

REVIEW

The use of hydrofluoric acid as a surface treatment material on bond strength in repair system of lithium disilicate – Literature Review

Ludwika Patricia Razalie,¹ Putri Welda Utami Ritonga,^{2*} Syafrinani²

ABSTRACT

Keywords: Bond strength, Hydrofluoric acid, Lithium disilicate, Surface treatment

Lithium disilicate is a glassy ceramic material that mimics the aesthetics and strength of natural tooth structure, making it very popular in recent years. Lithium disilicate has good flexural strength, translucency, and high mechanical strength of up to 360 ± 60 MPa which is superior to feldspathic porcelain or leucite-reinforced glass ceramic. These excellent properties make it suitable for both anterior and posterior use. Naturally, ceramic materials are inherently brittle and tend to fracture easily in repetitive function. There are several methods of fracture treatment, one of which is repair. Composite resin is frequently used for ceramic repair as a simple and fast solution. The bond strength of ceramic repairs on lithium disilicate using hydrofluoric (HF) acid is higher compared to other methods. Hydrofluoric acid that has been used as a surface treatment for lithium disilicate is 4% and 5% HF acid. This article reviews the effect of HF as a surface treatment material on bonding strength between lithium disilicate and composite resin. The application of hydrofluoric acid in ceramic surface promotes the reaction with the glass matrix that contains silica and form hexafluorosilicates. This glass matrix is selectively removed and the crystalline structure is exposed. As a result, the surface of the ceramic becomes rough and this roughly etched surface helps to provide more surface energy prior to combining with the silane solution. Silane coupling agent forms a chemical covalent bond between silica on the lithium disilicate surface and composite resin. This bond will increase the micromechanical interlock. (IJPD 2024;5(2):102-106)

Introduction

Metal-ceramic restoration has been widely used for decades as a gold standard, for either single restoration or bridges, but nowadays, ceramic restoration without metal layer has been considered as a restoration with more optimum aesthetic value therefore increasing its demand.¹⁻⁷ Ceramic materials, especially lithium disilicate and zirconia, are being used extensively for the fabrication of crowns and bridges.⁸ Lithium disilicate ($\text{Li}_2\text{O}5\text{Si}_5$) is a glassy ceramic with an average flexural strength of 400 Mpa and a favourable translucency, making it suitable for both anterior and posterior use. Lithium disilicate has many advantages over the traditional metal materials, macromolecule materials, and older ceramics. These include high mechanical and flexural strength, good wear resistance and excellent aesthetics.⁹

Lithium disilicate has a unique structure that helped improve the fracture toughness and increase its flexural strength.¹⁰ Retrospective studies on success rates of lithium disilicate ceramic restorations from between three to ten years of follow up, showed survival rates (i.e. restorations that had remained in place without complications) of over 95%, with the monolithic crowns having less reported structural problems than layered crowns.⁹ Overall cumulative survival probability of lithium disilicate restorations for up to years was 85.08%.¹¹ However, ceramic in nature is a brittle material that highly susceptible to be cracked, which leads to chipping and fracture of the restoration.

Ceramic fractures are often considered an emergency and become a challenge for dentists. Ceramic fractures can occur from several factors, namely intra-ceramic defects, pressure from parafunction, contamination during manufacture, improper planning, endodontic factors, differences in thermal coefficient expansion of core and veneering ceramic, inadequate tooth preparation and trauma.¹²⁻¹⁵ Fractures of ceramic are classified into small chipping, moderate chipping and severe chipping. Small chipping can be treated with polishing, moderate chipping can be repaired with resin composites and grade 3 chipping led to replacement of the entire restoration.

