Repair bond strength of resin composite to a novel CAD/CAM hybrid ceramic using different repair systems

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This study evaluated the repair bond strength of a nanohybrid resin composite to a novel CAD/CAM hybrid ceramic based on four intraoral ceramic repair systems. Vita Enamic (VE) CAD/CAM hybrid ceramic was used in this study. Specimens were divided into five test groups according to the repair method performed on the ceramic surface: Gr C (No treatment; control); Gr CZ (Cimara Zircon); Gr PR (Porcelain Repair); Gr CR (Clearfil Repair); and Gr CS (CoJet system). Nanohybrid resin composite (GrandioSO) was packed onto treated ceramic surfaces for adhesion testing using microtensile bond strength test. Debonded specimens were examined with a stereomicroscope and SEM to determine the fracture mode. Data were analyzed using ANOVA and Tukey's HSD test. PR and CZ repair systems significantly enhanced the bond strength of nanohybrid resin composite to VE CAD/CAM hybrid ceramic when compared with the other tested repair systems.

Keywords: Adhesion, CAD/CAM hybrid ceramic, Microtensile bond strength, Repair, Surface treatment

INTRODUCTION

The use of CAD/CAM all-ceramic restorations, such as inlays, onlays, veneers, crowns, and bridges, has increased in recent years because of patients' increasing esthetic demands[1-3]. On the other hand, ceramic restorations in clinical service are susceptible to fractures[4]. Intraceramic defects, trauma, and parafunctional habits are frequently reported to cause the fracture of all-ceramic restorations[5]. Replacing a failed restoration is not necessarily the most practical solution when the following factors are taken into account: replacement cost, more loss of tooth structure, and further trauma to the tooth[1,6].

For localized failures, intraoral repair could be an alternative treatment to restoration replacement which is clinically unacceptable and no longer satisfactory[1,4,7]. Intraoral repair is a minimally invasive approach that entails the addition of a restorative material —with or without prior preparation in the restoration[1,8,9]. However, the repair of fractured ceramic restorations is a challenging clinical situation. To date, documentation on the clinical performance of repaired restorations is sparse and scanty[10].

Different repair protocols have been developed, and evaluated by researchers, to enhance the functionality, longevity, and esthetics of ceramic restorations: acid etching (*e.g.*, hydrofluoric acid, acidulated phosphate fluoride, and phosphoric acid)[2,11-14], airborne particle abrasion with aluminum oxide[15], and airborne particle abrasion with silica coating[16-18]. However, there is no agreement on which repair system is the efficient one that guarantees favorable clinical outcome[16]. This is because various factors influence the repair bond strength of resin composites to dental ceramics: type of ceramic, repair protocol, aging condition, and type of resin composite[1].

Silica coating technique was reportedly the best treatment for alumina ceramics, zirconia[6], and leucite-reinforced glass ceramics[16]. For lithium disilicate ceramics, airborne particle abrasion and acid etching produced the highest tensile bond strength values to a resin composite[6]. On the other hand, etching alumina-reinforced feldspathic ceramic with hydrofluoric acid followed by silanization provided higher repair bond strength than the combined treatment of airborne particle abrasion with 110-µm aluminum oxide or 30-µm silicon oxide followed by silanization[19]. In another study, silane coating after airborne particle abrasion and acid etching was the most effective surface treatment in terms of improved bond strength for a lithium disilicate ceramic to a resin composite[10].

On the effect of aging on alumina-reinforced feldspathic ceramics, the hydrolytic stability of repair protocols based on silica coating and silanization was reportedly superior to the other repair protocols[18].

Hybrid ceramics are based on the concept of combining the positive characteristics of ceramics and composites[20,21]. A CAD/CAM hybrid ceramic was recently developed for indirect restorations. It consists of a feldspar ceramic network (86 wt%) that is fully integrated with a polymer network (14 wt%)[20] (Fig. 1). To date, no studies have evaluated the repair bond strength of CAD/CAM hybrid ceramic. The aim of the present study was to evaluate the repair bond strength of a nanohybrid resin composite to CAD/CAM hybrid ceramic based on four intraoral ceramic repair systems. The null hypothesis of this study was that repair bond strength would not differ among the four ceramic repair systems tested.

