



## Original Article

# The Effect of Drying Time of Silane Coupling Agent on the Hardness of Fiber-Reinforced Composite for Dental Applications

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## ABSTRACT

Treatment of tooth decay includes caries or pulp necrosis, necessitates crown restoration and the creation of a post. Non-metallic pegs, one of which is fiber-reinforced composite (FRC), have become increasingly popular in recent years. FRC is a combination of composite with fiber that is bound by a silane coupling agent. The addition of silane serves several purposes, one of which is to strengthen the bond between the fiber and the composite. The chemical reactions of silane coupling agents are divided into two stages: hydrolysis and condensation reactions. The drying time of the silane coupling agent has an effect on the success of the hydrolysis reaction. Good fiber and composite bonds can improve the mechanical properties of fiber-reinforced composites, including the hardness of FRC. This research aims to determine the effect of silane coupling agent drying time on the hardness of FRC. This research is a pure laboratory experiment, with a sample amounted to 24, and was divided into four treatment groups based on drying time, namely group A 15 seconds, group B 30 seconds, group C 60 s, and group D 120 s. The sample was in the form of a disk with dimensions of 5 mm in diameter and 2 mm in height. The Vickers test was used for testing the sample. The results showed that group C had the highest hardness value of 87.33 Mpa with a drying time of 60 s. Statistical tests using the One-Way ANOVA and LSD tests revealed a difference in influence between groups with a significance value of  $p=0.000$  ( $p<0.05$ ). There is an effect of drying time of silane coupling agent on the hardness value of FRC with the highest hardness value at drying time of 60 s.

## GRAPHICAL ABSTRACT



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## Introduction

Dental and oral disease is an essential issue of the health sector in general; however, in developing countries, there is a tendency to increase dental and oral diseases, one of which is dental caries, which, if untreated, can lead to tooth necrosis [1–10]. Treatment of tooth decay, such as caries or pulp necrosis, necessitates crown restoration and the creation of a post [11]. Non-metallic pegs, one of which is fiber-reinforced composite (FRC), have become increasingly popular in recent years [12]. FRC posts have good aesthetic properties, a modulus of elasticity, compressive strength, a coefficient of thermal expansion nearly equal to dentin, better flexibility than metal, are not susceptible to corrosion, can reduce the risk of fracture, and can be easily removed from the root canal with an ultrasonic device [13–15]. Fiber-reinforced composite (FRC) is a material made up of a composite resin reinforced with fiber using a silane coupling agent [16].

A silane coupling agent is a silicon-based chemical substance [17, 18]. The use of a silane coupling agent that functions as a binder on the FRC can change the surface of the composite from hydrophilic to hydrophobic, which will have an impact on increasing the adhesion and mechanical strength of the composite material. Furthermore, the silane coupling agent has physical and chemical properties that increase the strength of the bonding and prevent interfacial debonding during composite use [19]. Silane coupling agents, in general, work at interfaces derived from organic and inorganic materials with the goal of bonding the two dissimilar materials [20]. The silane coupling agent chemical reaction process is divided into hydrolysis and condensation.

The hydrolysis process initiates the silane coupling agent's mechanism of action. A rapid and reversible protonation of the alkoxy silane group occurs during the hydrolysis process, resulting in a bimolecular nucleophilic substitution reaction on silicon [21]. The next step is the condensation process. The shape of the silanol molecule will change during the condensation process into an oligomer [22].

A silane coupling agent is a technique-sensitive material. The drying time is one of several factors that can influence the working mechanism of the silane coupling agent. The drying time of the silane coupling agent is critical because it affects the quality of the resulting FRC bond, which can directly impact mechanical strength, one of which is hardness. During the drying process, a reaction between the organofunctional groups and the functional groups of the silane coupling agent will form and strengthen the bond at the interface of the composite resin and fiber [22]. A shorter drying time of the silane coupling agent will result in excess water material, which will result in excessive polymerization in the solvent phase [23]. Water absorption in the polymer may result in an expansion pressure between the composite matrix and the fiber. This condition will cause the release of components from the fiber or the rest of the monomer, affecting the cytotoxicity. High water absorption can reduce the mechanical strength, one of which is the material's hardness [24, 25]. A longer drying time causes the FRC to lose water, which affects the formation of an imperfect monolayer. Then, it will influence the mechanical strength of the material, one of which is hardness [26]. Based on this background, this research aims to determine the effect of silane coupling agent drying time on fiber-reinforced composites' hardness.

