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RESEARCH AND EDUCATION

Influence of different evaluation pastes on the bond of resin cement to lithium disilicate ceramic: An in vitro study

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ABSTRACT

Statement of problem. Residue from evaluation pastes may remain on the bonding surface of indirect restorations, requiring specific cleaning protocols. However, it is not clear how much the composition of different evaluation pastes affects the bond strength to resin cement.

Purpose. The purpose of this in vitro study was to determine the effectiveness of using 37% phosphoric acid to clean different evaluation pastes and its effect on the long-term bond strength of lithium disilicate ceramics to resin cement.

Material and methods. Lithium disilicate slices (IPS e.max CAD) with simulated computer-aided design and computer-aided manufacturing (CAD-CAM) topography were crystallized, etched (20 seconds - 5% hydrofluoric acid -HF), and received the application of different evaluation pastes (CTRL – no contamination; AC – Allcem; NX – NX3; ML – Multilink; RX – RelyX). All specimens were actively cleaned for 60 seconds with 37% phosphoric acid, except for the CTRL group. After applying silane, resin cement cylinders were produced and light-polymerized. Micro-shear bond strength testing was carried out on half of the cylinders (n=40) at baseline (24 hours) and after aging (220 days of water storage at 37 °C and 25 000 heat cycles between 5 °C and 55 °C). Failure modes, surface topography via scanning electron microscopy (SEM), and chemical composition through energy-dispersive X-ray spectroscopy (EDS) were assessed. Statistical analysis used 2-way analysis of variance (ANOVA) and Bonferroni post hoc tests (α=.05).

Results. At baseline, the AC and NX groups exhibited bond strength similar to the CTRL group. All groups experienced a considerable decline in bond strength after aging, but there was no difference between the AC group and the CTRL group. In both scenarios, the RX and ML groups displayed the weakest bonds. SEM analysis revealed similar surface topography, confirming the EDS results, where similar elemental composition was observed among the groups.

Conclusions. Cleaning lithium disilicate surfaces with 37% phosphoric acid after contamination with an evaluation paste is not universally effective for the tested pastes. Some pastes leave a residue that still compromises bond strength in the long term. Therefore, alternative cleaning methods should be considered. (J Prosthet Dent 2026;135:580.e1-e7)

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Clinical Implications

Relying solely on phosphoric acid may be insufficient to eliminate different evaluation paste residue from lithium disilicate surfaces, potentially compromising bonding. Selecting an effective cleaning protocol is essential to ensure optimal and durable adhesive bonding between lithium disilicate ceramic and resin cement.

Lithium disilicate ceramics have been widely used in computer-aided design and computer-aided manufacturing (CAD-CAM) systems because of their high fracture strength,[1] stain resistance,[2] and translucency that mimics natural teeth.[3] These properties make them suitable for durable, esthetic, restorations with favorable long-term performance in single crowns.[4,5]

Their clinical longevity, however, depends on proper bonding protocols,[6] including the appropriate luting technique and material.[7] Hydrofluoric acid (HF) etching has been used on glass-ceramics before bonding, selectively dissolving the glassy phase.[8] While HF can be applied chairside, safety concerns have led to its restriction in some countries, limiting its use to controlled environments,[9–11] resulting in workflows where restorations are evaluated, then returned to the dental laboratory for etching, adding time and cost.

Following HF conditioning, a silane agent is typically applied to promote chemical adhesion between the ceramic and resin cement.[12–14] Adhesive cementation also improves mechanical performance,[6,15] and influences restoration shade.[16] To enhance shade predictability, manufacturers offer evaluation pastes matched to resin cements,[16–18] allowing shade verification before bonding. However, effective removal of these pastes, along with saliva, is essential to avoid compromised bonding.[19–24]

Evaluation pastes vary in composition, often based on polyethylene glycol, glycerol, or water-soluble agents, designed for removal with water, alcohol, or in ultrasonic baths.[25] In vitro studies have examined how these pastes affect bonding to lithium disilicate and how different cleaning protocols interact with surface treatments.[19,26–29] Pilecco et al[27] reported that 37% phosphoric acid applied for 60 seconds removed glycerin-based paste residues and preserved bond strength and fatigue behavior.[30] Santos Junior et al,[19] using scanning electron microscopy (SEM), reported that cleaning effectiveness was paste-dependent, reinforcing the need for further investigation.

