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ORIGINAL ARTICLE

Tensile bond strength of resin composite repair in vitro using different surface preparation conditionings to an aged CAD/CAM resin nanoceramic

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Abstract
Objectives This study was conducted in order to assess the pretreatment method (air abrasion, both wet and dry, and Al_2O_3 grinder), the conditioning method (comprised of different adhesive systems), the repair resin composite (low and high modulus of elasticity), the contamination of CoJet air-abraded surfaces with water, and the effect phosphoric acid on the macrotensile bond strength (TBS) to aged CAD/CAM resin nanoceramic (RNC).
Materials and methods Aged RNC substrates (LAVA Ultimate, 3M ESPE; N=900; 10,000 cycles, 5 °C/55 °C) were air-abraded (CoJet 3M ESPE) with and without water contamination or treated with an Al_2O_3 grinder (Cimara, Voco). Immediately after pretreatment, half of the specimens were additionally cleaned with phosphoric acid, while the rest were only rinsed with water. Four intermediate agents (Futurabond U/VOCO, Scotchbond Universal/3M ESPE, One Coat Bond/Coltène Whaledent, visio.link/bredent) were selected for conditioning the surface, while no conditioned specimens acted as control groups. Specimens were thereafter repaired using two direct resin composites (Arabesk Top and GrandioSo, VOCO), stored for 24 h at 37 °C in H_2O, and thermally aged for 10,000 cycles (5 °C/55 °C; n=15/subgroup). TBS and failure types were determined and evaluated with four- and one-way ANOVA and χ^2 test ($p<0.05$).
Results The highest influence on TBS was exerted by the conditioning method (partial eta-squared (η_P^2)=0.273, $p<0.05$), followed by the resin composite repair (η_P^2=0.07, $p<0.05$) and the surface pretreatment method (η_P^2=0.032, $p<0.05$), while an acid contamination after surface pretreatment was insignificant (p=0.154).
Conclusions Air abrasion produced superior TBS compared to grinding of the surface with Al_2O_3 prior to repair. The tested universal adhesives proved to be effective intermediate agents for repairing aged CAD/CAM RNC, while visio.link and Scotchbond Universal performed slightly better than Futurabond U.
Clinical relevance Phosphoric acid or water contamination of the air-abraded surface does not affect the repair bond strength.

Keywords Repair · Surface conditioning · Tensile bond strength · Resin nanoceramic · RNC · CAD/CAM resin · Resin composites · LAVA Ultimate

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Introduction

Currently, dental restorations milled out of resins by the use of computer-aided design/computer-aided manufacturing (CAM/CAM) technology are likely useful for high-quality long-term restorations [1]. The design and manufacturing of the restoration can be made either in the dental laboratory or directly in the dental office, with the advantage of reduced treatment time and the elimination of temporary chairside dentures [2].

Based on the industrial standardized polymerization of CAD/CAM resin blanks under high pressure and temperature, significantly higher physical and mechanical properties can be achieved compared to conventionally polymerized (direct/indirect temporaries) materials [2–5]. Not only improved mechanical behavior but also fewer discolorations [6] and higher abrasion resistance [7] in relation to the conventional polymerized resins were obtained. Furthermore, the operator had a considerable effect on the quality of conventional polymerized resins [6]. Especially for thin dental restorations, CAD/CAM milled resins showed a higher fracture resistance than restorations of glass-ceramics [7–9].

LAVA Ultimate is a composite of resin and nanofillers, called in the dental market as resin nanoceramic (RNC). This material contains nanomer and nanocluster fillers (silica nanomers of 20 nm diameter and zirconia nanomers of 4–11 nm diameter) with a total nanoceramic material content by weight of approximately 80 %. The engineered nanoparticles are treated with a silane-coupling agent using a proprietary method. This functionalized silane bonds chemically to the nanoceramic surface as well as to the resin matrix. The manufacturer has approved this CAD/CAM resin for long-term restorations.

Likewise, as a consequence of their mechanical properties and enamel wear characteristics, resin-based materials offer further advantages over glass-ceramics because they cause distinct enamel wear in antagonists [10–13]. However, loss of the CAD/CAM materials itself is very high compared to glass-ceramics [7]. Due to all of these dependent factors, and the possibility of producing dental restorations with lower costs and exposure times when using CAD/CAM materials [2], it should be clarified whether these resins can be seated into the patient's mouth for a longer period. This raises the question of whether these CAD/CAM materials would benefit from an additional protection in the form of a resin composite, or if they could be properly repaired, if necessary. Currently, difficulties are being encountered regarding the durable repair/restorations of CAD/CAM materials. Due to the standardized polymerization procedure, these resins have barely sufficient carbon-carbon double bonds on the surface to which the resin-luting agent can bond. Studies on the bond strengths between CAD/CAM materials and resin composites have shown that besides surface roughening, an additional application of adhesive systems is required [14–17].

