

## RESEARCH AND EDUCATION

# Repair bond strength and nanoleakage of artificially aged CAD-CAM composite resin



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Indirect composite resins are an alternative to direct composite resins for large restorations in posterior teeth as indirect materials facilitate the control of proximal contacts, anatomic form, esthetics, and polymerization shrinkage.<sup>1</sup> Computer-aided design and computer-aided manufacturing (CAD-CAM) high-density polymers may also offer additional benefits such as enhanced mechanical properties,<sup>2,3</sup> adequate wear resistance,<sup>4,5</sup> less discoloration,<sup>6</sup> and a higher degree of conversion with less residual monomer.<sup>7</sup> Moreover, when compared with ceramic materials, these polymers can be milled to a very thin dimension with low risk of chipping,<sup>3,8-11</sup> are not fired, have a high load-bearing capacity,<sup>5,12,13</sup> are less abrasive to the opposing enamel,<sup>5,14</sup> and are easier to repair in the case of failure.<sup>11,15</sup> These characteristics make CAD-CAM composite resin restorations suitable for clinical

### ABSTRACT

**Statement of problem.** The polymerization of computer-aided design and computer-aided manufacturing (CAD-CAM) composite resins during their manufacture enhances their physical properties and biocompatibility but might compromise their reparability.

**Purpose.** The purpose of this in vitro study was to determine the microtensile bond strength and nanoleakage (NL) of aged LAVA Ultimate (LU) CAD-CAM composite resin after different repair protocols.

**Material and methods.** Fifty-eight LU miniblocks were prepared, thermocycled (10 000 cycles, 5°C to 55°C), and assigned to 10 surface pretreatment and bonding protocols: (1) tribochemical silica coating (CoJet, CoJet Sand; 3M ESPE)+Scotchbond Universal Adhesive (SBU; 3M ESPE); (2) CoJet+silane (Si, ESPE Sil; 3M ESPE)+Adper Scotchbond 1 XT Adhesive (XT; 3M ESPE); (3) CoJet+10-methacryloyloxydecyl dihydrogen phosphate-based silane (MO; Monobond Plus; Ivoclar Vivadent AG)+XT; (4) CoJet+XT; (5) 30- $\mu$ m alumina airborne-particle abrasion (AL)+SBU; (6) AL+Si+XT; (7) AL+MO+XT; (8) AL+XT; (9) no pretreatment+SBU; and (10) no pretreatment+XT. All blocks were repaired using the Filtek Supreme XTE (3M ESPE) composite resin. Stick-shaped specimens (0.9 $\times$ 0.9 mm) were obtained and submitted to microtensile bond strength ( $\mu$ TBS) and %NL testing after 24 hours.  $\mu$ TBS data were analyzed with 1-way ANOVA, followed by the Tukey post hoc test, and NL data with nonparametric Kruskal-Wallis and Dunn tests ( $\alpha=.05$ ).

**Results.** For  $\mu$ TBS, CoJet, and AL pretreatments showed significantly higher mean  $\mu$ TBS, especially when used together with SBU. No pretreatment+XT yielded the lowest mean  $\mu$ TBS. For NL, marginal sealing improved significantly after the use of SBU regardless of the surface treatment. This improvement was only statistically different after tribochemical silica coating.

**Conclusions.** Airborne-particle abrasion with alumina particles, silica coated or not, together with the application of SBU resulted in the highest mean  $\mu$ TBS. The lowest %NL was recorded when aged LU blocks were repaired using SBU. (*J Prosthet Dent* 2019;121:523-30)

use, with a reported success rate of 85.7% after 24 months,<sup>16</sup> and for long-term interim restorations in complex rehabilitation treatments.<sup>2,8,17</sup>

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## Clinical Implications

Treating the surfaces with tribochemical silica coating or alumina airborne-particle abrasion is essential for repairing aged Lava Ultimate restorations. The use of a universal adhesive effectively increases repair bond strength and provides better sealing.