## Methodology

This research is a pure laboratory experiment with a post-test only control group design. There was a total of 24 fiber-reinforced composite samples. The materials used included E glass fiber, flowable nanohybrid composite resin, and silane coupling agent. The sample was created in the shape of a disk 5mm in diameter and 2 mm in height. The samples were divided into four treatment groups: group A for silane coupling agent drying time of 15 s, group B for silane coupling agent drying time of 30 s, group C for silane coupling agent drying time of 60 s, and group D for silane coupling agent drying time of 120 s.

The sample was prepared by cutting the fiber along 5 mm with fiber scissors and then weighing it to standardize the fiber weight, which is 0.56

mg. The fiber was placed on a glass plate, then silane coupling agents were applied using a micropipette with a silane volume of 0.58 l, and the fiber was allowed to stand for 60 s before drying for 15 s with an electric fan at a distance of 30 cm for group A. The next step was to fill half of the mold with composite resin. Using fiber tweezers, silane-treated fiber was placed on top of the composite. After reapplying the composite resin to the fiber until the mold was full, the FRC surface was covered with a celluloid strip and irradiated for 20 s. If there was excess FRC in the mold, it was removed with a polishing disk. The steps were repeated for the other sample groups, with the only difference being a 30 s drying time for group B, a 60 second drying time for group C, and a 120 s drying time for group D.

The sample was then placed in a conical tube filled with distilled water and stored for 24 h in an incubator at 37 oC. The sample was removed and dried with absorbent paper for 10 s before

being tested for hardness with a Micro Vickers Hardness Tester. The sample was placed on the test table by clamping the sample with a clamp on the Micro Vickers Hardness Tester table. Turn the knob with the load used of 100 gr to select the appropriate test load, then set the dwell time to 20 s. Press the penetrator button; the diamond penetrator will immediately descend, as indicated by a green light; after 20 s, the diamond penetrator will ascend, obtaining the diameter value from the sample; the data is then entered into the formula "HV ="  $1,8544 P/d^2$  where Hv is the Vicker hardness number, p is the load (N), and d is the average diagonal (1/1000 mm). Each group's mean and standard deviation were compared and analyzed using one-way ANOVA followed by the LSD test ( $p < 0.05$ ).

## Results and Discussion

Table 1 displays the mean and standard deviation of the hardness of each group:

**Table 1:** The mean values and standard deviation of FRC hardness

Treatment Group	N	Mean $\pm$ standard deviation
Group A	6	53.05 $\pm$ 5.81
Group B	6	67.82 $\pm$ 3.65
Group C	6	87.3 $\pm$ 9.46
Group D	6	23.69 $\pm$ 7.86

As seen in Table 1, the group with the drying time of 120 s has the lowest fiber-reinforced composite hardness value (23.69 7.86 Mpa), while the group with the silane coupling agent drying time of 60 s has the highest fiber-reinforced composite hardness value (87.3 9.46 MPa).

The one-way ANOVA test results (Sig= 0.000) show a significance value of  $p=0.000$  ( $p < 0.05$ ). From these results, it is obvious that the drying

time of the silane coupling agent has an effect on the hardness of the fiber-reinforced composite.

The data was then analyzed using a Post Hoc analysis test with the least significant difference (LSD) test to determine the difference in FRC hardness between groups. Table 2 displays the results of the LSD test.

**Table 2:** The results of the Least Significant Different (LSD) test

Drying Time	15 Seconds	30 Seconds	60 Seconds	120 Seconds
Group A		0.002	0.000	0.000
Group B	0.002		0.000	0.000
Group C	0.000	0.000		0.000
Group D	0.000	0.000	0.000	

Table 2 shows that there is a significant difference ( $p < 0.05$ ) between groups in the drying time of the silane coupling agent for 15 s, 30 s, 60 s, and 120 s.

