

Vol 13, No 1 (2025) ISSN 2167-8677 (online) DOI 10.5195/d3000.2025.979

Fluoride Release of New Bioactive Orthodontic Adhesive with Color Change and Fluorescent Property

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Abstract

Objective: This study aimed to assess the effect of adding fluorescent dyes & color change dyes in different concentration to bioactive orthodontic adhesive on fluoride release. **Material and Methods**: We used Bioactive BEAUTIFIL Injectable XSL (S-PRG), from (Giomer, Shofu, Japan) mixed with color change dye, Black changing to Colorless, (Atlanta chemical engineering, USA). 0.02%, 0.2% and 2% of weight concentrations were tested and with fluorescence dye (Strontium aluminate), and White Glow in the Dark Powder (Techno Glow Inc., USA), using in 5%, 10% and 15% of weight concentrations. For fluoride release, 40 samples prepared and divided into 4 groups with 10 samples as following: Group 1: BEAUTIFIL Injectable XSL Adhesive (control group), Group 2: BEAUTIFIL Injectable XSL with 0.02% color change material and 5% fluorescence material. Group3: BEAUTIFIL Injectable XSL with 0.2% color change material and 10% fluorescence material. Group 4: BEAUTIFIL Injectable XSL with 2% fluorescence material and 15% fluorescence material. fluoride Ion Selective Electrode. Eutech ION 2700. (Thermo Fisher scientific inc. Singapore) used to measure the release of fluoride ion. **Results**: The use of dyes with bioactive adhesive showed statistically significant differences. There was a decrease in fluoride ion with increase dye concentration. **Conclusion**:

Acceptable fluoride ion release within bioactive adhesive with color change and fluorescence properties was obtained but with increase concentration of dyes the ion release decreased.

Open Access

Citation: Younis MT, et al. (2025) Fluoride Release of New Bioactive Orthodontic Adhesive with Color Change and Fluorescent Property. Dentistry 3000. 1:a001 doi:10.5195/d3000.2025.979

Received: July 3, 2025 Accepted: July 4, 2025 Published: August 21, 2025

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Introduction

Orthodontic treatment involves using fixed or removable appliances to correct the positions of teeth. Fixed appliance treatment is a traditional and widely used form of orthodontic treatment to correct malpositions of the teeth and occlusal discrepancies [1,2]. The movement of teeth is achieved by forces

The movement of teeth is achieved by forces generated and directed to the teeth via arch wires and brackets [3]. During the active treatment, the arch wires are changed as the treatment progresses, but the brackets remain attached to the enamel for the whole active treatment period. A wide variety of orthodontic adhesives are available for the bonding brackets and orthodontic attachments [4,5]. Resin adhesives are a good

choice for orthodontic bonding as they have good mechanical and aesthetic properties and low failure rates. Orthodontic adhesives should provide sufficient strength to retain the appliance during treatment and allowing its easy removal at the end [6].

Patients with fixed orthodontic appliances show increased risk of white spot lesions (WSL) [7] and caries due to difficulty in maintaining oral hygiene [8,9].

Attempts were made to reduce enamel demineralization by introducing fluoride releasing adhesives [10], e.g. amorphous calcium phosphate (ACP) containing adhesives [11]. We tested Light Bond from Reliance company, Transbond Plus Adhesive from 3M Unitek company [12], glass ionomer and

resin modified glass ionomer [13], fluoridate varnish [14], and topical fluoride agents [15]. Another solution proposed by research teams is introduction of bioactive glass (BAG) into the composition of orthodontic adhesives [16].

Bioactive glass material included the Giomers, as a surface pre-reacted glass core (Sprg). It helps prevent and treat white spots lesions [17]. Giomers are useful in the process of collating orthodontic brackets with mechanical properties like composite resins and it offers protection against carious lesions [18]. S-prg releases various ions (fluoride, sodium, silicate, aluminum, borate and strontium ions that provide multiple biological functions, including the release and

recharge of fluoride, anti-plaque, anti-biofilm effects and pH modulation that providing protection against caries [19].

Another problem associated with orthodontic adhesive is the presence of excess of adhesive escaping from under the bracket base which promotes the accumulation of food debris and creates favorable area for bacterial collection which led to facilitating the demineralization and formation of WSL [20,21].