Given the variation in paste formulations, a universal cleaning protocol may not be possible. This study aimed to evaluate the effectiveness of 37% phosphoric acid in cleaning different evaluation pastes and its impact on the long-term bond strength between lithium disilicate ceramics and resin cement. The null hypotheses were that phosphoric acid cleaning would result in similar bond strength across paste groups and that aging would not reduce bond strength regardless of the paste used.

MATERIAL AND METHODS

The materials used are detailed in Table 1. The experimental design consisted of groups divided according to commercially available evaluation pastes at 5 levels and aging at 2 levels. The primary response variable was the micro-shear bond strength (μSBS) between resin cement and ceramic, as described in Table 2. A statistical software program (G*power v. 3.1.9.7; Heinrich-Heine Universität Düsseldorf) with the following parameters: effect size f=0.52, α=.05, power=.80, actual power=.80 was used to calculate the sample size based on data from a pilot study (n=12). A minimum of 39 resin cement cylinders were required, and a sample size of 40 resin cylinders was assigned to each group, distributed across 10 ceramic slices per group, with 8 cylinders per ceramic slice (4 to be tested at baseline and 4 after aging).

Prefabricated lithium disilicate blocks (IPS e.max CAD; Ivoclar AG) were sliced into dimensions of 12.4×14.5×2 mm under continuous water-cooling using a precision cutting machine (Isomet 1000; Buehler). To standardize surface roughness, specimens were polished under water-cooling using silicon carbide (SiC) abrasive papers (#400, #600, and #1200 grit; Norton Saint-Gobain) on a polishing unit (EcoMet/AutoMet 250; Buehler). A standardization protocol was then applied to the bonding surface to simulate the roughness after CAD-CAM milling by polishing with #60-grit SiC paper (Norton Saint-Gobain) for 15 seconds along both the x- and y-axes of each specimen.[31] The assessment of surface roughness was conducted before the crystallization process using a contact stylus profilometer (SJ-410; Mitutoyo). Three measurements were made along both the x- and y-axes of each ceramic slice, and the mean surface roughness (Ra) and maximum peak-to-valley height (Rz) were calculated to confirm uniform roughness across specimens, in accordance with International Organization for Standardization (ISO) 21920–2:2021 standard.[32] The slices were subsequently crystallized in a furnace (Vacumat 6000 MP; VITA Zahnfabrik) following the manufacturer's guidelines: starting at 403 °C, the temperature was raised by 90 °C/minute to 820 °C, followed by a second increase at 30 °C/minute to a final temperature of 840 °C, which was held for 15 minutes. A vacuum was applied between 450 °C and 840 °C. Following crystallization, the ceramic slices were randomly

