step adhesive resin composite cements (total and self-etch) and self-adhesive resin composite cements [11]. The latter does not require any pre-treatments and might be easier and more robust in clinical use [12,13]. The bond strength of resin composite cements varies greatly. While some self-adhesive resin composite cements perform equal to adhesive resin composite cements [14] others show inferior bond strength to tooth substrates [15]. Luting cements must not only bond to tooth structure but also to restoration materials like ceramics, metal alloys, and indirect resin composites [12]. For a satisfactory bond of

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restoration materials tested in this study, further conditioning of the surfaces is necessary [9,10].

Although, in-vitro testing in the laboratory is unable to reproduce intra-oral conditions in detail, they provide some information about the reliability of the bond [9] and the quality of adhesion can be assessed [16]. In the literature, a lot of bond strength studies exist – mostly either based on µ-shear bond strength tests or µ-tensile bond strength (µTBS) setups [17], more laborious crown retention tests are also common [18].

It is important that new restoration materials work with common and established dental cements. Here, the interface to the restoration material is on interest but limited information is available regarding bond strength of resin composite cements to industrially manufactured polymeric materials used for CAD/CAM manufacturing. The aim of the study was to figure out the influence of different conditioning methods of polymeric CAD/CAM materials on µTBS initially and after aging and to determine the failure modes of debonding. The null hypothesis tested was that independent of the substrate, conditioning method, resin composite cement, and aging level comparable µTBS could be achieved.

2. Material and methods

The objective of this study was to determine the bond strengths of two polymeric CAD/CAM materials (PMMA: artBlock Temp and resin composite: exp. CAD/CAM resin composite) to two different resin composite cements in combination with different conditioning agents by using the µTBS testing (Table 1).

CAD/CAM polymeric blocks were cut under water cooling into disks of 5.1 mm and 20.1 mm using a Secotom-50 (Struers, Ballerup, Denmark) and polished under constant pressure with a series of silicon carbide papers (SiC) up to P2400 under water application (Abramin, Struers). The specimens were air-abraded with 50 µm alumina powder with a mean size of 50 µm for 20 s with 2 bar at an angle of 45° in a distance of 1 cm (Basic Quattro, Renfert, Hilzingen, Germany) and subsequently cleaned in an ultrasonic bath in distilled water (Sonorex RK102H, Bandelin electronic Berlin, Germany). CAD/CAM materials were conditioned as follows: i. Monobond Plus (MPH) was applied and air-dried for 60 s. Heliobond was applied and light cured for 10 s using an (Elipar S10 curing light, 3M, Seefeld, Germany); ii. visio.link (VL) was applied and light cured for 90 s (bre.Lux Power Unit, Bredent); iii. Ambarino P60 (AP60) was applied and air-dried for 120 s: iv. VP connect (VPC) was applied and air-dried for 180 s, and v. the control groups were non-conditioned. After conditioning, the same substrate material was bonded using a self-adhesive resin composite cement Clearfil SA cement (SARC for Self-Adhesive Resin Cement) or a resin composite cement Variolink II (ARC for Adhesive Resin Cement) and axially loaded with 750 g. The cements were polymerized by light curing for 10 s from each site (Elipar S10 LED curing light, 3 M, Seefeld, Germany). The excess cement was careful removed with a scalpel. For each group PMMA: 5 and composite: 6 bonded blocks were fabricated. The blocks were cut under water cooling perpendicular to the bonding interface with a cross-sectional area of 1 mm^2 (Secotom-50). After the first serial section, the blocks were positioned again but exactly 90° rotated to the first position. The cutting length allowed for bar specimens of 10 mm. From each block 10 specimens were cut.

The specimens were stored in deionized water at 37 °C for 24 h. Thereafter, half of the specimens of each group were randomly selected to be measured directly. The remaining specimens were thermocycled (Thermocycler THE-1100 (SD Mechatronik, Feldkirchen-Westerham, Germany) for 5000 cycles between 5 °C and 55 °C with a dwell time of 20 s. Prior to µTBS testing, Specimens were stored in deionized water for 1 h at room temperature (23 °C).