The solution turned out to be introduce adhesive characterized by a contrasting color before cross-linking, which facilitates removal of the excess prior to curing, e.g. Transbond Plus (3M Unitek, USA), and Grengloo and Blugloo (Ormco Corporation, USA) [22]. Color change adhesive is manufactured by adding chromatic indicators, which facilitate the visibility of excess orthodontic adhesive around orthodontic brackets before bonding procedure [23]. This color characteristic allowed the operator to see the adhesive flash around the bracket base and remove it before it polymerized [241].

The third problem associated with orthodontic treatment is debonding [25], Leaving the remnant of orthodontic adhesive on the enamel surface facilitates plaque and dental caries formation [26]. Also, using rotating dental instruments to remove the remaining white or transparent adhesive can cause damage to tooth enamel [27].

To overcome this problem, fluorescent orthodontic adhesive has been developed to improve the visibility of the remaining adhesive after debonding by using ultraviolet light [28,29]. Fluorescent additives will facilitate the discrimination between the enamel and remnants of the adhesive. This modification can maximize the preservation of tooth structure after debonding procedure [30].

The aim of this research was to evaluate the effect of adding color change and fluorescent dies to bioactive composite to be used as an orthodontic adhesive by evaluate the degree of conversion.

Materials and Methods

This study was conducted at Mosul University, Dentistry College, Dental Hospital Central Laboratory and was approved by the Ethics Committee of College of Dentistry, Mosul University, Iraq (under the code UoM.Dent.23/49).

To measure fluoride release, 40 samples of the tested materials (10 sample for each group) were used:

Group 1: BEAUTIFIL Injectable XSL Adhesive (control group)

Group2: BEAUTIFIL Injectable XSL with 0.02% color change material and 5% flourcence material.

Group3: BEAUTIFIL Injectable XSL with 0.2% color change material and 10% flourcence material.

Group4: BEAUTIFIL Injectable XSL with 2% color change material and 15% flourcence material.

Plastic cylinder molds measuring 4 mm in diameter and 2 mm in height were used to construct the specimens for each group. Celluloid mylar strips and glass slides were placed over the mold's top and bottom surfaces. as at room temperature, the adhesive then put within the mold. The central of samples were pressed between a celluloid strip and a glass cover slip and placed in plastic mold rings to extrude the excess material, to obtain a smooth flat surface, to prevent air bubble formation & to prevent oxygen layer formation. Then light cure the materials by using LED with at 1500 mw/cm² were cured from the top & bottom for 20sec. After light curing, each set specimen was released from the mold and placed into a polyethylene test tube. filled with 5 ml of deionized water. After that, each test tube was sealed, labelled, and arranged as previously indicated before being kept for 24 hours at 37°C with 95% relative humidity [31=-33].

Fluoride measurements were performed on 1st day (24 h), 7 days (1 week), and 30 days (1 month). All of the samples were maintained at 37°C in a incubator throughout the experiment. In this examination before testing the containers were thoroughly shaken and the specimens removed, washed, returned and immersed into a new 5 ml of fresh deionized water fresh solution. Meanwhile, the fluoride ions concentration in the storage media was measured. The 1st measurement was done after 24 h from sample preparation, and then 24 hours before day 7th and day 30th. The storage medium was changed with new 5 ml of fresh deionized water fresh solution every 24 h to avoid cumulative effects and because it is possible that the medium may get saturated with the released fluoride ions, preventing further fluoride ion release. This was done by using a fluoride Ion Selective Electrode (Eutech ION 2700 meter, Thermo Fisher Scientific Inc., Singapore) attached to an ion selective electrode meter. For statistical analysis, the amount of fluoride in each solution was measured and recorded as part per million (ppm). Before and after each measurement, the electrode tip was washed and lightly dried with deionized water to remove any residual fluoride ions which may affect the measurement [34,35] (Figure 1).

Results

Fluoride release values are represented in units of part per million (ppm) and shown in Table 1. The analysis of variance of one way

(ANOVA) test for each group revealed significant differences ($p \le 0.05$).