Table 1. Material, brand name, manufacturer, composition, and batch number

Material	Brand name	Manufacturer	Composition	Batch Number
Lithium disilicate-based glass-ceramic	IPS e.max CAD LT	Ivoclar AG	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, Al_2O_3, MgO, coloring oxides	Z02TK1
37% phosphoric acid	N-Etch Etching Gel	Ivoclar AG	37% phosphoric acid	Z01KSX
5% hydrofluoric acid	Condac porcelana	FGM	5% hydrofluoric acid	030523
Silane agent	Monobond N	Ivoclar AG	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulfide methacrylate	Z04OSJ
Dual polymerization resin cement	Multilink N Transparent	Ivoclar AG	Barium glass, ytterbium trifluoride, Bis-EMA, HEMA, Bis-GMA, Si-Zr mixed oxide, barium aluminum fluorosilicate glass, UDMA, highly dispersed silicon dioxide. Inorganic filler content ~40 vol%. Particle size of inorganic fillers 0.15–15.5 μm	Z02WJ4
Evaluation paste	Allcem Veneer Try-In OW	FGM	Polyethylene glycol, silica	100123
Evaluation paste	NX3 Nexus Third Generation Try-In Gel	Kerr Australia Pty Limited	Glycerol, silica, amorphous, fumed, cryst. – free glass, oxide, chemicals, titanium dioxide	9458902
Evaluation paste	Multilink Automix Try-In W	Ivoclar AG	Glycerin, mineral fillers, dyes	Z04XH6
Evaluation paste	RelyX Try-In Paste WOT	3M ESPE	Polyethylene glycol, ceramic powder, titanium oxide	7614WOT

Bis-EMA, ethoxylated bisphenol A-glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenol A-glycol methacrylate; UDMA, urethane dimethacrylate

Table 2. Experimental design and division of groups tested

Group	Surface Treatment	Evaluation Paste	Cleaning Method	Surface Treatment	Aging*	Analysis
CTRL	5% hydrofluoric acid (20 s)	-	37% phosphoric acid actively (60 s)	Silane agent (60 s)	No Yes	μSBS Bond strength (n=40)
AC		Allcem Veneer evaluation paste			No Yes	Topography (n=1) EDS (n=1)
NX		NX3 Nexus evaluation gel			No Yes	
ML		Multilink Automix evaluation paste			No Yes	
RX		RelyX evaluation paste			No Yes	

* 25 000 thermocycles + 220 days of water storage.

assigned to experimental groups using a random sequence generator (random.org/sequences).

The crystallized ceramic slices were embedded in acrylic resin (JET; Clássico) using polyvinyl chloride cylinders (PVC; Krona), leaving the bonding surface free. The surface treatment protocol is detailed in Table 2. Each specimen was subjected to an initial cleaning process using an ultrasonic bath with isopropyl alcohol for a duration of 5 minutes. This was followed by etching with a 5% hydrofluoric acid solution (Condac Porcelana 5%; FGM) for 20 seconds, followed by rinsing with an air-water spray for 30 seconds. The evaluation pastes (CTRL – no contamination; AC – Allcem; NX – NX3; ML – Multilink; RX – RelyX; Table 1) were spread onto the bonding surfaces of the experimental groups using a spatula. The specimens were then positioned with the contaminated surface in contact with a clean glass plate and loaded with 2.5 N for 5 minutes. After this period, the glass plate was removed, and the residual paste was cleaned off using an air-water spray for 30 seconds. Specimens in the control group were not coated with the evaluation paste. The contaminated surfaces were gently air-dried and actively cleaned with 37% phosphoric acid (N Etch Etching Gel; Ivoclar AG) for 60 seconds, followed by rinsing with an air-water spray for 30 seconds, and final air-drying. Then, a silane coupling agent (Monobond N; Ivoclar AG) was applied to the ceramic surface (60 seconds for reaction). Starch matrices (1 mm in height; 1.13 mm internal diameter) (Isabela; M. Dias Branco S.A. Indústria e Comércio de Alimentos) were then fixed onto the treated ceramic surfaces using sticky wax (Lysanda). A dual-polymerizing resin cement (Multilink Automix; Ivoclar AG) was mixed in a 1:1 ratio on a pad for 10 seconds and introduced into the starch matrices using a spatula. A dental explorer was employed to reduce the occurrence of bubbles or voids in the resin cement which was light polymerized (Radii-Cal; SDI) for 40 seconds at 1200 mW/cm², with the light tip positioned directly against the resin cement cylinders. The specimens were immersed in distilled water and stored at 37 °C for 24 hours. Following storage, the starch matrices were removed, and each resin cement cylinder was inspected for the presence of bubbles, defects, or voids. Forty resin cement cylinders from each group were evaluated after 24 hours of water storage to establish a baseline (short-term aging), while the other 40 underwent long-term aging, comprising 220 days of distilled water storage at 37 °C followed by 25 000 thermal cycles[33] with a 30-second dwell in water baths at 5 °C and 55 °C, with a 5-second transfer interval (521–6D, Ethik Technology; Nova Ética).