Replacing the fractured ceramic restoration is however not feasible, because it costs more and is less conservative of the natural tooth structure. Restoring crown and bridge restorations cannot be done in the patient's mouth, takes a long time, and requires more complex skills and tools. Removing a bridge or crown restoration is an unpleasant experience for the patient and tiring for the dentist. That is why replacing all restorations is not considered as the best solution because of the high costs and difficulties.^{2,13,14} Therefore, a direct repair of such restorations has to be considered as a more viable option. The repair procedure involves surface treatment of the fractured ceramic interface to create roughness through mechanical means to increase

¹Specialist Program in Prosthodontics, Faculty of Dentistry, Universitas Sumatera Utara, Medan, Indonesia

²Department of Prosthodontics, Faculty of Dentistry, Universitas Sumatera Utara, Medan, Indonesia

*Corresponding author: putri.welda@usu.ac.id

Table 1. Comparison of various surface roughening methods on ceramics

Type of Ceramic	Diamond Burs	HF Acid Etching	Sand blasting	Triboche-mical Silica Coating	Lasers	Recommended Method
Feldspathic Porcelain e.g. IPS Classic (Ivoclar Vivadent, Inc., Amherst, New York), VITA Mark II (Vident, Brea, California)	Effective	Most effective	Effective	Long term low stability	Low bond strength	HF Acid Etching
Lithium Disilicate based Ceramic e.g. IPS e.max Press, Ivoclar Vivadent, Inc., Amherst, New York	Effective	Most effective	Reduces bond strength	n/a	Low bond strength	HF Acid Etching
Leucite-Reinforced Glass Ceramic e.g. IPS Empress, Ivoclar Vivadent, Inc., Amherst, New York	Effective	Low bond strength	Effective	Effective	Low bond strength	Sand blasting with alumina particles
Glass-infiltrated Aluminium oxide Ceramic e.g. In-Ceram Alumina; Vita Zahnfabrik, Bad Säckingen, Germany	Ineffective	Ineffective	Effective	Most effective	Low bond strength	Tribochemical Silica Coating
Densely Sintered Aluminium Oxide Ceramic e.g. ProCera All-Ceram, Nobel Biocare, USA, Inc., Yorba Linda, California	Ineffective	Ineffective	Effective	Most effective	Low bond strength	Tribochemical Silica Coating
Zirconia based Ceramics e.g. In-Ceram Zirconia (Vita Zahnfabrik, Bad Säckingen, Germany), Cercon (Dentsply, York, PA, USA), Lava (3M ESPE, St. Paul, Minnesota)	Ineffective	Ineffective	Effective	Most effective	Low bond strength	Tribochemical Silica Coating

surface area, such as air abrasion; laser irradiation or chemical means to increase the wettability for bonding, by acid etching with hydrofluoric (HF) acid and bonding with silane coupling agents. The treated surface is then ready to receive an adhesive composite resin material to restore the defect.¹⁶

The resin composite adheres well to dental ceramics when the substrate surface is mechanically prepared and a silane coupling agent applied.¹³ The standard protocol for the treatment of vitreous ceramic is etching with HF acid followed by application of silane coupling agent.^{4,5,17-19} HF acid dissolves the glassy surface on the ceramic matrix so that the crystalline structure is exposed, and silane coupling agent acts as hybrids of inorganic-organic compounds that create strong microscopic bonds between the two materials.¹⁹⁻²¹ Although HF acid is the suggested pretreatment for ceramic, this etchant is very toxic and may lead to accident in practice and also weakening the ceramic surface.^{12,17,22} The purpose of this literature review is to review the effect of HF as a surface treatment material on bonding strength between lithium disilicate and composite resin.

Literature Studies

Repair System of Lithium Disilicate Lithium Disilicate Repair Technique

Lithium disilicate repair technique is divided into 2 types, namely direct and indirect. Indirect repair is carried out in the laboratory using porcelain without removing the restoration first. Meanwhile, direct repair is a technique that is carried out directly in the mouth on damaged restoration by applying composite resin. The advantage of the indirect technique is more aesthetic because it is made through laboratory procedure. Disadvantages of indirect technique require additional time and cost. Some of the advantages of the direct technique include shorter time required, lower cost and easy application. While the disadvantages are lower strength, quality of

use and lack of aesthetics.²³

A possible classification and treatment recommendation for a chipped ceramic restoration was published by Heintze and Rousson. A chipping grading scale comprising three grades was established according to the treatment that followed the chipping as: small veneer chippings receive grade 1, moderate veneer chipping receive grade 2 and severe chipping receive grade 3. Grade 1 chippings can be treated with polishing, grade 2 chippings can be repaired with resin composites and grade 3 chippings led to replacement of the entire restoration. The criteria for replacement (grade 3) are the fracture surface extends into a functional area and repair is not feasible, recontouring will result in a significant unacceptable alteration of the anatomic form from the original anatomy, recontouring will significantly increase the risk of pulp trauma by the generation of heat, repair with a resin composite will result in esthetic changes that are unacceptable to the patient.²⁴

