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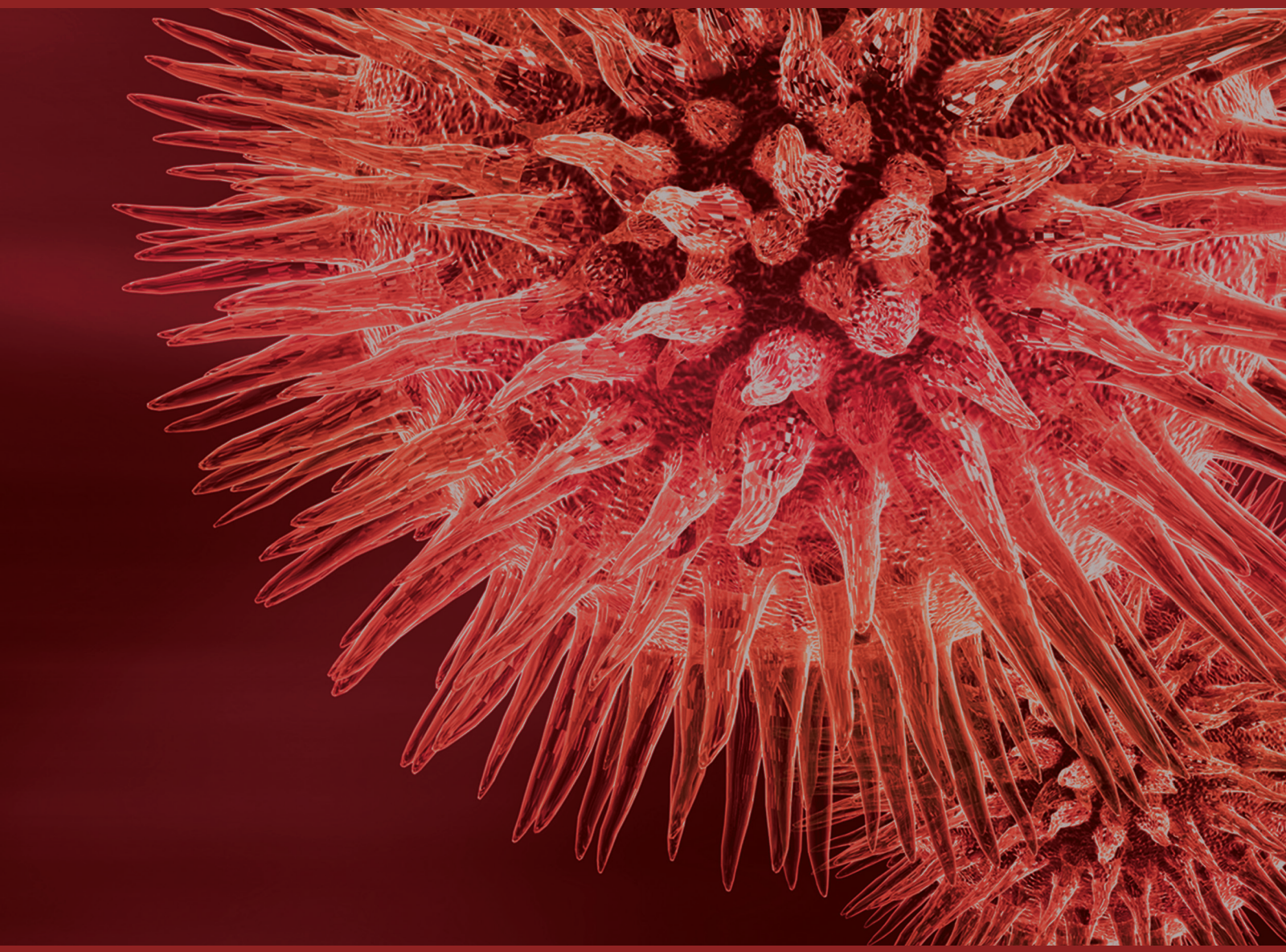
Metal-Free Restorations: Esthetic Considerations, Treatment Planning, Preparation, Manufacturing, Luting, and Followup

Guest Editors: Hamit S. Çötert, Handan Yılmaz, Cem Kurtoğlu, Flávio H. B. Aguiar, and Ahmet U. Güler



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Editorial

Metal-Free Restorations: Esthetic Considerations, Treatment Planning, Preparation, Manufacturing, Luting, and Followup

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The science and art of dental medicine are progressing with a vertiginous speed. Restorative and prosthetic dentistry can be considered as the fastest growing branches due to their close interactions with material science, chemistry, engineering, and computer science. Only two decades ago did dental practitioners had corrected the unsightly appearance of their patients' teeth by using full-coverage crown restorations. Ceramometal combinations were the material of choice for such restorations. In spite of the fact that these crowns were quite esthetic, resistant, and long lasting, they were still requiring invasive tooth preparation techniques. After the improvements in resin composite restorative materials and ceramics and manufacturing, bonding, and luting techniques and improvements in the preoperative design approaches, most of the dental operators have substituted the ceramometal full-coverage crowns with metal-free full or partial-coverage restorations made from resin composites or ceramics. Honestly, initial signs of these improvements have been introduced earlier, in the second half of the last century. These initial signs were the acid etching of the tooth enamel described by Buonocore and the chemical composition of the Bis-GMA introduced by Bowen. During the nineties, the velocity of the scientific investigations and researches shifted to describe, progress, compare, and observe the several restorative materials, manufacturing techniques, bonding, and/or luting agents and methods. Performed restorations have also been collected as case series, followed, analyzed, and reported. Nevertheless, the magnificent enlargement and the growing speed of the cumulative data subjecting the metal-free restorations are required to be reviewed and upgraded as frequent as possible. For this purpose the present special issue has been conducted to collect the most recent research reports that subjected the esthetic considerations, treatment planning, tooth preparation, manufacturing, luting, and the followup of the metal-free restorations.

Ten precious articles written by fifty authors from all over the world have been included in this special issue. The recent knowledge about the surface treatment methods of the hard tooth tissues as well as the glass-ceramics and zirconia; marginal fit and surface finishing of CAD-CAM fabricated monolithic and bilayered zirconia crowns; surface characteristics of the direct resin composite restoratives after bleaching are discussed. A meticulous literature review objecting glass-ceramic crowns is also included in order to help the readers to refresh their knowledge.

C. R. G. van den Breemer et al. systematically organized the current knowledge regarding the cementation of glass-ceramic materials and restorations in a comprehensive review. They additionally discussed the benefits of Immediate Dentin Sealing (IDS). D. P. Lameira et al. evaluated the effect of design and surface finishing on fracture strength of monolithic and bilayered zirconia crowns after

artificial aging. K.-H. Lee et al. investigated the marginal fit of metal-free crowns made by three different computer-aided design/computer-aided manufacturing (CAD/CAM) systems and G. Pompa et al. compared the marginal fit of the zirconia crowns with the laser sintered and cast metal crowns. Y. Lee et al. analyzed the microshear bond strength of self-adhesive resin cement on leucite-reinforced glass-ceramic, with or without surface treatment using pure silane or a universal adhesive. J.-S. Ahn et al. evaluated the effects of different phosphate monomer-containing primers on the shear bond strength between zirconia and MDP-containing self-adhesive resin cement. M. Piemjai and N. Nakabayashi reported the tensile strength and characteristics of dentin restored with all-ceramic, resin composite and cast metal prostheses cemented with resin adhesives. C. Bernard et al. assessed the effect of radiotherapy on bond efficiency of two different adhesive systems. R. J.-Y. Kim et al. observed the temperature changes at various sites within the composite and on the pulpal side of dentin during polymerization of composite increments. B. A. Irawan et al. exhibited the effects of the vital tooth bleaching on the color stability and 3D surface profile of dental restorative filling materials.

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Research Article

Tensile Bond Strengths of Two Adhesives on Irradiated and Nonirradiated Human Dentin

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The aim of this study was to assess the effect of radiotherapy on bond efficiency of two different adhesive systems using tensile bond strength test. Twenty extracted teeth after radiotherapy and twenty nonirradiated extracted teeth were used. The irradiation was applied *in vivo* to a minimal dose of 50 Gy. The specimens of each group were randomly assigned to two subgroups to test two different adhesive systems. A three-step/etch-and-rinse adhesive system (Optibond FL) and a two-steps/self-etch adhesive system (Optibond XTR) were used. Composite buildups were performed with a nanohybrid composite (Herculite XTR). All specimens were submitted to thermocycling ageing (10000 cycles). The specimens were sectioned in 1 mm^2 sticks. Microtensile bond strength tests were measured. Nonparametric statistical analyses were performed due to nonnormality of data. Optibond XTR on irradiated and nonirradiated teeth did not show any significant differences. However, Optibond FL bond strength was more effective on nonirradiated teeth than on irradiated teeth. Within the limitations of an *in vitro* study, it can be concluded that radiotherapy had a significant detrimental effect on bond strength to human dentin. However, it seems that adhesive choice could be adapted to the substrata. According to the present study, the two-steps/self-etch (Optibond XTR) adhesive system tested could be more effective on irradiated dentin compared to three-steps/etch-and-rinse adhesive system (Optibond FL).

1. Introduction

"Radio-induced caries" are a well-known consequence of the radiotherapy of head and neck cancer malignant tumors. Hyposalivation which is induced by irradiation [1, 2], dietary changes [3], and oral flora modifications [4, 5] are considered as the most important etiological factors of these caries [6]. Radio-induced caries begin near the gum and surround the cervical zone of the tooth leading to coronoradicular fracture [7]. The loss of mechanical autocleaning of these surfaces as a result of decreased salivary flow probably explains this location.

While there is lack of data published on this topic, evidences suggest a conservative approach using adhesive restorations [8]. Haveman and Redding have shown that conventional glass-ionomer cement (GIC) had poorer results than the resin-modified glass-ionomer cements (RMGICs) and composite fillings in patients treated by radiotherapy [9]. Moreover, according to several studies, it is not recommended to use the GIC as restorative material for patients suffering hyposalivation and having a daily fluoride application [10–12]. Composite resin restorations are an alternative for both esthetic and wear resistance.

The loss of adhesive restorations can be due to an alteration of dental tissues as a consequence of head and neck irradiation. A significant decrease of dentin microhardness has been observed after irradiation [13]. These observations were accompanied by reduction of the stability of the enamel/dentin junction [14]. The disturbance of

enamel/dentin junction could result in the formation of a gap (10 μm), loss of prismatic structure, and bacterial colonization associated with the obliteration of the dentinal tubules and odontoblastic process atrophy [15, 16]. These characteristics can be observed via scanning electron microscopy (SEM) [17, 18]. Furthermore, the radiogenic destruction of the dentin collagen could result in bonding failure [19].

As the loss of these restorations is time dependent, it was suggested as a reliable method to test the durability of the bond strength by accelerated ageing [20–27]. Thermocycling tests evaluate the stress of adhesive interface to water infiltration, mechanical and contraction/expansion tension by an alternative immersion in cold water (5°C) and hot water (55°C) [28]. This can result in cracks which propagate along the adhesive interface, a process known under the name of "percolation" [29]. This method of ageing is suitable for dental adhesive systems and recommended by the International Organization for Standardization (ISO, TR 11450) [30].

The purpose of this study was to evaluate the incidence of the radiotherapy on tensile strength of two adhesives on the human irradiated and nonirradiated dentin.

2. Material and Methods

2.1. Sample Preparation. Forty human extracted teeth (incisors, canines, premolars, and molars) were collected (gathered following informed consent). Twenty came from irradiated patients suffering from head and neck cancer. These teeth received a minimal dose of 50 Gy and were extracted because of periodontal disease. Twenty other teeth came from nonirradiated patients and were used as control group. All teeth were collected and stored in physiological solution for a period not exceeding two weeks; then, they were stored in distilled water at a temperature of 5°C. Class I cavities on molars and class V cavities on other teeth ($4 \times 4 \times 2$ mm) were prepared with a cylindrical medium-grit (100 mm) diamond bur (FG 068-040, Komet France SA, Paris, France) under constant water irrigation. The burs were changed for every 8 teeth.

2.2. Experimental Design and Bonding Procedures. Each group was randomly divided into 2 subgroups of 10 teeth. The subgroups were restored using a two-step/self-etch adhesive system (Optibond XTR, batch number 5092152, Kerr France, Créteil, France) or a three-step/etch-and-rinse adhesive system (Optibond FL, batch number 4995918, Kerr France, Créteil, France). The adhesive materials were used following manufacturer's instructions (Table 1).

Restorations were made using a nanohybrid composite resin (Herculite XRV Ultra, Kerr France, Créteil, France) with 2 layers of 1 mm thickness. Photopolymerization of the resin-based materials was performed using a LED light curing unit (Elipar S10, 3M ESPE, Cergy-Pontoise, France) at 1450 mW/cm^2.

Subsequently, the resin-bonded samples of each group underwent artificial ageing using thermocycling machine (10000 cycles for 2 weeks) with baths at temperatures of 5°C and 55°C (Table 2) and 30-second dwelling time. The storage solution of thermocycling baths was changed weekly.

2.3. Sticks Preparation. Thermocycled teeth were included in resin to allow fixation during microtensile sample preparation. Four to six slices, 1 mm thick, were cut perpendicularly and through to the bonded interface using Diamond Disk Wafering Blades 15HC (Buelher, Düsseldorf, Germany) under constant irrigation (IsoMet Low Speed Saw, Buelher, Düsseldorf, Germany). The sticks were then individualized and measured (±1 mm wide square section). The most peripheral sticks with residual enamel were excluded. A maximum of 4 sticks of the tooth central part were used trying to minimize the regional variability of dentin. The bonded surface area was calculated before each test by measuring the width with digital caliper.

2.4. Microtensile Bond Strength Testing (μTBS). Each specimen was attached following the methodology described by Perdigao et al. [31]. An aluminum device constituted of two symmetric parts, having a central notch (2 mm of depth and width) in order to allow autoalignment. Device surfaces were cleaned with alcohol. Tensile load was applied with a universal testing machine (DY34, Adamel Lhomargy SARL, Roissy-en-Brie, France), at a crosshead speed of 1 mm/min, to obtain the ultimate tensile strength, using a load cell of 1 KN.

2.5. Failure Mode Analysis. Fracture mode was determined at ×50 magnification with a stereoscopic microscope (Wild Heerbrugg TYP 376788, Wild Heerbrugg, Switzerland) and recorded as cohesive failure and adhesive failure.

2.6. Statistical Analysis. The experimental design included (i) two fixed crossed factors: irradiation [yes(I)/no(NI)] and adhesive system (XTR/FL) leading to 4 subgroups and (ii) a random factor (tooth) nested in each subgroup: 10 teeth per subgroup with one to four replicates per tooth. The conditions for the application of statistical treatment were carefully verified. The effect of the tooth factor on the explained variable (bond strength of sticks: μTBS) was first assessed by a mixed linear model on the full dataset. In case of nonapplicability of this mixed model, we conducted a one-way nonparametric ANOVA per subgroup using Kruskal-Wallis test. Missing data were supposed to be missing at random and no data imputation was performed.

In case of no tooth effect on μTBS, normality of μTBS data was checked graphically and using the normality Shapiro test for each of the 4 subgroups. In case of nonrespect of normality in one subgroup, pairwise distributions comparisons were performed between subgroups. Four comparisons were *a priori* of interest: between the two control subgroups (NI:XTR versus NI:FL), between the two irradiated subgroups (I:XTR versus I:FL), and for each adhesive system: (NI:XTR versus I:XTR) and (NI:FL versus I:FL). Correction for multiple comparisons was performed to maintain the family-wise error rate at the significant level of 5%. For 4 pairwise comparisons, Bonferroni correction gave a significant level of 2-tailed single test equal to 0.05/4, that is, 0.0125. Data were reported as mean ± SD per subgroup. Statistics were performed using the R language, version 3.1.2 available on the https://cran.r-project.org/ website. Package nlme was used to perform mixed linear model.

Table 1: Adhesive systems reference and composition.

Product name (manufacturer)	Class of adhesive	Composition	Batch number
Optibond FL, Kerr France, Créteil, France	3-step/etch-and-rinse adhesive	*Gel etchant*: 37.5% H_3PO_4, water, and fumed silica *Primer*: (Ph = 1.8): HEMA, GPDM, MMEP, water, ethanol, photoinitiator (CQ), and BHT *Adhesive*: Bis-GMA, HEMA, GPDM, GDMA, photoinitiator (CQ), ODmab, and fillers (fumed SiO_2, barium aluminoborosilicate, and Na_2SiF_6)	4995918
Optibond XTR, Kerr France, Créteil, France	2-step/self-etch adhesive	*Primer*: (pH = 2.4 before application, reduction in 1.6 to the application in dental structure). Acetone, water, ethanol, HEMA, photoinitiator (CQ), and GPDM *Adhesive*: ethanol, HEMA, sodium hexafluorosilicate, MEHQ; nanosilica, barium; photoinitiator (CQ)	5092152

HEMA: 2-hydroxyethyl methacrylate.
GPDM: glycerol dimethacrylate dihydrogen phosphate.
MMEP: mono(2-methacryloyloxy)ethyl phthalate.
CQ: camphorquinone.
BHT: butylated hydroxytoluene.
Bis-GMA: bisphenol A glycidyl methacrylate.
GDMA: glycerol dimethacrylate.
MEHQ: monomethyl ether of hydroquinone.

Table 2: Different constituents and brands of the thermocycling machine.

Bath of hot water	Brand, Fisherbrand (water bath heated, digital PID control UK plug 12L)
Bath of cold water	Fisher (Bioblock Scientific 18201)
Waterproof box for electric system	Schneider Electric, Telemecanique crouzet (ACM)
Timer for arm	Crouzet (Top 948, LCD MULTI-FUNCTION TIMER)

3. Results

Three teeth and one tooth out of 10 were missing in NI:XTR and I:FL subgroups, respectively. Linear mixed model was not appropriate because of the nonnormality of the normalized residuals ($p < 10^{-3}$). No effect of factor tooth was significant in each subgroup using Kruskal-Wallis test with p values ranging from 0.46 (I:XTR) to 0.84 (I:FL).

Due to the different number of samples by tooth, we obtained 15 observations for subgroup NI:XTR, 31 for NI:FL, 25 for I:XTR, and 27 for I:FL. The two subgroups relative to XTR exhibited nonnormal skewed distribution with p values $< 10^{-2}$.

Means and standard deviation of μTBS are graphically presented in box plots in Figure 1.

On irradiated dentin, both adhesive systems (XTR and FL) did not show any significant difference with μTBS in I:XTR subgroup equal to 12.2 ± 5.3 MPa (mean ± SD) and in I:FL subgroup 11.3 ± 2.8 MPa ($p = 0.97 > 0.0125$). On nonirradiated dentin, they did not show any significant difference on bond strength with μTBS in NI:XTR subgroup equal to 14.5 ± 4.8 MPa and in NI:FL subgroup 16.4 ± 6.2 MPa ($p = 0.42 > 0.0125$).

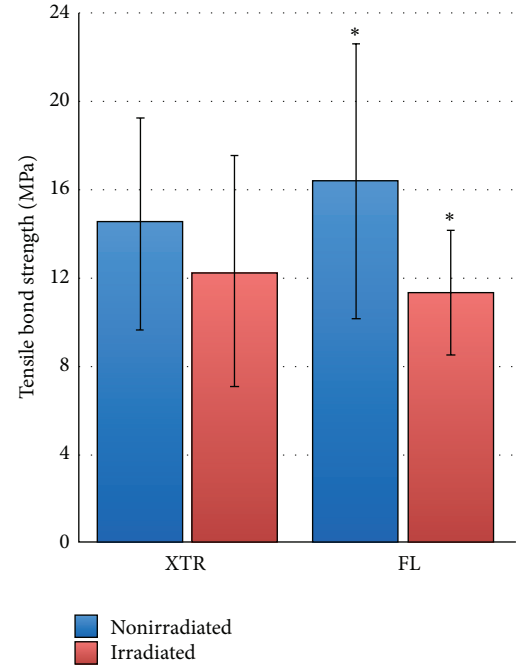

Figure 1: Mean of microtensile bond strength with standard deviation (MPa) according to the irradiation and the adhesive system (XTR/FL). *Significant difference between results ($p < 0.0125$).

Regarding FL groups, the value was significantly different between nonirradiated and irradiated dentin ($p = 0.0009 < 0.0125$). μTBS was observed 1.5 times higher in nonirradiated subgroup in case of FL (33% decrease from nonirradiated to irradiated subgroups). On the other hand, no statistical differences were found for XTR adhesive system ($p = 0.040 > 0.0125$) with μTBS observed 1.2 times higher in nonirradiated

Table 3: Adhesive and cohesive failure distribution.

	Adhesive fracture	Cohesive fracture
FL nonirradiated	48%	52%
FL irradiated	52%	48%
XTR nonirradiated	67%	33%
XTR irradiated	78%	22%

subgroup (16% of decrease from nonirradiated to irradiated subgroups).

The failure type for each group is summarized in Table 3. Adhesive failures at the composite resin/dentin interface were mainly observed for specimens treated with XTR. For FL adhesive system, there were as many adhesive fractures as cohesive failure.

4. Discussion

Head and neck cancers are one of the most common cancers [32]. Surgery and/or radiotherapy are the treatment of choice for such cancers [33]. Among the adverse effects like xerostomia or osteoradionecrosis, it has been demonstrated by several authors that radiation affects hard tissues [13, 34–37]. Regarding these consequences, some studies evaluated the bond strength on irradiated teeth. However, thermocycling for a sufficient time was not considered [38, 39]; furthermore, teeth were irradiated outside the oral cavity and after extraction [39, 40].

The procedure of the present study considers both the use of *in vivo* irradiated teeth and a sufficient thermocycling ageing protocol.

Teeth were stored in physiological saline solution immediately after extraction at the dental clinic and then in distilled water at 5°C. Even though Goodis et al. noticed that the physiological salt solution could have an action on dentin permeability and on traction resistance, unlike distilled water [41], Retief et al. have shown that saline solution does not influence the chemical and physical properties of human dentin [42].

Cavities were prepared using diamond bur under continuous water cooling to bring a higher traction resistance, compared with the abrasive 80-grits paper and to the diamond bur without irrigation [43]. As the experimental conditions should be standardized, dental composite resins were bonded on flat surface despite the overestimated bonding strength resulting in comparison to clinical conditions [44].

Regional differences in dentin anatomy and permeability have a significant influence on dentin bond strength [45, 46].

Photopolymerization time was applied according to the manufacturer recommendations and using the same light curing unit [47]. For all groups, the same resin composite and the same shade were used to avoid any influence of the composite material on bonding [48]. Several studies have shown the influence of the thermocycling ageing on adhesive systems strength [23, 26]. The standard (ISO TR 11450) recommends 500 cycles [30]. To simulate one-year ageing, as in the study of Gale and Darvell, a 10000-cycle experiment has been performed [28].

Several studies involving the two adhesive systems used in this work have been performed and have shown similar results [26, 49, 50]. Furthermore, according to De Munck et al. meta-analysis [51], Optibond FL, is the current reference in term of dentin bonding efficiency, on all the adhesives. These studies have been made on normal dentin. Nevertheless, the results obtained in this study showing μTBS decrease (33%) in irradiated dentin for FL subgroups are consistent with literature. It is reported that the ionizing radiations may have an effect on the collagen fibers of dentinal tubules [19, 52]. Moreover, the changes described in the crystalline structure of dental hard tissues after irradiation seem to affect tensile strength [53–56].

With the XTR adhesive system, the weak decrease of μTBS (16%) in irradiated dentin could be due to the chemical connections between the carboxylic or the phosphate groups of functional monomers and the phases of dissolved hydroxyapatite. These chemical connections would contribute to a better cohesion of the infiltrated resin after polymerization and, probably, in better resistance in the hydrolysis of this zone [57].

The results are in agreement with those of Naves et al. and S. Yadav and H. Yadav [40, 57]. Nevertheless, another similar study [58] did not find significant differences between irradiated and nonirradiated groups according to four adhesive systems, taking in consideration that no process of artificial ageing has been applied. In the present study, teeth were irradiated *in vivo* and, then, underwent adverse effects like hyposalivation.

5. Conclusion

The changes resulting from the irradiation on the hardness, the crystalline structure or the collagen matrix, seem to influence the adhesive agents bond strength to dentin. The dental substrate might have experienced radiation effects that could compromise bonding ability by impairing hybrid layer formation.

Under the limitations of this *in vitro* study, it appears that, regarding the type of adhesive system, radiotherapy may affect the microtensile bond strength of composite restorations on irradiated dentin. Therefore, it is advisable for a clinician to restore all cavities before radiotherapy and initiate caries prevention modalities in patients undergoing radiation therapy.

Further studies are needed to help the practitioner to adapt the choice of the adhesive system after radiotherapy of head and neck.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Research Article

Fracture Strength of Aged Monolithic and Bilayer Zirconia-Based Crowns

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The purpose of this study was to evaluate the effect of design and surface finishing on fracture strength of yttria-tetragonal zirconia polycrystal (Y-TZP) crowns in monolithic (1.5 mm thickness) and bilayer (0.8 mm zirconia coping and 0.7 mm porcelain veneer) configuration after artificial aging. Bovine incisors received crown preparation and Y-TZP crowns were manufactured using CAD/CAM technique, according to the following groups ($n = 10$): Polished monolithic zirconia crowns (PM); Glazed monolithic zirconia crowns (GM); Bi-layer crowns (BL). Crowns were cemented with resin cement, submitted to artificial aging in a chewing simulator (2.5 million cycles/80 N/artificial saliva/37°C), and tested for fracture strength. Two remaining crowns referring to PM and GM groups were submitted to a chemical composition analysis to measure the level of yttrium after aging. One-way ANOVA and Tukey's test ($P = .05$) indicated that monolithic zirconia crowns presented similar fracture strength (PM = 3476.2 N ± 791.7; GM = 3561.5 N ± 991.6), which was higher than bilayer crowns (2060.4 N ± 810.6). There was no difference in the yttrium content among the three surfaces evaluated in the monolithic crowns. Thus, monolithic zirconia crowns present higher fracture strength than bilayer veneered zirconia after artificial aging and surface finishing does not affect their fracture strength.

1. Introduction

The increase of esthetics' demand has led to the development of metal-free restorations without metallic components [1]. Dental ceramics present numerous favorable characteristics including biocompatibility and excellent potential to simulate the optical characteristics of natural teeth [2, 3]. However, the evaluation of clinical survival rates of posterior all-ceramic crowns and fixed dental prostheses (FDPs) reveals the vulnerability of those systems to various failure modes [4–6]. Therefore, several attempts have been made to improve the fracture strength of all-ceramic restorations, including the use of Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) due to its higher flexural strength [7] that allows the manufacturing of fixed partial prostheses (FPPs) in areas of high masticatory loads [8].

However, the strength of all-ceramic crowns relies upon the core as well as the veneer material, whereby a bilayer system with a strong and tough Y-TZP core veneered with translucent but brittle porcelain tends to fail prematurely. Moreover, these bilayer systems have several disadvantages including the multistep manufacturing process, low toughness of the veneer material, and weak bonding between veneer layer and coping [6]. Therefore, zirconia prostheses veneered with porcelain rarely undergo framework fracture, and chipping or cracking of the porcelain veneer is the most commonly reported complication [9–12]. The clinical survival rate of zirconia-based veneer restorations can be as high as 79–100% after 5 years [13–15] and chipping of the veneer layer is mostly reported for bilayers crowns in powder build-up technique [16, 17].

The alternative to circumvent all the bilayer systems' disadvantages is to replace the veneer/core bilayer with a monolithic restorative system [6]. Monolithic lithium disilicate fracture resistance appears promising while submerged in a wet environment [18], and its fatigue load-to-failure showed higher values than veneered Y-TZP crowns [19].

Fabricating zirconia monolithic restorations could improve the mechanical stability and increase the range of indications of those prostheses. However, its wear behavior and chemical stability have not yet been fully clarified. Zirconia presents three different crystal configurations depending on the temperature: monoclinic from room temperature to 1170°C; tetragonal from 1170°C to 2370°C; and cubic at temperatures above 2370°C. When cooling after sintering, this material undergoes volume expansion of 3% to 5%, which is related to the transition from tetragonal to monoclinic phase. Nonetheless, many oxides such as calcium (CaO), magnesium (MgO), yttrium (Y_2O_3), or ceria (CeO_2) may be added to zirconia to stabilize the tetragonal and stronger phase at room temperature [20, 21].

The concentration of the stabilizer plays a decisive role in the performance of this material under fatigue and the addition of 2-3 mol% of Y_2O_3 results in partially stabilized tetragonal zirconia, which is the most attractive composition for "transformation toughening" [22]. This mechanism is primarily responsible for the superior mechanical properties of zirconia, since it may undergo phase transformation from tetragonal to monoclinic under localized stress, with a subsequent increase of about 4 to 5% of local volume, inhibiting crack propagation [9, 23]. However, due to its metastable nature, zirconia-based materials are susceptible to unfavorable phase transformation at room temperature, and this phenomenon is known as "low temperature degradation" (LTD) [24, 25]. Aging occurs through an uncontrolled slow transformation of superficial grains from tetragonal-to-monoclinic phase in contact with water. This creates surface roughness and formation of microcracks, creating possibilities for water penetration causing further phase transformation and consequent loss of mechanical strength [24–26].

The aging process may induce yttrium loss and compromise the stability of the tetragonal phase of zirconia-based restorations, leading to uncontrolled tetragonal-to-monoclinic transformation [27]. It has been hypothesized that this mechanism occurs as a result of the reaction between water (H_2O) and yttrium (Y_2O_3) to form yttrium hydroxide ($Y(OH)_3$), which steadily drains the stabilizer, allowing for local conversion to the monoclinic phase [21, 28]. Apart from the aging controversy, the application of full-contour zirconia restorations is currently discussed as an alternative to bilayer veneered restorations based on the fact that clinical failures are observed mainly in the veneer layer [29]. In spite of reducing the possibility of early fracture by eliminating the weak phase (veneer layer) from the restorative complex, phase transformation is a reason for concern, since the direct contact with saliva under masticatory loads may aggravate the water penetration and crack propagation.

Hence, the purpose of this study is to compare the fracture strength and failure mode of two Y-TZP monolithic systems, either polished or glazed, and bilayer veneered Y-TZP crowns after prolonged artificial aging. The content of yttrium of the monolithic crowns after artificial aging was also investigated. The null hypothesis was that the crown design, monolithic or bilayer, had no effect on fracture strength of aged zirconia crowns.

Figure 1: Standardized crown preparation on a bovine incisor.

2. Material and Methods

2.1. Specimens' Preparation. Thirty-two healthy bovine incisors were used in this study, and a standardized crown preparation was performed in a lathe machine (Magnum-Cut; FEL-2680 GZJ) with the following dimensions: 4.2 mm diameter occlusal base, 6.0 mm diameter cervical base, and 7.0 mm axial height (Figure 1). The taper was established as 8 degrees for all axial walls and the cervical finish line was rounded shoulder. The tooth inner angles were rounded with fine grain diamond burs (KG Sorensen).

Specimens were randomly distributed in three groups ($n = 10$) according to the crown fabrication technique: PM group: monolithic zirconia polished crowns (1.5 mm thickness); GM group: monolithic zirconia glazed crowns (1.5 mm thickness); BL group: zirconia copings with hand-layered porcelain veneering (0.8 mm core and 0.7 mm porcelain thickness). Two additional crowns, referring to PM and GM groups, were submitted to an electron probe microanalysis (EPMA) to quantify the yttrium content after aging.

For fabrication of the nonanatomical crowns, all preparations were scanned by a noncontact optical 3D scanning device (Lava Scan system scanner; 3M ESPE). All zirconia crowns and copings were designed by the same technician with Lava Scan Design System. Then, zirconia blocks (Lava Plus for monolithic crowns, and Lava Frame for by-layer crowns) were milled by using the Lava CNC 500 milling machine (3M ESPE). After the milling procedure, all copings and crowns were sintered in a furnace (Lava Furnace 200) for approximately 11 hours. The fully sintered crowns referring to PM were finished and polished with diamond wheels and bristle brushes (Brasseler; dental instruments). The crowns referring to GM received glaze firing after the sinterization. A silicone impression was taken from one finalized specimen of PM in order to duplicate its 1.5 mm thickness to control the final thickness of the veneered crowns. Copings referring to BL were veneered with the powder build-up technique with Lava Ceram veneer ceramic (3M ESPE). The thickness of

the veneered porcelain and the contour of the final crown were verified by measuring the crown at different locations with a digital caliper, and the firing cycle was controlled by an experienced dental technician to ensure standardized crowns.

The crowns were cleaned for 10 min in an ultrasonic bath (Bransonic ultrasonic cleaner 3510 E-DTH; Branson), and 10 specimens of each group were cemented on their respective prepared tooth with a self-adhesive phosphate-based luting resin (RelyX Unicem 2 Automix; 3M ESPE). A static load of 5 kg [11, 30] was applied for 7 minutes following the cementation procedure following the manufacturer's instructions. The crowns for chemical analysis were cemented with temporary cement (RelyX Temp NE; 3M ESPE).

2.2. Specimens Aging. After luting, specimens were stored in distilled water at 37°C for 24 hours and submitted to an aging procedure: 2 500 000 cycles, 80 N, at 37°C under artificial saliva bath [31]. Loading was applied with a vertical displacement of 0.2 mm and horizontal (occlusal) displacement of 0.5 mm in a chewing simulator CS-4 (SD Mechatronik). As a substitute for human enamel, hydroxyapatite steatite indenters (3 mm diameter) were used as antagonists and were replaced for each specimen [32].

2.3. Fracture Strength Measurement. Aged specimens were loaded in a universal testing machine (Instron; model 8501) under deionized water bath at room temperature, with a 5 mm diameter ball indenter (stainless steel) at a crosshead speed of 0.5 mm/min. The maximum fracture load was measured by applying compressive load to the occlusal surface until the crown failed. Catastrophic fracture failure was considered as either the presence of visible cracks or sudden load drops or even acoustic events of chipping or fracture.

