# THE RELEASE OF FLUORIDE IONS OF BIOACTIVE RESIN IN THE SOLUTION OF LACTIC ACID AND ARTIFICIAL

*by* Dewi Puspitasari

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# THE RELEASE OF FLUORIDE IONS OF BIOACTIVE RESIN IN THE SOLUTION OF LACTIC ACID AND ARTIFICIAL SALIVA

## INTRODUCTION

Dental caries are multifactorial disease of dental hard tissue that damages the structure of the email, dentin and sementum.[1] Caries process begins with the presence of continuous demineralization of tooth due to irreversible solubilization of the tooth mineral caused by acid proceeded by certain bacteria such as Streptococcus mutans. The dominant acid produced by Streptococcus mutans is lactic acid.[2,3,4] Caries management to preserve and restore natural tooth structure are determined by the type of lesion, restorative material applications are usually to be the preferred approaches. [3,4] One of the most common causes of restoration failure is secondary caries.[5] Secondary caries are caries lesions lied at the marginal of restoration or caries tied to the restoration or sealant.[6]

As the times progress, the use of resin based restorative materials is significantly increased.[7,8] Composite resins are globally used due to good aesthetics with high strength. In 2013

bioactive composite resin was introduced as a bioactive restorative material with the "smart behaviour".[7,9] The bioactivity of these materials can be attributed to its ability to release ions and provide ionic exchange with saliva and tooth structure thereby remineralization could be promoted surrounding tooth structure. Bioactive composite resins consist of matrix and filler from that differed from convention composite resins. Filler of this resin contains reactive glass filler (21.8 wt%), inorganic filler (56 wt%), rubberized resin, amorphous silica (6.7%), sodium fluoride (0.75%) and water. Matrix is a mixture of diurethane and methacrylate modified with 44.6% polyacrylic acid.[10] Bioactive composite resins have water absorption and solubility properties due to the presence of amorphous silica.[8] Mechanically, bioactive composite resins are stronger than conventional composite resins due to their rubberized content, ionic-resin which contains less water therefore it is more resistant to shock.[11] Bioactive composite resins have chemical properties that are able to release and recharging phosphate, calcium and fluoride.[8]

Bioactive composite resins release more phosphate, fluoride and calcium ions than glass ionomers. Phosphate, fluoride and calcium ions, stimulate remineralization and apatite formation.[8,10] Fluoride has remineralization activity that helps strengthen tooth tissue structure and inhibits demineralization by combining fluoride with calcium to form fluoroapatite.[8,12,13] This bond is more stable and more resistant to acids due to the bond structure formed is more complex.[13]

The ability of bioactive resin to take and release fluoride is dependent on factors such as fluoride content and also changes of oral pH.[14] Oral environment pH including salivary pH may turn to acid condition due to the metabolism of Streptococcus mutans, this bacteria can metabolize carbohydrates into organic acids, namely lactic acid.[15] This state may affect the properties of bioactive resin including the release of fluoride ions.[15,16] Conventional composite resin-based restorative materials have no ability to release fluoride ions for tooth remineralization as do as GIC, but conventional GIC cannot conform the strength of conventional composite resin. Therefore, the use of bioactive resins is expected to represent both of restorative materials in terms of strength and ion releasing properties.[14] Bioactive resins released fluoride ions without a burst effect on the first day, but the level of fluoride ion release remained relatively constant for a week (7 days).[16] There is still a limited study of the release of fluoride ions from the first day until the seventh day, especially in the lactic acid immersion and artificial saliva to represent the oral environment. The purpose of the study is to analyse the release of fluoride ions of bioactive resin in the solution of lactic acid and artificial saliva.

## MATERIALS AND METHODS

The study was approved by the ethical clearance test published by the Faculty of Dentistry, Lambung Mangkurat University No. 012/KEPKGFKGULM/EC/II/2021. The samples were prepared using bioactive resin (Activa ™ Bioactive Restorative, Pulpdent) with 15 mm in diameter and 1 mm in thickness of plastic mould in a total of fourtytwo specimens.