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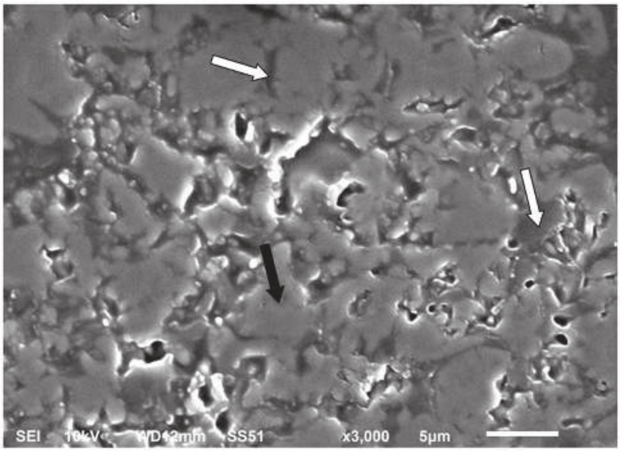

Fig. 1 SEM micrograph (×3,000) of VE CAD/CAM hybrid ceramic surface.
White arrows represent polymer phase and black arrow represents ceramic phase.

MATERIALS AND METHODS

Specimen preparation

Vita Enamic (VE) CAD/CAM hybrid ceramic was used in this study. Manufacturers and compositions of the materials used in this study are presented in Table 1. A total of 15 blocks (10 mm×10 mm×6 mm) were cut from VE CAD/CAM hybrid ceramic using a water-cooled diamond blade (Diamond Wafering Blade, Buehler, Lake Bluff, IL, USA) with a low-speed cutting saw (Isomet, Buehler, Lake Bluff, IL, USA). Specimens were wet-ground on one surface only using 1,200-grit silicon carbide paper (Leco Corp., St. Joseph, MI, USA), and then ultrasonically cleaned (Sonorex, Bandelin, Germany) in distilled water for 5 min.

Specimens were aged in an artificial saliva (*Artificial Saliva* Gal Fovet; SAGF) medium (NaCl 125.6 mg L^{-1}; KCl 963.9 mg L^{-1}; KSCN 189.2 mg L^{-1}; KH_2PO4 654.5 mg L^{-1}; urea 200.0 mg L^{-1}; $NaSO_4.10H_2O$ 763.2 mg L^{-1}; NH_4Cl 178.0 mg L^{-1}; $CaCl_2.2H_2O$ 227.8 mg L^{-1}; $NaHCO_3$ 630.8 mg L^{-1}) at 37°C for 30 days. After which, specimens were subjected to thermocycling between 5°C and 55°C for 1,000 cycles with a 30-s dwell time[16,22].

Grouping of specimens

Fifteen specimen blocks (10 mm×10 mm×6 mm) were divided into five test groups (*n*=3 blocks/group) according to the repair method performed on the VE CAD/CAM hybrid ceramic surface: Gr C (No treatment; control); Gr CZ (Cimara Zircon); Gr PR (Porcelain Repair); Gr CR (Clearfil Repair); and Gr CS (CoJet system) (Table 2).

Application of repair resin composite

Nanohybrid repair resin composite (GrandioSO; GS) was packed onto treated VE CAD/CAM hybrid ceramic surfaces using a split Teflon mold (10 mm×10 mm×12 mm). Resin composite layers were incrementally condensed into the mold to fill up the mold, and each layer (2 mm) was cured for 40 s using a LED light curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA; light output: 1,200 mW/cm^2). After curing, the ceramic-resin composite block was removed from the mold and the adhesive interface was exposed to additional 40 s of irradiation. Bonded specimens were stored in artificial saliva at 37°C for 24 h prior to microtensile bond strength (μTBS) testing[16].

μTBS test

After 24-h storage, ceramic-resin composite blocks were vertically sectioned into serial slabs and then into beams with a cross-sectional area of approximately 1 mm^2 using a water-cooled diamond blade with a low-speed cutting saw. Fifteen beams were obtained from each ceramic-resin composite block (*n*=45 beams/group). Only the central beams were used for μTBS testing. Peripheral beams were discarded so that results would not be influenced by either excess or insufficient amount of resin composite at the margins[18,19].

Each beam was attached to a Bencor Multi-T testing device (Danville Engineering Co., Danville, CA, USA) using a cyanocrylate adhesive (Zapit, Dental Ventures of America Inc., Anaheim, CA, USA). It was stressed in tension to failure using a universal testing machine (Model TT-B, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min. Bond strength (σ) (MPa) was calculated using the following formula: $\sigma=L/A$, where L is the load at failure (N) and A is the surface area (mm^2) of each beam[18,23]. A digital caliper (Digimatic Caliper, Mitutoyo Corp., Tokyo, Japan) was used to measure the cross-sectional area of fracture site.

