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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 Chi²-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.

PMMA: 99%, composite: 86% given a standard deviation equal to PMMA: 2.8 MPa, composite: 8.5 MPa given a relevant effect of PMMA: 15 MPa, composite: 20 MPa between the tested conditioning groups. In the second analysis, the number of specimens optimal to investigate the difference between aging and resin composite cement levels was computed. According to the two-sample t-test with a significance level 0.05 the optimal sample size of PMMA: 5, of experimental resin composite: 6 blocks in each group is needed. The power is equal to PMMA: 99%, experimental resin composite: 95% given a standard deviation equal to PMMA: 2.8 MPa, experimental resin composite: 8.5 MPa given a relevant effect of PMMA: 15 MPa, composite: 20 MPa between the groups. Overall, 10 bars were manufactured out of one block. The normality of the μTBS data was examined using the Kolmogorov-Smirnov test. Descriptive statistics were calculated. To determine the significant differences between the conditioning methods within resin composite luting cement, aging and substrate groups the 4-way ANOVA was computed together with eta-squared (η_P^2) parameters. Kruskal-Wallis and Mann-Whitney U tests were performed to evaluate the significant differences between the groups. In order to discover differences in the frequency of failure modes between tested groups a Chi^2 test was computed. The relative frequencies of the failure types were provided using Wilson's method. Statistical analysis was performed using SPSS 23.0 (SPSS, IBM Corp., Version 23.0. Armonk, NY, USA) ($\alpha = 0.05$).

3. Results

The highest influence on the μTBS was exerted by the conditioning method (partial eta squared $\eta_P^2 = 0.715$, $p < 0.001$), followed by the aging level ($\eta_P^2 = 0.260$, $p < 0.001$), resin composite cement ($\eta_P^2 = 0.196$, $p < 0.001$) and the substrate ($\eta_P^2 = 0.135$, $p < 0.001$). Also, the effect of the binary, ternary or quaternary combinations of the four parameters was significant ($p < 0.001$). The fixed effects could not be compared directly, as the higher order interactions between them were found to be significant. Consequently, several different analyses were computed and divided by levels of substrate, conditioning method, resin composite cement and aging level. The results of the descriptive statistics (mean ± SD) are presented in Fig. 1 and Table 2. The Kolmogorov-Smirnov test indicated a violation of the assumption of normality in approximately 27% of the tested μTBS groups, therefore data was analyzed using non-parametric tests.

3.1. Impact of substrate

VL ($p = 0.006$) and VPC (without TC: $p = 0.009$, TC: $p = 0.028$) combined with SARC showed higher μTBS values on PMMA substrate than on experimental resin composite substrate. Within non-aged MPH ($p = 0.144$) in combination with SARC group no impact of substrate on μTBS values was observed. The remaining groups ($p = 0.004$–0.044) showed higher μTBS values on experimental resin composite substrate.

3.2. Impact of conditioning method

Within all *PMMA substrate* groups a significant impact of conditioning method was observed ($p < 0.001$). VL generally showed the highest μTBS values, regardless of the substrate and aging level ($p < 0.001$). None of the aged groups luted with SARC and conditioned using MPH presented significantly higher μTBS values than after conditioning using AP60 ($p < 0.001$). Both groups, CG and VPC were in the same value range with AP60 and MPH. Within no aged groups luted using ARC the conditioning with MPH showed significantly higher μTBS values than the CG or the groups conditioned using AP60 and VPC ($p < 0.001$). Within aged SARC groups, the lowest values were observed for CG followed by the group conditioned using VPC while conditioning using AP60 and MPH proved not to be significant compared to CG and VPC ($p > 0.160$). Within aged ARC groups, no statistical differences between CG, AP60, VPC and MPH were found ($p > 0.799$).

With respect to experimental resin composite substrate a significant impact of conditioning method was observed ($p < 0.001$). VL generally showed the highest μTBS values, regardless of the substrate and aging level ($p < 0.001$). Within groups cemented using SARC non-conditioned groups showed comparable initial μTBS values to VL ($p > 0.165$). Within specimens luted with SARC MPH and AP60 showed significantly higher initial μTBS than VPC ($p < 0.001$). Within ARC the second highest μTBS was observed for MPH, followed by CG, VPC and AP60 groups ($p < 0.001$). Within aged SARC groups, VPC presented lower μTBS than MPH and AP60 ($p < 0.001$). Within aged ARC groups, significantly higher μTBS for MPH than for AP60, VPC or CG group was found ($p < 0.001$).

