Effect on diametral pull strength of applying a nanofiller coating agent to glass ionomer cement contaminated with artificial saliva

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Abstract. Glass ionomer cement (GIC) is very sensitive to contact with intraoral fluids before it sets. The aim of this study was to determine how the diametral tensile strength (DTS) of GIC that is either contaminated or uncontaminated with artificial saliva is affected by applying a nanofiller coating agent (NCA). The GIC used was Fuji IX Extra SIK comprising a powder and a liquid, and the NCA used was G-Coat Plus. There were six groups of GIC restoration with six of each specimen soaked in aquabides for 1 h, 1 d, and 1 wk at 37°C. Test statistical analysis using nonpaired t-test. The results obtained at 1 h immersion ($p = 0.051$) and 1 d immersion ($p = 0.528$) ($p > 0.05$), which means no significant difference, whereas in 1 wk $p = 0.030$ ($p < 0.05$), which means there are significant differences. Applying an NCA to GIC contaminated with artificial saliva, until one day, may provide a nonsignificant change in DTS.

1. Introduction

Glass ionomer cement (GIC) is a restorative material that is used by most dentists because it requires minimal preparation, bonds chemically with dental tissues, releases fluoride ions in the long term, and is esthetic, biocompatible, weakly soluble, transparent, and antibacterial [1]. GIC comprises acid-soluble calcium fluoroaluminosilicate glass in the form of powder and polyacrylic acid in the form of liquid, and it sets in 6–8 min from when the two are mixed. The reaction in which GIC sets and hardens involves the transfer of metal ions, calcium, and aluminum to the polyacrylic acid, which causes the liquid phase to become a gel. During the transfer process, the matrix that forms the metal ions is soluble and therefore susceptible to fluid ingress [2]. This makes GIC deficient compared to other materials because of its brittleness.

Water plays an important role during hardening: excess water absorption from the oral cavity can alter the physical properties of GIC [3]. As a liquid, saliva in the oral cavity can contaminate GIC during hardening; during that 24-hour period, GIC is sensitive to saliva and therefore some protection should be used to avoid the saliva contamination (SC) that causes GIC to dissolve and adhere less to the teeth. GIC is also susceptible to water loss sometime after filling; if left unprotected and exposed to the air, its surface will crack because of desiccation. Furthermore, desiccation and water contamination can continue to alter the structure of GIC for several weeks after cessation. Therefore, in order to obtain the best results while GIC hardens, it must be protected with an effective waterproof...
insulating material to avoid being contaminated by saliva and air [4]. Nanofiller coating agents (NCAs) represent a new type of material that offers excellent protection and has a single-dispersion technology. Evenly dispersed nanofiller particles increase wear resistance, prevent abrasion, and discoloration, and provide resistance to coated restorative materials such as GIC [5]. A characteristic of GIC is that it can see the effect of dissolution and decrease the drop in adhesion force (known as the diametral tensile strength, DTS) due to SC, that is, against force causing the material to stretch or lengthen before eventually breaking. This characteristic affects the strength of the GIC when receiving mastication loads [2]. Furthermore, this diametral tensile test is an easy and common way of measuring the tensile strengths of brittle materials such as GIC.

There appears to have been no research on how coatings affect the DTS of GIC that is either contaminated or uncontaminated with artificial saliva. Most previous studies compared the effects of different coating types and assessed the surface hardness of GIC after being coated. In fact, when using GIC for restoration in the oral cavity, many aspects (e.g., SC and moisture) affect the DTS. Therefore, the aim of this study was to analyze how coatings affect the DTS of GIC that is either contaminated or uncontaminated with artificial saliva.

2. Methods
The specimens were coated after 30 min and then tested after immersion for 1 hour, 24 hours, and 1 week; the controlled variables are temperature, humidity, artificial saliva, pH, how to manipulate the GIC, duration of lighting G-Coat Plus coating, and the universal mechanical testing machine.

Each specimen comprised Fuji IX Extra GIC placed into a 6 mm diameter stainless-steel mold with a thickness of 3 mm. A total of 36 specimens were divided into six groups. Groups 1, 3, and 5 contained specimens contaminated by artificial saliva at 2 min over 20 s for 10 s before setting time, followed by coating at 30 min; the specimens were then well immersed in aquabides at 37°C for 1 h, 1 d, and 1 wk, and then tested for DTS. Groups 2, 4, and 6 contained specimens not contaminated by artificial saliva; they were coated at 30 min, taken from the mold, immersed in aquabides at 37°C for 1 h, 1 d, and 1 wk, and then tested for DTS.

The tools used in this research were a stainless-steel mold (diameter: 6 mm; thickness: 3 mm), a plastic spatula/plastic filling, Mylar strips, glass preparation, impermeable paper, gloves, a mask, a 2 cc syringe, an incubator (WT Binder, Brand Heraeus, Germany), and a Shimadzu Automotive AG Mechanical Testing Machine 5000 E. The materials used were Fuji IX Extra glass ionomer, G-Coat Plus NCA, and artificial saliva.

