

## Evaluation of wear resistance and fluoride release from glass ionomer covered with nanofilled self-adhesive coat: An in vitro study

Zahra Lari<sup>1</sup>, Fariba Ezoji<sup>2</sup>, Hemmat Gholinia<sup>3</sup>, Effat Khodadadi<sup>4✉</sup>

1. Dental Student, Student Research Committee, Babol University of Medical Sciences, Babol, IR Iran.

2. Assistant Professor, Dental Materials Research Center, Health Research Institute, Babol University of Medical Sciences, Babol, IR Iran.

3. MSc in Statistics, Health Research Institute, Babol University of Medical Sciences, Babol, IR Iran.

4. Associate Professor, Oral Health Research Center, Health Research Institute, Babol University of Medical Sciences, Babol, IR Iran.

✉**Corresponding Author:** Effat Khodadadi, Department of Pediatric Dentistry, Faculty of Dentistry, Babol University of Medical Sciences, Babol, IR Iran.

**Email:** Dr\_ekhodadadi@yahoo.com

**Tel:** +981132291408

**ORCID** (0000-0002-3196-7615)

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### Abstract

**Introduction:** The resin-modified glass ionomer (RMGI) materials are hybrid materials including conventional glass ionomer and small addition resin. They have the advantages of both, such as adhesion to tooth structure, esthetics and fluoride release. The purpose of this study was to compare the fluoride release and wear resistance of two types of RMGI.

**Materials & Methods:** In this in vitro study, specimens for measuring the fluoride release were made with brass mold from two types of RMGI EQUIA Forte glass ionomer and Fuji II conventional glass ionomer. After incubating the specimens in a plastic vial containing 7 ml of distilled water, the release of fluoride for each specimen was measured on days 1, 7 and 15 using a PH/Ion meter device. For evaluating the wear resistance, 8 specimens were prepared using a brass mold in each group. Then, each specimen was exposed to 5000, 10000, 20000, 40000, 80000 and 120000 cycles in wear simulator. The specimens' weight was measured by an electronic weight balance before and after each wear cycle. The data were analyzed by SPSS-17 through repeated-measurement ANOVA and independent T-test.

**Results:** Fluoride released from Fuji II glass ionomer was significantly higher in the first 24 hours and on day 7. During the day 15, the differences in fluoride release between the two glass ionomer groups were not statistically significant. ( $p < 0.001$ ). The EQUIA Forte glass ionomer exhibited significantly greater wear resistance in different wear cycles. ( $p < 0.001$ )

**Conclusion:** Due to its good wear resistance, acceptable fluoride release, ease of clinical use and infection control, the capsular glass ionomer can be a useful material in people at high risk of caries, temporary restoration techniques, pediatric dentistry and root caries.

**Keywords:** Glass ionomer cements, Fluorides, Glass ionomer

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## بررسی مقاومت سایش و آزاد سازی فلوراید از گلاس آینومر پوشش داده شده با پوشش نانو سلف ادهزیو: یک مطالعه آزمایشگاهی

زهرا لاری<sup>۱</sup>، فریبا ازوجی<sup>۲</sup>، همت قلی نیا<sup>۳</sup>، عفت خدادادی<sup>۴\*</sup>

۱. دانشجوی دندانپزشکی، کمیته تحقیقات دانشجویی، دانشگاه علوم پزشکی بابل، بابل، ایران.
  ۲. استادیار، مرکز تحقیقات مواد دندانپزشکی، پژوهشکده سلامت، دانشگاه علوم پزشکی بابل، بابل، ایران.
  ۳. کارشناسی ارشد آمار، پژوهشکده سلامت، دانشگاه علوم پزشکی بابل، بابل، ایران.
  ۴. دانشیار، مرکز تحقیقات سلامت و بهداشت دهان، پژوهشکده سلامت، دانشگاه علوم پزشکی بابل، بابل، ایران.
- \*نویسنده مسئول: عفت خدادادی، گروه دندانپزشکی کودکان، دانشکده دندانپزشکی، دانشگاه علوم پزشکی بابل، بابل، ایران.  
پست الکترونیکی: dr\_ekhodadadi@yahoo.com تلفن: +۹۸۱۱۳۳۲۹۱۴۰۸

### چکیده

**مقدمه:** رزین مدیفايد گلاس اینومر موادی هیبریدی مشتمل بر گلاس اینومر معمولی و مقادیری رزین هستند. این مواد مزایایی از هر دو جز مانند اتصال به بافت دندان، زیبایی و آزاد سازی فلوراید را دارا هستند. هدف این مطالعه مقایسه مقاومت سایش و آزاد سازی فلوراید دو نوع رزین مدیفايد گلاس اینومر بود.