The direct repair procedure using composite resin is carried out in several stage, including examination of the fracture classification. Isolation of the fracture part using a rubber dam. Form a bevel on the ceramic fracture suffix using a low-speed green stone bur. Apply HF acid to the porcelain surface, rinse with water and dry. Application of silane material on ceramic surfaces. The bonding material was applied to the ceramic fracture surface and then light cured. The application of the composite resin restorative material using incremental technique and light cured each layer. Finishing and polishing using a disc bur with conventional method.²⁵ The success of ceramic repair by direct technique is clinically determined by the intact bond between ceramic and composite resin. This complete bond is achieved by chemical and mechanical bond. Chemical bonding is obtained from the application of silane, while

mechanical bonding is obtained from the surface treatment.²⁶⁻²⁸

Surface Treatment

Surface treatment is divided into mechanical methods, chemical methods and combination. Mechanical methods include airborne particle abrasion and diamond rotary bur grinding, chemical methods involve etching the ceramics with acid. Several surface treatment methods that have been carried include diamond bur, air abrasion, laser and acid etching. Clinical application of the adhesive method on ceramic requires surface treatment to optimize the adhesion of the composite resin to the ceramic.²⁹ Surface treatment on ceramic is performed to produce mechanical retention which can increase the bond strength of ceramic repair from the roughened ceramic surface.³⁰ This surface roughness results in a micromechanical bond between the ceramic and the repair material obtained from the surface treatment. This makes surface treatment procedure important in determining the success of intraoral repair.³¹

Acid etching provides a clean surface by increasing the capacity for micromechanical retention and, as a consequence, increasing potential bond strength.³² Acid etching on dental ceramics was several types of acid, such as orthophosphoric (OP), sulfuric, nitric, ammonium hydrogen difluoride, acidulated phosphate fluoride (APF) and HF acid are recommended as surface treatment materials for ceramic restoration. The most commonly used acid etching is HF acid.³³ HF acid is an organic acid capable of etching the surface of glass. HF acid reacts with silicon oxide (SiO₂) in the glass phase of ceramics, resulting in surface microporosity, which allows the formation of mechanical interlocks with the composite resin.^{32,34}

HF acid is considered a relatively weak acid from a chemical point of view because of its low tendency to dissociate into H⁺ and F⁻ ions. This does not mean that HF is harmless. Quite the opposite; HF has the ability to easily penetrate skin tissue (often without causing external burns) due its low dissociation potential. These conditions can cause extensive internal tissue damage, as well as alter blood calcium levels (due to CaF₂ formation), which can lead to cardiac arrhythmias. The use of HF during intra-oral repair procedures, exposes the patient to a high risk of acid damage, in particular, soft tissue. Thus, specific protocols should be followed including isolation of the rubber dam, careful use of a triple air water syringe, removal of excess acid and use of a high volume aspirator to maximize preventive measures.³²

HF acid with a concentration of 5% is the type commonly used in etching lithium disilicate restorations and intraoral repair of fractures. HF acid can be safely used in dental procedures within this concentration range, including intraoral repairs, with caution and reasonable care when used. Recommended HF etching time is in the range of 20 seconds to 20 minutes, depending on acid concentration and type of ceramics.^{32,34} The use of composite resin to repair fractures in ceramic has been introduced in various methods. Micromechanical retention of composite resin can be obtained from all surface treatment methods performed on ceramic surface. However, etching porcelain using HF acid is a commonly used procedure. The use of HF acid to achieve a clean microretention surface before bonding or repairing ceramic can be produced. This is because the acid can dissolve the glass matrix on the ceramic, thereby creating a

mechanically retentive surface. Several selections on surface treatment methods on ceramic surfaces that provide effective results can be seen in table 1.³⁰

