



School of
Dental Medicine

**Fluoride Release, pH change and Recharge Ability of Different
Types of Glass Ionomer Restorative Materials: A Comparative
In-Vitro Study**

A Thesis

**Presented to the Faculty of Tufts University School of Dental
Medicine in Partial Fulfillment of the Requirements for the Degree of
Master of Science in Dental Research**

by

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August 2016

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ABSTRACT

Purpose: The aim of this in-vitro study was to evaluate and compare fluoride release, levels and change in pH, and recharge ability of four types of glass ionomer restorative materials at one day, seven days, 15 days, 16 days, and 30 days. **Materials and Methods:** Samples were prepared from Fuji IX, Photac Fil, Riva LC and Activa Bioactive restorative. Each specimen was immersed in 5 mL of artificial saliva for 24 hours to be measured for fluoride release and pH level. Samples were then transferred to new containers to be measured again at 7 days and 15 days. After the 15th day reading, samples were recharged using 5% sodium fluoride varnish. Fluoride release and pH levels were assessed again at the 16th day and 30th day. **Results:** All materials, except Fuji IX, scored highest at day 1 then decreased gradually at day 7 only to increase at day 15. Fuji IX showed the highest scores until the day of recharging and decreased gradually from day 1 to 15. Fuji IX and Activa showed similarly the highest scores at day 16 (1 day post recharging with NaF). Activa was significantly highest at day 30 followed by Riva and Fuji IX showing comparable amounts. pH levels showed instability and exhibited median values between 4.65 and 5.95. **Conclusion:** Within the limitations of this in-vitro study, we can conclude that when tested in artificial saliva, Fuji IX had the highest scores of fluoride release. Activa exhibited the highest recharge ability at day 30. pH levels fluctuated and avoided potentially destructive levels throughout most of the study's duration.

Dedication

My efforts here are dedicated to my family. Everything I do, everything I am, I owe to every single one of you.

To my grandfather Abdulrahman who passed away during my studies, there is not a day that passes without me wishing you were here.

To my mother Ezdehar and my wife Najlaa, without your incredible strength and unconditional love, I would not have been who or where I am today.

I thank you. I love you.

Acknowledgments

I would like to thank my principal advisor Dr. Loo for her continuous support throughout my time at Tufts. She has been an excellent source of advice and guidance.

I would also like to extend my gratitude to Dr. Matthew Finkelman. His contribution and input have been indispensable to this project and my education.

I will be forever indebted to Dr. Mohamed Hassan for his vital help inside the clinic and out. He has been an outstanding mentor.

My sincerest appreciation goes to Dr. Gerard Kugel. Without his key insight, this project would not have been possible.

You all have my most respect and gratitude. I will forever be your student.

Many thanks go to the leadership of my home country Saudi Arabia. Their vision and support have made this dream of mine, and thousands of others, come true.

A special thank you goes to Jeffery Daddona for his technical assistance and contribution during this project.

Literature Review and Introduction

Fluoride:

Fluoride is a natural mineral that is found in soil and water. Natural levels of fluoride vary depending on the source and are generally not high enough to prevent tooth decay. As early as the 1930s, researchers found that people who had access to water with naturally higher fluoride content had a lower rate of tooth decay than those who did not. In the mid 1940s, communities started to add fluoride to the water supply in what is known today as community water fluoridation.¹ In a 2012 census, 75% of US communities reported water fluoridation.²

Fluoride's main activity is characterized by the inhibition of the demineralization process. Utilizing calcium and phosphate, its mechanism has been described to change the hydroxyapatite structure on tooth enamel into fluorohydroxyapatite. This composition is known to be less acid soluble thus making it harder to break down.³

Today, fluoride is present in many forms; the wider and most cost-effective use is community fluoridation. The US public Health Service has established the level of 0.7-1.2 mg fluoride ion/L (ppm) to be both safe and effective.⁴ In areas where levels are below 0.6 ppm, the American Academy of Pediatric Dentistry (AAPD) recommends prescribing supplemental fluoride to children.^{5,6}

Supplemental and over the counter fluoride comes in a few different forms. One of the most popular methods is fluoridated toothpaste and mouth rinse for daily use. Fluoride content in such products comes in very low, yet effective doses. This low dose is balanced by high frequency use. There is an abundance of evidence to suggest the efficacy of fluoridated dentifrices and low dose (0.05%) sodium fluoride mouth rinses.^{7,8} Fluorosis has been described as a chronic condition in which white spots are seen on teeth enamel and pitting in more severe cases. It is caused by excessive intake of fluoride during early stages of teeth development in young children.⁹ Research has discovered that if the recommended guidelines are followed with care and supervision, these amounts are safe to use in children with very low risk. Fluorosis is the result of cumulative fluoride intake and its severity depends on dose, duration and phase of enamel development at the time of high exposure.¹⁰

Professionally applied fluoride has also shown high success rates in meta analyses in permanent teeth and multiple clinical trials for primary teeth. The most common forms of office use are 5% sodium fluoride (NaF) and 1.23% acidulated phosphate fluoride (APF) gels.^{11, 12} Clinically, fluoride gels and varnishes have been used to coat early carious lesions. Varnishes have also been found to leave a calcium rich layer that protects the tooth and helps reverse the cavitation process.¹³

Fluoride foams have also been used clinically. However, there is limited clinical data indicating the effectiveness of foams compared to gels and varnishes. In addition, there is not sufficient evidence suggesting superiority of varnishes over gels or vice versa. The American Dental Association emphasizes the importance of establishing a level of risk for each individual based on his or her history and clinical presentation. Patients are typically categorized into low, moderate or high-risk patients. Once the level of risk is established and clinical experience of the provider is integrated, treatment decisions are much easier taken chair side.¹⁴

Dietary supplements of fluoride have also been recommended for children in areas with less than optimal water fluoridation levels. The AAPD presents clear guidelines on appropriate indications and doses for the provider.⁶

The process of Demineralization and Remineralization:

The process of cavitation is dynamic in nature. Before a carious lesion can be visually detected clinically, the lesion starts as an area of demineralization with minor color change. As bacteria start to produce acid from sugar present in foods on tooth enamel and pH levels begin to drop, certain minerals are leached from tooth surfaces. This decrease in pH levels causes calcium and phosphate ions to diffuse from enamel into the oral cavity.¹⁵ This is normally balanced by the buffering effect of salivary calcium and phosphate back into enamel known as remineralization where the initial process of demineralization is neutralized. Typically under normal circumstances, this cycle is in balance and no changes would occur. Some authors have urged clinicians to further focus their efforts in prevention and intervention in these early stages rather than addressing lesions of the disease in the form of restoring lost tooth structure. It has been shown that when identified in initial stages with proper intervention, this process of cavitation can be

arrested and even reversed.^{16, 17} Moreover, since acidity is an integral part of the demineralization and cavitation process,¹⁵ any potential change in pH levels caused by these materials is an area of great clinical interest. Rytomaa et al suggested that the level of which enamel dissolution occurs is 5.5 with a seven to eight factor increase with each decrease by 1 unit of pH.^{18,19}

Glass Ionomer Restoratives:

Wilson and Kent first introduced glass ionomers in 1970. Glass polyalkenoate cements, given the name “glass ionomer” by Kent, are materials containing a base and an acid. The base part is usually made of calcium or aluminofluorosilicate glass powder whereas the acid is made of a water-soluble polymer. Upon mixing, a setting reaction takes place and hardened cement is produced. Many improvements have been made in such materials over the years. Of these improvements was the inclusion of resin into glass ionomers. Resin modified glass ionomers (RMGI), as the name implies, have resin particles incorporated in their composition. Resin components increase physical strength and wear resistance aspects. Research has also shown that different ingredients have been added to expedite the hardening process and increase esthetic outcomes. Polymerization by visible light has also added clinical value to RMGIs. This “on command” setting has gained popularity among clinicians especially the pediatric dentist.^{20,21}

The AAPD defines Early Childhood Caries (ECC) as “the presence of one or more decayed (noncavitated or cavitated lesions), missing (due to caries) or filled tooth surfaces in any primary tooth in a child under the age of six.”²² Glass ionomer restorations have been one of the essential materials at the dentists’ disposal in efforts to address and treat dental caries. They were promoted for use in primary dentition due to their ability to release fluoride and adhere chemically to tooth surfaces.²³ In their early phases, they had some disadvantages such as poor wear resistance and difficult handling. They have shown much improvement in features and consequently popularity along the years nonetheless.²⁴

Glass ionomer materials are known to be less technique and moisture sensitive than the typical composite resin materials.²⁵ This is one of the most important advantages when managing less than cooperative children and the special needs population.^{25, 26} Another main advantage is the incorporation of fluoride releasing ability.^{25, 26} There have been studies where decrease of caries causing bacteria has been noted for up to six months with such materials.^{29, 30} They are the material of choice when conventional preparation and ideal isolation cannot be achieved.²⁶

All of these features offer a great deal to the clinician and more specifically to the pediatric dentist when behavior of preschoolers and children with special needs present a challenge. Children's perception and reaction to a dental setting varies depending on multiple factors.³¹

