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Effect of luting agent on the load to failure and accelerated-fatigue resistance of lithium disilicate laminate veneers

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ABSTRACT

Objective. The aim of this study was to investigate the influence of the luting agent on the application of laminate veneers (LVs) in an accelerated fatigue and load-to-failure test after thermo-cyclic aging.

Methods. Sound maxillary central incisors (N = 40) were randomly divided into four groups to receive LVs ($Li_2Si_2O_5$) that were adhesively bonded: Group CEMF: Adhesive cement (Variolink Esthetic LC), fatigue test; Group CEMLF: Adhesive cement, load-to-failure test; Group COMF: Resin composite (Enamel HFO), fatigue test; Group COMLF: Resin composite, load-to-failure test. The specimens were thermo-mechanically aged (1.2×10^6 cycles at 1.7 Hz/50 N, 8000 cycles 5–55 °C) and then subjected to either accelerated fatigue (5 Hz, 25 N increasing after each 500 cycles) or load to failure (1 mm/min). Failure types were classified and data analyzed using chi-square, Kaplan Meier survival, Log Rank (Mantel–Cox) and independent-samples t-test.

Results. After thermo-mechanical aging, fracture resistance ($p < 0.000$) was higher in the composite groups. Kaplan Meier survival rates showed significant difference ($p < 0.001$) between the composite (mean load: 1165 N; mean cycles: 22.595) and the cement groups (mean load: 762.5 N; mean cycles: 14.569). The same differences were observed in the load to failure test (cement M = 629.4 N, SD ± 212.82 and composite M = 927.59 N, SD ± 261.06); $t(18) = -2.80$, $p = 0.01$. Failure types were observed as fractures and chipping in group CEMF, all other groups were predominantly adhesive failures between the luting agent and the laminate veneer.

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Significance. The delivery of laminate veneers using a direct restorative composite rather than a resin cement resulted in significantly less chipping and fractures, higher fracture strength in both accelerated fatigue and load-to-failure.

1. Introduction

Laminate veneers (LV) are indicated as minimal invasive treatment options as an alternative to full coverage crowns. Since retention of the LV restorations does not rely on mechanical retention principles, durable adhesive luting of such restorations is crucial for long-term clinical success [1,2]. In clinical studies, survival rates of ceramic LVs range between 82 and 96% in 10–21 years [3–8]. Fractures of ceramic (5.6–11%) and marginal defects (12–20%) are the main reasons for failure [3,4,8–12]. Successful luting increases the retention, fracture resistance of the tooth and the restoration, and reduces the incidence of micro-leakage [2,13]. Adhesively bonded restorations offer the advantage of sealing the margins of the restorations, while solubility of cements is avoided. Also, adhesive luting of bonded restorations does not only provide minimal invasive restorations but also reinforces the glassy matrix ceramics [1].

For the conditioning of glassy matrix ceramics, hydrofluoric acid (HF) etching followed by the application of a silane coupling agent is a well established method [14–17]. For the luting of LVs, in most laboratory and clinical studies a photo-polymerized resin composite is suggested [2,5,8,18–21]. This kind of resin luting agent has some advantages over dual-polymerized ones. Photo-polymerized resin cements have better handling properties and they allow increased time for the clinician to seat the restoration. Furthermore, in some studies, with photo-polymerized resin materials increased bond strength was reported when compared to dual-polymerized resin cements [22–28]. In a study by Kameyama et al. [22], a dual-polymerized resin cement was compared with a direct composite as a luting material for ceramic inlays in a micro-tensile bond-strength test. The direct resin composite experimental group resulted in ca. 30 MPa of bond strength with only 1 pre-test failure whereas dual-polymerized resin composite cement delivered values below 10 MPa with almost half of the specimens presenting pre-test failures [22].

When testing durability of restorative materials in a laboratory setting, different aging methods are proposed. Besides different aging protocols such as water storage, thermocycling or thermo-mechanical aging, two different methods for fracture testing could be applied, namely load to failure test or accelerated fatigue test [26]. No consensus is available as to which method of durability test should be used to simulate the intra-oral situation ideally.

The objectives of this study were to (a) compare two different LV luting agents, (b) compare the outcome of the two different test methods on the survival of the bonded laminate veneers. The following null-hypothesis were tested: (a) aging would not have a significant effect on ceramic LVs luted with two resin composite materials (b) different luting materials would not have an influence on the survival rate or fracture resistance, (c) the test method would not have an influence on the outcome of the LVs.

2. Material and methods

2.1. Experimental groups and specimen preparation

The brands, types, main chemical compositions, manufacturers and batch numbers of the materials used for the experiments are listed in Table 1. Schematic description of the experimental design is presented in Fig. 1.

Sound human maxillary central incisors (N = 40) of similar size, free of restorations and root canal treatment were selected from a pool of recently extracted teeth. All teeth were screened on the presence of cracks under ultraviolet light and those with cracks were eliminated and replaced with new teeth. The teeth were then randomly divided into 4 groups (n = 10).