2.4. Failure Types' Analysis. The crowns were optically examined after fracture testing, and failure modes were divided into total core fracture, chipping of the veneer, or fracture at core/veneer interface. One representative specimen from each group was mounted on stubs with carbon adhesive tape and colloidal silver paint. Then, specimens were gold-sputtered and observed under scanning electron microscopy (SEM).

2.5. Surface Compositional Analysis. The two remaining specimens referring to PM and GM were used for quantification of yttrium content. The yttrium level was measured in 10 points starting from the worn occlusal surface (occlusal dimple) up to the most inner point of the coping of PM and GM and in a surface away from the occlusal load in PM undamaged. Compositional analyses were performed by using electron probe microanalysis (EPMA) on an electron microprobe (camera SX-50/51 DCI 1300 DLL) with 40-degree take-off angle and beam energy of 15 keV.

2.6. Statistical Analysis. Fracture strength and yttrium content were separately analyzed by using SPSS 19.0 for Windows (SPSS Inc.). One-way analysis of variance (ANOVA) and Tukey's test were used to compare mean and standard deviation (SD), with 95% confidence levels for both fracture strength and yttrium content.

Table 1: One-way ANOVA test results for fracture strength effect indicating significant difference amongst the groups.

Source	Sum of squares	df	Mean square	F	P
Between groups	12563505.80	2	6281752.900	7.571	.002
Within groups	22401284.20	27	829677.193		
Total	34964790.00	29			

Table 2: Mean fracture strength and Tukey's test results at 95% significance level.

Experimental group	Fracture strength (N)	Std. deviation	Tukey ($P = .05$)
Polished monolithic (PM)	3492.5	748.21	a
Glazed monolithic (GM)	3344.7	1159.45	a
Bilayered veneered (BL)	2051.8	764.76	b*

*Statistical difference among experimental groups ($P = .002$).

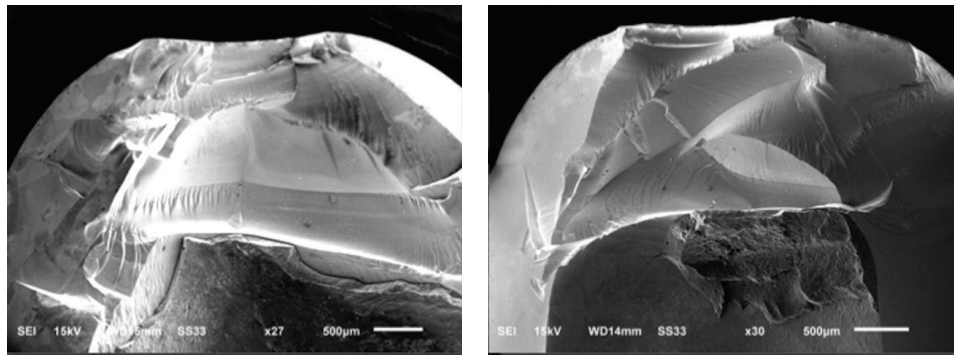
3. Results

3.1. Fracture Strength. All crowns withstood the artificial aging in the chewing simulator. One-way ANOVA indicated a significant difference among the groups ($P = .002$, Table 1). The fracture strength of monolithic zirconia crowns polished (PM = 3492.5 ± 748.2 N) and glazed (GM = 3344.7 ± 1159.4 N) was statistically similar ($P = .930$) and significantly ($P = .002$) higher than the results for the bilayer crowns (BL = 2051.8 ± 764.7 N, Table 2).

3.2. Failure Types Analysis. The failure pattern observed in PM and GM showed total crown fracture (Figure 2). All the specimens from group BL showed fracture at core/veneer interface without infrastructure damage.

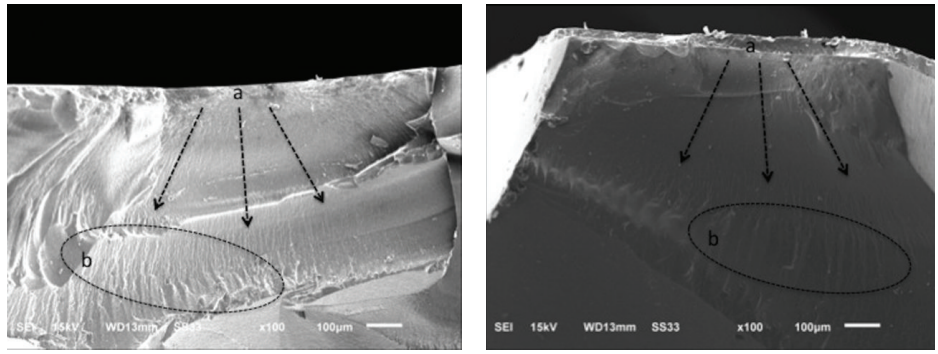
Fractographic analysis of PM and GM indicates that the direction of the crack propagation occurs from the occlusal surface to the center of the restoration. Based on failure patterns, hackles and lines are perpendicular to the crack origin (Figure 3). In BL, fractographic analysis shows that the critical flaw is located in the middle of the surface damaged inside the veneer layer (Figure 4).

3.3. Surface Compositional Analysis. One-way ANOVA of the yttrium content indicated statistically similar ($P = .935$, Table 3) concentration of yttrium among the surfaces. Mean and standard deviation for yttrium content may be observed in Table 4.



(a) (b)

Figure 2: Overview of scanning electron micrographs of polished monolithic crown (PM) ((a) ×27) and glazed monolithic crown (GM) ((b) ×30) fractured specimens.



(A) (B)

Figure 3: SEM micrographs of polished monolithic (PM) (A) and glazed monolithic (GM) (B) fractured specimens, indicating similar fracture mechanism between them, whereby crack propagation (arrows) starts at occlusal surface (a), and hackles and lines (b) perpendicular to crack origin may be observed.

Table 3: One-way ANOVA test results for yttrium content, indicating statistically similar values amongst the groups.

Source	Sum of squares	df	Mean square	F	P
Between groups	.001	2	.000	.067	.935
Within groups	.157	27	.006		
Total	.158	29			

Table 4: Mean yttrium content (wt%) in monolithic crowns and Tukey's test results at 95% significance level.

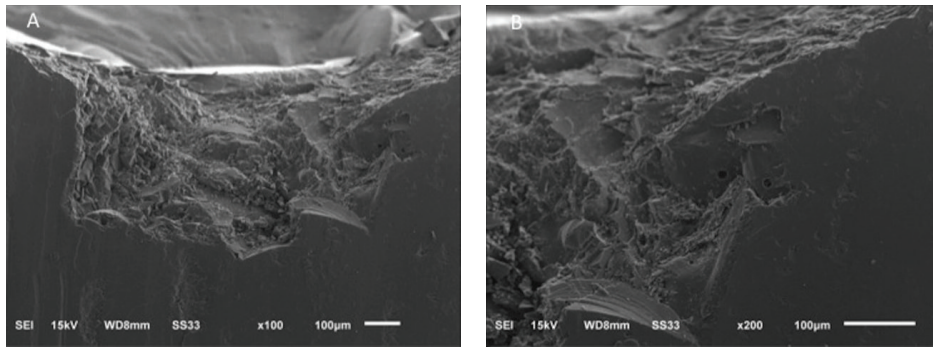
Experimental group	Yttrium content (wt%)	Std. deviation	Tukey ($P = .05$)
PM worn occlusal	2.0785	0.9361	a*
GM worn occlusal	2.0822	0.6728	a
PM undamaged	2.0700	0.6443	a

*Similar letters indicate statistically similar results among all groups ($P = .935$).

4. Discussion

The application of artificial aging before the fracture strength test aimed to simulate the effect of the oral environment on zirconia-based crowns by associating cyclic loading, an antagonist tooth, and artificial saliva. This reproduction of the in vivo condition was designed to observe changes representative of the expected clinical in vivo changes, which might result in the undesired phenomenon of low temperature degradation (LTD). 2.5 million mechanical cycles were selected to simulate 5-year aging in the oral environment, considering that an average adult would perform around 500 000 loading cycles/year [33, 34]. However, there is a large variation between number of cycles and the vertical loading applied in aging studies in the literature, with in vitro studies reporting the application of 5 000 to 400 000 cycles [32, 35–39]. Indeed, several studies performed 1 200 000 cycles with 50 N of vertical load [29, 40–42].

All crowns survived the artificial aging in the chewing simulator. This result indicates a stable performance



(a) (b)

Figure 4: SEM micrograph of porcelain fractured surface showing critical flaw (crack) in bilayer (BL) veneered fractured crown. Note chipping at the occlusal surface (a) and voids inside veneering layer (b).

of zirconia-based crowns under a constant load of 80 N during 5 years. Previous studies that evaluated the clinical performance of zirconia-based restorations demonstrated a survival rate of 79–100% after 5 years [13–15], with the most frequent clinical problem being the fracture of the veneering ceramic. Those results may be explained by the uneven masticatory loads presented in vivo, which also varies according to the type of food to be triturated by the posterior teeth. Moreover, other variables are present in the mouth, such as pH and temperature variations, and these effects on the fracture strength and chemical stability of all-ceramic crowns are not well known.

The null hypothesis that the Y-TZP crown design, monolithic or bilayer, has no effect on fracture strength was rejected. Therefore, the present study showed higher fracture strength of monolithic zirconia crowns in comparison to the bilayer configuration.

Previous studies analyzing the fracture strength of all-ceramic monolithic crowns indicate a superior performance for the monolithic design. Monolithic lithium disilicate restorations and hand-layer veneered Y-TZP core evidenced that the highest fatigue load-to-failure values were presented by monolithic crowns and the lowest for veneered Y-TZP crowns [19]. Even though the monolithic system was made of lithium disilicate, better results were obtained when compared to bilayer Y-TZP. According to the authors, the enhanced performance of monolithic crowns may be caused by the elimination of the interface between core and veneer, which is believed to be the weak link in bilayer systems.

Another in vitro study evaluated the load-bearing capacity of four different zirconia based crowns, including zirconia core with veneer layer produced either by powder build-up or CAD/CAM technique, glazed monolithic zirconia, and polished monolithic zirconia. The results showed that zirconia in bilayer configuration had significantly lower load-bearing capacity than the other crowns' design [16]. Nevertheless, it is important to consider that fracture load presented by all groups (PM = 3492.5 N; GM = 3344.7 N; BL = 2051.8 N) was still higher than maximum chewing forces reported in the literature, which is expected to be around 700 N for healthy young adults [43, 44]. Therefore, the results indicated that the fracture load presented by all groups tested in the study may tolerate the clinical applications without restrictions. However, clinical reports of failed bilayer zirconia-based restorations due to chipping or cracking are still commonly reported in the literature [10–12].

In the present study, the groups referring to monolithic crows, polished and glazed, showed a total core fracture pattern. This result was expected, since PM has only one material layer and GM has a thin glaze layer which leads to a bulk structural fracture. On the other hand, all the bilayer crowns showed fracture at core/veneer interface. Failure mode at the veneer layer has been reported for bilayers crowns, most commonly in powder build-up technique rather than in the sintering or pressed veneering technique [16, 17]. This technique, which is highly sensitive and more susceptible to variability due to the individual operator and the many firing cycles required, was used in the present study. The process may result in the addition of impurities and porosities, which maximizes the risk of crack propagation (Figure 3). Therefore, the technique and the low mechanical properties of the veneer material may be the reason for this mode of failure as well as for the lower fracture strength presented by specimens in BL group, since the inner coping was still intact after the mechanical testing. In contrast, there are some researches reporting complete failure (core/veneer) of all Lava CAD/CAM crowns [7] and total coping fracture [17].

There is still no consensus about the system triggering LTD, but three different rationales have been suggested in the literature. The first hypothesis is that water (H_2O) interacts with yttrium (Y_2O_3) generating yttrium hydroxide ($Y(OH)_3$), which totally compromises the stabilizer, leading to local yttrium deficiency that results in transformation of tetragonal to monoclinic phase. Another mechanism suggested is that water breaks the bond between Zr and O, resulting in localized stress growth as a result of –OH movement inside the crystal structure. This motion causes lattice fault that acts as nucleating agents for posterior crystalline changes. And the last theory is that O_2– from water breakdown fills oxygen vacancies [21].

The content of yttrium after aging was evaluated in previous studies. An in vitro study reported yttrium decrease (from 6.76 wt% to 4.83 wt%) after aging Vita In-Ceram YZ in boiling water for 7 days, confirming the first hypothesis for LTD's origin [27]. However, another research with the same experimental method reported no difference in the yttrium content after aging, even showing increase in monoclinic phase concentration (from 2 to 21%) [21]. The contradictory results between the first and the latter references may be related to the distinct chemistry of the zirconia substrates used.

In the current study, there was no difference in the yttrium content among occlusal worn surfaces and undamaged surfaces. Thus, this result can support the hypothesis that the chemical composition of monolithic crowns was not affected by the occlusal loading.

The results of this study demonstrated that monolithic zirconia-based crowns might have reliable fracture strength after 5 years of occlusal loading. Indeed, the fabrication of monolithic zirconia restorations might allow for extended clinical application, reducing a major drawback, which is fracture of veneering ceramic. However, future researches concerning whether temperature or ph variations can influence the fracture strength and chemical stability of monolithic zirconia crowns after artificial aging should be conducted. And in vivo studies should be performed to evaluate the clinical behavior of monolithic zirconia restorations.

5. Conclusion

According to the results of this study, Y-TZP monolithic crowns (polished and glazed) present higher fracture strength than bilayered veneered Y-TZP crowns. There was no evidence of yttrium depletion after 2.5 million cycles in artificial aging.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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Research Article

Comparison of Conventional Methods and Laser-Assisted Rapid Prototyping for Manufacturing Fixed Dental Prostheses: An In Vitro Study

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This study assessed whether there are differences in marginal fit between laser-fusion and conventional techniques to produce fixed dental prostheses (FDPs). A master steel die with 2 abutments was produced to receive a posterior 4-unit FDPs and single copings. These experimental models were divided into three groups ($n = 20$/group) manufactured: group 1, Ni-Cr alloy, with a lost-wax casting technique; group 2, Co-Cr alloy, with selective laser melting (SLM); and group 3, yttria-tetragonal zirconia polycrystal (Y-TZP), with a milling system. All specimens were cut along the longitudinal axis and their adaptation was measured at the marginal and shoulder areas on the right and left sides of each abutment. Measurements were made using a stereomicroscope (×60 magnification) and a scanning electron microscope (×800 magnification). The data were analyzed using one-way analysis of variance and the Bonferroni post hoc test, with a significance cutoff of 5%. Significant differences ($P < 0.05$) were observed between group 3 and the other groups. The marginal opening was smallest with Co-Cr alloy substructures, while the shoulder opening was smallest with Ni-Cr alloy substructures. Within the limitations of this study, the marginal fit of an FDP is better with rapid prototyping (RP) via SLM than conventional manufacturing systems.

1. Introduction

Fixed dental prostheses (FDPs) are the rehabilitation of choice after endodontic and operative treatments, especially among all over the world [1]. Moreover, the introduction of implant restorations has increased the popularity of fabricating crowns and bridges to rehabilitate the edentulous area. The development of both casting gold alloys and dental precision casting systems has contributed to the application of metallic restorations. However, patients are increasingly requesting metal-free restorations for aesthetics and biosafety reasons [2].

A poor marginal fit of crowns is responsible for 10% of prosthetic failures; these failures are mainly due to secondary caries, periodontal diseases, pulpitis, necrosis, and technical errors [3, 4]. Clinical trials have demonstrated the importance of the marginal fit in the quality assessment of fixed restorations and for their clinical success [5]. There are various opinions about the required marginal fit in the literature [6–9], but gaps of 100–150 μm are generally considered to be clinically acceptable [8, 10–14]. The marginal fit of FDPs is a fundamental requirement [15, 16] to achieve a clinically acceptable result with a good long-term prognosis [17–20].

Noble-metal alloys are generally preferred to base-metal alloys for the manufacture of FDPs due to their biocompatibility, good mechanical properties, and excellent ceramic-metal bonding [21]. However, nonprecious alloys, such as nickel-chromium (Ni-Cr) and cobalt-chromium (Co-Cr), are now commonly used for the substructure of metal-ceramic restorations due to economic considerations [22, 23]. The trend in modern dentistry is to use metal-free restorations, but metal-ceramic crowns are still the most

Table 1: Composition and properties of materials used and their manufacturing processes.

Product	Process	Alloy composition (Wt %)	Elastic modulus (GPa)	Vickers hardness (Kg/mm^2)	Yield strength (MPa, 0.2%)	Thermal expansion coefficient 10^{-6} μm/mK (25–500°C)
Cercon base (DeguDent) Ni-Cr alloy	Lost wax casting	Co 0.1 Cr 14–16 Al 1–3 Ni 71–75 Mo 8–10 Be 0.1–1.9 Traces of Ti	218	360	586	13.9
StarLoy LS (DeguDent) Co-Cr alloy	Selective laser melting (SLM)	Co 55.2 Cr 18.4 W 18.4 Fe 6.0 Al 2.0	210	390	938–1024	14.3
Biologic NA (Conero Dental) Y-TZP	Milling	ZrO_2 Y_2O_3 5% HfO_2 <2% Al_2O_3 and SiO_2 <1%	210	1200	900–1200	10.5

widely used ones due to their excellent mechanical properties and clinical performance, low cost compared to metal-free restorations, simple cementation technique, and role in natural reproduction of lost dentition in most restorative treatments [24]. The conventional technique for fabricating a metal structure is lost-wax casting. However, computer-aided design and manufacturing (CAD/CAM) technologies now allow the precise design of elements produced by specialized computerized equipment [25]. The use of CAD/CAM systems has been considered by several authors in the dentistry field, especially for the manufacture of FDPs [26–34]. Laser-assisted rapid prototyping (RP) is a CAD/CAM technology that was originally developed to fabricate prototypes for industrial purposes, and the use of RP systems can reduce the sensitivity and the technical complexity involved in the creation of a dental prosthesis [35].

This in vitro study compared the internal and marginal precisions of different posterior FDPs manufactured using three different methods: a milling system, laser-assisted RP through selective laser melting (SLM), and the lost-wax casting technique. The aim was to quantify the differences in accuracy between copings produced with SLM and the other techniques so as to provide an experimental basis for clinical research. The hypothesis of the study is the objective of allowing the clinician to rely on technology in increasingly more reliable and reproducible way, with a minimum margin of error in the realization of FDPs.

2. Materials and Methods

In the study the following methods have been used: (1) the CAD/CAM Cercon system and the Compartis system (DeguDent, Hanau, Germany) for manufacturing Y-TZP (zirconium oxide stabilized with yttrium oxide) single copings and bridges using a milling technique, (2) SLM for Co-Cr alloy, and (3) the conventional lost-wax technique for the realization of single crowns and bridges in Ni-Cr alloy. The compositions of the materials selected for this study and their processing techniques are provided in Table 1.

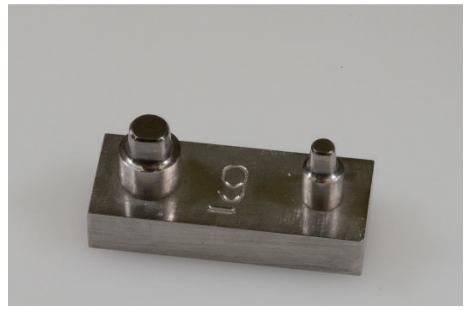


Figure 1: Master stainless-steel model.

2.1. Manufacturing the Models. A stainless-steel model with two abutments simulating a first premolar and a second molar screwed tightly on a holder (40 mm long, 16 mm wide, and 8 mm thick) was machined at the Mechanical and Aerospace Department of "Sapienza" University of Rome (Figure 1).

The abutments were positioned on the platform to receive posterior four-unit FDPs or two single crowns. The preparation had a finish line with a 1.0 mm radius, a 10-degree angle of convergence of the axial walls, and a shoulder margin, thereby simulating clinical conditions. A vertical flat surface parallel to the long axis of the abutments allowed the correct insertion of the structures and also ensured the stability of the platform. Specimens were measured using a digital caliper (Aura Dental, Germany) with an accuracy of 0.01 mm. The steel sample was used as the master model, and it was duplicated using silicone (Elite Double 22, Zhermack, Germany).

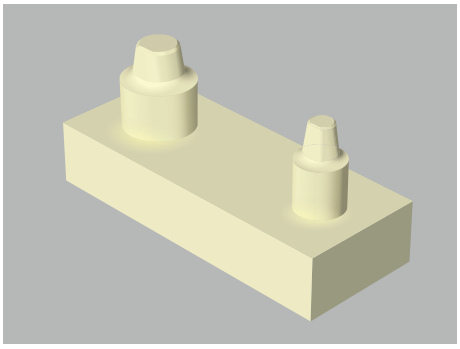


Figure 2: 3D model.

The master steel model was placed in a rigid plastic container with a top opening, and the silicone material was then poured in, with the expected hardening times following the manufacturer's instructions. Casts of polyurethane resin were then made (K.W. New Color, Techim Group), which were used as working dies. Thirty resin casts were constructed, and they were divided into three groups: group 1, Ni-Cr alloy; group 2, Co-Cr alloy; and group 3, Y-TZP. Each group comprised five resin casts for single copings and five resin casts for four-unit bridges ($n = 10$/group). The resin casts with single copings were then dissected to analyze the copings separately. The same protocol was used for the bridges. All of the samples used in the study were marked with an identification code ($n = 20$), with 10 single structures and 10 elements of bridges being fabricated for each group.

2.2. Manufacturing the Framework. The dies for copings in the Ni-Cr alloy group were coated with a layer of die spacer (total thickness of 20 μm) applied 0.5 mm above the margin (Yeti Die Spacer, Yeti Dental, Engen, Germany). The Ni-Cr alloy was cast by a private dental laboratory (SaviDent, Rome, Italy) using the conventional lost-wax technique and single casting to fabricate the restorations. Wax patterns were prepared and invested with carbon-free phosphate bonded investment material (GC Stellavest, GC Corporation, Belgium) in accordance with the manufacturer's instructions (Figure 2). The patterns were casted in Ni-Cr alloy (Biologic NA, Conroe Dental, Ancona, Italy) using an induction and centrifugation casting machine (Seit Elettronica, Italy). After divesting, the castings were cleaned with 50 μm Al_2O_3 using an air-borne-particle abrasion device (Basic Master, Renfert, Hilzingen, Germany). Each FDP was fabricated according to the manufacturer's instructions by the same dental technician. Y-TZP samples were digitized using the 3Shape D700 scanner (3S, DeguDent), which uses a laser-based optical scanning method. The 3S has a three-axis movement system that allows for an individualized scanning position of the casts. The CAD process was performed with the 3Shape Dental System software (3Shape A/S, Copenhagen, Denmark). All copings had a minimum thickness of 0.5 mm, which was consistent with the manufacturer's recommendation. All FDPs produced with the 3Shape system were fabricated in Y-TZP (Cercon Base, DeguDent) by a centralized milling center (Compartis, DeguDent) after transmitting the data via the Internet. Co-Cr alloy (StarLoy LS, DeguDent) copings were fabricated using the SLM technique. Scanning patterns and phase CAD were performed by the same method used for the Y-TZP samples. The production of substructures was outsourced to the Compartis Center (DeguDent).

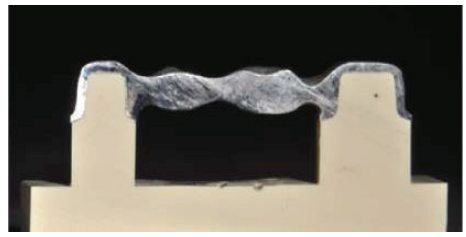


Figure 3: Sectioned framework.

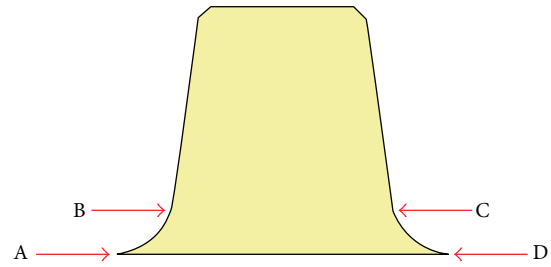


Figure 4: Representation of the four measuring points on each abutment.

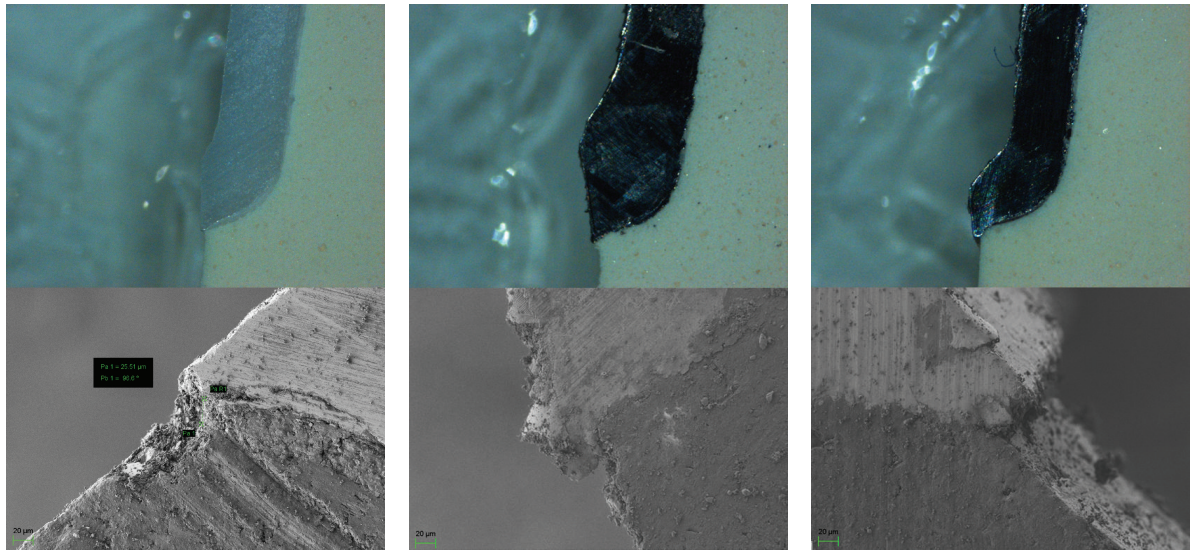
All of the structures were repositioned on their models and then checked for the correct positioning. If the positioning was incomplete, the structure was adapted using a standardized protocol according to the literature and clinical practice [35–39]. Areas to be corrected were identified by applying a spray lacquer (Contact-Spray, Protechno, Girona, Spain). The colored spots inside the cap were removed with a tungsten carbide bur while using a water spray to clean the debris. The same dental expert adapted and verified all of the restorations.

2.3. Cementation Process. All FDPs were cemented using a conventional glass ionomer (Ketac Cem Easymix, 3M ESPE, USA), mixed following the manufacturer's instructions. The cement was placed on the axial surfaces of the abutments so that cementation would simulate the clinical procedure as closely as possible.

Each restoration was set on pillars and subjected to a pressure of 50 N [8, 40–43] for 10 minutes using a compression testing machine with automatic recognition of calibration data at 50 N (Mecmesin, United Kingdom).

2.4. Analyses and Measurements of Marginal Fit. At 24 hours after cementation, each framework was sectioned centrally in the mesiodistal direction (Figure 3) with the aid of a cutting machine (Micromet, Remet, Bologna, Italy). The fit of the substructures was evaluated as illustrated in Figure 4.

For each substructure, the four measurement locations were used to determine the precision of the marginal and



(a) (b) (c)

Figure 5: Stereomicroscope and SEM micrographs of the marginal precisions of (a) a Y-TZP premolar abutment, (b) a Co-Cr premolar abutment, and (c) an Ni-Cr alloy premolar abutment.

internal fits: the marginal opening (points A and D), at the point of closest approximation between the model and the substructure, and the shoulder area (points B and C), corresponding to the internal adaptation of the substructure at 1 mm from the margin. Image analysis software (AxioVision Rel. 4.8, Zeiss, Germany) in combination with a stereomicroscope (Stemi 200-C, Zeiss) at ×60 magnification and a camera (AxioCam ICc1, Zeiss) were used for analyzing the marginal fit. The specimens were positioned in a base perpendicular to the optical axis of the microscope.

Given the thinness of the cement, additional micrographs were acquired using image analysis software (SmartSEM, Zeiss) in combination with field-emission scanning electron microscopy (SEM) at ×800 magnification (Auriga, Zeiss) at the Nanotechnology and Nanoscience Laboratory, SNN-Lab, Sapienza University of Rome, which was capable of performing low-voltage imaging of highly nonconductive materials. All measurements were performed by the same investigator, and the accelerating voltage was fixed at 1 kV (Figures 5(a), 5(b), and 5(c)) Measurements for each FDP were averaged, and these were used to determine the mean discrepancy in the marginal fit in each group ($n = 20$).

2.5. Statistical Analyses. The statistical analysis was carried out using Stata 12.0 software. Descriptive statistics included the calculation of mean ($\overline{x}$) and standard deviation (SD) values for all available measurements at each point. A one-way analysis of variance (ANOVA) was carried out to identify statistically significant differences between the investigated systems in terms of marginal fit at the different measurement locations confirmed with the nonparametric Kruskal-Wallis test. A Bonferroni post hoc test was used to compare the different groups. The cutoff for significance was set at 5% in all tests.

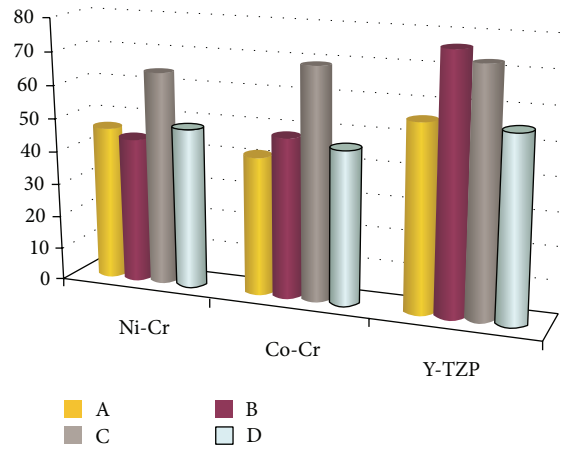


Figure 6: The total mean of marginal and internal discrepancies of 4 measurement points for each group (values in μm).

3. Results

The mean and SD values of the marginal and internal adaptations for all measurement points, tooth sizes, and production methods are presented in Tables 2 and 3. Measurements have been made in the same locations in stereomicroscope and SEM. The mean values for all measurement points are shown in Figure 6. The discrepancies were largest in the Y-TZP group. The discrepancies in tooth size between the premolar bridge, molar bridge, single premolar, and single molar were largest between the single premolar and the premolar bridge ($P < 0.05$).

Significant differences were present for the position parameter, with higher discrepancies at points A and B

Table 2: Mean values ($\overline{x}$) and standard deviation values (SD) of points A and B for all abutments in each group (values in μm).

Point A	Y-TZP $\overline{x}$ (±SD)	Co-Cr $\overline{x}$ (±SD)	Ni-Cr $\overline{x}$ (±SD)	pV
Premolar abutment	51.37 (10.62)	43.88 (15.25)	48.49 (15.12)	0.696
Molar abutment	60.88 (6.56)	47.06 (18.96)	58.92 (15.20)	0.302
Single abutment	56.37 (9.09)	33.08 (7.56)	31.53 (5.30)	0.001
Single abutment	56.03 (5.45)	43.80 (17.26)	43.68 (21.68)	0.571
Point B	Y-TZP $\overline{x}$ (±SD)	Co-Cr $\overline{x}$ (±SD)	Ni-Cr $\overline{x}$ (±SD)	pV
Premolar abutment	78.62 (18.25)	39.61 (12.90)	27.23 (16.18)	0.001
Molar abutment	75.47 (20.00)	73.60 (24.54)	69.64 (20.19)	0.911
Single abutment	77.87 (12.86)	30.79 (9.60)	36.08 (21.95)	0.002
Single abutment	74.25 (23.33)	48.65 (19.06)	42.07 (21.52)	0.181

Table 3: Mean values ($\overline{x}$) and standard deviation values (SD) of points C and D for all abutments in each group (values in μm).

Point C	Y-TZP $\overline{x}$ (±SD)	Co-Cr $\overline{x}$ (±SD)	Ni-Cr $\overline{x}$ (±SD)	pV
Premolar abutment	52.02 (24.20)	77.60 (32.55)	73.87 (21.49)	0.404
Molar abutment	96.98 (17.13)	93.26 (43.39)	79.03 (18.05)	0.597
Single abutment	57.72 (11.21)	44.12 (15.96)	40.40 (16.38)	0.262
Single abutment	79.24 (17.01)	63.15 (22.38)	60.69 (26.21)	0.488
Point D	Y-TZP $\overline{x}$ (±SD)	Co-Cr $\overline{x}$ (±SD)	Ni-Cr $\overline{x}$ (±SD)	pV
Premolar abutment	45.94 (8.05)	46.42 (17.61)	40.75 (10.56)	0.743
Molar abutment	60.02 (9.48)	53.66 (18.87)	64.11 (18.28)	0.600
Single abutment	56.74 (29.73)	36.51 (13.09)	50.27 (6.92)	0.292
Single abutment	58.48 (22.72)	48.18 (26.27)	35.70 (21.53)	0.427

($P < 0.05$). The best fit—independent of the different parameters—was at points A and D for the SLM technique and at points B and C for the lost-wax technique. The mean marginal discrepancy was 47.56 μm for the Ni-Cr alloy, 55.6 μm for Y-TZP, and 43.92 μm for the Co-Cr alloy. The mean internal gap was 54.11 μm for the Ni-Cr alloy, 74.73 μm for Y-TZP, and 58.76 μm for the Co-Cr alloy. ANOVA revealed statistically significant differences ($P < 0.05$) in marginal and internal adaptations among the groups at the four measurement points.

4. Discussion

RP technology has attracted enormous interest among researchers because it greatly facilitates the realization of bespoke three-dimensional (3D) objects. A focused high-power laser beam can selectively melt layers of metal alloy powder in a mass using thermal energy and so can be used to produce any desired 3D object under computer control. After each section is scanned, the thickness of the powder bed of an alloy of the base metal is lowered by one layer, and a new layer of metal-based alloy is applied on top. This process is repeated until the part is completed.