**مواد و روش ها:** در این مطالعه آزمایشگاهی، نمونه ها برای اندازه گیری آزاد سازی فلوراید با مولد برنجی از دو نوع رزین مدیفايد گلاس اینومر (*EQUIA Forte glass ionomer and Fuji II conventional glass ionomer*) ساخته شدند. نمونه ها در ویال پلاستیکی حاوی ۷ میلی لیتر آب مقطرانکوبه و آزاد سازی فلوراید با دستگاه *PH/Ion meter* اختصاصی یون فلوراید، روزهای ۷، ۱۵ و ۱۵ اندازه گیری شد. برای سنجش مقاومت سایشی از هر گروه ۸ نمونه با استفاده از مولد برنجی آماده شدند. سپس هر یک از نمونه ها تحت سیکل های ۵۰۰۰، ۱۰۰۰۰، ۲۰۰۰۰، ۴۰۰۰۰، ۸۰۰۰۰ و ۱۲۰۰۰۰ دور با استفاده دستگاه سایش قرار گرفتند. نمونه ها قبل و بعد هر سیکل سایشی با ترازوی دیجیتالی وزن شدند. جهت انجام آنالیز آماری نرم افزار *SPSS 17* و آزمون آماری (*Repeated Measurement ANOVA*) و *T-test* مستقل استفاده شد.

**یافته ها:** فلوراید آزاد شده *Fuji II* در ۲۴ ساعت اول و روز ۷م اندازه گیری به طور معناداری بالاتر بود ( $p < 0.001$ ). میزان فلوراید آزاد شده روز ۱۵م دو گروه گلاس اینومر تفاوت معناداری نداشت. مقاومت به سایش گلاس اینومر *EQUIA Forte* در سیکل های مختلف سایشی، به طور معناداری بالاتر بود. ( $p < 0.001$ )

**نتیجه گیری:** به دلیل مقاومت به سایش خوب و آزاد سازی فلوراید قابل قبول و سهولت استفاده بالینی، رعایت کنترل عفونت، گلاس اینومر کپسولی می تواند ماده مفید در افراد با ریسک پوسیدگی بالا، تکنیک های ترمیم موقت، دندانپزشکی کودکان و پوسیدگی ریشه باشد.

**واژگان کلیدی:** سمان گلاس اینومر، فلوراید، گلاس اینومر

### Introduction

Glass Ionomer Cements (GICs) were introduced to dental practice in the early 1970s and are now in widespread use primarily as direct restorative material owing to their unique property of being able to chemically bond with enamel and dentin and to release fluoride. The GICs are made of powder and liquid components; the powder consists of calcium–aluminum–fluorosilicate glass, and the liquid is 35–65%

polyacrylic acid. [1] Glass ionomer is a good alternative when the use of amalgam and composite becomes undesirable and is recommended for patients with mental or physical disabilities, children with poor cooperation and in atraumatic restorative technique (ART). [2, 3] Today's modern restorative dentistry focuses on hard tissue removal by minimally invasive dental procedures, and on application of restorative materials that may have a therapeutic effect on

demineralized tissues. Chemical adhesion to tooth structures, remineralization, caries prevention effects due to fluoride release, LCTE (Linear Coefficient of Thermal Expansion) similar to tooth structures, biocompatibility and low cellular toxicity have made glass ionomer cements a clinically preferred restorative material. However, despite many advantages, they have lower mechanical and physical properties in comparison with amalgam and composites, such as low fracture strength, toughness and wear resistance, and they are very sensitive to moisture and dehydration.<sup>[4]</sup>

Wear of dental restorative materials is one of the main concerns in long-term clinical services. It is defined as a loss of material mass or change in anatomic contour, which occurs on dental materials and restorations.<sup>[4]</sup> There are different wear mechanisms including abrasion, attrition, adhesion, chemical degradation and fatigue; among them abrasion is the most common mechanism associated with occlusion in oral environment.