The bioactive resin was mixed and extruded from a syringe through a spiral gun and applied to the mould. The top of the mould was covered by a celluloid strip followed by glass slide and gently pressed. Specimens were light cured with a LED Light Curing Unit with an output of 800 mW/cm2 for 20s on the top of mould surfaces and the tip touching the glass, subsequently stored in an incubator for 24 h at 37ºC. All specimens were immersed based on the group treatment as follows: (1) immersion on lactic acid solution for 1 day, (2) artificial saliva for 1 day, (3) distilled water for 1 day, (4) lactic acid solution for 7 days, (5) artificial saliva for 7 days, (6) distilled water for 7 days; all immersion media are treated with pH measurement (lactic acid solution pH 5.2, artificial saliva pH 6.7 and distilled water pH 7), replaced every day during the study process and stored in incubator with the temperature of 37º C.

The release amount of ion release measurement was conducted by using pH Meter (Lutron-208). The bioactive resins specimens were ground using a mortar and pastle until fine particles were obtained and then diluted using 10 ml of distilled water and 10 ml of sodium fluoride buffer. The electrode was immersed into the sample solution for 3 minutes using a pH meter (Lutron-208). The calculation of the amount of fluoride release was carried out by subtracting the baseline bioactive composite resin by the number of the fluoride calculated using a pH meter (Lutron-208).

Data analysis was carried out with a two-way test ANOVA with a confidence level of 95% ($\alpha$ = 0.05) and continued using the Bonferroni Post Hoc test to determine the value of significance.

## RESULTS

The mean values of the amount of fluoride ion release in the bioactive resin are summarized in table 1.

The mean and standard deviation of the number of fluoride ion release of bioactive resin.

| Immersion media and time | Mean± SD (μg) |
|---|---|
| Lactid acid 1 d | 6.37 ± 0.74[A] |
| Artificial saliva 1 d | 1.30 ± 0.48 [B] |
| Distilled water 1 d | 3.78 ± 0.97 [C] |
| Lactid acid 7 d | 22.90 ± 0.81 [D] |
| Artificial saliva 7 d | 19.00 ± 0.81 [E] |
| Distilled water 7 d | 21.60 ± 1.01 [F] |

Two Way Annova test
*Different superscript letters indicate significant differences.

The table 1 showed that the highest mean amount of fluoride ion release was in the group of bioactive resin specimens immersed in lactic acid solution for 7 days, while the group with the lowest mean amount of fluoride release was the group of artificial saliva immersion for 1 day.

The results of Two-Way Annova test showed that the significance value of the immersion media group was p = 0.000 (p <0.05), which means that there was a significant difference in the number of the fluoride ion release of bioactive resin immersed in lactic acid, artificial saliva and distilled water. The significance value of the immersion time group was p=0.000 (p<0.05), which means that there was a significant difference in the amount of fluoride ion release in the bioactive resin that was immersed for 1 and 7 days. The significance value of the immersion media and time was p=0.138 (p>0.05), which means there was no interaction between the immersion media and time. Bonferroni Post Hoc test shows that there are significant differences among all groups.

## DISCUSSION

The capability of composite resin-based bioactive materials to release fluoride ions may attributed to the remineralization of the surrounding tooth.[16] The released fluoride which taken up by the enamel and dentin, making the tooth structure more resistant to acids by a combination of decreasing solubility and impairing the activity of cariogenic bacteria. Fluoride ion dynamics provide an advantage for tooth that have a high susceptibility to caries.[7]

In the oral cavity, many factors may affect the amount of fluoride ion release in bioactive composite resins, one of which is the pH value of oral cavity and also saliva. In the present study, to represent the condition of oral cavity we conducted for the treatment of bioactive resin in the immersion of lactic acid and artificial saliva. Lactic acid has the most acidic degree of acidity (4.2) followed by artificial saliva (6.7) and distilled water (7.0). The lower the pH value, the more the reservoir releases fluoride ions. Our study is incline with Abdallah (2019) that bioactive resins may release more fluoride ions in acidic pH than neutral pH.[13] This is due to an acidic state contain more $H^+$ ions, result in the release of the chemical structure of the bioactive composite resin and stimulating the release of fluoride ions.[14,17,18] Meanwhile, artificial saliva and distilled water have a neutral pH, which does not ionize more to produce $H^+$ ions, resulting in the release of fluoride ions. The more acidic pH may alter the properties of materials by releasing more ions and result in the loss of material. This loss may reduce the life and durability of the materials. The loss of material may attributed to the higher amount of resin matrix leads to increase water absorption and softening the specimen ion release from filler particles can result in separating of the matrix and filler, and also by micro-cracks forming in the interface of filler-matrix interface pH.[7,18]