Lava Ultimate (LU; 3M ESPE) was the first CAD-CAM reinforced composite resin material introduced, followed by CERASMART (GC Corp), Shofu Block HC (Shofu), BRILLIANT Crios (Coltène), Katana Avencia (Kuraray Noritake Dental Inc), Brava Block (FGM), and AMBARINO High-Class (Creamed).

LU is a nanoparticulate prepolymerized composite resin that contains 4 to 11 nm of zirconia and 20 nm of silica nanoparticles agglomerated into clusters,<sup>18</sup> with a total filler content of approximately 80 wt% and a volume filler loading of 65 vol%.<sup>19</sup> The nanoparticles are treated with a silane-coupling agent that bonds the filler surface to the highly cross-linked polymer matrix.<sup>20,21</sup> The polymerization process is carried out under standardized high pressure and temperature, leading to a highly homogeneous internal structure.<sup>14,22</sup> Therefore, the manufacturers of LU claim that it offers ease of handling of a composite resin with a surface gloss and finish retention similar to those of porcelain.<sup>18</sup>

According to the manufacturer, LU is indicated for veneers, inlays, and onlays, with an internal retentive design element. The material is not indicated for complete crowns because there is potential for debonding.<sup>19,20</sup> Repairable failures of LU restorations may be a result of secondary caries or fracture.<sup>23,24</sup> The direct repair of defective restorations with composite resin is the preferred option because it is more conservative and less costly and time-consuming, reducing dental tissue loss and pulpal trauma.<sup>25-27</sup> The repair has similar or even increased longevity compared with a replacement restoration.<sup>28,29</sup>

During the manufacturing process, CAD-CAM composite resins are polymerized in a controlled environment to obtain a highly compact internal structure with fewer flaws and pores<sup>14</sup> and a higher degree of conversion with less residual monomer.<sup>20,30,31</sup> These characteristics are advantageous with regard to biocompatibility but make retention problematic and compromise repair predictability.<sup>20,30</sup> LU properties might change over time because it undergoes degradation after water immersion, cyclic loading, and thermocycling.<sup>32-36</sup>

Different materials and techniques can be used to roughen the intaglio surface with the objective of increasing the bonding area and enhancing the

micromechanical interlocking; these include hydrofluoric acid,<sup>37-40</sup> airborne-particle abrasion with aluminum oxide powder (27 to 50  $\mu\text{m}$ ),<sup>37-39,41-52</sup> or surface roughening with a coarse-grit diamond rotary instrument.<sup>37,43,48,53</sup> Other methods combine airborne-particle abrasion with an enhanced chemical interaction method, such as tribochemical silica coating, followed by the application of a silane.<sup>28,36,41,46,48,52,54</sup> A silane<sup>29,40</sup> and/or a dental adhesive might affect the in vitro repair bond strength.<sup>29,45,55-57</sup> Silanes establish a covalent bond to the resin matrix monomers by carbon double bond polymerization and to the inorganic fillers by siloxane bonds,<sup>58</sup> whereas dental adhesives improve the wettability of the composite resin surface.<sup>59</sup> The recent introduction of universal dental adhesives that contain silane and other functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) may simplify the bonding procedure.<sup>60,61</sup> Likewise, MDP-containing silanes are available.<sup>62,63</sup> All these new products might be alternatives for composite resin repair.<sup>41,64,65</sup>

The current literature on LU repair is limited<sup>20,66-69</sup> and mainly focused on assessing repair bond strengths to find the best repair protocols. The authors are unaware of studies comparing the effect of MDP- and silane-containing universal adhesives with the application of a silane as a separate step or with the use of an MDP-containing silane. Moreover, studies that determined the leakage of the interface after LU repair are lacking.

Therefore, the purpose of this in vitro study was to evaluate the effect of different protocols to repair aged LU on microtensile bond strength ( $\mu\text{TBS}$ ) and nano-leakage (NL). The null hypothesis tested was that different repair protocols, including surface treatments such as airborne-particle abrasion with alumina particles (coated with silica or uncoated), and different bonding procedures such as a silane followed by an adhesive, an MDP-containing silane, or a silane-containing universal adhesive would not influence  $\mu\text{TBS}$  or NL after LU repair.