Abrasion occurs when two surfaces are in contact with each other by sliding with or without a third component like tooth paste or food particles which make it three or two-body abrasion.<sup>[5]</sup>

Salinovic et al. and Moshaverinia et al. compared mechanical properties like hardness and compressive strength in EQUIA Forte and other glass ionomers as well as showed that there were no significant differences between these materials. Nevertheless, it is noteworthy that wear resistance of EQUIA Forte glass ionomers hasn't been studied yet in great detail.<sup>[1, 6]</sup>

There is no question about the importance of fluoride in the prevention and treatment of dental caries. Fluoride adsorbs to the surface of partially demineralized crystals, inhibits demineralization by forming more acid-resistant crystals (fluoroapatite), and increases the oral environment pH above 5.5 which protects the tooth surface from dissolution. It enhances the deposition of calcium and phosphate into the tooth, rising the remineralization of partially demineralized tissues.<sup>[7]</sup>

Releasing fluoride and forming chemical bond to dental tissue are two main features of glass ionomer which made it the material of choice for different special cases like high-risk patients with multiple cervical caries and ART.<sup>[8]</sup> Jafari et al. evaluated fluoride release and recharge ability of two different GICs (EQUIA Forte and Fuji II) in the presence of various fluoride sources after using CPP- ACFP, and

they found that Fuji II had higher fluoride release levels.<sup>[9]</sup> In another study, Dasgupta et al. compared fluoride release of EQUIA Forte Fil with other glass ionomers (GP IX Extra, Beautifil Bulk, Dyract XP, and Tetric NCeram) and their results indicated that EQUIA Forte Fil had the highest fluoride release.<sup>[10]</sup>

GI restorative systems made of powder and liquid can be prepared by hand-mixing or using sealed capsules. Glass ionomers are usually supplied as a powder and liquid which are dispensed using a scoop and dropper bottle system before hand-mixing. This fabrication process can lead to considerable variations in powder/liquid ratio from what recommended by the manufacturer. Any change in powder/liquid proportion recommended by the manufacturer can compromise physical properties of glass ionomer. Encapsulated glass ionomers decrease operator induced variability and void formation by standardizing the relative proportions of powder and liquid in a sealed capsule.<sup>[11]</sup>

EQUIA Forte was launched as a new material based on glass hybrid technology consisted of EQUIA Forte Fil (a highly viscous conventional GIC) and EQUIA Forte coat (a nanofilled coating material). EQUIA's powder consists of 95% strontium fluoroaluminosilicate glass including the newly added highly reactive small particles and 5% polyacrylic acid.<sup>[12]</sup> The liquid component consists of 40% aqueous polyacrylic acid. EQUIA coat is composed of 50% methyl methacrylate and 0.09% camphor Quinone. This hydrophilic low-viscosity nanofilled surface coating seals the GIC surface and improves aesthetics by increasing luster and translucency.

The aim of this study was to evaluate and compare the amount of fluoride release and wear resistance of EQUIA Forte encapsulated glass ionomer with Fuji II conventional glass ionomer.

## Materials & Methods

This study was approved by the Ethics Committee of Babol University of Medical Sciences, Babol, Iran (with the code of IR.MUBABOL.REC.1397.036). In the current experimental study, the amount of fluoride release and wear resistance of encapsulated EQUIA Forte glass ionomer (GC America, IL, USA) were compared with Fuji II conventional glass ionomer (GC America, IL, USA). General composition of materials used in this study are provided in table 1.

**Table1. Materials used in this study**

Material	Type	Manufacturer	General composition
Fuji II	Conventional glass ionomer	GC America,IL, USA	Powder – fluoroaluminosilicate Liquid –polyacrylic acid
EQUIA Forte	Glass hybrid restorative	GC America,IL, USA	Fluoroaluminosilicate glass, polyacrylic acid powder, surface treated glass
EQUIA Forte Coat	Nanofilled resin	GC America,IL, USA	Methyl methacrylate, colloidal silica, camphor Quinone, urethane, methacrylate, phosphoric ester monomer

**EQUIA Forte group:** The materials were handled according to the manufacturer's instructions. In order to activate the EQUIA Forte capsule (GC America, USA), the plunger was depressed and inserted on the capsule applicator and clicked once to activate. Then, the capsules were mixed in a capsule mixer (4000-4500 rpm) for 10 seconds which were removed and placed in the molds with the help of the GC capsule applicator III, and next the top surface of each specimen was covered with a glass slide to ensure smooth surface of glass ionomer.<sup>[1, 3, 13]</sup> The specimen was allowed to set at room temperature for 2.30 minutes. Then, a thin layer of EQUIA Forte coat (GC America,USA) was applied on the surface of the specimen using a micro brush and light cured using a LED light curing unit (Valo, Ultradent, USA) with intensity of 1000 mw/cm<sup>2</sup> for 20 seconds. EQUIA Forte coat was free of fluoride and contained low-viscosity monomer methyl methacrylate, phosphoric acid ester monomer and photo initiator.

