The Effect of Surface Treatment on Shear Bond Strength of Resin Matrix Ceramics and Dual Cure Resin Cement

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Abstract

The aim of this study was to evaluate the effect of Tetrahydrofuran (THF) on shear bond strength (SBS) of resin matrix ceramics (RMC) to resin cement.

RMC (Enamic, Cerasmart, Shofu block HC) were cut into square piece size 6x6x2 mm^3 and randomly divided into 10 groups according to the surface treatments: no treatment(C), adhesive agent (Ad),THF 1min(T1), silane/adhesive agent (Si/Ad), THF 1min/adhesive agent(T1/Ad), THF 1 min/silane/adhesive agent(T1/Si/Ad), THF 2 mins/silane/adhesive agent(T2/Si/Ad), THF 3 mins/silane/adhesive agent(T3/Si/Ad), THF 4 mins/silane/adhesive agent(T4/Si/Ad), THF 5 mins/silane/adhesive agent(T5/Si/Ad). Specimens were cemented to composite resin rod with resin cement, then kept in water at 37°C for 24 hours. The SBS was tested with universal testing machine and data were analyzed with two-way ANOVA and Bonferroni's post hoc tests (α=0.05). Failure modes were examined by stereomicroscope.

The highest mean SBS for all RMC was found in group T3/Si/Ad (25.37±4.73 MPa) significantly greater than almost all groups (p<.05), except for T4/Si/Ad and T5/Si/Ad. Enamic showed the highest SBS value (28.12±5.45MPa) followed by Cerasmart and Shofu block HC, respectively. Mixed failure was the most common found in THF with silane and adhesive agent groups.

Tetrahydrofuran with silane and adhesive agent affected to the SBS of RMC and resin cement.

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Introduction

One of the main purposes of restorative dentistry is to create functional and esthetic restorations. Ceramics are extensively used as indirect restorations due to their esthetic appearance, good fracture resistance and low wear rate.[1,2] However, ceramics have limitations on success rates because of their toughness, brittleness, and potential to wear opposing teeth.[3,4] Nowadays, not only esthetic expectations, but also chairside fabrication of restorations are necessary. As the results, computer - aided design / computer - aided manufacturing (CAD/CAM) technology is wildly used and different types of machinable materials such as ceramics, acrylic resins, and composite resins developed to complete the requirements.[3,5,6]

Resin matrix ceramics (RMC) have been recently developed for CAD/CAM technology. RMC combines the benefits of composite resins, improved flexural properties and low abrasiveness, as well as color stability and durability of ceramics.[7,8] Available commercial products of resin matrix ceramic materials include a polymer-infiltrated ceramic network (PICN) material (VITA ENAMIC), nanohybrid composite resin materials such as resin nanoceramic (Shofu Block HC ,Lava Ultimate) and a nanoparticle-filled resin (Cerasmart). In addition, RMC have the ability to distribute stress due to modulus of elasticity near to dentine and the capacity of milling-adjusting which is more convenient and safes compared to glass matrix or polycrystalline ceramics.[3,9,10]

The bond strength between cement and resin or ceramic CAD/CAM materials has a major role in providing the improvement of fracture resistance and keeping the marginal integrity of the restorations.[11,12] To create a sufficient bond, mechanical or chemical pre-treatments are

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essential.[13,14] Depending on the composition of the material, various surface treatment techniques such as silanization, silica coating (Co-jet), etching with hydrofluoric acid (HF) and sandblasting could be applied.[15-17] Many studies attempted to improve the bond strength of RMC materials to different resin cements by using different surface treatments. However, some methods of surface treatments are still inconclusive.[18-22] Tetrahydrofuran (THF) is an organic solvent and it could be used as solvent in dental adhesive systems to form bond strength stability.[23] THF could also be used with silane for improving the shear bond strength of glass fiber post.[24] There is still not enough information on THF to be used as surface treatment for enhancing the bond strength between resin cement and RMC. Therefore, the purpose of this study was to evaluate the effect of THF on bond strength between RMC and dual cure resin cements. The null hypothesis is that surface treatment by using THF with silane and adhesive agent would not affect the shear bond strength of RMC to dual cure resin cement.