The sequence of preparing the samples was as follows. First, the GIC was stirred on a paper pad with a 1:1 ratio of powder to liquid (according to the manufacturer’s recommendation); due to the fact that the size and diameter of the mold were large, a 2:2 ratio was used. The entire mass of GIC was then placed in the stainless-steel mold, and all 36 specimens were covered first with Mylar strips and then with preparatory glass that was pressed down manually for 20 s. The specimens were divided into six treatment groups of six specimens each. They were immersed in aquabides at 37°C in the incubator and then measured diametrically of the GIC using the universal mechanical testing machine for three different immersion times, namely, 1 h, 1 d, and 1 wk. Data of measured diametrically of the GIC ionomer were analyzed statistically with an unpaired t-test. The tested values were (i) the average diameter glass tensile magnetic strength between the SC groups and the non-SC groups and (ii) the comparison of the average values of the intergroup specimens.

3. Results
The results were obtained after calculating the DTS test with the final unit of MPa, that is, GIC (Fuji IX Extra SIK) contaminated with artificial saliva and which without artificial SC was given application of NCA, and stored in aquabides for 1 h, 1 d, and 1 wk. From the research, we get the average DTS values of GIC as given in Table 1.
Table 1. Average DTS (unit: MPa) of GIC with coating applied.

| Long immersion | Average DTS GIC (MPa) | P-value |
|----------------|-----------------------|---------|
|                | Contaminated          | Uncontaminated |
| 1 h            | 15.3 ± 1.7            | 18.9 ± 3.4          | 0.051** |
| 1 d            | 13.1 ± 2.5            | 14.1 ± 2.7          | 0.528** |
| 1 wk           | 11.5 ± 1.9            | 15.8 ± 3.5          | 0.030*  |

**p > 0.05; *p < 0.05

Those GIC specimens that (i) were contaminated with artificial saliva, (ii) had NCA applied, and (iii) were immersed for 1 h had an average DTS of 15.3 ± 1.7 MPa. Those that were not contaminated, had NCA applied, and were immersed for 1 h had an average DTS of 18.9 ± 3.4 MPa. Therefore, contamination with artificial saliva decreased the DTS of the NCA-coated samples immersed for 1 h.

Those GIC specimens that (i) were contaminated with artificial saliva, (ii) had NCA applied, and (iii) were immersed for 1 d had an average DTS of 13.1 ± 2.5 MPa. Those that were not contaminated, had NCA applied, and were immersed for 1 d had an average DTS of 14.1 ± 2.7 MPa. Therefore, contamination with artificial saliva decreased the DTS of the NCA-coated samples immersed for 1 d.

Those GIC specimens that were (i) contaminated with artificial saliva, (ii) had NCA applied, and (iii) were immersed for 1 wk had an average DTS of 11.5 ± 1.9 MPa. Those that were not contaminated, had NCA applied, and were immersed for 1 wk had an average DTS of 15.8 ± 3.5 MPa. Therefore, contamination with artificial saliva decreased the DTS of the NCA-coated samples immersed for 1 wk.

The differences in the average DTS between the SC and non-SC NCA-coated GIC groups for different soaking times (1 h, 1 d, or 1 wk) can be seen more clearly in the bar chart in Fig. 1.

Figure 1. Diagram of DTS differences for SC and non-SC NCA-coated GIC samples for immersion times of 1 h, 1 d, and 1 wk.

The results for the average DTS values of SC and non-SC GIC for immersion times of 1 h, 1 d, and 1 wk were then subjected to statistical analysis in the form of an unpaired t-test, the results of which are given in Table 1. An immersion time of 1 h was associated with a significance of $p = 0.051$ ($p > 0.05$), and an immersion time of 1 d was associated with a significance of $p = 0.528$ ($p > 0.05$), meaning that the DTS values between the SC and non-SC GIC groups showed no significant difference. By contrast, an immersion time of 1 wk was associated with a significance of $p = 0.030$.
(p < 0.05), meaning that the DTS values between the SC and non-SC GIC groups showed a significant difference.

4. Discussion

The results from this research are given in Tables 1 and Figure 1. Immersion in aquabides for 1 h, 1 d, and 1 wk increased the average DTS in each case. For each immersion, the non-SC samples had higher DTS than the SC samples, all of which had been coated with a layer of G-Coat Plus NCA on the entire surface. This may have occurred because the SC samples were damaged in the setting time, thereby lowering the DTS of the GIC. This would accord with the explanation by Zoergiebel (2012) that because one of the basic ingredients of GIC powder is silicate (SiO$_2$), when the powder and liquid are stirred together to form a paste, the powder reacts with polyalkenoic acid or poly(acrylic acid) to form a hydrogel salt. In this early phase, the silica base material is susceptible to solubility due to moisture and SC [6].

According to Nicholson et al. (1992), if the cement comes into contact with intraoral fluid before the former hardens, the ion-forming matrix (Ca and Al) disappears and is replaced by an unfavorable matrix formation [7], while Mojon (1996) adds that this would make low mechanical character [8]. Therefore, conventional GIC is susceptible to moisture contamination during reaction settings.