**Fuji II group:** One scoop of powder and two drops of liquid were poured on a glass slab, and the powder was divided into two equal portions. The first portion was mixed with liquid for 15 seconds, and after that, the remaining was added and mixed for 10 seconds. Then, the material was inserted in the mold and covered with a glass slab for two minutes. The specimens were subsequently removed from the molds by applying pressure and the excess extruded material was removed by gentle pressure. All samples were stored independently, in a moist environment at 37°C for 24 h.

**Wear resistance:** Eight<sup>[4]</sup> cubic specimens of each glass ionomer were prepared for wear test in a customized

brass mold (10 mm length × 10 mm width × 2 mm depth).<sup>[3,5]</sup> Through conducting the wear tests, we observed that the prepared samples broke in high wear cycles, so to avoid this, we reasonably redesigned the mold and increased the depth(height) to 3 mm and made 8 specimens of each glass ionomer in this new molds (10 mm length × 10 mm width × 3 mm depth).

The wear test was performed in P.D.B wearing machine (Pajooesh Dandanpezeski Babol, Babol, Iran) that simulates occlusal wear. It is a compression-cycling motion system in which the specimens are moved rotationally and has the ability of three-body wear with diluted (1: 2 with distilled water) toothpaste (Paveh, Paxan Company, Iran).

The cylindrical abrading bar is made up of chromium-cobalt, and the samples of restorative materials were subjected to wear testing against circular, flat-ended chromium-cobalt abrading cylindrical bar with circular contact areas of 1.98 mm<sup>2</sup> at 1-2.5 cycles/sec. Before performing wear test, the diameter of chromium-cobalt bar was measured using a caliper to verify that its dimensions are correct.

Before test, each sample was weighed in analytical scale with an accuracy of up to 0.1 mg (Sartorius ED224s, Sartorius AG, Germany). After specimens were placed in the wear machine, they were abraded in 5000, 10000, 20000,40000, 80000 and 120000 cycles, respectively as well as their weights were measured after each cycle by an analytical scale. The weight loss due to the wear was calculated as the difference between the weight of each specimen before and after each wear cycle.

**Fluoride releasing measurement:** Eight<sup>[13]</sup> cubic specimens of each glass ionomer was prepared in customized brass mold (with the dimension of 10 mm length × 10 mm width × 2 mm depth) for measuring the amount of fluoride release. After preparation, each specimen was immersed in 7 ml of deionized water in polyethylene vials and incubated at constant temperature of 37°C for 24 hours. The first measurements of fluoride concentration was made 24 hours after preparation of the suspension. The samples were removed from the vials, and the concentration of fluoride ions in the deionized water was measured using a fluoride ion-selective electrode (Ion Sensitive Electrode Metrohm AG, Switzerland) in PH/ion meter device (PH/Ion meter metrohm, Switzerland). Before measuring the fluoride content, the deionized water in the plastic container was buffered with 0.5 ml of TISAB

III (Total Ionic Strength Adjustment Buffer) to achieve constant ionic strength (ion concentration) and PH as well as to prevent the generation of fluoride ion complexes. Furthermore, the specimens were rinsed with deionized water, dried with absorbent papers and next, immersed into new containers with 7 ml of deionized water. The same procedure was repeated on days 7 and 15 and expressed in ppm. After measuring fluoride concentration, the electrode was rinsed with deionized water and after being dried, was used for the next sample.

The obtained data were statistically analyzed by SPSS 17 (SPSS Inc.; Chicago, 11, USA) through repeated-measurement ANOVA and independent T-test.

## Results

**Fluoride release:** In the beginning of the study, all specimens were weighted to ensure that there were no voids in their structure.<sup>[12]</sup> Mean weight for EQUIA Forte glass ionomer and for Fuji II glass ionomer was  $0.57 \pm 0.01$ g and  $0.56 \pm 0.02$ g, respectively, and the differences between them were found to be statistically insignificant ( $p=0.57$ ). Repeated Measurement ANOVA analysis showed that the amount of fluoride release significantly decreased with time. ( $F=4258.42$ ,  $df=2$ ,  $p<0.001$ ) The amount of fluoride released from the two glass ionomers during the study period is illustrated in figure 1.

