Original Article

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Effect of cementation technique on fracture resistance of glass-ceramic material

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Objective: To evaluate the effect of cementation technique on the fracture resistance of glass-ceramic material.
Methods: Sixty third molar teeth were collected. The tooth surface was prepared by cutting on occlusal table to obtain 5 mm x 5 mm of dentin surface, and the tooth was fixed in acrylic resin cylinder. Sixty 4 mm x 4 mm x 2 mm zirconia-reinforced lithium silicate plates were made and divided into four groups (n = 15). The ceramic bonding surfaces of group 1, 2 and 3 were etched with 5% hydrofluoric acid coated with single bond universal adhesive and cemented to tooth surfaces with self-adhesive resin cement using three techniques, untreated, self-etch bonding, and etch and rinse bonding techniques, respectively. For group 4 (Control group), untreated ceramics were cemented with zinc phosphate cement on untreated tooth surfaces. The specimens were tested under compression with a 5 mm steel ball using a universal testing machine. The maximum failure load was recorded in newton. Fracture surfaces were examined under a scanning electron microscope to determine the origin and the fracture pattern. The data were analyzed using one-way analysis of variance (ANOVA), and Tukey's HSD test.
Result: The mean fracture loads of group 1, 2, 3, and 4 were 1698 ± 126 N, 1760 ± 231 N, 1842 ± 172 N and 1018 ± 151 N, respectively. Fracture load of group 4 was significantly lower than that of the other groups ($p < 0.01$). However, there was no significant difference between other groups.
Conclusion: The use of self-adhesive resin cement showed higher fracture load than zinc phosphate cement. The cementation techniques with self-adhesive resin cement exhibited negligible effect to the fracture resistance of glass-ceramic materials.

Keywords: cementation technique, fracture analysis, fracture resistance, glass-ceramic, zirconia-reinforced lithium silicate

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Introduction

Esthetics outcome becomes a main factor for material selection to reflect the patient's character and beauty. In the past decades, all-ceramic restorations have been increasingly used to replace the metal-ceramic restorations in both anterior and posterior teeth. All-ceramic materials such as lithium disilicate ceramics are very popular because it fulfils the demand in the esthetic aspect of patients [1].

Several studies recommended that lithium disilicate ceramic can be used as a monolithic all-ceramic crown. They do not need metal substructure to support glass veneer as in porcelain fused to metal crown (PFM) [2]. Besides that, lithium disilicate ceramics have good clinical survival rate [3], and they have higher fracture resistance than bilayer zirconia/fluorapatite all-ceramic crown [4]. However, other researchers reported that lithium disilicate crowns have an excellent survival rate within 1 – 5 years but the survival rate declined after 5 – 10 years [5-8].

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The main failure of all-ceramic restorations is fracture after being use. The properties of ceramic material are stiff but brittle, therefore it tends to crack when exposed to the tensile stress [9]. Normally, ceramics always have flaws in their structure, and these flaws may occur during powder compaction, forming, drying, firing and grinding. The fracture generally initiates from the most critical flaw and propagates through the structure of the restoration, and it finally fails [9-12].

Factors, which affected the fracture resistance of ceramic restoration were preparation design, restoration's geometry, restoration thickness, the mechanical properties of ceramics, occlusal force, cementation material and bonding technique [12-17]. For the film thickness, the result showed that there was no effect of the film thickness on the fracture resistance of bonded glass-ceramic/resin plate except when the film thickness of resin cement was over 300 μm where the fracture resistance was gradually decreased [18].

Several studies investigated whether the type of resin cement affected the fracture resistance. A study indicated that type of the resin cement exhibited no effect on the fracture resistance [19]. However, it has been reported that the bonding system distinctively affected the fracture resistance of all-ceramic restoration [20]. In addition, some studies suggested that the use of adhesive bonding system between the ceramic and the tooth structure improved the fracture resistance of laminate ceramic and feldspathic porcelain [16, 21]. However, they found that only bonding system affected the fracture resistance. They did not state whether the different bonding techniques affected the fracture resistance.