An advantage of CAD/CAM material restorations is seen in the lowered risk of treatment for an intraoral restoration repair, since the use of hydrofluoric acid, indicated for surface treatment of glass-ceramic restorations, is no longer necessary [18]. The poor esthetic properties of composite CAD/CAM restorations, when compared with glass-ceramics, can be amended, since efficient methods for esthetic modification of the restoration after machine milling have been proposed [19]. Similar to repairs of resin composite restorations [18], this includes conditioning the surface with air-borne-particle abrasion and the use of a silane-coupling agent and an adhesive resin as intermediate agents, followed by the application of a resin composite [19]. As for the repair strength of aged resin composites, literature data reported within 50 resin composite combinations shear bond strength values ranging from 10.27 to 43.8 MPa. This repair strength was 35.4–90.9 % of the cohesive strength of the original composites, while the filler loading of the considered materials ranged from 81 to 92 wt% [20].

In an attempt to simplify a restoration procedure, universal adhesive systems were recently launched on the market, with fewer steps and less chance of error in the application process. Their chemical composition includes—in addition to methacrylic monomers—silane or phosphate monomers, allowing them to prime metal, silica-based ceramic, and zirconia restorations. The aim of this study was therefore to analyze the effectiveness of repairing aged NRC substrates by using different surface pretreatment and conditioning methods and different resin composites as repair material. Since a contamination of the air-abraded surface with water or phosphoric acid might occur clinically during a restoration procedure, the study aims to simulate these conditions and to determine their impact on repair efficiency.

The hypotheses tested were as follows: (i) the pretreatment method (air abrasion, both wet and dry, and Al_2O_3 grinder); (ii) the conditioning method (comprised of different adhesive systems); (iii) the repair resin composite (low and high modulus of elasticity); (iv) the contamination of CoJet air-abraded surfaces with water; and (v) phosphoric acid shows no impact on the tensile bond strength (TBS) to aged CAD/CAM RNC substrates.

Material and methods

This study tested the TBS of a CAD/CAM resin nanoceramic LAVA Ultimate (3M ESPE, Seefeld, Germany), in combination with different methods of conditioning for repair with two different resin composites: GrandioSo and Arabesk Top (both from VOCO, Cuxhaven, Germany). For the repair of industrially polymerized CAD/CAM resins, there is no practical way to include a control group. Therefore, no cohesive bond strength was measured. The compositions and batch number of all tested materials are shown in Table 1.

Nine hundred slices of 3 mm thickness×5 mm width×5 mm length were cut from CAD/CAM blocks during water cooling using a fully automatic cut machine (Secotom-50, Struers, Ballerup, Denmark; rotational speed 2,200 rpm, feed speed of 0.1 mm/s) and then embedded in a self-cured two-component acrylic resin (ScandiQuick, ScanDia, Hagen, Germany; Lot. No: 542125/142125). Bonding surfaces were polished during water cooling with a series of silicon carbide papers up to SiC P2400 (Tegramin-20, Struers). Thereafter, all polished surfaces were aged for 10,000 thermal cycles between 5 and 55 °C with a dwelling time of 20 s in each bath (Thermocycler THE-1100, SD Mechatronik, Feldkirchen-Westerham, Germany). The 900 specimens were then randomly divided into three pretreatment methods (n=300): (i) CoJet dry air-abrading (3M ESPE), (ii) CoJet wet air-abrading (3M ESPE), and (iii) Cimara grinding (Voco). The detailed