Glass ionomer restorative materials are also used as interim therapeutic restorations (ITR) as a caries control tool in patients with rampant caries.³² Stepwise excavation techniques, where carious lesions are carefully accessed and restored more than once in a period of a few months to protect pulp tissue, have been validated in a Cochrane review in 2013 for both primary and permanent molars.³³ A more definitive type of restoration after a certain period of time usually follows this step.^{32, 34}

In addition, in a split mouth design study, Resin Modified Glass Ionomers (RMGI) have also been shown to be more effective in reducing counts of *Mutans S* and *Lactobacilli* when compared to amalgam counterparts.³⁵

Another important feature in these materials is not only fluoride release, but also the ability to uptake fluoride.^{36, 37} They have the ability to absorb the additional fluoride introduced into the oral cavity by prescription supplementary fluoride or fluoridated mouthwash and toothpastes.³⁸ This feature helps the restoration act as a reservoir of fluoride and maintains constant release adjacent to enamel and dentine, which is much needed in patients with high caries susceptibility.^{28, 21}

Compomers:

Compomers are a type of restorative material that was developed and marketed in the 1990s. As the name suggests, they were formulated in an attempt to abridge the gap between glass ionomers and composite resins in a polyacid modified resin-based composite. Manufacturers strived to combine the advantages of both materials in one. However, research has shown that while it offered an improvement in handling and esthetic properties, it did not present superior abilities in wear resistance over resin composites or fluoride release, recharge ability and marginal adaptation over resin modified glass ionomers.^{20, 21, 39-41,}

Aims:

To evaluate and compare fluoride release, levels and change in pH and recharge ability of four types of glass ionomer restorative materials at one day, seven days, 15 days, 16 days, and 30 days.

Hypothesis:

There will be a difference between the groups in fluoride release, pH levels and change, and recharge ability with Fuji IX being highest, followed by Photac Fil, Riva LC and Activa Bioactive Restorative to exhibit the least amounts*.

*Based on a pilot study finding.

Research Design and Methods:

Sample Size Calculation:

A calculation was conducted using nQuery Advisor Version 7.0 (Statsols, MA, USA) to determine the power of the study's primary aim: the comparison of 24-hour fluoride release between groups. Assuming means of 88.4, 29.0, 17.1, and 12.7 PPM* for Fuji IX, Photac Fil, Riva LC, and Activa Bioactive, respectively, and a within-group standard deviation of 24.8 PPM*, a sample size of n=15 per group was deemed adequate to obtain a Type I error rate of 5% and power greater than 99%.

* Results obtained from pilot study

Study Design:

This study was designed to be a blinded controlled in-vitro experiment. A blinded co-investigator recorded all measurements and entered data in a spreadsheet. Samples were each given a code composed of a letter and a number.

Materials: Shown in Table 1:

Group I - Fuji IX (**F IX**) (GC, Tokyo, Japan) (Self cure)

Group II - Photac Fil (**PF**) (3M, MN, USA) (Dual cured)

Group III - Riva LC (**RL**) (SDI, Sydney, Australia) (Light cured)

Group IV- Activa Bioactive Restorative (**AB**) (Pulpdent, MA, USA) (Triple cure)

Methods

Preparation of artificial saliva:

List of ingredients:

- 4.76g HEPES (2-Hydroxyethyl)-1-piperazineethanesulfonic acid)
- 2.23g Potassium Chloride (KCL)
- 0.544g Potassium Dihydrogen Phosphate (KH₂PO₄)
- 0.077g Calcium Chloride (CaCl₂)
- 0.049g Magnesium Chloride Hexahydrate (MgCl₂ 6H₂O)
- 0.019g of Sodium Azide (NaN₃)

Ingredients were mixed and dissolved into deionized water and adjusted to a pH of 5.6, measured by a pH electrode (Ross Ultra 8102 BN, Thermo Scientific, MA, USA). (Figure 1)

Specimens from each material (n=15 per group) for a total of 60 specimens were prepared and molded into disc shaped samples (10mm in diameter and 1.5 mm in thickness) using plastic molds to insure standardization in size and amount of material.⁴² Then, clear Mylar strips were placed on both sides and glass slabs were placed over each sample. Hand pressure was applied to insure no air was trapped and excess material was removed.⁴³ Manufacturer's instructions were followed for mixing and dispensing for each material. Light curing, when indicated, was achieved using LED light from both sides for a total of 40 seconds (Translux Wave, Heraeus Kulzer Hanau, Germany) (Figure 2). Samples were carefully removed from molds and placed in polypropylene plastic tubes containing 5 mL of artificial saliva and incubated in a constant temperature of 37±0.5°C⁴⁴ (LabX, ON, Canada) (Figure 3). After 24 hours, the initial reading of pH and fluoride release was taken. Levels of pH were measured first for each sample using a pH electrode (Ross Ultra 8102 BN, Thermo Scientific, MA, USA) connected to a digital ion analyzer (Orion Star A214, Thermo scientific, No 0809, MA, USA) (Figure 1). Total Ionic Strength Adjustment Buffer (TISAB II, Orion research, Inc., MA, USA) containing 1.2-

cyclohexylenedinitrotetraacetic acid (CDTA) was then added by ratio of 1:1 to provide a constant background and decompose fluoride ion for detection.⁴⁵

Fluoride levels were obtained following buffering using a fluoride ion specific electrode (Thermo scientific, No 0809, Beverly, MA) (Figure 1) and recorded in Parts Per Million (PPM). Samples were then removed from containers, rinsed with distilled water and placed in a new container with 5 mL of fresh artificial saliva and re incubated for the next reading at 7 days.^{36, 43} This procedure was repeated for the third reading at the 15 day time point.³⁴ Calibration was carried out for both electrodes before each reading with different pH solutions (4.01, 7.00 and 10.01) and standard fluoride solutions with ionic concentration (1, 2 and 10 PPM). To avoid possible overlap, electrodes were rinsed using deionized water and dried with delicate wipes following each reading.

After the 15th day reading, all samples were immersed in 5% sodium fluoride NaF (Vanish, 3M, MN, USA) for 5 minutes.⁴⁴ After immersion, samples were rinsed 3 times with 5 mL distilled water and dried using absorbent paper before placing them in artificial saliva and re incubating. Fluoride analysis was repeated post exposure in 24 hours (day 16) and day 30 applying the same methods of measurements for fluoride release and pH as pre exposure to fluoride varnish. Measurements were taken at room temperature of $23 \pm 1^\circ\text{C}$ at all times.

Statistical Analysis

For the comparison between different groups at each time point, the Kruskal-Wallis test was used supplemented by the Mann-Whitney U test with Bonferroni correction for post-hoc comparisons. The Friedman test was used to analyze fluoride release and pH levels between the first three time points (1, 7 and 15 days) for each group followed by the Wilcoxon signed-rank test with Bonferroni correction for post-hoc comparisons. The Wilcoxon signed-rank test was used to compare recharge ability and pH levels for the last two time points (16th day and 30th day). Data were analyzed using SPSS Version 24 (IBM Corp. Armonk, NY).

Results

Fluoride Release:

Intergroup comparison: Medians and interquartile ranges of fluoride release for each group as well as time points are shown in Table 2 and illustrated in Figures 4, 5 and 6. On day 1, the Kruskal-Wallis test revealed there was a statistically significant difference ($p < 0.05$) in fluoride release between study groups. The post-hoc tests revealed there was a statistically significant difference between all groups except (RL) and (AB). (F IX) had the highest median of 84.60, with an interquartile range (IQR) of 22.3. This was followed by (PF) with a median (IQR) of 14.2 (3.2). On day 7, there was a statistically significant difference between all groups with highest release again shown by (F IX) followed by (PF), (RL) and lastly (AB). The same result was found for day 15 in the same order as day 7. P-values of post-hoc tests are presented in Table 3.

Intragroup comparisons: (F IX) showed a statistically significant difference in fluoride release between days 1 and 7 with day 1 being significantly higher. Results from day 7 and 15 were comparable and not statistically significant ($p=0.910$) (Table 2). (PF), (RL) and (AB) all showed similar release patterns, with day 7 showing the least amount of fluoride release and day 1 showing the highest (Table 2). There was a statistically significant difference between all tested time points. P-values of post-hoc tests are shown in Table 4.

pH Levels:

Intergroup comparison: Medians and interquartile ranges for pH levels for all groups and time points are shown in Table 5 and illustrated in Figures 7, 8 and 9. The Kruskal-Wallis test showed there was a statistically significant difference between the study groups (p value < 0.05) on day 1. The post-hoc tests showed statistical significance between (F IX) and (RL) ($p=0.001$) with (RL) showing a higher median, and (RL) and (AB) ($p=0.006$) with (RL) showing a higher median as well. There was no statistically significant difference between all other groups. On day 7, there was a statistically significant difference between the groups (p -value < 0.05). (F IX) tended to have the

highest pH value with a median of 5.95. The post-hoc tests showed there was a statistically significant difference between all groups except between (RL) and (AB)($p=0.653$). On day 15, there was also a statistically significant difference found between all groups, with (AB) having the highest median of 5.73. Table 6 shows P-values of all post hoc tests.