Discussion

Fluoride is clearly known as an anti-caries agent, and fluoride release is an important part of restorative materials, fluoride can help reduce tooth decay by reducing bacterial metabolism and increasing the resistance of enamel and dentin [36,37]. Giomer is one of modern restorative material that contain in its chemical structure combination of fluoroaluminosilicate glass, polyalkenoic acid and water, with resin included. What differentiates giomer from other fluoride-containing restorative materials is that they contain a pre-reacted glass (S-PRG) filler in their matrix. This filler facilitates the release of fluoride ions [35,38]. Due to the absence of an acid-base reaction, giomer materials do not have a glass ionomer matrix phase. The quantity of fluoride released by giomer materials was discovered to be less than that of GIC since they only include S-PRG particles as a fluoride component. The materials' antibacterial properties rely on metal ions such aluminum, strontium, zirconium, and barium in addition to the fluoride that is emitted [39,40]. Numerous studies shown that giomer possesses physical qualities that might compete with other composite resin, as well as a high fluoride release and rechargeability [41]. The quantity of water absorbed, the giomer's porosity, the filler, the water content, the solubility of ytterbium trifluoride in water, and the resin's permeability all affect how many ions are released from the giomer [42]. Giomer has a fluorine concentration of only 4.13% [43].

Fluoride release from the material is important due to the formation of fluorapatites as well as the anti-caries property that can prevent the formation of microorganisms. The smallest quantity of fluoride that must be released to inhibit demineralization and promote the remineralization has not been precisely determined [44]. Some authors reported that this value would be between 0.02 and 0.06 ppm [45]. Others said that 0.2 ppm significantly reduces the risk of dental caries lesions [46].

Fluoride released from the restorative material reduced the solubility of dental tissue in acidic environments, this property being based on the fluoride capacity to incorporate itself into the crystalline structure of the hydroxyapatite of the dental hard tissue, resulting in a mineral phase which was less soluble & more resistant to the cariogenic challenge. So, since enamel solubility is low when fluoride ions are present in saliva & biofilm, it is desirable to select dental materials with the highest & longest fluoride release [47,48].

Many methods have been employed to estimate the sum of fluoride releases such as spectrophotometry, ion chromatography, fluoride ion-specific electrodes and capillary electrophoresis [49]. Ion-specific electrode with an ion analyzer was used in this study because it is simple, inexpensive and does not require the use of complex laboratory equipment. Also, it gives an accurate and direct estimate of the free fluoride present in the solution [50].

To measure the fluoride release of restorative materials, a variety of media, including deionized water, artificial saliva and acidic media, may be selected. Since deionized water is readily available & contains no ions, it is believed that fluoride release may be more accurately quantified. For this aim, deionized water used in our research protocol to be consistent with previous investigations [48,51].

In this study, we can see two events. One, it is related with decrease the rate of fluoride release with time for all groups. So, the 1^{st} day show the highest rate then followed by 7th day while the 30 day show the lowest fluoride rate. The 2nd one related to the groups was that the control group (adhesive only) showed the highest followed by adhesive +5% fluorescence+0.02% color change, then adhesive +10% flourcence +0.2% color change and finally adhesive +15% fluorescence +2% color change showed the lowest fluoride release rate. These occurred due to the many reasons; one of them related to the time, it because to the statement that fluoride release drop with time due to the mechanism of its release as suggested by many authors. According to this mechanism, S-PRG is composed of three layers: a multifunctional glass core is the innermost layer, followed by a glass ionomer phase from the acid-base reaction that contains the polyacrylic acid chain and ions trapped in the phase, and the surface-modified layer (porous inorganic silica glass layer) at the outermost layer. Diffusion of the fluoride ion from the intermediate layer to the environment is the mechanism of fluoride release. A fluoride ion is traded for a hydroxyl ion in an ion exchange process, which is the primary mechanism of fluoride ion release in giomers [52].

The complex process of fluoride ion release is influenced by both internal and external factors, including the type and permeability of the filling material, the frequency of fluoride exposure, the type and concentration of the fluoridating agent, temperature, preparation method, material solubility, composition, powder-liquid ratio, surface area of the specimen, matrix, filler composition, storage medium, saliva composition and acidity and the type and concentration of the fluoridating agent. The precise quantity of fluoride

released, which inhibits demineralization and encourages remineralization, is unknown. However, since fluoride ions in the oral cavity decrease enamel solubility, it is better to utilize materials with a high and sustained fluoride ion release [53-55].

Although the exact mechanism behind the ion release from S-PRG filler is unknown, it is thought that the existence of a glass ionomer phase around the filler's glass core is connected to the ion release [56].