In addition, the remaining unprocessed powder can be reused, in contrast with conventional methods in which most of the material is wasted and there are spatial limitations restricting the production of complex shapes [44]. SLM technology is characterized by remarkable precision, the possibility of building virtually any required dental geometry, and a constant surface speed and therefore a high-quality milling (especially of undercuts) thanks to the availability of four-axis simultaneous milling.

The optimal clinical marginal gap remains controversial. McLean and von Fraunhofer [44] found that a prosthetic restoration is successful if the marginal gap is less than 120 μm. Based on this criterion as the limit of clinical acceptance, the mean marginal and internal discrepancy values were clinically acceptable in all three groups in the present study.

While further research is necessary to optimize the process parameters and clinical applications, the laser-assisted RP procedure reported herein provides an efficient and rapid method for digitally designing and manufacturing complex metal structures for FDPs. It should be noted that many of the samples exhibited wide variations of the marginal gap; for example, while one surface was accurate to a few microns, there were large openings on the contralateral side. This may have been due to small displacements of the structure during corrections, which could have resulted in incomplete seating of the substructure and the largest marginal openings,

or there may have been inaccuracies during cutting. All restorations were evaluated after the corrections had been made by the dental laboratory. Since the master model was duplicated in numerous resin models, there were many variables that could potentially change the results during the laboratory work, including the duplication time, small changes in water/powder ratio, water temperature, and wax distortion. In this study all of the substructures were placed and cemented in their respective resin models to test the differences between the different manufacturing techniques and the different materials used at the levels of the marginal gap and shoulder area.

The study was subject to several limitations.

(1) All structures were adapted using a standardized protocol, and retouching was performed by the same technician in order to avoid large inaccuracies.

(2) There was a possibility of samples being damaged during the cutting process. This risk was minimized but cutting under a water spray and using low feeding rates.

(3) All samples were produced and tested under ideal conditions, which might not accurately reflect typical clinical use.

(4) If the position of the framework was incomplete, the structure was adapted manually by technician using a standardized protocol with a margin of human error.

5. Conclusion

This study investigated the application of different techniques for the manufacture of FDPs. Within the limitations of this in vitro study, the following conclusions can be drawn.

(1) Copings produced with SLM technology have better marginal adaptation within an acceptable range.

(2) The type of metal alloy did not significantly affect the measurements.

(3) The marginal and shoulder areas presented greater discrepancies in values between metal alloys and Y-TZP.

(4) The cement gap was wider in the region of the shoulder than at the point of closure.

(5) All of the techniques and materials tested resulted in acceptable marginal openings in vitro.

The RP technique is a substantial innovation for the manufacture of dental prostheses, allowing dentists to work more easily and faster while still ensuring the production of a high-quality finished product, due to significantly decreases in the risk of human error. It was concluded that, within the limitations of this study, the RP system can compete with conventional systems and can achieve a good marginal fit in vitro.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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Research Article

3D Surface Profile and Color Stability of Tooth Colored Filling Materials after Bleaching

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This study aims to evaluate the effects of vital tooth bleaching with carbamide peroxide home bleaching and in-office bleaching on the color stability and 3D surface profile of dental restorative filling materials. Thirty discs ($n = 30$) measure 6 mm in diameter and 2 mm thick for each of three restorative materials. These are nanofilled composite Filtek Z350 XT, the submicron composite Estelite Σ Quick, and nanofilled glass ionomer Ketac N100 nanoionomer and were fabricated in shade A2. Each group was further divided into three subgroups ($n = 10$): subgroup A (Opalescence PF), subgroup B (Opalescence Boost in-office bleaching), and subgroup C (distilled water) serving as control. Samples were bleached according to the manufacturer's instructions for a period of two weeks. The Commission Internationale de L'Eclairage (CIE L^*, a^*, b^*) system was chosen for image processing, while 3D surface profile was tested with atomic force microscopy (AFM). Statistical analyses were performed with the Mann-Whitney tests and Krusal-Wallis with a P value of ≤0.05. The three restorative materials showed significant color changes (ΔE); $P \leq 0.05$. In diminishing order, the mean color changes recorded were Estelite Σ (3.82 ± 1.6) > Ketac Nano (2.97 ± 1.2) > Filtek Z350 XT (2.25 ± 1.0). However, none of the tested materials showed statistically significant changes in surface roughness; $P > 0.05$.

1. Introduction

Home bleaching has gained considerable acceptance among dentists and patients as a simple, effective, and safe procedure to lighten discolored teeth. Since its introduction by Haywood and Heymann in 1989 [1], tooth whitening has become one of the most popular esthetic procedures offered by dentists. There are several types of bleaching methods, but all of them share the common principle of the degrading of peroxides from hydrogen peroxide or its compounds such as carbamide peroxide (CP) into unstable free radicals. These radicals are further broken down into large pigmented molecules through either oxidation or reduction reaction [2].

The oxidation or reduction process changes the chemical structure of the interacting organic substances of the tooth, which results in the change in color [2]. Furthermore, Maleknejad et al. [3] reported an increase in the diameter of dentinal tubules at a concentration of 45% CP as a result of different intracoronal tooth-bleaching procedures. They also reported alterations in the mineral content of the dentin.

Bleaching methods include nonvital bleaching, in-office professional bleaching, and home bleaching. Tray-delivered home bleaching uses a relatively low concentration of whitening gel, which is applied to the teeth by means of a custom fabricated tray, which is worn at night for the duration of at least two weeks [4].

Considerable research has been carried out to identify the effects of bleaching on the tooth surface and dental restorative materials. Jacob and Dhanya Kumar [5] reported that bleaching with CP might affect the marginal leakage of resin composite restorations; however, amalgam restorations were not adversely affected *in vitro*. The CP agents were observed to have a profound influence on the color behavior

of tooth colored restorations [6] or perhaps even cause deterioration [7]. Interaction between the bleaching agents and the restorative material may result in noticeable color change especially if the color closely matched the tooth structure before bleaching [8]. Thus, as result of bleaching, the end result could be an esthetic failure due to an incorrect color match. As a result, replacement of the existing restoration may be required. Studies have shown that the color stability of tooth colored restorative materials depends on the type of material [8].

Composite resin and glass ionomers are the most popular dental restorative materials. They offer superior esthetics, require minimal tooth preparation, and are widely used for anterior and posterior restorations. Recently a nanofilled resin composite and glass ionomer were introduced and exhibit a high initial polish while retaining this over time combined with excellent physical properties [10].

In composite resin technology, particle size and quantity are crucial when determining how to best utilize the restorative materials. Alteration of the filler component remains the most significant development in the evolution of composite resins [11]. The filler particle size, distribution, and the quantity incorporated dramatically influence the mechanical properties and clinical success of composite resins [12].

Filtek Z350 XT is nanohybrid resin composite material. To moderate the shrinkage, PEGDMA has been substituted for a portion of the TEGDMA resin. The fillers are a combination of nonagglomerated/nonaggregated 20 nm silica filler, nonagglomerated/nonaggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles). The nanocomposites have an average cluster particle size of 0.6 to 10 microns while the inorganic filler loading is about 72.5% by weight (55.6% by volume).

Estelite Σ Quick is microhybrid composite resin which contains BisGMA and triethylene glycol dimethacrylate (TEGDMA) with filler size of 0.1–0.3 μm and filler loading is 82% by weight or 71% by volume.

Generally, glass ionomer restoratives can contain a broad range of particle sizes. Filler particle size can influence strength, optical properties, and abrasion resistance. By using bonded nanofillers and nanocluster fillers, along with FAS glass, nanoionomer restorative has improved esthetics and low wear yet still provides the benefits of glass ionomer chemistry, such as fluoride release. Overall, nanoionomer restorative exhibits impressive surface characteristics.

It has been reported that bleaching agents reduce the microhardness of enamel and promote an increase in surface roughness [13]. There exists a significant and positive correlation between surface roughness and bacterial adhesion [14]. Roughness has a major impact on esthetic appearance, discoloration of restorations, plaque accumulation, secondary caries, and gingival irritation [15]. Interaction between bleaching agents and the restorative material may result in noticeable color change especially if the color matched the tooth structure closely before bleaching.

On the other hand, studies have also shown that the effect of bleaching agents is minimal with regard to roughening of composite resin surfaces and that they do not perceptibly change the shade of tooth colored materials [16]. However, another study concluded that nanofilled resin composites are more resistant and are preferred as a restorative material when bleaching treatment is indicated [17].

In this study, samples were analyzed using the CIELAB color technique. Standard Commission Internationale de L'Eclairage (CIELAB) is color system that assesses chromatic differences in colorimetry. The use of digital cameras to capture accurate color in dentistry is advantageous in the color replication process for any craniofacial prosthesis, given the potential to capture the polychromatic color of the structure, as well as form, texture, and perceived translucency [9]. A color difference of $\Delta E = 2$ in the CIELAB color systems was detectable by the human eye under uniformly controlled conditions [18].

There is limited data on the effects of bleaching agents on microhybrid and nanofilled composite resins, as well as the new nanofilled glass ionomers. There is also scant knowledge concerning the effect of the in-office and home bleaching systems on these latest developments. Current study hypothesized that there are no differences between in-office and home bleaching systems on the color stability and 3D surface profile of tooth colored restorative materials. The aim of this *in vitro* study was to evaluate the effect of home versus in-office bleaching systems on the color stability and 3D surface profile of tooth colored restorative materials.

2. Materials and Methods

The Ethics Committee of the Universiti Sains Malaysia (Kelantan, Malaysia) reviewed and approved the research project. This *in vitro* study evaluated the color stability and 3D surface profile of three tooth colored restorative materials after bleaching. Two commercially available nanohybrid and microhybrid, Bis GMA-based resin composites and one nanoionomer, all with an A2 shade, were used in the present study (Table 1). Properties of the research materials were presented in Table 2. Samples were fabricated in 2 mm thick plexiglass with a circular opening of 6 mm. After insertion of the test material polyethylene was applied and the material pressed down with glass slabs. Excess material was removed. A total of 90 samples were prepared, thirty samples ($n = 30$) for each test material (Group I ($n = 30$): Filtek Z350 XT nanohybrid composite[1], Group II ($n = 30$): Estelite Σ microhybrid composite[2], and Group III ($n = 30$): Ketac N100 nanoionomer cement[1]).

All samples were light-cured from the top and bottom using an Elipar Freelight 2[1] according to the manufacturer's instructions with an output of 1000 mW/cm^2 and wave length of emitted light of 430–480 nm. The nanoionomer cement after placement was light-cured for 20 s, while the resin composite materials were placed incrementally into the circular opening and light-cured for 20 seconds each increment.

All samples were then polished using Sof-Lex[1] from coarse (55 μm) to medium (40 μm) to fine (24 μm) and ultrafine (8 μm), using a mandrel and a slow-speed hand piece. Polishing was carried out without water for 10 seconds

Table 1: Composites resin and bleaching agents tested.

Materials	Composition	Manufacturer	Batch number
Filtek Z350 XT (nanohybrid composite resin)	BisGMA, Bis-EMA, UDMA, and TEGDMA Filler size of 5–20 nm Filler loading is 78.5% by weight or 58.5% by volume	3M ESPE, St. Paul, MN, USA	N179865
Estelite Σ Quick (microhybrid composite resin)	BisGMA and triethylene glycol dimethacrylate (TEGDMA) Filler size of 0.1–0.3 μm Filler loading is 82% by weight or 71% by volume	Tokuyama Dental, Tokyo, Japan	E542
Ketac N100 (resin-modified nanoglass ionomer)	Based on the methacrylate-modified polyalkenoic acid Deionized water Methacrylate: blend including HEMA Polyalkenoic acid: VBCP Glass: acid-reactive FAS glass, nanoparticles, and nanoclusters	3M ESPE, St. Paul, MN, USA	N389644
Opalescence home bleaching: Opalescence PF	20% carbamide peroxide Potassium nitrate 0.11% fluoride ions	Ultradent Products Inc., South Jordan, UT, USA	
Opalescence in-office bleaching chair-side Whitening: Opalescence Boost	40% hydrogen peroxide	Ultradent Products Inc., South Jordan, UT, USA	

Bis-GMA: bisphenol-glycidyl methacrylate; BIS-EMA: ethoxylated bisphenol A glycol dimethacrylate: UDMA: urethane dimethacrylate: TEGDMA: triethylene glycol dimethacrylate: HEMA: hydroxy ethyl methacrylate; VBCP: vitrebond copolymer; FAS: fluoroaluminosilicate.

Table 2: Properties of research materials.

Material	Type of material	Properties
Filtek Z350 XT	Nanohybrid composite resin	(i) Nanofiller improves compression strength and/or hardness, flexural strength, elastic modulus, coefficient of thermal expansion, water absorption, and wear resistance (ii) Optimizing the adhesion of restorative biomaterials to the mineralized hard tissues of the tooth is a decisive factor enhancing the mechanical strength, marginal adaptation, and seal, while improving the reliability and longevity of the adhesive restoration
Estelite Σ Quick	Microhybrid composite resin	(i) Outstanding polishability (ii) Wide shade matching range (chameleon effect) (iii) High gloss retention over time (chameleon effect) (iv) High wear resistance (v) Low shrinkage (vi) Good radiopacity
Ketac N100	Resin-modified nanoglass ionomer	(i) Nanoionomer is the first paste/paste, resin-modified glass ionomer developed with nanotechnology (ii) Using fluoroaluminosilicate (FAS) technology (iii) Exhibiting impressive surface characteristics (iv) High fluoride release (v) Improved wear resistance (vi) Radiopaque (vii) Light cure on demand
Opalescence PF	Home bleaching	(i) Low concentration of 20% carbamide peroxide, potassium nitrate, and fluoride ions
Opalescence Boost	In-office bleaching chair-side Whitening	(i) High concentration of 40% hydrogen peroxide

per disk. An effort was made to standardize downward force and number of strokes for each disk. After polishing, the samples were cleaned ultrasonically using a Sonica 2200 ETH[3] for 5 minutes and then stored in distilled water at 37°C for 24 hours prior to the bleaching treatment.

Each group was further divided into three subgroups of 10 specimens each ($n = 10$). The specimens were subjected to bleaching agents following the manufacturers' instructions. Samples in subgroup A (control group) were not bleached but stored in a vibrating distilled water bath[4] for 14 days at 37°C.

Samples in subgroup B were subjected to Opalescence home bleaching PF, a 20% CP[5], for four hours per day for 14 days according to the instructions of the manufacturer.

Subgroup C was treated with Opalescence in-office bleaching chair-side Whitening Boost, a 40% CP[5], for 40 minutes (2 × 20 minutes) per day for a total of 5 days. The mixing procedure of the Opalescence Boost bleaching gel and application of a 0.5–1.0 mm thick layer of gel on the sample was carried out according to the manufacturer's instructions.

In subgroups B and C, prior to each bleaching procedure, the samples were removed from the distilled water bath and air-dried with an oil-free air jet spray for 60 seconds. The bleaching agent was applied on one surface using a microbrush[6] and left in place for the duration suggested by the manufacturer. After each bleaching procedure, the samples were washed with an air/water spray for 60 seconds before they were stored again in distilled water at 37°C until the next bleaching session.

Duration time of bleaching for each subgroup followed the manufacturer's instructions. The bleaching protocol was carried out daily for two weeks.

2.1. Analysis of Color Stability. The samples were placed on a neutral grey card and photos were taken with a Nikon D200 digital camera in a darkened room with the main source of light coming from two Phillips F15TS 15 watt bulbs at 45 degrees (Figure 1). CIELAB color values were analyzed from digital raw images taken from the samples using software Photoshop CS3 Ver 10.0. All specimens were measured twice and the average values were calculated. The calculations of the color variations (ΔE) were made between two color positions.

The CIE LAB-based color difference formula, introduced in 1976 and recommended by the International Commission on Illumination [9], defines a color space ($L^*a^*b^*$) in which L^* represents lightness, a^* represents the chromaticity coordinate for red-green (Ca^*Z red direction; Ka^*Z green direction), and b^* represents the chromaticity coordinate for yellow-blue (Cb^*Z yellow direction; Kb^*Z blue direction). The magnitude of total color difference (between baseline and after bleaching measurements) is represented by a single number ΔE (Commission Internationale de L'Eclairage, 1979):

$$\Delta E = \left[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right]^{1/2}, \quad (1)$$

where ΔL^*, Δa^*, and Δb^* are the respective difference between the measured and predicted CIE LAB values of the shade.

2.2. 3D Surface Profile Measurements. Six samples from each group were subjected to 3D surface profile evaluation using atomic force microscopy (AFM)[7]. The mean 3D surface profile was assessed with a contact mode. Five different randomly selected areas were scanned with an area of 40 × 40 μm and resolution of 512 × 512 pixels to obtain surface roughness values (Ra). Ra analysis was done by ScanAtomic SPM control software. Then, three-dimensional (3D) images with 10 × 10 μm sizes were acquired for each group of materials (Figures 2, 3, and 4).

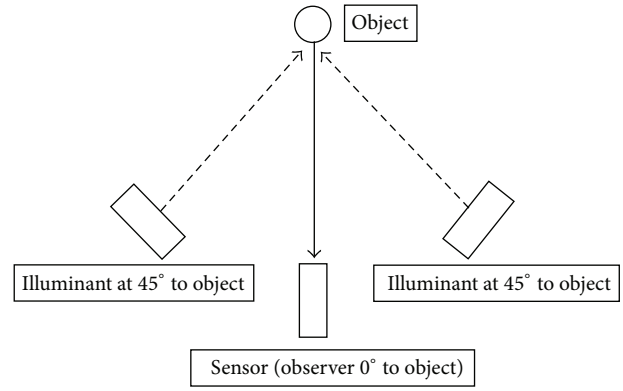

Figure 1: Schematic view of the experimental set-up [9].

Table 3: Comparison of ΔE between 3 different tooth colored restorative materials after bleaching (between restorative materials).

Restorative material	Bleaching agent	Mean ± SD	P value
Filtek Z350 XT	20% CP HB	2.2 ± 1.02	0.0390*
	40% CP OB	2.6 ± 1.1	
Estelite Sigma Quick	20% CP HB	3.7 ± 1.5	0.0020*
	40% CP OB	3.0 ± 1.2	
Ketac N100	20% CP HB	3.1 ± 1.2	0.0160*
	40% CP OB	2.7 ± 1.2	

Mann-Whitney test; * P value < 0.05 is significant; CP: carbamide peroxide.

The data collected were analyzed using SPSS version 16.0. All of the statistical analysis was conducted at a significance level of $P < 0.05$ using the Mann-Whitney and Kruskal-Wallis test.

3. Results

ΔE is compared within the groups and between the subgroups.

Table 3 shows a comparison of ΔE value between restorative materials when treated with a different bleaching agent. Filtek Z350 XT has a higher mean color change as a result of in-office bleaching compared to home bleaching. In contrast, Estelite Sigma Quick and Ketac N100 demonstrated a higher ΔE value after home bleaching compared to in-office chair-side bleaching.

Table 4 shows the comparison of mean color change of the restorative materials between the two bleaching agents. The mean color change of Ketac N100 was the highest, followed by Estelite Sigma Quick for both in-office and home bleaching agents. Filtek Z350 XT showed the least color changes.

Table 5 presents the roughness numbers (Ra) of the restorative materials that were bleached. Statistically insignificant changes were found in roughness for all three materials tested after 14 days of bleaching with 10 and 20% CP compared to the control group.

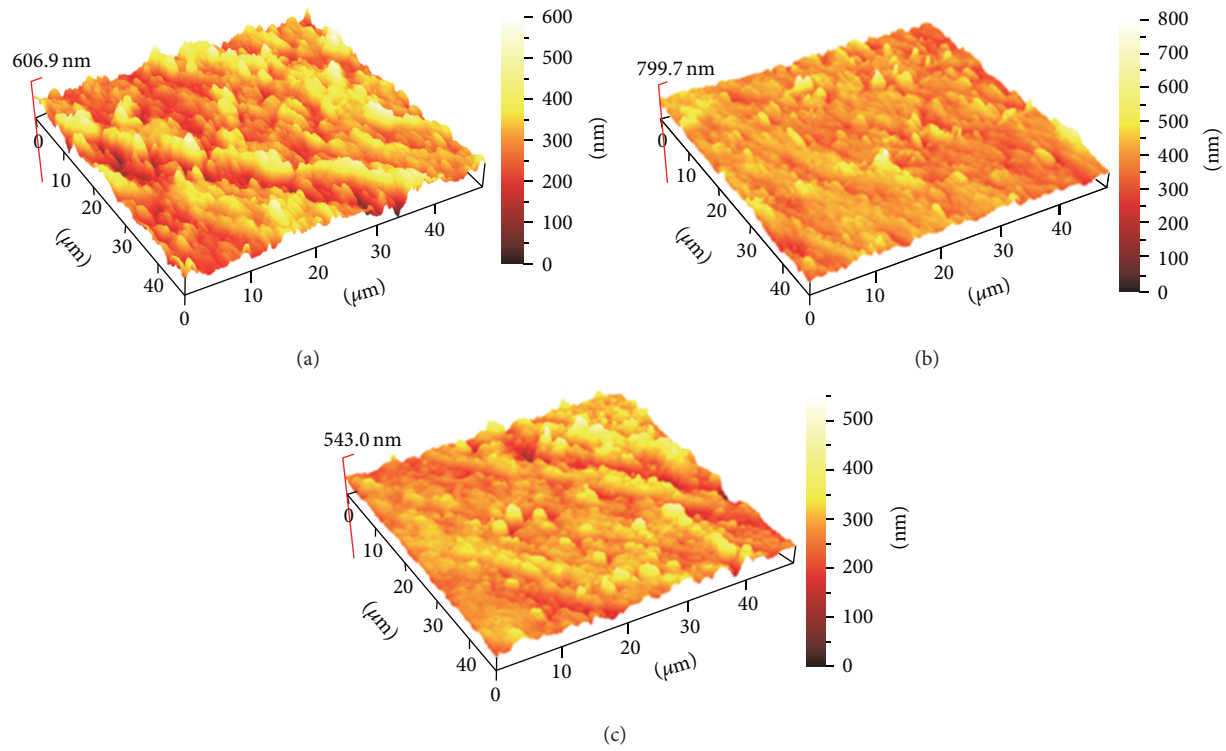

Figure 2: The AFM-3D images of Z350 XT surface roughness: (a) Z350 XT without bleaching, (b) Z350 XT with home bleaching, and (c) Z350 XT with in-office bleaching.

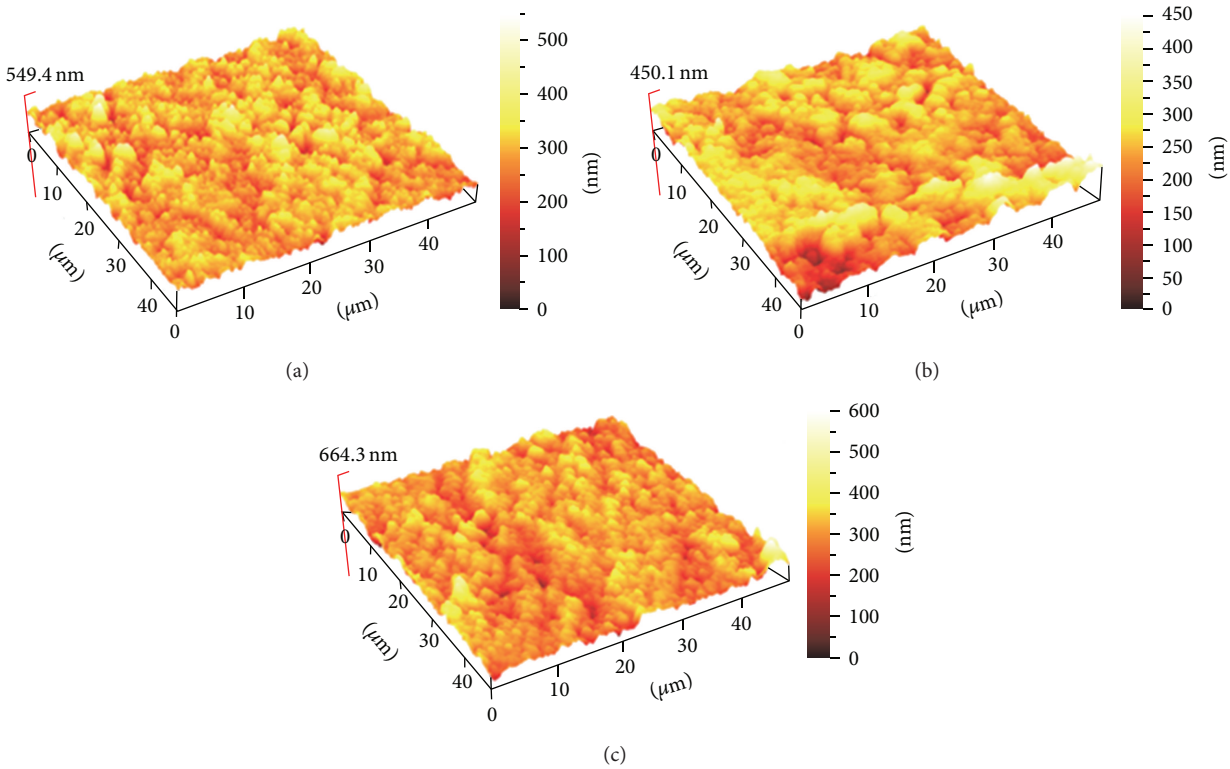

Figure 3: The AFM-3D images of Ketac N100 surface roughness: (a) Ketac N100 without bleaching, (b) Ketac N100 with home bleaching, and (c) Ketac N100 with in-office bleaching.

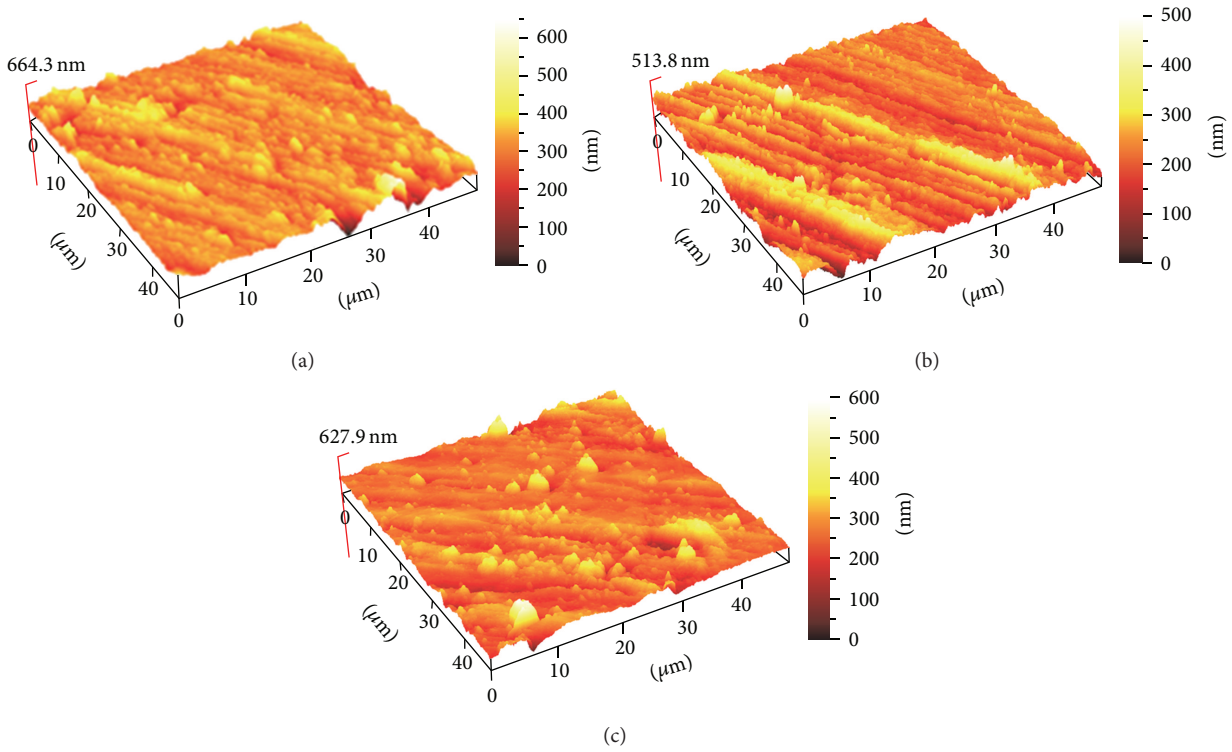

Figure 4: The AFM-3D images of Estelite Σ Quick surface roughness: (a) Estelite Σ without bleaching, (b) Estelite Σ with home bleaching, and (c) Estelite Σ with in-office bleaching.

Table 4: Comparison of ΔE value of the tooth colored restorative materials after bleaching (between the bleaching agents).

Bleaching agent	Restorative material	Mean ± SD	P value
Home bleaching 20% CP	Filtek Z350 XT	2.2 ± 1.0	0.0001*
	Estelite Σ Quick	3.7 ± 1.5	
	Ketac N100	3.1 ± 1.2	
In-office bleaching 40% CP	Filtek Z350 XT	2.6 ± 1.1	0.0001*
	Estelite Σ Quick	3.0 ± 1.2	
	Ketac N100	2.7 ± 1.2	

Mann-Whitney test; * P value < 0.05 is significant; CP: carbamide peroxide.

4. Discussion

Home bleaching and in-office bleaching are popular treatment modalities that are attractive to dentists and patients, as they constitute a simple, safe, and effective procedure to lighten discolored teeth. However, preexisting Classes I, II III, IV, and V tooth colored restorations may be affected by the bleaching gels. Bleaching agents may result in a color change of a restoration that may be perceived by the patient and determined to be unacceptable. If a restorative material has a perfect color match with the surrounding tooth before bleaching, this may no longer be the case after bleaching when the teeth have become lighter and brighter as a result of the CP treatment. Within the limits of this study, it was observed that even low concentrations of bleaching agents had an effect on the color of restorative materials.

Considering the active ingredients available for vital tooth bleaching, carbamide and hydrogen peroxide are the most commonly used agents for different bleaching modalities. Carbamide peroxide degrades into approximately one-third of hydrogen peroxide and two-thirds of urea [19]. The free radicals that are formed eventually combine to form molecular oxygen and water. Some aspects of this chemical process might accelerate the hydrolytic degradation of restorative materials, as described by Söderholm et al. [20]. Chemical softening of the restorative materials might also occur if the bleaching products have a high concentration of hydrogen peroxide [21].

GIC's color change is due to its polyacid content, while the composite color changes are influenced by many factors such as resin shades, the chemical activator, initiator, and inhibitor. The resin component was determined to be the source of discoloration [18].

The A2 shade was chosen for composite materials to minimize the effect of shade variation. Two marketed bleaching systems that differed with respect to peroxide concentration and regimen were compared: Ultradent Opalescence Boost (40% hydrogen peroxide) for in-office bleaching and Ultradent Opalescence PF (20% hydrogen peroxide) for home bleaching. Control specimens were used against which the effects of bleaching were compared.

The color of dental esthetic restorative materials is routinely measured with commercial DSLR cameras and

Table 5: Median roughness number (Ra, nm) and interquartile range of the three tested composite resins after bleaching with home and in-office bleaching agent.

Material	Control $n = 10$ Median (IqR)	20% CP $n = 10$ Median (IqR)	40% CP $n = 10$ Median (IqR)	P value
Filtek Z350XT	73.87 (13.73)	71.73 (10.47)	68.43 (14.25)	0.537
Estelite Σ Quick	77.86 (17.55)	75.26 (11.76)	74.87 (15.84)	0.491
Ketac N100	72.49 (10.31)	72.85 (12.36)	70.22 (13.79)	0.635

Kruskal-Wallis test; P value < 0.05 is significant; IqR: interquartile range; CP: carbamide peroxide.

appropriate calibration protocols. In assessing chromatic differences, CIELAB was used in this study.

The lightening of the specimens was depicted as an increase in L while the actual hue-chroma change was demonstrated in changes in A or B. The amount of discoloration after a given period was represented by the color difference value (ΔE). The accepted change caused by these bleaching preparations produces a ΔE value of 2, which is less than that of visual perception [22]. Thus, the human eye cannot detect a change in color of a material that has undergone a change of less than ΔE of 2 [22]. Therefore, a minimum difference of 2 can be used as criteria for the comparison of color changes in the restorative materials [23]. Wee et al. [9] concluded that perceptible color differences range from a ΔE of 1 and 2 in *in vitro* studies to 3.7 in an *in vivo* study, while acceptable color differences range from a ΔE of 2.72 and 3.3 in *in vitro* studies to 6.8 in an *in vivo* study. In another study, Yalcin and Gurgan [24] reported that bleaching regimens may also cause a change in gloss of restorative materials.

Among the materials tested, Estelite Sigma Quick showed the largest color change ($\Delta E = 3.8$), followed by Ketac N100 ($\Delta E = 3.1$) and Filtek Z-350 XT ($\Delta E = 2.2$). This can be explained by the degradation of metal polyacrylate salts. Color changes of composites may be influenced by the differences in resin shades, the chemical activator, initiator, and inhibitor, polymer, type and quantity of filler, oxidation of C=C double bonds, resin thickness, or storage methods of specimens during observation [24]. Filtek Z-350 showed the least color change followed by Ketac N100. This may be attributed to the amount of nanofiller particles present in the composite resin [7]. Canay and Çehreli [8] have also reported that the change in color is associated with the matrix content, the amount of filler, and the type of filler material.

The size and morphology of filler particles influence the mechanical and physical properties while nanoparticles and clusters in the nanofilled materials improved it [25]. Higher discoloration of the Estelite Sigma Quick may be due to the greater volume of the resin composite matrix when compared with Filtek Z-350 [26]. The bleaching agents may also cause a decline of silica and silicon content, indicating erosion of the resin composite material [27]. In addition, the color changes of composites were also influenced by the differences in curing conditions, background colors for color measuring, color measuring methods, type of color measuring instruments, and observation methods [24].