Intragroup comparison: (F IX) had a statistically significant difference between days 1, 7 and 15 with day 7 showing the highest median and (IQR) of 5.95 (0.34). (PF) showed no statistical significance between day 1 and 7 ($p=0.570$). The highest median and (IQR) scored was 5.45 (0.03) on day 1 and then slightly decreased gradually. The difference between day 1 and 15 was statistically significant ($p=0.001$). (RL) had a statistically significant result ($p=0.001$) between day 1 and 7, as well as between 1 and 15 ($p=0.001$), but not between 7 and 15 when the Bonferroni correction was used ($p=0.050$), with a similar pattern to (PF). (AB) had statistical significance between day 1 and 7, 1 and 15, and 1 and 15 ($p=0.001$) with day 15 showing the highest level of pH with a median and (IQR) of 5.73 (0.05). P-values of post-hoc tests are shown in Table 7.

Recharge Ability:

Intergroup comparison: Following fluoride recharge using 5% sodium fluoride (NaF) on day 15, all samples were subject to fluoride release analysis at 1 day post recharging (day 16) and day 30. Results are shown in Table 8 and illustrated in Figures 10 and 11. For day 1, the Kruskal-Wallis test showed statistically significant results between groups ($p < 0.05$). The post-hoc tests revealed (F IX) and (AB) with a median and (IQR) of 13.10 (9.97) and 14.30 (7.34) respectively had the highest amounts of fluoride released with comparable results ($p=0.744$). (PF) with a median and (IQR) of 4.57 (1.19) and (RL) with 6.62 (4.49) ($P=0.116$) were also comparable. There was a statistically significant difference between all other groups. P-values of post-hoc test are shown in Table 9.

On day 30, (AB) showed the highest amount released with a median and (IQR) of 27.10 (1.80) and (PF) had the lowest 8.18 (0.86). The Kruskal-Wallis test showed a statistically significant difference between study groups ($p<0.05$). The post-hoc tests revealed a statistically significant difference between all study groups except (RL) and (AB) ($p = 0.775$) and (PF) and (RL) ($p= 0.089$).

Intragroup comparison: Table 10 shows p-values of the Wilcoxon signed-rank tests. (F IX) did not show a statistically significant difference between day 16 and day 30 ($p=0.167$), per the Wilcoxon signed-rank test. All other groups showed statistically significant differences in fluoride release between day 16 and day 30 readings with the latter being significantly higher.

pH levels post recharge:

Intergroup comparison: Presented in Table 11 and Figures 12 and 13, on day 16, the Kruskal-Wallis test showed a statistically significant difference in pH scores among groups ($P<0.05$). (F IX) showed the highest median 5.78 (IQR = 0.12) and (RL) with the lowest 5.61 (0.07). The post-hoc tests showed there was a statistically significant difference between (F IX) and (PF) ($p=0.001$), (F IX) and (RL) ($p< 0.001$), and (RL) and (AB) ($p<0.001$).

For day 30, (PF) exhibited the highest median of pH levels 5.74 (IQR = 0.08) and (RL) exhibited the lowest 5.47 (0.18). The Kruskal-Wallis test showed there was a statistically significant difference between the groups ($p <0.05$). The post-hoc tests showed there was a statistically significant difference between (F IX) and (RL) ($p<0.001$), (PF) and (RL) ($p<0.001$), and (PF) and (AB) ($p<0.001$). P-values are reported in Table 12.

Intragroup comparison: Medians and interquartile ranges are reported in Table 11. For (F IX), the Wilcoxon signed-rank test showed no statistically significant difference between pH values on day 16 and day 30 ($p=0.059$) with day 16 being slightly higher. For all other groups, there was a statistically significant difference found between the two values (PF $p=0.002$) (RL $p=0.004$) and (AB $p=0.001$). P-values of post-hoc tests are presented in Table 13.

Discussion

The objective of this study was to assess the fluoride release and pH levels of four different types of fluoride releasing glass ionomer restorative materials. All tested materials released fluoride with varying amounts.

Fluoride is indeed one of the most essential and effective methods in controlling caries and lowering its rates among populations.⁹ Fluoride presents itself in multiple ways with various efficacy rates.^{47, 48} It is abundantly available in low doses with typically high frequency use as in the cost effective community water fluoridation,^{49, 50} dentifrices and mouth rinses.⁵¹ It is also available to the clinician in high doses and low frequency as in gels and varnishes in addition to low dose with close contact with affected or susceptible areas like these restorative materials presented in this study.⁵¹

This study also evaluated the levels and change of pH these materials may cause. Review of the literature showed many studies that assessed behavior of different materials in different media with various pH levels but none, to our knowledge, assessed their potential effect on surrounding media. It was shown that the storage solution could influence the amount of fluoride released from restorative materials.^{53, 54} In our study, artificial saliva, along with incubation, was chosen as a storage medium in efforts to simulate the clinical conditions as best as possible. Fluoride released from such materials has been shown to have cariostatic effects by elevating salivary fluoride, calcium and phosphate content. They have also shown cariostatic potential on demineralized tooth structure in in-vitro studies.⁵⁵ However, there is very little research done on how much fluoride is necessary to achieve clinical effectiveness. Loyola-Rodriguez et al. suggested that 140 ± 25 ppm of fluoride release is necessary to inhibit *S. Sobrinus* activity in an in-vitro study.⁵⁶ According to our findings, (F IX) had the highest amounts of fluoride released on the initial 'burst' in 24 hrs. Although the presence of (RL) and (AB) is scarce in published research, our results are comparable to previous studies on (F IX) and (PF). Ghajari et al found that Fuji IX released more fluoride in the initial 24 hours as well as post recharging using NaF and APF gels when compared to Chem Flex (hand mixed GI) and Fuji II.⁵⁷ Other authors have also reported similar mean values to ours for the 24hrs fluoride release with (PF).⁵⁸ In another study where three RMGIs were compared with a

compomer, (PF) reported amounts only below Fuji II light cure but higher than Vitremer with Dyract (Compomer) showing the least amounts.⁵⁹ Freedman et al showed that (PF) had the highest total fluoride release and recharge ability when exposed to multiple daily fluoride compared to Ketac-Fil and Dyract.⁶⁰ All groups except (F IX) then had a slight decrease of fluoride release on day 7 only to increase again on day 15. This varying amount and rate of fluoride release could be explained by the interaction between polyacrylic acid content with fluoride compounds and resin amounts in each material and the photochemical initiation polymerization process.⁶¹ Other researchers suggested that there is a ‘rinsing effect’ that leads to this initial ‘burst’ while the fluoride diffuses through materials pores for the next few days.⁶² All groups released significantly more fluoride on day 15 than day 7 except for (F IX), whose fluoride release was slightly lower at day 15 than at day 7.

All samples were assessed for fluoride release after immersion in 5% sodium fluoride (NaF) for five minutes. On day 16, all materials released fluoride with mixed degrees. (AB) and (F IX) showed similarly the highest amounts with (AB) being marginally higher. On day 30, (AB) scored significantly higher amounts of fluoride released than all other groups. This ability to recharge is in accordance with previously published research on glass ionomer materials.^{41, 63}

Moreover, since pH levels are critical for the process of remineralizing affected tooth structure,^{64, 65} it was an important objective to assess whether these materials can affect those levels. All materials were able to keep pH level medians in the range of 4.65 to 5.95. Research has shown that the levels of which *Streptococcus mutans*, a strain that has been identified as key in the process of cavitation,⁶⁶⁻⁶⁸ would be highly active is to likely be in the acidic range of 4.41 to 4.46.⁶⁹ In our study, there were fluctuations in pH levels; the closest to the above range was (RL), which had a median and (IQR) of 4.65 (0.14) at day 15. Out of 15 samples per group, 20% of group (AB) samples fell into the critical pH range on day 7. On day 15, one sample (7%) of group (RL) was found with a level in that same range.

This study assessed specific aspects of these materials. There are many other features to each of these materials that have not been compared. For instance, researchers have suggested that light cured resins may offer superior marginal adaptation while self-cured

materials may be advantageous in areas where light curing is challenging.⁷⁰ Pulpdent® also claims that (AB) offers the advantage of calcium and phosphate release.⁷¹ Both of these minerals have been shown to be important in the dynamics of reversing the early phases of cavitation.⁷²⁻⁷⁴ In fact, calcium and phosphate are main ingredients, along with fluoride, in marketed remineralizing agents such as Casein Phosphopeptide-Amorphous Calcium Phosphate (CPP-ACP).⁷⁵⁻⁷⁷

Although it may be difficult to establish an association between each materials composition and its performance, research has discovered that some components may play a role in their respective material clinical behavior. For example, (F IX) is a conventional glass ionomer cement with an acid and a base mixed in an aqueous medium and indeed showed the highest amounts of initial fluoride release in agreement with previous research.^{62, 41} (PF) has a camphor-quinone element that provides on demand hardening.⁷⁸ (RL) has tartaric- acid incorporated in its mix. This acid was found to not only increase esthetic properties in the form of providing lighter shades but also it accelerates the polymerization process.²⁰ (AB) had the highest resin content amongst our study groups. High resin content has been linked to higher physical strength and better wear resistance.^{20, 21} In addition, (AB) had NaF representing its fluoride content, which is unique compared to other fluoride releasing materials that typically contain aluminofluorosilicate. In a 2013 review of glass ionomers and bioactive glass ionomers, the authors concluded that “smart” bioactive materials and components have been increasingly more incorporated in available materials in today’s dentistry. With focus on conservation of tooth structure and remineralization, these materials have shown great potential improving bioactivity and adaptation to the dynamics of the oral cavity.⁷⁹

Limitations of the Study and Future Studies

In-vitro experiments can only simulate clinical conditions to a certain extent. The oral environment is complex and dynamic in nature and cannot be fully imitated. The short-term nature of this study should also be considered in future studies. It may also be prudent to investigate other aspects of these materials such as marginal adaptation, compressive and shear bond strength.