Failure mode analysis

After μTBS testing, fractured beams were examined under a stereomicroscope (SZX-ILLB100, Olympus Optical Co. Ltd., Tokyo, Japan) at ×40 magnification and by scanning electron microscopy (SEM; JSM-6510LV, JEOL Ltd., Tokyo, Japan) at ×500 magnification to determine their failure modes. For SEM examination, the fractured surface of each beam was rinsed with 96% ethanol, air-dried, mounted on metallic stub, and sputtered with a gold layer (SPI-Module Sputter Coater, Structure Probe Inc., West Chester, PA, USA). Failure modes were classified as follows: Type 1 —Adhesive failure for fractured surfaces with no remains from other materials; Type 2 —Cohesive failure within adhesive layer (primer, silane, and/or adhesive); Type 3 —Cohesive failure in resin composite (failure totally within the resin composite) or cohesive failure in ceramic (failure totally within the ceramic); and Type 4 —Mixed failure[10,24,25].

Statistical analysis

Normality of data distribution was tested using Kolmogorov-Smirnov and Shapiro-Wilk tests. Data were normally distributed. μTBS (MPa) mean values was analyzed using one-way analysis of variance (ANOVA) and multiple comparisons were made using Tukey's HSD test with SPSS 15.0 (Statistical Package for Social

Table 1 Materials used in this study

Material	Product (composition)†	Code	Manufacturer
CAD/CAM Ceramic	Vita Enamic (86 wt% feldspar ceramic, 14 wt% polymer)	VE	Vita Zahnfabrick, Bad Säckingen, Germany
Cimara Zircon	Grinding burs, Zircon-primer: organic acids and silane, Zircon-adhesive: Bis-GMA, HEMA, TEGDMA, BHT, acetone	CZ	VOCO, Cuxhaven, Germany
Porcelain Repair	Ultradent porcelain etch: 9% hydrofluoric acid, Ultradent silane: 8% methacryloxypropyl-trimethoxysilane, isopropyl alcohol, acetic acid, Peak Universal Bond: 7.5% ethyl alcohol, 0.2% chlorhexidine, methacrylic acid, 2-HEMA	PR	Ultradent Products Inc., South Jordan, UT, USA
Clearfil Repair	K-etchant gel: 40% phosphoric acid Clearfil-SE Bond Primer: 10-methacryloyloxydecyl dihydrogen phosphate (MDP), HEMA, dimethacrylate monomer, water, photoinitiator, Clearfil-SE Bond: silanated colloidal silica, Bis-GMA, 10-MDP, Clearfil Porcelain bond activator: bisphenol A polyethoxy dimethacrylate 3-methacryloyloxypropyltrimethoxy silane (MPS)	CR	Kuraray Medical, Okayama, Japan
CoJet system	CoJet-Sand: aluminum trioxide particles coated with silica, particles size: 30 µm, ESPE-Sil: 3-methacryloxypropyl-trimethoxysilane, ethanol, Visio-Bond: dicyclopentyldimethylene diacrylate, 2-propenoic acid, 2-methyl,2-(2-hydroxylethyl) (3-methoxypropyl) aminoP ethyl ester	CS	3M ESPE, St. Paul, MN, USA
GrandioSO	Nanohybrid composite (A2 Shade, 89% wt inorganic fillers: glass ceramic filler (particle size 1 µm), silicon dioxide nanoparticles (20–40 nm), Bis-GMA, Bis-EMA, TEGDMA, initiators, inorganic pigments, BHT)	GS	VOCO, Cuxhaven, Germany

† Manufacturers' data.
Bis-GMA: Bisphenol A glycidyl methacrylate, HEMA: hydroxyethyl methacrylate, TEGDMA: Triethylene glycol dimethacrylate, Bis-EMA: Ethoxylated bisphenol A glycidyl methacrylate, BHT: butylated hydroxytoluene