For µTBS, the specimens were fixed using adhesive (Liquicol, Renfert, Hilzingen, Germany) on aluminum adapters, positioned in the testing machine (MDT-500, SD Mechatronik) and loaded at a cross-speed of 0.5 mm/s until fracture. µTBS was calculated according to the equation: $\sigma = F/A$ (σ: tensile bond strength; F: load at fracture [N]; A: cross-sectional area [mm^2]).

Specimens that failed before µTBS measurements were excluded and not considered for further analysis. Directly after µ-tensile bond measurements, the specimens were analyzed with magnifying glasses at 2.5× magnification (GTX 2,5 Zeiss, Oberkochen, Germany). The debonding was classified as follows into 3 categories: (I) adhesive failure directly at the interface, (II) cohesive failure either within cement layer or the substrate, and (III) mixed mode failure.

For planning of this study, a power analysis for both CAD/CAM materials was performed. The analysis for the PMMA blocks was based on pre-test results of the means of 3 blocks each consisting of 3 bars and for experimental CAD/CAM resin composite on the means of 4 blocks each consisting of 3 bars. The blocks were air-abraded, conditioned using visio.link and luted with Clearfil SA Cement. The first analysis was computed for the differences between pre-treatments. According to the two-sample t-test with a Bonferroni corrected significance level of PMMA: 0.005 (0.05/10), experimental resin composite: 0.017 (0.05/3) the optimal sample size of PMMA: 5 and of experimental resin composite: 6 blocks in each group was computed. The power is equal to

Table 1
Summary of the products, manufacturers, composition, Lot. numbers used in this study.

	Brand	Composition	Manufacturer	Lot
Substrate	artBloc Temp	PMMA unfilled	Merz Dental, Lütjenburg, Germany	44308
	exp. CAD/CAM composite	Polymeric composite with inorganic fillers	Ivoclar Vivadent, Schaan, Liechtenstein	b.28923
Adhesive system	Monobond Plus	Silane methacrylate, phosphoric acid methacrylate, sulfide methacrylate in an alcoholic solvent	Ivoclar Vivadent, Schaan, Liechtenstein	S05679 R22281
	Heliobond	Bis-GMA, TEGDMA, initiators, stabilizers		
	visio.link	MMA, PETIA, photoinitiator	Bredent, Senden, Germany	114784
	Ambarino P60	DMA based on phosphor acidesters and phosphon acidesters	Creamed, Marburg, Germany	2011004057
	Exp. VP connect	MMA	Merz Dental, Lütjenburg, Germany	22912
Resin composite cement	Clearfil SA Cement	Paste A: MDP, Bis-GMA, TEGDMA, DMA, Ba-Al fluorosilicate glass, SiO_2, benzoyl peroxide, initiators Paste B: Bis-GMA, DMA, Ba-Al fluorosilicate glass, SiO_2, pigments	Kuraray Med., Sakazu, Okayama, Japan	058AAA
	Variolink II	Bis-GMA, TEGDMA, UDMA, benzoyl peroxide, inorganic fillers, ytterbium trifluoride, Ba-Al fluorosilicate glass, spheroid mixed oxide, initiators, stabilizers, pigments	Ivoclar Vivadent, Schaan, Liechtenstein	Base: R46653 Catalyst: R42290

PMMA: Polymethylmethacrylate; Bis-GMA: bisphenol-A-diglycidylmethacrylate; TEGDMA: triethylenglycol dimethacrylate; MMA: methylmethacrylate; PETIA: pentaerythritol triacrylate; DMA: dimethacrylate; MDP: 10-methacryloxydecyl dihydrogenphosphate; UDMA: urethane dimethacrylate.