The cementation technique of resin cement is related to the bonding system. The bonding system can be classified into three groups [22]; 1) adhesive resin cement using etch and rinse adhesive; 2) adhesive resin cement using self-etch adhesive; and 3) self-adhesive resin cement. Etch and rinse adhesive is an effective system; however, the technique is rather sensitive [23]. Hence, this lead to the development of self-etch adhesive which is more simple and user-friendly compared to the early system [24]. Some studies showed that there was no significant difference in bond strength and longevity between 3 steps etch and rinse and 2 steps self-etch methods. But, there was the difference between 3 steps etch and rinse and 1 step self-etch (all-in-one) methods [25, 26].

A self-adhesive resin cement is popular for luting an indirect restoration because the cement does not require tooth surface treatment before cementation. The clinical application can be finished in one step. It was proposed that the smear layer removal is unnecessary, and the incidence of post-operative sensitivity after cementation is low [22]. As a result, the self-adhesive resin cement is widely used in cementation of fixed dental prostheses. Although the self-adhesive resin cement is effective by itself, the bond strength is increased when it was used with the dentin bonding systems [22, 26, 27].

Modifying the bonding surface of the crowns improved the fracture resistance of all ceramic restorations [17]. In the cementation procedure, the surface was treated with acid, and the gap between flaws was filled with resin cement and adhesive agent. However, there have been very few studies of the effect of bonding system on the fracture resistance of all-ceramic restoration. Therefore, the objective of this study was to evaluate the effect of using different cementation techniques on the fracture resistance of glass-ceramic materials.

Materials and methods

Specimens preparation

Tooth preparation

This study was ethical approved by the

institutional review board, Faculty of Dentistry/ Faculty of Pharmacy, Mahidol University: IRB COE2018/034.3008. Sixty unrestored and caries-free third molar human teeth were collected and thoroughly washed in running water. The adherent tissue was removed by periodontal scalers or sharp hand instruments. They were stored in 1.0% chloramine-T trihydrate bacteriostatic/ bactericidal solution at room temperature for 7 days. After that, the teeth were stored in 4°C distilled water for 2 days [28, 29].

The teeth were cut using a low speed diamond saw. (IsoMet, Buehler, Lake Bluff, Illinois, USA). The occlusal table of the teeth was cut flat at the distance of 2-3 mm below the cusp tip, in which the area of 5 mm x 5 mm of dentin was obtained. The root of the tooth was secured in custom-made auto-polymerizing acrylic resin cylinders (20 mm in diameter). The tooth was fixed along their long axis where the cementoenamel junction was 2 mm emerged from the resin [20].

Fabrication of ceramic specimens

A commercial glass-ceramics, zirconia-reinforced lithium silicate (Celta Duo, Dentsply Sirona Restorative, Milford, USA) was selected for this study. Sixty ceramic plates were made in the size of 4 mm x 4 mm x 2 mm. and divided into 4 groups of 15 each. The surface of group 1, 2 and 3 were acid-etched using 5% hydrofluoric acid (HF; IPS Ceramic Etching Gel 5%, Ivoclar Vivadent, Schaan, Liechtenstein) for 20 sec, rinsed and gently blew until dried. Single bond universal adhesive (3M ESPE) was applied on the etched ceramics for 20 sec and gently blew for 5 sec. The ceramics of group 4 was no surface treatment.

Cementation methods

The cementation techniques were classified into four groups as showed in table 1.

Group 1: No tooth surface treatment. Treated ceramics were cemented with RelyX™ Unicem, 3M ESPE.

Group 2: Tooth surface treatment by self-etch bonding technique: dentin on the occlusal surface was treated with Single bond universal adhesive, 3M ESPE for 20 sec. Treated ceramics were cemented with RelyX™ Unicem, 3M ESPE.

