Effect of salivary pH on diametral tensile strength of glass ionomer cement coated with coating agent

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Abstract. The aim of this study was to analyze the effect of salivary pH to diametral tensile strength of glass ionomer cement (GIC) coated with a coating agent. GIC specimens coated with varnish and nano-filled coating agent were stored in artificial saliva at pH values of 4.5, 5.5, and 7 for 24 h at 37°C, then the diametral tensile strength was tested by universal testing machine. Results showed that there was no significant difference in the diametral tensile strength of the GIC coated with varnish and nano-filled coating agent with decreasing of salivary pH (p < 0.05). It can be concluded that salivary pH does not affect the diametral tensile strength of GIC coated by varnish or nano-filled coating agent

1. Introduction
Caries is a disease characterized by loss of tooth structure due to acid produced by microorganisms in dental plaque. One of the most important factors in the process of caries occurrence is the presence of saliva. Saliva acts as a buffer that can maintain the pH of bacteria-generated acids in the oral cavity. The process of caries occurrence begins from bacteria in the plaque that metabolize carbohydrates into acids that cause salivary pH decrease. If the saliva pH reaches 5.5, the dental hydroxyapatite crystals will dissolve and cause tooth demineralization. If a fluoride ion is present in the tooth structure, then the ion replace the hydroxyapatite arrangement into a fluorapatite that is more resistant to acid so that demineralization does not continue. However, if the saliva fails to neutralize the acid, the pH decrease will continue, and if the saliva pH reaches 4.5, then the fluorapatite will dissolve causing demineralization to continue. Demineralization that is not compensated by remineralization will cause caries. Therefore, salivary pH is an important factor in the occurrence of caries, the lower the saliva pH the higher the caries risk [1]. Glass ionomer cement (GIC) is one of the most appropriate restorative materials for individuals with high caries risk because GIC can release fluorides that can prevent the occurrence of caries. As a restorative material, GIC is also widely used because of its many advantages, including chemical binding to the tooth structure, good biocompatibility, and other dental restoration properties such as strength and toughness [2]. However, GIC also has a disadvantage of being vulnerable to the water at the beginning of hardening to maturation. Contamination with water will change the physical and mechanical properties of GIC because water can dissolve the aluminum and calcium ions that needed in the acid-base reactions, thus disrupting the formation of the matrix and maturation of the GIC [3]. Therefore, a material that can protect GIC during the period of hardening to maturation called a coating agent is used. The coating agent serves to maintain the water balance in the GIC and maintain the stability of the GIC color. Various kinds of coating agents have been developed, including varnish and nano-filled coating agent.
Clinically, many factors can affect the restoration resistance in the oral cavity, one of which is contact with acid. Individuals with a high caries risk tend to have a low salivary pH. Saliva with a low pH makes the mouth become acidic. Acid can decrease the physical and mechanical properties of a non-completely maturated GIC. Therefore, the selection of the coating agent is very important. This coating agent must be resistant to and not easily soluble in acids. Solubility of the coating agent before the GIC is fully maturated will cause a decrease in the physical and mechanical properties of the GIC. Deterioration of the physical and mechanical properties of GIC can be identified by measuring the GIC diametral tensile strength. This measurement is important to determine the tensile strength of GIC in accepting mastication loads [4]. Therefore, this study aims to determine the effect of artificial saliva with pH values of 4.5, 5.5, and 7 on the diametral tensile strength of GIC coated with varnish and nano-filled coating agent. Immersion of GIC with the coating agent in artificial saliva pH 4.5 and 5.5 will simulate the restoration situation in individual oral cavity with high caries risk, because these pH values represent of the critical pH for fluorapatite and hydroxyapatite crystals. Conversely, immersion of the GIC with the coating agent in saliva with pH 7 will simulate the restoration in the oral cavity of individuals with low caries risk. The results of this study are expected to provide consideration for the clinicians in the selection of GIC coating agents for individuals with low salivary pH.

2. Materials and Methods
This laboratory experimental study begins with the manufacture of GIC specimens. The mould was initially coated with thin silicon oil. The powder and GIC solution were weighed to determine the ratio of powder and GIC solution. After that, GIC was manipulated according to the obtained ratio of the powder and the solution. After obtaining a homogeneous mixture, it was placed into a mold using plastic filling. The top surface of the GIC was covered in mylar plastic, and the glass slab was placed on top of it. A load was placed on top of the glass slab to remove the excess of the GIC, leveling the specimen height with the mold and producing a smooth surface.

After the sixth minute, specimens from groups 1, 2, and 3 were removed from the mold, and varnish was coated on the entire surface using a micro brush, while specimens from groups 4, 5, and 6 were removed from the mold and the nano-filled coating agent was applied on the entire surface using a microtip applicator, after which the specimens were irradiated With LED light for 20 seconds on the entire surface. All groups of specimens were removed from the mold and soaked in artificial saliva in plastic pots. Groups 1 and 4 were immersed in artificial saliva with pH = 7. Groups 2 and 5 were immersed in artificial saliva with pH = 5.5. Groups 3 and 6 were immersed in artificial saliva with pH = 4.5. All specimens were then placed in an incubator at 37 °C for 24 h. After being soaked in artificial saliva for 24 h, each specimen’s diametral tensile strength was measured using a Shimadzu Universal Testing Machine with a compression load of 100 Kgf and a test speed of 0.5 mm/min [5].