Table 1 Materials, composition, and form of application as used in the study

	Brands	Compositions	Manufacturer	Lot no.	Application
CAD/CAM RNC	LAVA Ultimate	Polymer with approx. 80 wt% inorganic filler[a]	3M ESPE, Seefeld, Germany	N392136	
Pretreatment method	CoJet dry	Silicatized sand (30 μm)[b]	3M ESPE, Seefeld, Germany	516365	10 s, 10 mm perpendicular, 3 bars
	CoJet wet	Silicatized sand (30 μm)[b] + distilled water	3M ESPE, Seefeld, Germany	516365	10 s, 10 mm perpendicular, 3 bars, 10 s water spray
	VOCO Cimara grinder	Silicon carbide[c]	VOCO, Cuxhaven, Germany	PP-2012-890, 027258	Roughening surface with 10,000 rpm
	Scotchbond Universal Etchant phosphoric acid 34 %	Water, phosphoric acid, silica, polyethylene glycol, aluminum oxide[d]	3M ESPE, Seefeld, Germany	520594, R22282	Application for 30 s and cleaning with distilled water for 30 s
Conditioning method	Futurabond U	HEMA, Bis-GMA, HEDMA, acidic adhesive monomer, UDMA[e]	VOCO, Cuxhaven, Germany	1327615	Application and light curing for 10 s
	One Coat Bond	HEMA, hydroxypropylmethacrylate, methacrylate-modified polyacrylic acid, UDMA, glycerol, DM, amorphous silicic acid, water[f]	Coltène Whaledent, Altstätten, Switzerland	F24467	Application and light curing for 10 s
	Scotchbond Universal	MDP phosphate monomer, DM, HEMA, Vitrebond copolymer, filler, ethanol, water, silane[g]	3M ESPE, Seefeld, Germany	521215	Application and light curing for 10 s
	visio.link	Methyl methacrylate, pentaerythritol triacrylate, pentaerythritol tetraacrylate, diphenyl(2,4,6,-trimethylbenzoyl)-phosphineoxide[h]	bredent, Senden, Germany	114784	Application and light curing for 30 s
Resin composite	Arabesk Top GrandioSo	Bis-GMA, UDMA, TEGDMA, 77 wt% filler[i] Bis-GMA, Bis-EMA, TEGDMA, 89 wt% filler[j]	VOCO, Cuxhaven, Germany	1246419 1327362 1334312	2 mm increments, 20 s light curing, subsequently light curing from 3 directions

Data are provided by the manufacturers

Bis-EMA bisphenol A polyethylene glycol diether dimethacrylate, *Bis-GMA* bisphenol A diglycidyl ether dimethacrylate, *TEGDMA* triethyleneglycol dimethacrylate, *UDMA* urethane dimethacrylate, *HEMA hydroxyethylmethacrylate*, *DM* dimethacrylate, *HEDMA* hydroxyethyldimethacrylate

[a] 3M ESPE, LAVA Ultimate CAD/CAM Restorative, Technical Product Profile, http://multimedia.3m.com/mws/mediawebserver?66666UuZjcFSLXTtMXfamxz6EVuQEcuZgVs6EVs6E666666–&fn=Lava_Ult_TPP.pdf

[b] 3M ESPE, CoJet Product Dossier, http://multimedia.3m.com/mws/mediawebserver?mwsId=SSSSSufSevTsZxtUMx_GMx_GevUqevTSevTSevTSeSSSSSS–&fn=cojet_tp_en.pdf

[c] According to the manufacturer's information, VOCO

[d] 3M ESPE, Material Safety Data Sheet, Scotchbond Universal Etchant, June 2012, http://multimedia.3m.com/mws/mediawebserver?mwsId=SSSSSuUn_zu8l00xMY_eN8mGov70k17zHvu9lxtD7SSSSSS–

[e] VOCO, Safety data sheet Futurabond U, Feb 2013, http://www.voco.com/en/products/_products/futurabond-u//sdb-Futurabond-U-E-rev_-00-130227.pdf

[f] Coltene, Technical Product Profile, One Coat Bond, http://www.coltene.com/download.php?file_id=4108

[g] 3M ESPE, Scotchbond Universal Adhesive, Technical Product Profile, http://multimedia.3m.com/mws/mediawebserver?mwsId=SSSSSuH8gc7nZxtUo8_Zo8_UevUqe17zHvTSevTSeSSSSSS–

[h] bredent, Safety data sheet, visio.link, Jan 2008, http://www.xpdent.com/MSDS/sdb_visio-link_GB.pdf

[i] VOCO, Safety data sheet, Arabesk Top, July 2013, http://www.voco.com/en/products/_products/arabesk_top/sdb-Arabesk-Top-E-rev_-00-130701.pdf

[j] VOCO, GrandioSo Scientific Product Information, http://www.voco.com/us/products/_products/GrandioSO/VC_84_002610__1110_GB_V.pdf

steps of the pretreatment are described in Table 1. Immediately after pretreatment, half of each treated group (n=150) was additionally cleaned with phosphoric acid (Table 1), while the other half (n=150) was only rinsed with distilled water.