From the results we also determined that the mean color change of all tested restorative materials was greater for home bleaching than in-office bleaching. This may be due to the longer application time, in spite of the fact that the concentration of hydrogen peroxide is lower for home bleaching agents. According to Meireles et al. [23], lower carbamide peroxide concentrations were more effective in the first week of their study. It appears that total contact time of bleaching gels is more important than the concentration.

Another study showed that higher concentrations of bleaching agents achieve the same postbleaching result as lower concentration. However, the higher concentration accomplished the whitening result more quickly [28]. The results of our study suggest that the final color change does not depend on the concentration of the bleaching agent but rather on the application time.

The AFM method senses any irregularities on the surface of the specimen and in this study no significant differences between the materials were recorded. This is in agreement with findings of Silva et al. [29]. However, our data contradicts Hafez et al. [30] who reported an increased surface roughness of composites resin, which they determined depending on the bleaching agent as well as the type and shade of composite material tested.

Generally, the 3D surface profile that was recorded had a reading of below 0.2 μm or 200 nm. It has been reported that Ra above 0.2 nm results in an increase in plaque accumulation resulting in a higher risk for caries and periodontal disease [31]. According to Chung [32], when Ra was lower than 1 μm, the surfaces were visibly smooth. Therefore, all composites surfaces evaluated after bleaching demonstrated a smooth surface, which from a clinical point of view is favorable as it reduces plaque accumulation.

As was reported here, even low concentrations of bleaching agents had an effect on the color of restorative materials. Patients should be informed that existing restorations may not match their natural teeth after bleaching and replacement may be required for esthetic reasons. However, this has to be further investigated with *in vitro* studies evaluating the effects of saliva as well as controlled clinical trials.

This study evaluated the effect of home versus in-office bleaching systems on the color stability (CIELAB) and 3D surface profile (AFM) of nanofilled tooth colored restorative materials. This combination study was the advanced and different method to previous bleaching studies.

Limitations of Study. The study was carried out to compare color stability and surface roughness of tooth colored filling material *in vitro*. Oral simulating condition cannot be achieved, especially saliva.

5. Conclusion

Submicron filled resin composites showed the highest color changes followed by Ketac nanoionomer after bleaching. The nanofilled composite was found to be highly stable in terms of color. Nanofilled composite and a glass ionomer showed better color stability compared to a microfilled tooth colored material.

Based on the result of this study, it can be concluded that 20% CP home bleaching and 40% CP in-office bleaching agents did not cause changes in surface roughness of the three tested composites. The AFM evaluation of surface roughness observed in the 3D images proved to be an effective technique. Nanohybrid resin composite, microhybrid composite resins, and nanoionomer bleached with 20 or 40% CP bleaching agents resulted in the same 3D surface profile.

Clinical Significance in Dentistry. Dental practitioner should make sure that their patients with dental restorations (especially those with polymer content) are aware of the changes that may occur during whitening, as well as the possibility that their bleached restorations may need to be polished or replaced at the end of the treatment.

As was reported here, even low concentrations of bleaching agents had an effect on the color of restorative materials. Patients should be informed that existing restorations may not match their natural teeth after bleaching and replacement may be required for esthetic reasons.

For dental society, this result will give information to the dental practitioner regarding the effect of home and in-office bleaching to the new available tooth colored filling materials. This information is important for dental practitioner to make decision on material to be chosen in tooth whitening treatment, for the benefit of patients.

Recommendations. Further clinical study should be conducted on color stability of tooth colored filling material after tooth whitening evaluating the effects of saliva and other oral environments. Controlled clinical trials are necessary to determine any clinical implication.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Authors' Contribution

(1) Bryant Anthony Irawan, Stacey Natalie Irawan, Sam'an Malik Masudi, Ninin Sukminingrum, and Mohammad Khursheed Alam contributed to conception and design of the work. (2) Bryant Anthony Irawan, Stacey Natalie Irawan, and Sam'an Malik Masudi contributed to acquisition of data or analysis and interpretation of data. (3) Drafting the paper or revising it critically for important intellectual content was performed by Bryant Anthony Irawan, Stacey Natalie Irawan, Sam'an Malik Masudi, Ninin Sukminingrum, and Mohammad Khursheed Alam. (4) Bryant Anthony Irawan, Stacey Natalie Irawan, Sam'an Malik Masudi, Ninin Sukminingrum, and Mohammad Khursheed Alam contributed to final approval of the version to be published.

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Endnotes

1. 3M ESPE, Minneapolis, MN, USA
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7. Ambios Technology, Santa Cruz, CA, USA.

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Review Article

Cementation of Glass-Ceramic Posterior Restorations: A Systematic Review

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Aim. The aim of this comprehensive review is to systematically organize the current knowledge regarding the cementation of glass-ceramic materials and restorations, with an additional focus on the benefits of Immediate Dentin Sealing (IDS). *Materials and Methods*. An extensive literature search concerning the cementation of single-unit glass-ceramic posterior restorations was conducted in the databases of MEDLINE (Pubmed), CENTRAL (Cochrane Central Register of Controlled Trials), and EMBASE. To be considered for inclusion, *in vitro* and *in vivo* studies should compare different cementation regimes involving a "glass-ceramic/cement/human tooth" complex. *Results and Conclusions*. 88 studies were included in total. The *in vitro* data were organized according to the following topics: (micro)shear and (micro)tensile bond strength, fracture strength, and marginal gap and integrity. For *in vivo* studies survival and quality of survival were considered. *In vitro* studies showed that adhesive systems (3-step, etch-and-rinse) result in the best (micro)shear bond strength values compared to self-adhesive and self-etch systems when luting glass-ceramic substrates to human dentin. The highest fracture strength is obtained with adhesive cements in particular. No marked clinical preference for one specific procedure could be demonstrated on the basis of the reviewed literature. The possible merits of IDS are most convincingly illustrated by the favorable microtensile bond strengths. No clinical studies regarding IDS were found.

1. Introduction

Bonded glass-ceramic restorations have gained popularity, particularly after new materials, bonding systems, cements, and cementation techniques became available in recent years. Nowadays different ceramics are introduced for the use of posterior restorations, being either an oxide-ceramic or a glass-ceramic. Glass-ceramics are of special interest in this review because their silica content and micromechanical interlocking structure allow adhesive cementation to enamel and dentin. Consequently, glass-ceramic restorations can withstand tensile forces without cement failure, even if the preparation of the tooth is nonretentive. Since the surface treatment of feldspathic porcelain in 1983 [1] became available, new materials have evolved into high strength and esthetic glass-ceramics such as lithium disilicate. This higher strength compared to earlier glass-ceramics is reached because of a different firing process [2]. Contemporary glass-ceramic fixed dental crowns possess good optical and mechanical properties, thus mimicking natural teeth to a large extent [3–5].

To ensure proper attachment of an indirect restoration, basically two aspects have to be taken into consideration: conditioning of the ceramic material and conditioning of the tooth substrate followed by cementation. The most commonly used conditioning method for the glass-ceramic surface these days is application of hydrofluoric acid and silanisation, as reviewed by Tian et al. [6]. Cements are considered necessary to obtain durable retention of the restoration and good marginal seal, as well as maintaining original

color and marginal outline. The first dental luting agents were water based cements like zinc phosphate and glass ionomer cements. With the introduction of resin cements, properties like solubility and adhesion improved, thereby allowing a minimally invasive preparation design [7]. Contemporary resin cements vary in properties like viscosity, whether or not they need light curing, and whether they are adhesive, self-etching, or self-adhesive. However these cements require some kind of conditioning procedure of the tooth substrate and indirect restoration.

In addition, sealing of dentin tubules with a filled adhesive resin directly after tooth preparation and prior to (digital or analogue) impression taking is presumed to result in improved bond strength, less gap formation, decreased bacterial leakage, and reduced dentin sensitivity [8]. This procedure may be highly clinically relevant and was first tested *in vitro* by Pashley et al. [9] and described in 1996 as the dual application of dentin bonding agents [10]. Later Magne referred to it as "Immediate Dentin Sealing" (IDS) [8].

Compared to luting with water based cements, adhesive cementation is more difficult and time-consuming and moisture control is more important. A clinical study showed a tendency to higher fracture rates among posterior crowns compared to anterior crowns, and indirect bonded restorations in molars revealed higher failure rates than premolar crowns [11]. Hence cementation of glass-ceramics in the posterior region appears clinically the most challenging and thus is of clinical relevance for further investigation. There is little homogeneity between studies in terms of materials, test method, and analysis. For *in vitro* studies four types of testing are predominantly applied: (micro)shear bond strength, (micro)tensile bond strength, fracture strength, and marginal gap. The outcomes of these studies are of importance as this could predict the long term results of indirect restorations.

A shear bond strength test evaluates the degree to which two attached specimens resist shear. A true shear test is difficult to perform because one of the specimens is always fixed to the test device. Instead, a microshear bond strength test is preferable, in which a cross-sectional area of 1 mm^2 is generally used for greater uniformity of stress distribution. This test results in more adhesive failures at the bonding interface instead of cohesive failures in the substrate, which is considered to be more realistic [6].

A tensile bond strength test is performed perpendicular to the bonded interface and is therefore generally adopted as the most valid bond strength test at this moment [12]. However it is hard to control the alignment of specimen, and nonuniform stress distribution across the bonding surface occurs. With a microtensile test the small size of the specimen leads to a more favorable stress distribution and to bond failures that lie closer to their ultimate strengths [13].

Fracture loading, fracture resistance, load-to-failure, breaking strength, and fracture strength are considered synonymous terms. They are used to indicate the stress at which a specimen fails by occlusal loading, and, in the following, the term "fracture strength" will be adopted. In general, restored teeth are progressively, occlusally loaded until fracture by means of a stainless steel ball. Fracture strength and fracture type are the most common outcome parameters.

The marginal gap reflects the quality of marginal adaptation and is commonly studied by means of microleakage experiments (e.g., with dye penetration or silver staining and/or by scanning electron microscopy (SEM)), either with or without thermocycling and with or without loading in a chewing simulator. With conventional nonadhesive restorations the size of the marginal gap is considered of paramount importance for the (quality of) survival of the restoration and should be as small as possible. The size of the marginal gap may not be as critical when using materials that can be luted adhesively to the tooth substrate, such as glass-ceramics.

There appears to be a plethora of materials, cements, bonding systems, and cementation techniques for luting glass-ceramics to posterior teeth. The aim of this systematic review is to focus on cements and organize the current knowledge and the manner in which cements are used for the cementation of glass-ceramic materials and restorations, with an additional focus on the benefits of IDS.

2. Materials and Methods

2.1. Search Strategy. A comprehensive literature search was undertaken in the databases of MEDLINE (1950–1 January 2015) (Pubmed), CENTRAL (1800–1 January 2015) (Cochrane Central Register of Controlled Trials), and EMBASE (1966–1 January 2015) by means of a combination of MeSH terms and text words. The English language restriction was applied and articles without an available abstract were not considered. The search strategy is outlined as follows.

Search Strategy

MEDLINE. ((“Ceramics”[Mesh] OR ceramic*[tw]) AND (“Cementation”[Mesh] OR “Dental Cements”[Mesh] OR cementation*[tw] OR immediate dentin seal*[tw] OR luting[tw] OR lute[tw] OR dental adhesives[tw] OR resin coat*[tw])) NOT (veneer*[TI] OR posts*[TI] OR implant*[TI] OR zirconi*[TI] OR alumina[TI] OR “zirconium oxide”[Supplementary Concept]) NOT (“Case Reports”[Publication Type] OR “Review”[Publication type]) AND English[lang].

Run data search: January 1, 2015 (1868 results).

EMBASE. “dental ceramics”/exp OR ceramic*:ab,ti AND (“cementation”/exp OR “tooth cement”/exp OR cementation*:ab,ti OR “immediate dentin sealing”:ab,ti OR luting:ab,ti OR lute:ab,ti OR “dental adhesives”:ab,ti OR “resin coating”:ab,ti).

NOT (veneer*:ti OR posts*:ti OR implant*:ti OR zirconi*:ti OR alumin*:ti) NOT (“case report”/exp OR “review”/exp) AND[english]/lim.

Run data search: January 1, 2015 (806 results).

COCHRANE Library (Trials) (Search in ti,ab,kw). ceramic* AND (cement* OR immediate dentin seal* OR luting OR lute OR dental adhesive* OR resin coat*).

Run data search: January 1, 2015 (332 results).

2.2. Study Selection. Titles and abstracts of the identified publications were screened by one of the authors. Full text

Table 1: Assessment of risk of bias of included *in vitro* ((micro)shear bond strength) studies ($n = 17$) according to Cochrane collaboration's tool.

Adequate sequence generation?	Allocation concealment?	Blinding?	Incomplete outcome data addressed?	Free of selective reporting?	Free of other bias?	References
Unclear	NA	NA	Unclear	Yes	Yes	[22]
Unclear	NA	NA	No	Yes	Yes	[16]
Unclear	NA	NA	Yes	Yes	Yes	[27]
Unclear	NA	NA	Yes	No	Yes	[25]
Unclear	NA	NA	Yes	Yes	Yes	[26]
Unclear	NA	NA	Unclear	Yes	Yes	[29]
Unclear	NA	NA	Unclear	Yes	Yes	[28]
Unclear	NA	NA	Unclear	Yes	Yes	[23]
Unclear	NA	NA	Unclear	Yes	Yes	[30]
Unclear	NA	NA	Unclear	Yes	Yes	[31]
Unclear	NA	NA	Yes	Yes	Yes	[24]
Unclear	NA	NA	No	Yes	Yes	[21]
Unclear	NA	NA	Unclear	Yes	Yes	[20]
Unclear	NA	NA	Unclear	Yes	Yes	[15]
No	NA	NA	Yes	Yes	Yes	[19]
Unclear	NA	NA	Yes	Yes	Yes	[17]
Unclear	NA	NA	Yes	Yes	Yes	[18]

Table 2: Assessment of risk of bias of included *in vitro* ((micro)tensile bond strength) studies ($n = 14$) according to Cochrane collaboration's tool.

Adequate sequence generation?	Allocation concealment?	Blinding?	Incomplete outcome data addressed?	Free of selective reporting?	Free of other bias?	References
Unclear	NA	NA	Unclear	Yes	Yes	[34]
Unclear	NA	NA	Yes	Yes	No	[37]
Unclear	NA	NA	Yes	Yes	Yes	[33]
Unclear	NA	NA	Unclear	Yes	Yes	[44]
Unclear	NA	NA	Yes	Yes	Yes	[42]
Unclear	NA	NA	Unclear	Yes	Yes	[40]
Unclear	NA	NA	Unclear	Yes	Yes	[32]
Unclear	NA	NA	Yes	Yes	Yes	[35]
Unclear	NA	NA	Unclear	Yes	Yes	[45]
Unclear	NA	NA	Unclear	Yes	Yes	[43]
Unclear	NA	NA	Yes	Yes	Yes	[38]
Unclear	NA	NA	Yes	Yes	Yes	[39]
Unclear	NA	NA	Yes	Yes	Yes	[36]
Unclear	NA	NA	Unclear	No	Yes	[41]

documents were obtained for all articles meeting the inclusion criteria. Additional hand searching was performed by following up on the reference lists from included articles. Full text analysis to decide on inclusion/exclusion was subsequently performed by two reviewers and Cohen's Kappa was used as the measure of agreement. Disagreements were resolved by manner of discussion.

Methodological quality regarding the risk of bias in selected articles was assessed by one of the authors according to the criteria as set by the Cochrane Collaboration (Tables 1, 2, 3, 4, and 5). In case of multiple clinical studies in which the same restorations were analyzed at different time intervals, leading to different publications, the study with the longest follow-up was selected for definitive analysis.

2.3. Inclusion Criteria. Only articles about glass-ceramic materials were considered. Clinically, the focus was on single-unit posterior restorations. Included studies should compare different cementation regimes and involve a "glass-ceramic/cement/human tooth" complex. Studies regarding the benefits of IDS attracted special attention. Descriptive studies (e.g., technical notes), systematic reviews, case

Table 3: Assessment of risk of bias of included *in vitro* (fracture strength) studies ($n = 11$) according to Cochrane collaboration's tool.

Adequate sequence generation?	Allocation concealment?	Blinding?	Incomplete outcome data addressed?	Free of selective reporting?	Free of other bias?	References
Unclear	NA	NA	Unclear	No	Yes	[52]
Unclear	NA	NA	Unclear	Yes	Yes	[49]
Unclear	NA	NA	Unclear	Yes	Yes	[47]
No	NA	NA	Unclear	No	Yes	[48]
No	NA	NA	Unclear	No	Yes	[54]
Unclear	NA	NA	Unclear	Yes	Yes	[55]
Unclear	NA	NA	No	Yes	No	[59]
Unclear	NA	NA	Unclear	No	No	[53]
Unclear	NA	NA	Unclear	Yes	Yes	[58]
Unclear	NA	NA	Unclear	Yes	Yes	[56]
Unclear	NA	NA	Unclear	Yes	Yes	[60]

Table 4: Assessment of risk of bias of included *in vitro* (marginal gap) studies ($n = 26$) according to Cochrane collaboration's tool.

Adequate sequence generation?	Allocation concealment?	Blinding?	Incomplete outcome data addressed?	Free of selective reporting?	Free of other bias?	References
No	NA	NA	Unclear	Yes	Yes	[72]
Unclear	NA	NA	Unclear	Yes	Yes	[76]
Unclear	NA	NA	Unclear	No	Yes	[50]
Unclear	NA	NA	Unclear	Yes	Yes	[79]
Unclear	NA	NA	Unclear	Yes	Yes	[74]
Unclear	NA	NA	Unclear	Yes	Yes	[73]
Unclear	NA	NA	Yes	Yes	Yes	[71]
Unclear	NA	NA	Unclear	Yes	Yes	[63]
Unclear	NA	NA	Yes	Yes	Yes	[78]
Unclear	NA	NA	Unclear	No	Yes	[77]
Unclear	NA	NA	No	Yes	Yes	[70]
Unclear	NA	NA	Unclear	No	Yes	[62]
Unclear	NA	NA	Unclear	Yes	Yes	[66]
Unclear	NA	NA	Unclear	No	Yes	[67]
Unclear	NA	NA	Unclear	Yes	Yes	[80]
Unclear	NA	NA	Yes	Unclear	Yes	[75]
Unclear	NA	NA	Yes	Unclear	Yes	[57]
Unclear	NA	NA	Yes	Yes	Yes	[82]
Unclear	NA	NA	No	Yes	Yes	[46]
Unclear	NA	NA	Unclear	No	Yes	[65]
Unclear	NA	NA	Yes	No	Yes	[61]
Unclear	NA	NA	Unclear	Yes	Yes	[51]
Unclear	NA	NA	Unclear	No	Yes	[64]
Unclear	NA	NA	Unclear	Yes	Yes	[81]
Unclear	NA	NA	Yes	No	Yes	[68]
Unclear	NA	NA	Yes	Yes	Yes	[69]

reports, or studies with less than ten patients were excluded (Figure 1). Descriptions such as "selective double-bond technique," "resin coating technique," or "adhesive resin liner" were considered synonymous for IDS.

2.4. Data Extraction. The included studies were divided into *in vitro* and *in vivo* studies. For *in vitro* studies the data were organized according to the following topics: (micro)shear and (micro)tensile bond strength, fracture strength, and finally

Table 5: Assessment of risk of bias of included *in vivo* studies ($n = 20$) according to Cochrane collaboration's tool.

Adequate sequence generation?	Allocation concealment?	Blinding?	Incomplete outcome data addressed?	Free of selective reporting?	Free of other bias?	References
Unclear	Unclear	Unclear	Yes	Yes	Yes	[83]
Unclear	Unclear	Unclear	Yes	No	Yes	[99]
No	Unclear	Unclear	No	No	No	[94]
Unclear	Unclear	Unclear	Yes	Yes	Yes	[93]
Unclear	Unclear	Unclear	Yes	No	Yes	[101]
Unclear	Unclear	Unclear	Yes	Yes	Yes	[91]
Unclear	Unclear	Unclear	Yes	No	No	[87]
Unclear	Unclear	Unclear	Yes	No	No	[89]
Unclear	Unclear	Unclear	Yes	Yes	Yes	[97]
Unclear	Unclear	Unclear	Yes	Yes	Yes	[98]
Unclear	Unclear	Unclear	Yes	Yes	Yes	[92]
Unclear	Unclear	Unclear	Yes	Yes	Yes	[84]
Unclear	Unclear	Unclear	Yes	No	Yes	[88]
Unclear	Unclear	Unclear	Yes	No	Yes	[102]
Yes	Unclear	Yes	Yes	Yes	Yes	[100]
Unclear	Unclear	Unclear	Unclear	No	No	[95]
Unclear	Unclear	Unclear	Yes	No	Yes	[96]
Unclear	Unclear	Unclear	Yes	Yes	No	[86]
Unclear	Yes	Unclear	Yes	Yes	Yes	[85]
Unclear	Yes	Unclear	Yes	Yes	Yes	[90]

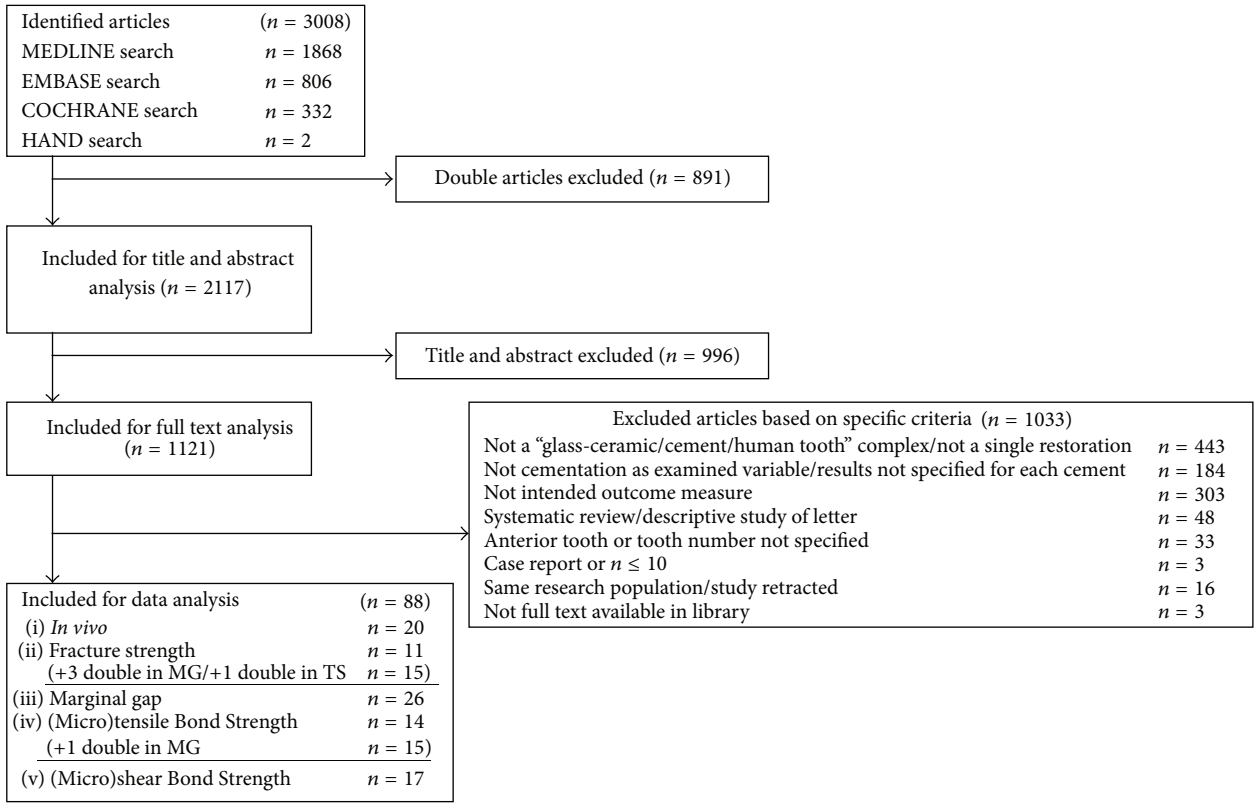

Figure 1: Algorithm of study selection procedure.

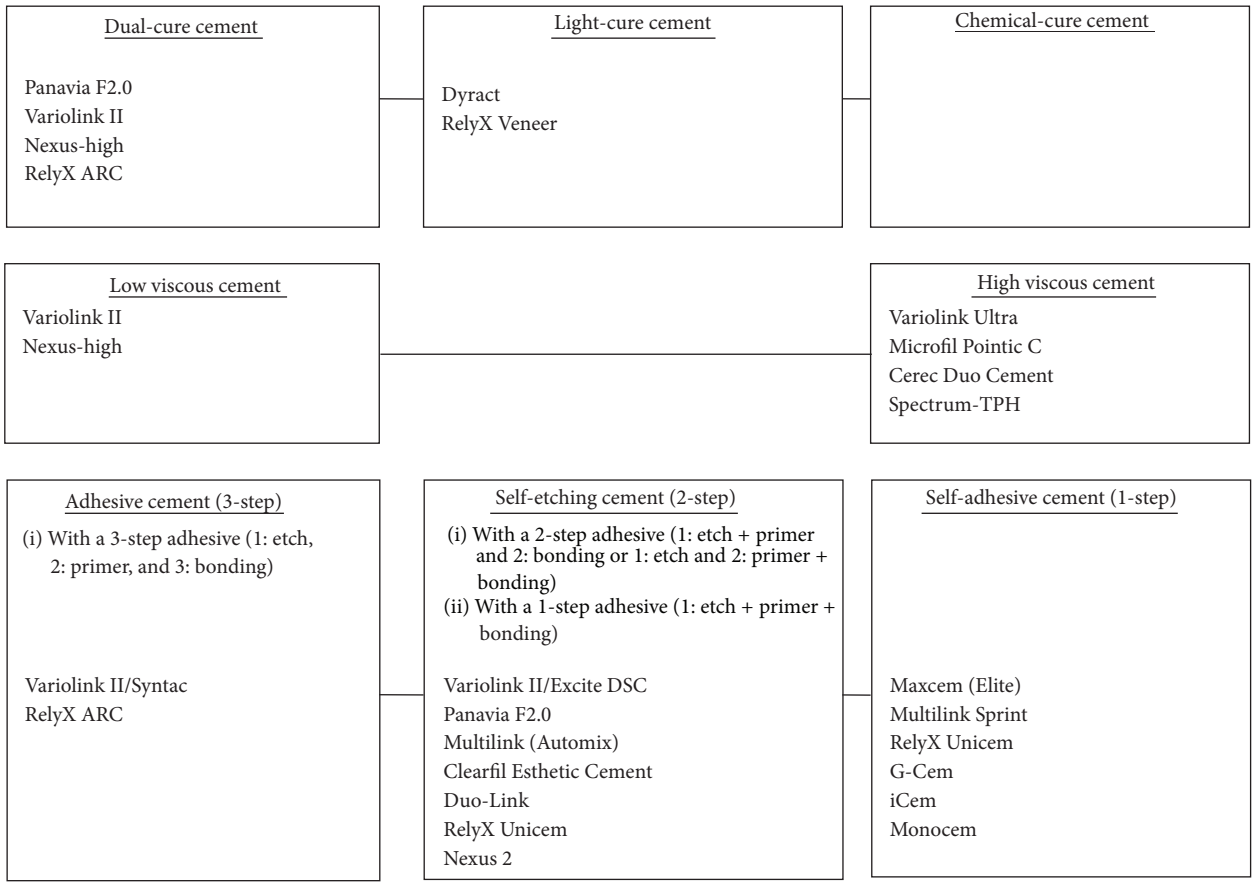

Figure 2: Choices in commonly used resin composite cements.

marginal gap and integrity. For *in vivo* studies survival and quality of survival were considered.

3. Results

The searches of MEDLINE (Pubmed), CENTRAL (Cochrane Central Register of Controlled Trials), and EMBASE resulted in 3008 publications. After exclusion of double publications, 2117 publications remained for title and abstract analysis. 1121 articles were hereafter included for full text analysis. Only a limited additional number of publications were found after checking the references of the included studies. Application of specified exclusion criteria resulted in 88 publications that could be included in the review. The exclusion criteria are described in Figure 1.

Interobserver agreement (Cohen's Kappa) regarding final inclusion or exclusion of studies that were proposed after full text analysis was 0.80 (IBM SPSS 22), which is generally considered to be a strong level of agreement [14]. Initial disagreements were generally caused by ambiguities in the study design or the characterization of materials used.

The included studies were assessed for their risk of bias according to the Cochrane library (Tables 1, 2, 3, 4, and 5). Assessment of allocation concealment and blinding of participants, personnel, and outcome assessors for included *in vitro* studies proved difficult and hardly ever applicable. Sequence generation and incomplete outcome data for *in vitro* studies are not explained in most cases but just named. Assessment "unclear" on incomplete outcome data generally implies that no missing data were reported. Most studies in this review did not report sequence generation; for *in vitro* studies the relevance of this can be subject of debate. For *in vivo studies* sequence generation, allocation concealment, and blinding were often assessed as "unclear," because studies often did not describe these procedures. Overall the included studies had a low risk of bias. More specifically, a low risk of bias was assessed for shear bond strength studies, tensile strength studies, and marginal gap studies. An unclear risk of bias was assessed for fracture strength studies and *in vivo* studies.

Because of their great variety it is important to divide contemporary resin cements into subgroups regarding their curing type, their viscosity, and whether they are either adhesive (with a 3-step adhesive), self-etching (with a 2-step or 1-step adhesive), or self-adhesive. This terminology is not used consistently in literature. An overview is presented in Figure 2. Cements that are named in this study will be specified as one of these three types, which usually depends on the adhesive used. Cement and adhesive system brand names, manufacturers, city, and countries of origin are presented in Table 6. Generally, different cement brands, cement types, or cementation techniques were compared in the included studies (e.g., water based cements among which are zinc

Table 6: Cement and adhesive system brand names, manufacturers, city, and countries of origin.

Cement and adhesive system brand names	Manufacturers	City	Countries of origin
Adapter SingleBond 2	3M ESPE	Seefeld	Germany
All-bond 2	Bisco Inc.	Schaumburg, IL	USA
Authentic	Ceranay	Stuttgart	Germany
Aquacem	Dentsply deTrey	Konstanz	Germany
Biomer	Dentsply Caulk	Milford, DE	USA
Cavex Clearfil F2	Cavex	Norden	Germany
Cergo	DeguDent	Hanau	Germany
Cergogold	DeguDent	Hanau	Germany
Chemiace II	Sun Medical	Moriyama City	Japan
Clearfil Esthetic Cement	Kuraray	Tokyo	Japan
Clearfil Protect Bond	Kuraray	Tokyo	Japan
Clearfil SA	Kuraray	Tokyo	Japan
DeTrey Zinc	Dentsply deTrey	Konstanz	Germany
Definite Multibond primer	DeguDent	Hanau	Germany
Definite cement	DeguDent	Hanau	Germany
Dicor cement	Dentsply	York, PA	USA
Dicor LAC	Dentsply deTrey	Konstanz	Germany
Ducere LFC	Ducere	Rosbach	Germany
Duo-Link	Bisco Inc.	Schaumburg, IL	USA
Dycal	Dentsply Caulk	Milford, DE	USA
Dyract-Cem	Dentsply DeTrey	Konstanz	Germany
ED primer II	Kuraray	Tokyo	Japan
Enforce	Dentsply	São Paulo	Brazil
Excite (DSC)	Ivoclar Vivadent	Schaan	Liechtenstein
Finesse	Dentsply Ceramco	Burlington, NJ	USA
Fleck's	Mizzy Inc.	Cherry Hill	USA
Fuji I	GC Corp.	Tokyo	Japan
Fuji Plus (F)	GC Corp.	Tokyo	Japan
G-Cem	GC Corp.	Tokyo	Japan
Geristore	Dent-Mat	Santa Maria	USA
GC Fuji Cem	GC Corp.	Tokyo	Japan
Go!	3M ESPE	Seefeld	Germany
Harvard	Richter-Hoffman	Berlin	Germany
Harvard cement	Harvard Dental	Berlin	Germany
iCem	Heraeus Kulzer	Hanau	Germany
Illusion Universal Cementation System	Bisco Dental Products	Richmond, BC	Canada
IPS E.max Press	Ivoclar Vivadent	Schaan	Liechtenstein
IPS Empress (I) (II)	Ivoclar Vivadent	Schaan	Liechtenstein
Ketac-Cem	3M ESPE	St. Paul, MN	USA
Linerbond 2V	Kuraray	Osaka	Japan
Metabond	Sun Medical	Moriyama City	Japan
Maxcem	Kerr-Hawe	Orange, CA	USA
Microfil Pontic C	Heraeus Kulzer	Hanau	Germany
Mirage	Chameleon Dental	Kansas City, KA	USA
Mirage ABC	Chameleon Dental	Kansas City, KA	USA
Mirage FLC	Chameleon Dental	Kansas City, KA	USA
Multilink (Automix)	Ivoclar Vivadent	Schaan	Liechtenstein
Multilink primer	Ivoclar Vivadent	Schaan	Liechtenstein
Multilink Sprint	Ivoclar Vivadent	Schaan	Liechtenstein

Table 6: Continued.