## MATERIAL AND METHODS

The material used in the present study included 5 LU blocks and are listed in Table 1. Each CAD-CAM block was cut into 12 small blocks (6×6×5.5 mm) with a slow-speed, water-cooled diamond saw (IsoMet 5000; Buehler). The bonding surface of each specimen was ground with 600-grit SiC paper for 30 seconds (Buehler-Met II; Buehler) under water cooling using a grinding machine (Beta Grinder-Polisher; Buehler).

After ultrasonic cleaning in distilled water for 10 minutes (Ultrasonic Cleaner 3510 E-DTH; Branson), the blocks were submitted to an aging protocol that consisted of 10 000 thermocycles in distilled water baths at 5°C and 55°C, with a dwell time of 30 seconds in each bath.<sup>35,70,71</sup>

**Table 1.** Chemical composition of materials used

Material, Abbreviation, (Batch Number); Manufacturer	Composition
Lava Ultimate CAD-CAM restorative BL-LT for CEREC, LU, (N369384); 3M ESPE Dental Products	Bis-GMA, UDMA, Bis-EMA, TEGDMA with 80%wt 20-nm silica and 4- to 11-nm zirconia nanoparticles, and zirconia/silica nanoclusters
CoJet Sand, CoJet, (535723); 3M Deutschland GmbH Dental Products	Tribochemical silica coating with 30- $\mu$ m alumina particles modified by silica
RONDOflex Abrasive Powder, AL; KaVo Dental GmbH	27- $\mu$ m alumina particles
Scotchbond Universal Adhesive <sup>a</sup> , SBU, (533699); 3M Deutschland GmbH Dental Products	MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, and silane
ESPE Sil, SI, (532902); 3M Deutschland GmbH Dental Products	3-MPS silane and ethanol
Adper Scotchbond 1 XT <sup>b</sup> , XT, (N515260); 3M ESPE Dental Products	Bis-GMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system, a methacrylate functional copolymer of polyacrylic, and polyitaconic acids
Monobond Plus, MO, (S44734); Ivoclar Vivadent AG	Ethanol, 3-trimethoxysilylpropyl methacrylate, MDP, and disulfide acrylate
Scotchbond Universal Etchant, PA, (537103); 3M Deutschland GmbH Dental Products	32% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, and aluminum oxide
Filtek Supreme XTE <sup>c</sup> Universal Restorative A4B Body Shade, XTE, (N673584); 3M ESPE Dental Products	Bis-GMA, UDMA, TEGDMA, Bis-EMA, 20-nm silica, and 4- to 11-nm zirconia nanoparticles, and zirconia/silica nanoclusters.

MDP, 10-methacryloyloxydecyl dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; 3-MPS, 3-methacryloyloxypropyltrimethoxy; Bis-GMA, bisphenylglycidyl dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate. <sup>a</sup>Also known as Single Bond Universal in other countries. <sup>b</sup>Also known as Adper Single Bond Plus or Adper Single Bond 2 in other countries. <sup>c</sup>Also known as Filtek Supreme Ultra or Filtek Z350 XT in other countries.

The specimens were then divided into 10 experimental groups according to the surface pretreatment and/or the bonding protocol (Table 2).

A composite resin (Filtek Supreme XTE, A4B shade; 3M ESPE) was applied to the aged surface-treated blocks in three 2-mm-thick increments, photopolymerized for 10 seconds, and then for an additional 10 seconds on each free surface after the last increment. A light-emitting diode polymerization unit (Elipar S10; 3M ESPE) was used with an output of 1200 mW/cm<sup>2</sup>.

The repaired blocks were sectioned (IsoMet 5000) under water cooling in both the x- and y-directions perpendicular to the adhesive interface to obtain sticks with a cross-section of approximately 0.9×0.9 mm. Individual sticks were retrieved from each block, and peripheral sticks of each block were discarded. The specimens were stored in artificial saliva<sup>72</sup> for 24 hours at 37°C. Two sticks from each repaired block were selected for NL testing. The remaining sticks were used for microtensile bond strength ( $\mu$ TBS) testing.