CEMF: Ceramic LV, Photo-polymerizing luting agent, accelerated fatigue test.

CEMLF: Ceramic LV, Photo-polymerizing luting agent, load-to-failure test.

COMF: Ceramic LV, Restorative resin composite, accelerated fatigue test.

COMLF: Ceramic LV, Restorative resin composite, load-to-failure test.

Prior to the LV preparation, impressions were made using a condensation silicone (Provil Novo putty fast set, Heraeus, Hanau, Germany) in order to obtain moulds for the provisional restorations. Window type tooth preparations without incisal overlap were made under an optical microscope (OPMI pico, Zeiss, Oberkochen, Germany). After marking the preparation outline, depth cuts of 0.3 mm were made (801-014, Komet, Besigheim Germany), preparations were finalized using a round-ended tapered diamond chamfer bur (879m-014 FG, Komet, Besigheim, Germany). The preparations ended completely in enamel, 1 mm above the cemento-enamel junction. Smooth margins were created to prevent stress concentration zones using finishing discs (Sof-Lex Contouring and Polishing Discs, 3M ESPE, St Paul, Minnesota, USA). After preparations were finished and enamel surfaces were polished, impressions were made using an polyvinyl-silicon impression material (Aquasil Ultra Heavy and XLV, Dentsply, Milford, USA) and these were checked for irregularities under an optical microscope (×10 magnification, OPMI pico, Zeiss). Provisional LVs (Protemp 4, 3M ESPE, St Paul, Minnesota, USA) were made and applied using a spot etch technique where etching was performed for 10 s in the cervical and incisal part of the preparation. After adjusting the temporary restorations using

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Table 1 – The brands, types, chemical compositions, manufacturers and batch numbers of the materials used for the experiments.

Brand	Type	Chemical composition	Manufacturer	Batch number
Top dent	Etching agent	38% Phosphoric acid	DAB, Malmö, Sweden	140919, 140128, 141031
Universal adhesive	Universal adhesive	2-Hydroxyethyl methacrylate, bis-GMA, ethanol, 1,10-decandiol dimethacrylate, methacrylated phosphoric acid ester, campherquinone, 2-dimethylaminoethyl methacrylate, ethanol	Ivoclar Vivadent, Schaan, Liechtenstein	T28040
IPS Empress etching gel	Ceramic etching gel	<5% Hydrofluoric acid	Ivoclar Vivadent	T34823
Monobond Plus	Silane coupling agent	Ethanol, 3-trimetho-xysilsylpropylmethacrylaat, methacrylated phosphoric acid ester	Ivoclar Vivadent	T21454
Enamel Plus HFO	Photo-polymerized resin composite	1,4-Butandioldimethacrylate, urethandimethacrylate, Diurethandimethacrylate, Iso-propyliden-bis (2(3)-hydroxy-3(2)-4(phenoxy)propyl)-bis(methacrylate), glass filler: mean particle size 0.7 μm; highly dispersed silicone dioxide	Micerium, Avegno, Italy	2014004869
Variolink Esthetic LC	Dual-polymerized resin cement	Urethane dimethacrylate, ytterbium trifluoride, 1,10-decandiol dimethacrylate, glycerine-1.3-dimethacrylate, 2,6-di-tert-butyl-*p*-cresol	Ivoclar Vivadent	T21748

polishing discs (Sof-Lex Contouring and Polishing Disks, 3M ESPE), specimens were stored in distilled water at 37 °C for 2 weeks.

One dental technician fabricated all lithium-disilicate ($Li_2Si_2O_5$) LVs (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) according to the instructions of the manufacturer. LVs were first sintered in a calibrated ceramic oven (Programat P3000, Ivoclar Vivadent), glazed and hand polished (CeraGlos HP, Edenta, Au, Switzerland). The final thickness of the laminate veneers was 0.3 mm at the incisal and 0.1 mm at the cervical regions.

2.2. Adhesive cementation

A photo-polymerizing resin luting agent (Variolink Esthetic LC, Ivoclar Vivadent) was used for group CEMF and CEMLF, and a restorative resin composite (Enamel HFO, Micerium, Avegno, Italy) was used for group COMF and COMLF. Before luting, provisional restorations were removed, teeth were cleaned with pumice and the fit of ceramic LVs were controlled under optical microscope (×25, OpmiPico, Zeiss). LVs were then checked for fractures in the ceramic using ultraviolet light.

Intaglio surfaces of the ceramic LVs were conditioned using hydrofluoric acid (Ceramic etching gel <5% hydrofluoric acid, Ivoclar Vivadent) for 20 s, rinsed and ultrasonically cleaned (Emag, Valkenswaard, The Netherlands) in distilled water for 5 min. They were then silanized (Monobond Plus, Ivoclar Vivadent) and 1 min heat dried at 100 °C (DI500, Coltene, Altstatten, Switzerland) and coated with a thin layer of adhesive resin (Adhese Universal, Ivoclar Vivadent).