A one way ANOVA statistical test was used to compare the mean values of the diametral tensile strength of GIC in the varnish-coated GIC group for immersion in artificial saliva with pH of 4.5, 5.5, and 7, followed by Post-Hoc Tukey test with significance \( \alpha = 0.05 \). Data analysis was performed using SPSS V.20. The Kruskal Wallis test was carried out to compare the mean value of the diametral tensile strength of GIC in the GIC groups with nano-filled coating agent for immersion in artificial saliva with pH of 4.5, 5.5, and 7 with significance \( \alpha = 0.05 \). Then, the independent t-test and Mann–Whitney test were carried out to compare the average diametral tensile strength values between the varnish-coated GIC group and the GIC group coated with nano-filled coating agent for immersion in each artificial saliva pH, with significance \( \alpha = 0.05 \).

3. Results and Discussion
3.1 Results
The results of the diametral tensile strength of GIC coated with varnish or the nano-filled coating agent for immersion in artificial saliva with pH of 4.5, 5.5, and 7 are shown in Table 1.
Table 1. Mean diametral tensile strength values of GIC coated with different coating agents after immersion in artificial saliva with different pH values

| Coating agent          | Mean Diametral Tensile Strength Value (MPa) ± SD | Sig.  |
|------------------------|-----------------------------------------------|-------|
|                        | pH 7            | pH 5.5           | pH 4.5 |       |
| Varnish                | 11.07 ± 1.76    | 9.12 ± 1.14      | 11.74 ± 1.68 | 0.028 |
| Nano-filled coating agent | 10.89 ± 1.61    | 11.33 ± 1.45     | 11.39 ± 1.69 | 0.895 |
| Sig                    | 0.818           | 0.009            | 0.727     |

After the distribution and homogeneity of the data were tested, it obtained the results of data distribution normal and homogeneous in the varnish coated specimen group, so one way ANOVA test used in order to see the change in the diametral tensile strength values of the specimen group in artificial saliva with pH 7, 5.5, and 4.5. The average significance value of the diametral tensile strength was obtained at 0.028 or p < 0.05. It can be interpreted that the mean diametral tensile strength value in the varnish-coated specimen group has a significant difference. The post hoc Tukey test was then performed to observe the significance of the diametral tensile strength values between the specimen groups. The results showed that there was significant difference only in the average diametral tensile strength between the specimen groups with artificial saliva immersion at pH 5.5 and 4.5, with p < 0.05. Furthermore, in the group of specimens coated with the nano-filled coating agent, the results showed abnormal data distribution but homogeneous data, so the Kruskal–Wallis statistical test was carried out to observe the significance of the diametral tensile strength change values between the specimen groups. The mean significance value of the diametral tensile strength was obtained at 0.895 or p > 0.05. It can be interpreted that the average diametral tensile strength value in the group of specimens coated with the nano-filled coating agent has a not significant difference.

The Mann–Whitney statistical tests were then performed to determine the mean value of the diametral tensile strength for the varnish-coated group and the group with nano-filled coating agent for artificial saliva immersion with pH 7. The mean significance value of the diametral tensile strength was 0.818 or p > 0.05. It can be interpreted that the average diametral tensile strength value in the varnish-coated specimen group and the specimen group with nano-filled coating agent for artificial saliva immersion with pH 7 has anot significant difference. Furthermore, for the specimen group with artificial saliva immersion at pH 5.5, the Mann–Whitney statistical test was carried out to determine the mean value of the diametral tensile strength for the varnish-coated specimen group and the specimen group coated with the nano-filled coating agent. The mean significance value of the diametral tensile strength is obtained at 0.009 or p < 0.05. It can be interpreted that the average diametral tensile strength value in the varnish-coated group and the group with nano-filled coating agent with artificial saliva immersion at pH 5.5 has a significant difference. In the group with artificial saliva immersion at pH 4.5, the independent t test was performed. The average significance value of diametral tensile strength is obtained at 0.727 or p > 0.05. It can be interpreted that the average diametral tensile strength value in the varnish-coated specimen group and the group coated with the nano-filled coating agent with artificial saliva immersion at pH 4.5 has a not significant difference.