All sticks were measured using a digital caliper with an accuracy of 0.001 mm (Mitutoyo Corp). The specimens were attached to a notched stainless steel Geraldelli jig<sup>73</sup> with cyanoacrylate resin (Loctite Super Glue-3 gel; Henkel) and stressed to failure in tension mode in a universal testing machine (Instron 3345; Instron Corp) at a cross-head speed of 1 mm/min. The  $\mu$ TBS values were calculated in MPa by dividing the load at failure by the cross-sectional bonding area.

Fractured sticks were observed by a single operator (C.A.) using a stereomicroscope (Olympus SZX7; Olympus Corp) at ×40 magnification to determine failure mode: (1) Adhesive, between LU and composite resin; (2) cohesive, within LU or composite resin; (3) or mixed, if fractures were simultaneously adhesive and cohesive.

**Table 2.** Groups tested

Surface Pretreatment (Mode of Use)*	Bonding Protocol (Mode of Use)
CoJet (tribochemical silica coating at 10-mm distance, for 10 s, and 200 kPa)	SBU (active application for 20 s, solvent evaporation for 5 s, and photopolymerization for 10 s with Elipar S10 LPU)
	SI (application for 60 s) and XT (2 layers following these steps: application for 15 s, solvent evaporation for 5 s, and photopolymerization for 10 s with Elipar S10 LPU)
	MO (application for 60 s) and XT
	XT
AL (airborne-particle abrasion at 10-mm distance, for 10 s, and 200 kPa)	SBU
	SI and XT
	MO and XT
	XT
None	SBU
	XT

AL, RONDOflex Abrasive Powder; MO, Monobond Plus; SBU, Scotchbond Universal Adhesive; SI, ESPE Sil; XT, Adper Scotchbond 1 XT, LPU, light-polymerization unit. \*Bonding surfaces cleaned as follows: After CoJet pretreatment, 96% ethanol for 15 s+air-drying for 10 s. After AL pretreatment, phosphoric acid (PA; Scotchbond Universal Etchant; 3M ESPE) for 15 s+water-spray for 15 s+air-drying for 10 s.

Sticks for NL assessment were coated with 2 layers of nail polish, except for the bonded interface and a 1-mm rim surrounding the interface. The sticks were subsequently placed in an ammoniacal 50% silver nitrate solution (pH=9.9) in darkness for 24 hours at 37°C. Then, the specimens were rinsed thoroughly with distilled water for 1 minute and immersed in a photo-developing solution (Rapid Access Ref 183 8379; Carestream Dental) for 8 hours at room temperature under a fluorescent light to reduce silver ions to metallic silver grains, as described by Tay et al.<sup>74</sup> The specimens were embedded in a low-viscosity epoxy resin (EpoxiCure 2; Buehler) using silicone molds. After polymerization, the specimens were

**Table 3.** Mean  $\pm$ standard deviation (SD) in MPa, number of specimens tested (n), and relative frequencies of failure in percentage

Surface Pretreatment	Bonding Protocol	Mean $\pm$ SD	N	Adhesive Failure	Cohesive Failure	Mixed Failure
CoJet	SBU	64.6 $\pm$ 19.9 <sup>ab</sup>	38	57.9	42.1	0.0
	SI+XT	55.3 $\pm$ 21.3 <sup>abc</sup>	40	92.5	5.0	2.5
	MO+XT	60.6 $\pm$ 17.0 <sup>abc</sup>	44	79.5	16.0	4.5
	XT	50.2 $\pm$ 16.8 <sup>c</sup>	42	85.7	14.3	0.0
AL	SBU	68.6 $\pm$ 20.3 <sup>a</sup>	25	88.0	12.0	0.0
	SI+XT	50.9 $\pm$ 19.2 <sup>c</sup>	35	88.6	8.6	2.8
	MO+XT	55.6 $\pm$ 20.7 <sup>abc</sup>	57	86.0	12.3	1.7
	XT	52.7 $\pm$ 22.0 <sup>bc</sup>	35	86.2	10.3	3.5
None	SBU	46.1 $\pm$ 27.0 <sup>bc</sup>	37	86.5	10.8	2.7
	XT	5.3 $\pm$ 12.1 <sup>d</sup>	49	100.0	0.0	0.0