In all groups, enamel was etched with 38% H_3PO_4 (Top Dent, DAB, Malmö, Sweden) for 30 s followed by 30 s of rinsing with copious water. Then, the adhesive resin (Adhese Universal, Ivoclar Vivadent) was applied, air-thinned but not photo-polymerized. While in groups CEMF and CEMLF the photo-polymerizing resin luting agent (Variolink Veneer, Ivoclar Vivadent) was used for delivery of the ceramic LVs, in group COMF and COMLF the pre-heated (40 °C, EASE-IT, Rønvig, Daugaard, Denmark) restorative resin composite (HFO UD2, Micerium) was used for the luting of the laminate veneers. This composite was chosen for its certain practical advantages and optimized rheological properties for luting [27]. The luting agent was applied at the intaglio surface of the LV and applied to their corresponding teeth under finger pressure until complete adaptation.

Excess resin was removed using a dental probe and a brush (GC, Leuven, Belgium). Glycerine gel (liquid strip, Ivoclar Vivadent) was applied at the margins of the LVs and photo-polymerized for 40 s from labial, lingual and incisal (≥1000 mW/cm^2, Bluephase, Ivoclar Vivadent) each. The output of the polymerization device was 1000 mW/cm^2 throughout the experiment (Bluephasemeter, Ivoclar Vivadent).

The margins were polished using rubber points (Astropol, Ivoclar Vivadent). Each specimen was embedded in polymethylmethacrylate (Autoplast, Condular, Wager, Switzerland) up to the cemento-enamel junction in the middle of a plastic ring (PVC, diameter: 2 cm, height: 1 cm).

2.3. Aging, accelerated fatigue and load-to-failure tests

After luting, all specimens were artificially aged in a chewing simulator (SD Mechatronik CS-4.8 Chewing Simulator, Feldkirchen-Westerham, Germany) using a flat ceramic antagonist (50 N) at the incisal edge (Fig. 3a, 1.200.000 cycles, 1.7 Hz) and hydrolytically aged (8000 cycles in 5–55 °C distilled water). Changes as chipping/fractures and incisal wear were evalu-

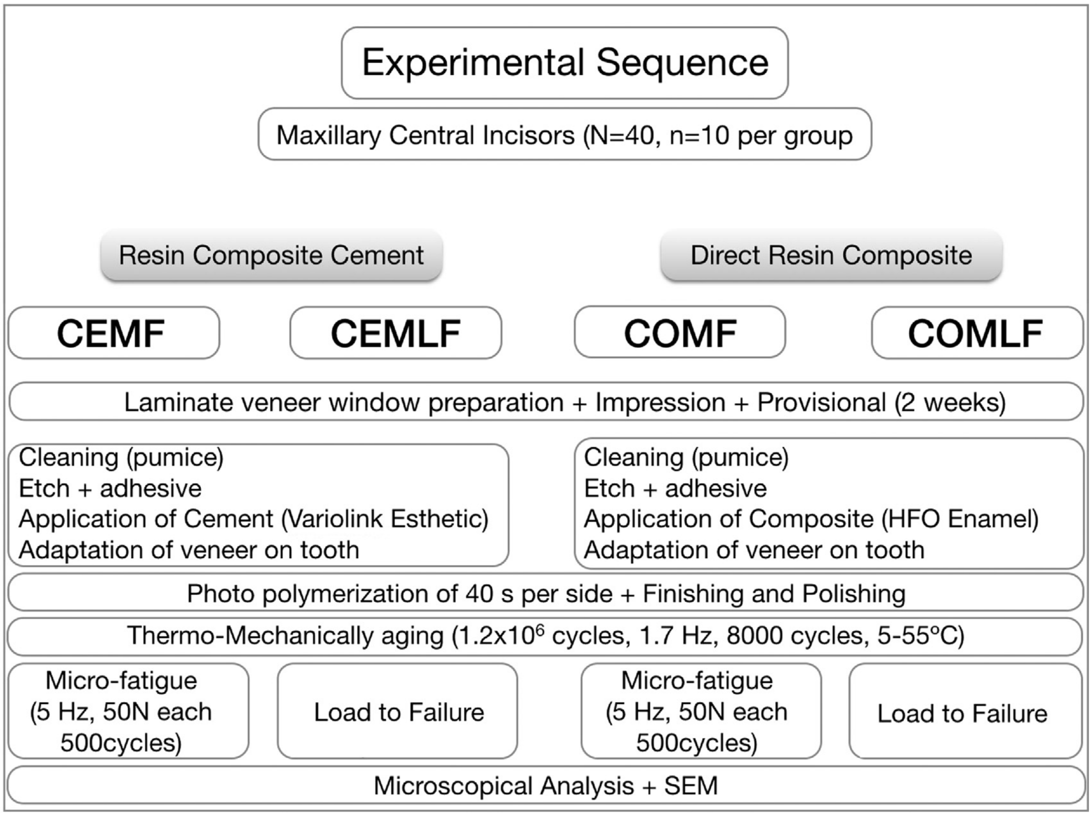

Fig. 1 – Flow-chart showing experimental sequence and allocation of groups.