Based on the results of study that has been carried out, the least amount of fluoride ion release was found in the bioactive composite resin immersed in artificial saliva for one day, although the pH value of artificial saliva was lower than that of distilled water. This possibility may occur due to the difference in viscosity between artificial saliva and distilled water. The specimens were immersed in distilled water to prevent any influence of minerals or organic molecules which might be presented in the solutions or artificial saliva.[13] The presence of dissolved substances $Na_2HPO_4$, KSCN, NaCl, $KH_2PO_4$, KCl and $NaHCO_3$ contained in artificial saliva may cause the particle density to be greater and the viscosity to be higher.[16,19] Meanwhile, distilled water is pure $H_2O$ which has a smaller density and has a lower viscosity. High viscosity can make it difficult for artificial saliva to reach the filler in the bioactive composite resin, therefore the amount of fluoride ion release is less. The solute in artificial saliva is also a mineral salt with a normal pH that does not produce $H^+$ and $OH^-$ ions due to an imperfect ionization process, the low $H^+$ content resulting in the ions released from artificial saliva was less.[20]

Another factor that may cause the bioactive composite resin immersed in artificial saliva to release less fluoride ions when compared to distilled water is the presence of bioactive glass in the bioactive composite resin. The fluoride ion in the bioactive composite resin is found in the filler. The resin contains bioactive glass consisting of $SiO_2$, CaO, $Na_2O$ and $P_2O_5$. Therefore, when water diffused and contact with the bioactive glass, a

reaction occurs in the formation of hydroxyapatite (HA). Acid conditions and the presence of moisture due to the diffusion of water ($H_2O$) into the matrix of resin causes the breakdown of $H_2O$ into $H^+$ and OH, then the silicon dioxide bond (Si-O-Si) is attacked by hydroxyl ions. The surface of the bioactive glass exposed to water results in the exchange of sodium and calcium ions with hydrogen ions from solution, forming Si-OH groups and forming a layer rich in silica, which undergoes polymerization, binding calcium ions and phosphate ions in the form of Amorphous Calcium Phosphate (ACP), which then crystallized into HA.[21] If HA can bind to fluoride ions, fluoroapatite can be formed which can be a better remineralizing agent than HA. In order for the formation of HA and fluoroapatite can be conducted, the structure of the bioactive composite resin must be able to allow the diffusion of water to reach the bioactive glass and fluoride ions released from the structure of the bioactive composite resin.[22]

Immersion the composite resin in lactic acid media, artificial saliva and distilled water for one day and seven days showed no interaction with the amount of fluoride ion release. Based on the statistical analysis test of Two Way Anova on the 1st and 7th day of immersion the amount of fluoride ion release of the bioactive composite resin showed the same results, namely lactic acid which was the highest followed by distilled water and the lowest was artificial saliva. According to Francois (2020) study, bioactive composite resins in contact with an aqueous environment will release fluoride ions, calcium ions and phosphate ions.[14] The release of fluoride ions can also occur when bioactive composite resins are exposed to saliva and solutions with critical pH.[7] The use of immersion media also has an effect on the amount of fluoride ion released. The study conducted by Eriwati (2020) showed that the immersion of bioactive resins in a solution of critical pH (4.5) and normal pH (7.0) released more fluoride ions in critical pH.[23] The more acidic the environment around the bioactive composite resin, the more a lot of fluoride ions are released. The immersion time also has an effect on the amount of fluoride ion release. The longer the immersion time for the bioactive composite resin, the more fluoride will be released. In line with Ruengrungsom's study on day 4, more fluoride ions were released than on day 1.[16] Ibrahim et al (2020) also stated that restoration materials based on composite resin released more fluoride ions on day 7 than day 1.[24] The higher the fluoride ion released by bioactive resin, the higher the fluoride that can be deposited on the tooth enamel. Therefore, even in an acidic environment remineralization will be achieved

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