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The effect of prolonged immersion of giomer bulk-fill composite resin on the pH value of artificial saliva and resin surface roughness

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Abstract. This study aimed to determine the effect of immersion time on the surface roughness of Giomer Bulk-Fill composite resin and on the pH value of artificial saliva. Sixty-three specimens were divided into nine groups and immersed in artificial saliva with pH values of 7, 5.5, and 4.5 for 1 hour, 24 hours, and 72 hours at 37 °C. The changes in artificial saliva pH were measured using a pH meter and the surface roughness was measured using a surface roughness tester. Longer immersion time increases the pH of artificial saliva and the surface roughness of Giomer Bulk-Fill composite resin.

1. Introduction

As time passes, composite resins continue to evolve. An aesthetic restorative material made of Bulk-Fill composite resins that can be used to fill anterior or posterior teeth; it is capable of achieving a 4 mm depth of cure [1-3]. One type of resin with unique characteristics is Giomer Bulk-Fill composite resin, a Giomer material (combined glass ionomer and polymer). Glass Ionomer Cement (GIC) derivative content of Giomer Bulk-Fill composite resin filler called S-PRG (Surface Pre-reacted Glass Ionomer). The S-PRG Filler is capable of releasing and retrieving fluorine ions and is also capable of releasing five other ions: sodium, strontium, aluminum, silicate, and borate. The ions generally play a role in acid neutralization when exposure to lactic acid comes from bacteria in plaque. Acid neutralization reduces the likelihood of secondary caries and makes the restoration last longer [4,5]. Research on Giomer composite resin showed an increase from an initial pH of 3.80 of the lactic acid solution to a pH value of 4.57 ± 0.09 after immersion for 1 hour. After soaking for 24 h, the pH increased to 6.05 ± 0.07 [6]. Research revealed higher values for surface roughness of Giomer composite resin in comparison to those of compomers after immersion in beverages having a low pH, namely soft drink and orange juice [7]. Meanwhile, other studies found that the Giomer composites were less hard than nanohybrid composite resins immersed in low-pH beverages: i.e., apple juice, orange juice, soft drink, coffee, and beer [8].

Saliva is a natural protective factor that plays a role in preventing or inhibiting the development of caries. This is because saliva contains Ca^{2+} and HPO_4^{2-} ions that can replace ion loss in the tooth [9]. However, if the saliva pH reaches 5.5, the hydroxyapatite will be demineralized. Therefore, a pH of 5.5 is a critical condition for hydroxyapatite [10]. The critical pH may be surpassed if there is fluorine exposure due to the oral environment, such as exposure to fluorine in toothpaste, food, or drink capable of contributing to the remineralization that would form fluoroapatite. However, if the acidity in the mouth falls to a pH of 4.5, then the fluoroapatite can be demineralized. So a pH of 4.5 is critical

for fluoroapatite. In general, high levels of oral acidity are present in people with high caries risk [9-11]. The potential of Giomer composite resins to increase the pH of the lactic acid solution6 suggests that the Giomer Bulk-Fill composite resin can increase the critical pH for hydroxyapatite and fluoroapatite. Therefore, a study was conducted to determine the ability of Giomer Bulk-Fill composite resins to increase the critical pH for hydroxyapatite and fluoroapatite. In addition, this study was also conducted to determine the effect of critical pH values for hydroxyapatite and fluoroapatite on the surface roughness of Giomer Bulk-Fill composite resin.

2. Materials and Methods

Specimens were prepared using a mold 1 mm thick with a diameter of 15 mm. Before filling, ends of yarn were placed in the mold; then a thin layer of silicon oil was applied on the entire mold surface. Thereafter, the mold was filled with Giomer Bulk-Fill composite resin using a plastic filling instrument and then compacted, and a thread was placed in the interior of the filling. The surface area was coated with a Mylar Strip, and an object was placed on top of the glass. A load was placed on top of the object glass to compact the composite resin. After that, the excess material was discarded. Irradiation was carried out four times on the surface of the specimen. After irradiation, the specimen was released from the mold, and then the initial surface roughness of each specimen was tested before immersion.

