# **Research Report**

# Shear bond strength between porcelain and nano filler composite resin with or without 9% hydrofluoric acid etching

#### Kun Ismiyatin

Department of Conservative Dentistry Faculty of Dentistry, Airlangga University Surabaya-Indonesia

### ABSTRACT

**Background:** Reparation technique on restorations with broken or damaged porcelain which are still attached with the teeth are difficult, because it is very hard to remove the porcelain restoration without damaging it, and it needs a long time. Various ways have been developed to repair the broken porcelain, one of them is the use of composite resin as the material for the restoration of fractured porcelain. Repairing porcelain inside the mouth without removing the restoration of the damaged porcelain using light cured composite resins material seems to be an advantageous option because it is relatively simple, has low risks, good esthetically and cheap. **Purpose:** The objective of this study was to find out the difference of shear bond strength in porcelain reparation using nano filler composite resin with or without 9% hydrofluoric acid etching by using Autograph measuring device. **Methods:** Twenty pieces of the porcelain samples devided into 2 groups. Group I: etching process using 9% hydrofluoric acid, and group II : without etching process. **Result:** The data was analyzed using t test in a p value of 0.0001 (pÝ0.05), which means there is a significant different of shear bond strength between treated group I and II. The biggest shear bond strength was in treatment group I. **Conclusion:** The use of 9% hydrofluoric acid on the surface of porcelain can increase the shear bond strength between porcelain and nano filler composite resin.

Key words: shear bond strength, porcelain, nano filler composite, 9% hydrofluoric acid, etching

*Correspondence*: Kun Ismiyatin, c/o: Departemen Konservasi Gigi, Fakultas Kedokteran Gigi Universitas Airlangga. Jl. Mayjend. Prof. Dr. Moestopo No. 47 Surabaya 60132, Indonesia. E-mail: kunismiyatin@gmail.com

# INTRODUCTION

Porcelain restoration material has advantages in its strength which resembles email, translucent, not easily worn out, stable and esthetically close to the tooth color, while in dentistry, porcelain material is used for inlay, onlay, laminate veneer, and crown.<sup>1,2</sup> However, porcelain has disadvantages in its fragile character, thus porcelain has fracture potential, even in light pressure.<sup>1</sup> Some factors that may cause porcelain restoration undergo defect are the heavy pressure of occlusion, excessive chewing, micro defect of the material it self, inappropriate design, trauma and its fragile character.<sup>3</sup>

Reparation treatment towards restorations of broken or damaged porcelain which are still well attached to teeth is difficult to do, because it is very hard to remove porcelain restoration without damaging it, and it needs a long time. Various ways have been developed to repair the broken porcelain, one of them is what is now being developed, that is the use of composite resin as the material for the restoration of fractured porcelain. Composite resins has become the chosen material because it is esthetically good, strong enough, not easily abraded, easy to manipulate, able to stick to porcelain surface, provided in various colors so that one may choose the color matching to porcelain color.<sup>4–6</sup> Repairing porcelain inside the mouth without removing the restoration of the damaged porcelain using light cured composite resins material seems to be an advantageous option because it is relatively simple, has low risks, esthetically good and relatively cheap.<sup>7,8</sup>

Composite resins containing very small particles are nano filler composite resins, whose particle diameter ranges from 20–27 nm. The compressive strength and fracture resistance of this nano composite is the same as, or a little higher than other kinds of composites, in addition, nano composite shows better polishing result than that of micro hybrid composite.<sup>9</sup> The shear ability between porcelain and composite resins becomes significant when reparation on porcelain restoration is going to be done, therefore a lot of studies to increase the shear bond strength of composite resins and porcelain have been done. The shear bond strength of the material is determined by the nature of the physical shear, the mechanism and the chemistry of the surface of the material.<sup>2</sup> One of the efforts to obtain the characteristic of chemical shear between composite resins and porcelain is the use of silane solution. Some researchers have proved that composite resins is able to adhere well on porcelain with silane solution.<sup>4</sup> Silane is the combination of organic-inorganic materials which function as a mediator, a coupling agent to obtain shear between different organic and inorganic materials.<sup>8</sup> The silane mostly used in dentistry is  $\gamma$ -methacryloxypropyltrimethoxysilane ( $\gamma$ -mpts), and this material functions as the mediator between composite shear and porcelain because the methacryloxypropyl clusters of  $\gamma$ -mpts match with the ones of dimethacrylate used in resin technology.<sup>10</sup> Besides, this silane has alcoxy clusters which can react on hydroxyl clusters of inorganic silicone substrate of the porcelain,<sup>8</sup> and this will enable silane to become a chemical bond mediator between porcelain and composite resins.