Materials and methods

Three different resin matrix ceramics materials were used in this study. Manufacturers and compositions of the materials are presented in table 1. The RMC materials were cut with a diamond disk (Slow speed cutting machine, Model Isomet, Buehler, IL, USA) under cooling water to a square shape (6 × 6 × 2 mm^3). The specimens were embedded in polyvinyl chloride pipe (PVC) diameter 0.5 inch with epoxy resin. After the epoxy resin reached its final setting time, the mounted specimens were polished using a polishing machine (Minitech 233, Presi, Le Locle, Switzerland) with 300 and 600-grit silicon carbide abrasive paper, respectively. The specimens were then ultrasonically cleaned in distilled water for 5 minutes, and air-dried for 15 seconds.

The specimens of each RMC were randomly divided into 10 groups and each group was subdivided into 3 subgroups (n=10) according to surface modification methods as shown in Fig. 1. The surface modification details are shown in table 2. For the THF groups, lead sheets had been punched as a square shape size 5x5 mm^2, to limit the area of applying agent, then removed after finishing this treatment steps. To control the bonding area, an 80-micron thick single-sided adhesive tape (Scotch blue Painter's tape, 3M, St. Paul, MN, USA) was cut into a square shape with a size of 5x5 mm^2. A 3-mm diameter hole was made in the center of the adhesive tape using a hole- puncher. The adhesive tape was firmly placed and attached to the specimen surfaces, this procedure was performed before applying other chemical agents and cementation.

Three hundred composite resin rods were prepared using a custom-made silicone mold (4 mm diameter × 4 mm height). Composite resin (Filtek™ Z350. XT shade A3, 3M ESPE, MN, USA) was condensed with a hand instrument in 2-mm incremental layers and light-polymerized with light curing unit (1000 mW/cm^2, Elipar Freelight 2 LED curing light, 3M ESPE, St. Paul, MN, USA) for 40 seconds. The ends of composite resin rods were blasted with 50-micron alumina. Composite resin rods were bonded to the treated specimens with dual-cure resin cement (RelyX U200, 3M Deutschland GmbH, Neuss, Germany) by light-polymerization. Luting was performed under constant weight of 1,000 g applied to the composite rod during the bonding procedure for 10 seconds at room temperature. The cement was activated by a light-curing unit at the 4 proximal sides and the top surface, 20 seconds each. The bonded specimens were kept in 37^{o}C distilled water for 24 hours in an incubator (Contherm 160M, Contherm Scientific Ltd, Korokoro, Lower Hutt, New Zealand) according to ISO/TS 11405 to allow for post-polymerization. The bonded specimens were test with the notched-edge shear bond strength test modified from ISO 29022:2013 using a universal testing machine (Shimadzu, EZ-S 500N model, Japan). The specimen was placed in a metal sample holder, notched-edge shear blade was mounted on the universal testing machine and placed over the composite resin rod on the aligned specimen. The blade was positioned precisely over the composite resin rod and force fitted without premature contact to ensure that the load was applied directly to the cylinder at a crosshead speed of 1.0 mm/min until failure occurred. Shear bond strength values were calculated in megapascal (MPa) by dividing the maximum load at failure (N) with the bonding area (mm^2). Subsequently, the failure modes were investigated under a stereomicroscope (Olympus Stereo Microscopes, SZ61, Japan) at a magnification of ×40. The failure modes were classified into the following categories: adhesive failure at the cement–materials interface, cohesive failure within the luting cement, cohesive failure in RMC materials and the mixed failure. The bond strength values were analyzed by two-way analysis of variance (ANOVA) to determine the significant differences between the surface treatment methods and the different types of RMC at significance level of 0.05 with post hoc comparisons by Bonferroni tests (IBM SPSS Statics for Windows, Version 22.0. Armonk, NY, USA). The specimens from each RMC material in the control group and the group applying THF for 3 minutes were evaluated with scanning electron microscope (SEM) analysis (FEI Quanta 250) at ×2000 magnification. For the group THF application for 3 minutes, specimens were analyzed by Energy Dispersive X-ray Spectroscopy (EDX) point-measurements. Specimens in group No.1-10 were not investigated by SEM.