3.3. Impact of resin composite cement

Experimental resin composite substrate combined with VL and luted using ARC ($p = 0.004$) showed significantly higher μTBS than luted with SARC. No impact of resin composite cements was observed in the following groups: PMMA substrate without conditioning ($p > 0.999$) and conditioned using VL ($p = 0.465$) and composite conditioned using VPC ($p = 0.144$) regardless of the aging level.

3.4. Impact of aging level

Specimens luted using ARC on PMMA ($p = 0.050$–0.999) as well as on exp. resin composite substrate in combination with VL ($p = 0.150$) and AP60 ($p = 0.439$) showed no impact of thermal aging. In contrast, all remaining groups presented a significantly decrease of μTBS values after thermal cycling compared to non-aged specimens ($p = 0.004$–$0.0.009$).

3.5. Classification of failure modes

For PMMA groups, the adhesive failure mode was dominant. Only visio.link groups showed partly mixed or cohesive failures. On experimental resin composite substrate, different observations were made. A lot of cohesive and mixed failures were observed especially for the groups with high μTBS values above approximately 10 MPa (Table 2).

4. Discussion

This study examined the influence of different conditioning methods of polymeric CAD/CAM materials on μTBS. The failure mode of debonding has been determined. The null hypothesis that independent of the conditioning method, substrate, aging level, and cement comparable bond strength could be achieved needs to be rejected.

The conditioning method shows the highest impact on μTBS. Groups with visio.link as conditioning agent achieved best results to both cements and both substrates tested initially and after aging. The finding is in accordance to Keul et al. [19] who reported good bonding performance of visio.link on same substrates but in a crown pull-off test design.

Visio.link contains MMA and highly reactive triacrylate monomers (PETIA). That might explain the good bonding performance to resin composite cements and polymeric restoration materials based on methacrylate chemistry. MMA and PETIA can penetrate into the resin matrix of the polymeric restoration material and can create entanglements [19] which function as mechanical connection. Even in polymeric restoration materials with a high conversion rate due to industrial curing, a lot of unreacted double bonds are still prevalent. MMA and PETIA allow for covalent bindings to methacrylates in polymeric restoration materials respectively resin composite cements. PETIA specifically leads to an increased high crosslinking density at the interface and within the layer of the primer. It can be expected, the high crosslinking density of visio.link contributes to good mechanical values of