Cement and adhesive system brand names	Manufacturers	City	Countries of origin
Nexus	Kerr Corp.	Orange, CA	USA
Nexus 2	Kerr Corp.	Orange, CA	USA
Nexus 3	Kerr Corp.	Orange, CA	USA
Nexus-high	Kerr Corp.	Orange, CA	USA
Noritake Super porcelain	Noritake Dental Supply Co., Ltd.	Nagoya	Japan
One Coat Bond	Coltene/Whaledent AG	Altstätten	Switzerland
Optibond FL	Kerr Corporation	Orange	United States
Panavia 21	Kuraray	Osaka	Japan
Panavia F2.0	Kuraray	Osaka	Japan
Panavia F	Kuraray	Osaka	Japan
Protect Liner F	Kuraray	Osaka	Japan
Prodigy	Kerr Corp.	Orange, CA	USA
RelyX ARC	3M ESPE	St. Paul, MN	USA
RelyX Veneer	3M ESPE	St. Paul, MN	USA
RelyX Unicem (Clicker)	3M ESPE	St. Paul, MN	USA
Single Bond	3M ESPE	Seefeld	Germany
Self-etching primer A+B	Ivoclar Vivadent	Schaan	Liechtenstein
SmartCEem 2	Dentsply Caulk	Milford, DE	USA
Spectrum-TPH	Dentsply Caulk	PA	USA
SpeedCEM	Ivoclar Vivadent AG	Schaan	Liechtenstein
Super-Bond C&B	Sun Medical	Moriyama City	Japan
Super porcelain EX-3	Noritake Kizai Co.	Nagoya	Japan
Syntac (classic)	Ivoclar Vivadent	Schaan	Liechtenstein
Temp Bond	Kerr	Corporation, Orange	United States
Tetric flow	Ivoclar Vivadent	Schaan	Liechtenstein
Universal glass ionomer	Super Dent	Westbury, NY	USA
Variolink II	Ivoclar Vivadent	Schaan	Liechtenstein
Variolink II base	Ivoclar Vivadent	Schaan	Liechtenstein
Variolink II refill	Ivoclar Vivadent	Schaan	Liechtenstein
Variolink II Ultra	Ivoclar Vivadent	Schaan	Liechtenstein
Vitadur Alpha	Vita	Bad Säckingen	Germany
Vita Cerec Duo Cement	Coltene/Whaledent AG	Altstätten	Switzerland
Vita Mark II	Vita	Bad Säckingen	Germany

phosphate (Harvard); polycarboxylate cement (Harvard); glass ionomer (Fuji I; Ketac-Cem; Dyract-Cem) and resin cements (Panavia 2; RelyX Unicem; Multilink; Maxcem; G-Cem; Prodigy; Nexus; Vita Cerec Duo Cement and Clearfil Esthetic cement)) in combination with several brands of glass-ceramic restorations. An overview of contemporary resin cements is presented in Figure 2.

3.1. In Vitro Studies

3.1.1. (Micro)shear Bond Strength (n = 17 Studies). Seventeen studies could be identified that met the inclusion criteria; their risk of bias is overviewed in Table 1.

In only one study different groups of luting agents were used and the authors concluded that zinc phosphate cement and glass ionomer cements produced the lowest shear bond strengths, whereas the highest shear bond strengths were found with two self-etching cements (Panavia F2.0 and Multilink) and one self-adhesive resin cement (RelyX Unicem) [15].

Several studies ($n = 7$, [16–22]) compared different resin cements in a shear bond strength test. Adhesive cements produced significantly higher shear bond strength values to dentin [16, 17]. When comparing self-adhesive cements with self-etching cements, the self-etching cements showed the highest bond strengths to dentin [18]. To enamel a self-etching cement (Variolink II/Excite DSC) produced better results compared to another self-etching cement (Clearfil Esthetic cement/ED primer II) [19]. When different self-etch resin cements were compared, Duo-Link showed the highest bond strength, followed by Variolink II (with Excite DSC), and Nexus 2 showed the lowest [20]. To dentin and enamel the adhesive cement Variolink II and the self-etch cement Panavia F2.0 showed the highest shear bond strengths, with Variolink II reaching the highest values [21]. In another study

a similar conclusion was reached, but with no difference between Panavia F2.0 and Variolink II [22].

Others, using a push-out test, concluded that an adhesive cement (Variolink II/Syntac) did not perform better than three self-adhesive cements [23].

To enamel three different self-etching resin cements with different setting modes (dual-cure, light-cure, and flow) were compared in a microshear bond strength test; no significant differences were seen [24].

Four studies [25–28] focused specifically on the presumed benefits of IDS compared to Delayed Dentin Sealing (DDS). In two studies different dentin adhesives acted as an IDS and the authors concluded that they did not alter the retentive strength of adhesively luted ceramic restorations using either of the tested bonding systems [25, 26]. Two other studies concluded that IDS using Clearfil SE Bond resulted in improved shear bond strength compared to DDS [27, 28].

The application of fluoride or triclosan based desensitizing agents prior to adhesive cementation did not influence the shear bond strength [29], nor did laser-etching of the dentin compared to a self-etch (Clearfil Esthetic) and an etch-and-rinse cementation procedure (Variolink II) [30]. Application of a silane coupling agent to the ceramic surface after etching with hydrofluoric acid increases the shear bond strength [31].

In summary, some evidence supports the use of adhesive cement with respect to the shear bond strength compared to self-adhesive and self-etch systems when luting all ceramic materials to human dentin. There is little evidence to support the assumption that IDS improves the shear bond strength especially when Clearfil SE Bond was used.

3.1.2. (Micro)tensile Bond Strength (n = 15 Studies). Fifteen articles could be included investigating the effect of different cements on glass-ceramic restorative materials with a (micro)tensile bond strength test; their risk of bias is overviewed in Table 2.

When comparing different cement groups, glass ionomer cement (Aquacem) yielded far lower tensile bonding strengths (2-3 times) compared to a self-etch resin cement (Dicor LAC) [32].

In studies comparing different resin cements results were opposite or similar about which cement, self-etching or self-adhesive, resulted in the highest tensile bond strength [33–35] or obtained similar results for each cement, be it adhesive, self-etching, or self-adhesive [36]. Values were still worse than those obtained using adhesive luting agents [37] (personal communication) and [38]. But in another study this was contradicted because the self-etching cement did better than the adhesive cement [39]. When a less commonly used self-etching adhesive system (Super-Bond C&B) was used, a higher tensile bond strength was obtained compared to two other self-etching cements [40].

It was hypothesized that the tensile bonding strength is not so much dependent on the type of adhesive approach but more so on the chemical composition and viscosity of the cement used. Interestingly, the use of self-etch adhesive combined with a restorative composite (Clearfil SE Bond with Clearfil APX) yielded higher tensile bond stresses to dentin than dedicated self-adhesive, self-etch, and adhesive cements [39]. But no such difference was found when the same material (Clearfil APX) was used with another bonding system (Linerbond 2V) [41].

Overall, autocure leads to a lower microtensile bond strength when compared to dual-cure cement modes [42, 43]. Precuring of the adhesive layer increased tensile bond strengths [43].

As before, tensile bond strengths were also higher for enamel than for dentin, that is, in a study by Habekost et al. [44].

The effect of IDS on microtensile bond strength was tested in two studies. An IDS layer (one or two resin coatings) applied directly after preparation yielded higher values compared to applying it just prior to cementation or not at all. No temporary restorations were made [45, 46].

In summary, no one particular cement or adhesive system, be it self-etching, self-adhesive, or adhesive, showed overall superior results with respect to (micro)tensile bond strength. IDS improved microtensile bond strength in both included studies.

3.1.3. Fracture Strength (n = 15 Studies). Fifteen studies could be identified that met the inclusion criteria; their risk of bias is overviewed in Table 3. Seven studies [47–53] examined the effect of different cement groups like zinc phosphate, glass ionomer, or resin cements. Regardless of the preparation type, specimens with crowns that were adhesively cemented were stronger upon occlusal loading than those with conventionally cemented crowns [47]. Several other researchers came to a similar conclusion: zinc phosphate cements were associated with the lowest fracture loads [48] and adhesive cements increased fracture load significantly compared to glass ionomer and zinc phosphate cement [49, 50]. When comparing two self-adhesive cements with an adhesive cement and a glass ionomer cement, the self-adhesive cement (RelyX Unicem) revealed the highest fracture strength [51]. In one study the authors concluded that the cement type had no statistical significant effect on fracture resistance within the ceramic system [52] and in another study there were no differences found in fracture strength between glass ionomer, zinc phosphate, and composite resin cements [53].

Seven studies [44, 54–59] were included that examined the performance of different resins cements. Different variations of dentin bonding agents and resin luting materials were tested ((1) Mirage ABC and Mirage FLC; (2) Metabond; (3) All-bond 2 and Duo-Link; (4) Scotchbond multipurpose and 3M indirect porcelain bonding kit; (5) Mirage ABC and 3M indirect porcelain bonding kit). Mirage porcelain crowns were luted to premolars. The last two groups produced higher fracture strengths than the other three, suggesting that 3M indirect bonding kit was of significant influence [54]. In a study comparing two different dual-cure resin cements, it was unclear which adhesive system was used for each cement so the cements cannot be considered adhesive, self-etching, or self-adhesive. The authors hypothesize that cements with a higher flexural modulus exhibit higher values of fracture resistance for the ceramic/tooth assembly [55]. Others also suggest that the modulus of elasticity or the preparation design may be of larger influence than the adhesiveness of

resin cements [44, 56]. In one study the authors concluded that the cement type had a significant effect on fatigue resistance in favor of the self-etching Panavia F2.0 [57], but other authors concluded Panavia F did the poorest, compared to other dual-cured resin cements [58]. When comparing a dual-cure cement (RelyX ARC) with a light-cure cement (RelyX Veneer), no significant differences in loads at failure among the tested cement group [59] were seen.

One study described the effect of the thickness of IDS materials (Clearfil SE Bond and Protect Liner F) on the fracture strength of IPS Empress II crowns cemented with Panavia F. The film thickness formed by Clearfil SE Bond and Protect Liner F increased the fracture load of IPS Empress II crowns [60].

In summary, teeth that are restored with an indirect glass-ceramic restoration, with respect to *in vitro* fracture strength of posterior adhesively cemented specimen, exhibit higher fracture strength with adhesive cements. Literature is inconclusive about the type of resin cement used. The modulus of elasticity is considered more important than the type of resin cement. There are no data found in the literature on fracture strength using contemporary glass-ceramics, such as lithium disilicate. So extrapolation of the findings to current materials and cementation protocols should only be done with great reservations. Little evidence supports the use of IDS in increasing the fracture load [60].

3.1.4. Marginal Gap and Marginal Integrity (n = 26 Studies). Twenty-six studies could be identified that met the inclusion criteria; their risk of bias is overviewed in Table 4. The effect of different viscosities was given special attention by several authors. The *in vitro* studies focusing on marginal gap and marginal integrity are too numerous to allow for individual discussion. Therefore the relevant findings evolving from these studies are outlined below.

A consistent finding is that the least microleakage and the best marginal adaptation are obtained when using a resin cement [50, 61–64]. These cements are also the least affected by artificial ageing. A glass ionomer cement exhibited a considerable drop in marginal adaptation after thermocycling, and such a finding seems relevant to clinical practice [51].

Four studies [65–68] focused on the effect of resin cements with different viscosities on marginal adaptation when luting a glass-ceramic restoration. The degree of viscosity was generally referred to as "high" (e.g., Variolink Ultra; Microfil Pontic C; Cerec Duo cement; Spectrum-TPH) or "low" (e.g., Variolink II; Nexus-high), without further physical description of the terms "high" or "low." Both the initial size of the gap and the viscous properties of the luting agent were found to influence the final marginal (and also internal) gap width and marginal integrity. For relatively small discrepancies between the outline of the preparation and the margin of the restoration, low and high viscous cements result in similar interface widths after cementation [65]. Highly viscous cement is recommended for restorations with a larger luting space [66, 67]. Even luting spaces greater than 100 μm can be partially compensated by a resin cement. In such cases highly viscous, filled composite cements are recommended when considering the quality of postcementation marginal integrity [68].

When applying resin cements, the degree of microleakage is generally higher on dentin margins than on enamel margins [57, 69–75]. Cement systems involving an etch-and-rinse approach result in higher percentages of gap-free margins in enamel than other luting systems, although in one study no difference is found between the etch-and-rinse cement (Panavia F2.0) and a self-adhesive resin cement (RelyX Unicem) [76]. However, self-etch adhesives and self-etch cements are also capable of sealing dentin tubules [77–79] or were even considered superior to the etch-and-rinse approach regarding this aspect [80].

In a study involving the cementation of partial crowns, preparation design was of no influence with respect to the size of the marginal gap [63].

Five studies [46, 75, 80–82] investigated the potential benefit of an IDS on the marginal gap. A temporary restoration was provided in only one of the studies [80]. In two studies the flowable composite extended to the cervical margin [75, 81], whereas in the other studies contamination of the margin with resin material was avoided [80, 82], which seems a relevant difference when looking at marginal adaptation. In most studies, less microleakage was seen when applying IDS compared to no IDS [75, 80–82]. However, one study found little difference in reducing microleakage at the dentin interface and even increased it at the enamel interface [46].

In summary, adhesive resin cements showed the least microleakage and are least affected by artificial aging. With a large marginal gap a highly viscous cement is recommended, when the gap is smaller there is no advantage but also no disadvantage of using a highly viscous cement. "Small" and "Large" are not further specified. Compared to enamel, there was generally more microleakage in dentin. There was little proof that with etch-and-rinse systems a higher percentage of gap-free margins could be obtained in enamel, compared to dentin. With self-etching systems and self-adhesive systems equivalent or even more gap-free margins were reached in dentin. IDS was generally considered of merit in reducing microleakage.

3.2. In Vivo Studies (n = 20 Studies). There were twenty clinical studies on glass-ceramic restorations comparing different cementation protocols, but protocols and materials were seldom similar among different studies. Their risk of bias is overviewed in Table 5. Clinical performance is described as survival or success, often with additional qualitative measures such as USHPS criteria (United States Public Health Services criteria) and CDA-criteria (California Dental Association criteria).

Mirage fired feldspathic restorations were luted with either a dual-cure composite (Mirage) or a glass ionomer luting cement (Fuji I), resulting in 2% and 15% lost or fractured restorations, respectively, after a maximum observation period of 3 years. The predominant complication was adhesive bond failure at the cement-porcelain interface [83] as also concluded by others [84]. Clinically, good marginal adaptation and marginal seal and consequently little marginal discoloration, as well as good wear resistance, were observed,

as expressed according to the USHPS criteria. No difference was seen in the cementation procedure. Marginal breakdown of this type of restoration cement with glass ionomer was also seen in a different study [85].

In another similar study restorations could be evaluated after 6 years with 12% and 26% failures, respectively. The difference was already obvious at the 3-year recall period [86]. In contrast to the former study, a deterioration of qualitative parameters was seen during the initial 3 years when judged according to USPHS-criteria regarding marginal adaptation and surface roughness for the dual-cure cement group and even more so for the glass ionomer group. The use of a light-cured (Mirage) instead of a dual-cured adhesive cement (Mirage FLC) presumably caused incomplete curing of the cement because of insufficient penetration of the light through the inlays, with concomitant reduction in fracture strength [87]. The insufficient penetration was associated with 80% versus 20% fracture of the Mirage restorations after a mean observation period of just over one year, especially in thin restorations (<2 mm). These restorations were so thin because a lining cement was used in case of deep preparations (Dycal or a glass ionomer). A similar protocol to protect the vital pulp was adopted in the study by van Dijken et al. [86], which should be kept in mind when extrapolating the results to other situations or current cementation protocols.

In another split mouth study, Cerec (Vita Mark II) inlays were cemented with either a dual-cured (Vita Cerec Duo Cement, Vita) or chemically cured resin cement (Cavex Clearfil F2) and evaluated according to the criteria of the California Dental Association. Twenty-three percent of the restorations were replaced, all from the dual-cured resin cement group within a 10-year period. Possibly, the self-curing capacity of the dual-cured resin cement was insufficient to achieve adequate hardening in order to withstand the stresses and strains that can arise in posterior regions. Although no differences in qualitative parameters were reported between baseline and the period after 10 years, acceptable scores for marginal discoloration after 10 years were seen more frequently in the dual-cured than in the chemically cured cement group (58% versus 78%) [88].

Klink and colleagues also used Vitablocs Mark II full crowns, partial crowns, and inlays luted with either Variolink II or RelyX Unicem. According to the CDA-criteria inlays and partial crowns performed well. Prevalence of complications or failure was highest for crowns. They concluded that success was related to patient factors and restoration type, not luting protocol [89]. Others also found that resin cement type had no influence on success using the same ceramic material [90]. It is noteworthy that the margins were entirely in enamel.

In a study by Gemalmaz and colleagues two adhesive cements (Variolink Ultra and Enforce) and a glass ionomer cement (Geristore) were used to lute Ducere LFC ceramic inlays resulting in 13%, 13%, and 33% failures, respectively, after a little more than 2 years. Margins were evaluated by SEM on gypsum models. Deterioration of marginal adaptation, rate of submargination, and marginal discoloration of surviving restorations luted with the glass ionomer cement were markedly inferior to those luted with the other two cements, with the restorations cemented with Variolink Ultra performing the best [91].

In a prospective dual-center study, the clinical behavior of adhesively luted pressed glass-ceramic restorations (Cergogold) was evaluated using two cementation regimens (personal communication). One group of restorations was luted with Definite Multibond primer with corresponding adhesive and definite cement and the other with Syntac classic (3-step) with Variolink Ultra cement. Survival rates were 93% and 95%, respectively, after 4 years, with the first group exhibiting more hypersensitivity shortly after cementation of the restoration (27% versus 0%). Hence both luting protocols provided similar results when compared according to USPHS criteria and by SEM [92]. A similar conclusion was reached in a different study by the same group involving other patients after 4 years of clinical service [93]. Two operators luted Cergogold inlays in 39 patients using protocols same as those previously described. Considerable interoperator differences were observed with respect to annual failure rate (0.6 versus 6.2%).

Lithium disilicate restorations were cemented with either a commercially available self-etching dual-curing cement (control, Multilink Automix) or a self-adhesive dual-curing "experimental" cement originating from the same company (experimental). Both cements had qualitatively similar results after 2 years of function as assessed by the modified USPHS criteria. All restorations functioned for 2 years without crown fracture or surface chipping. The undisclosed nature of the experimental cement leaves little room for practical comparison or interpretation. The publication did not mention the type of restoration that was provided (full, circumferential, or partial) [94]. For this restoration type, inlays luted with resin-modified glass ionomer cement (Fuji Plus F) or a self-cured resin composite cement (Panavia 21) yielded similar results after 5 years [95]. IPS Empress (leucite reinforced glass-ceramic) restorations were cemented with different adhesive approaches and can function successfully for 15 years [96]. Others also saw good long term results but described a significant amount of deterioration of marginal adaptation in the long run, even though modern adhesive procedures were used. Overall failure rates of this type of restoration were in the order of 8–10% after 10 years [97–99]. A classic etch-and-rinse approach (Syntac classic/Variolink II) produced better marginal integrity when cementing leucite reinforced glass-ceramic inlays than a contemporary self-adhesive resin cement (Relyx Unicem) after 2 years in function [100]. Another author favored dual-cure cements based on 12-year results [101], whereas the viscosity of the cement (low versus high) had no influence on success in a large prospective study after 10 years [102].

In conclusion, most included, rather heterogeneous clinical studies involve relatively old, no longer available restoration types or systems. The use of lining cements in several older protocols challenges external validity. Cementation protocols involving glass ionomer cements generally (but not always) result in more fracture and loss of restorations as well as poorer qualitative performance of surviving restorations compared to protocols involving adhesive resin cements. Studies comparing cementation protocols for more

contemporary restorative materials (lithium disilicate) are rare and involve self-etching, self-adhesive, or adhesive procedures. None of these cementation protocols can be considered clearly superior in clinical performance on the basis of the reviewed literature.

There is limited evidence that light-cured resin cements perform worse than dual-cured cements, whereas solely chemically cured resin cements perform the best. Results obtained with technically challenging adhesive cementation procedures may be operator-dependent. Marginal deterioration is frequently reported, also when using adhesive cements.

No clinical studies evaluated the potential benefits of IDS protocols that were identified.

4. Discussion

This review is aimed at organizing knowledge regarding the cementation of glass-ceramic restorations, particularly posterior, single-unit ones, with a special emphasis on the possible merits of IDS. The topic is of interest to the clinician because of the growing number of all ceramic restorations that are being placed. They substitute metal and metal-ceramic crowns and are advantageous because they are relatively cheap in light of the current gold price and their manufacturing price and because of their superior esthetics. In early years, glass-ceramics were cemented with conventional cements like glass ionomers, with limited adhesive properties. This reflects on the results, as demonstrated in this comprehensive review, and consequently challenges the external validity of data subtracted from these studies to contemporary, strengthened glass-ceramics (leucite reinforced glass-ceramic and lithium disilicate). By removing superficial glass content by etching, glass-ceramics can be cemented adhesively and as a result allow nonretentive preparation forms, maintaining sound tooth tissue. This may help in avoiding endodontic complications.

Bonding to dentin has traditionally been considered to be more challenging than to enamel. IDS may provide better results with respect to the bonding capacity and it is possibly also more friendly to the pulp.

Over 3000 studies were initially identified for this review, but many were discarded, predominantly because they did not compare different cementation protocols or evaluated a "glass-ceramic/cement/human tooth" complex. The selection on articles in the English language only may have introduced some bias.

The *in vitro* and *in vivo* studies that were included proved dramatically heterogeneous. Consequently, they do not allow meta-analysis or relevant grouping because of different test methods (e.g., tooth and substrate preparation, dimension and geometry of the restoration or tested ceramic, tooth number, storage conditions, artificial aging/thermocycling or not, cyclic loading or not, cementation protocols (e.g., a single or a double adhesive layer), testing machines, standardization of the test method, crosshead speed of the testing device and the size of the steel ball during instrumentation, the use of a "stress breaker" such as a rubber dam, film thickness of luting cements, or (lack of) definition of outcome parameters, particularly the mode of failure). It was decided to include studies only if they compared cements or cementation procedures, thus correcting for the heterogeneity in some manner. Often it was complicated to categorize the cementation procedures into "adhesive," "self-etching," or "self-adhesive" because of the chosen bonding agents and the confusing way that they were applied and described.

With respect to the application of IDS, terminology and the clinical application in the literature regarding this procedure are different. The present authors regard IDS as a procedure in which a resin layer is applied immediately after preparation, followed by impression taking and the provision of a temporary restoration in combination with a temporary cement. Eventually, this restoration is replaced by a glass-ceramic one, which is luted to the reactivated IDS layer and the uncovered tooth structure by means of a resin cement. In the current review, when no temporary restoration was provided in an evaluated study, it is referred to as a "resin coating," which is fundamentally different. The manner in which such an intermediate layer is applied and conditioned is also expected to be of influence and often different among studies that were included.

Nevertheless and possibly as a result of the rather rigorous inclusion and exclusion criteria, the included studies in the review are generally considered of good methodological quality as evaluated by Cochrane's collaboration tool of bias.

In vitro studies identify some differences in outcome resulting from the tested protocols or variables. These are generally not reflected in rather more crude, clinical outcome measures, such as survival of a restoration, presented in *in vitro* studies. Therefore it is tentatively suggested that when luting modern glass-ceramics to posterior teeth, adhesive protocols that are the most operator and patient friendly may be preferred.

5. Conclusion

Bearing in mind the shortcomings and limitations of this review as described above, the following conclusions are drawn.

From *in vitro* studies it can be concluded that adhesive systems (3-step, etch-and-rinse) show the best (micro)shear bond strength values compared to self-adhesive and self-etch systems when luting to human dentin. For (micro)tensile strength values or evaluation of the marginal gap no such preference can be identified on the basis of the reviewed literature. The highest fracture strength is obtained using adhesive cements, rather than water based cements like glass ionomer.

Clinical studies comparing cementation protocols for contemporary restorative glass-ceramic materials (lithium disilicate) are rare and involve self-etching, self-adhesive, and adhesive procedures. No marked clinical preference for one specific procedure could be demonstrated on the basis of the reviewed literature.

Few studies focus on the possible merits of IDS. The benefits are most convincingly illustrated by the favorable microtensile bond strengths when compared to negative or

positive controls *in vitro*. No clinical trials have been performed and deleterious clinical consequences, be it objective or subjective, were not reported.

Conflict of Interests

The authors declare that they have no conflict of interests.

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Research Article

Real-Time Analysis of Temperature Changes in Composite Increments and Pulp Chamber during Photopolymerization

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Objective. The aim of this study was to evaluate the temperature change at various sites within the composite and on the pulpal side of dentin during polymerization of two composite increments. *Materials and Methods*. Class I cavities prepared in third molars were restored in two composite increments ($n = 5$). Temperatures were measured for 110 s using eight thermocouples: bottom center of cavity (BC), top center of 1st increment (MC), top center of 2nd increment (TC), bottom corner of cavity (BE), top corner of 1st increment (ME), top corner of 2nd increment (TE), pulpal side of dentin (PD), and center of curing light guide tip (CL). *Results*. Maximum temperature values (°C) measured during polymerization of 1st increment were MC (59.8); BC (52.8); ME (51.3); CL (50.7); BE (48.4); and PD (39.8). Maximum temperature values during polymerization of 2nd increment were TC 58.5; TE (52.6); MC (51.7); CL (50.0); ME (48.0); BC (46.7); BE (44.5); and PD (38.8). *Conclusion*. Temperature at the floor of the cavity was significantly higher during polymerization of 1st increment compared to 2nd increment. Temperature rise was higher at the center than at the corner and at the top surface than at the bottom surface of each increment.

1. Introduction

Composite resins have become the most widely used direct restorative materials in meeting the patient demand for esthetic dentistry. Despite the many advantages of composites, heat is inevitably generated during photopolymerization by the exothermic reaction of composite resin and by the light-curing unit *per se* [1]. It is well known that excessive heat is detrimental to living tissues such as the dentin-pulp complex and periodontal tissue. Possible outcomes of thermal injuries during dental procedures include transient pulpal inflammation, irreversible pulpal inflammation or necrosis [2, 3], bone resorption, and tooth ankylosis [4].

Numerous studies have demonstrated a positive correlation between the light-curing unit intensity and temperature rise [5–11]. The degree of dentin mineralization [12] and the remaining dentin thickness [12–14] showed a negative correlation with a temperature rise during polymerization of light-cured composites. Previous studies have evaluated the temperature rise during polymerization of composite resin by using a thermistor [5, 9], thermocouples [6–8, 10, 12, 15], differential scanning calorimetry [16], differential thermal analysis [17, 18], and infrared thermography [11, 14].

However, these studies measured the temperature change only at the bottom surface of composites [5, 6, 9], at the pulpal surface of dentin [7, 10, 12], or at the center of

composites [8, 15]. Recently, Chang et al. [19] measured the polymerization temperature at multiple spots along the external surface of a Class II cavity according to the curing depth and approximation to the cavity wall using infrared thermography. Nevertheless, to date there is no published study that has performed the simultaneous measurements of the temperature changes within the composite resin and at the pulpal side of the dentin using human teeth under simulated *in vivo* conditions. Moreover, no studies exist evaluating the effect of incremental curing of composites on the temperature changes.

The aim of this paper was to compare the temperatures measured at various sites along the bottom and top surfaces of composite increments and the pulpal side of dentin during photopolymerization of two increments of composites in real-time using multiple thermocouples.

2. Materials and Methods

2.1. Specimen Preparation. The experimental setup is shown in Figure 1. Five freshly extracted, intact caries-free third molars stored in 0.5% chloramine-T solution were used for the study. The occlusal surface of each tooth was ground to a flat surface and prepared with Class I cavities (mesiodistal length, 5 mm; buccolingual width, 4 mm; and depth, 3 mm) using a flat end cylindrical diamond bur. The lower portion of the crown was horizontally sectioned at 4 mm below the occlusal surface of the cavity, leaving a 1 mm thick dentin remaining between the internal bottom surface of the cavity and the external horizontally sectioned surface.

The temperature measurement sites are as follows, assigned according to the position of the thermocouples:

BC: bottom center of the cavity,

MC: top center of the 1st increment composite resin,

TC: top center of the 2nd increment composite resin,

BE: bottom corner of the 1st increment composite resin,

ME: top corner of the 1st increment composite resin,

TE: top corner of the 2nd increment composite resin,

PD: bottom center on the pulpal aspect of dentin,

CL: center of the curing light guide tip.

Two vertical grooves (one for the center and another for the corner positions) extending from the occlusal surface to the bottom of the cavity were made for each tooth by cutting the corners of the teeth using a diamond bur to accommodate K-type thermocouples with a diameter of 0.5 mm (TT-K-36, Omega, Stamford, CT, USA) within the cavity. A flowable composite resin (Charisma, Shade A2, Lot 10128, Heraeus-Kulzer, Hanau, Germany) was applied around the thermocouple wires within the grooves and light-cured for 20 s to secure the thermocouples, which were connected to a thermocouple conditioner and setpoint controller (AD597, Analog Devices, Norwood, MA, USA). The thermocouple signals were digitized in real-time with a data acquisition board (cDAQ-9174, National Instruments, Austin, TX, USA) equipped with an analog input module (NI 9205, National Instruments) using a data acquisition software (LabVIEW, National Instruments).

2.2. Temperature Measurement. Temperature changes were recorded during polymerization of two increments of composite resin (Filtek Z250, Shade A2, Lot N506344, 3M ESPE, St. Paul, MN, USA). For temperature measurement, thermocouples were position at BC, BE, PD, and CL, and then after the cavity was filled with the 1st increment (1.5 mm thick, 30 mm^3), additional thermocouples were placed at MC and ME. A modified periodontal probe with a marking at 1.5 mm from the tip was positioned along the axis of the tooth cavity in order to control the thickness of the composite increment. A baseline was obtained for 10 s without light exposure, after which the curing light was turned on for 20 s at 750 mW/cm^2 (Elipar S10 LED curing light, 3M ESPE). The tip of the curing light was positioned 2 mm away from the specimen. After recording the temperatures once every second for 110 s, a subsequent 2nd increment of composite (1.5 mm thick, 30 mm^3) was placed over the light-cured 1st increment up to the top of the cavity, and additional thermocouples were placed at TC and TE. As in the 1st increment, the temperatures were recorded for 110 s, including a 10 s baseline and a 20 s light-curing. Temperatures were measured at six sites except TC and TE for the 1st increment and at all eight sites for the 2nd increment. During temperature measurements, each specimen was secured in a water bath maintained at 36.5°C, whereby the 1 mm thick remaining dentin was immersed in water with the rest of the tooth above the water level. Each experimental condition was replicated five times.

2.3. Statistical Analysis. The mean maximum temperatures of the assigned measurement sites were analyzed using one-way ANOVA followed by Tukey's HSD post hoc comparison analysis. The analyses were conducted with SPSS software version 21 (IBM, New York City, NY, USA) at a significance level of 0.05.

3. Results

Mean temperatures as a function of time during polymerization of the 1st and 2nd increments are shown in Figure 2. A rapid increase and decrease in temperature corresponded to the curing light being turned on and off, except at PD.

The mean maximum temperatures during polymerization of the 1st and 2nd composite increments are summarized in Table 1. The maximum temperatures were significantly increased with a decrease in distance between the curing light and the measurement site. Also, the maximum temperatures were significantly higher at the center when compared to the corner of the composite within the same depth. During polymerization of the 2nd composite increment, the maximum temperatures measured at BC and MC were significantly lower than those obtained during the initial curing of the 1st increment ($P < 0.05$) (Figure 3). The pulpal aspect of the dentin at PD exhibited the lowest mean maximum temperatures of 39.8 ± 1.5°C during polymerization of the 1st increment

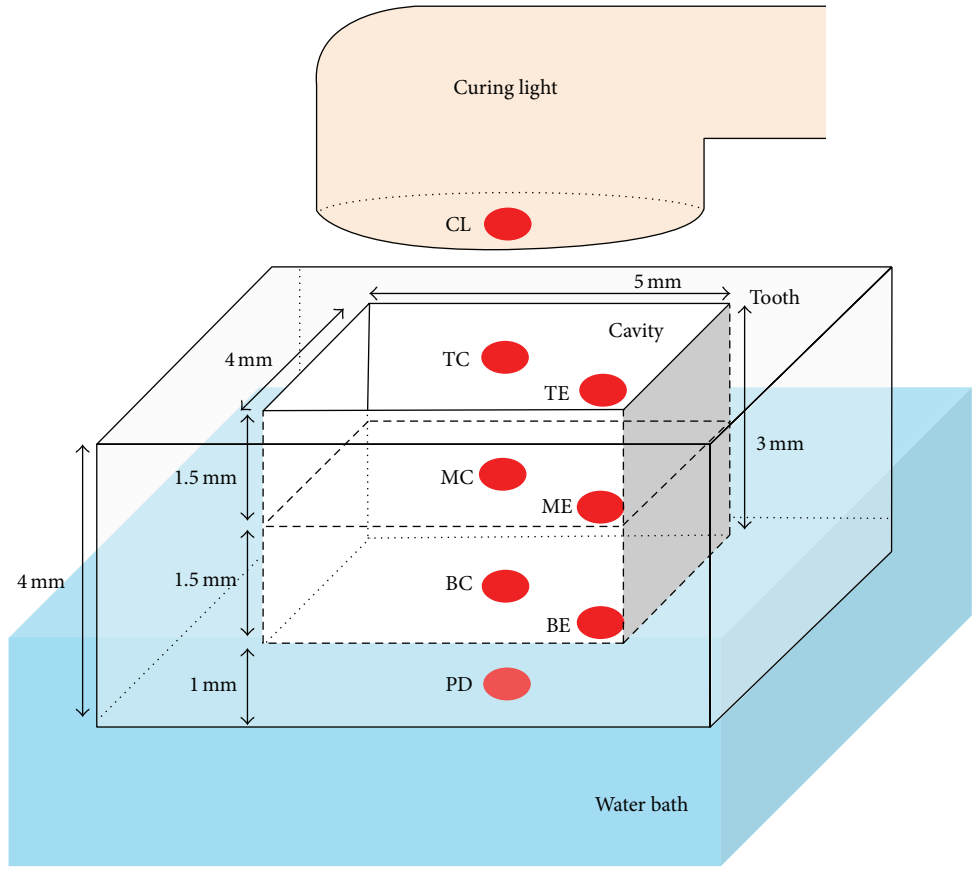

Figure 1: Schematic diagram of specimen setup showing the position of thermocouples and dimension of tooth cavity (BC: bottom center of the cavity; MC: top center of 1st increment; TC: top center of 2nd increment; BE: bottom corner of 1st increment; ME: top corner of 1st increment; TE: top corner of 2nd increment; PD: bottom center on pulpal aspect of dentin; and CL: center of curing light guide tip).