AL, RONDOflex Abrasive Powder; MO, Monobond Plus; SBU, Scotchbond Universal Adhesive; SI, ESPE Sil; XT, Adper Scotchbond 1 XT. Different superscript letters within same column indicate statistically different mean  $\mu$ TBS ( $P < .05$ ).

wet-polished with SiC paper of decreasing abrasiveness (600-, 800-, 1000-, and 1200-grit) to a width of 0.4 mm. All specimens were then ultrasonically cleaned in distilled water for 10 minutes, air-dried, and mounted on aluminum stubs.

Interfaces were observed at 20 kV under a scanning electron microscope (XL30 ESE; FEI Company) in back-scattered mode. The presence of silver in the interface was confirmed using energy-dispersive X-ray spectroscopy. For each stick, 1 micrograph was made at  $\times 250$  (general view of the interface) and 3 micrographs at  $\times 1000$ . The first of these 3 micrographs was obtained at the center of the stick, whereas the other 2 were obtained on the left and right edges of the interface. A blinded evaluator measured the relative percentage of silver nitrate uptake (%NL) in the interface. Additional micrographs were made at  $\times 2500$  to characterize the NL pattern.

The  $\mu$ TBS data were analyzed using descriptive statistics, and a value of zero was assigned to pretesting failures.<sup>75</sup> Normality of data distribution was tested using the Shapiro-Wilk test. One-way ANOVA followed by the Tukey post hoc test was performed to determine the effects of different surface treatments on  $\mu$ TBS. The %NL scores were analyzed with the Kruskal-Wallis and the post hoc Dunn tests. Pretesting failures that occurred during the NL specimen preparation were excluded from the analysis. A statistical software program (IBM SPSS Statistics, v20.0; IBM Corp) was used for the analysis ( $\alpha = .05$  for all tests).

## RESULTS

Mean  $\mu$ TBS (MPa) and standard deviations are shown in Table 3. A normal distribution was found in 80% of the groups (8 out of 10). Thus, for all statistical tests, a normal distribution was assumed. One-way ANOVA revealed that different surface pretreatments and bonding protocols had a significant influence on LU repair bond

strength ( $P < .001$ ). Post hoc comparisons showed that specimens treated with 30- $\mu$ m alumina airborne-particle abrasion (AL) plus Scotchbond Universal Adhesive (SBU; 3M ESPE) achieved the highest mean  $\mu$ TBS, but not significantly higher than the means of groups using AL+Monobond Plus (MO; Ivoclar Vivadent AG)+Adper Scotchbond 1 XT Adhesive (XT; 3M ESPE), CoJet (CoJet Sand; 3M ESPE)+SBU, CoJet+ESPE Sil (SI; 3M ESPE)+XT, or CoJet+MO+XT. However, the use of SBU alone achieved statistically similar mean  $\mu$ TBS compared with groups using CoJet or AL, except for AL+SBU. The use of XT alone resulted in the lowest mean  $\mu$ TBS.

The percentages of failures recorded for the experimental groups are displayed in Table 3. The most prevalent failure was adhesive. Cohesive failures and mixed failures were rarely observed and mostly located in the repair composite resin. However, the CoJet+SBU group resulted in a similar percentage of cohesive and adhesive failures.

Median, first, and third quartiles of NL data are shown in Table 4. Surface pretreatment and bonding protocols had a significant influence on %NL within the interface between LU and Filtek Supreme XTE ( $P < .001$ ). The 3 experimental groups in which SBU adhesive was applied (CoJet+SBU, AL+SBU, and None+SBU) had a statistically similar %NL. Specifically, CoJet+SBU resulted in a significantly lower %NL (better sealing) than the other experimental groups.