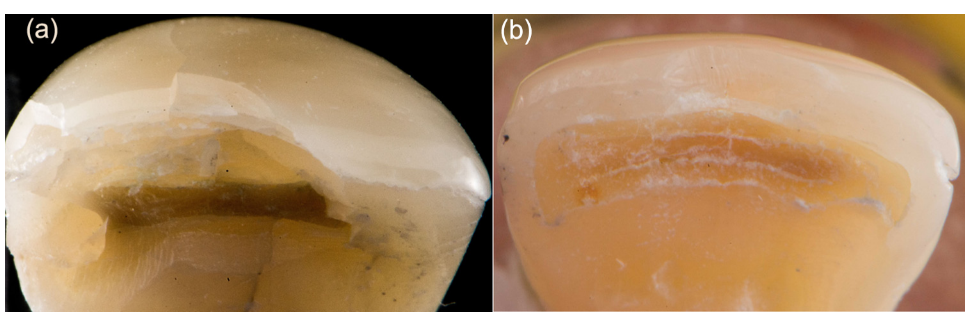

Fig. 2 – (a) Representative specimens from groups (a) CEMF after aging, note the fracture/chipping at the incisal edge, (b) COMF, note the wear of the ceramic at the incisal edge.

ated. Digital photos of the specimens were made (Figs. 2 and 3) before and after aging.

Specimens in groups CEMF and COMF were subjected to accelerated fatigue using a closed-loop electrodynamic system (Acumen 3, MTS Systems, Eden Prairie, MN USA) [28]. The force was applied through a flat composite resin antagonist (MZ100, 3 M, St Paul, USA). Each specimen was placed in the load chamber contacting two thirds of the incisal edge parallel to the long axis of the tooth. Ultrafine silicone carbide abrasive paper (600 grid, Norton Abrasives, Worcester, MA, USA) was used to adjust the incisal edge and assure uniform contact. The load chamber was filled with distilled water until complete immersion of the specimen. A frequency of 5 Hz was used starting with a load of 50 N and increasing 25 N after each 500 cycles until catastrophic failure. The fracture load of the specimen was noted as the machine stopped by a failure detection module. The number of load at failure and endured cycles were then recorded.

The load-to-failure test was performed on the specimens of groups CEMLF and COMLF in a Universal Testing machine (810 Material Test System, MTS, Eden Prairie, USA) at a crosshead speed of 1 mm/min with the same load configuration as before (Fig. 3b). The maximum force to produce fracture was recorded in Newton.

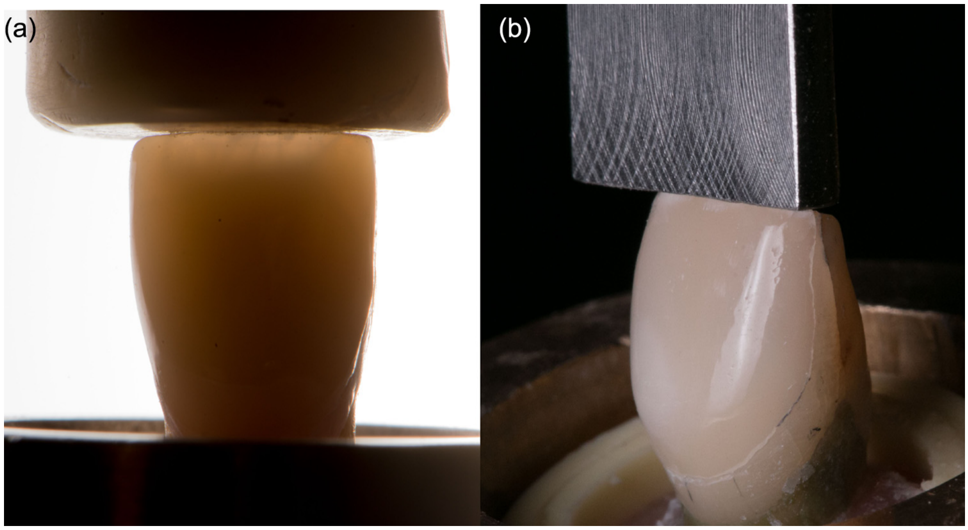

Fig. 3 – The position of the load cell actuator (a) in the accelerated fatigue device and (b) during maximum load to failure test, in relation to the laminate veneer-tooth interface.