Group 3: Tooth surface treatment by etch and rinse bonding technique: dentin on the occlusal surface was etched using 37% phosphoric acid (Scotchbond™ Universal Etchant) for 15 sec, rinse and treated with Single bond universal adhesive, 3M ESPE for 20 sec. Treated ceramics were cemented with RelyX™ Unicem, 3M ESPE.

Group 4: No tooth surface treatment. Untreated ceramics were cemented with zinc phosphate cement, S.S. White group. (Control group)

Table 1 showed variables between four groups of cementation techniques

Groups	Tooth surface treatment	Cementation materials
1	No tooth surface treatment	Self- adhesive resin cement (RelyX™ Unicem, 3M ESPE)
2	Self-etch bonding technique (Single bond universal adhesive, 3M ESPE)	
3	Etch and rinse bonding technique (Scotchbond™ Universal Etchant and Single bond universal adhesive, 3M ESPE)	
4	No tooth surface treatment (control group)	Conventional cement (Zinc phosphate cement, S.S. White group, Gloucester, England)

Table 2 Materials: type, manufacturer, composition and batch number used in this study

Materials	Type	Manufacturer	Composition	Batch number
Scotchbond™ Universal Etchant	Etching gel	3M ESPE Dental Products, St. Paul, MN 55144 - 1000, USA	37% phosphoric acid	664117
Single Bond Universal	Universal Adhesive	3M ESPE Dental Products, St. Paul, MN 55144 - 1000, USA	MDP phosphate monomer, HEMA, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane	70818A
RelyX™ Unicem, shade A3	Self-adhesive universal resin cement	3M ESPE Dental Products, St. Paul, MN 55144 - 1000, USA	Powder: glass fillers, silica, calcium hydroxide, self-curing initiators, pig ments, light-curing initiators (filler load 72% wt, particle size < 9.5 μm) Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators	660827
Zinc cement improved; shade 12 tooth yellow	Zinc Phosphate cement	S.S. White group, Stephenson drive, Gloucester GL2 2HA, England	Powder: zinc oxide, magnesium oxide, coloring agents Liquid: phosphoric acid, aluminum hydroxide, zinc oxide, distilled water	Powder: 626 321 Liquid: 101621-1
IPS Ceramic Etching Gel	Etching gel for dental ceramic	Ivoclar Vivadent AG, FL-9494 Schaan, Liechtenstein	5% Hydrofluoric acid	W95356
CELTRA® DUO, shade A3	Zirconia-Reinforced Lithium Silicate (ZLS)	Dentsply Sirona Restorative 38 West Clarke Avenue, Milford, DE 19963, USA	Zirconia-reinforced lithium silicate (ZLS), type II dental ceramic material, Class 2a pursuant to DIN EN ISO 6872 (CTE 25–500°C: 11.6 10-6 K-1)	18028457

80-micron painter's tapes (Scotch blue Painter's tape, 3M, Minnesota, USA) were used as spaces in each group. The tapes were pressed at borders of cementation area on tooth surface. All ceramic specimens in group 1, 2 and 3 were bonded to the teeth using self-adhesive resin cement (RelyX™ Unicem, 3M ESPE). The ceramic was positioned at the center of tooth between the tapes and was pressed by a flat end steel bar with a constant force of 10 N. The force was applied to the ceramics in direction of long axis of the teeth during cementation. The cement was light-activated using light-curing unit (Bluephase® G2, Ivoclar vivadent) for 1-2 seconds at each border of the ceramic. Excess cement was removed, and the cemented specimens were light activated for 40 sec at each border of the specimens. All ceramic specimens in group 4 were cemented with zinc phosphate cement in the same manner. The specimens were kept in absolute humidity container at 37°C for 2 days before testing.

Mechanical testing

The specimens were tested under compression using a universal testing machine (Instron 5566; Instron Ltd., Buckinghamshire, England) at a crosshead speed of 1 mm/min. A 5-mm steel ball was used to load at the center of the specimen until fracture, and the maximum failure load was recorded in newton [17].