For AL pretreatment, differences were found depending on the bonding protocol used. AL+SI+XT resulted in significantly higher %NL (worse sealing) than AL+SBU, which achieved significantly lower %NL. NL was observed in different patterns depending on the combination of silane and/or adhesive. For SBU, regardless of surface pretreatment, a rectangular silver accumulation with clear margins was located on the edge of the interface, whereas the rest of the interface was free of silver deposits (Fig. 1A). Silver spots that coalesced near the edge of the interface were identified when CoJet

**Table 4.** Percentiles 25, 50, and 75 of NL (%) for all experimental groups

Surface Pretreatment	Bonding Protocol	NL			N
		P25	P50	P75	
CoJet	SBU <sup>a</sup>	0.77	1	1.22	12
	SI+XT <sup>bc</sup>	6.16	13.48	38.43	12
	MO+XT <sup>bc</sup>	4.12	9.16	17.28	11
	XT <sup>bc</sup>	3.69	14.50	25.60	12
AL	SBU <sup>ab</sup>	1.00	1.60	7.59	9
	SI+XT <sup>c</sup>	15.16	24.41	57.96	10
	MO+XT <sup>bc</sup>	3.56	6.02	20.90	10
	XT <sup>bc</sup>	6.15	14.58	23.24	12
None	SBU <sup>ab</sup>	3.26	5.59	10.80	12
	XT <sup>bc</sup>	8.58	10.49	100.00	7

AL, RONDoflex Abrasive Powder; MO, Monobond Plus; SBU, Scotchbond Universal Adhesive; SI, ESPE Sil; XT, Adper Scotchbond 1 XT. Different superscript letters indicate statistically different %NL between experimental groups.

or AL+MO+XT was used (Fig. 1B). In the case of AL+SI+XT, the silver pattern was identified as a continuous line on the top of LU, with dendritic ramifications within the adhesive interface, as seen in Figure 1C. Coalescent silver spots were found on top of LU when silane was not used (Fig. 1D).

**DISCUSSION**

In the present study, the μTBS test was used to assess the repair bond strength, whereas the NL test was used to assess the marginal seal of the aged LU repair. The μTBS test is reliable, allowing a uniform and homogeneous loading stress distribution over small-sized specimens.<sup>26,39</sup> NL evaluation has been used to assess the integrity of dentin-adhesive bonds, as it can indicate the presence of voids or deficient adhesion.<sup>26</sup> Thermo-cycling was used to age the LU material artificially before repair<sup>20</sup> because the method, in addition to water storage, has the highest impact on the flexural properties of LU<sup>35,36</sup> and on its repair strength.<sup>54,69</sup> Filtek Supreme XTE was used to repair LU as both have identical composition and filler content.<sup>76</sup> The use of identical composite resin in repair procedures has been recommended.<sup>27</sup>