Soaking was done in 5 ml of artificial saliva prior to storage in an incubator at 37 °C. After immersion of specimens for 1 hour, 24 hours, and 72 hours in artificial saliva with pH values of 7.5, 5, and 4.5, the pH of the artificial saliva was measured using pH meters and then recorded. A surface roughness test was performed using a surface roughness tester with a cut-off length of 0.8 mm and an evaluation length of 4 mm. Roughness testing was performed before and after immersion [12]. An initial surface roughness test was performed on each specimen. Testing was done five times from different directions. A final surface roughness test was also performed. After immersion of specimens for 1 hour, 24 hours, and 72 hours, in artificial saliva with pH values of 7.5, 5, and 4.5, specimens were washed using an ultrasonic cleaner for 5 minutes. Then each specimen was dried and a final roughness test was performed on it. Testing was done five times from different directions.

3. Results and Discussion

3.1 Results

The mean values for the pH of the artificial saliva after immersion of the Giomer Bulk-Fill composite resin specimens can be seen in Table 1.

Table 1. Mean pH values and their standard deviations of artificial saliva after immersion of Giomer Bulk-Fill composite resin specimens

Immersion Media Artificial Saliva	Immersion Time		
	1 Hour	24 Hour	72 Hour
pH 7.0	7.08±0.02	7.18±0.02	7.69±0.05
pH 5.5	6.07±0.02	6.68±0.02	7.65±0.03
pH 4.5	5.45±0.03	6.11±0.02	7.13±0.01

Statistically, normality and homogeneity tests on the artificial saliva pH value after immersion showed a normal, homogenous distribution. Then a one-way ANOVA test revealed the presence of a significant difference in pH value ($p < 0.05$) between the immersion groups (1 hour, 24 hours, and 72 hours). After that, a Tukey HSD post hoc test indicated significant increases in the artificial saliva pH value across the immersion times. Another Tukey HSD post-hoc test showed generally significant differences in pH value across the pH treatment groups (initial pH values of 7, 5.5, and 4.5). However,

there was no significant difference between the groups with an initial pH of 7 and of 5.5 at an immersion time of 72 hours (Table 2).

Table 2. Mean surface roughness values in μm for the composite resin Giomer Bulk-Fill

Immersion Media Artificial Saliva	Surface Roughness			
	Immersion Time			
	0 Hour	1 Hour	24 Hour	72 Hour
pH 7.0	0.20±0.01	0.21±0.01	0.22±0.01	0.22±0.01
pH 5.5	0.20±0.01	0.23±0.01	0.25±0.01	0.41±0.01
pH 4.5	0.20±0.01	0.25±0.01	0.28±0.00	0.45±0.01

Statistically, the mean values for surface roughness after the specimens were immersed in artificial saliva showed a normal, homogeneous distribution. Accordingly, a one-way ANOVA test was conducted to determine the significance of the test results. The duration of immersion produced an overall significant difference in surface roughness. A Tukey HSD post hoc test showed that, for the pH groups 5.5 and 4.5, the surface roughness of the resin became significantly coarser after soaking for 1 hour, 24 hours, and 72 hours. However, within the artificial saliva group pH 7, the roughness value of the resin surface after 1 hour, 24 hours, and 72 hours of immersion did not produce any significant differences.

3.2 Discussion

The results showed that the Giomer Bulk-Fill composite resin increased the pH value of the artificial saliva after immersion for 1 hour, 24 hours, and 72 hours. The greatest increase in pH occurred in the group of specimens immersed in artificial saliva with an initial pH value of 4.5 for 72 hours, and rose into pH to 7.13, or a percentage increase of 58.44%. In contrast, the smallest increase in pH occurred in the group of specimens soaked in artificial saliva with a pH value of 7 for 1 hour with a rise in pH into 7.08 with a percentage increase of 1.14%. The increase in pH of the artificial saliva occurs because the composite resin of Giomer Bulk-Fill is a type of composite resin containing an S-PRG (Surface Pre-Reacted Glass Ionomer) filler capable of releasing ions such as fluorine, sodium, strontium, aluminum, silicate, and borate [6]. The increase in pH in this study was due to the presence of strontium ions released during immersion in artificial saliva. This is in accordance with the theory that the strontium ion plays a role in neutralizing the acid [13]. Previous studies demonstrated an increase in the pH of lactic acid after immersion of the Giomer composite resin specimens for 1 hour and 24 hours. The results of the analysis of the lactic acid solution showed that the largest number of ions released to neutralize the acid solution were strontium ions (Sr^{2+}) [6].