Fracture analysis

Fracture surfaces of the specimens were carbon-coated (SPI; SPI structure prope., West Chester, USA) and examined under a Scanning Electron Microscope (SEM) (JSM-6610LV; JEOL Ltd., Tokyo, Japan). The SEM images were taken to determine the origin of fracture and the fracture pattern.

Statistical analysis

The data were analyzed using one-way analysis of variance (ANOVA). Tukey's HSD test was used for post-hoc multiple comparison. The significance value was set at $p = 0.05$

Result

The result of fracture load is shown in table 3. The mean fracture load of group 1, 2, 3, and 4 were 1698 ± 126 N, 1760 ± 231 N, 1842 ± 172 N 1018 ± 151 N, respectively. Fracture load of group 4 was significantly lower than that of the other groups ($p < 0.01$). However, there was no significant difference between the other groups ($p > 0.05$).

Scanning electron microscopic evaluation of the top surface and fractured surface from all groups showed similar fracture pattern. According to Figure 1, the arcs of circle (ac) or crack line was found on the top surface of ceramics around the compressive area (ca) in all groups. When look at the fracture surface as illustrated in Figure 2, the classical fracture pattern was presented. The images presented a form of yield deformation (y) beneath the compressive area (ca) and showed classical cone cracking (cc) beside the yield deformation (y) area.

Discussion

Cementation method significantly affected fracture resistance of ceramics [20]. Studies reported that the most recommended bonding method for lithium disilicate glass ceramics was to treat the surface with hydrofluoric acid in combination with silane primer [30, 31]. Cementation with adhesive resin cement significantly strengthened the ceramics [32, 33]. The fracture resistance of ceramic was increased when these protocols were followed. The study aimed to evaluate the effects of different tooth surface treatments to fracture resistance of glass-ceramic material by following the recommended protocols.

Table 3 showed the average loading force (N) in each group. The fracture load values with the same superscript indicate no significant difference ($p>0.05$)

Cementation technique	Fracture load (N)
Group 1: no tooth surface treatment and cemented with self-adhesive resin cement.	1698 ± 126 ^{A}
Group 2: self-etch bonding technique and cemented with self-adhesive resin cement.	1760 ± 231 ^{A}
Group 3: etch and rinse bonding technique and cemented with self-adhesive resin cement.	1842 ± 172 ^{A}
Group 4: no tooth surface treatment and cemented with conventional cement.	1018 ± 151 ^{B}

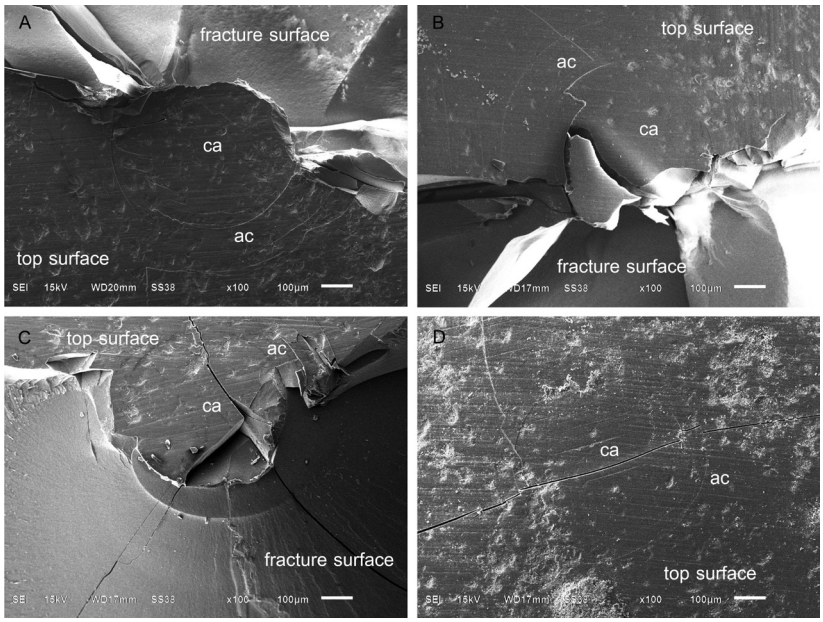


Figure 1 SEM images show top view of ceramic plates after compressive testing. The arcs of circle (ac) were founded around compressive area (ca).