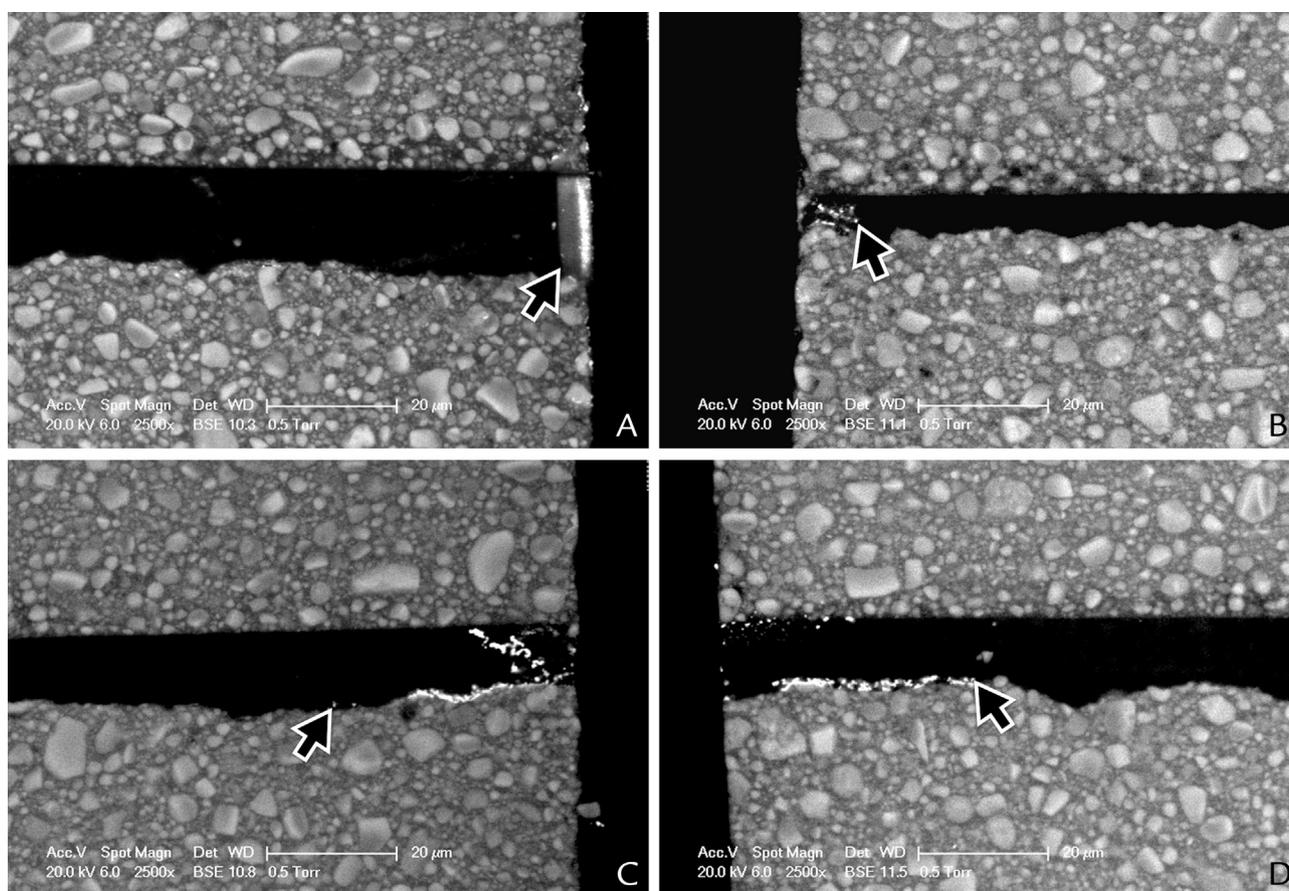
In the present study, both the airborne-particle abrasion methods resulted in higher repair mean bond strength than the use of XT without any pretreatment. Particle abrasion methods are considered an essential repair step, as they expose a fresh and clean rough surface that enhances micromechanical retention.<sup>20,45-47,66</sup> This positive effect of airborne-particle abrasion on LU repair, in comparison with no pretreatment or with rotary instrument roughening, has been described by other authors,<sup>20,40,51,66,69</sup> who reported that airborne-particle abrasion with alumina and tribochemical silica coating play the most important role in retaining luted LU.

Nevertheless, tribochemical silica-coating treatment (CoJet) did not increase mean bond strength over alumina airborne-particle abrasion in the present study. These results are consistent with those of Wiegand et al<sup>66</sup> and Loomans et al.<sup>69</sup> The similar particle size of both abrasion methods (27 μm for alumina and 30 μm for silica-coated alumina) might explain these results. Different particle size has a more relevant influence on bond strengths than the respective chemical composition.<sup>41,48</sup> In fact, the effect of abrasion with 50-μm alumina particles on the LU repair bond strength has been recently associated with the creation of irregular and rough surfaces with several cracks in the large filler particles and the resin matrix.<sup>49</sup> Therefore, smaller abrasive particles with low and controlled air pressure, as used in the present study, are recommended for LU repair.

The application of silane as a separate step after AL or CoJet was not associated with an increase in mean μTBS, which is consistent with previous studies.<sup>41,50</sup> The role of silane in composite resin repair is controversial.<sup>29</sup> Whether the use of silane enhances the chemical reactivity between individual silica filler particles in LU or with silica particles deposited on the surface by the silica-coating procedure, creating siloxane bonds between them, is unclear.<sup>63</sup> In a recent study, Loomans et al<sup>69</sup> did not observe any effect of applying silane on LU repair. In addition, a separate application of silane and adhesive results in a multiphase layer, which may introduce flaws in each application step,<sup>26</sup> making the interface more prone to leakage (Fig. 1C).