In the μSBS test, each PVC cylinder containing a ceramic slice was placed in a universal testing machine (EMIC DL-2000; EMIC). The wire-loop technique used

a Ø0.2 mm stainless steel wire placed in proximity to the ceramic interface, ensuring alignment of shear force on each resin cement cylinder. Shear force was applied at a crosshead rate of 1 mm/minute until failure occurred. Bond strength (MPa) was determined by dividing the failure load (N) by the bonded area (1.13 mm^2 for all specimens). Fractured specimens were evaluated using a stereomicroscope (Stereo Discovery V20; Carl Zeiss) at ×50 magnification to categorize the failure mode as adhesive (predominantly interfacial failure, affecting >50% of the bonded area) or cohesive (failure within the resin cement, affecting >50% of the cylinder area). All classifications were performed by a single trained examiner (C.P.).

Energy-dispersive X-ray spectroscopy (EDS) integrated with a scanning electron microscope (SEM) (VEGA-3G; Tescan) featuring a secondary electron detector was employed to evaluate the chemical composition of an additional specimen from each group. The specimens were gold sputter-coated and analyzed at ×200 magnification under an acceleration current of 30 mA for 120 seconds. Additional specimens were prepared to evaluate the surface topography and verify the presence of evaluation paste remnants. These were analyzed under SEM at ×250 and ×1000 magnification using the same imaging parameters.

A software program (IBM SPSS Statistics, v21; IBM Corp) was used for all statistical analyses, and for bond data analysis, the resin cement cylinder was used as the experimental unit. As reported by Blanca et al,[34] the outcomes of 2-way analysis of variance (ANOVA) are robust to violations of assumptions such as normality and homoscedasticity. Therefore, the data were rank transformed to enable a 2-way ANOVA, with evaluation paste and storage condition as the independent variables. For pairwise comparisons, the Bonferroni post hoc test was applied. Only data from specimens exhibiting adhesive failures were included in the statistical analysis. Descriptive statistics were used to report failure mode distributions and EDS data. The SEM topographic images were analyzed qualitatively.

RESULTS

The 2-way ANOVA revealed that both the type of evaluation paste ($P<.001$; F=39.89) and the aging process ($P<.001$; F=149.94) significantly influenced bond strength. No substantial interaction was observed between the 2 variables (evaluation paste × aging) ($P=.176$; F=1.66). The μSBS values and the corresponding failure modes for each group are listed in Table 3.

Under baseline conditions, the AC and NX groups demonstrated adhesion data statistically similar to the control group, CTRL ($P>.999$), and outperformed those of the RX and ML groups ($P<.005$). The aging process considerably decreased bond strength across all groups ($P<.001$). After aging, only the AC group maintained statistically similar values to the CTRL group ($P=.405$), while NX showed intermediate performance. In contrast, the RX and ML groups exhibited the lowest bond strength values ($P<.02$). Most failures across all groups were adhesive (86.5% to 98.9%), with only a small percentage of cohesive failures observed (1.1% to 13.5; Table 3).