Table 2 Repair procedures

Surface treatment	Procedures
Control	No treatment was applied to the ceramic surface; apply GS composite; light-curing 40 s
CZ	Surface grinding with Universal Ceramic grinding burs at 6,000–10,000 rpm; clean the bonding surfaces under dry conditions with the short-bristled brushes; Zircon-primer 60 s; dry; Zircon-adhesive; blow thin; light-curing 20 s; apply GS composite; light-curing 40 s
PR	Surface grinding with standard flame-shaped diamond bur 10 strokes; Ultradent porcelain etch 90 s; rinse 20 s; dry 5 s; Ultradent silane: 60 s; Peak Universal Bond; blow thin 10 s; light-curing 10 s; apply GS composite; light-curing 40 s
CR	Surface grinding with standard flame-shaped diamond bur 10 strokes; K-etchant gel 5 s; rinse; dry; mix 1 drop of SE Bond primer with 1 drop of Porcelain Bond activator; mix; apply 5 s; dry; apply SE Bond Bond 15 s; blow thin; light-curing 10 s; apply GS composite; light-curing 40 s
CS	Air-abrasion with 30 µm aluminum trioxide particles coated with silica; distance 10 mm; pressure 2.5 bar 15 s; sand particle remnant gently air blown; ESPE-Sil: 5 min; Visio-Bond: air thinned and light-curing 20 s; apply GS composite; light-curing 40 s

Science, SPSS Inc., Chicago, IL, USA). Statistical significance was set at α=0.05.

RESULTS

Table 3 presents the mean and standard deviation values of µTBS (MPa). PR and CZ repair systems revealed significantly higher (p<0.05) bond strength values (17.17±3.10 and 16.71±2.46 MPa respectively) than CS and CR systems (12.91±2.25 and 10.58±2.04 MPa respectively). There was no significant difference between PR and CZ repair systems (p>0.05). The control group, which had no surface treatment, demonstrated the lowest bond strength value (9.58±1.13 MPa). In summary, improved bond strengths (MPa) were yielded by the tested repair systems in the following descending order: PR>CZ>CS>CR>control group (Table 3).

On failure modes, mixed failure (Type 4) was the predominant failure mode identified for specimens treated with CS, PR, and CZ (86.66%, 84.44%, and 82.22% respectively). On the other hand, adhesive failure (Type 1) was mainly observed for the control and CR groups (88.89% and 77.78% respectively). In addition, cohesive failure within the adhesive layer (Type 2; 6.22%) and cohesive failure within the resin composite (Type 3; 4%) were also observed (Fig. 2). No cohesive failures were observed within the ceramic. Representative SEM micrographs of fractured beams are presented in Fig. 3.

Table 3 Mean (standard deviation) of the μTBS (MPa) of CAD/CAM hybrid ceramic/resin composite with different repair treatments and Tukey's analysis

Surface treatment	VE/GS
Control (no treatment)	9.58^{C} (1.13)
CZ	16.71^{A} (2.46)
PR	17.17^{A} (3.10)
CR	10.58^{C} (2.04)
CS	12.91^{B} (2.25)

Mean values represented with different superscript uppercase letter are significantly different according to Tukey's test (p<0.05).

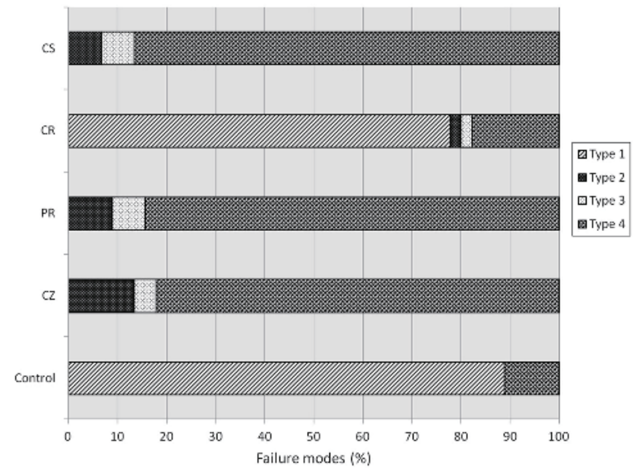

Fig. 2 Failure mode distributions of different test groups, where Type 1: Adhesive failure for fractured surfaces with no remains from other materials; Type 2: Cohesive failure within the adhesive layer (primer, silane and/or adhesive); Type 3: Cohesive failure in resin composite (failure totally within the resin composite) or cohesive failure in ceramic (failure totally within the ceramic); and Type 4, Mixed failure.