Long-term clinical trials are needed to better evaluate the fluoride release efficacy along with establishing guidelines for acceptable rates of fluoride release in restorative materials.

Conclusion

With respect to the limitations of this in-vitro study, we can conclude that when stored in artificial saliva:

- Fuji IX tended to score the highest levels of fluoride release.
- Fuji IX and Activa Bioactive tended to show comparably the highest recharge ability on day 16. Riva LC had comparable recharge ability with Fuji IX on day 30 with Activa Bioactive showing significantly the highest amounts.
- During this study's duration, most samples were able to stay in pH values higher than potentially destructive levels.

Appendix A: Tables

Table 1: Materials used in the study

Material	Symbol	Manufacturer	Composition	Cure type	Shade	Lot and serial number
Fuji IX	(F IX)	GC Tokyo, Japan	Water, carboxylic acid, polyacrylic acid, polybasic aluminofluoro silicate glass	Self (chemical) cure	A2	1511201 D65842504204
Photac Fil	(PF)	3M MN,USA	Glass ionomer compatible monomers and oligomers Copolymer acids (acrylic- and maleic acids) Camphor-quinone Na-Ca-Al-La-fluorosilicate-glass Activator (Amine)	Dual cure(chemical/light)	A2	611157 70201143578
Riva LC	(RL)	Southern Dental Industries (SDI) Sydney, Australia	Polyacrylic Acid Tartaric Acid 2-Hydroxyethyl Methacrylate Dimethacrylate Cross-linker Acidic Monomer Fluoroaluminosilicate glass powder	Light cure	A2	J1603041EG DO3687000021J

Activa Bioactive Restorative	(AB)	Pulpdent MA, USA	Diurethane dimethacrylate Bis (2- (Methacryloyl oxy) Ethyl) Phosphate Barium glass Ionomer glass Polyacrylic acid/maleic acid copolymer Sodium fluoride	Triple cure (Light cure, self-cure resin chemistry, and self-cure glass ionomer reaction)	A2	160324 D701VRA225
Vanish		3M MN, USA	5% Sodium Fluoride			N763813 7020108815

Table 2: Median and Interquartile Range of fluoride release in Parts Per Million (PPM) measured at day 1, 7 and 15.

Material	1 Day Fluoride Release	7 day Fluoride Release	15 Day Fluoride Release
	Median (IQR)	Median (IQR)	Median (IQR)
Fuji IX	84.60 (22.30)	26.80 (18.40)	25.60 (19.40)
Photac Fil	14.20 (3.20)	4.28 (0.96)	8.85 (1.59)
Riva LC	6.66 (5.09)	3.35 (1.67)	6.35 (2.67)
Activa Bioactive	6.77 (1.48)	1.09 (0.31)	3.19 (2.08)

Table 3: P-values of post-hoc test for intergroup fluoride release:

Group	Group	P- value		
		1Day	7 Days	15Days
Fuji IX	Photac Fil	<0.001	<0.001	<0.001
Fuji IX	Riva LC	<0.001	<0.001	<0.001
Fuji IX	Activa Bioactive	<0.001	<0.001	<0.001
Photac Fil	Riva LC	<0.001	0.001	0.001
Photac Fil	Activa Bioactive	<0.001	<0.001	<0.001
Riva LC	Activa Bioactive	0.412	<0.001	<0.001

P-value \leq 0.0081 was considered statistically significant.

Table 4: P-values of post-hoc tests for intragroup fluoride release:

Day	Day	P-value			
		Fuji IX	Photac Fil	Riva LC	Activa Bioactive
1	7	0.001	0.001	0.001	0.001
1	15	0.001	0.001	0.047	0.001
7	15	0.910	0.001	0.001	0.001

P-value \leq 0.017 was considered statistically significant.

Table 5: Median and Interquartile Range of pH levels at day 1, 7 and 15.

Material	1 Day pH	7 Day pH	15 Day pH
	Median (IQR)	Median (IQR)	Median (IQR)

Fuji IX	5.45 (0.02)	5.95 (0.34)	5.27 (0.64)
Photac Fil	5.47 (0.03)	5.33 (0.60)	4.90 (0.12)
Riva LC	5.46 (0.01)	4.82 (0.34)	4.65 (0.14)
Activa Bioactive	5.45 (0.05)	4.83 (0.19)	5.73 (0.05)

Table 6: P-values of post-hoc test for intergroup pH values

Group	Group	P- value		
		1Day	7 Days	15Days
Fuji IX	Photac Fil	0.011	<0.001	0.004
Fuji IX	Riva LC	0.001	<0.001	<0.001
Fuji IX	Activa	0.250	<0.001	0.001
Photac Fil	Riva LC	0.624	<0.001	0.001
Photac Fil	Activa	0.081	<0.001	<0.001
Riva LC	Activa	0.006	0.653	<0.001

P-value \leq 0.0081 was considered statistically significant.

Table 7: P-values of post-hoc test for intragroup pH values:

Day	Day	P-value			
		Fuji IX	Photac Fil	Riva LC	Activa Bioactive
1	7	0.001	0.570	0.001	0.001
1	15	0.256	0.001	0.001	0.001
7	15	0.003	0.001	0.050	0.001

P-value \leq 0.017 was considered statistically significant.

Table 8: Median and Interquartile Range of fluoride release post recharge with 5% sodium fluoride measured at day 16 and 30.

Material	16 Day Fluoride	30 Day Fluoride
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	Release	Release
	Median (IQR)	Median (IQR)
Fuji IX	13.10 (9.97)	15.40 (7.10)
Photac Fil	4.57 (1.19)	8.18 (0.86)
Riva LC	6.62 (4.49)	17.30 (13.98)
Activa Bioactive	14.30 (7.34)	27.10 (1.80)

Table 9: P values of post-hoc test for intergroup fluoride release values

Group	Group	P- value	
		16 Days	30 Days
Fuji IX	Photac Fil	<0.001	<0.001
Fuji IX	Riva LC	<0.001	0.775
Fuji IX	Activa	0.744	<0.001
Photac Fil	Riva LC	0.166	0.089
Photac Fil	Activa	<0.001	<0.001
Riva LC	Activa	<0.001	<0.001

P-value \leq 0.0081 was considered statistically significant.

Table 10: P values of post-hoc test for intragroup fluoride release values

Day	Day	P-value			
		Fuji IX	Photac Fil	Riva LC	Activa Bioactive
16	30	0.167	0.001	0.002	0.001

P-value \leq 0.05 was considered statistically significant.

Table 11: Median and Interquartile Range of pH levels at day 16 and 30.

Material	16 Day pH	30 Day pH
	Median (IQR)	Median (IQR)
Fuji IX	5.78 (0.12)	5.67 (0.23)
Photac Fil	5.67 (0.03)	5.74 (0.08)
Riva LC	5.61 (0.07)	5.47 (0.18)
Activa Bioactive	5.69 (0.05)	5.57 (0.04)

Table 12: P values of post-hoc test for intergroup pH values:

Group	Group	P- value	
		16 Days	30 Days
Fuji IX	Photac Fil	0.001	0.250

Fuji IX	Riva LC	<0.001	<0.001
Fuji IX	Activa	0.016	0.015
Photac Fil	Riva LC	<0.001	<0.001
Photac Fil	Activa	0.116	<0.001
Riva LC	Activa	<0.001	0.016

P-value ≤ 0.0081 was considered statistically significant.

Table 13: P values of post-hoc test for intragroup pH values:

Day	Day	P-value			
		Fuji IX	Photac Fil	Riva LC	Activa Bioactive
16	30	0.059	0.002	0.004	0.001

P-value ≤ 0.05 was considered statistically significant.