Surface treatment using HF acid to obtain adequate adhesion between ceramic materials and composite resin is acceptable. Etching of ceramics also has the potential to significantly increase the bond strength of composite resin. Generally, ceramic consists of a glass matrix phase and a crystalline phase. HF acid as an acid that selectively dissolves the glass matrix in ceramic so as to increase the porosity of the surface, it is high energy, microretentive and provides a large surface area for the bonding of composite resin. In principle, these conditions are the same as enamel surface after etching with phosphoric acid. The hydroxyl groups are also exposed after etching using HF which are important for chemical bonding through the solute-pairs present in the silane.³⁵

Silane Coupling Agent

Silanes are a class of organic molecules that contain one or more silicon atoms (3-methacryloxypropyl trimethoxy silane), which act as a wetting agent and help to form covalent chemical bonds at the involved interfaces. Single-bottle silanes that are pre-hydrolysed typically consist of 1% to 5% silane in a water/ethanol solution with added acetic acid to achieve the desired pH of 4 to 5. They perform optimally if left for 5 minutes.

Silane hydrolysis creates terminal hydroxyl groups on each silane molecule. These hydroxyl groups react directly with corresponding hydroxyl groups on the surface of glass ceramic through the oxidation of SiO₂. A condensation polymerization reaction creates bonds between the silane and porcelain when the opposing hydroxyl groups interact with one another via hydrogen and covalent bonding. Clinically, the surface of the porcelain should appear matte after silane application and drying. The treated surface is then ready to receive an adhesive composite material to restore the restoration defect.⁹

Lithium Disilicate Repair Material

Different techniques for repairing fractured ceramic restorations:

Acrylic resin

The attempt for repairing of fractured ceramic used for making denture teeth started early as a trial for preparing the fractured denture teeth. Older techniques utilize acrylic resin as a repairing material for fractured ceramic restorations, these techniques includes: fabrication of a pin only with an acrylic resin veneer cemented to the labial surface, directly formed acrylic resin facing which are cemented to place and the use of Nuva-Fil to replace fractured porcelain.³⁶

Glass ionomer cement

Glass ionomer cement may be used for ceramic repairs. This material is opaque and can be matched to a variety of tooth shades. Its characteristic of local fluoride release presents the advantage of increased resistance to the onset of carious lesions.³⁷

Resin composites

Because of their physical, mechanical and optical properties, hybrid resin composites and their variations are best suited for ceramic repairs. Resin composite systems that offer opaque and translucent resins, in addition to the basic shades, should preferably be used to reestablish esthetics after ceramic repair.³⁷ There are several mechanical properties of composite resin as repair system of lithium disilicate, one of which is bond strength. According to The Glossary of Prosthodontic Terms, the definition of bond strength is the force required to break a bonded assembly with failure

occurring in or near the adhesive/adherent interface. Interfacial bond strength is a key factor influencing the overall mechanical properties of composites. Bond strength tests are relatively easy to perform and can be done without expensive equipment.³⁸

Discussion

The fracture of ceramic restorations can severely compromise the esthetics and function of the restorations, affecting their longevity. In most clinical trials, the failure of ceramic restorations was related to chipping of the veneering layer. Where applicable, the intraoral repair completed with resin composites is a conservative minimally invasive approach with a very good cost-to-benefit ratio. This type of repair is associated with reduced cost, treatment time, and increased longevity of the restoration for the patient.²³ Depending on the type and size of the fracture, different substrates may be exposed, requiring different treatment methods involving etching agents, adhesion promoters and/or sandblasting to ensure good adhesion of intraoral repair material to the ceramic surface. These treatments can promote mechanical interlocking, chemical bonding, or both.

The acids that are used as ceramics etchants are the HF acid, the acidulated phosphate fluoride (APF), and the ammonium hydrogen difluoride. The hydrofluoric acid is the most frequently used acid, which when applied onto the ceramic surface reacts with the silica matrix creating silicon tetrafluoride and molecules of water that are released.³⁹ A scanning electron microscope analysis has shown that ceramic surface etched with 4% APF for 2 min has irregular etching pattern with pores. An insignificant effect of APF may be explained by low concentrations of HF acid and available fluoride ions. However, when the application time of 1.23% APF gel (clinically used as a topical fluoride for reduction of the incidence and progress of caries) was prolonged up to 60 min, increasing in surface roughness was detected, with numerous pores and deposits of particles in the form of precipitate or degradation material in the glassy matrix. The strongest immediate bond was achieved by etching of the ceramic surface with HF acid (23.7 Mpa).³⁹