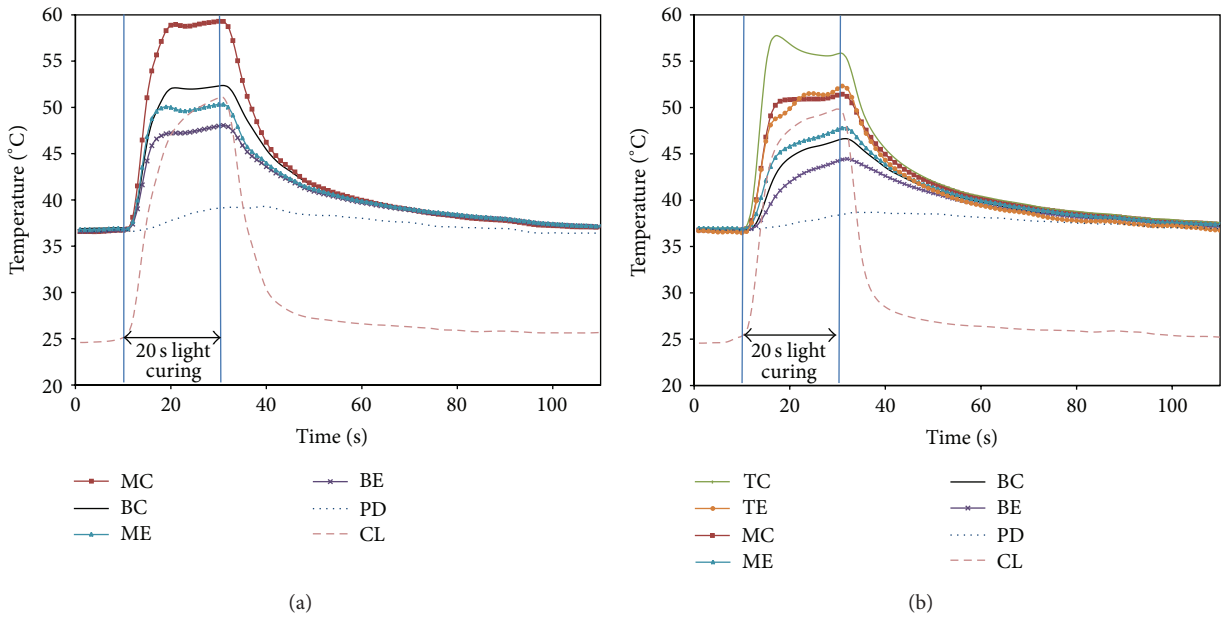

Figure 2: Mean temperatures measured during polymerization of (a) 1st composite increment and (b) 2nd composite increment (BC: bottom center of the cavity; MC: top center of 1st increment; TC: top center of 2nd increment; BE: bottom corner of 1st increment; ME: top corner of 1st increment; TE: top corner of 2nd increment; PD: bottom center on pulpal aspect of dentin; and CL: center of curing light guide tip).

Table 1: Mean maximum temperatures (°C) at assigned measurement sites during polymerization of each increment.

	Measurement sites							
	BC	MC	TC	BE	ME	TE	PD	CL
1st increment	52.8 (2.5)^{b}	59.8 (3.8)^{a}	N/A	48.4 (4.0)^{b}	51.3 (5.9)^{b}	N/A	39.8 (1.6)^{c}	50.7 (1.4)^{b}
2nd increment	46.7 (1.1)^{df}	51.7 (1.5)^{bc}	58.5 (2.1)^{a}	44.5 (1.4)^{ef}	48.0 (2.1)^{cde}	52.6 (4.0)^{b}	38.8 (0.1)^{g}	50.0 (1.3)^{bd}

Standard deviations are in parentheses.
Values with identical superscript letters are similar within the same increment (Tukey's HSD, $P > 0.05$).
N/A: Not applicable.
BC: bottom center of the cavity; MC: top center of 1st increment; TC: top center of 2nd increment; BE: bottom corner of 1st increment; ME: top corner of 1st increment; TE: top corner of 2nd increment; PD: bottom center on pulpal aspect of dentin; and CL: center of curing light guide tip.

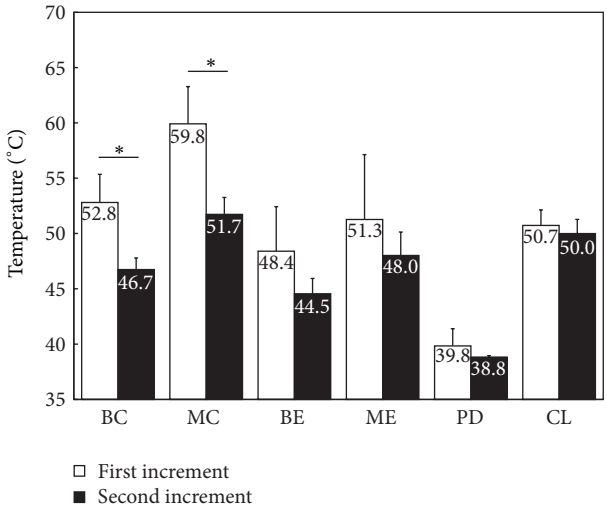

Figure 3: Means and standard deviations of maximum temperature during polymerization of 1st and 2nd composite increments. Asterisk indicates statistical difference between the 1st and 2nd composite increments (BC: bottom center of the cavity; MC: top center of 1st increment; TC: top center of 2nd increment; BE: bottom corner of 1st increment; ME: top corner of 1st increment; TE: top corner of 2nd increment; PD: bottom center on pulpal aspect of dentin; and CL: center of curing light guide tip).

and 38.8 ± 0.2°C during polymerization of the 2nd increment. The maximum temperatures recorded at MC (59.8 ± 3.2°C) and TC (58.5 ± 2.1°C) were highest of all the measurement sites during polymerization of the 1st increment and 2nd increment, respectively ($P < 0.05$).

4. Discussion

The temperature rise of light-cured composites is typically caused by both an exothermic reaction process and the transmission of heat from the curing light itself [1]. An exothermic reaction takes place as carbon-to-carbon double bonds in the monomer change to a single bond during polymerization [11, 14]. Previous studies have reported that the temperature rise is greater with increasing power density [5–8, 10] and irradiation time [9, 10] of the curing light and with decreasing the distance from the curing light tip [9].

In the present study, multiple thermocouples were used to provide a comprehensive understanding of temperature changes at various sites within the cavity and at the pulpal aspect of dentin during polymerization of two increments of composites. Within the cavity, temperatures were measured at the center as well as the corner of each increment since the temperature change in the corner is also of value to note considering the tooth is a living tissue; the corner adjacent to the cavity wall is in communication with the dentin-pulp complex via odontoblasts and their processes through the interconnecting dentinal tubules.

At the same depth within the composite increments, the temperature rise at the center was higher than at the corner adjacent to the cavity walls. It could be explained by the difference in the amount of reacted composites. Studies have shown that there is a proportional relationship between the temperature rise from the exothermic reaction and the amount of composite resin [11, 14]. In this study, the thermocouples placed at the center were surrounded by a much greater amount of composites to be polymerized, which in turn resulted in a higher temperature rise due to greater heat generation via the exothermic reaction, than those placed in the peripheral composites. Considering the greater heat generation with increasing amount of composite resin to be polymerized, clinicians should consider placing a smaller amount of composite resin for the initial first layer in order to minimize potential thermal irritation to the pulp.

An inversely proportional relationship was found between the temperature rise and distance from the curing light to the measurement sites in each incremental layer of composite resin. The top surface received the maximum energy from the curing light as it was closer to the curing light. Al-Qudah et al. [11] have reported that the temperature rise on the lower surface of the composite was lower with an increase in the thickness of the composite specimens. This observation can be explained by the exponential reduction in the light intensity, due to light scattering, with increasing composite thickness [20]. Thus, as the light passes through the thicker composite, the effectiveness of the light cure at the bottom surface is impaired, as evidenced by the reduction in the degree of conversion [17, 21]. Such a pattern of temperature rise emphasizes the important role the distance from the light source and thickness of composite material have in the temperature rise. Knežević et al. [22] also showed a higher degree of conversion and temperature rise on the

surface than at a depth of 1 mm, supporting the current findings of a higher temperature on the top surface of each increment. A light-emitting diode (LED) curing light was employed in the present study to help clinicians understand how much the temperature may rise during a composite restoration in the clinical setting using the currently most commonly used curing light type for photopolymerization of the composite. With regard to the type of light-curing units, LED curing units are reported to generate less heat as a result of having narrower emission spectra, compared to halogen curing units, which have broad emission spectra far into the ultraviolet A range [23].

As the curing light was turned on and off, a corresponding rapid increase and decrease in temperature was observed at all measurement sites except the pulpal side of the dentin. The lowest temperature rise was observed after a latent phase of approximately 10 s at the pulpal side of dentin during polymerization of both increments (Figure 2). The surface temperature on the pulpal aspect of the dentin was further reduced from 39.8 ± 1.5°C (3.3°C rise) in the 1st increment to 38.8 ± 0.2°C (2.3°C rise) in the 2nd increment. Zach and Cohen [2] evaluated the histological responses of dental pulp to thermal stress in Macaca rhesus monkeys. They found 15% of the healthy teeth failed to recover when the pulp temperature was increased by 5.6°C, 60% after an increase of 11.1°C, and almost 100% when the temperature was raised above 11.1°C. Thus, an increase of intrachamber temperature by 5.6°C has been considered a critical threshold for irreversible pulpal damage [2]. As per the current results, the temperature rise in the pulpal aspect of dentin, underneath the 1 mm thick remaining dentin, during polymerization of both increments was below the critical value. Since the dentin is known to be an excellent thermal insulator [14], having a 2.5-fold lower thermal diffusivity than enamel [24], the temperature rise was significantly reduced and slowed down on the pulpal side of the dentin. The remaining dentin thickness is therefore an important protective factor against thermal injury to the pulp [12–14]. In addition to the insulating effect of dentin, the cooling effect of water within the pulpal chamber reduced the temperature rise on the pulpal side of the dentin. Temperature rise would be even greater, increasing the potential for jeopardizing pulpal health during composite restoration, in the following situations where the remaining dentin thickness is reduced: (1) when more remaining mineralized dentin [12] associated with caries or previous treatment history exists due to the presence of greater amount of reparative dentin that has a higher thermal diffusivity; (2) when local anesthesia including vasoconstrictor is administered, reducing the cooling effect of pulpal blood due to the reduction of pulpal blood flow [25, 26]; and (3) when the volume of the pulp chamber is reduced, especially in aged people, thereby relatively reducing the cooling potential.

The present study demonstrated that the values and patterns of temperature changes were different among various sites within the composite during photopolymerization. Further studies are required to evaluate the effect of light-curing intensity and mode, remaining dentin thickness, and thickness of each incremental composite resin layer on temperature rise, thereby providing clinical recommendation in the context of minimizing temperature rise.

5. Conclusions

Temperature measured at the floor of the cavity was higher during polymerization of the 1st increment. The temperature rise was higher at the center than the corner and at the top surface than at the bottom surface of each increment. Given the higher temperature rise with the increasing amount of composites, it is suggested to avoid placing a large amount of the composite as a first layer to minimize potential thermal damage to the pulp, especially in the case where the remaining dentin is thin. Within the limitations of this experiment, the temperature rise on the pulpal aspect of dentin underneath the 1 mm thick remaining dentin does not seem to cause detrimental effect on the pulpal health.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Authors' Contribution

Ryan Jin-Young Kim and In-Bog Lee contributed equally to this work.

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Research Article

Effects of Computer-Aided Manufacturing Technology on Precision of Clinical Metal-Free Restorations

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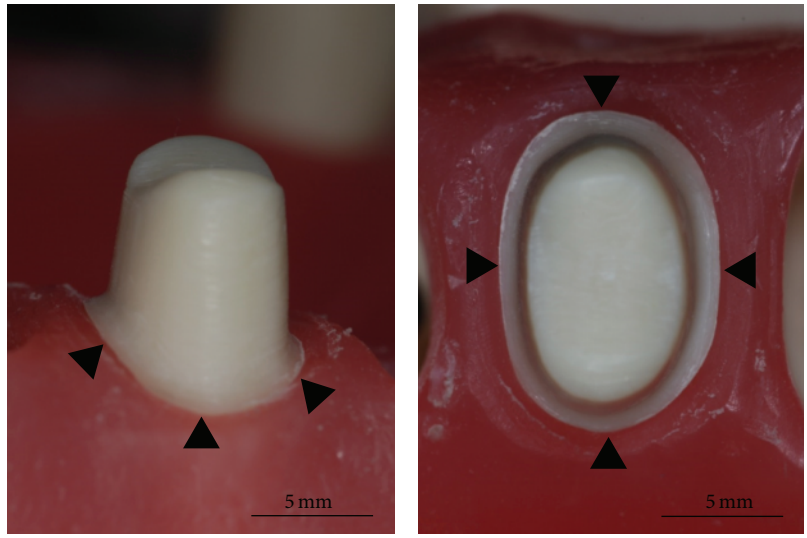
Purpose. The purpose of this study was to investigate the marginal fit of metal-free crowns made by three different computer-aided design/computer-aided manufacturing (CAD/CAM) systems. *Materials and Methods*. The maxillary left first premolar of a dentiform was prepared for all-ceramic crown restoration. Thirty all-ceramic premolar crowns were made, ten each manufactured by the Lava system, Cercon, and Cerec. Ten metal ceramic gold (MCG) crowns served as control. The marginal gap of each sample was measured under a stereoscopic microscope at 75x magnification after cementation. One-way ANOVA and the Duncan's post hoc test were used for data analysis at the significance level of 0.05. *Results*. The mean (standard deviation) marginal gaps were 70.5 (34.4) μm for the MCG crowns, 87.2 (22.8) μm for Lava, 58.5 (17.6) μm for Cercon, and 72.3 (30.8) μm for Cerec. There were no significant differences in the marginal fit among the groups except that the Cercon crowns had significantly smaller marginal gaps than the Lava crowns ($P < 0.001$). *Conclusions*. Within the limitation of this study, all the metal-free restorations made by the digital CAD/CAM systems had clinically acceptable marginal accuracy.

1. Introduction

With increasing demand for aesthetics, many studies on zirconia, which is the most representative element for metal-free restoration in the field of restorative dentistry, have been recently performed due to its acceptable aesthetics and high strength that is comparable with the strength of a metal ceramic crown [1–8]. Yttria-stabilized tetragonal zirconia polycrystal is provided as a block form to secure the maximum strength [6, 7]. A new precise mechanical subtracting process has been introduced instead of the previous adding method including waxing, investing, and casting to fabricate a prosthodontic shape from the block. A computer-aided design/computer-aided manufacturing (CAD/CAM) system has been further developed in dentistry over the last 20 years to handle very precise data acquisition, complex restoration design, complete task processing, and high-end cutting system [9].

One of the most important elements in evaluating a fixed prosthodontic device is marginal accuracy. Every prosthodontic restoration process, from abutment preparation to cementation, has effects on the marginal fit of the restoration [10]. Unlike the traditional analogue methods, the CAD/CAM system needs the precision of the system itself, including the accurate digital conversion of acquired information and calibration of the digitalized data according to materials used in CAM. Therefore, it is important in clinical CAD/CAM application to prosthodontic restoration to understand both the differences between the CAD/CAM systems and the accuracy of the resulting crowns.

This study aimed to investigate the marginal fit of zirconia crowns made by widely used CAD/CAM systems: Lava (3M ESPE, Seefeld, Germany), Cercon (DeguDent, Hanau, Germany), and Cerec (Sirona Dental Systems GmbH, Bensheim, Germany). This study also compared the marginal fit of the zirconia crowns with that of a metal ceramic gold (MCG)



(a) (b)

Figure 1: The prepared premolar resin tooth from the buccal (a) and occlusal viewpoints (b). Note the circumferential cervical margin of 1 mm width (black arrowheads).

crown, which is one of the restoration forms clinically used for the longest period.

2. Materials and Methods

The maxillary left first premolar (#24) of the dentiform (Columbia Dentoform Corp., New York) was prepared to form an abutment tooth. Two millimeters of the occlusal surface and 1.0–1.4 mm of the lateral side were reduced. The completed convergence angles of the abutment were about 8–10° both mesiodistally and buccolingually. The margin was assigned with 1 mm of a heavy chamfer margin in the overall range of the cervical aspect (Figure 1). After the abutment preparation, the resin tooth was invested onto the plaster, and the impression was acquired by using the additional silicone impression products of putty and light body (Exafine, GC Co., Tokyo, Japan). Forty original resin models (Exakto-Form, Bredent, Senden, Germany) were manufactured from the silicone impression. These resin models were subsequently used for the measurements of the marginal openings after the final restorations were cemented to these models. The models were divided into 4 groups by assigning 10 models to each group. Lava, Cercon, and Cerec systems were used to fabricate final restorations. Ten single MCG premolar crowns served as control, which were made by the conventional casting method. The other all-ceramic crowns were fabricated according to the manufacturers' recommendations of the systems evaluated in this study. The gap for cement was all assigned as 60 μm.

The working die productions for the MCG, Lava, and Cercon crowns were performed using high strength dental stone (GC Fujirock EP, GC Europe N.V., Leuven, Belgium) after taking impressions of the original resin models with the additional silicone impression materials of putty and light body (Exafine). The virtual working dies for the Cerec crowns were produced by direct scanning method. For the production of MCG crowns, a wax pattern was produced by using a conventional method with high strength dental stone model. The die spacer (Pico-Fit Die Spacer Varnish (silver), Renfert USA, Il, USA) was coated 3 times on high strength dental stone. Considering the fact that 1 time die spacer coating creates a layer thickness of 14–20 μm from the manufacturer's technical data, the practice allowed for a cement space of approximately 42–60 μm. The gold (Bio Herador SG, Heraeus, Germany) coping was produced by following the investing and casting procedures and then veneered with porcelain. For the Lava crowns, the high strength dental stone dies were scanned with a scanner (Lava Scan Scanner) and zirconia copings were designed under a CAD system (Lava CAD), which gave the cement space of 60 μm. The copings were produced by milling zirconia blocks (Lava zirconia blocks) with a CAM system (Lava Form Milling Unit). The copings were manufactured by setting the thickness of the coping at 0.5 mm. The final crowns were completed by veneering porcelain (Lave Ceram) on the copings after sintering. In the manufacturing of Cercon crowns, the working dies were also scanned using a scanner (Cercon EYE) and zirconia frameworks were designed using a CAD software (Cercon ART). Zirconia blocks (Cercon zirconia blocks) were then milled using a CAM system (Cercon BRAIN), to make the frameworks that were 0.5 mm thick. The milled zirconia frameworks were sintered and were veneered with a heat-pressed material (IPS e.max Ceram, Ivoclar Vivadent AG, Benderer Str. 2, Liechtenstein) and technique, to manufacture the final crowns. For the fabrication of Cerec crowns, the original resin models were directly scanned (CEREC Bluecam) to make the software working dies and the final crowns were designed using a CAD software (CEREC 3D). Zirconia blocks (IPS e.max ZirCAD,

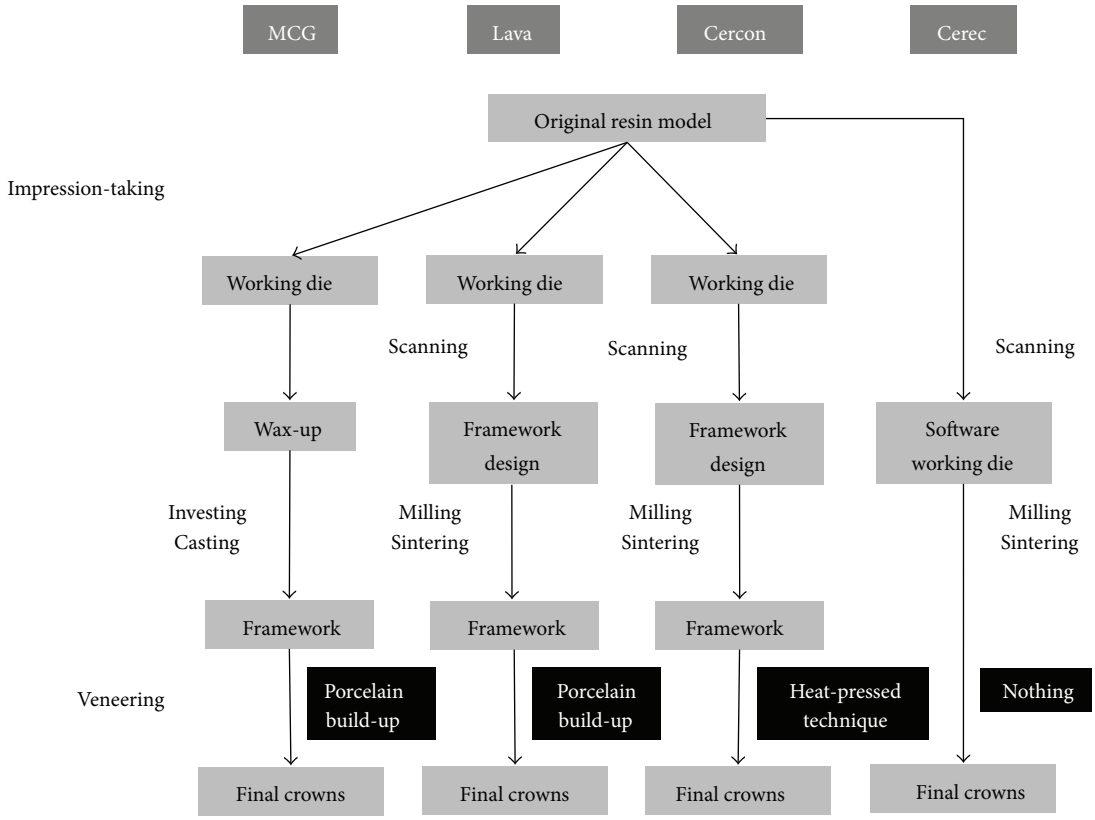


Figure 2: Summarized fabrication procedures for each system. Note that there was neither impression-taking nor veneering procedure in the Cerec system, which manufactured the purely digitalized crowns. Also, notice that each all-ceramic system used a different veneering technique. MCG; metal ceramic gold crowns.

Ivoclar Vivadent AG, Schaan, Liechtenstein) were milled by a CAM system (CEREC in Lab MC XL milling machine) and sintered to make the final restorations with no veneering procedure. The procedures, instruments, and materials to make the specimens are summarized in Figure 2.

The MCG, Lava, Cercon, and Cerec crowns were, respectively, cemented to their own resin models by using a resin cement (RelyX Unicem Clicker, 3M ESPE, Germany). During cement setting time, 50 N loading was applied with finger pressure by the person who had trained to calibrate the 50 N load with a laboratory scale. Excessive cement material was cleaned with cotton pellets. The marginal fit of each sample was measured by using a stereoscopic microscope (Nikon DS-Fi 1, Nikon, Japan) at 75x magnification. The marginal gap was defined in this study as a distance on the microscope from a point of the tooth margin to the intersecting point between the restoration margin and the line perpendicular to the tangent line to the tooth margin at the tooth margin point. For each crown, the gap was measured at one point of the labial, lingual, mesial, and distal surface. The marginal gap of a crown was calculated as the mean of the measured four gaps.

The mean and standard deviation (SD) were calculated for the measured marginal gaps of each group. One-way ANOVA and a post hoc test, Duncan's test, were used to find any statistically significant difference among the groups at the level of significance of 0.05.

3. Results

The mean marginal gaps (SD) of MCG, Lava, Cercon, and Cerec crowns were determined to be 70.5 (34.4) μm, 87.2 (22.8) μm, 58.5 (17.6) μm, and 72.3 (30.8) μm, respectively. The descriptive statistics including the mean, SD, minimum, and maximum measured values of each group are presented in Table 1. One-way ANOVA and Duncan's post hoc test showed that there were no significant differences in the marginal fit among the groups except that the Cercon crowns had significantly smaller marginal gaps than the Lava crowns ($P < 0.001$).

4. Discussion

There are many and various criteria about the clinically acceptable marginal fit of prosthodontic restoration [11–15]. ADA specification number 8 defined that the range should be 25–40 μm, and Ostlund stated that the value should not exceed 50 μm [11]. Unfortunately, those values appear to be very difficult to obtain clinically. Christensen reported that a maximum marginal distance of 119 μm was allowed by dentists for the proximal surface of gold inlays through observations using eyes, probes, and radiographic images and stated an approximate 39 μm maximum marginal distance for the occlusion surface [12]. McLean and von Fraunhofer stated that a marginal gap of about 100 μm does not cause

Table 1: Mean, SD, and minimum and maximum values of the marginal fit for each of the groups.

Group	Mean (SD)	Minimum–maximum
MCG	70.5 (34.4)^{AB*}	31.9–207.7
Lava	87.2 (22.8)^{B}	45.1–140.9
Cercon	58.5 (17.6)^{A}	34.8–97.3
Cerec	72.3 (30.8)^{AB}	21.8–164.1

*The groups with the same superscript letters (A and B) were not significantly different (unit; μm).

any clinical problems in a study observing 1,000 dental restorations performed over more than 5 years, concluding that the clinically allowable maximum marginal discrepancy was 120 μm [13]. Another previous study evaluated that a marginal gap up to 100 μm was clinically acceptable, while still another extended the clinically acceptable marginal gap to 200 μm [14, 15]. There is still controversy over the clinically acceptable marginal fit standard. However, most authors are considered to agree upon the fact that the marginal discrepancy should be less than 200 μm [16–23].

The measurement values that were acquired in the present study were in the clinically acceptable range for all the test groups. Most of the currently used CAD/CAM systems were found to show appropriate clinical marginal fit by exhibiting a mean marginal discrepancy value of less than 200 μm. Bindl and Mörmann found no significant difference in the marginal fit of crowns, when comparing the marginal fit of CAD/CAM all-ceramic crowns of Cerec inLab, DCS, Decim, and Procera, the slip cast type crown of In-Ceram zirconia, and heat-pressing type crown of Empress 2, showing a marginal opening range of about 20–70 μm [24]. The marginal fit of the 4-unit fixed dental prostheses made by four CAD/CAM systems (Cercon, Cerec inLab, Digident, Everest) was evaluated to be 57.9–206.3 μm [25]. Another previous study investigating the marginal accuracy of 3-unit fixed dental prostheses showed the mean marginal gaps of 77–92 μm for the Cerec inLab, Digident, and Lava systems [26]. The previous results were similar to those of this study although there were some numerical differences according to the experimental conditions including the restored teeth (anterior, posterior), the restoration types (single, multiple), and the fabrication procedures.

The Cercon premolar crowns exhibited significantly superior marginal fit to the Lava crowns in this study. However, these statistics were unable to be interpreted as superiority of one system in precision to the other because there were no significant differences either between the Cercon and the control (MCG) groups or between Lava and control. Differences in the veneer techniques, not those in the CAD/CAM systems, could explain some causes of the results shown in this study. Some previous studies showed the differences in accuracy between the restorations with and without the porcelain build-up procedures and the significant effects of the veneering methods on restoration precision [27, 28]. This investigation, however, did not consider a CAD/CAM system and a veneer technique as two independent variables, which was one of the limitations. Further studies are required to evaluate and to compare the effects of those two factors, the systems and veneering methods, on the marginal accuracy of prosthodontic restorations. In addition, this study indicated that the accuracy of a dental restoration fabricated by digital technology may be clinically acceptable, when compared with that by conventional analogue method. However, various approaches were found according to the CAD/CAM systems: pure digital techniques and digital-analogue combinations, as shown in Figure 2. Further studies are needed to compare each step in digital procedures with that in analogue.

5. Conclusions

Computer-aided digital technologies may manufacture metal-free restorations that are clinically acceptable in precision. Considering the results in this study, the marginal gaps of the digitalized metal-free crowns were similar to those of the conventional metal ceramic gold crowns. All the accuracy investigated in this study may be within the generally agreed clinically acceptable marginal fit standard.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Authors' Contribution

Ki-Hong Lee and In-Sung Yeo contributed equally to this work.

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Research Article

Analysis of Self-Adhesive Resin Cement Microshear Bond Strength on Leucite-Reinforced Glass-Ceramic with/without Pure Silane Primer or Universal Adhesive Surface Treatment

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Objective. To evaluate the microshear bond strength (μSBS) of self-adhesive resin (SA) cement on leucite-reinforced glass-ceramic using silane or universal adhesive. *Materials and Methods*. Ceramic blocks were etched with 9.5% hydrofluoric acid and divided into three groups ($n = 16$): (1) negative control (NC) without treatment; (2) Single Bond Universal (SBU); (3) RelyX Ceramic Primer as positive control (PC). RelyX Unicem resin cement was light-cured, and μSBS was evaluated with/without thermocycling. The μSBS was analyzed using one-way analysis of variance. The fractured surfaces were examined using stereomicroscopy and scanning electron microscopy (SEM). *Results*. Without thermocycling, μSBS was highest for PC (30.50 MPa ± 3.40), followed by SBU (27.33 MPa ± 2.81) and NC (20.18 MPa ± 2.01) ($P < 0.05$). Thermocycling significantly reduced μSBS in SBU (22.49 MPa ± 4.11) ($P < 0.05$), but not in NC (20.68 MPa ± 4.60) and PC (28.77 MPa ± 3.52) ($P > 0.05$). PC and NC predominantly fractured by cohesive failure within the ceramic and mixed failure, respectively. *Conclusion*. SBU treatment improves μSBS between SA cement and glass ceramics, but to a lower value than PC, and the improvement is eradicated by thermocycling. NC exhibited the lowest μSBS, which remained unchanged after thermocycling.

1. Introduction

Because of its simplified cementation procedure, self-adhesive resin cement (SA cement) is becoming more widely used in dentistry. Specifically, it has been claimed that no additional surface treatment of the tooth surface is required when this new type of resin cement is applied [1, 2]. Therefore, this approach is time-saving and convenient for both the dentist and patient when compared with the conventional cementation procedure, which is time-consuming and technique-sensitive and which requires various products for use in multiple steps [3].

One of the most important properties of dental cement is high, durable bond strength, which is required for successful restorative treatment [3]. In previous studies, SA cements have exhibited clinically acceptable bond strength to both zirconia [4, 5] and metal restorations [6]. However, recent improvements in computer-aided design/computer-aided manufacturing (CAD-CAM) technology and patients' demand for esthetic treatment have led to increased use of

Table 1: Materials used in this study.

Materials (Lot number)	Composition	Manufacturer
IPS Empress CAD (R04751)	Silicon dioxide, aluminium oxide, potassium oxide, sodium oxide, other oxides, pigments (leucite-reinforced glass-ceramic)	Ivoclar Vivadent, Schaan, Liechtenstein
Porcelain Etchant (9.5%) (120006991)	Hydrofluoric acid, polysulfonic acid	Bisco Inc., Schaumburg, IL, USA
Single Bond Universal (539321)	MDP, Bis-GMA, HEMA, decamethylene DAM, ethanol, water, silane treated silica, 2-propenoic acid, -methyl-, reaction products with 1,10-decanediol and phosphorous oxide, copolymer of acrylic and itaconic acid, dimethylaminobenzoate(-4), camphorquinone, (dimethylamino)ethyl methacrylate, methyl ethyl ketone	3M ESPE, St. Paul, MN, USA
RelyX Ceramic Primer (N526043)	Ethyl alcohol, water, methacryloxypropyl-trimethoxysilane	3M ESPE, St. Paul, MN, USA
RelyX Unicem U200 (548681)	*Base*: methacrylate monomers containing acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizer *Catalyst*: methacrylate monomer, alkaline fillers, silanated fillers, initiator components	3M ESPE, St. Paul, MN, USA

ceramic restorations [7–9]. Hence, clinicians frequently use SA cements to bond ceramic restorations.

Silica-based ceramics have highly esthetic properties and have exhibited high bond strength to resin cement. In application, the surfaces of these ceramics are etched with 9.5% hydrofluoric acid to yield a micromechanically retentive surface [10, 11]. Then, for chemical bonding, silane is applied so that covalent and hydrogen bonds are formed [12–14]. This is followed by application of an adhesive [15]. Thus, manufacturers recommend silane pretreatment of silica-based ceramics before SA cements are used [16]. However, universal adhesives have recently been introduced for use in simple and convenient bonding procedures, and manufacturers claim they may be used on silica-based ceramics because they contain silane [16].

Based on the above findings, clinicians often use universal adhesives for silane application prior to application of SA cements. In contrast, some clinicians even assume that SA cements can be applied without any additional treatment of the ceramic surface. Thus, they apply SA cement directly onto an untreated ceramic surface, as in metal and zirconia restoration cementation procedures.

Scientific data regarding the effectiveness of silica-based ceramic surface treatment before cementation using SA cement is not widely available. Therefore, the purpose of this study is to evaluate the microshear bond strength (μSBS) of self-adhesive resin cement on leucite-reinforced glass-ceramic both with and without surface treatment, using pure silane containing primer and universal adhesive before and after thermocycling. The null hypothesis is that no differences in the μSBS between self-adhesive resin cement and leucite-reinforced glass-ceramic exist, regardless of whether pure-saline-containing primer or universal adhesive surface is used.

2. Materials and Methods

2.1. Specimen Preparation. The materials used in this study are shown in Table 1, and the experimental procedure is schematically explained in Figure 1. Using up to 600-grit silicon carbide sandpaper (Rotopol-V, Struers, Ballerup, Denmark), 48 leucite-reinforced glass-ceramic blocks (IPS Empress CAD, Ivoclar Vivadent, Schaan, Liechtenstein) with $14 \times 14\,mm^2$ surface were polished under running water. The ceramic blocks were acid-etched with 9.5% hydrofluoric acid (Porcelain Etchant, Bisco, Schaumburg, IL, USA) for 1 min, rinsed with water, and then cleaned ultrasonically in isopropyl alcohol for 3 min. The specimens were randomly divided into three groups of 16 samples each. These groups were then subjected to one of the following surface treatment methods:

NC: negative control (NC); no additional treatment;

SBU: Single Bond Universal (SBU) adhesive (3M ESPE, St. Paul, MN, USA) was applied for 20 s and the sample was then air-dried for 10 s;

PC: positive control (PC); RelyX Ceramic Primer (3M ESPE), which consists of silane, was applied for 20 s and the sample was then air-dried for 10 s.