Appendix B: Figures



Figure 1: Orion Star A214 pH and fluoride electrodes (Thermo scientific, No 0809, Beverly, MA)



Figure 2: Translux Wave (Heraeus Kulzer Hanau, Germany)

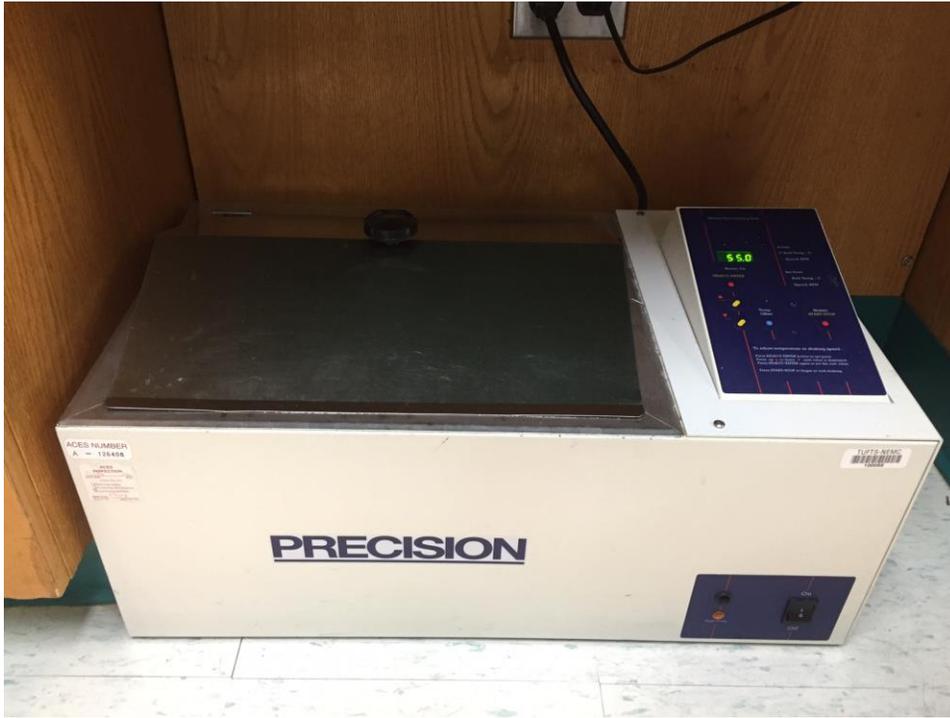


Figure 3: Precision water bath incubator (LabX, ON, Canada)

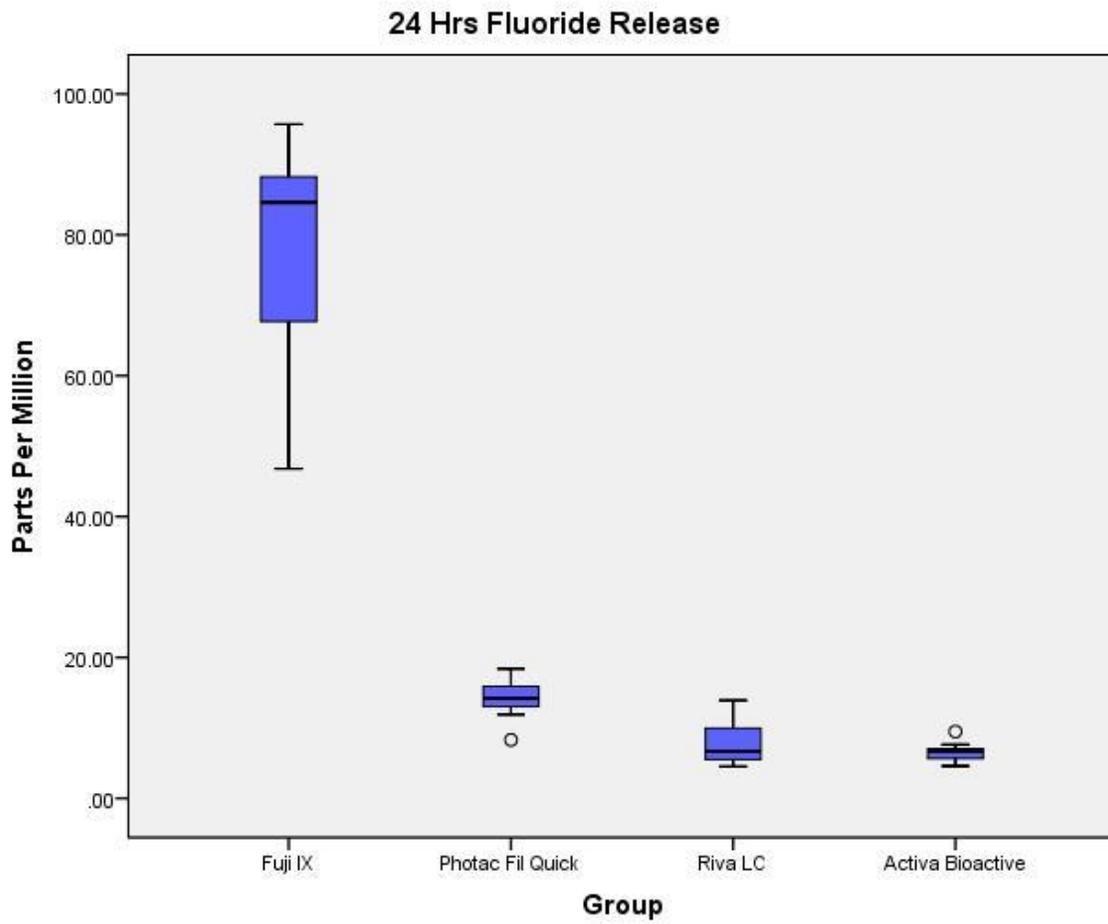


Figure 4: Side-by-side boxplots showing 24 hrs fluoride release scores.

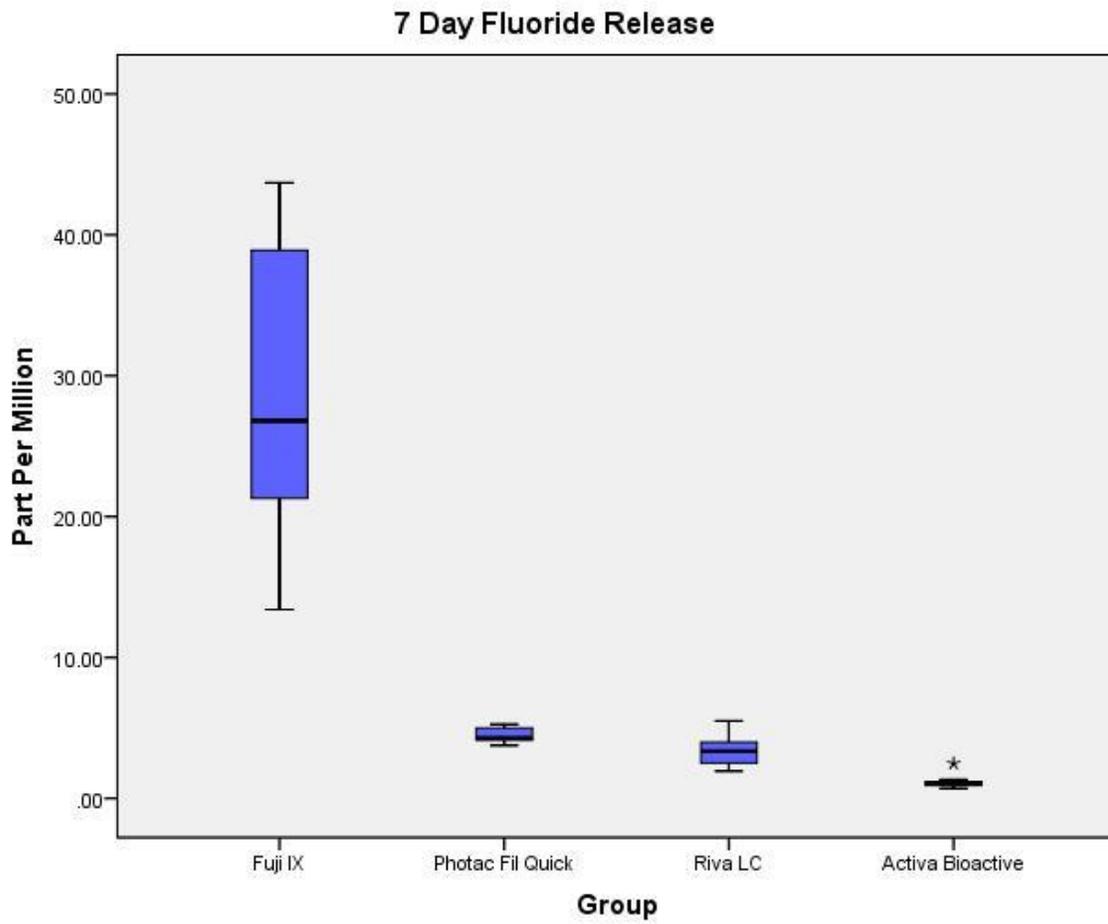


Figure 5: Side-by-side boxplots showing day 7 fluoride release scores.