3.2 Discussion

In this research, the mean value of diametral tensile strength of GIC GC Fuji II after soaking in artificial saliva with pH of 4.5, 5.5, and 7 for 24 h is 9.12–11.74 MPa. The results are not different than those obtained in a study by Moshaverinia et al. (2008) who measured the diametral tensile strength test of GIC GC Fuji II by soaking in aquadest, and found average diametral tensile strength values of 11.8 ± 2.4 MPa [6]. This result corresponds to the diametral tensile strength of GIC type II of 7–14 MPa [7]. Thus, it can be said that the immersion medium does not affect the diametral tensile
strength of GIC. The results in Table 1 show that there is a fluctuation in the diametral tensile strength value of varnish-coated GIC immersed in artificial saliva with pH of 7, 5.5, and 4.5. Significant differences were found in the group immersed in artificial saliva with pH of 5.5 and 4.5. The diametral tensile strength for the group immersed in artificial saliva with pH of 4.5 was significantly higher than that of the group immersed in artificial saliva with pH of 5.5. This may be due to GIC rehydration. Research conducted by Blenet et al. (2014) states that GIC will shrinkage when hardened that causes a deterioration of the physical and mechanical properties of GIC. Therefore, water absorption is required in order to compensate for the shrinkage [8]. The lower the salivary pH, the more the dissolution of hydrogen ions is, so the GIC will absorb more water for rehydration. The use of the coating agent can prevent rehydration, but varnish contains volatile materials that may facilitate the occurrence of GIC rehydration. Although there is a significant difference, the diametral tensile strength value in the varnish-coated GIC group of 9.12–11.74 MPa is still in line with the diametral tensile strength value for GIC type II 7–14 MPa. This illustrates that the varnish is sufficiently good to tolerate the acidic environment. This statement is supported by research conducted by Khusla et al., which states that varnish applications can protect GIC particles from dissolution or degradation due to APF gel exposure at pH 3.5 [9]. Varnish resistance to acidic is maybe due to varnish content in the form of vinyl chloride and vinyl copolymers acetate, which is an organic compound that can only dissolve in organic solvents such as ether, acetone, or chloroform [10]. The APF gel in the Khosla et al. (2014) study and the artificial saliva solution in this study both contain inorganic compounds that cause the varnish to be insoluble. In addition, the nature of varnish, which is only physically attached to GIC, causes the varnish not to dissolve in the saliva, but the varnish will dissolve if there is a mechanical force causing the layer to be removed from the restoration surface [11].

Conversely, in the group of specimens coated with the nano-filled coating agent, there was no significant difference in the mean value of the diametral tensile strength of GIC on immersion in saliva with pH of 7, 5.5, and 4.5. In other words, it can be concluded that saliva pH has no effect on the nano-filled coating agent. Similar results were also reported by Reddy D (2014) in his research stating that GIC coated with the nano-filled coating agent did not experience significant differences in roughness after immersion in citric acid solution with different pH values (2, 3, 4, 5, 6, and 7) compared with the roughness of GIC coated with petroleum jelly, which significantly decreased with pH = 12. This is because the nano-filled coating agent has a 40-nm filler that is developed in a single dispersion. The single dispersion of the nano-filler is spread evenly on the surface of the restoration, thus enhancing the wear resistance of the restoration and increasing the hardness and resistance to acids [12]. If a ratio of diametral tensile strength between the varnish and the nano-filled coating agent group is measured for each saliva immersion, it is found that the diametral tensile strength of the GIC group coated with the nano-filled coating agent is more stable compared to that of the varnish-coated GIC. This is possibly because the nano-filled coating agent has a higher final thickness of 35–40 μm compared with the final thickness of varnish of 5–10 μm, and hence, the nano-filled coating agent can better protect the restoration surface compared to varnish [11,13]. In addition, the nano-filled coating agent also has infiltration capabilities into the underlying restoration material, so as to provide internal protection from cracks and avoid increased fracture tendencies [13]. Based on research conducted by Bonifacio, the nano-filled coating agent infiltration produces mechanical interlocking between the nano-filled coating agent and the GIC, so it can increase the resistance of fracture [14]. Earl et al. and Hotta et al. in Saleh's (1994) study indicated that light-cured resin coating agent is the best coating agent for limiting the outflow of water in GIC and for maintaining the stability of the GIC color [15]. Research conducted by Eh Kato et al. (2008) on the influence of various coatings on flexural strength and tensile bond strength of GIC also showed similar results. The results obtained indicate that GIC coated by the nano-filled coating agent has the highest flexural strength and tensile bond strength compared to GIC not coated with a protective material or coated with other protective materials, in this case Fuji varnish and Fuji COAT LC [16]. However, although the diametral tensile strength of GIC coated with the nano-filled coating agent is more stable and constant than that of the varnish-coated GIC, there is generally no significant difference between the diametral tensile strength values
of the two groups. Therefore, it can be concluded that saliva pH does not affect the diametral tensile strengths of the GIC coated with varnish and the nano-filled coating agent, and hence, both varnish and nano-filled coating agent can be used as coating agents for individuals with high caries risk.

4. Conclusion
Based on the results of this study, it can be concluded that saliva pH does not affect the diametral tensile strength of GIC coated with varnish or nano-filled coating agent, and thus, both coating agents can be used as GIC restoration coating agents on individuals with high caries risk.

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