2.4. *Microscopical analysis*

Failure types were evaluated using an optical microscope (×40 magnification, Wild M3Z, Heerbrugg, Switzerland, ×40). Additionally, after cleansing with alcohol, representative specimens from each group were first sputter-coated with a 3 nm thick layer of gold (80%)/palladium (20%) (90 s, 45 mA; Balzers SCD 030, Balzers, Liechtenstein) and analyzed using cold field emission Scanning Electron Microscope (SEM) (LyraTC, Tescan, Brno, Czech Republic). Images were made at 15 kV at a magnification of ×35 to ×5.000.

2.5. *Statistical analysis*

A chi-square test of independence was performed to examine the relation between the luting agent type and mode of failure (wear or wear with fracture) after aging. The data of the accelerated fatigue were drawn, in a Kaplan Meier survival curve to cycles and life table for load. Additionally a Log Rank test (Mantel–Cox) was performed to compare survival curves for cycles and Wilcoxon for load. A Shapiro–Wilk's test ($p > 0.05$) and a visual inspection of their histogram, normal Q–Q plots and box plots showed that the data were approximately normally distributed for group CEMLF with a skewness of 0.581 (SE 0.687) and a kurtosis of 1.206 (SE 1.334) and group COMLF with a skewness of −0.442 (SE 0.687) and a kurtosis of −0.206 (SE 1.334). An independent-samples t-test was conducted afterwards to compare groups CEMLF and COMLF in terms of maximum load to failure using a statistical software programme (SPSS 22.0, SPSS inc., Chicago, USA).

3. Results

A chi-square test of independence was performed to examine the relation between the luting agent type and mode of failure (wear or wear with fracture) after thermo-cyclic aging. The relation between these variables was significant, X^2 (1, N = 40) = 22.56, $p < 0.000$ meaning that fractures (n = 18) were more frequently observed in the luting cement groups (CEMF and CEMLF); and the incidence of wear (n = 17) was higher in the resin composite groups (COMF and COMLF) after thermo-cyclic loading (Fig. 2).

In the accelerated fatigue test, mean survival rates for load were 762.5 N for group CEMF and 1165 N for group COMF. Log-rank test X^2 (1, N = 20) = 10.98, $p < 0.001$ indicates that the fracture loads for the LVs cemented with a resin composite were statistical significantly higher than those luted with resin composite cement. Mean survival rates for amount of cycles were 14.569 for group CEMF and 22.595 for group COMF (X^2 (1, N = 20) = 10.44, $p < 0.001$, Log-rank test). The number of endured cycles of the LVs luted with a restorative resin composite was significantly higher than the resin composite luting agent (Fig. 4).

An independent-samples t-test was conducted after normality test in order to compare groups CEMLF and COMLF in the load to failure test. There was a significant difference in the scores for group CEMLF (M = 629.4, SD = 212.82) and group COMLF (M = 927.59, SD = 261.06); $t(18) = -2.80$, $p = 0.01$ showing that the luting agent type affected the results in maximum load to failure test (Fig. 5).

Failure types were predominantly adhesive between the resin cement and the LV in groups CEMF, COMF, COMLF while Group CEMLF presented chipping of the ceramic more frequently (Fig. 6). None of the teeth restored with LVs showed fractures of the root or large amount of tooth structure.

SEM images clearly showed chipping in group CEMLF (Fig. 7a) or detachment in groups CEMF and COMF (Fig. 7b–d) of the LV from the luting agent with some remnants of luting agent still attached on the tooth surface.

4. Discussion

The strength of LV restorations rely highly on the adhesion protocol used where surface conditioning of the ceramic and

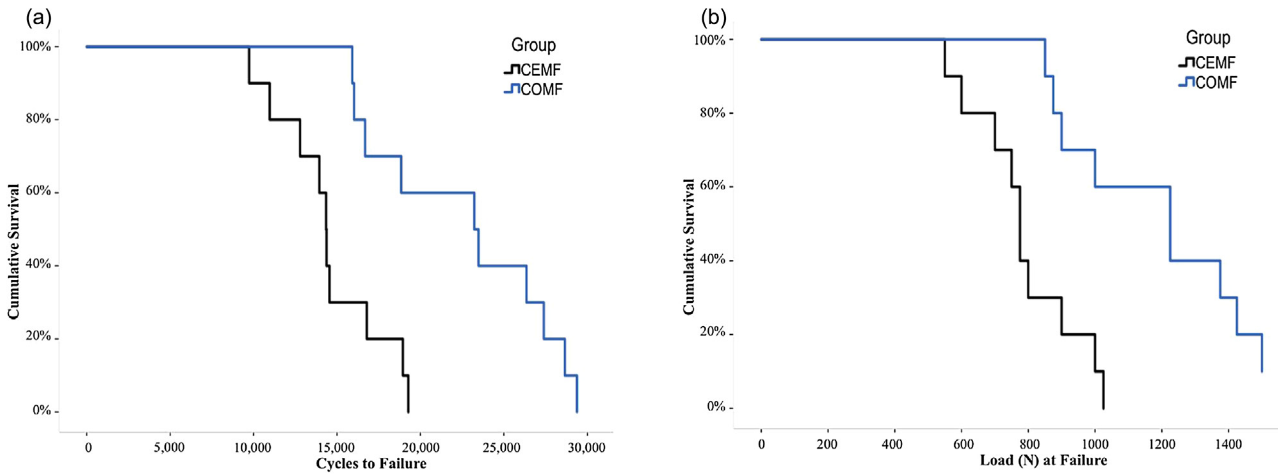

Fig. 4 – Survival functions in relation to (a) the amount of cycles and (b) load of the accelerated fatigue test for group CEMF: Variolink Veneer and group COMF: Micerium HFO.