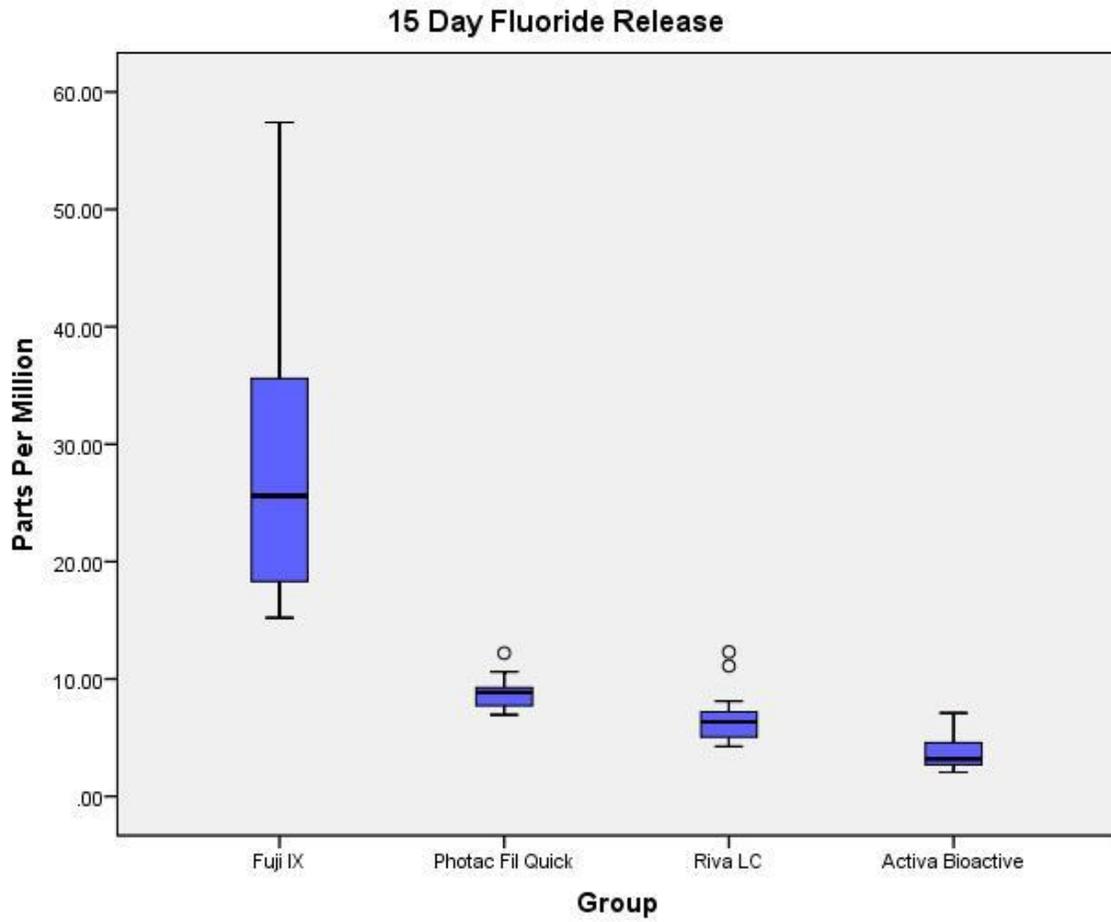


Figure 6: Side-by-side boxplots showing 15th day fluoride release scores.

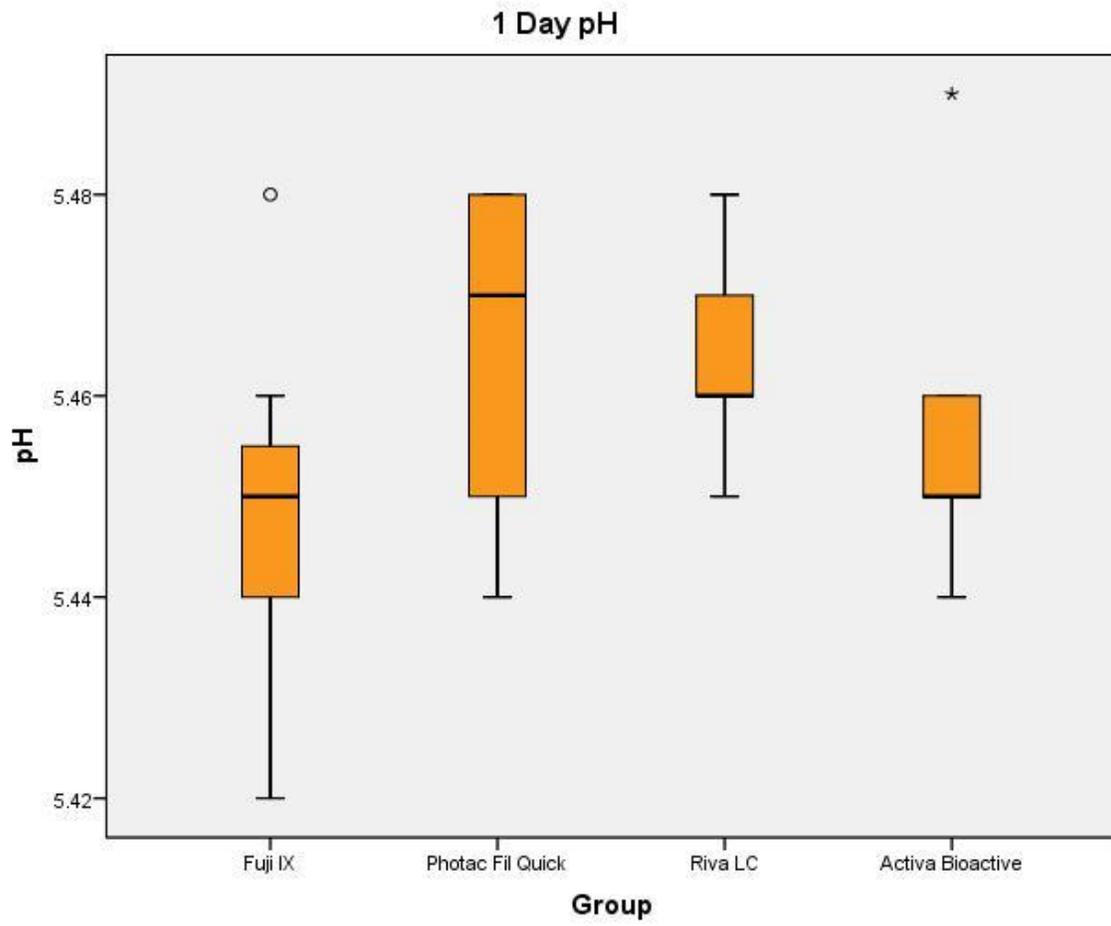


Figure 7: Side-by-side boxplots showing 24 hrs pH levels.

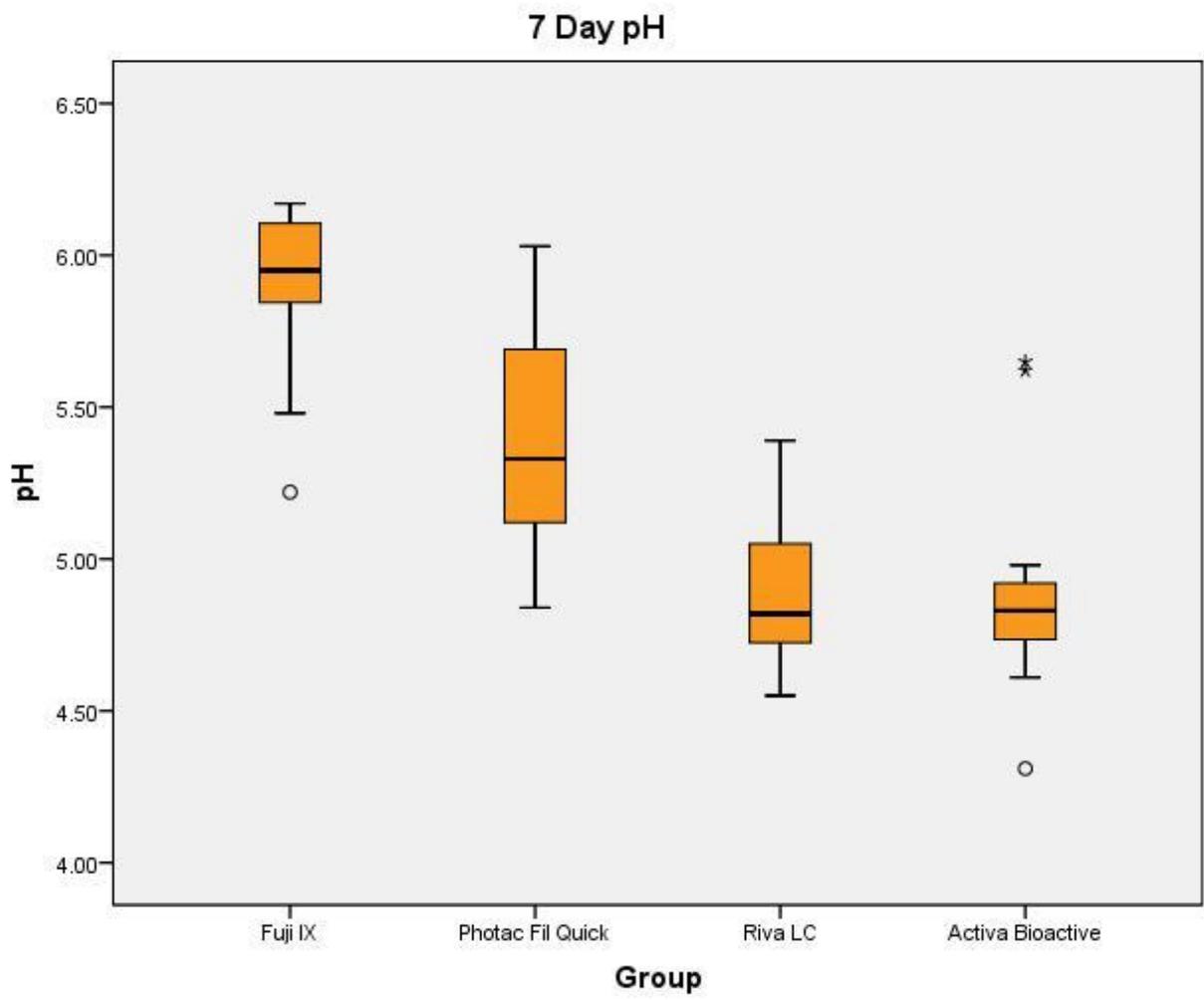


Figure 8: Side-by-side boxplots showing 7 days pH levels.