The percentage increase in the pH of lactic acid after immersion for 1 hour was 20% ± 0.09, from an initial pH of 3.80 to 4.57 ± 0.09. That result means a percentage increase in pH that is not much different from the results of this study [6]. In this study, after soaking for 1 hour, the percentage increase due to the artificial saliva treatment with a pH value of 4.5 was 21.11%. However, after 24 hours of immersion, the percentage increase in lactic acid pH was 59.00% ± 0.05, while the increase due to our artificial saliva treatment with a pH value of 4.5 was 46.89%. In addition to the strontium ions, other ions released from the S-PRG filler, for example, water-soluble sodium (Na^{+}) ions, have other roles: they are able to induce the release of five other ions, including borate ions (BO^{3-}), to prevent bacterial adhesion; in addition, these ions have anti-plaque properties. The aluminum ion (Al^{3+}) plays a role in the control of hypersensitivity. Subsequently, silicate ions (SiO^{2-}) play a role in dentine calcification, and fluoride ions play a role in remineralization to prevent caries [13]. Other references suggest that the presence of strontium and fluorine ions can increase tooth resistance to acids by forming strontiumapatite and fluoroapatite [14]. Thus, ions released from the S-PRG filler

also play a role in neutralizing acid and in preventing secondary caries and are capable of remineralizing dentine, thus strengthening the tooth structure [13].

With respect to the surface roughness test, the duration of immersion in artificial saliva had a greater effect on the roughness than did the pH treatments of 7, 5.5, and 4.5of the composite resin surface. The mean increase in roughness was due to immersion across all pH treatments, and the mean increase in roughness was due to pH treatment across all immersion treatments. After immersion for 1 hour, all specimens showed an increase in surface roughness value. The associated percentage increases in the surface roughness at the pH values of 7, 5.5, and 4.5 are, respectively, 5%, 10%, and 25%, all significantly different, with the highest surface roughness value for the artificial saliva group pH 4.5. These results indicate that the stronger the acidity of the artificial saliva is, the rougher the surface is. After 24 hours and 72 hours immersion, there were also increases in surface roughness, with the highest percentage increase occurring again at an artificial saliva pH value of 4.5. The highest roughness value occurred in the specimens immersed for 72 hours in artificial saliva with a pH of 4.5: an increase in surface roughness of 125%. These results are similar to those of other studies on the surface roughness of composite resin: after immersion for one year in a solution with a pH of 5.8, there was no change [15]. Increased surface roughness after a short immersion was probably due to the presence of GIC derivative components in the composite resin Giomer Bulk-Fill. GIC is a material that is highly soluble in acidic conditions [4, 16]. So longer immersions in artificial saliva with a lower pH value lead to the release of filler particles of the composite resin, such that its surface is degraded and becomes coarser [7].

The surface roughness of restorative materials is an important factor in food debris retention and bacterial adhesion. A previous study showed that the relationship between surface roughness and bacterial adhesion at an roughness value of 0.06 μm did not produce significant results [17], but at roughness values between 0.06 and 0.2 μm, surface roughness were associated with the early stages of bacterial adhesion in the restorative material [17]. Based on those results, the surface roughness of the composite resin in this study may have caused bacterial adhesion due to its surface roughness exceeding Ra 0.06 μm. Thus, Giomer Bulk-Fill composite resins are effective in increasing the critical pH of artificial saliva and are capable of exceeding a neutral pH value. Protection against acidic solutions on teeth can be obtained from the release of ions through the application of resin, as stated above. Therefore, an accurate resin bulk-fill polishing procedure is required to obtain the least possible surface roughness. As mentioned in previous research, a polishing procedure using a nanoscale technique (nano grit) results in very little surface roughness (0.02μm) [17].

4. Conclusion

Based on the results of this study, it can be concluded that immersion of Giomer Bulk-Fill composite resin for 1 hour, 24 hours, or 72 hours in artificial saliva that has pH values of 7, 5.5, and 4.5 increases the pH of the artificial saliva and the surface roughness of the resin. The surface roughness of the composite resin may have caused bacterial adhesion due to its surface roughness. Thus, Giomer Bulk-Fill composite resins are potentially effective in increasing the critical pH of saliva and are capable of exceeding a neutral pH value. Accurate resin bulk-fill polishing procedure is required to obtain the least possible surface roughness.

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