EDS analysis corroborated that contaminants were not detected at the elemental level. Similar atomic compositions were observed across all groups (Table 4), suggesting effective chemical decontamination regardless of the evaluation paste used. Lithium disilicate in all groups exhibited consistent topography, characterized by the typical rough pattern of CAD-CAM milling (Fig. 1). Ceramic surfaces contaminated with evaluation paste and subsequently cleaned with 37% phosphoric

Table 3. Micro-shear bond strength values * (mean, standard deviation), 95% confidence intervals, failure mode distribution (%), and number of specimens lost after aging (premature debondings) for each evaluation paste group

Group	Baseline		Aging		Failures		
	Mean (SD)	95% CI	Mean (SD)	95% CI	% Adhesive Failure	% Cohesive Failure	Number of Aging Failures
CTRL	25.89 (6.14)^{Aa}	24.0 – 27.8	19.95 (6.05)^{Ab}	18.2 – 21.7	86.54	13.46	7
AC	25.85 (7.40)^{Aa}	23.7 – 28.1	16.45 (6.90)^{ABb}	14.6 – 18.3	93.52	6.48	2
NX	23.21 (6.26)^{Aa}	21.4 – 25.0	15.14 (4.95)^{BCb}	13.7 – 16.6	93.20	6.80	1
ML	17.90 (6.91)^{Ba}	15.9 – 19.9	11.64 (8.15)^{CDb}	9.1 – 14.2	98.90	1.10	7
RX	17.50 (7.52)^{Ba}	15.4 – 19.6	8.58 (6.25)^{Db}	6.7 – 10.5	97.75	2.25	6

*Only specimens with predominantly adhesive failures included in bond strength analysis.

For each storage condition (baseline or aging), different uppercase letters within same column indicate statistically significant differences between groups ($P<.05$).

Different lowercase letters within same row indicate significant difference between baseline and aged values within each group ($P<.05$).

Table 4. Chemical analysis of surface composition (% elementary atomic content) after application of evaluation pastes removal with 37% phosphoric acid

Element	CTRL	AC	NX	ML	RX
Group					
O	75.56	80.17	76.87	75.93	78.62
Si	17.68	17.10	17.25	20.61	17.87
K	1.07	0.96	0.97	1.37	1.05
Al	0.98	1.03	1.04	1.16	1.00
P	0.69	0.63	0.60	0.77	0.68
Ce	0.13	0.11	0.10	0.15	0.12
C	3.90	-	3.15	0.02	0.66

acid exhibited surface textures closely resembling those of the uncontaminated group, with no visible evaluation paste residue.

DISCUSSION

This study explored, under in vitro conditions, the effectiveness of 37% phosphoric acid in removing different evaluation pastes from lithium disilicate ceramics and its influence on their long-term bond strength to resin cement. The results indicate that the cleaning efficacy of 37% phosphoric acid varied depending on the composition of the evaluation paste. SEM and EDS analyses confirmed the absence of visible evaluation paste contaminants; however, statistically significant differences in bond strength were still noticed. Consequently, the null hypotheses that phosphoric acid cleaning would result in similar bond strength across paste groups and that aging would not reduce bond strength regardless of the paste used were rejected, as all groups revealed a significant reduction in bond strength after aging.

Evaluation procedures have been routinely performed to verify color matching and marginal adaptation.[16] If evaluation pastes are not completely removed, the long-term integrity of the adhesive interface may be compromised.[19,26,27] Evaluation procedures may leave behind an organic layer of salivary proteins and paste residues that has been reported to be resistant to simple water rinsing.[20] Therefore, various decontamination strategies have been proposed, most commonly involving water, ethanol, phosphoric acid, or HF etching.[19,22,25–27]

The present data highlighted the potential risk associated with residual contaminants even when they are not visible on the ceramic surface, especially after HF etching. Contaminants may be more difficult to remove from the etched ceramic because of the increased roughness and contact area.[13,22] Despite manufacturers' claims that evaluation pastes are water-soluble and can be easily removed, their organic components and colorants could penetrate or absorb into the surface, requiring more rigorous decontamination protocols. Clinicians should consider selecting pastes that are less prone to interfering with adhesion and, when needed, adopt alternative or adjunctive cleaning methods, such as ethanol, an ultrasonic bath, or even consider the use of evaluation paste before HF etching.