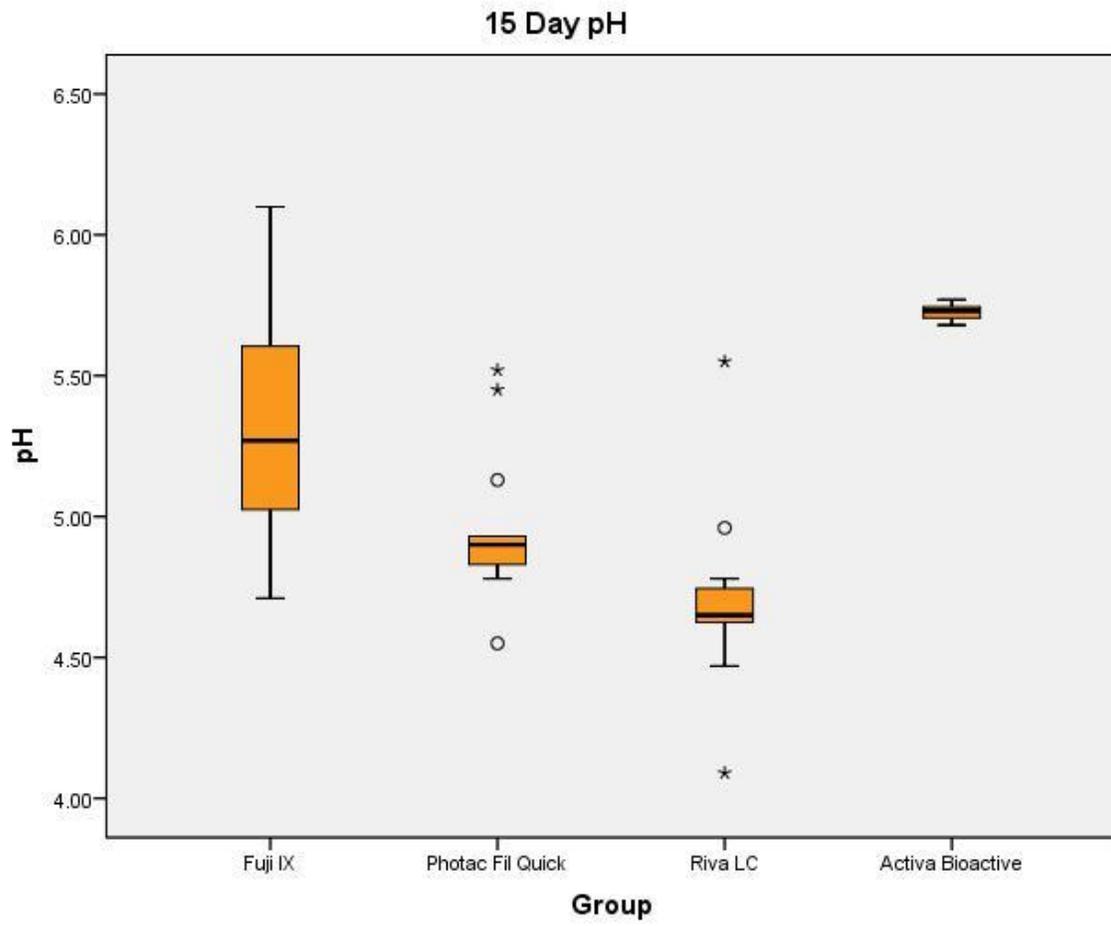


Figure 9: Side-by-side boxplots showing 15 days pH levels.

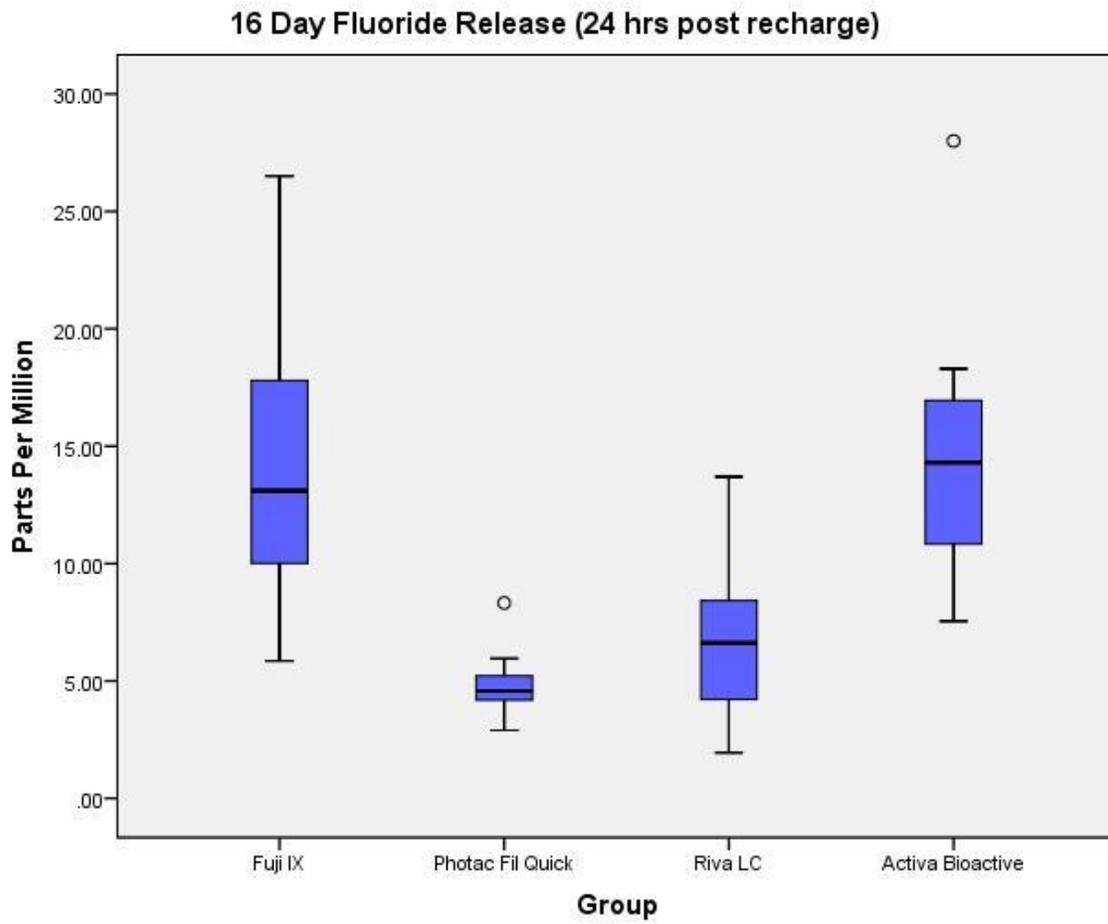


Figure 10: Side-by-side boxplots showing 24 hrs post recharge fluoride release scores.