As ceramic materials are rich in silica, hydrofluoric acid interacts with the microstructure of this material, creating a porous surface that aids in mechanical retention of the resin composite used in the intraoral repair technique. This approach favors the bonding procedure and increases the repair longevity. However, hydrofluoric acid needs to be carefully used intraorally due to possible damage to soft tissues and therefore the use of proper isolation is essential.²³

Etching with acid has multiplied effect on ceramic: cleansing the bonding surface by removing the unwanted oxides, debris and grease, increasing the roughness thus increasing the bonding area and wettability of the ceramic surface and create micro retention that can be easily infiltrated with uncured flowable composite. This will significantly increase resin-ceramic bond strength.²¹ The use of medium-grain diamond burs can produce roughness values comparable to those of hydrofluoric acid but it does not provide sufficient bond strength to be an alternative to acid etching.⁴ This is because the acid etching creates more hydroxyl groups on the surface and increases micro-mechanical retention.¹⁸

A study comparing the difference in shear bond strength between ceramic repair systems using grinding and universal primers with those using etching HF acid and silane on lithium disilicate showed that the shear bond strength achieved in acid etching and silane group was higher than the grinding group. The manufacturer of IPS e.max Press (EMX; Ivoclar

Vivadent, Schaan, Liechtenstein), a commercial brand that presents ± 70 vol% of lithium disilicate crystals dispersed in an amorphous vitreous phase, recommends that EMX should be etched with a 4.8% HF concentration for 20 seconds. However, several clinical reports and in vitro studies, published from 2011 to date, have reported different HF concentrations and etching periods on lithium disilicate ceramic, such as 10% for 15 seconds, 10% for 20 seconds, 9.6% for 30 seconds, 9.5% for 60 seconds, 4.8% for 60 seconds, 5% for 20 seconds.⁴⁰

That etching with 5% HF for 20 seconds can be recommended for lithium disilicate and leucite-reinforced CAD/CAM ceramics. However, for pressed lithium disilicate ceramic, 10% HF for 60 s showed significantly higher bond strength.⁴¹ On the contrary, the highest shear bond strength values were obtained with surface treatment with 9% HF and not affected significantly by the conditioning time (20 s or 90 s). They assumed that surface treatment with 9% HF for only 20 s will provide better bonding strength than using 5% HF acid for 20 s, as per the manufacturer recommendation.⁴² That optimal bond strength to lithium disilicate is achieved by exposure at least 20 s of 5% HF acid followed by a coat of silane. If an additional silane step is not taken prior to applying a universal adhesive, the use of 9.5% HF for 60 s can increase bond strength.⁴³ Sunfeld et al suggested to clinically use previously heated 1% or 2.5% hydrofluoric acid instead of higher concentrations when etching to lithium disilicate glass-ceramic.⁴⁴ The 10% HF acid concentration and exposure time from 20 to 40 s showed the best results.⁴⁵

Over-etching on the etched ceramic surface is clearly visible as "white residue." Sometimes, due to the large amount of residue deposits, it can be very broad covering the ceramic surface. This is related to the concentration of HF acid, time and type of ceramic. The condition cannot be removed by air/water spray and wiping with acetone-soaked cotton. Cleaning can be done by placing it in ethanol followed by ultra-sonication. It takes 15 minutes of ultrasonication to release the white residue. The etching efficiency of HF depends on the concentration, etching time, temperature, and dilution of the acid solution. A consensus regarding the most suitable etching protocol for glass ceramics is not clear, especially for lithium disilicate glass ceramics.⁴⁰

Conclusion and Suggestion

It is concluded that the use of HF acid as a surface treatment material in increasing the bond strength of lithium disilicate and composite resin is an option that can be considered because the material is quite easy to obtain in the market, affordable and easy to apply. The right time and concentration of HF acid to obtain maximum bond strength between lithium disilicate and resin composite is also concerning. The selection of the right time and concentration in the application of HF acid as a surface treatment material is necessary in repair system of lithium disilicate.

Further research is needed on the effect of time and concentration of HF acid as a surface treatment material in lithium disilicate repair system to obtain maximum increase in bond strength between lithium disilicate and composite resin, so that the minimum application time is obtained with the right concentration of HF acid.

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