Results

The two-way ANOVA analysis revealed that the shear bond strength values were significantly influenced by the RMC materials, surface treatment methods, and the interaction between the RMC materials and the surface treatment methods. (p = .001, .001, .222, F = 118.96, 16.91, 1.25, η_p^2 = .80, .11, .08, respectively) Moreover, RMC had a moderate effect size while surface treatment methods and interaction between the RMC materials and the surface treatment methods had a small effect size.[25]

The SBS are presented in table 3. Most of the groups showed that the SBS significantly greater than the C group ($p < .05$), except for the T1 group. Also, Si/Ad group presented significantly higher SBS than group C, Ad, T1, T1/Ad ($p < .05$), but still lower than T1/Si/Ad, T2/Si/Ad, T3/Si/Ad, T4/Si/Ad and T5/Si/Ad group .For T1/Si/Ad, T2/Si/Ad, T3/Si/Ad, T4/Si/Ad and T5/Si/Ad showed SBS value between 17.44 - 28.12 MPa, which were significantly higher than Ad and Si/Ad groups ($p < .05$). The highest SBS was found in group T3/Si/Ad, but there was no significant difference compared to T4/Si/Ad and T5/Si/Ad groups. In addition, Enamic showed the highest SBS value (28.12 ± 5.45) MPa in group T3/Si/Ad followed by Cerasmart and Shofu block HC, respectively. The comparison of mean SBS among brands of RMC from table 4 demonstrated that VE showed the highest SBS (16.99 ± 8.01 MPa) followed by CS (14.52± 8.31 MPa) and HC (13.96 ± 8.88 MPa), respectively. The significant difference was observed in VE with CS and HC groups, but there was no significant difference between CS and HC groups. The frequencies of the failure mode observed are presented in table 3. Adhesive failure pattern was the most common failure mode found in C, Ad, T1, T1/Ad group. Mixed failure was also the most common failure mode showed in Si/Ad, T1/Si/Ad, T2/Si/Ad, T3/Si/Ad, T4/Si/Ad and T5/Si/Ad.

SEM photograph at 2000x magnification in Fig.2 showed the different surface morphology of three RMC brands between the control and the THF groups. The specimen's surface treated with THF presented more irregularities and whiter spot than the control group. The surface of the THF group showed more inorganic particle related to results of EDX (yellow circle). The majority of the inorganic particle was silicon element.

Discussion

The purpose of the present study was to evaluate the effects of surface treatment by using Tetrahydrofuran (THF) with silane and adhesive agent on bond strength of three different types of CAD/CAM Resin matrix ceramics (RMC) materials to resin cements. From previous studies, THF could be used as a solvent in dental adhesive systems. THF not only showed increased bond strength stability and had an intermediate cytotoxicity close to HEMA, but also increased bond strength value to glass fiber post by application with silane.[11,23,26] In the present study, THF with silane and adhesive agent could be used as pre-treatment for improvement of shear bond strength to different RMC. Thus, the null hypothesis was rejected. THF is an organic compound that is classified as heterocyclic compound, specifically a cyclic ether. THF is used as solvent for many polymers such as polyvinyl chloride, unvulcanized rubber , vinyl, polymer coating, cellophane, protective coatings.[27] According to Inoue and Hayashi study THF was used as the solvent to find that the residual Bis-GMA in resin composite.[28] Väkiparta et al. found residual monomers, Bis-GMA and

TEGDMA, in fiber-reinforced composites by using THF.[29] RMC materials combined two phases of materials polymer matrix and condensed filled ceramics particles.[7,9] Thus, the increased bond strength between RMC and the resin cement of present study could be explained by the fact that THF dissolves partial polymer part at the surface of material. Consequently, the surface of material shows more inorganic part (silica) which reacts and promotes adhesion by applying silane.