A: group 1, no tooth surface treatment and cemented with self-adhesive resin cement

B: group 2, self-etch bonding technique and cemented with self-adhesive resin cement.

C: group 3, etch and rinse bonding technique and cemented with self-adhesive resin cement.

D: group 4, no tooth surface treatment and cemented with conventional cement.

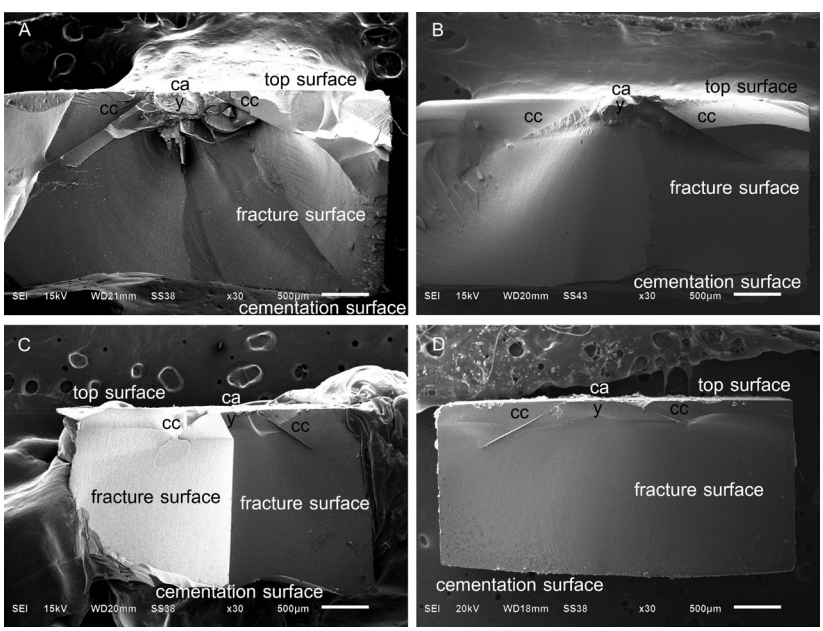


Figure 2 SEM images show fracture pattern on fracture surface. The fracture pattern of every groups occurred as classical cone cracking (cc) and yield deformation (y) under compressive area (ca). The pattern started from top surface and propagated to cementation surface.

A: group 1, no tooth surface treatment and cemented with self-adhesive resin cement

B: group 2, self-etch bonding technique and cemented with self-adhesive resin cement.

C: group 3, etch and rinse bonding technique and cemented with self-adhesive resin cement.

D: group 4, no tooth surface treatment and cemented with conventional cement.

According to the study design, the ceramic's surface was treated with 5% hydrofluoric acid and coated with silane primer in single bond universal adhesive. As the tooth surface treatments were varied in the experimental design, the result showed that resin bonding technique did not affect the fracture resistance of 2 mm thick ceramic plates. However, cementation with adhesive resin cement is a more effective method when compared with conventional cementation. In this study, zinc phosphate cement was used as a control group. Ceramic surface always has flaws, which are normally the origin of fracture. The studies had the assumption that the resin cement or the adhesive cement could reduce the effect of flaws by coating and sealing the ceramic surface flaws with their adhesive ability [32, 33]. The resin cement may improve fracture resistance by this mechanism while the conventional cement like zinc phosphate cement cannot. Moreover, the significant difference of fracture load may relate to mechanical properties of the cement. Quality of the mechanical support of the cementation material is important. A study reported that when loading force applied to ceramic crowns, the multiple cracking was found in the zinc phosphate cementation layer whereas it did not occur when the resin cementation was used. Tension beneath the ceramic restorations can readily build up when the supporting cementation layer has low mechanical strength [32]. Consequently, ceramic plates which was cemented with zinc phosphate cement were more susceptible to fracture than the resin cement. Therefore, ceramic restorations can be strengthened by cementing with resin cement following the recommended protocols.