For the first time some researchers did on porcelain etching, they used hydrofluoric acid, then the glass/silica molecules in the porcelain reacted on the hydrofluoric acid was following this reaction:

$$\mathrm{SiO}_{2(\mathrm{s})} + 6\mathrm{HF}_{(\mathrm{aq})} \rightarrow \mathrm{H}_{2}[\mathrm{SiF}_{6}]_{(\mathrm{aq})} + 2\mathrm{H}_{2}\mathrm{O}_{(\mathrm{l})}$$

The result of the reaction, which is  $H_2[SiF_6]_{(aq)}$ , dissolves, resulting a micro porous porcelain surface.<sup>11</sup> The study on the etching of porcelain surface using 9% hydrofluoric acid for one minute produces as wide as 50 µm pore diameter of porcelain surface,<sup>12</sup> so that the resins applied to the surface of the porcelain will penetrate into those pores and form resin tag, producing micro mechanical interlocking between porcelain and composite resin. The hydrofluoric acid used for intra oral is a buffer solution in the shape of gel, which in general has 9% content and 1.57 pH. This solution will produce constant hydrogen fluoride, but during the etching process it will not do any harm.

Repairing porcelain reparation using composite resins needs to result in good bond strength. Therefore a test to asses the shear strength of composite resins to the porcelain which undergoes treatment needs to be done. Nowadays, different opinions whether porcelain surface treatment will develop a better bond strength as well as the use of 9% hydrofluoric acid due to its irritating characteristic to oral tissues still exist. The purpose of this study was to know shear bond strength between porcelain and nano filler composite resin with or without 9% hydrofluoric acid etching.

#### MATERIALS AND METHODS

Experiment samples are 20 pieces of porcelain, metal kind porcelain of Ivoclar made according to the factory instruction, 4 mm in diameter, 4 mm height, no cracking part, having flat surface, soft, and no glazing has been done.<sup>5</sup> The porcelain samples are made more than 20 pieces. Then, the 20 pieces which match with the criteria are taken randomly, 10 each for one group of treatment. The composite resin used is nano filler composite resins polimerated with the help of visible light curing unit.

Group I: The porcelain's surfaces are made rough using No. 100 Taiyo sand paper with medium pressure for one minute, then etching process using 9% hydrofluoric acid is done for one minute to the porcelain's surfaces, and then they are washed with aquadest spray using 2,5 cc syringe, next they are dried using air blow. After that, the porcelain's surfaces were smeared with silane solution twice, and let them dry in one minute.

Group II: The surfaces of the porcelains are made rough, first by means of number 100 Taiyo sand paper, using medium pressure for one minute then they are washed with aquadest spray using 2.5 cc syringe for two times, and after that they are dried using air blower, next the surfaces of the porcelains smeared twice with silane solution, and then they are let dry in one minute after having been treated, each porcelain samples group is placed on a plastic tray. In order to prevent scraping during composite aplication, the treated surfaces must face the composite molds. The composite mould ring was inserted and applied approximately 2 mm thick composite, and cured it with blue light for 40 seconds. Next, approximately 2 mm thick composite was applied for the second time, and then celluloid strip and thin glass were put on it, weighted with one kilogram of weight for one minute, then the excessive composite was removed using scalpel, and cured with blue light of visible light cure unit for 40 seconds. After the composite application was finished, the specimen was taken out from the composite laying aid and the mold ring was removed, at last the finished samples were put into a plastic tube containing aquadest for 24 hours prior to the shearing test.

The test of shear bond strength for each sample group was measured with Autograph AG-10 TE (Shimadzu, Japan) with a cross head speed of 1/10 mm/second and Load cell capacity of 5 kN/500 kgf (1 kgf = 9.81 N).

# RESULT

From the result of the experiment to inquire the effect of 9% hydfluoric acid on the porcelain surface to shear bond strength using composite resins, the data as depicted in table 1 are gotten:

 Table 1.
 Mean and standar deviation as the results of shear bond strength measurement between composite resins materials and the porcelains undergo acid etching and without acid etching (N/mm<sup>2</sup>)

Λ	SD
245.77	± 24.28
148.42	$\pm 13.12$

Notes:

X : Mean, N: Numbers of samples, SD: Standard Deviation

The result of the experiment of treated group I gave a mean value of 245.77 and the deviation standard is  $\pm$  24.28; the treated group II gave a mean value of 148.42 and the deviation standard is  $\pm$  13.12. Before the deviation standard test between both treated groups was done, first, normal distribution test using Kolmogorov Smirnov test has been given to each group to see the data distribution in each group. From the Kolmogorov Smirnov test done to all groups, a value of p > 0.05 was got, this result showed that all groups distribute normally. Next, t test was done to see the difference of the shear strength in treated group I and treated group II. T-test results in a p value of 0.0001 (p  $\geq$  0.05), which means there was a significant different of shear bond strength between treated group I and II.