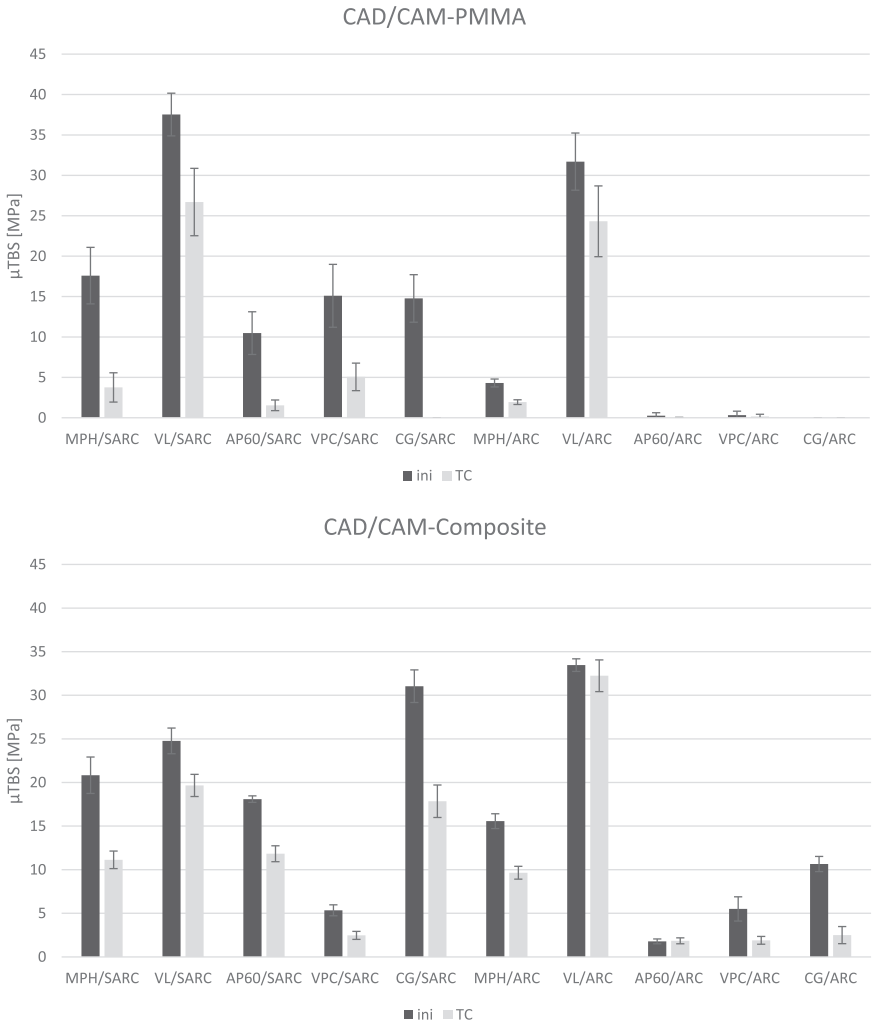

Fig. 1. Bar graphs (mean ± SD) representing µTBS values for PMMA- and composite-substrates, separately.

the interface after curing as well.

Another aspect to explain the good performance of visio.link might be the good wetting behavior to other polymers which is a prerequisite for chemical interactions at the interface and for a good mechanical interlocking in micro pores of the surfaces. A poor wetting behavior might be an explanation for the worse results of the exp. VP connect. Assuming adequate curing, the MMA containing exp. VP connect should have shown better adhesion performance especially to PMMA.

Cleaning and enlargement of the surface to increase the mechanical retention is intended by air abrasion treatment step which is a standard for all dental restoration materials except glass ceramics. When treating resin composites in that way, fillers become present at the surface. The ratio of fillers and resin matrix at the surface is dependent on the filler content [19]. The filler content of resin composites intended for permanent restorations is typically in the range of 50 vol%. For an adhesive bond to fillers acidic groups are instrumental because they can create ionic interactions with the filler surfaces. How well acidic monomers can contribute to the adhesion performance to inorganic fillers is indicated by the good values of the control group with the self-adhesive resin composite cement on resin composite. Clearfil SA cement contains MDP, a phosphoric acid derivate.

According to manufacturer's information, visio.link does not contain acidic groups conclusively the good adhesion performance is a result of the bonding mechanisms to the resin matrix of the restoration materials and the resin composite cements. The composition of visio.link seems to be highly compatible to the polymeric parts of the systems tested.

Using cements without conditioning of the restoration surface might work for resin composites when using a self-adhesive resin composite cements. In all other cases, there were no or very low bond strength values observed. One reason might be the fast reaction kinetic of radical polymerization. Due to the polymerization shrinkage, the stress at the interface is very high. The initial attachment cannot withstand the shrinkage induced stress resulting in initial gap formation. Another reason of low or zero bond strength values might be the higher viscosity of cements compared to the adhesives which results in poor wetting of the restoration surface. An explanation why the self-adhesive resin composite cement worked without conditioning of the experimental resin composite can be that for resin composites the air abraded surface shows to a great extend uncovered hydrophilic inorganic filler surfaces which might allow the also more hydrophilic acidic self-adhesive resin composite cement to wet the surface and to bond to the fillers especially.

For aging, the specimens were thermocycled 5000 times between 5 °C and 55 °C. This method can be seen as the best method to expose all specimens to reproducible thermal stress [20]. A significant decrease of µTBS was observed for the majority of the groups. Only the groups with visio.link in combination with the adhesive resin composite cement showed high values and lowest adhesive failure rate independent from aging. The µTBS level of these groups is significantly higher than with the dedicated primer system Monobond Plus/Heliobond. Monobond Plus contains silane (molecules with terminal Si-OH groups and reactive double bindings) which creates a good bond to fillers. On the other

Table 2
Descriptive statistics of μTBS results for all tested groups.