The present findings were consistent with a previous study reporting that the effectiveness of paste removal is

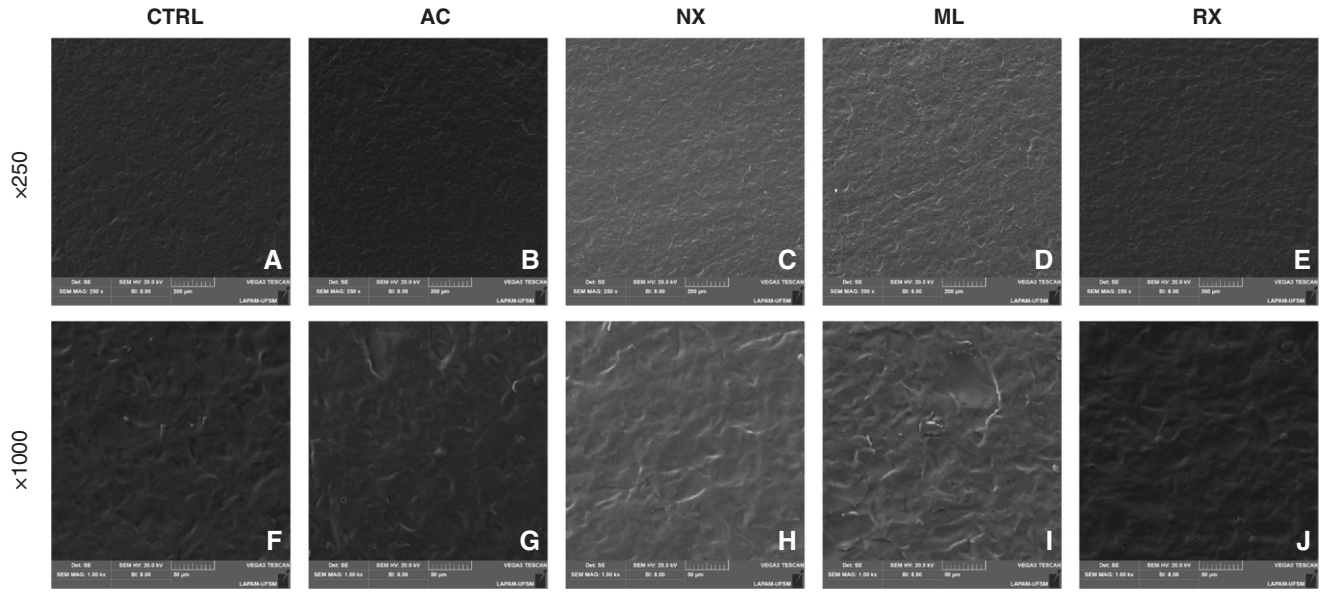

Figure 1. Scanning electron microscopy (SEM) images (original magnification ×250 and ×1000) of lithium disilicate ceramic surfaces after contamination with different evaluation pastes and subsequent cleaning with 37% phosphoric acid. All groups exhibited similar topography, characterized by roughened surface pattern typical of CAD-CAM simulated milling. No visible residues detected, and surface morphology closely resembled that of uncontaminated control group (CTRL), supporting apparent efficacy of cleaning protocol.