The silane-coupling-agent acts as bifunctional monomer and adhesion promoter in silica-containing materials. There have been studies describing that silane-coupling-agent reacts to inorganic fillers exposed on surface of material. The other functional monomeric ends molecules of silane can react with the methacrylate groups of the adhesive resin and the integrated polymer parts of RMC materials.[30,31] The primer in bonding also increased efficiency to bond the CAD/CAM composites.[11,32,33] In addition, the use of methacrylate-containing primer combine with a silane-coupling-agent increased the bond strength.[32] Another explanation of adhesion mechanism is due to methacrylate monomers of the adhesive agent penetrate to the resin matrix of materials and polymerize to form the interpenetrating network.[32,34] All explanations correspond to the results of this present study that using THF with silane and adhesive agent shows better improvement of the shear bond strength of RMC. In addition, Enamic has the highest bond strength value of RMC in the present study. This could result from the difference in the percentage of inorganic component. Thus, Enamic has more percentage of inorganic part when compared to within the RMC groups. Mixed failure is correlated with the improved bond strength, but adhesive failure means lower bond strength,[30] which corresponds with the results in table 3 that mixed failure was the predominant type found in THF with silane and adhesive agent. Adhesive failure was commonly found in other groups. But the Si/Ad group mostly found mixed failure due to chemical reaction from silane.

Many surface treatment methods for the RMC materials were observed from previous studies,[18-22] chemical and mechanical methods were often used to increased bond strength value. The chemical pre-treatment method which used HF and silane as chemical agents can improve bond strength. HF acid treatment in Enamic caused by partial dissolved the glassy phases and polymer which created microporosities and micromechanical retention surface. Silane application can increase the surface wetting of bonding area and improve a chemical bond to the resin cement and better bond strength as a consequence.[21] However, Cekic-Nagas et al. showed that RMC treated with 10% HF acid gel did not have an effect on bond strength value between resin cement and RMC.[18] In addition, HF acid causes irritation to tissue and considerable health hazard because of toxicity and volatility.[35] Due to, the controversial effects of HF acid to the bond strength of RMC, the surface treatment protocol by HF was not used in this study. Sandblasting is the mechanical method which is used to increase the bond strength by improving micromechanical interlocking, and increasing wettability and surface area.[16,20] However, there is a study stating that sandblasting to ceramics should be avoided because of the huge volume loss.[36] Also, Yoshihara et al. revealed that sandblasting created surface damage to Shofu Block HC and silane treatment cannot improve the bond strength for these materials.[22] Thus, Tekçe et al. stated that surface sandblasting for 60 seconds showed lower micro-tensile bond strength value when compared with shorter time of duration for Enamic.[37] However, sandblasting to RMC seem still have controversy for surface treatment because it may create microcracks in the surface and lead to premature failures. It also affects internal and marginal adaptation.[16,20,38] As the results, there are no definite conclusion whether chemical or mechanical surface pre-treatment method is more appropriate for RMC materials. Self-adhesive resin cement was chosen in this study because self-adhesive resin cement such as RelyX U200 is dual-cured resin cement, easy to use and has the improved mechanical and bonding properties in one step. Moreover, etching, priming and bonding are not necessary for this cement type and self-adhesive resin cement is mostly used in the dental practice.[39] Due to the limitation of this study, shear bond strength was used in the present study because shear test is convenient to prepare specimen and a simple test process. However, shear test could not interpret interface failure as good as mini-dumbbell test.[40] In addition, the test was performed 24 hours after cementation which should have further investigation for increasing time storage, thermocycling or vary other resin cement systems.

Conclusions

THF could be used as pre-treatment with silane and adhesive agent showed improvement of shear bond strength of RMC. Mixed failure pattern was the most common failure mode in group of THF with silane and adhesive agent. Among RMC groups, Enamic showed the highest value of bond strength when compared with other materials.

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Declaration of Interest

The authors report no conflict of interest.

Material	Composition	Manufacturer	Lot No.
Vita Enamic (VE)	86 wt% feldspar ceramic, 14 wt% polymer,UDMA,TEGDMA	Vita Zahnfabrik H. Rauter, Bad Sackingen, Germany	071601
Cerasmart (CS)	Nanoparticle-filled resin containing 71 wt% silica and barium glass filler,UDMA	GC Corp., Tokyo, Japan	1706151
Shofu Block HC(HC)	Filler composition: 61%, include zirconium silicate, silicon dioxide, UDMA, TEGDMA	Shofu Inc., Kyoto, Japan	77671
Filtek Z350	Bis-GMA, UDMA, TEGDMA, and Bis-EMA resins and filler	3M ESPE, St. Paul, MN, USA	NA26994
One Coat Bond SL	HEMA,UDMA,GDMA amorphous silicic	Coltene/Whaledent GmbH, Langenau, Germany	179850
Monobond N primer	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	Ivoclar Vivadent, Schaan, Liechtenstein	Y29210
Tetrahydrofuran	Tetrahydrofuran 99.5 %	Loba Chemie Pvt Ltd., Mumbai,Maharashtra, India	LM04671706
RelyX U200	Multifunctional phosphoric acid methacrylates, dimethacrylates, acetate, initiator/stabilizer, powdered glass, silica, substituted pyrimidine, calcium hydroxide, peroxide compound, pigments	3M Deutschland GmbH, Neuss, Germany	4819681