Thereafter, the specimens were randomly divided into five main groups for different conditioning methods (n=30), as follows:

1. Futurabond U (VOCO, Cuxhafen, Germany)
2. One Coat Bond (Coltène Whaledent, Altstätten, Switzerland)
3. Scotchbond Universal (3M ESPE, Seefeld, Germany)
4. visio.link (bredent, Senden, Germany)
5. no conditioning, serving as the control group

The application steps are described in Table 1. Thereafter, the conditioned specimens were repaired using two different resin composites (Arabesk Top and GrandioSo, n=15 per resin composite). For the repair procedure, the specimens were positioned into a holding device and an acrylic cylinder (SD Mechatronik) with an inner diameter of 2.9 mm and a height of 4.5 mm and was fixed on the conditioned CAD/CAM resin surface, filled with resin composite, and axially loaded with 100 g. Light polymerization was performed with a blue-violet LED curing unit (VALO, Ultradent Products Inc., South Jordan, UT, USA, standard power mode, 1,176 mW/cm^2), with three sequences of 20 s each, by applying the curing unit perpendicular directly onto the acrylic cylinder from three directions. Subsequently, the specimens were stored for 24 h at 37 °C in distilled water to allow for post-polymerization and then additionally aged for 10,000 thermal cycles between 5 and 55 °C with a dwelling time of 20 s.

The Universal Testing Machine (MCE 2000 ST, Quicktest, Langenfeld, Germany) was used for tensile strength measurements by positioning the specimens in a special device that provided a moment-free axial force application (Fig. 1). A collet held the acrylic cylinder while an alignment jig allowed for self-centering of the specimen. The device was attached to the load cell and pulled apart by the upper and lower chain, allowing the whole system to be self-aligning. The specimens were loaded at a crosshead speed of 5 mm/min until debonding of the cylinders occurred. Values were recorded at the time of the debonding of the cylinders. Bond strength was expressed by dividing the force by the bonded surface area.

The fracture pattern was determined by analyzing the specimens under a stereomicroscope (Axioskop 2Mat, Carl Zeiss Microscopy, LLC, Thornwood, NY, USA). The fracture mechanism was divided into three different types: (1) adhesive, when the failure occurred in the interface between the CAD/CAM material and the resin composite; (2) cohesive, when the failure was in the CAD/CAM material or in the resin composite; and (3) mixed. Fractures occurring during the thermal aging process (prefailure) were recorded as prefailure and considered as 0 MPa.

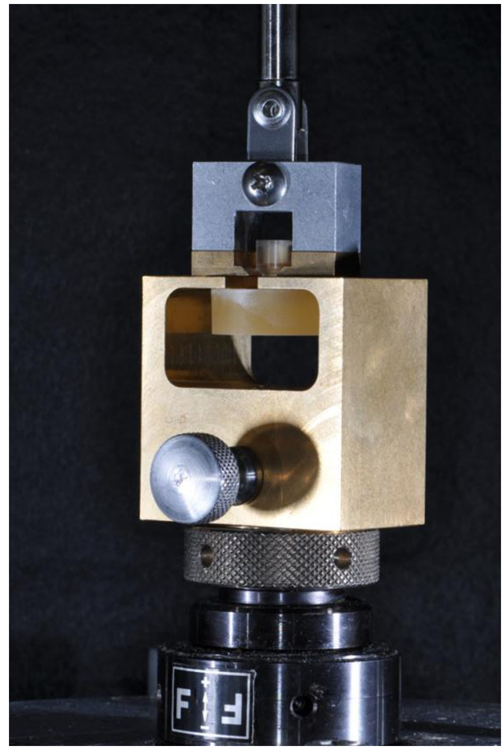

Fig. 1 Design of the macrotensile strength test

The measured data were analyzed using descriptive statistics such as mean and standard deviation. Normality of data distribution was tested using the Kolmogorov-Smirnov and Shapiro-Wilk tests. Four- and one-way ANOVA followed by the Scheffé post hoc test were computed to determine the significant differences among the pretreatment or conditioning method groups. The impact of cleaning using phosphoric acid or the impact of resin composite type was calculated using an unpaired two-sample t test. Relative frequencies of failure types were provided. A χ^2 test was used to detect differences in frequencies of failure types in different groups. The statistical tests were performed with SPSS Version 20.0 (SPSS Inc, Chicago, IL, USA).

Results

The highest influence on the TBS was exerted by the conditioning method (partial eta-squared (η_P^2)=0.273, p<0.05), followed by the resin composite repair (η_P^2=0.07, p<0.05) and the surface pretreatment method (η_P^2=0.032, p<0.05), while acid contamination after surface pretreatment was not significant (p=0.154). The effect of the binary, ternary, or quaternary combinations of the four parameters was significant only for the combinations of surface pretreatment method coupled with acid contamination (η_P^2=0.011, p<0.05), surface pretreatment method coupled with resin composite repair (η_P^2=0.015, p<0.05), and conditioning methods coupled with acid contamination (η_P^2=0.015, p<0.05).