A self-adhesive resin cement (RelyX Unicem Self-Adhesive Universal Resin Cement, 3M ESPE) was mixed and polyethylene tubes (Tygon R-3603 tubing, Saint-Gobain Co., Courbevoie, France) were filled and placed on the specimens. The tubes were light-cured for 20 s each from all four directions at $800\,mW/cm^2$ (Elipar Free Light 2, 3M ESPE). After the tubes were removed, a resin cement cylinder of 0.8 mm in diameter and 1 mm in height remained on the ceramic specimen surfaces.

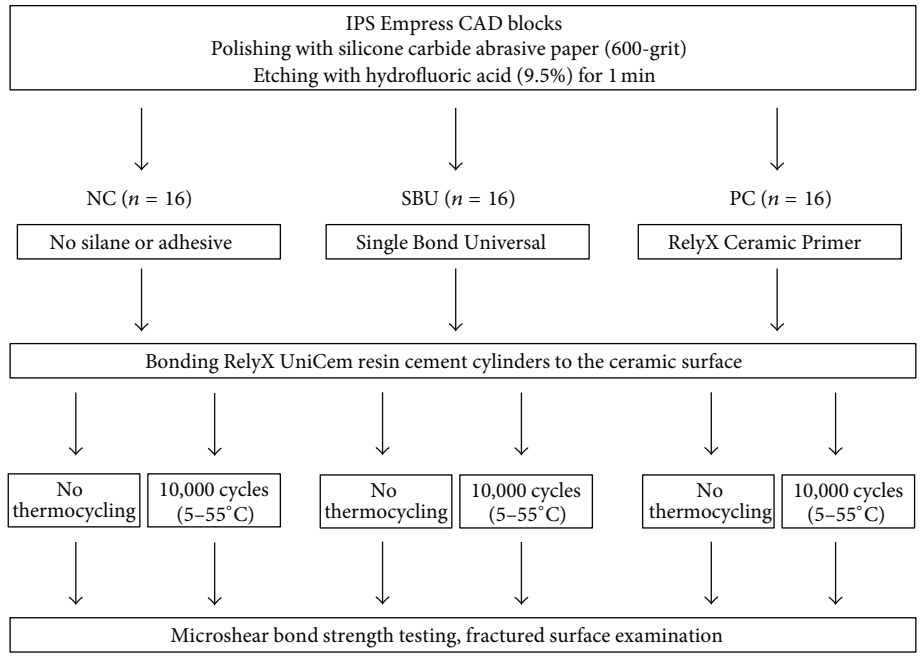

Figure 1: Experimental procedure. NC: negative control; SBU: Single Bond Universal; PC: positive control.

2.2. Microshear Bond Strength Measurements. From the total 16 specimens in each group, eight were subjected to μSBS testing after 24 h storage in distilled water at 37°C, while the remaining specimens were subjected to thermocycling for 10,000 cycles at 5 and 55°C, with 25 s dwell time before testing. Using a universal testing machine (LF Plus, Lloyd Instruments, Fareham, UK) at a crosshead speed of 0.5 mm/min, shear force was applied until failure through a stainless steel orthodontic wire of 0.2 mm in diameter, which was positioned as close as possible to the ceramic/resin bond interface.

The μSBS values were analyzed using one-way analysis of variance, and the Tukey honest significant difference (HSD) was used for post hoc testing of any difference between the various surface treatment groups. The effect of thermocycling on each surface treatment group was analyzed using a paired t-test. (SPSS software version 21, IBM, New York City, NY, USA) at an α level of 0.05.

2.3. Examination of Fractured Surfaces. Following μSBS testing, the fractured specimen surfaces were studied under a stereomicroscope (SZ4045, Olympus Optical Co. Ltd., Tokyo, Japan) to determine the mode of failure at 40x magnification, and the precise failure mode was determined. If the fracture occurred within the ceramic, it was categorized as cohesive failure, and if the fracture occurred within both the ceramic and the resin cement, it was deemed a mixed failure. The fractured specimens were also examined with a scanning electron microscope (SEM) at 200x magnification (S-4700 FESEM, Hitachi, Tokyo, Japan).

3. Results

The mean and standard deviation of the μSBS (MPa) value for each group are shown in Table 2. Before and after thermocycling ($P < 0.05$), the highest μSBS was exhibited by the PC group specimens, followed by the SBU and NC groups. However, the SBU group exhibited a significant reduction in μSBS after thermocycling ($P < 0.05$), while the other two groups were unaffected. After thermocycling, the PC group exhibited the highest μSBS compared to the other two groups ($P < 0.05$), and the μSBS of the SBU group did not differ significantly from the NC group ($P > 0.05$).

Table 2: Mean and standard deviation (SD) of microshear bond strength (in MPa).

Group	Water storage (24 hours)	Thermocycling (10,000 cycles)
NC	20.18 (2.01)^{c}	20.68 (4.60)^{b}
SBU	27.33 (2.81)^{b}	22.49 (4.11)^{b*}
PC	30.50 (3.40)^{a}	28.77 (3.52)^{a}

Within the same column, values with different superscript lower case letters are statistically significantly different (Tukey HSD, $P < 0.05$).
$*$ indicates a significant reduction in bond strength for each group after 10,000 thermocycles (paired t-test, where $P < 0.05$).
NC: negative control; SBU: Single Bond Universal; PC: positive control.

The distributions of the failure modes for each group are shown in Figure 2. It can be seen that the silane PC group predominantly fractured because of cohesive failure within the ceramic both before and after thermocycling, while the NC group exhibited predominantly mixed failure both before and after thermocycling. However, for the SBU group, thermocycling resulted in an increase in mixed failure and a reduction in cohesive failure within the ceramic. Representative SEM images are shown in Figure 3. More resin remnants are seen in the NC group specimen, while larger and deeper cohesive ceramic fractures are seen in the SBU and PC group specimens.

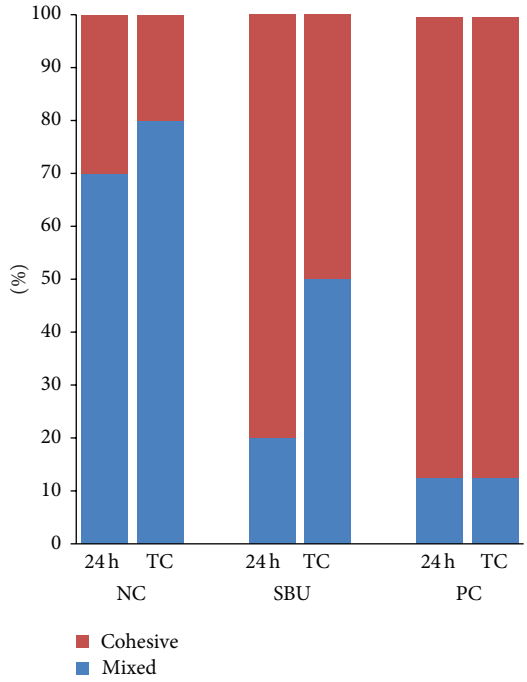

Figure 2: Failure mode distribution after 24 h and 10,000 thermocycles. NC: negative control; SBU: Single Bond Universal; PC: positive control.

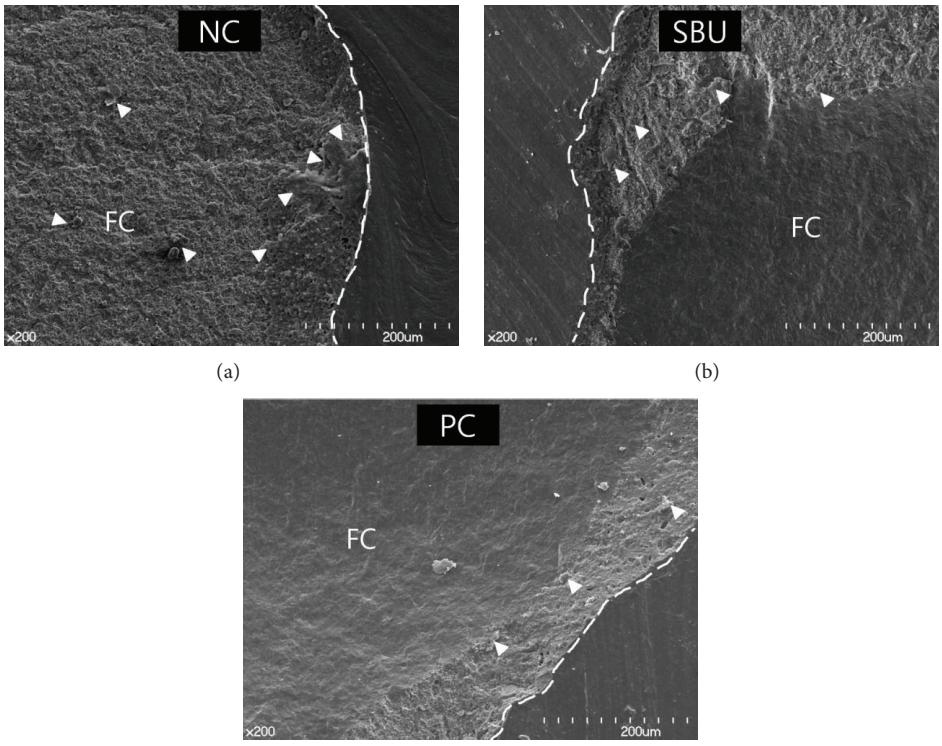

Figure 3: Representative SEM images of fractured ceramic specimens. (a) Facture (dashed lines) occurred within the cemented area in the negative control (NC) group. ((b) and (c)) Fracture occurred (dashed lines) beyond the initial cemented surface in Single Bond Universal (SBU) and pure-saline positive control (PC) groups. The arrowheads show the resin cement remnants on the fractured ceramic (FC). Magnification: 200x.

4. Discussion

This study evaluated the μSBS between a self-adhesive resin cement and leucite-reinforced glass-ceramics before and after thermocycling, according to the following different surface treatment methods: no additional treatment (NC group), universal adhesive application (SBU group), and pure-saline application (PC group).

The highest μSBS performance of the self-adhesive resin cement, RelyX Unicem, to the leucite-reinforced glass-ceramic was measured for the PC group, for which pure silane was applied on the ceramic specimen surfaces. This is consistent with the findings of many previous studies, which recommend silane application to increase the bonding between silica-based ceramics and resin composite [12–14, 17, 18]. This performance is expected, as silane's organofunctional terminal groups bond with resin and its hydroxyl groups bond with silica [18, 19].

However, although SBU also contains saline, the μSBS of the SBU group was significantly lower than that of the PC group. In addition, while the SBU μSBS was markedly higher than that of the NC group before thermocycling, the μSBS value decreased significantly after thermocycling to the NC group value. This indicates that while SBU contains silane it is not as effective as pure silane in improving the bond strength between RelyX Unicem and silica-based ceramics. This finding is consistent with the results of Kalavacharla et al. [16], who reported that a silane pretreatment step significantly improved the bond strength when lithium-disilicate was bonded using SBU. This implies that SBU alone is not as effective as pure saline in improving the ceramic-resin bond. The ineffectiveness of the silane in SBU may be due to the fact that various components such as acidic methacryloyloxydecyl dihydrogen phosphate (MDP) and bisphenol A-glycidyl methacrylate (BisGMA) are mixed in a single bottle with silane [18, 20]. As SBU is in an acidic condition due to the acidic monomer, MDP, the silanol groups in silane may undergo premature self-condensation reactions [18]. In addition, BisGMA may prevent the reaction of silane with the hydroxyl group of the ceramic surface containing silica [20].

In the SBU group, a significant reduction in μSBS occurred after thermocycling, lowering the μSBS value to that of the NC group. After thermocycling, the effect of the SBU is lost, and only the micromechanical retention effect remains in place [15]. Since the initial μSBS before thermocycling was significantly higher than that of the NC group, it may be inferred that the increased bond strength of the SBU group after 24 h storage is attributed to the increased wettability and flow in the irregularly etched ceramic surface, rather than true chemical bonding. The observed reduction in the μSBS of the SBU group after thermocycling was consistent with the fracture mode distribution. The favorable cohesive failure within the ceramic decreased after thermocycling, while the mixed failure increased.

The results also show that although the NC group with no additional surface treatment exhibited the lowest μSBS of approximately 20 MPa this value is clinically acceptable. In fact, 10–13 MPa has been suggested as the minimum clinically acceptable μSBS value for bonded restorations [21]. Moreover, no significant reduction in the μSBS of the NC group occurred after thermocycling. The measured 20 MPa μSBS is most likely due to the micromechanical retention achieved by the partial dissolution of the silica-based ceramic surface after etching with hydrofluoric acid [22, 23]. However, when compared with other groups, the NC group exhibited a greater mixed failure distribution with larger resin cement remnants and less ceramic fracture.

Within the limitations of this study, when self-adhesive resin cement was applied to silica-based ceramics, the μSBS values exhibited by specimens with applied SBU or no surface treatment were significantly lower than those exhibited by specimens undergoing separate saline application. While the use of SBU improved the μSBS value, it was not as effective as a pure-saline treatment, and the μSBS of these specimens decreased significantly to that of the untreated group after thermocycling. However, although the μSBS of the untreated group was the lowest measured value, it was clinically acceptable even after thermocycling.

5. Conclusions

The μSBS of Rely X Unicem U200, a self-adhesive resin cement, to hydrofluoric acid-etched glass-ceramics was significantly improved by the additional application of SBU or silane. Thermocycling significantly reduced the μSBS of SBU treated group while it did not affect the μSBS of untreated group and the saline treated group.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Authors' Contribution

Yoon Lee and Jae-Hoon Kim contributed equally to this work.

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Research Article

Shear Bond Strength of MDP-Containing Self-Adhesive Resin Cement and Y-TZP Ceramics: Effect of Phosphate Monomer-Containing Primers

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Purpose. This study was conducted to evaluate the effects of different phosphate monomer-containing primers on the shear bond strength between yttria-tetragonal zirconia polycrystal (Y-TZP) ceramics and MDP-containing self-adhesive resin cement. *Materials and Methods*. Y-TZP ceramic surfaces were ground flat with #600-grit SiC paper and divided into six groups ($n = 10$). They were treated as follows: untreated (control), Metal/Zirconia Primer, Z-PRIME Plus, air abrasion, Metal/Zirconia Primer with air abrasion, and Z-PRIME Plus with air abrasion. MDP-containing self-adhesive resin cement was applied to the surface-treated Y-TZP specimens. After thermocycling, a shear bond strength test was performed. The surfaces of the Y-TZP specimens were analyzed under a scanning electron microscope. The bond strength values were statistically analyzed using one-way analysis of variance and the Student–Newman–Keuls multiple comparison test ($P < 0.05$). *Results*. The Z-PRIME Plus treatment combined with air abrasion produced the highest bond strength, followed by Z-PRIME Plus application, Metal/Zirconia Primer combined with air abrasion, air abrasion alone, and, lastly, Metal/Zirconia Primer application. The control group yielded the lowest results ($P < 0.05$). *Conclusion*. The application of MDP-containing primer resulted in increased bond strength between Y-TZP ceramics and MDP-containing self-adhesive resin cements.

1. Introduction

Yttria-tetragonal zirconia polycrystal (Y-TZP) that is currently used in restorative dentistry contains over 90% zirconium oxide without silica [1]. Y-TZP provides higher fracture toughness and strength compared to other dental ceramics [2]. Unlike other silica-based ceramics, Y-TZP shows a critical weakness in failing to form reliable and durable bonds due to its resistance to hydrofluoric-acid etching [2].

Predictable cementation is one of the most important factors for achieving clinical success with any restorative material, including Y-TZP [3]. However, the cementation method using mechanical and chemical adhesion remains controversial for Y-TZP, unlike for glass or alumina-based ceramics [4]. Previous studies have proven that air abrasion provides micromechanical bonding and that the use of resin cements that consist of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) strengthens the bonds [5–7].

In order to simplify adhesive cementation procedures, self-adhesive resin cements requiring fewer clinical steps have been developed [8, 9]. Various self-adhesive resin cements consist of phosphate monomers, including MDP, and manufacturers suggest that clinicians apply self-adhesive cements to Y-TZP without additional Y-TZP primer [10]. However, studies [6, 9–11] of the bonding efficiency of phosphate monomers in self-adhesive resin cements to Y-TZP have not

provided much information on the bonding itself. Thus, it is necessary to evaluate the bond strength between the MDP-containing self-adhesive resin cement and Y-TZP ceramics when each new phosphate monomer-containing primer is used.

Therefore, the aim of this study was to evaluate the effects of different phosphate monomer-containing primers on the shear bond strength between MDP-containing self-adhesive resin cements and Y-TZP ceramics. The null hypothesis was that zirconia primer application would not influence the bonding strength to Y-TZP ceramics.

2. Materials and Methods

2.1. Specimen Preparation. Ceramic disks of 4 mm thickness, 19 mm diameter, and 100 mm height were obtained by sectioning Y-TZP blocks, which were composed of 97% zirconium dioxide stabilized with a 3% Yttria-Lava Frame (3M ESPE, St. Paul, MN, USA), using a low-concentration diamond blade (Allied High Tech Productions Inc., CA, USA). Under water cooling, the surfaces of each specimen were ground and polished using silicon carbide abrasives of 600-grit. The Y-TZP ceramic specimens were ultrasonically cleaned for 3 min in distilled water prior to sintering as the manufacturer's instructions. The specimens were then embedded in polyethylene molds of 19 mm inner diameter, 21 mm outer diameter, and 12 mm height. For cement bonding, a single side of each disk was left exposed.

2.2. Surface Treatments and Bonding Procedure. Depending on the surface treatment method and the resin cement used, 60 specimens were randomly assigned to six groups with 10 specimens per group. The specimens were grouped based on the resin cement used and method of surface treatment. The experimental design and materials used in this study are shown in Tables 1 and 2, respectively. The three groups without air-abrasion treatment were treated with either Metal/Zirconia Primer (Ivoclar Vivadent, Schaan, Liechtenstein) or Z-PRIME Plus (BISCO, Schaumburg, USA) or did not undergo any primer treatment. The three remaining groups were treated with 50 μm grain-sized Al_2O_3 particles at a standoff distance of 10 mm and 3.5 bar press for 15 s using air abrasion. The surface was rinsed for 30 s and then air-dried for 30 s after the air abrasion treatment. Either Metal/Zirconia Primer, Z-PRIME Plus treatment, or no primer treatment was performed on the three groups with air abrasion. After being mixed according to the manufacturer's instructions, self-adhesive resin cement (Clearfil SA Luting, Kuraray, Kurashiki, Okayama, Japan) was placed inside a #5 size gel cap (area 16.8 mm^2). Each specimen with a gel cap was light-cured from all four sides at 600 mW/cm^2 for 20 s, using an LED curing light unit (Elipar S10, 3M ESPE, St. Paul, MN, USA). At 23 ± 1°C, all specimens were left to polymerize further for 1 h. The specimens were then stored in 37°C distilled water for 23 h. The specimens were subjected to thermocycling (5–55°C for 5000 cycles). The transfer time between baths was 2 s, with a dwelling time of 30 s at each temperature.

2.3. Bond Strength Test and Surface Analysis. At a 0.5 mm/min crosshead speed, the adhesive interface of each specimen was loaded with a jig of the universal testing machine (LF-plus, AMETEK Inc., Largo, FL, USA) until failure occurred. The failure modes were observed under a stereomicroscope (45x). The resin bonding on the Y-TZP and fractured surfaces was examined using a scanning electron microscope (SEM; S-4700 FESEM, Hitachi, Tokyo, Japan) at 600x magnification and 10 kV accelerating voltage.

2.4. Statistical Analysis. For data analysis, the R programming language (R Foundation for Statistical Computing, Vienna, Austria) [12] was used. The normality of the data and equality of the variance were confirmed. A one-way analysis of variance (ANOVA) and Student–Newman–Keuls multiple comparison test were carried out. The mean difference was considered significant at the level of $P < 0.05$.

3. Results

The means and standard deviations for shear bond strength of all groups are presented in Table 3. One-way ANOVA was used to calculate the statistical significance for the different surface treatments ($P < 0.05$). Air abrasion and the use of Z-PRIME Plus were more effective than the control group treatment. The group treated with Z-PRIME Plus after air abrasion showed the best results.

Figure 1 shows representative SEM images (magnification 600x) for Clearfil SA Luting cement residues on the contact area of the Y-TZP specimens. The failure mode distribution for all samples is shown Figure 2.

4. Discussion

This study investigated the effects of different phosphate monomer-containing primers on the shear bond strength between MDP-containing self-adhesive resin cements and Y-TZP ceramics.

The null hypothesis was rejected for the case of MDP as the effective functional monomer in this experiment, and the untreated Y-TZP surfaces showed the lowest bond strengths. High incidence of adhesive failure was observed, leaving the Y-TZP surfaces free of any luting material remnants, which explains the significantly lower bond strength between the self-adhesive resin cement and the untreated Y-TZP surfaces in the control group. This result may be caused by the poor chemical interaction at the interface between the components, at the interface between the MDP component of the Clearfil SA Luting cement and the hydroxyl groups of the Y-TZP ceramics. Other studies [5, 6, 13, 14] have also reported low bond strength when conventional resin cements are used on untreated Y-TZP ceramic surfaces.

Our results indicate that bond strength can be affected by treatment with primer containing MDP, as well as by conventional methods including air abrasion. The bond strength was influenced greatly by 50 μm sized particle air abrasion regardless of the zirconia primer pretreatment. This result is consistent with previous studies [4–6, 15]. The air abrasion

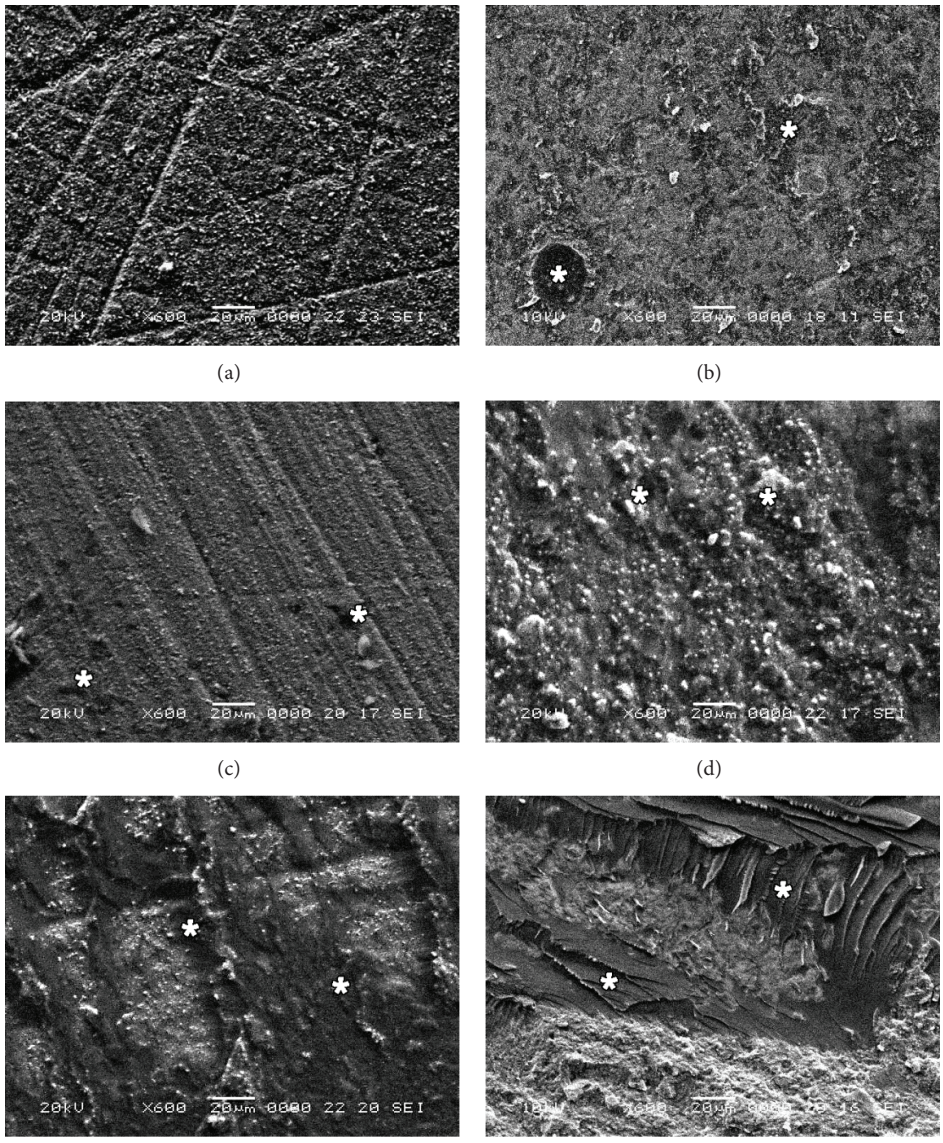

Figure 1: Representative scanning electron microscope images (600x original magnification) of Y-TZP ceramic specimens debonded after cementation with Clearfil SA Luting. (a) Polished Y-TZP; (b) airborne abrasion with 50 μm grain-sized Al_2O_3; (c) Metal/Zirconia Primer, a zirconia primer applied on polished Y-TZP; (d) Metal/Zirconia Primer, a zirconia primer applied on Y-TZP after airborne abrasion; (e) Z-PRIME Plus, a zirconia primer applied on polished Y-TZP; and (f) Z-PRIME Plus, a zirconia primer applied on Y-TZP after airborne abrasion. The regions marked with white stars indicate the remaining resin cements.

Table 1: Experimental design for each surface treatment on Y-TZP specimens in this study.

Y-TZP blocks (ground with 600-grit silicon carbide abrasive paper and sintered)					
↓			↓		
No air abrasion			Air abrasion		
↓			↓		
None	Metal/Zirconia Primer	Z-PRIME Plus	None	Metal/Zirconia Primer	Z-PRIME Plus
↓			↓		
Bonding with Clearfil SA Luting cement (Kuraray, Kurashiki, Okayama, Japan) 5000 thermal cycles between 5 and 55°C, shear bond test ($n = 10$)					

Table 2: Characteristics of experimental materials.

Materials	Brand	Product	Manufacturer
Y-TZP	LAVA	97% zirconium dioxide stabilized with 3% Yttria-Lava frame	3M ESPE, St. Paul, MN, USA
Primer	Z-PRIME Plus	HEMA, BPDM, ethanol, and MDP	Bisco Inc., Schaumburg, IL, USA
	Metal/Zirconia Primer	Dimethacrylate, tertiary butyl alcohol, methyl isobutyl ketone, phosphonic acid acrylate, and benzoyl peroxide containing primer	Ivoclar Vivadent, Schaan, Liechtenstein
Resin cement	Clearfil SA Luting	Bis-GMA, TEGDMA, MDP, barium glass, silica, and sodium fluoride	Kuraray, Kurashiki, Okayama, Japan

HEMA: hydroxyethyl methacrylate, BPDM: biphenyl dimethacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, MPS: 3-methacryloxyprophyltrimethoxy silane, Bis-GMA: bisphenol A-glycidyl methacrylate, and TEGDMA: triethylene glycol dimethacrylate.

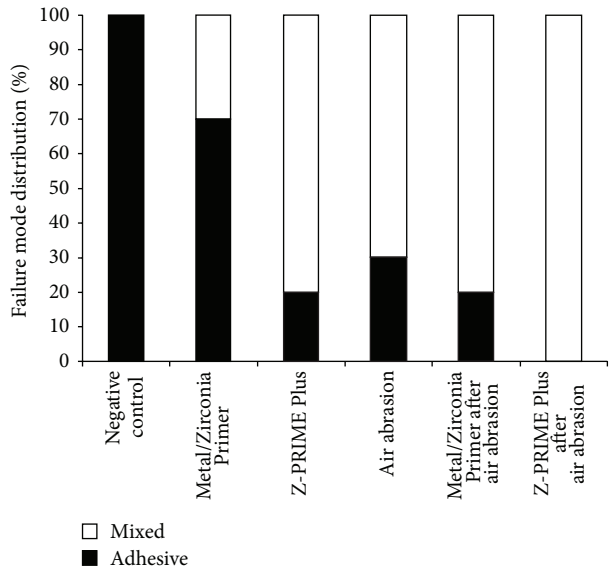

Figure 2: Distribution of failure modes after 10,000 thermocycles.

Table 3: Means and standard deviations of the shear bond strength (MPa) of the samples with Clearfil SA Luting cement and different surface treatments on Y-TZP ($n = 10$).

Priming conditions	Surface conditions	
	No air abrasion (polished)	Air abrasion
None	4.61 (1.13)^{A}	9.84 (2.36)^{B}
Metal/Zirconia Primer	5.41 (1.66)^{A}	10.00 (2.29)^{B}
Z-PRIME Plus	10.72 (1.70)^{B}	15.23 (1.97)^{C}

Different superscripts indicate a statistical difference ($P < 0.05$), and identical superscripts indicate no statistical difference in the designated group after the Student–Newman–Keuls multiple comparison test.

method is thought to assist in the progress of resin cement flow into microretentions due to increased roughness and surface energy, which create micromechanical interlocking between the resin cements and Y-TZP [6, 16]. Moreover, air abrasion may generate hydroxyl groups on the Y-TZP surfaces, facilitating the chemical reaction with phosphate monomers [17, 18].

The results of this study show that the MDP containing Z-PRIME Plus promotes durable bonding to Y-TZP. Even though the self-adhesive resin cement included MDP, its functional monomer properties in terms of the amount and flow seemed insufficient to increase the Y-TZP adhesion ability without any pretreatment [6, 19]. Therefore, MDP functional monomers need to be applied to Y-TZP surfaces even if the self-adhesive resin cement contains such monomers. This interpretation is consistent with the results of previous studies [5, 6, 20, 21]. The phosphate ester group of the adhesive monomers and zirconia oxides chemically creates direct bonds [7, 22, 23]. MDP has bifunctional ends that consist of long organic hydrophobic chain molecules. Hydrophilic phosphate ester groups at one end bond strongly to Y-TZP, and vinyl groups react with the monomers of the resin cement at the other end [20, 24].

In the present study, only the MDP-based product, Z-PRIME Plus, had a significantly higher bond strength than did the phosphonic acid-based Metal/Zirconia Primer. The reason is probably that MDP was more effective than phosphonic acid acrylate in Y-TZP surface treatment, though the same phosphate monomer was included in both products [22]. In other studies, MDP-based primers were proven to have higher bond strengths on Y-TZP compared to other primers [7, 21, 22, 25].

It is notable that adhesive failure can be observed in the control group images, whereas the specimens treated with air abrasion, Metal/Zirconia Primer, Z-PRIME Plus, and air abrasion in combination with Z-PRIME Plus presented mixed failures with resin cement. The resin cement remnants can be seen to a relative degree (Figures 1(a)–1(f)). Figure 1(f) shows a unique ridged appearance, with more resin cement residues in the group with a combination of air abrasion and Z-PRIME Plus application.

The highest bond strength was achieved through the combination of air-abrasion treatment and MDP-based product application. This result may be due to the air-abrasion treatment enhancing the surface wettability and the MDP-containing primer increasing the bond strength, thus improving the chemical affinity. In this group, all specimens indicated mixed fracture patterns, which may be due to the combined effects of the increased contact area with the Y-TZP ceramic surface and the improved chemical interaction

with the MDP monomer in Z-PRIME Plus [6, 7]. The combination of MDP-containing primer application and air-abrasion treatment is recommended to achieve strong and durable bonds to Y-TZP using self-adhesive resin cements containing MDP monomers.

5. Conclusion

Within the limitations of this study, the following can be concluded.

(1) The application of MDP-containing self-adhesive resin cement without pretreatment was not sufficient to improve the bond strength to an untreated Y-TZP surface.

(2) MDP-containing primer application seems to be a reliable method for increasing the bond strength between Y-TZP ceramics and MDP-containing self-adhesive resin cements.

Conflict of Interests

The authors declare that they have no conflict of interests.

Acknowledgment

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Research Article

Direct Tensile Strength and Characteristics of Dentin Restored with All-Ceramic, Resin-Composite, and Cast Metal Prostheses Cemented with Resin Adhesives

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A dentin-cement-prosthesis complex restored with either all-porcelain, cured resin-composite, or cast base metal alloy and cemented with either of the different resin cements was trimmed into a mini-dumbbell shape for tensile testing. The fractured surfaces and characterization of the dentin-cement interface of bonded specimens were investigated using a Scanning Electron Microscope. A significantly higher tensile strength of all-porcelain (12.5 ± 2.2 MPa) than that of cast metal (9.2 ± 3.5 MPa) restorations was revealed with cohesive failure in the cement and failure at the prosthesis-cement interface in Super-Bond C&B group. No significant difference in tensile strength was found among the types of restorations using the other three cements with adhesive failure on the dentin side and cohesive failure in the cured resin. SEM micrographs demonstrated the consistent hybridized dentin in Super-Bond C&B specimens that could resist degradation when immersed in hydrochloric acid followed by NaOCl solutions whereas a detached and degraded interfacial layer was found for the other cements. The results suggest that when complete hybridization of resin into dentin occurs tensile strength at the dentin-cement is higher than at the cement-prosthesis interfaces. The impermeable hybridized dentin can protect the underlying dentin and pulp from acid demineralization, even if detachment of the prosthesis has occurred.

1. Introduction

Dentin is more susceptible to degradation by acid and wear when exposed to the acidic oral cavity compared with enamel due to the smaller crystal size of hydroxyapatite, the presence of tubules, and its greater organic content. This natural phenomenon has not been well recognized in our community. Good retention has been believed to be one of the major requirements in achieving long-term success of restorations and fixed prostheses, as the explanation for the detachment of restorations was due to poor retention. Therefore researchers have attempted to increase retention either by removal of sound tooth substance to create mechanical interlocking for abutments or by increasing the strength of the cement bond to both tooth and restorations [1–4]. However the most common failure found in either direct or indirect restorations and fixed partial dentures is secondary caries [4–8], especially located at the gingival margins [6].