Table 1. Name, composition, manufacturers, lot number of materials used in this study.

Group	Code	Surface Treatment details
1	C	No Surface treatment (Control)
2	Ad	Two microliters of bonding agent (One Coat Bond SL, Coltene/Whaledent GmbH, Langenau, Germany) were applied to the specimen surface using micropipette (Eppendorf AG, Hamburg, Germany) and rubbed with a disposable microbrush (Citisen Micro Applicator, Huanghua Promisee Dental, Hebei, China) for 10 seconds. The excess adhesive agent was removed with a new disposable microbrush, gently air-dried for 20 seconds and light activated for 40 seconds.
3	T1	Two drops of THF were applied to the specimen surface and left undisturbed for 1 min, gently air-dried 10 seconds.
4	Si/Ad	Two microliters of Monobond N were applied to the specimen surface and rubbed with a disposable microbrush for 10 sec and *left* undisturbed for 1 min, gently air-dried for 20 seconds. The bonding was applied as described in group 2.
5	T1/Ad	THF was applied as described in group 3 and the bonding agent was the applied as described in group 2.
6	T1/Si/Ad	THF was applied as described in group 3 and the monobond N and the bonding agent was the applied as described in group 4, respectively.
7	T2/Si/Ad	Two drops of THF were applied to the specimen surface for 2 times. Each round was left undisturbed for 1 min. After the second application, the treated surface was air-dried for 10 seconds. The monobond N and bonding agent were applied as described in group 4, respectively.
8	T3/Si/Ad	Two drops of THF were applied to the specimen surface for 3 times. Each round was left undisturbed for 1 min. After the second application, the treated surface was air-dried for 10 seconds. The monobond N and bonding agent were applied as described in group 4, respectively.
9	T4/Si/Ad	Two drops of THF were applied to the specimen surface for 4 times. Each round was left undisturbed for 1 min. After the second application, the treated surface was air-dried for 10 seconds. The monobond N and bonding agent were applied as described in group 4, respectively.
10	T5/Si/Ad	Two drops of THF were applied to the specimen surface for 5 times. Each round was left undisturbed for 1 min. After the second application, the treated surface was air-dried for 10 seconds. The monobond N and bonding agent were applied as described in group 4, respectively.

Table 2. Shows group, code and surface treatment details.