The four-way ANOVA interactions between the effects were significant ($p<0.011$). Therefore, the fixed effects cannot 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 surface pretreatment, as well as the use of acid, adhesive systems, and resin composites, depending on the hypothesis of interest. The results of the descriptive statistics (mean, SD) are presented in Table 2.

Impact of the pretreatment method on CAD/CAM RNC

The groups where no acid cleaning was used showed significantly lower TBS values following a pretreatment using the Cimara grinder compared to the TBS values of CoJet air-abraded groups (dry or wet) for Scotchbond Universal combined with GrandioSo ($p=0.019$), no conditioning combined with GrandioSo ($p<0.001$), and visio.link combined with Arabesk Top ($p=0.009$).

In contrast, within the acid-cleaned groups, a significant impact of the substrate pretreatment was observed only among groups conditioned by using One Coat Bond. In the groups repaired with GrandioSo, a significantly higher TBS was found for a CoJet wet air abrasion compared to CoJet dry air abrasion and Cimara grinding ($p=0.01$). Within the groups repaired with Arabesk Top, the group pretreated with the Cimara grinder showed significantly lower values than the CoJet wet air-abraded group ($p=0.036$).

Impact of acid cleaning after pretreatment of CAD/CAM RNC surface

No consistent results were found for acid cleaning after pretreatment of CAD/CAM restoration. Within groups repaired

Table 2 Descriptive statistics (mean and standard deviation (SD)) for the tensile bond strength as a function of the repair method using repair composites: (a) GrandioSo and (b) Arabesk Top

Treatment	Acid	Adhesive	GrandioSo Mean±SD (MPa)	Arabesk Top Mean±SD (MPa)
CoJet dry air-abraded	Yes	Futurabond U	19.7±6.1	20.3±5.7
		One Coat Bond	12.8±5.6	13.8±3.9
		Scotchbond Universal	19.7±5.6	19.3±5.0
		visio.link	20.1±7.1	20.0±4.8
		No conditioning	10.7±5.2	8.8±6.2
	No	Futurabond U	17.4±7.5	16.4±10.7
		One Coat Bond	14.7±6.8	16.8±5.5
		Scotchbond Universal	23.3±5.9	21.4±5.9
		visio.link	18.8±9.5	22.4±8.2
		No conditioning	11.5±5.6	13.3±5.2
CoJet wet air-abraded	Yes	Futurabond U	17.5±4.6	18.4±5.2
		One Coat Bond	18.7±5.7	12.1±4.0
		Scotchbond Universal	20.8±5.9	22.5±6.0
		visio.link	18.7±3.9	21.2±6.8
		No conditioning	11.2±3.6	10.3±4.4
	No	Futurabond U	15.1±6.3	13.4±3.4
		One Coat Bond	17.0±7.2	16.2±6.0
		Scotchbond Universal	19.4±6.4	17.8±4.6
		visio.link	18.3±4.8	21.8±5.1
		No conditioning	11.3±3.1	10.5±3.4
Cimara grinder	Yes	Futurabond U	14.4±6.4	18.2±5.8
		One Coat Bond	10.8±5.0	16.1±4.3
		Scotchbond Universal	17.0±7.1	21.4±8.0
		visio.link	18.2±8.8	21.7±7.9
		No conditioning	9.0±4.8	8.3±6.0
	No	Futurabond U	12.9±5.6	17.0±6.5
		One Coat Bond	13.2±6.1	15.4±7.6
		Scotchbond Universal	16.7±6.3	18.9±5.5
		visio.link	14.5±7.1	15.1±7.0
		No conditioning	4.8±5.5	9.1±5.0

with GrandioSo, substrate grinded with the Cimara grinder without further conditioning showed significantly higher TBS after acid cleaning than without acid cleaning (p=0.033). Among the groups repaired with Arabesk Top, a positive impact of acid cleaning was observed for CoJet wet air-abraded and conditioned groups with Futurabond U (p=0.005) and Scotchbond Universal (p=0.023), as well as for the Cimara-grinded and visio.link-conditioned groups (p=0.021). A negative impact of acid cleaning was detected for the CoJet dry air-abraded nonconditioned group (p=0.04) and CoJet wet air-abraded group in combination with One Coat Bond (p=0.036). The remaining groups exhibited no impact from acid cleaning on their TBS results (p>0.05).