Fluoride was released from the surface in a burst in the first step, which is followed by a significant reduction in elution and a second bulk diffusion process that releases minute quantities of fluoride into the surrounding medium. Fluoride is thus released on the first day due to a surface rinsing impact and on following days its diffusion via pores and fractures is responsible for the release. For the fluoride ions to diffuse, the resin monomer gradually absorbs water [57,58].

So, in the first phenomenon of fluoride release called the "Burst Effect". The second bulk diffusion phase, which releases fluoride in minute quantities via the material matrix pores, occurs concurrently with the later slow release. This could have to do with the kind of fillers used. Another aspect may be the bonding between the matrix and the fillers. It was discovered that more microporosities may have resulted from the dye particles' inability to connect with the adhesive matrix, which might have facilitated the release of fluoride which agrees with Bansal & Bansal in 2015 & Dawood et al in 2019 [57-59]. It is shown that giomers have a lower fluoride release than glass ionomers, with no early fluoride burst impact, but that fluoride release levels are mostly constant. The initial intense release of these ions may also be due to surface leaching, while its subsequent stabilization results from the diffusion of fluoride ions through the pores and fractures of the material [38,58].

Additionally, the ionic interaction on the surface of the glass particles or water absorption after the dissolution of the glass filler particles might cause fluoride release [60]. In addition, the 1st step of fluoride released from the surface of giomer after which the elution is markedly reduced, accompanied by the second bulk diffusion process by which small amounts of fluoride continue to be released into the surrounding media [61]. As during water penetration through diffusion, the surface layers will be more saturated than the inner mass leading the material can leach ions from the mass that have been penetrated by water & the penetration of water is different for different materials, depending on the permeability of materials [62,63]. Anyway the release of fluoride from giomer is more water-exposure dependent [64]. When the S-PRG encounters water, the ions that are not bonded in the polymerization chain created in giomer are dissolved. For example, the polymer in the giomer will react to create a polymer chain when exposed to light. Many ions that are not part of the polymer chain are present in the polymer chains in order for them to dissolve in the immersion solution. Fluoride is one of the ions not included in this polymer chain [42,65].

The results of this study agree with Sangeetha in 2005 who approved that the fluoride release rate was maximum at 1st day then subsequently dropped to a lower level after one week and had reached a near constant level at 30th day [66]. Salmerón-Valdés et al in 2016 said, in vitro, the degree of fluoride released from giomer was maximum during the 1st 24 hs, then after 8 days showed minimum levels of released fluoride [67]. Garoushi et al (2018) who calculated the daily release of fluoride over a period of 10 days from bioactive materials, he said that fluoride start to decrease from 1st day with until the 10th day [48]. Also Nahum et al in 2021 said that all materials analyzed in his study demonstrate the greatest fluoride release in the first 24 h, followed by a marked decrease after 5th days (68). Feiz et al in 2022 approve that the supreme mean of fluoride released during the days 1st, 3rd, and 7th then decrease on day 14th [37]. Pastrav et al in 2021 and Marnani & Kazemian 2024 suggested in their studies that the dental materials which release fluoride ions show highest activity on the 1st day after setting, followed by a gradual decrease in the number of ions released over the following days, months and years [34,69]. Harhash et al in 2019 discovered that after the first day, the commercial giomer Beautifil Flow Plus F03, A2 color, emitted 1.0020 ppm of fluoride, 0.4140 ppm after the first week, and 0.3165 ppm after the fourth week [42]. While Rusnac et al in 2021, according to his research, the experimental giomer emitted 1.87 ppm of fluoride after the first day, 0.766 ppm after a week, and 0.307 ppm after 30 days. Fluoride levels in the giomer B-F03 were 3.1 ppm after the first day, 0.442 ppm during the first week, and 0.242 ppm after 30 days [70].

While in the other side like Zabokova *et al* in 2011 said that the amount of fluoride after 3 & 6 months it was higher compared to initial values this may be due the material & technique that he used it in his research [71].

The second phenomena occur due to the many reasons one of them is the presence of filler within constant sample lead to decrease the size of adhesive which leading to decrease the quantity of fluoride in the testes sample when compare with the control sample. However, other writers hypothesized

that the high degree of conversion from a double to a simple bond, which results in the cohesiveness of polymer networks and lowers the mobility of ions like fluoride, might be the cause of the reduced release. A double bond's high degree of conversion to a single bond (-c=c-) (c-c). which mean that increase in polymerization would result in entrapment of fluoride ions inside the lattice of the polymer, so the amount of fluoride release will be decreased [72,73]. This can be disagreeing with our results related with DC, this may be due to the difference between size of samples & presence of dyes filler that produce some voids within mixture leading to release fluoride from this voids this agree with Alinda et al (2021) who said, voids play a significant role in releasing fluorine ions [40].