The additional application of a dental adhesive is required to improve the repair bond strength. Adhesives must be able to wet the roughened surface and establish an adequate interlock between the substrate and the composite resin used for repair.<sup>11,20,29,30,44,57</sup> This suggests that micromechanical retention alone does not result in adequate bond strengths between LU and the composite resin used to repair LU.

The application of SBU without any pretreatment resulted in a high mean μTBS (54 MPa), which was not statistically different from that obtained when SBU was applied after tribochemical silica-coating pretreatment. In agreement with this finding, Tantbirojn et al<sup>64</sup> reported that SBU might regain the failure strength of the original monolithic composite resin after composite resin repair. Universal adhesives can be used for direct restorations as well as for luting indirect restorations, reducing steps in the bonding protocol. SBU, in particular, contains MDP, which achieves direct bifunctional adhesion to surface oxides (including zirconia<sup>60</sup>) through a phosphate ester group and to the resin matrix through a methacrylate group.<sup>77</sup> As LU contains a high percentage of zirconia filler, the interaction between MDP and zirconia might be responsible for the high mean μTBS in the group in



**Figure 1.** Scanning electron micrographs of nanoleakage specimens. LAVA Ultimate phase is located in lower part of images and limit of silver intake marked with arrows. All specimens pretreated with CoJet or AL. Original magnification  $\times 2500$ . A, CoJet+SBU. B, CoJet+MO+XT. C, AL+SI+XT. D, AL+XT.

which only SBU was used,<sup>20,41,65</sup> which makes the additional use of a silane superfluous. Accordingly, Yoshihara et al<sup>60</sup> determined that the silane incorporated in SBU was not as effective as a separate silane primer or a silane freshly mixed with the adhesive. The bond strength to LU remained less stable, most likely, because the low pH of SBU promotes hydrolysis and dehydration condensation.<sup>60</sup>

Consequently, the chemical composition of the adhesive itself can influence LU repair, as the mean  $\mu$ TBS obtained with SBU alone was significantly higher than the mean  $\mu$ TBS obtained with XT. The latter resulted in the lowest mean  $\mu$ TBS of all groups (5 MPa). Similarly, different NL patterns were observed for SBU versus XT. Groups in which SBU was applied, alone or after CoJet or AL, exhibited an interface free of silver ions, with minimal silver accumulation at the edge of the specimen. Because data were collected only 24 hours after repair, this association cannot be extrapolated for longer wet-storage periods.

Even though the repair bond strength should be identical to the cohesive strength of the composite resin

to reestablish its original properties,<sup>48</sup> there is no practical way to include a control group.<sup>20</sup> In general, the results of the failure mode analysis showed predominant adhesive failures, whereas cohesive failures and mixed failures were more infrequent and located in the direct composite resin phase.<sup>20</sup> Because of the manufacturing process of LU blocks which leads to improved physical and mechanical properties, adhesive failures are expected to be more frequent than cohesive failures after LU repair. However, Loomans et al<sup>69</sup> reported more cohesive failures in the LU phase after submitting aged LU specimens repaired with Filtek Supreme XTE to microshear bond strength testing; this was in spite of higher cohesive resistance of aged and nonaged LU than that with Filtek Supreme XTE. These differences might be explained by the different tests used to assess repair bond strength.

Finally, the present study only evaluated the immediate repair bond strength to aged LU substrates with different protocols. However, because some of the materials used may have degraded after water storage, evaluation after aging the repaired specimens would be an avenue of further research.

## CONCLUSIONS

Based on the finding of this *in vitro* study, the following conclusions were drawn:

1. Micromechanical retention with airborne-particle abrasion methods, such as CoJet and AL, are essential for repairing aged LU.
2. The use of a universal adhesive after airborne-particle abrasion is recommended to increase the repair bond strengths and sealing ability of aged LU.

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