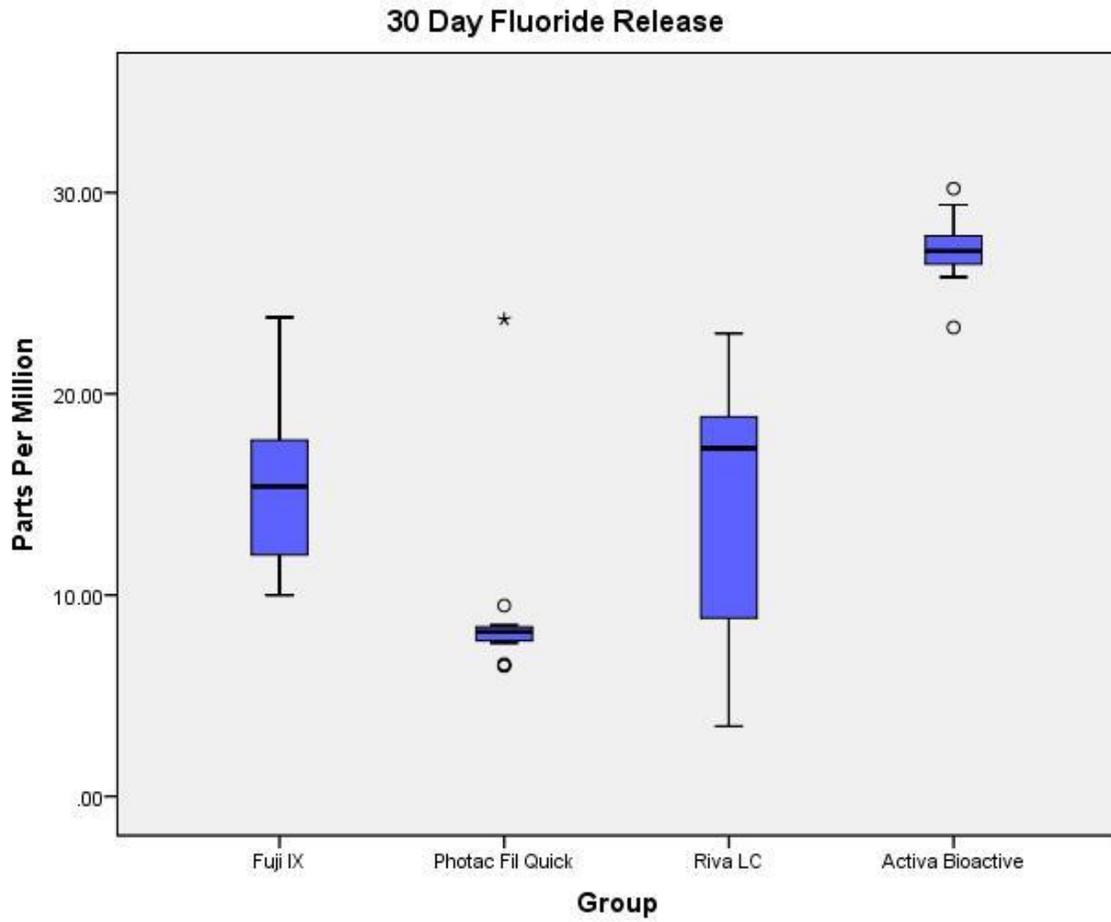


Figure 11: Side-by-side boxplots showing day 30 fluoride release scores.

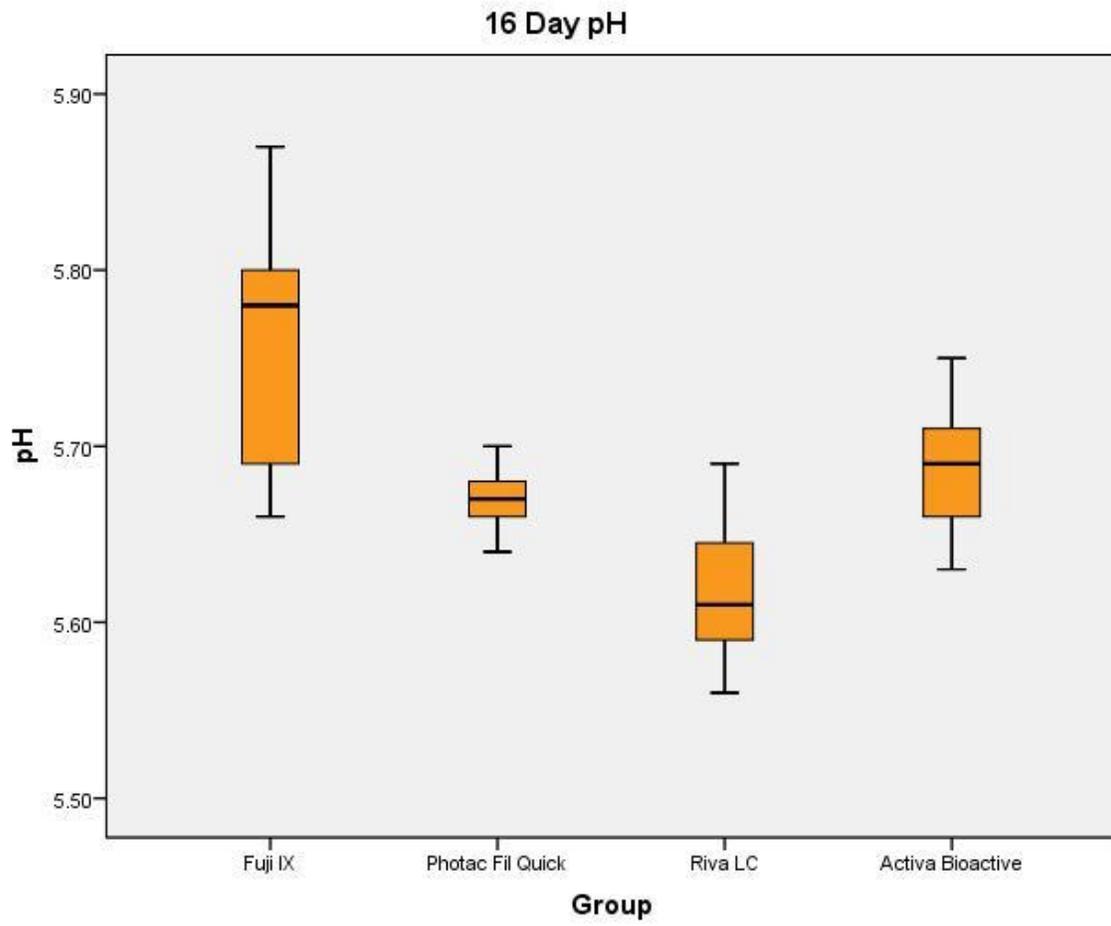


Figure 12: Side-by-side boxplots showing 24 hrs post recharge pH levels.

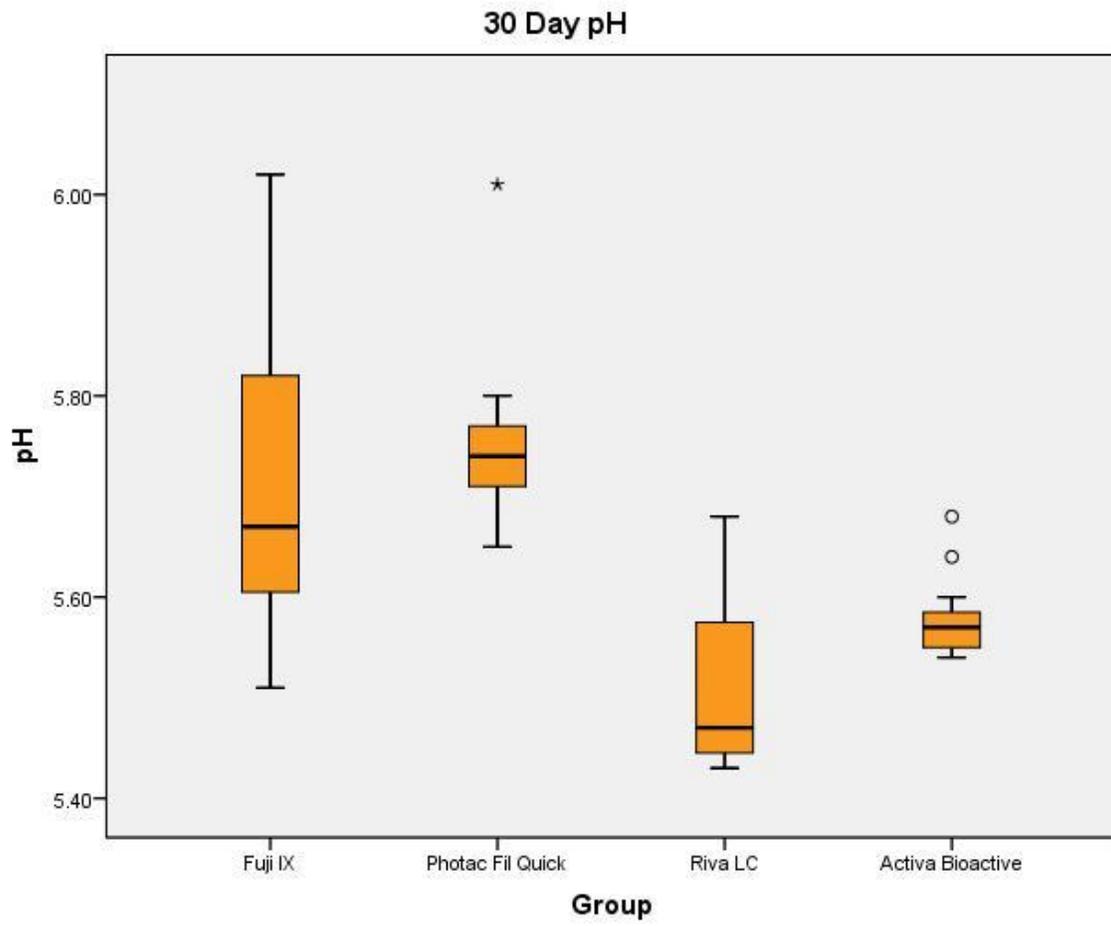


Figure 13: Side-by-side boxplots showing day 30 pH levels.

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