Adhesive resin cements have been increasingly used as they provided better retention than acid-base cements [9–11]. Mostly the retention provided by acid-base cements has been evaluated in terms of crown retention by using the pulling force required to remove the crown [11–13]. With this technique it is difficult to control the size of the interfacial area and the stress distribution, mainly shearing stress, through the tooth-cement-prosthesis junctions. The method recommended to measure the bond strength to tooth substrates in the ISO/TC 106 Dentistry Standard is not suitable to identify defects created in the subsurface dentin, as the cross-sectional area of the tooth substrate is wider than that of the bonded interface [10, 14]. Tensile strength measurement using dumbbell specimens is a widely accepted methodology

in materials science and engineering to detect the defect in materials [15]. A mini-dumbbell shaped specimen ($3 \times 2 \times 1.2$ mm rod forming the resin-dentin junction) modified to obtain reliable data for the full dentin depth was proposed to find defects or the weakest part in the restored dentin [10, 16–19]. A reliable tensile strength of resin bonded to dentin depends on the quality of the hybridized dentin or hybrid layer [10]. It has been reported that specimens with initial high tensile bond strength as measured using a microtensile test degraded after 1–3 years [20, 21]. Tensile testing using mini-dumbbell specimens and a chemical challenge using hydrochloric acid (HCl) and sodium hypochlorite (NaOCl) solutions have been shown to be effective in detecting defects such as demineralized dentin and smears in the restored dentin [16–19]. Adhesive failure and cohesive failure in the remaining demineralized dentin suggest that the resin infiltration into the conditioned dentin was not complete. The dual immersion in HCl and NaOCl solutions removes the mineral phase and the collagen fibers that have not been enveloped by impregnated resin. Degradation of dentin-resin interfacial layer after chemical challenge confirms the existence of demineralized dentin. These defects can lead to leakage under restorations bonded to dentin [22, 23]. Demineralized dentin permeable to dyes, whether formed by acid-base cements before setting or formed by incomplete infiltration of resin [22, 23], permits diffusion of acid which subsequently may result in tooth hypersensitivity, short-term detachment of restorations, secondary caries, and pulpal pathology [4–8, 24–26]. Microleakage-free restorations can be achieved on restored dentin when complete hybridization of dentin occurs, because an impermeable hybridized dentin layer is formed [22, 23, 27].

Although bonding resin to enamel and dentin protected by a barrier impermeable to acids may maintain the retention for restorations, preparation of impermeable hybridized dentin is not as simple as for enamel as dentin contains more organic substances and water which can interfere with the diffusion of monomers [17, 20, 21]. Numerous adhesive resin systems, both for direct bonding and for cementation, have been developed and released into the market over the last 40 years. The adverse effect of phosphoric acid in removing a weak smear layer must be carefully studied in order to understand dentin substrates suitable for clinically reliable bonding. The acid dissolves water soluble hydrophilic glycosaminoglycans (GAGs), immobilized in intact dentin with hydroxyapatite, into the demineralized dentin. This must be the reason why demineralized dentin is so hydrophilic and difficult to dehydrate [28–30].

Many methods have been introduced to ensure sufficient tensile strength between resin cements and prosthetic materials. These include preparations for mechanical retention by grinding with burs, air abrasion with aluminum oxide, and/or etching with either acidic solutions or electrolysis [31–33]. The use of silane coupling agents was reported to increase the bond strength of resin to porcelain as well as to cured resin-composite, as it promoted chemical adhesion [33–35]. Surface treatment using an alloy primer has also been reported to significantly increase the tensile bond strength both for base metal and noble alloys [36].

The authors hypothesized that direct tensile strength of dentin-cement-prosthesis restoration using mini-dumbbell shaped specimens [10, 16–19] and the characteristics of the dentin-cement interface may indicate that the high strength of the restorative materials is not as important as the protection of exposed basic dentin with an impermeable barrier resistant to acid demineralization for long-term function. In other words protecting exposed dentin with a barrier impermeable to acid for demineralization is critical for the long-term success of restored dentin with either brittle tooth-colored materials or stronger cast metal.

The objective of this study was to detect the weakest area in the restored dentin-cement-prosthesis complex when restored with cast metal, cured resin-composite, and all-porcelain cemented to dentin with different resin cements, using a direct tensile strength test and the characterization of dentin-cement interface.

2. Materials and Methods

2.1. Part I: Direct Tensile Strength Test

2.1.1. Preparation of Dentin Slabs. Extracted human molars that had been removed and frozen for less than three months were root-embedded in acrylic blocks (Taklon Co., Milan, Italy). The teeth required extraction and the patients gave written informed consent for their use in this project. A 4.0 mm occlusal portion was cut off horizontally and vertically sectioned to prepare 2.0 mm dentin slabs (Figure 1) using a sectioning machine (Isomet, Struers Co., Copenhagen, Denmark). Twelve dentin slabs for each group were cut. The cross-sectioned surfaces of the slabs were prepared using a diamond cylinder bur (GC International Co., Aichi, Japan) for cementing with the prostheses. A mini-dumbbell template was used to outline the bonding interface area on each dentin slab for tensile testing (Figure 1(a)).

2.1.2. Fabrication of Prostheses. A standardized mini-dumbbell plastic mold was prepared. Mini-dumbbell patterns using self-cured acrylic resin (Taklon Co., Milan, Italy) with a 2.0×3.0 mm cross-section on the center [17] were prepared using the standardized mold. Acrylic resin patterns were sprued and invested using PowerCast investment (Whip Mix Co., Kentucky, USA) with a powder/liquid ratio of 100 g/23 mL. The pattern mold was cast into base metal alloys (Ni-Cr alloy, Wiron 99, Bego Co., Bremen, Germany) using an electronic induction casting machine (Degutron, Degussa, Germany) at the casting temperature of approximately 1450°C. Each casting was quenched, divested, and finished with stones and rubber finishing burs. A carborundum disc (Jota Co., Ruthi, Switzerland) was used to cut through the center of the mini-dumbbell to prepare two half mini-dumbbells (Figure 1(b)).

Resin-composite (Filtek Z 250, 3M ESPE, St. Paul, MN, USA) mini-dumbbell specimens were prepared using the standardized plastic mold. The mold was filled with resin-composite with a bulk placement and light-cured for 60 s on each side using a light curing machine (3M Elipar Trilight, St. Paul, MN, USA). Standardized mini-dumbbell investment molds were prepared to make all-porcelain specimens using

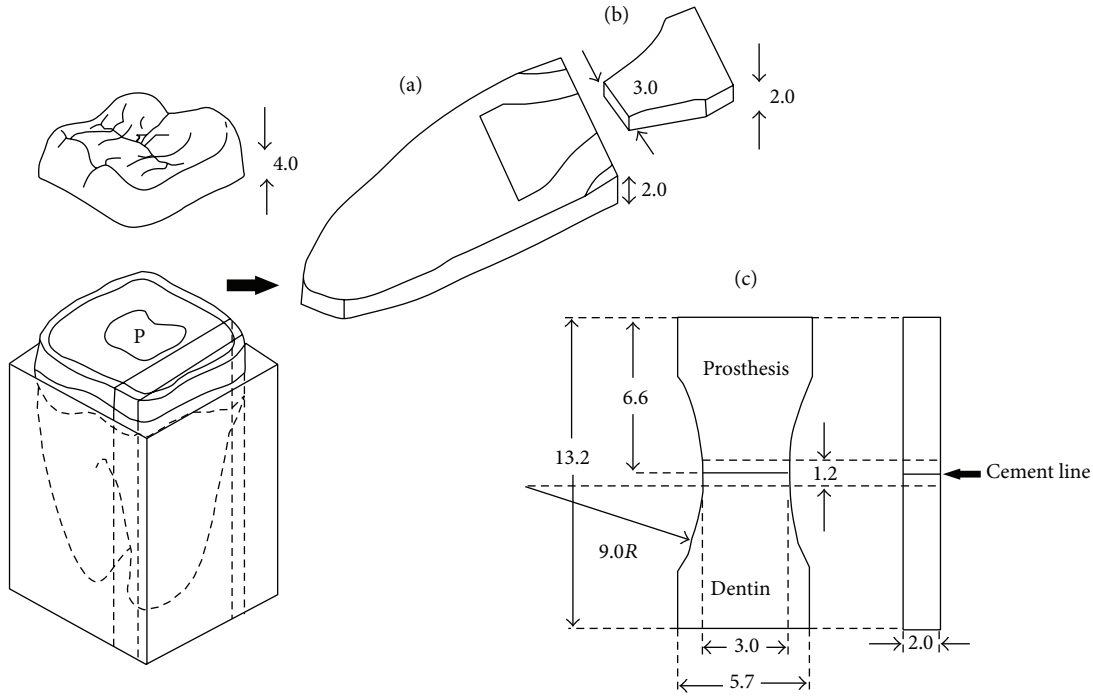


Figure 1: Schematics (in mm) of a dentin slab (a), restored with half mini-dumbbell prosthesis (b), to prepare a mini-dumbbell specimen (c). P: pulp chamber.

Table 1: The firing cycle of all-porcelain mini-dumbbell specimens.

Porcelain	Predrying		Heating-up		End firing		Vacuum
build-up	(°C)	(min)	(min)	(°C/min)	(°C)	(min)	(min)
1st firing	600	6.00	6.00	55	930	1.00	6.00
2nd firing	600	6.00	6.00	53	920	1.00	6.00
3rd firing	600	6.00	6.00	51	910	1.00	6.00

the dentin powder (Vita Omega, VITA Zahnfabrik, Bad Säckingen, Germany). The firing cycle of porcelain furnace (Vita Vacumat, Bad Säckingen, Germany) recommended by a manufacturer was scheduled (Table 1). Three layers of porcelain build-up were applied into the mold. After being finished with the stones and rubber finishing burs all resin-composite and porcelain mini-dumbbell specimens were sectioned with diamond discs (Jota Co., Ruthi, Switzerland) to prepare two half mini-dumbbells for each specimen (Figure 1(b)).

2.1.3. Mini-Dumbbell Preparation of Restored Dentin. All the twelve half mini-dumbbell shape prostheses for each group were air-blasted with 50 μm alumina for 10 s on the surface areas to be cemented. A silane coupling agent specific for each cement system was applied on resin-composite and porcelain surfaces before fixing on dentin slabs with resin cements. The bonding procedures followed the manufacturer's recommendation as shown in Table 2. The same operator cemented all the restorations using finger force. After light curing or initial autocuring of resin cements, each bonded sample was trimmed into a mini-dumbbell shaped specimen with the cross-section of 2.0 × 3.0 mm and 1.2 mm rod high (Figure 1(c)), using a diamond cylinder bur and high speed handpiece (KaVo Dental Co., NC, USA) with air-water spray.

2.1.4. Preparation for Tensile Testing. After storing in water at 37°C for 24 h, all mini-dumbbell bonded specimens in all groups were affixed to the poly(methyl methacrylate) (PMMA) jigs using Super-Bond C&B (Sun Medical, Shiga, Japan) and self-cured acrylic resin for tensile testing. With a cross-head speed of 1.0 mm/min, a tensile force was applied using a universal testing machine (Lloyd Co., Hampshire, UK) on the assembled specimen (Figure 2). The cross-sectional areas of fractured specimens were remeasured using a digital micrometer (Mitutoyo 293, Tokyo, Japan). The tensile strength data were calculated in MPa and statistically analyzed using an analysis of variance (ANOVA) and Scheffe's test. Fracture surfaces of specimens in each group were investigated using a light microscope (Nikon, Tokyo, Japan) and a Scanning Electron Microscope (SEM, JSM-5008LV, JEOL, Tokyo, Japan) to categorize the mode of failures.

2.2. Part II: Characterization of the Dentin-Cement Interfacial Layer. Three dentin slabs similarly prepared as mentioned in

Table 2: Cementing procedures.

Procedures	Super-Bond C&B	PanaviaF	Variolink II	Single-Bond + RelyX
Primer for all-porcelain and cured composite	Porcelain liner M Liquid A : B (1 drop : 1 drop) Mixed and applied with brush	Clearfil porcelain bond activator Applied with brush	Monobond S Applied for 60 s and gently air-dried	3 M Scotchbond ceramic primer Applied and gently air-dried
Conditioner	10-3	ED primer	37% phosphoric acid	32% phosphoric acid
(i) Application on dentin surface	Applied 10 s, rinsed off 10 s, and air-dried 10 s	Applied 60 s and air-dried 2-3 s	Applied 10 s, rinsed off 15 s, and air-dried 2-3 s	Applied 15 s, rinsed off 10 s, blot-dried, and kept moist
Bonding agents/resin cements (i) Manipulations	4-META/MMA : TBB = 4 drops : 1 drop Mixed and applied on conditioned dentin and prosthesis using brush-dip technique with PMMA powder, cemented, and self-cured	Base : catalyst (paste) = 1 : 1 Hand mixed, cemented, Oxyguard coated, and light-cured 20 s (each side)	Applied Syntac primer 15 s, gently air-dried 2-3 s, applied Syntac adhesive 10 s, gently air-dried and light-cured for 20 s, applied Heliobond on both dentin and prosthesis, gently air-dried Hand mixed base and catalyst paste (1 : 1), applied on the prosthesis, cemented, and light-cured 40 s (each side)	Applied Single-Bond, gently air-dried 2–5 s (twice) on prepared dentin (light-cured 10 s) and prosthesis Hand mixed base and catalyst paste (1 : 1), applied on the prosthesis, cemented, and light-cured 40 s (each side)

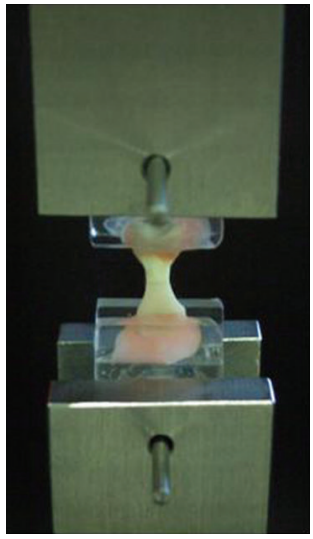


Figure 2: Direct tensile testing of restored dentin with porcelain using a universal testing machine.

part I for each cement group were restored with three veneers of light-cured resin-composite (2 × 4 × 1 mm) using each resin cement to characterize the dentin-cement interface. The manipulation procedures followed the manufacturer's recommendation as previously described (Table 2). Without epoxy embedding, two cross-sectional specimens of 1 mm thickness were prepared from each restored dentin specimen using a diamond disc and low-speed handpiece. The prepared surface was abraded on 600-grit and then 1,200-grit abrasive papers and then polished with 0.05 μm alumina paste. Specimens were ultrasonically cleaned for 30 min and air-dried. One was immersed in 6 mol/L HCl for 30 s followed by 1% NaOCl for 60 min. All the polished and chemically treated specimens were desiccated and gold sputtered. The thickness of the dentin-resin interfacial layer on the chemically treated and the originally polished specimens was compared using SEM micrographs at ×500 and ×2000 magnification.

3. Results

The mean tensile strength ± standard deviation (SD), failure mode, and the amount of detached specimens during trimming of each group are shown in Table 3. A two-way ANOVA found significant differences in tensile strength among the types of cements and prostheses. A Scheffe test at $P < 0.05$ revealed significant differences between groups of cement and prosthesis types (Table 3). The highest tensile strength of restored dentin was found when using Super-Bond C&B cement with a cohesive failure in the cured resin and adhesive failure on the prosthesis side interface (Figure 3(a)). No significant difference in tensile strength between PanaviaF (Kuraray Medical Inc., Okayama, Japan) and Variolink II (Ivoclar Vivadent, Liechtenstein) cements was found. The failure mode of PanaviaF specimens mostly occurred with mixed failure of adhesive on the dentin side interface and cohesive failure in the hybridized smear layer and resin (Figure 3(b)), while adhesive failure on the demineralized dentin interface was mostly found in Single-Bond + RelyX (3M Dental Products, St. Paul, USA) and Variolink II specimens (Figures 3(c) and 3(d)). Dentin restored with Single-Bond + RelyX showed the lowest tensile strength and

Table 3: Mean tensile strength ± SD, failure mode in restored dentin, and numbers of detached specimens during trimming of each group.

Groups ($n = 12$)		Mean ± SD (MPa)	Failure mode in restored dentin (numbers of specimens)	Numbers of detached specimens
Cements	Prostheses			
PanaviaF[a]	Metal	4.3 ± 1.7	A/D (2), A/D + Hs + R (7), A/P + Hs + R (2)	1
	Composite	5.7 ± 4.2	A/D (2), A/D + Hs + R (7), A/P + Hs + R (3)	—
	Porcelain	6.0 ± 3.0	A/D + Hs + R (5), Hs + R (3), A/P + Hs + R (4)	—
Super-Bond[b]	Metal*	9.2 ± 3.5	A/P + R (12)	—
	Composite	11.7 ± 2.1	R (2), A/P + R (10)	—
	Porcelain*	12.5 ± 2.2	R (2), A/P + R (10)	—
Single-Bond[c]	Metal	2.2 ± 1.2	A/D (8), A/P (2)	2
	Composite	1.3 ± 1.1	A/D (9)	3
	Porcelain	1.5 ± 1.0	A/D (9)	3
Variolink II[a]	Metal	2.0 ± 1.3	A/D (10)	2
	Composite	3.9 ± 4.0	A/D (10)	2
	Porcelain	5.0 ± 3.6	A/D (9), A/P + R (2)	1

[a,b,c]Significant differences in tensile strength between cements indicated by the different superscripts ($P < 0.05$).

*Differences in tensile strength between prostheses are significant.

A/D = adhesive failure at dentin side interface, A/P = adhesive failure at prosthesis side interface, R = cohesive failure in resin, Hs = cohesive failure in hybridized smear, and + = mixed failure.

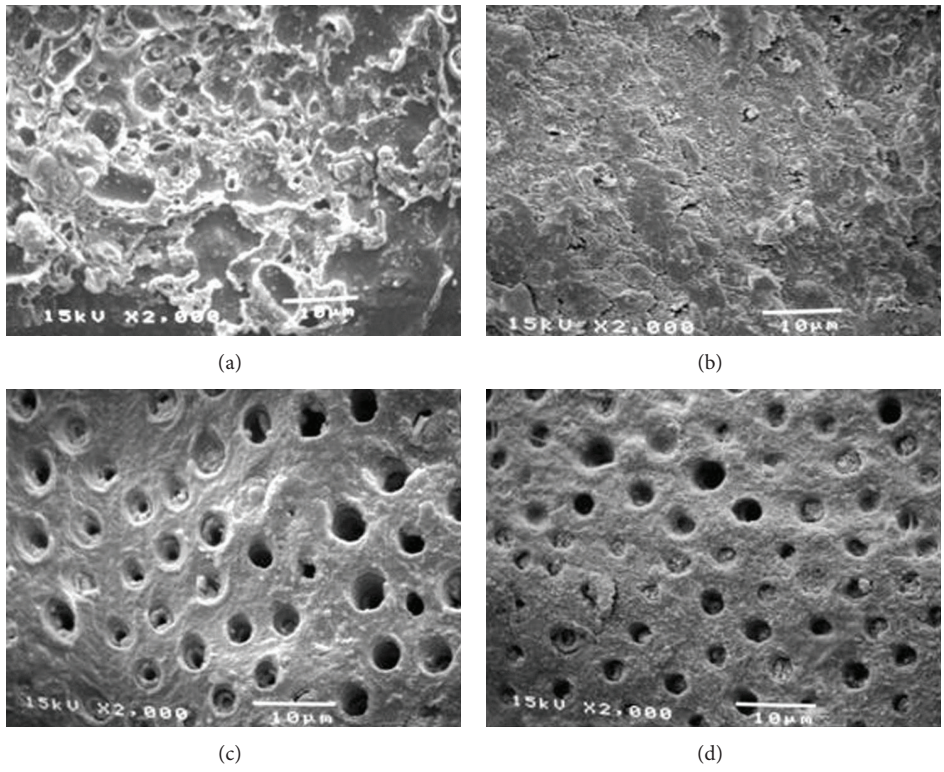
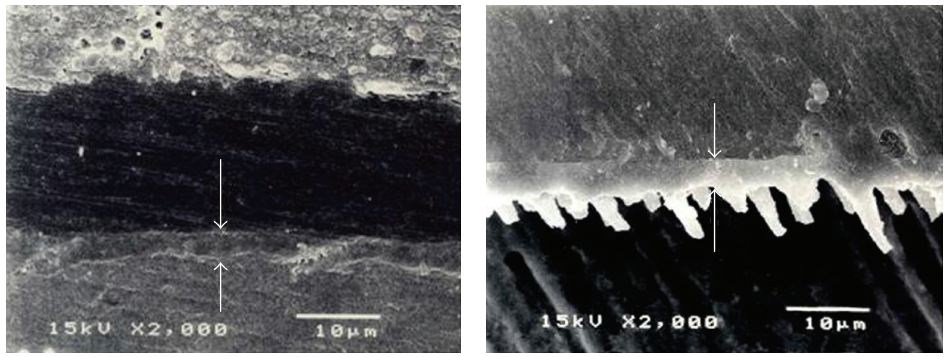


Figure 3: Fracture surface of restored dentin demonstrating (a) cohesive failure in resin and at prosthesis side interface in Super-Bond C&B specimen; (b) cohesive failure in hybridized smears and resin in PanaviaF specimen; (c) adhesive failure at demineralized dentin interface in Single-Bond + RelyX and Variolink II (d) specimens.

greatest number of detached specimens during dumbbell preparation with adhesive failure on the dentin side interface (Figure 3(c)).

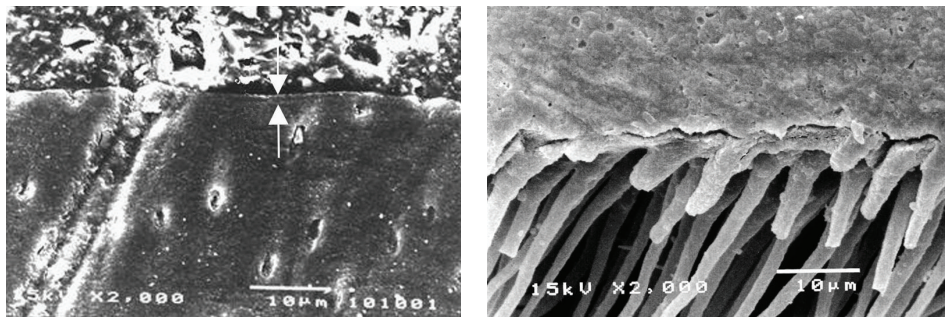
None of the specimens was detached while trimming and no adhesive failure on the dentin side interface was found in the Super-Bond C&B groups (Table 3). Most failures occurred on the prosthesis side interface, with significantly higher tensile strength for all-porcelain compared with those of cast metal restorations being revealed. No significant difference between types of prosthesis was found in the other three cement groups.

The dentin-cement interfacial layer of Super-Bond C&B specimens was consistent and continuous for 3-4 μm both before and after chemical modification using HCl and NaOCl



(a) (b)

Figure 4: Characteristics of dentin-cement interfacial layer before (a) and after (b) HCl and NaOCl modifications demonstrated the consistent and continuous hybridized dentin (arrowed) in Super-Bond C&B specimen.



(a) (b)

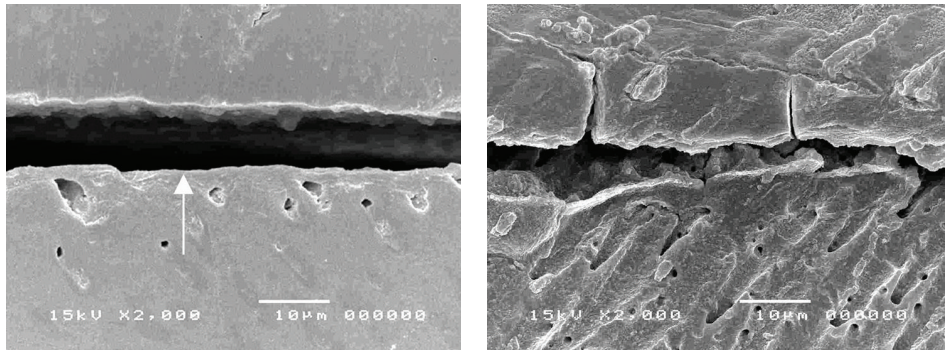
Figure 5: Characteristics of dentin-cement interfacial layer in PanaviaF specimen demonstrating (a) the thin layer of polished specimen (arrowed) and (b) the degraded and detached layer after HCl and NaOCl modifications.

(Figure 4), whereas that in PanaviaF specimens was detached and degraded (Figure 5). In Variolink II and Single-Bond + RelyX specimens, where dentin was demineralized by phosphoric acid and kept moist, the interfacial layer was detached on the dentin side interface and was degraded after the chemical modification (Figures 6 and 7).

4. Discussion

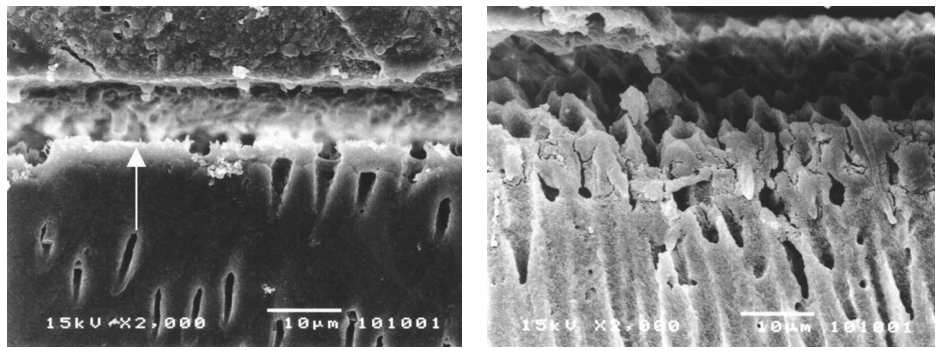
A significantly higher tensile strength of restored dentin was found for Super-Bond C&B specimens with cohesive failure in cement and failure on the prosthesis side interfaces (Figure 3(a)). The hybridized dentin before and after chemical immersion was consistent and continually attached (Figure 4). These results suggested that dentin conditioned with 10% citric acid and 3% ferric chloride (10-3) solution, rinsed, and gently air-dried could provide permeability for complete infiltration of 4-methacryloyloxyethyl trimellitate anhydride in methyl methacrylate initiated by tri-*n*-butyl borane (4-META/MMA-TBB) in the presence of PMMA resin to form an impermeable hybridized dentin layer which could resist the acid and NaOCl challenge (akin to caries formation). This means that the hydroxyapatite was well encapsulated and protected with impregnated impermeable copolymers against acid demineralization and exposed collagen was also well enveloped and protected against NaOCl degradation. The well encapsulated hydroxyapatite crystals in the hybrid layer contribute to the longevity of bonding [37, 38]. The hybridization of dentin substrate with the resin gave a higher tensile strength than did the interface of cured cement-restorative materials irrespective of whether being cast metal, cured resin-composite, or porcelain. Nevertheless, the tensile strength was sufficient to resist stress during the mini-dumbbell shape preparation as none of these restored dentin specimens was detached prior to tensile testing.

The significantly lower tensile strength of dentin restored with PanaviaF, Variolink II, and Single-Bond + RelyX specimens resulted from adhesive failure on the dentin side of the interface. This clearly suggested that good retention to restored dentin did not depend on the strength of resin cement but was due to the complete hybridization of the resin into dentin which is the substrate [27]. Adhesive failure on the dentin interface suggested that phosphoric acid conditioned dentin rinsed and kept moist had less permeability for impregnation by monomers; thus complete hybridization of resin into the conditioned dentin did not occur. This adhesive failure must be due to the weak layer of demineralized dentin in the restored dentin. It is important to discover how to



(a) (b)

Figure 6: Characteristics of dentin-cement interfacial layer in Single-Bond + RelyX specimen demonstrating (a) the detachment at dentin side interface (arrowed) of polished specimen which was degraded (b) after HCl and NaOCl modifications.



(a) (b)

Figure 7: Characteristics of dentin-cement interfacial layer in Variolink II specimen demonstrating (a) the detachment at dentin side interface (arrowed) of polished specimen which was degraded (b) after HCl and NaOCl modifications.

eliminate this weak layer from the restored dentin. Thus the influence of GAGs dissolved in demineralized dentin by these etching agents used to remove the weak smear layer and the effect of ferric ions to aggregate GAGs to improve bonding to dentin need further study [28–30].

Cohesive failure in hybridized smear layers was also confirmed in the PanaviaF group (Figure 3(b)) as this self-etching cement bonded through the smear layers. Observation of the dentin-cement interfacial layer using SEM showed a degraded and detached layer after chemical modification (Figure 5). These results suggested that the smear layer could reduce the amount of monomer infiltration into underlying dentin and also contribute to the weakness of hybridized smear layer [10, 39]. The failure mode of Single-Bond + RelyX specimens was mostly adhesive failure on the demineralized dentin interface as for the Variolink II specimens (Figures 3(c) and 3(d)). The dentin-cement interfacial layer of these two groups demonstrated detachment of polished specimen (Figures 6(a) and 7(a)) which was degraded after chemical challenge (Figures 6(b) and 7(b)). This confirmed that monomer infiltration was difficult and could not fill the phosphoric acid demineralized dentin [29]; therefore any exposed collagen which was not enveloped in Variolink II and Single-Bond + RelyX groups was liable to degradation with NaOCl as shown in Figures 6 and 7, respectively. Thus it must be difficult for these adhesive resins to inhibit the detachment of restorations when being under stress.

The number of detached specimens during the preparation of mini-dumbbell shape found in three cement groups (Table 3) was 8, 5, and 1 out of 36 in Single-Bond + RelyX (22%), Variolink II (14%), and PanaviaF (3%), respectively. SEM micrographs of the detached surfaces showed adhesive failure on the dentin side interface. The higher percentage of detachment of restorations was probably due to restored dentin in the presence of demineralized dentin, the weakness of the dentin itself, and not because of the weakness of dental materials. These results also suggested that these cements must be carefully applied in the clinic as any demineralized dentin introduced during treatment could later be penetrated by acid produced in the mouth. The demineralized dentin resulting from the incomplete infiltration of monomers into conditioned dentin leads to leakage [22, 40, 41], degradation [38, 40], and detachment [10, 42] at this area. Thus prostheses or restorations cemented with these resins may not provide leakage-free restorations, and tooth hypersensitivity and restoration detachment could be expected in the short

term, where additional increased retention of restorations or prostheses has been gained from tooth preparation geometry, or bonding to enamel, secondary caries, or pulpal pathology could be the subsequent results. This suggests that retention-based dentistry may not be the solution for long-term function of restored teeth.

Hybridized dentin that resisted the HCl and NaOCl challenge suggested that it could protect prepared weak dentin against the demineralization with lactic acid under oral condition and thus inhibit recurrent caries formation. Dentin restored using Super-Bond C&B can provide not only a microleakage-free interface [22, 23, 27, 40, 41], but also a reliable and higher tensile strength on the dentin-resin cement interface than that of the cement-restorative material interface used in this study. Significant difference in the tensile strength of the cement-restoration interface was found between porcelain and cast metal cemented with Super-Bond C&B resin. This suggested that the roughened surface on the cast metal could provide retention to resin cements able to resist a tensile stress similar to that of cured resin-composite even with a silane application. However, the greater amount of silica coupled with silane in all-porcelain compared with cured resin-composite could create a higher resistance to tensile stress than that of cast metal. With the compressive strength higher than the bite force of posterior molars [43] all-porcelain restoration coupled with this complete hybrid layer can provide the retention, strength, and stability for long-term function of both anterior and posterior teeth.

No significant difference in tensile strength between types of restorative materials was found in the Single-Bond + RelyX, Variolink II, and PanaviaF groups (Table 3). As most failure occurred on the dentin side of the interface of these cements, this suggested that the strength of resin cements and/or prostheses and the marginal fit of restorations/prostheses had no influence on the protection of restored dentin when not coupled with a complete hybrid layer. On the contrary, a barrier impermeable to acids can protect weak exposed dentin from acid demineralization, and this must contribute to the reliability of the dental treatment. Should the restoration or prosthesis detach or fracture, the remaining tooth substance will still be protected from degradation in the oral environment and can be restored again with minimal or no further tooth reduction.

The direct tensile test of the restored dentin-cement-prosthesis complex with mini-dumbbell shape modified from dentin-cement-PMMA rod specimens [10, 16–19] to simulate clinical treatment can detect the weakest area in restored dentin. Pretest failure, adhesive failure at the dentin side interface and smears, and defects in the dentin-resin interface suggest that monomer impregnation of the resin adhesives into the conditioned dentin was not complete. The chemically resistant hybrid layer is more reliable in preventing caries related to restoration. The mini-dumbbell tensile test and the characterizing of dentin-resin interface can be the basic test method required for predicting clinical performance as it can detect any defects left in the restored dentin in 24 hours while a detachment of the resin-dentin interface was caused by gradual hydrolysis of the existing demineralized dentin appearing after soaking in water for 1–5 years [38].

5. Conclusion

Chemically impermeable hybridized dentin in the Super-Bond C&B group provided a higher tensile strength on the dentin-cement interface than on the cement-prosthesis interface with failure in the cured resin and on the prosthesis side of the interface. Types of restorative materials had no influence in terms of retention and dentin protection in Variolink II and Single-Bond + RelyX groups, as demineralized dentin introduced during treatment was the weakest point allowing acid penetration and chemical degradation of restored dentin. These results suggest dentin restored by providing an impermeable hybridized dentin is more significant in protecting weak dentin in a cavity or abutment from demineralization with oral acids, thus promoting longer-term function. Tensile strength of restored dentin using all-porcelain or resin-composite cemented with Super-Bond C&B was not less than that of cast metal alloy which confirms that metal-free restorations can be used as well as cast metal to provide retention, stability, and perfect seal for restored dentin abutments.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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