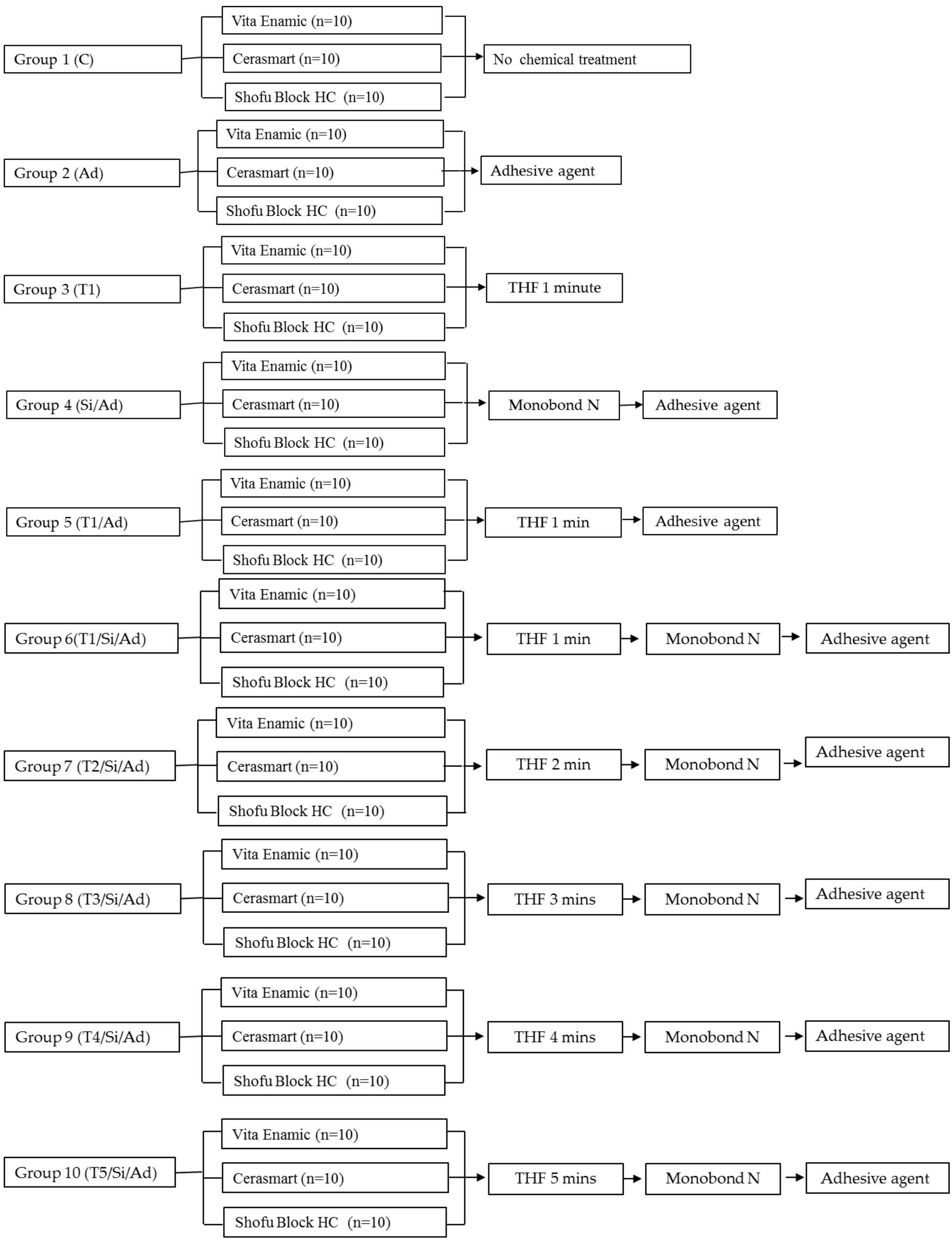

Figure 1. Schematic diagram of experimental procedure.

Group	Vita Enamic		Cerasmart		Shofu Block HC		Total
	Mean SBS ± SD	Failure mode AF/CR/CM/MF	Mean SBS ± SD	Failure mode AF/CR/CM/MF	Mean SBS± SD	Failure mode AF/CR/CM/MF	
C	8.54± 1.56	100 / 0 / 0 / 0	3.89 ± 2.02A	100 / 0 / 0 / 0	2.11± 1.22A	100 / 0 / 0 / 0	4.84 ± 3.17a
Ad	11.29± 1.27A	80 / 0 / 0 / 20	9.15 ± 1.32A	80 / 0 / 10 / 10	7.79± 2.12A	100 / 0 / 0 / 0	9.41 ± 2.14b
T1	6.22± 1.38A	100 / 0 / 0 / 0	2.44 ± 1.52AB	100 / 0 / 0 / 0	1.89± 1.42B	100 / 0 / 0 / 0	3.52 ± 2.40a
Si/Ad	18.48± 4.21	0 / 20 / 50 /30	14.10 ± 2.70A	40 / 0/ 10 / 50	12.58± 2.56A	40 / 0 / 0 / 60	15.05 ± 4.03
T1/Ad	9.69± 1.95A	80 / 10 / 0 / 10	10.21 ± 2.21A	90 / 0 / 0 / 10	9.57± 3.59A	80 / 10 / 0 /10	9.82 ± 2.60b
T1/Si/Ad	20.36± 5.40A	0 / 10 / 40 / 50	17.94 ± 5.81A	40 / 0 / 30 / 30	17.44± 4.50A	30 / 0 / 40 / 30	18.58 ± 5.24c
T2/Si/Ad	20.67± 3.36A	10 / 0 / 20 / 70	18.04 ± 4.54A	20 / 0 / 20 / 60	21.90± 7.91A	20 / 0 / 10 / 70	20.20 ± 5.66cd
T3/Si/Ad	28.12± 5.45A	0 / 0 / 30 / 70	24.69 ± 3.87AB	20 / 0 / 10 / 70	23.31 ± 3.68B	30 / 0 / 20 / 50	25.37 ± 4.73e
T4/Si/Ad	23.98± 3.84A	0 / 0 / 40 / 60	22.65 ± 4.00A	10 / 0 / 20 / 70	21.63 ± 1.68A	30 / 0 / 10 / 60	22.78 ± 3.37de
T5/Si/Ad	22.62± 5.70A	0 / 0 / 30 / 70	22.13 ± 5.86A	20 / 0 / 10 / 70	21.38 ± 7.12A	10 / 0 / 20 / 70	22.04 ± 6.06de

