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Research Article

The Effect of Immersion Time on the Amount of Fluoride Release in Fluoridated Composite Resin Materials

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ABSTRACT

Introduction: Fluoride released from composite resin restorations can strengthen tooth structure affected by caries and help prevent secondary caries through remineralization. Fluoride release increases under acidic oral conditions, where hydroxyl ions stimulate ion exchange. This release occurs continuously over time. This study evaluated the effect of immersion time on the amount of fluoride released from fluoridated composite resin materials.

Materials and Methods: This laboratory experimental study used 25 disc-shaped specimens of fluoride-containing composite resin Tetric N-Ceram Packable (Ivoclar Vivadent AG, Schaan, Liechtenstein), measuring 2 mm in thickness and 5 mm in diameter. Samples were divided into five groups according to immersion time: 1, 7, 14, 21, and 28 days. Each specimen was immersed in artificial saliva at pH 5.5 to simulate acidic oral conditions. The amount of fluoride released was measured using a UV–Vis spectrophotometer at a wavelength of 570 nm.

Results: Post Hoc LSD analysis showed that the 1-day group differed significantly from all other groups. Significant differences were also found between the 7-day group and the 21-day and 28-day groups, and between the 14-day and 21-day groups compared with the 28-day group. No significant differences were observed between the 7-day and 14-day groups or between the 14-day and 21-day groups. Fluoride release decreased as immersion time increased.

Conclusion: Immersion time significantly affected the amount of fluoride released from fluoridated composite resin materials, with higher release occurring during the early immersion period.

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INTRODUCTION

Dental caries is a pathological condition characterized by the progressive destruction of the hard tissues of the teeth. This condition is multifactorial in origin and involves the demineralization of tooth structures caused by acids produced from bacterial metabolism of dietary carbohydrates. The demineralization process is followed by the degradation of organic components of dentin, particularly collagen, which eventually leads to the formation of cavities and more extensive structural damage to the tooth.[1] One of the primary approaches to managing dental caries is dental restoration, which aims to restore the form, function, and integrity of the affected tooth structure. In addition to functional rehabilitation, aesthetic considerations have become an important factor in restorative dentistry. Composite resin is widely used as a restorative material because it provides good color matching with natural teeth and allows the restoration of tooth morphology to its original form.[2]

Composite resin materials offer several advantages, including superior aesthetics, good micromechanical bonding with tooth structures, and relatively conservative cavity preparation requirements. However, composite resin also presents certain limitations. One of the main disadvantages is polymerization shrinkage that occurs during the curing process, commonly referred to as polymerization shrinkage, which may affect the marginal integrity of the restoration.[3] Composite resins are classified based on the type and size of their filler particles into several categories, including macrofill, microfill, hybrid, and nanofill composites.[4] Recent developments in restorative material technology have focused on the incorporation of nano-sized filler particles to improve mechanical properties and aesthetic performance.[5] In addition, advancements have been made to enhance the preventive properties of composite resins by incorporating fluoride-releasing compounds such as strontium fluoride or ytterbium trifluoride into the filler components. These modifications aim to provide an additional anticariogenic effect through fluoride release.[6]

Unlike glass ionomer–based restorative materials, fluoride-containing composite resins generally do not exhibit a significant initial burst release of fluoride. Instead, they demonstrate a relatively low but sustained fluoride release over time.[7] Fluoride release is particularly important because it can inhibit the demineralization process and enhance remineralization of tooth structures. In acidic conditions within the oral cavity, fluoride ions can be released from the restorative material and contribute to the remineralization process by interacting with hydrogen ions (H^+), thereby reducing acidity and promoting a more neutral environment. Under these conditions, fluoride-releasing composite resin may act as a buffering agent that helps protect tooth structures from further demineralization.[8]

Previous studies have reported that fluoride nanohybrid composite resins generally release lower amounts of fluoride compared to other fluoride-releasing restorative materials such as glass ionomer cement (GIC), resin-modified glass ionomer cement (RMGIC), polyacid-modified composite resin, and giomer.[9] This phenomenon is influenced by several factors, including the amount of fluoride incorporated into the filler particles, the low solubility of ytterbium trifluoride (YbF_3) in aqueous environments, the relatively low water sorption of composite resins, and their limited permeability. As a result, fluoride release from composite resin tends to occur at a slower and more stable rate.[6]

Although numerous studies have investigated fluoride release from restorative materials, there remains limited information regarding how immersion time influences the cumulative amount of fluoride released from fluoride-containing nanohybrid composite resins under controlled conditions. Understanding the relationship between immersion duration and fluoride release is important because it may reflect the potential long-term preventive effect of restorative materials in the oral environment. Therefore, this study aims to evaluate the effect of immersion time on the amount of fluoride released from fluoride-containing composite resin materials.