Substrate	Pre-treatments	Cement	TC	Mean (SD)	95% CI	Min/Median/Max	Adhesive failure mode (95% CI)
PMMA	MPH	SARC	–	17.59 (3.50)	(13.2;22.0)	14.52/17.91/23.06	100 (91;100)
	VL			37.53 (2.64)	(34.2;40.9)	34.93/37.51/41.38	45 (31;60)
	AP60			10.48 (2.64)	(7.2;13.8)	7.58/9.89/14.20	100 (91;100)
	VPC			15.10 (3.89)	(10.2;20.0)	11.47/14.49/21.70	100 (91;100)
	CG			14.77 (2.94)	(11.0;18.5)	11.47/14.61/18.99	100 (91;100)
	MPH	ARC		4.31 (0.49)	(3.1;5.6)	3.75/4.61/4.58	100 (91;100)
	VL			31.70 (3.54)	(27.3;36.1)	28.83/29.72/36.00	48 (33;63)
	AP60			0.26 (0.37)	(0.1;0.8)	0/0/0.76	100 (91;100)
	VPC			0.33 (0.49)	(0.2;1.0)	0/0/1.10	100 (91;100)
	CG			0	–	0/0/0	100 (91;100)
	MPH	SARC	TC	3.76 (1.81)	(1.5;6.1)	2.39/2.73/6.63	100 (91;100)
	VL			26.70 (4.17)	(21.5;31.9)	22.66/25.05/33.27	70 (55;82)
	AP60			1.54 (0.66)	(0.7;2.4)	0.90/1.52/2.58	100 (91;100)
	VPC			5.06 (1.71)	(2.9;7.2)	2.46/5.36/6.77	100 (91;100)
	CG			0	–	0/0/0	100 (91;100)
	MPH	ARC		1.94 (0.29)	(1.2;2.7)	1.65/1.92/2.24	100 (91;100)
	VL			24.32 (4.38)	(18.8;29.8)	18.94/24.94/30.51	75 (60;86)
	AP60			0.02 (0.04)	(0;0.1)	0/0/0.08	100 (91;100)
	VPC			0.17 (0.26)	(0;0.5)	0/0/0.58	100 (91;100)
	CG			0	–	0/0/0	100 (91;100)
Comp	MPH	SARC	–	20.83 (2.09)	(18.6;23.1)	17.77/20.87/23.84	0 (0;7)
	VL			24.77 (1.47)	(23.2;26.4)	23.03/25.05/26.82	0 (0;7)
	AP60			18.09 (0.37)	(17.6;18.5)	17.36/18.18/18.43	0 (0;7)
	VPC			5.34 (0.63)	(4.5;6.2)	4.54/5.33/6.13	58 (44;71)
	CG			31.04 (1.87)	(29.0;33.1)	28.10/31.43/32.94	0 (0;7)
	MPH	ARC		15.57 (0.85)	(14.6;16.5)	14.40/15.61/16.90	0 (0;7)
	VL			33.46 (0.72)	(32.7;34.3)	32.78/33.32/34.37	0 (0;7)
	AP60			1.78 (0.28)	(0;4.4)	1.58/1.78/1.98	98 (89;100)
	VPC			5.50 (1.39)	(4.0;7.0)	2.87/5.85/6.75	44 (31;58)
	CG			10.65 (0.87)	(9.7;11.6)	9.39/10.69/11.99	48 (34;62)
	MPH	SARC	TC	11.13 (1.01)	(10.0;12.2)	9.95/10.88/12.93	0 (0;7)
	VL			19.66 (1.27)	(18.3;21.0)	17.45/19.83/21.39	0 (0;7)
	AP60			11.83 (0.91)	(10.8;12.8)	11.03/11.52/13.19	0 (0;7)
	VPC			2.47 (0.46)	(1.9;3.1)	1.98/2.30/3.04	65 (50;77)
	CG			17.85 (1.86)	(15.8;19.8)	16.04/17.32/20.24	2 (0;11)
	MPH	ARC		9.65 (0.74)	(8.8;10.5)	8.43/9.76/10.35	0 (0;7)
	VL			32.24 (1.82)	(30.3;34.2)	29.32/32.73/34.69	0 (0;7)
	AP60			1.85 (0.34)	(0;5.0)	1.61/1.85/2.09	86 (75;94)
	VPC			1.90 (0.45)	(1.4;2.4)	1.19/1.97/2.38	59 (44;71)
	CG			2.50 (0.98)	(1.4;3.6)	0.86/2.79/3.67	79 (66;88)