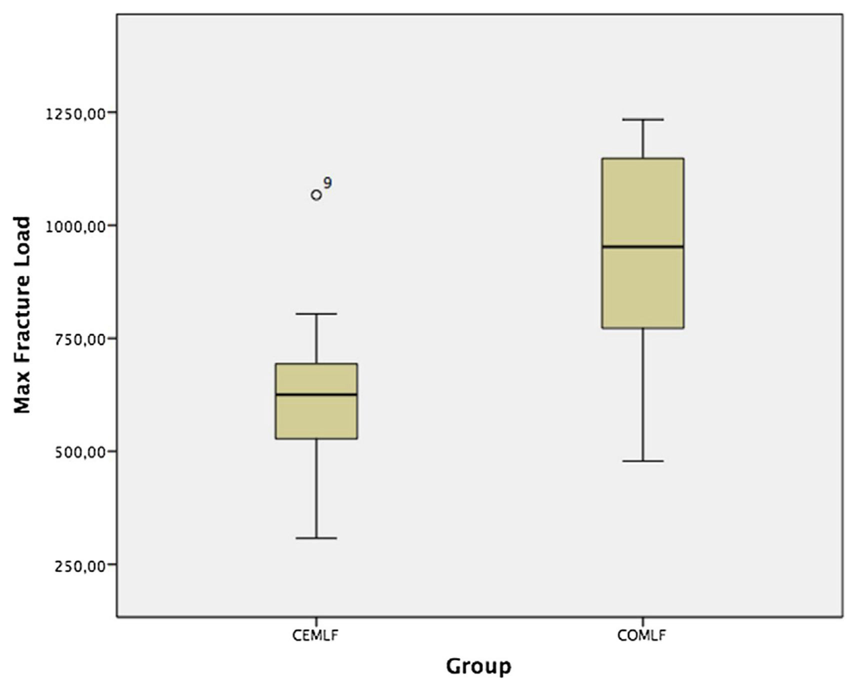

Fig. 5 – Boxplot of the maximum load to failure data of groups CEMLF and COMLF.

tooth substrates play a significant role [18]. Although procedures for adhesive luting are well-established, failures are still experienced in clinical studies and survival rates are reported to range between 82 and 96% in 10–21 years [3–8]. Fractures of ceramic (5.6–11%) and marginal defects (12–20%) were the typical reasons for failure [3,4,6–12]. In addition to surface conditioning, the luting agent can play an important role as seen in previous studies where there was a significantly positive effect of a restorative resin composite as a luting agent in comparison to different dual-polymerized cements [28,29,30]. For this reason, this study was undertaken in order to compare a luting agent with a restorative resin composite as a luting material. Besides thermo-mechanically aging, two different test methods were used namely the accelerated fatigue and the well-established load to failure test. Based on the results of this study, since there were significant differences between the experimental groups in terms of aging, survival and fracture resistance, the hypothesis that the aging and luting agent type would have no effect could both be rejected. The hypothesis that testing approach would deliver similar results could be accepted as both tests revealed similar significant differences between the test groups.

The load-to-failure test method for the assessment of luting agent effectiveness could be useful for ranking mate-

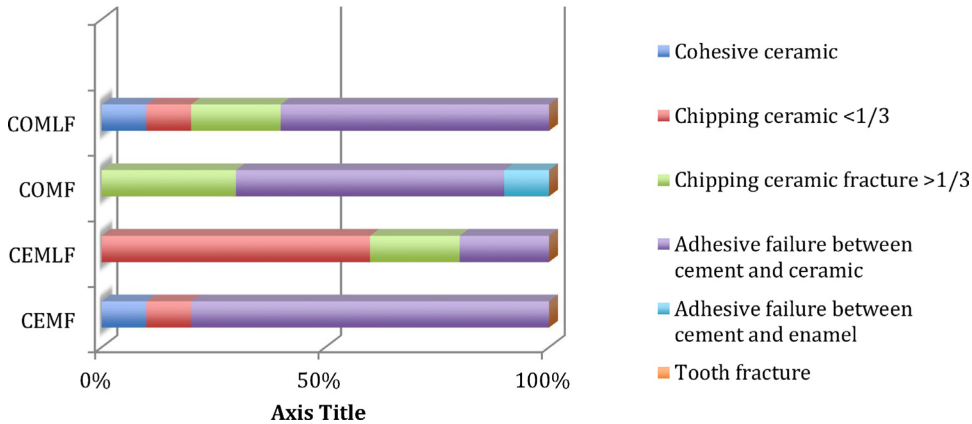

Fig. 6 – Frequencies of failure modes in percentages. Type I: cohesive ceramic fracture; Type II: chipping of the ceramic <1/3; Type III: chipping of the ceramic >1/3; Type IV: adhesive failure between luting agent and ceramic; Type V: adhesive between luting agent and enamel; Type VI: tooth fracture.