MATERIAL AND METHODS

This study employed a true experimental laboratory design using a posttest-only control group design. The research was conducted at the Laboratory of the Faculty of Dentistry, Muhammadiyah University of Surakarta. Ethical approval for this study was obtained from the Health Research Ethics Commission of Dr. Moewardi Regional General Hospital, Surakarta (No. 2.236/XII/HREC/2023).

The samples consisted of 25 fluoride-containing composite resin Tetric N-Ceram Packable (Ivoclar Vivadent AG, Schaan, Liechtenstein) specimens prepared in a disc shape with a height of 2 mm and a diameter of 5 mm. The specimens were randomly divided into five groups based on immersion time, namely 1 day, 7 days, 14 days, 21 days, and 28 days (n = 5 per group).

Each specimen was placed in a tube containing artificial saliva at pH 7 and stored in an incubator at 37°C according to the designated immersion period. After the assigned immersion time, the specimens were removed from the artificial saliva and gently dried with tissue. Subsequently, each specimen was immersed in artificial saliva at pH 5.5 for 15 minutes to simulate an acidic oral environment that may stimulate fluoride release. The saliva solution obtained from this immersion was then used to measure the fluoride release level using a UV–Vis spectrophotometer.[10]

Fluoride concentration was determined using the SPADNS spectrophotometric method. Standard fluoride solutions with concentrations of 2, 4, 6, 8, and 10 ppm were prepared to generate a calibration curve. A SPADNS–zirconyl acid reagent was used as the colorimetric indicator for fluoride detection. In this method, fluoride ions react with the zirconyl-SPADNS complex, resulting in a decrease in absorbance intensity as fluoride concentration increases.

Prior to sample analysis, the UV–Vis spectrophotometer was calibrated using a blank solution and the prepared standard solutions. Measurements were performed at a wavelength of 570 nm using quartz cuvettes. The calibration curve was generated from the absorbance values of the standard solutions. The saliva samples obtained from each immersion group were then analyzed under the same conditions to determine the fluoride concentration expressed in parts per million (ppm). The reduction in absorbance corresponds to an increase in fluoride ion concentration due to the dissociation of the SPADNS–zirconyl complex.[11]

The study began by testing the calibration of the standard fluorine solution with an initial concentration of 100 ppm. The standard fluorine solution was diluted into several concentrations, namely 2 ppm, 4 ppm, 6 ppm, 8 ppm, and 10 ppm.

The equation obtained from the calibration test will be used as a formula to calculate the fluorine content in ppm units. The collected data were analyzed using One-way Analysis of Variance (ANOVA) to determine the effect of immersion time on fluoride release levels. Prior to the ANOVA test, normality was assessed using the Shapiro–Wilk test, and homogeneity of variance was evaluated using the Levene test. If significant differences were identified, the analysis was continued with a Post Hoc Least Significant Difference (LSD) test to determine pairwise differences between groups. In addition, effect size and 95% confidence intervals were calculated to provide a stronger interpretation of the magnitude of the observed effects. Statistical analysis was performed with a significance level of $p < 0.05$.

RESULTS AND DISCUSSIONS

The standard calibration curve was obtained from spectrophotometric analysis of fluoride standard solutions at concentrations of 2, 4, 6, 8, and 10 ppm. The relationship between fluoride concentration and absorbance measured at 570 nm produced a linear regression equation $y = -0.0015x - 0.6139$ with a coefficient of determination (R^2 = 0,9348), indicating a strong linear correlation. The calibration curve was used to calculate the fluoride concentration released from the composite resin samples based on the absorbance values obtained during spectrophotometric measurement. The graph data of the standard fluorine solution is as follows.