paste-specific.[19] While 37% phosphoric acid was effective for AC and NX, yielding bond strengths statistically similar to the uncontaminated control at baseline ($P>.999$), ML and RX showed significantly reduced performance ($P<.005$), both initially and after aging. These results support prior findings indicating that specific paste components, such as glycerol derivatives, dyes, or viscosity modifiers, may hinder decontamination.[22,28] Notably, even though the SEM and EDS results confirmed similar surface morphology and elemental composition across groups, the lower μSBS values for ML and RX suggested the presence of chemical or physical residues not detectable through visual or elemental analysis alone. Unfortunately, manufacturers provide insufficient detail about the composition and concentration of ingredients, while factors like viscosity may also affect both the effectiveness of cleaning procedures and the quality of the adhesive bond. One of the limitations of using EDS for chemical analysis lies in its poor sensitivity to trace elements and its inability to reliably detect elements lighter than sodium, such as oxygen, hydrogen, and carbon – key constituents in the composition of evaluation pastes.[35] Additionally, as reported by Newbury and Ritchie,[36] successful quantitative EDS analysis requires the sample surface to be highly polished, with a surface roughness below 50 nm, to minimize geometric effects during detection. In contrast, the present study evaluated lithium disilicate specimens with surfaces that mimicked CAD-CAM roughness and were further modified by hydrofluoric acid etching. This combination likely introduced geometric artifacts,[37,38] especially influencing the detection of low-energy versus high-energy X-ray photon peaks. Additional chemical analysis, such as mid-infrared (MIR), far-infrared (FTIR), as well as Raman spectroscopy, should be considered in future studies to better illustrate such mechanisms.

Aging caused a substantial decrease in bond strength for all groups ($P<.001$), reflecting well-known effects of hydrolytic degradation and thermal stress.[6,8] The AC group was the only one to maintain statistically comparable bond strength to the control after aging ($P=.405$), suggesting that its composition may facilitate more efficient removal using 37% phosphoric acid. This result emphasized the need not only for an effective cleaning method but also for the selection of evaluation pastes that do not compromise the adhesive interface over time.

Among decontamination methods, Pilecco et al[27] demonstrated that actively applying 37% phosphoric acid can effectively remove residual evaluation paste and maintain bond strength. Phosphoric acid presents advantages such as ease of use and cost-effectiveness, performance that has been validated in previous studies.[20,23,24,27,29] However, the varied responses across the tested pastes reinforce that phosphoric acid cannot be considered a universal cleaning solution for ceramic, as paste composition significantly influences cleaning effectiveness.

The lithium disilicate ceramics were etched before evaluation paste application. Although not ideal, this reflects common clinical situations in practices where restorations are delivered pre-etched from dental laboratories because of HF handling restrictions.[9,11] In such situations, intraoral evaluation is conducted on already etched surfaces, as simulated here. However, the etched surface may retain more residue because of its increased roughness and surface area. Etching with HF after evaluation might reduce the impact of contaminants or even serve as a cleaning method.[27]

Other limitations of the study included that the aging protocol employed did not include intermittent functional loading or the complex dynamics of the oral environment. Additionally, while the CAD-CAM surface was simulated using standardized flat specimens, these did not completely replicate the geometry and variability of an anatomic milled restoration, which may limit direct clinical application. EDS presents intrinsic limitations, particularly in detecting elements lighter than sodium (Na).[35] Furthermore, its accuracy is compromised when analyzing rough surfaces.[36] The authors are unaware of a previous study that investigated the influence of different evaluation paste compositions on the bond strength of lithium disilicate ceramics following phosphoric acid cleaning. The implementation of an extended aging protocol, which included 220 days of water storage and 25 000 thermal cycles, significantly enhanced the clinical relevance of the findings by demonstrating the long-term stability of the adhesive interface.

Future studies should investigate alternative cleaning protocols, consider a broader range of paste formulations, and add clinically representative geometries to improve relevance and applicability.

CONCLUSIONS

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

1. The use of 37% phosphoric acid cannot be regarded as a universally effective cleaning method for lithium disilicate surfaces following contamination with distinct evaluation pastes.
2. While some products (such as, Allcem and NX3) demonstrated bond strength values comparable to the control at baseline, Multilink and RelyX consistently showed reduced performance, indicating that paste composition plays a critical role in cleaning efficacy.
3. All groups exhibited a statistically significant decrease in bond strength following long-term aging, highlighting the necessity of effective cleaning protocols to preserve the long-lasting integrity of adhesive restorations.

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