Mean values represented with same uppercase letters (row) or lowercase letters (column) are not significantly to Bonferroni multiple comparison test (p>0.05). Percentage of failure mode [AF : adhesive failure at the cement–materials interface / CR : cohesive failure within the luting cement / CM: cohesive failure in RMC materials / MF : mixed failure]

Table 3. Mean shear bond strength values (MPa ± SD) and Number (%) of specimens according to failure mode.

Brand	Mean ± SD (MPa)
VE	16.99^{A} ± 8.01
HC	13.96^{B} ± 8.88
CS	14.52^{B} ± 8.31

Table 4. Mean of shear bond SBS value.

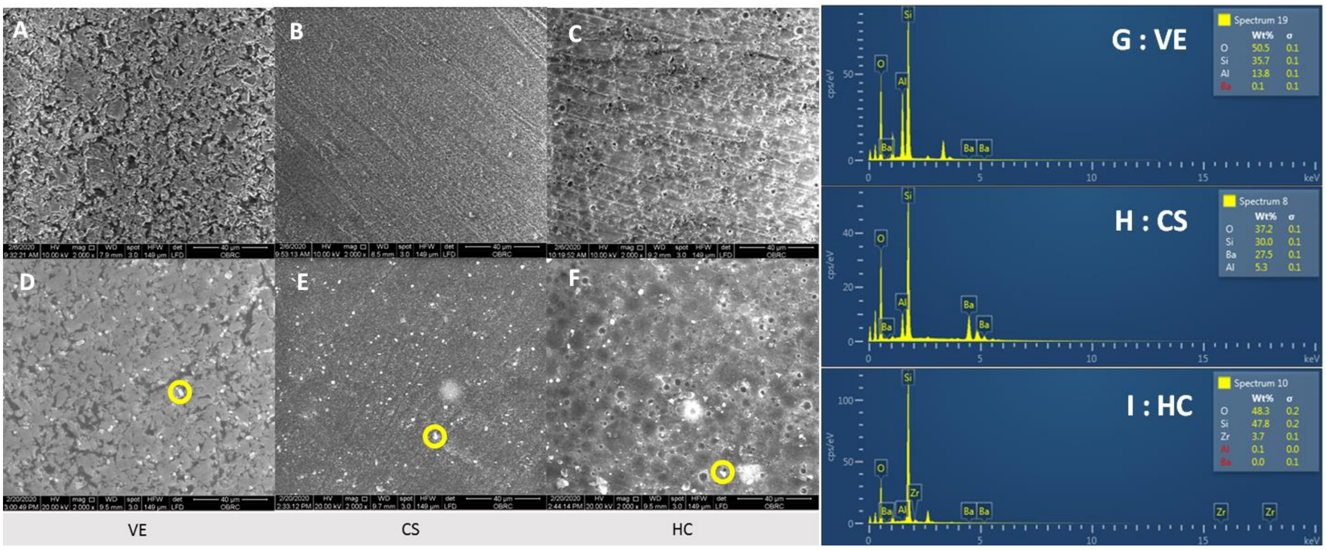

Figure2. SEM photograph at 2000x magnification Control: A-C THF 3 Mins : D-F and SEM/EDX point-measurements result : G-I.

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