hand, silane might weaken the bond to the polymer matrix since the Si-OH groups cannot contribute to the bond like covalent reactions. Only weaker hydrogen bonds can be built to the OH-groups that might be prevalent in the partnering resin matrix [19]. Generally, a decrease of μTBS after aging due to thermally induced stresses and the potential water uptake of polymeric materials can be expected [21]. In this study, especially all self-adhesive resin composite cement groups showed a significant decrease in μTBS values after aging. This leads to the assumption that the cement has an influence. Usually, self-adhesive resin composite cements are hydrophilic in nature to ensure good wetting capabilities of moist surfaces like dentin. On the other hand, this hydrophilicity can lead to higher sensitivity to water. Such interactions with water can jeopardize the bond strength. Petropoulou et al. [22] stated that interaction of resin composite cements with water is not type-related but they specifically reported high water sorption and solubility in water for Clearfil SA cement which has been used in this study.

In the present study, a μTBS test was used. In relevant points, the test setup was in strong accordance with the guidance recently published by Armstrong et al. [23]. The μTBS test was originally introduced 1994 by Sano et al. [24] to measure bond strength of small surfaces in order to assess the performance of dentin adhesives. Since then, μ-tensile bond testing has been employed in dozens of studies but is still discussed controversially. By some researchers, the test is seen as a versatile and reliable method to test the bonding performance [25]. With the rational that due to the reduced bonding area, a lower force is required which does not exceed the cohesive strength of the substrates, it is stated that true adhesive failures can be found by using this method [26]. In contrast, Söderholm et al. [27] concluded after a stress distribution analysis that the test does not predict clinical bond failure well. The highest stress levels were found in the substrates and not in the adhesive joint. With this finding, the authors speculate that this might be the explanation of often reported mixed cohesive failures.

Shear bond strength testing methods are discussed as competitive test methods. As Dünbar [28] stated in a comparison study of μTBS and shear bond strength testing, the testing method has a significant influence. In other studies, μTBS and μ-shear bond strength tests were compared [29,30]. Based on that, both micro bond tests lead to correlated results and therefore similar findings and conclusions might be expected when comparing results of studies where either one or the other test has been used.

A general disadvantage of all bond strength tests mentioned before is that the tests are conducted on flat surfaces and that the influence of the complex geometry of dental restorations with a different ratio of bonded to non-bonded surface is ignored [18].

Considering the discussions regarding the validity of findings based on different bond strength test methods, a comparison with other studies is mandatory. Here, it can be stated that the findings in this study are in strong accordance to the findings in a previous study published by Keul et al. [10] where same materials were tested but with a tensile bond strength method. The same is evident when comparing the results with another previous study published by Bähr et al. [9] where a shear bond strength test setup was used.

Laboratory tests definitely provide evidence of bonding

performances of different material systems. As discussed, the setup of the bond test has a high influence on the results. For this and other reasons, clinical studies need to be performed to validate such results. Within the limitation of this study the following can be concluded:

- Micromechanical retentions alone are not sufficient to obtain a sufficient bond between luting cement and polymeric CAD/CAM restoration material. For a durable bond, polymeric CAD/CAM restoration materials should definitely be pre-treated with suitable conditioning agents.
- The combination of visio.link and adhesive resin composite cement Variolink II shows the highest and most durable bond strength on polymeric CAD/CAM materials.
- Regardless of the substrate and luting cement used, visio.link generally shows the highest μTBS values and might therefore be the preferred conditioning method.

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