The unpaired t-test was performed with comparative objectives. In order to see the effect of NCA in protecting the SC GIC compared to non-SC GIC. There is an equation at 1 h immersion and at 1 d immersion, both of which gave p > 0.05. From the cases of 1 h immersion (p = 0.051) and 1 d immersion (p = 0.528), it can be concluded that there is no significantly different value. The DTS of NCA-coated GIC between the contaminated with the noncontaminated with artificial saliva. This is likely due to the self-adhesive monomer of G-Coat Plus with a 40 nm nanoscale filler size lining the entire surface of the GIC specimens. The nanoparticle distribution is homogeneous in the matrix. Nanofiller particles spread evenly throughout the surface so as not to clot. Equitable nanofiller particle dispersion increases wear resistance, prevents abrasion, and discoloration, and provides resistance to the coated restoration material [9].

According to Causton (1982), clinicians should keep the environment stable for newly placed restorations and coat them for at least an hour and better if done on the first day. This is to prevent the absorption of water into the cement, which can decompose the soluble polyacrylate calcium bond. If water absorption occurs at the stage where the formation of the polyacrylate calcium chain is occurring, the unstable divalent calcium polyacrylate bond will be dissolved and there will be a deterioration in the physical and cementitious properties of the cement. Calcium polyacrylate, which is more susceptible to water than aluminum polyacrylate, is more dominant in the newly hardened cement, and therefore there is a need for protection in newly hardened cement [10].

Based on other studies, using a light-cured coating agent can limit the movement of water through the cement surface that has been set. In addition, in 1990, the American Dental Association stated the importance of light-polymerized coating agents to coat conventional GIC restorations [11]. Tanaka et al. (2007) proposed a new restorative concept in the form of an application system that combines the posterior restoration of the tooth using GIC coated with an NCA. It is intended to combine the high hydrophilicity of GIC with the very low viscosity of NCA, thus making a perfect bond over the surface of the SIK. This nanofiller coating also protects the surface of the GIC against wear and tear, so it is important in the first months until the GIC is fully matured and capable of withstanding intraoral pressure [12].

A protective coating of G-Coat Plus allows for the complete maturation of glass ionomer reactions and, with delayed exposure to saliva, creates the possibility of the strongest hardening reaction of glass ionomer restorations. G-Coat Plus infiltration provides internal protection against cracks and reduces microleakage in the restoration of glass ionom cement [13].

It is found that p = 0.03 (p < 0.05), from which it can be concluded that there is a significant difference in the DTS value of GIC in G-Coat Plus coated between contaminated with noncontaminated saliva. This is related to several factors, such as the fact that initial contact between
cement and artificial saliva can cause damage. GIC absorbs water quickly, especially in the initial setting. If the cement is not sufficiently matured, swelling or loss of substance to the oral environment may damage the GIC surface, making it rougher [13]. This causes the 1 week saline-immersion SC group to have a significant mean difference from the equivalent non-SC group.

The subsequent difference occurs because of the increased DTS in the non-SC groups, thus causing significant differences from the SC groups. This relates to the GIC hardening reaction, namely, that perfect setting takes 24 h and that the cement must be protected from saliva in the mouth during that period. Conventional glass ionomer restoration is difficult to manipulate because it is sensitive to moisture absorption and desiccation during initial reaction settings before the material begins to harden. Even in the case of polymerization for resin modified reduced initial sensitivity for moisture, studies have shown that the properties of the material change significantly after exposure to moisture or SC [14]. This process is called maturation failure in GIC due to salivary saltation prior to setting time.

Based on Fig. 1 showing the average difference in DTS between SC and non-SC GIC, the pattern is formed in the SC group and, given the application of NCA, the DTS from soaking for 1 h, 1 d, and 1 wk decreases from 15.3 MPa to 11.5 MPa. This is possibly due to the effect of the application of NCA the longer the immersion can be soluble. There have been no explanations to date regarding how long-lasting and soluble coating agents are, especially NCAs, but the manufacturer’s information says that NCAs can dissolve. As for the non-SC groups, the pattern of average DTS generated for 1 h, 1 d, and 1 wk immersion was decreased for 1 d immersion and increased for 1 wk immersion. The decreased DTS occurs because of the lack of GIC maturation because it only reaches 24 h post initial setting; whereas perfect maturation occurs over 24 h. The increased DTS at 1 wk immersion is possible because the GIC has experienced perfect maturation so that, despite the solubility of the coating, there is no absorption of distilled water into the GIC.

In this research, the specimens were immersed in aquabides and fed into a 37°C incubator to match the conditions in the oral cavity. The water was intended to act as a reaction medium in the release of cations of calcium and aluminum to form cement to react with polyacid to form a polyacrylate matrix. Water also helps in hydrating silica hydrogels thereby forming polyacrylic metal salt. If as it sets the cement loses water because of desiccation, the cementitious reactions will cease [13].

5. Conclusion
This research has shown that applying an NCA to GIC contaminated with artificial saliva can increase the DTS value and has a nonsignificant difference compared to that uncontaminated with artificial saliva.

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