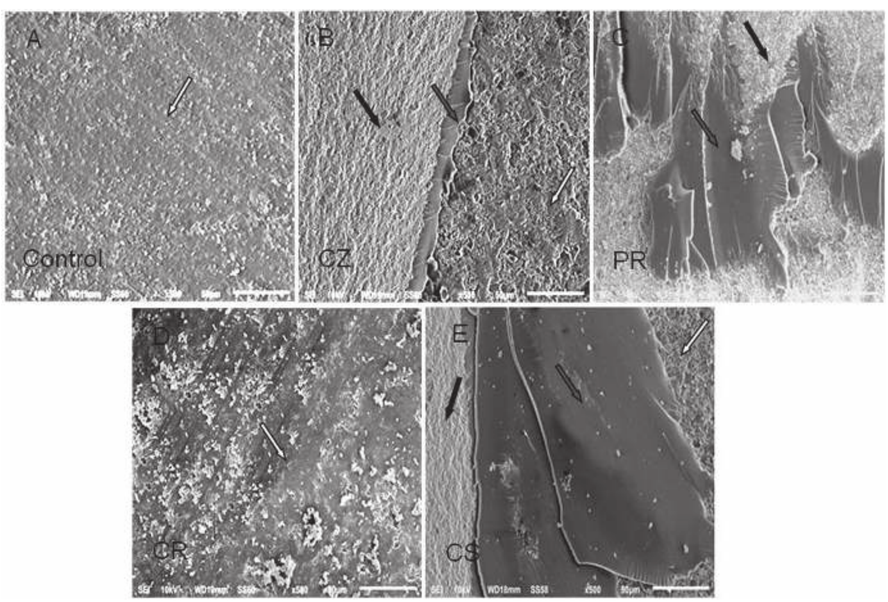

Fig. 3 Representative SEM micrographs (×500) of the debonded surfaces of GS/VE system after different repair treatments.
White arrow represents ceramic surface, black arrow represents retained composite, and gray arrow represents retained adhesive system (primer, silane and/or adhesive). Specimens repaired with CZ, PR, and CS showed predominantly mixed failure mode while control and CR groups showed mainly adhesive failure at the interface.

DISCUSSION

Results of this study led to the rejection of the null hypothesis: the four ceramic repair systems tested in this study demonstrated differing repair bond strengths.

Depending on the extent and reason of ceramic restoration fracture, intraoral repair with resin composite may be indicated as a simple alternative to extraoral repair[5,19,26,27]. Although various repair systems based on different conditioning protocols are available on the dental market, it is not easy for clinicians to choose the most appropriate system that would provide a reliable outcome[16,18]. Repair of ceramic restorations requires the surface to be conditioned to enhance the adhesion of repair resin composite to the ceramic surface[28]. In this study, the repair bond strength of a nanohybrid resin composite to VE CAD/CAM hybrid ceramic using four intraoral ceramic repair systems was determined by a μTBS test. The μTBS test is a reliable bond strength test as it provides a more uniform and homogeneous stress distribution during loading, and failure mainly occurs at the adhesive interface because of small bonded interfaces (approximately 1 mm^2) of the specimens used in this test[29-31].

Restorations typically fail after being aged in a humid oral environment. Aging conditions may cause some alterations on the surfaces of restorative materials[32,33]. For this reason, restoration aging should be considered and included in the planning of restoration repair[32]. In this study, specimens were aged in SAGF medium at 37°C for 30 days followed by thermocycling to obtain aged substrate surfaces.

In the present study, four different repair systems were tested —based on the initial mechanical surface preparation (diamond bur roughening for CZ, PR, and CR systems *versus* airborne particle abrasion for CS system) followed by the application of chemical component (primer, hydrofluoric acid, phosphoric acid, and silane respectively) (Table 2). PR and CZ systems significantly enhanced the bond strength of GS resin composite to VE CAD/CAM hybrid ceramic when compared with the other repair systems (Table 3). In PR system, the hydrofluoric acid dissolved the glass matrix of the ceramic and formed microporosity on the ceramic surface, thus enhancing micromechanical retention with the resin composite[13,18].

Silanization after hydrofluoric acid etching is widely advocated to improve adhesion between resin composites and ceramics[34,35]. Silane application enhances the chemical adhesion between organic and inorganic substrates, thereby achieving durable adhesion[26]. Silane is a bifunctional monomer which contains a silanol group that reacts with ceramic surfaces and a methacrylate group that co-polymerizes with the organic matrix of composites[18]. Thus, silane application improves the adhesion of resin composites to ceramics[27]. However, if silane is used without any prior surface conditioning of the ceramic surface, it results in inferior bond strength due to inadequate mechanical retention[36].