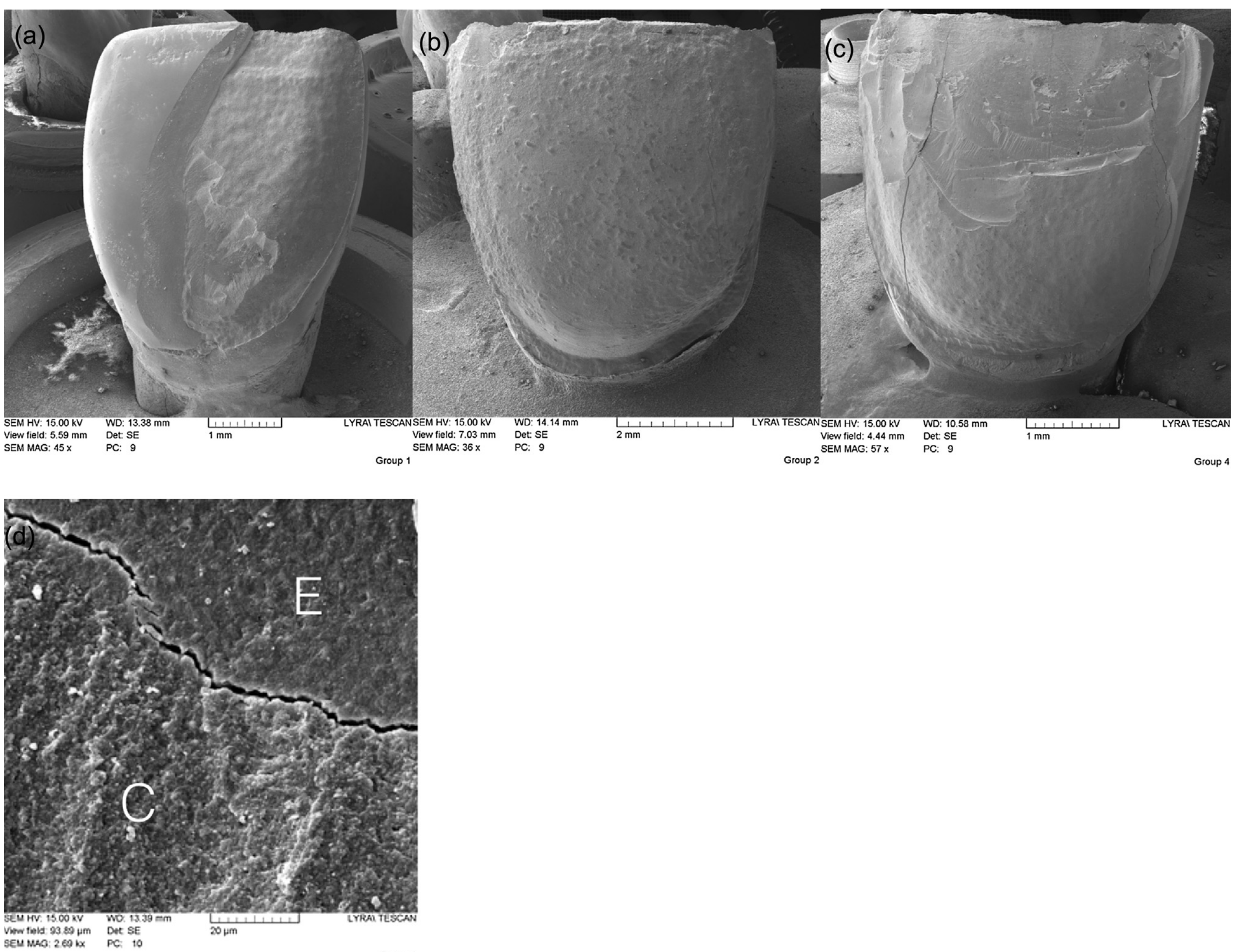

Fig. 7 – Typical failure types from (a) a specimen in Group CEMLF where a chipping occurred in >1/3 of the laminate veneer, (b) a specimen from group CEMF where delamination occurred between the ceramic and luting agent, (c) a specimen from group COMF where an adhesive failure occurred between the luting agent and enamel, (d) the corresponding SEM image (×2500) of group COMF of the enamel (E) and resin composite (C).