A silane coupling agent is a silicon-based chemical [13]. The silane coupling agent used in this research contains about 2.5 % organ silane, which reacts with cellulose on the surface of the

fiber and the surface of the composite resin [27], approximately 2.5 % phosphoric acid ester methacrylate, which is the active ingredient in the silane coupling agent, which serves to bind the composite resin and fiber, and 50-100 % ethanol, which is a solvent for organic and inorganic materials [28, 29].

The silane's mechanism of action begins when the silane coupling agent is applied to the fiber surface, causing the silane coupling agent to react with air, resulting in three layers on the silane: the outermost layer, the intermediate, and the innermost layer. Many oligomers exist in the outermost and intermediate layers, which can weaken the bond. The innermost layer is cross-linked with strong siloxane bonds. An optimal adhesion bond on the fiber requires only one layer, namely the innermost layer. The oligomers in the outermost and intermediate layers will be adsorbed to the deepest layer and dissolved during the silane drying process. The drying time of the silane coupling agent has a strong influence on its drying process. If the drying time is too short, the adsorption of the outermost and intermediate layers will be imperfect, weakening the bonds formed by the silane [30].

Silanization is a reaction consisting of two stages, called hydrolysis and condensation [31]. The hydrolysis process occurs at the beginning of the silane coupling agent drying process, where the hydroxyl groups on the fiber surface react with the silane groups in the silanes to form alkoxy silane groups. Silanes are converted to silanols by the formation of alkoxy silane groups, which is a rapid and reversible protonation of alkoxy silane groups followed by bimolecular nucleophilic substitution on silicon [32]. Every reaction in the alkoxy silane group necessitates the use of the appropriate solvent or water, with one alkoxy silane group necessitating the use of three molecules of water or solvent. The alkoxy silane group will change from a monoalkoxy silane group to a di group and a trialkoxy silane group during the hydrolysis process. This trialkoxy silane group contributes significantly to the adhesion strength of the fiber and composite resins in FRC [33]. The process of changing the group occurs concurrently with the

physical change of the silane coupling agent, which is liquid, becoming viscous or denser. Because the solvent or water at this stage serves to maintain hydrolytic conditions and regulate the solvent or water needed for the group alkoxy silane to react, the drying time plays an important role in the hydrolysis process in regulating the level of solvent or water contained in the silane coupling agent. The next process is the condensation process. The silanol molecules will change shape during the condensation process and become oligomers. Strong chemical bonds, known as covalent bonds, are formed during this process [34].

The length of the drying time of the silane coupling agent is proportional to the decrease in the solvent or water content of the material, which is followed by the formation of more and more bonds between the composite resin and the fiber [35]. A high solvent or water content in an FRC material will cause swelling, an increase in weight, an increase in solubility value, and a decrease in mechanical strength, one of which is the hardness of FRC [36], on the other hand, if the drying time is too long, the silane coupling agent will run out of solvent or water, resulting in a reduction in the bond strength formed between the composite resin and fiber due to imperfect monolayer formation on the FRC. If the bond between the fiber and the composite resin becomes too dry, it will be easily broken or brittle, resulting in a decrease in mechanical strength, one of which is the hardness of FRC.

At a sample thickness of 4 mm, fiber-reinforced composite has an ideal surface hardness value of more than 50 VHN and a ratio of more than 80% [37]. The hardness of the fiber-reinforced composite is proportional to the quality of the bond formed between the composite resin and the fiber, which is directly affected by the silane or water solvent content of the material [38, 39].

## Conclusions

In dentistry, FRCs are increasingly being used to replace metallic restorations like as periodontal splints, fixed partial dentures, endodontic posts, orthodontic appliances, and other indirect restorations. The followings are the main results of this research:

1. The drying time of the silane coupling agent affects the hardness of the fiber-reinforced composite.
2. The highest hardness value is found in the sample group with a drying time of 60 s.

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### Authors' contributions

All authors contributed toward data analysis, drafting and revising the paper and agreed to be responsible for all the aspects of this work.

### Conflict of Interest

We have no conflicts of interest to disclose.

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