In CZ system, surface preparation by bur grinding was followed by applications of silane-containing primer and unfilled adhesive resin. It was postulated that the silane-containing primer formed mainly chemical bonds with the VE CAD/CAM hybrid ceramic surface. The microstructure of VE CAD/CAM hybrid ceramic consisted of a feldspar ceramic network that was fully integrated with a polymer network[20] (Fig. 1). On the one hand, the methoxy groups of silane-containing primer chemically bonded with both the SiO_2 and integrated polymer components of VE CAD/CAM hybrid ceramic; on the other hand, these methoxy groups of silane molecules also reacted with the methacrylate groups of resin composite[26], thereby improving bond strength. In addition, the microretentive surface structure created by grinding increased the surface area. Accordingly, surface wettability after primer application was enhanced before unfilled adhesive resin was added[33].

Compared with the control and CR groups, CS repair system resulted in improved bond strength of GS resin composite to VE CAD/CAM hybrid ceramic (Table 3). CS system was based on the deposition of silica-coated alumina particles on substrate surface using chairside airborne particle abrasion. Applied silane coupling agent on the surface then caused covalent bonds to form among the alumina and silica particles and the adhesive[17,18], thereby improving the bond strength between resins and ceramics[16,26]. However, CS repair method had several shortcomings: vulnerability of ceramic surface contamination by sand particles, risk of health problems because of exposure to airborne particulate matter, and the additional cost of airborne abrasion device[18,26,29]. Moreover, sandblasting can damage ceramic surfaces and result in huge volume loss. Therefore, sandblasting of all-ceramic restorations with feldspar glass materials should be avoided[37].

CR repair system presented the lowest bond strength when compared with the other repair systems (Table 3). It was postulated that the surface roughness accomplished by diamond bur preparation and 40% H_3PO_4 was not efficient in improving the adhesion of GS resin composite to VE CAD/CAM hybrid ceramic. This finding agreed with previous studies in that lower bond strength was obtained with CR repair system[11,16,18], although different ceramics and resin composites were used in those studies. It was also reported that the role of H_3PO_4 was limited to surface cleaning after surface was ground with diamond burs, but not to change the ceramic surface[11,16].

Failure mode analysis of debonded specimens supported μTBS test results. The control and CR groups, which had significantly lower bond strength values, had higher incidence of adhesive failures (88.89% and 77.78% respectively). CS, PR, and CZ repair systems, which had higher bond strength values, had mainly mixed failures (86.66%, 84.44%, and 82.22% respectively) (Table 3, Fig. 2). A mixed failure mode indicates better adhesion, while adhesive failure is typically associated with low bond strength values[19,38,39]. It is noteworthy that all fractures occurred within the adhesive zone —the area where the intermediate material (primer, silane, and/or adhesive) interacted with the substrates (resin composite and ceramic)[24,25]. Therefore, data obtained in this study could be a reliable bond strength indicator of the tested GS resin composite/VE CAD/CAM hybrid ceramic system as all fractures occurred within the adhesive zone[24].

It has been reported that the type of resin composite used could influence its bond strength to ceramic[40]. In this study, nanohybrid resin composite restorative material (GS; Table 1) was used as the repair composite. GS resin composite, being a nanohybrid resin composite, contained silicon dioxide nanofillers besides the conventional hybrid-type fillers[41]. The main advantages of nanofillers are improved translucency effect and better polishability[42,43].

Results of this study suggested that PR and CZ repair systems are appropriate, practical, and cost-effective as these methods do not need more armamentarium purchase (*e.g.*, air abrasion device)[18]. In addition, the bond strengths produced by PR and CZ repair systems (17.17±3.10 and 16.71±2.46 MPa respectively, Table 3) were within the adequate repair bond strength range of 15 to 25 MPa indicated for clinical situations[44]. However, none of the individual surface treatments can be recommended as a universally optimal repair technique, as the efficiency of each repair technique is material-dependent[45]. Further studies are required to evaluate the effects of long-term aging and fatigue loading on the repaired GS resin composite/VE CAD/CAM hybrid ceramic system. In addition, the clinical performance of repaired ceramic must be evaluated to provide reliable recommendations for dental practitioners.

CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn:

1. PR and CZ repair systems significantly enhanced the bond strength of GS nanohybrid resin composite to VE CAD/CAM hybrid ceramic.
2. PR and CZ repair systems could be considered as alternative techniques to CS to avoid the contamination and damage of ceramic surfaces by sand particles and exposure to unfavorable working environment.
3. Surface treatment of VE CAD/CAM hybrid ceramic with CR system yielded the lowest bond strength value between ceramic and repair resin composite when compared with other repair systems.

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