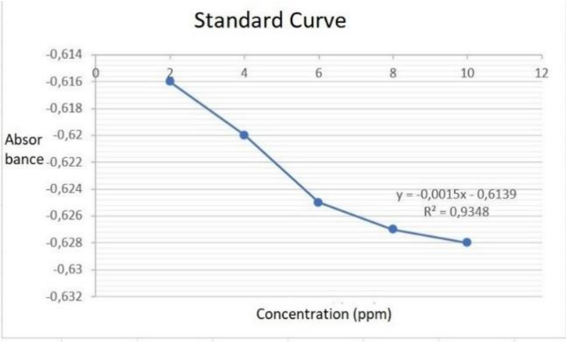

Figure 1. Standard solution curve of fluoride

Based on measurements using a spectrophotometer with a standard fluoride solution and a blank solution, a linear regression equation $y = -0.0015x - 0.6139$ was obtained with a correlation coefficient (R^2) of 0.9348. This result indicates a strong relationship between the absorbance value and the fluoride concentration in the working solution.

The correlation coefficient describes the strength and direction of the relationship between two variables. A coefficient value of +1 indicates a perfectly positive linear relationship, whereas −1 indicates a perfectly negative linear relationship. In spectrophotometric analysis, the calibration curve is used to determine the relationship between absorbance and concentration of the analyte.[12]

After obtaining the linear regression equation from the calibration curve, the fluoride concentration in the samples was determined using the absorbance values measured by the spectrophotometer. The measured absorbance value was substituted as y in the linear regression equation to calculate the corresponding x value, which represents the fluoride concentration expressed in ppm. The calculated fluoride concentrations represent the amount of fluoride released from the composite resin samples according to the formula presented in Table 1.

Table 1. Mean and SD results of fluoride release test (ppm)

Group	Mean ± SD
1 (1 day)	9.28 ± 0.855
2 (7 days)	8.2 ± 0.854
3 (14 days)	7.5 ± 0.863
4 (21 days)	6.82 ± 0.878
5 (28 days)	4.78 ± 0.363

The following are the mean values of fluoride release levels in each immersion group for 1 (1 day), 2 (7 days), 3 (14 days), 4 (21 days), and 5 (28 days).

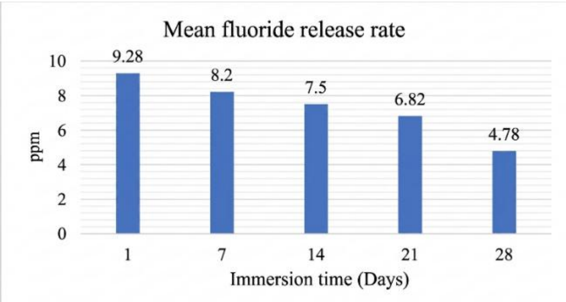

Figure 2. Fluoride release rate

The data obtained will then be subjected to statistical tests. Normality test with Shapiro-Wilk with normally distributed sample results ($p > 0.05$). Homogeneity test with Levene's test with a p-value of 0.237 ($p > 0.05$), so it can be concluded that the data is homogeneous. The data is then analyzed using one-way ANOVA. The results show the released fluor content from the resin-composite fluor. The obtained significance value (Sig.) of 0.000 indicates that the p-value is less than the significance level of 0.05 ($p < 0.05$). This finding suggests that immersion time has a statistically significant effect on the amount of fluoride released from the fluoride-containing composite resin**.** The subsequent step is promoting LSD analysis, the least significant difference, to determine the mean differences among groups. Table 2 shows the LSD.

Table 2. Post-Hoc LSD Test Results

Group	1 (1 day)	2 (7 days)	3 (14 days)	4 (21 days)	5 (28 days)
1 (1 day)					
2 (7 days)	0.043*				
3 (14 days)	0.002*	0.176			
4 (21 days)	0.000*	0.012*	0.188		
5 (28 days)	0.000*	0.000*	0.000*	0.001*	

Based on the results of the post-hoc LSD analysis above, it was concluded that there was a significant difference between the groups, except that the results showed no significant difference between groups 2 and 3, and also 3 and 4.