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rials in in-vitro settings. However, clinical performance of LVs is not only dependant on the tested variables but also on patient-related factors, materials and operator-related factors [32–34,38]. Moreover, clinical studies are expensive and it is controversial from an ethical standpoint to test materials in patients without preclinical tests. Therefore, laboratory aging and in vitro testing methods are applied in a manner to simulate the intra-oral situation as closely as possible, focussing on one or two variables while excluding confounding variables. During function, dental materials are exposed to various conditions and material properties are changing due to degradation and aging [31]. Changes in materials are usually due to chemical breakdown by hydrolysis, stress induced effect associated with hygroscopic expansion and applied stress, leaching and corrosion [31]. A widely used aging method is thermo-cycling. The ISO TR 11450 indicates that thermo-cycling of 500 cycles in water between 5 and 55 °C is an appropriate artificial aging test. However, it was concluded that 10.000 cycles corresponds to 1 year of in vivo functioning [40]. For posterior restorations, a true fatigue correlation for one year of clinical service is 250,000 cycles at only 13.6 N [41]. Besides thermo-cyclic aging, fatigue loading could also have further aging effect on materials [31]. In mechanical aging, it is crucial to simulate the stress/load as close as possible to the in vivo situation [41]. Monostatic testing has been questioned and low load fatigue testing is very time consuming. Therefore, in order to acquire more clinically relevant data with regard to the cementation of veneers, the accelerated fatigue test (step-loading) was performed. In this study, the thermo-mechanical as well as the accelerated fatigue and load to failure tests were performed on the incisal edge in a configuration representing edge-to-edge biting forces. Testing on the palatal side that is the most commonly used method that requires application of the loading jig acting on the palatal side of the tooth. This would however eventually lead to failure of the tooth structure itself and not the restoration-adhesive-tooth complex. In this study, the incisal edge position was chosen and a force of 400 N was reached. The average bite forces in human range between 20 and 1000 N but during actual chewing, the forces do not exceed 270 N [34]. Furthermore, the forces in the anterior region of the mouth are reported to be less than in the posterior region ranging between 155 and 200 N [34]. In this study, significant differences were present between the two groups and more severe deterioration in the form of fractures and chippings were observed in the groups where ceramic LVs were delivered with a dual-polymerized cement.

Over the years, there has been growing interest in adhesive luting of indirect restorations using highly filled restorative resin composite and by making them less viscous after pre-heating without detrimental changes to the properties of the material [35–37,39]. On the other hand, conventional resin composite luting agents have some advantages over a restorative resin composite with their lower viscosity that allows easy control during positioning and fitting the restoration on the tooth substance. Based on the results of this study from both the fatigue as well as the load to failure tests, it could be stated that adhesive luting by using a restorative resin composite instead of a resin composite cement would be beneficial. However, it should be noted the restorative resin composite was pre-heated. LVs were placed under finger pressure but in the restorative resin composite group, more pressure was applied in order to remove the excess resin. Reducing thickness of the cement layer in especially very thin ceramic LVs could increase the strength [29].

Using micro-tensile adhesion tests, comparison was made between dual-polymerized resin cements and a restorative resin composite but their adhesion was not compared for the application of LVs [24,28]. In a study by Sarr et al., restorative resin composite using a regular bonding system resulted in higher microtensile bond strength to dentin when compared to the frequently used, etch and rinse, self-etch or self adhesive resin cements [25]. In this study, LVs were bonded to enamel but cohesive strength of the resin composite with 63 v% fillers compared to dual-polymerized luting agent with 38 v% increased the strength of the tooth-cement-ceramic complex.

After surface conditioning of the ceramic with hydrofluoric acid etching and ultrasonic cleaning, silane coupling agent was applied, optimizing the resin penetration and increasing exposure of the silica to the silane in order to form siloxane bonds [35,39,42]. Heat treatment of the silane increased crosslinking, forming a uniform monolayer of silane molecules, which increases in turn the adhesion of resin-based materials to ceramics [42]. Although optimal surface treatment was performed, most of the observed failures (58%) after fatigue and load to failure tests were adhesive failures between the ceramic and the resin luting agent, which seems to indicate that potential improvement in adhesion of such materials to ceramics is still possible. In this study, no fractures of the root or severe enamel/tooth fractures were observed. Observed failures would be categorized as repairable and could be restored with direct resin composite chairside without necessitating replacement of the LV restoration.

5. Conclusions

From this study, the following could be concluded:

1. Luting of lithium disilicate laminate veneers using a preheated restorative resin composite resulted in significantly higher survival and fracture resistance.
2. Both test methods used (accelerated fatigue vs load to failure) presented similar results indicating the same significant differences between the two luting agents.
3. Failure analysis after thermo-cyclic aging showed predominantly wear facets together with chipping or fracture in LVs that were bonded with the regular luting agent while the groups luted with preheated restorative resin composite presented only wear.

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