Impact of intermediate agency

GrandioSo

Within CoJet dry air-abraded and nonacid-cleaned groups, conditioning using Scotchbond Universal resulted in significantly higher TBS than to nonconditioned groups and those conditioned with One Coat Bond (p<0.001). After acid cleaning, conditioning with One Coat Bond and lack of conditioning resulted in significantly lower TBS than conditioning with Futurabond U, Scotchbond Universal, or visio.link (p<0.001).

The wet-treated and nonacid-cleaned CoJet groups showed significantly higher TBS than the nonconditioned group (p=0.002), as did visio.link and Scotchbond Universal. Within the acid-cleaned groups, no conditioned group showed significantly the lowest TBS (p<0.001). The remaining conditioning methods showed no differences (p>0.05).

For the Cimara-grinded and nonacid-cleaned groups, no conditioning resulted in lower TBS than conditioning (p<0.001). Within the acid-cleaned groups, significantly higher TBS was found for Scotchbond Universal and visio.link compared to the nonconditioned group (p=0.001).

Arabesk top

Among the CoJet dry air-abraded and nonacid-cleaned groups, the group conditioned with visio.link showed significantly higher TBS than the nonconditioned groups (p=0.006). Within the acid-cleaned groups, the nonconditioned groups had significantly lower values than the groups conditioned with Scotchbond Universal (p<0.001). Conditioning using visio.link and Futurabond U caused a significantly higher TBS than the lack of conditioning or conditioning using One Coat Bond (p<0.001).

The CoJet wet-pretreated and nonacid-cleaned group showed significantly lower values without conditioning than the group conditioned with One Coat Bond and Scotchbond Universal and significantly higher TBS after conditioning using visio.link than without, One Coat Bond, or Futurabond U-conditioned groups (p<0.001). In contrast, within the acid-cleaned group, the lack of conditioning, as well as conditioning with One Coat Bond resulted in significantly lower values than conditioning with Futurabond U, visio.link, or Scotchbond Universal (p<0.001).

The Cimara-grinded group without acid cleaning and without conditioning exhibited significantly lower TBS than the groups conditioned with Futurabond U or Scotchbond Universal (p=0.001). Within the acid-cleaned groups, the nonconditioned group showed significantly lower TBS (p<0.001). The conditioning methods resulted in no differences within the acid-cleaned groups (p>0.05).

Impact of the resin composite

Repairing with GrandioSo results in significantly higher TBS compared to Arabesk Top within the CoJet wet air-abraded, acid-cleaned, and One Coat Bond-conditioned groups (p=0.001). Arabesk Top resulted in significantly higher TBS than GrandioSo for the Cimara-grinded, nonacid-cleaned, and nonconditioned groups (p=0.033) as well as those acid-cleaned and conditioned using One Coat Bond (p=0.004). All other groups showed no impact of the resin composite.

Failure types

The relative frequency of the failure types are shown as percentages in Table 3. According to the χ^2 test, significantly different failure types between the tested groups were observed (p<0.001). All groups showed predominant adhesiveness (13.3–100 %) or cohesiveness in their repair resin composite (0–86.7 %) failure. However, cohesive failures in the CAD/CAM resin, mixed failure, and prefailure were rarely observed.

Discussion

Material losses or small fractures of industrial polymerized CAD/CAM resin caused during the clinical period of wear must be repairable to allow for extended clinical use. Therefore, efficient bond properties should be generated between the CAD/CAM resin and repair resin composites. In this study, it was observed that the bond strength for CAD/CAM resin can be increased by surface pretreatment and an additional application of adhesive systems. Mechanical pretreatment principally cleans and increases the surface area, resulting in higher bond strength due to mechanical retention [21, 22]. Both pretreatment methods used in this study—air abrasion and grinding—resulted in an increase in surface area,

Table 3 Failure type analyses for all tested groups: (a) groups repaired using GrandioSo and (b) groups repaired using Arabesk Top