Advances in nanotechnology have enabled the development of composite resins such as fluoride to prevent recurrent caries.[6] The ability of composite resins to release fluoride is triggered by the oral cavity environment in acidic conditions. The mechanism of fluoride release occurs when bacteria in the oral cavity cause a decrease in saliva pH to making it acidic. Saliva conditions with acidic pH have an increased hydroxyl ion content. This plays an important role in stimulating fluoride release. The level of fluoride release varies according to the content of each material, such as composite resins that release lower fluoride every day.[8]

The results in Figure 2 show that the fluoride levels in the composite resin decreased after immersion for 1 day, 7 days, 14 days, 21 days, and 28 days. The 1-day immersion treatment group had the highest value with an average of 9,28 ppm. This was explained in the research that the amount of fluoride in the composite resin was greater during 24 hours or 1 day of immersion.[7] The release of large amounts of fluoride in the first 24 hours is because fluoride is needed in the polymerization process of fluoride-containing composite resin. The polymerization process of fluoride composite resin produces two types of bonds, namely micromechanical bonds and chemical bonds. Chemical bonds in composite resin are obtained through the content of ytterbium trifluoride. It can release fluoride. When fluorine is released, it will bind to hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$), and this results in the formation of a new molecule called fluorapatite ($Ca_{10}(PO_4)_6F_2$).[13]

The 7-day immersion treatment group had a value of 8,2 ppm. The 14-day immersion treatment group had a value of 7,5 ppm. The 21-day immersion group had a value of 6,82 ppm. And the 28-day immersion group had a value of 4,78 ppm. This explains that fluoride in fluoride composite resin is released during the polymerization process, and only in small amounts, is slowly released in the long term.[6] The fluoride levels in the composite resin decreased after immersion for 1 day, 7 days, 14 days, 21 days, and 28 days. It was explained that when demineralization occurs, fluoride will come out to turn into remineralization, so the longer the immersion, the lower the fluoride levels.[14]

The results are in Table 2. One-way ANOVA analysis showed a significant difference between the means in the 1-day, 7-day, 14-day, 21-day, and 28-day immersion groups. The amount of fluoride release decreased significantly over time for all filling materials.[7] The results are in Table 3. Post-Hoc LSD analysis showed that group 1 had a significant difference from the other groups, namely groups 2, 3, 4, and 5. Group 2 had a significant difference from groups 4 and 5. Groups 3 and 4 had a significant difference from group 5. As explained, on the first day, there was an increase in fluoride release due to the polymerization process. The polymerization process of fluoride composite resin produces two types of bonds, namely micromechanical bonds and chemical bonds. The chemical bonding mechanism in the polymerization process is when ytterbium trifluoride releases fluorine, and the fluorine binds to hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$), resulting in the formation of fluorapatite ($Ca_{10}(PO_4)_6F_2$).[13]

The results of the Post-Hoc LSD analysis in Table 3 show that there is no significant difference between groups 2 and 3, and also 3 and 4. Fluoride release occurs insignificantly because the composite resin formulation generally has a lower fluoride content. It is like the low solubility of ytterbium trifluoride in water, the low water content in the material, and the permeability of the composite resin.[14] The release of fluoride in composite resin is insignificant. It is because the content of fluoride composite resin, namely ytterbium trifluoride, has a low fluoride release, but in the long term.[15]

Fluoride levels can change every day, and the optimal fluoride levels to achieve the remineralization process require a minimum of 3 ppm. This study showed fluoride release at immersion times of 1 day, 7 days, 14 days, 21 days, and 28 days, which is more than 3 ppm, so that it has an optimal fluoride release to achieve the remineralization process. Composite resin has the potential to show continuous fluoride release in the long term. This is important to prevent recurrent caries.[16]

CONCLUSION

Based on the research results, it can be concluded that there is an effect of immersion time on reducing the amount of fluoride released from fluoride composite resin materials. Higher fluoride release was observed during the initial immersion period, followed by a gradual decrease as the immersion time increased. This indicates that fluoride release from the composite resin occurs more intensively at the early stage and subsequently stabilizes at lower levels over time.

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