In this study, the thickness of ceramics was 2 mm, simulating the recommended thickness of monolithic glass-ceramic crowns. According to the SEM images, all groups had similar pattern of ceramic fracture. As shown in figure 2, the images indicated that the fracture started on the loading area (top surface) and propagated downward to the ceramic/cement interface. On the other hand, the previous study reported that crack line initiated from the cementation surface in 1 mm thick ceramics [34]. The variances in origin of fracture was a result of ceramics thickness. It is generally believed that monolithic glass-ceramic restorations required the thickness typically between 1.5 to 2.0 mm to resist tensile stresses at the internal surface between ceramic and cement when they bended under occlusal loading [35]. In the brittle monolithic materials, cracking and damage modes are associated with various stress states. When the compressive contact remains small compared with the outer layer thickness, concentrated loads can induce flexural and other subsidiary stress states in the stiff outer layers on their soft underlayer supports. As a result, cone cracking and yield deformation were occurred at the upper surface near compressive contact and flexural radial cracking was occurred at the lower surface [36]. During occlusal loading on thin ceramic plate, tensile stress activated flexural radial cracking at the internal surface of ceramic and propagate upward to the occlusal surface [35]. Other damage modes induced from the contact stresses at the occlusal surface of the restoration. The damage showed the classical cone cracking and yield deformation [35]. All tested ceramic specimens showed the characteristic of cone cracking and yield deformation in this study. The classical cone cracking responded to the tensile stresses, whereas the form of yield deformation responded to the shear stresses [35]. This fracture can occur and propagate downward to the ceramic/cement interface and leading to failure of the restorations [36]. The radial cracks commonly occur when the ceramic thickness was less than 1 mm, whereas the occlusal surface fractures commonly occur when the thickness was between 1.5 - 2 mm [35]. Accordingly, in the current study, the origin of fracture was found on the top surface of the ceramic plates because the ceramic was thick.

In the present study, no significant difference was found for the mean fracture loads among three groups of ceramics cemented with resin cements. However, mean fracture loads in self-adhesive, self-etch, and etch and rinse adhesive technique presented in an increasing trend. It was suggested that etch and rinse adhesive technique achieved significantly higher fracture resistance value than that of self-etch adhesive technique in case of the 1 mm thick ceramic plate [34]. The dissimilarity in statistical analysis may result from the difference thickness of ceramic plates used in each study. Cementing with resin cement significantly affected fracture resistance when using 1 mm thick ceramic plates [34] because they cannot resist tensile stress at the cementation surface [35]. On the other hand, when the 2 mm thick ceramic was cemented with resin cement, the influence of ceramic thickness and cementation technique on fracture resistance are considerable. The thickness of ceramic was one of the factors which affected the fracture resistance of ceramic [15, 17]. It may have a pronounced influence on the fracture resistance comparing to the cementation technique. As a result, mean fracture loads of the 2 mm thick ceramics cemented with three different adhesive cements exhibited an increasing tendency with no statistical difference. For clinical application, simplification of luting procedure is one of the factors of clinical success. According to the result, clinician can use self-adhesive resin cement for luting 2 mm thick glass-ceramic restoration without any tooth surface treatment. This procedure is simple technique and reduces opportunity of contamination during cementation. However, the tooth surface treatment still has to concern when 1 mm thick glass-ceramic was cemented [34].

Further investigation on the correlation between different thicknesses of ceramics and cementation techniques on fracture resistance has to be experimented.

Conclusion

The cementation techniques with self-adhesive resin cement in the current study showed no significant effect on the fracture load with ceramics. Ceramics cemented with self-adhesive resin cement exhibited higher fracture load than that cemented with zinc phosphate cement. However, the fracture patterns observed in all groups were similar.

Within the limitations of this study, the results can be concluded that the cementation with different bonding systems did not affect the fracture resistance of glass-ceramic materials.

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