Treatment	Acid	Adhesive	Prefailure (%)	Adhesive (%)	Cohesive resin composite/ CAD/CAM polymer (%)	Mixed failure (%)
GrandioSo						
CoJet dry air-abraded	Yes	Futurabond U	0	40	53/7	0
		One Coat Bond	13	87	0/0	0
		Scotchbond Universal	0	73	27/0	0
		visio.link	0	67	33/0	0
		No conditioning	7	93	0/0	0
	No	Futurabond U	7	53	33/0	7
		One Coat Bond	7	87	7/0	0
		Scotchbond Universal	0	33	33/7	27
		visio.link	7	53	40/0	0
		No conditioning	0	100	0/0	0
CoJet wet air-abraded	Yes	Futurabond U	0	87	13/0	0
		One Coat Bond	0	80	20/0	0
		Scotchbond Universal	0	93	7/0	0
		visio.link	0	73	20/0	7
		No conditioning	0	100	0/0	0
	no	Futurabond U	7	60	33/0	0
		One Coat Bond	7	80	13/0	0
		Scotchbond Universal	0	67	33/0	0
		visio.link	0	87	13/0	0
		No conditioning	0	93	7/0	0
Cimara grinder	Yes	Futurabond U	7	27	60/0	7
		One Coat Bond	7	87	7/0	0
		Scotchbond Universal	0	53	40/7	0
		visio.link	7	60	33/0	0
		No conditioning	13	73	13/13	0
	No	Futurabond U	7	73	20/0	0
		One Coat Bond	0	87	13/0	0
		Scotchbond Universal	0	40	53/7	0
		visio.link	7	27	60/0	7
		No conditioning	47	53	0/0	0
Arabesk Top						
CoJet dry air-abraded	Yes	Futurabond U	0	40	53/0	7
		One Coat Bond	0	93	7/0	0
		Scotchbond Universal	0	40	60/0	0
		visio.link	0	27	67/0	7
		No conditioning	27	73	0/0	0
	No	Futurabond U	13	40	47/0	0
		One Coat Bond	0	73	27/0	0
		Scotchbond Universal	0	13	67/0	20
		visio.link	0	47	33/0	20
		No conditioning	0	67	7/0	27
CoJet wet air-abraded	Yes	Futurabond U	0	53	40/0	7
		One Coat Bond	0	93	7/0	0
		Scotchbond Universal	0	33	67/0	0
		visio.link	0	33	67/0	0
		No conditioning	7	93	0/0	0
	No	Futurabond U	0	53	47/0	0

Table 3 (continued)

Treatment	Acid	Adhesive	Prefailure (%)	Adhesive (%)	Cohesive resin composite/ CAD/CAM polymer (%)	Mixed failure (%)
		One Coat Bond	13	73	13/0	0
		Scotchbond Universal	0	40	53/0	7
		visio.link	0	47	53/0	0
		No conditioning	0	100	0/0	0
Cimara grinder	Yes	Futurabond U	0	47	40/0	13
		One Coat Bond	0	87	13/0	0
		Scotchbond Universal	7	80	7/0	7
		visio.link	0	20	67/0	13
		No conditioning	27	67	7/0	0
	No	Futurabond U	0	20	67/0	13
		One Coat Bond	13	67	20/0	0
		Scotchbond Universal	0	7	87/0	7
		visio.link	7	13	67/0	13
		No conditioning	13	87	0/0	0

inducing mechanical retention. However, the results showed that pretreatment with the CoJet system generated significantly higher bond strength than pretreatment with the Cimara grinder. It can be therefore assumed that air abrasion with a 3-bar pressure provides higher retentions (regarding surface area) than pretreatment with an Al_2O_3 grinder, and consequently, the bond strength results for treatment with 3-bar air abrasion are higher. Therefore, the first hypothesis—that pretreatment exerts no impact on bond strength—is rejected. While air abrasion by sand dust is not available in every dental practice, an Al_2O_3 grinder can be used anywhere quickly and easily. Although the bond strengths after using the Al_2O_3 grinder were significantly lower than those of air-abraded surfaces, these values were significantly higher compared to the groups that had not undergone surface conditioning, offering an alternative for repair. Surface pretreatment before the repair process should be performed in any case.