## DISCUSSION

This experiment was done to find out if 9% hydrofluoric acid etching on porcelain surfaces would cause an increase in the composite resin's shear bond strength towards the porcelains without acid etching. After having been acid etched or without acid etching the porcelain surfaces were smeared two times using silane solution that will increase the shear bond strength, and this is an important step which cannot be passed up in the procedure of fusing composite resins to porcelain.

Some test can be used to evaluate the shear bond strength between porcelain and composite resins, such as flexural test, shear test, tensile test. Each way of assessments has its own advantages and disadvantages and some writers state that shear test is the most adequate one to measure shear bond strength between two materials.<sup>4–6,8,9,13</sup> This kind of test is done through the potentials which directly develop on the surfaces of two materials which were going to be tested. The test on the shear bond strength with flat surface may direct most of the potential pressure to the fusing surface, without any influences of elasticity modules of the tested material as what happens in flexural test.<sup>5</sup> In this kind of test, the testing point has directed precisely to the surface of the material so that the oblique strength generated during the test could be minimized. It was appropriate with the research which stated that parallel directing of the strength to the unification field between two materials tested may minimize cohesive failure and the test result tends to result in adhesive failure.<sup>13</sup>

From the result of this experiment we get statistically significant differences on 95% trust in group I and II. This fact showed that the kind of surface treatment on porcelain determine the value of composite resin shear bond strength that will be got. The highest shear bond strength was gotten in treatment group I, which was suitable with the result of the experiment from another reseacher,<sup>13</sup> who did the experiment on micro shear bond strength, it might be because group I had two kinds of bonds between porcelain and composite resin were micromechanical bond and chemical bond, while in group II the bond was just chemically.

Another researcher said that hydrofluoric acid which is smeared to the surface of the porcelain may react with the glass/silica phase which form the porcelain. The result of the reaction which is  $H_2[SiF_6]_{(aq)}$  becomes dissolved and generating micro pores porcelain surface.<sup>11</sup> There is a research which promotes the micro pores of the material surface will cause the widening of the surface of the contact and it will cause a micromechanical interlocking between porcelain and composite resin.<sup>16</sup> Many kinds of acid solution can be used to etch a porcelain, but according to other researcher that comparison between the effect of the kinds of acid etching on porcelain surface, hydrofluoric acid showed the most effective result.<sup>16</sup>

The application of silane solution on the porcelain surface will result in chemical bond between porcelain and composite resin. A previous research said that silane solution functions as a promoter bonding which triggers chemical bonding between organic and inorganic surfaces. The fusion of composite resin on porcelain happens because of polymerization reaction between dimethacrylate cluster on resin matrix and the cluster methacryloxypropyl silane solution during composite resin curing process and through condensation reaction between hydroxyl (Si-OH) on the molecules of porcelain silica, with alcoxy cluster on hydrolysed cylanol molecule. This reaction generates cyloxane cluster (Si-O-Si) and water molecule (H<sub>2</sub>O) as the last product.<sup>16,17</sup>

The clean and dry surface are needed in forming optimum fusion because after the surface of the material is cleaned, the surface energy will increase and result in the surface of the material will be easier to absorb adhesive material,<sup>2</sup> and that is why the surface of the porcelain needs to be washed with aquadest and needs to be dried using air blow. Another researcher also said that silane solution also helps the bonding of porcelain and composite resin because there is an increase of the wetting of the porcelain surface.<sup>16,17</sup> The wetting on porcelain is proved with the small size of the contact angle of the surface and that the silane through out all part of the porcelain surface, including to the micro pores which has been formed because of acid etching.<sup>2</sup>

The use of 9% hydrofluoric acid is proved to be able to increase the shear bond strength between nano filler composite resin with porcelain, but because of the irritating characteristic of hydrofluoric acid, when it is used intra orally, optimal isolation method must be done to protect soft tissues,<sup>3</sup> and air vacuum needs to be used to lessen steam which might be inhaled during the etching process. If an open dentin is found, using silane solution without hydrofluoric acid etching is enough, because 9% hydrofluoric acid has an irritating effect on dentin.<sup>17</sup>

The conclusion of this study is the use of 9% hydrofluoric acid on the surface of porcelain can increase the shear bond strength between porcelain and nano filler composite resin.

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