Al-Shekhli & Al_Aubi in 2020 said that incorporation of filler in the composition of giomer restorative material tend to be affected by water exposure more than other filler types incorporated in restorative [74]. Jitaluk *et al.* (2022) implied that adding 20 weight percent nanofillers to resin improved the fluoride exchange compared to using microfillers at the same amount [35].

The difference in our result of fluoride release from results in another studies may be related to the difference in the type and size of material also due to the difference in the type and size of storage media that used this agree with Burtea *et al.* (2019) [52].

Anyway, the decrease in the fluoride release rate can be raised by fluoride recharge as suggested by Barakat & Abdelrahim in 2022, who claimed that after being exposed to fluoridated chemicals, the giomer could be recharged and re-release fluoride gradually [50]. Giomer can be recharged with fluoride ions by topically applied NaF gel and fluoridated toothpaste () ⁽⁶⁴⁾.

Finally, the quantity of fluoride that is released from the specimens cannot be anticipated to be the same as what happens within the mouth. The true effectiveness of restorations can only be ascertained by long-term clinical trials, even if laboratory investigations are crucial for providing quick answers to certain concerns & It should also be considered that the results were obtained in experimental conditions that cannot completely reproduce the conditions of the oral environment this agree with Naoum et al (2012) & Gateva et al (2023) [38,75]. Giomer can release and recharge fluoride & it showed a higher recharge potential compared to other fluoride-containing composite materials [50,76].

Conclusions

Acceptable fluoride ion release within bioactive adhesive with color change and fluorescence properties are obtained but with increase concentration of dyes the ion release decreased. It could advisable to use fluoridated supplements to compensate this decrease.

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Figure 1. Samples used to test fluoride release.

Table 1. Fluoride release values in ppm for all groups.

Groups	N	Time	Minimum	Maximum	Mean	Std. Deviation
Control	10	24 h	1.64	1.74	1.67	0.0374
		7 day	1.49	1.57	1.53	0.0251
		30 day	1.47	1.51	1.49	0.0139
5%+0.02%	10	24 h	1.60	1.72	1.66	0.0368
		7 day	1.49	1.62	1.52	0.0365
		30 day	1.43	1.51	1.48	0.0244
10%+0.2%	10	24 h	1.54	1.66	1.61	0.0442
		7 day	1.43	1.59	1.51	0.0463
		30 day	1.40	1.50	1.47	0.0294
15%+ 2%	10	24 h	1.54	1.64	1.59	0.0316
		7 day	1.47	1.57	1.51	0.0319

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Table 2. Duncan's analysis for determining the difference between the groups.

Groups	Time	1	2	3	4	5	6	7	8	9	10
Control	24hrs	1.73	1.65	1.64	1.65	1.68	1.74	1.64	1.64	1.65	1.68
	7 days	1.55	1.57	1.54	1.51	1.49	1.54	1.55	1.54	1.52	1.5
	30 days	1.49	1.49	1.51	1.47	1.48	1.51	1.49	1.51	1.48	1.49
5%+0.02%	24hrs	1.62	1.67	1.68	1.7	1.66	1.6	1.67	1.68	1.72	1.63
	7 days	1.5	1.53	1.54	1.49	1.52	1.5	1.62	1.53	1.52	1.51
	30 days	1.49	1.5	1.5	1.47	1.46	1.46	1.51	1.49	1.49	1.43
10%+0.2%	24hrs	1.64	1.65	1.64	1.54	1.59	1.65	1.66	1.65	1.55	1.6
	7 days	1.58	1.51	1.43	1.49	1.53	1.59	1.52	1.5	1.49	1.54
	30 days	1.49	1.46	1.4	1.48	1.5	1.5	1.47	1.48	1.46	1.49
15%+ 2%	24hrs	1.6	1.59	1.54	1.58	1.64	1.61	1.61	1.55	1.59	1.63
	7 days	1.56	1.51	1.49	1.5	1.48	1.57	1.52	1.51	1.52	1.47
	30 days	1.51	1.45	1.48	1.48	1.44	1.52	1.46	1.42	1.48	1.43

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