The results of this study demonstrate, furthermore, that an additional conditioning of the substrate with intermediate agents is necessary to achieve significantly improved adhesion between the CAD/CAM resin and resin composite. This indicates that micromechanical retention alone is not adequate to obtain a sufficient bond between the two materials. The CAD/CAM resins are industrially polymerized and present a higher degree of conversion than conventionally polymerized resins [23]. In spite of the low amount of unsaturated C-C bonds, the use of adhesive systems as intermediate agents significantly improved the bond strength. The efficiency of the analyzed intermediate agents was different, however, while the universal adhesives (visio.link, Scotchbond Universal, and Futurabond U) performed better than the adhesive that was based exclusively on methacrylic monomers (One Coat Bond). This variance in effectiveness recorded for the different adhesives should be attributed at least in part to the diversity of functional monomers included in the particular adhesive formulations. Universal adhesives such as Scotchbond Universal and Futurabond U contain silane or phosphoric acid monomers in addition to regular methacrylic monomers. This fact suggests a significant contribution to the bond of the silane or phosphoric monomers, which are able to prime the inorganic compound of the CAD/CAM materials that are made of nanoceramic particles embedded in a highly cured resin matrix. While approximately 80 wt% of the chemical composition of the CAD/CAM resins is attributed to nanoceramic particles (silica nanomers with a 20-nm diameter and zirconia nanomers with a 4–11-nm diameter), this result confirms the efficacy of universal adhesives in bonding to zirconia when air abrasion has already been applied [24]. As for visio.link, this adhesive does not contain phosphoric acid monomers, but rather high molecular weight acrylates such as pentaerythritol triacrylate ($C_{14}H_{18}O_7$) or pentaerythritol tetraacrylate ($C_{17}H_{20}O_8$). Acrylates are known to be more reactive than methacrylate. Nevertheless, visio.link and Scotchbond Universal induced comparable bond strength results, with the results of both superior to those measured for Futurabond U. Since the chemical composition of Scotchbond Universal and Futurabond U seems comparable, this difference might be attributed to a disparity in monomer amount. Particularly, for HEMA, a water-soluble OH-containing monomer present in both materials, a high amount of water was proven to be retained within the adhesive layer, which might adversely affect the latter mechanical strength and bonding effectiveness of the adhesives [25]. Since the data were collected after aging the specimens, differences in water uptake as a function of the HEMA amount might already have occurred. In summary, it was demonstrated that an additional application of adhesive systems leads to an increase of bond strength. Therefore, the second tested hypothesis—that an additional conditioning of the RNC surface has an effect on the TBS values—is rejected.

As for the resin composites used as repair materials in this study, their impact on TBS was low, but it was dependent on the surface pretreatment and phosphoric acid cleaning. The materials differed strongly in their mechanical properties, while the microhybrid Arabesk Top presented a lower indentation modulus than the nano-hybrid GrandioSo (16.09 vs. 24.23 GPa, measured after aging the materials similar to the repaired specimens), due to their different filler content (Table 1). A repair resin composite with a low viscosity might better wet the surface, generating fewer defects, but is also likely to shrink more. On the other hand, a high modulus of elasticity, as a consequence of increased filler content, is reflected in a lower volumetric shrinkage, as well as in increased shrinkage stress at the interface of the substrate-repair composite, thus affecting negatively the bond. These enumerated effects are contradictory, making the final effect difficult to predict. Within CoJet air-abraded specimens, a small advantage was observed when repairing with GrandioSo, while in the Al_2O_3-grinded groups, the opposite or no impact was confirmed.

This study used a post-repair thermal aging of 10,000 cycles between 5 and 55 °C with a dwell time of 20 s in each bath. Thermal cycling can influence TBS in two different ways. On one hand, the mechanical stress in the interface, caused by volumetric changes [26], may lead to cracks and result in lower bond strength. On the other hand, the thermal increase may enhance the post-polymerization of the luting area and result in higher bond strength [27]. Since, in this study, the specimens were thermally aged after allowing for post-polymerization for 24 h at 37 °C in distilled water, the first assumption might have proven accurate. Furthermore, several studies have stated that intraoral thermal changes occur due to the daily routines of eating, drinking [28, 29], and breathing [30]. At present, there is no systematic standardized procedure for fully mimicking in vitro testing conditions in the laboratory. However, laboratory thermocycling does provide a certain standardized and reproducible stress to all specimens. In this study, due to the high number of thermostat change cycles, it can be assumed that the aging process is quite similar to that found in clinical conditions. Although this in vitro study could not replicate all of the individual variations of intraoral exactly, it provides some hints for the reliable bond formation of CAD/CAM resin in dentistry. The in vitro bond strength tests assess the quality of adhesion. The tensile test chosen to determine the repair potential of CAD/CAM resin composites was proven to be more clinically relevant compared to shear bond strength [31]. Moreover, the macrotensile strength offer advantages compared to a microtensile strength test, since it allows for a sound specimen preparation with no additional mechanical preloading. Once a repair technique passes the in vitro testing, an in vivo test with a controlled, standardized study design should evaluate its long-term clinical performance.

Within the limitations of the present study, the following could be concluded:

– Air abrasion produced superior TBS compared to grinding of the surface with Al_2O_3 prior to repair (η_P^2=0.032, p<0.05) and the tested adhesive systems proved to be necessary intermediary agents for repairing aged CAD/CAM RNC substrates (η_P^2=0.273, p<0.05), while visio.link and Scotchbond Universal performed slightly better than Futurabond U.
– The use of repair resin composite showed a low but significant impact on the TBS (η_P^2=0.07, p<0.05).
– Phosphoric acid (p=0.154) or water contamination (p>0.05) of the air-abraded surface was proven not to affect repair bond strength.

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Conflict of interest The authors declare that they have no conflict of interest.

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