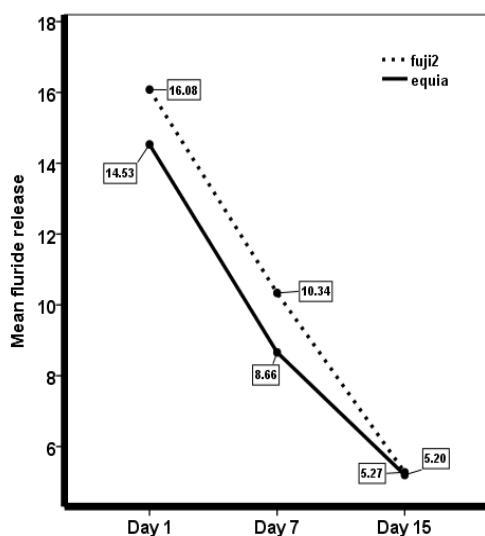


Figure1. Mean fluoride release (ppm) over time for two glass ionomers

In EQUIA Forte glass ionomer, the mean fluoride release was 14.53ppm, 8.65ppm and 5.19ppm on days 1, 7 and 15, respectively. The decrease rate was statistically significant ( $p<0.001$ ). For Fuji II glass ionomer, the mean fluoride release on days 1, 7 and 15 was 16.07ppm, 10.33ppm and 5.27ppm, respectively so that like in EQUIA forte glass ionomer, the decrease rate was statistically significant ( $p<0.001$ ). As shown in fig.1, the EQUIA Forte glass ionomer released considerably less fluoride than Fuji II glass ionomer over the time period of this study ( $F=14.52$ ,  $df=1$ ,  $p<0.001$ ). The differences in fluoride release between two glass ionomers were significant on days 1 (1.54ppm) and 7(1.67ppm) ( $p<0.001$ ). However, this difference almost gradually vanished (0.07) on day 15 ( $p=0.62$ ). For both materials, fluoride release was greatest in the first 24 hour, and after that an incremental decrease was observed.

**Wear resistance:** Repeated-measurement ANOVA demonstrated that there was a significant increase in the mean weight loss of both glass ionomers during the wear cycles. Mean weight loss for EQUIA Forte increases from 2.1mg to 16.5mg and this value enhances from 2.6mg to 16mg for Fuji II. ( $F=3643.86$ ,  $df=5$ ,  $p<0.001$ ) (fig.2). The mean weight loss in EQUIA Forte glass ionomer was significantly lower than Fuji II glass ionomer in all wear cycles ( $F=989.04$ ,  $df=1$ ,  $p<0.001$ ) (Fig.2).

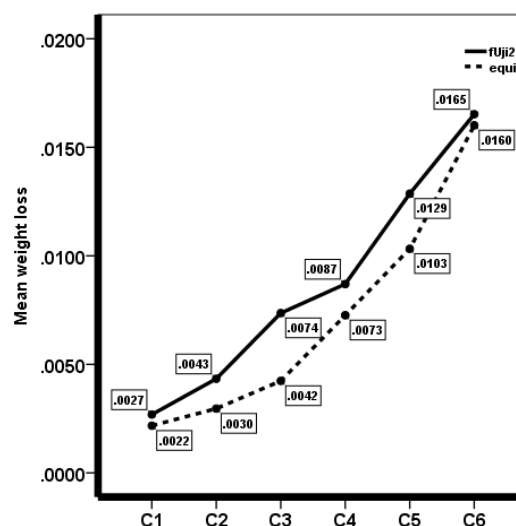


Fig2. Mean weight loss (g) in two groups of glass ionomer in different wear cycles (c1=5000, c2=10000, c3=20000, c4=40000, c5=80000, c6=120000)



## Discussion

Our findings suggested that both glass ionomers had a similar pattern of fluoride release. The highest amount of fluoride release was seen in the first 24h, and then its rate decreased on day 15. The interactions between glass particles and acid in primary setting cause the glass ionomers to release fluoride rapidly, and it is responsible for the initial high amount of fluoride release during first 24 hours. However, in the second stage, balancing glass particles in the glass ionomer structure and diffusing the fluoride ions in samples' matrix results in a decrease of fluoride concentrations, which confirm the previous studies.<sup>[12-15]</sup> The initial high level of fluoride release of glass ionomer restoration will reduce the growth of bacteria, induce remineralization of enamel/dentin and inhibit dental caries.<sup>[16]</sup>

In the current study, all the specimens were incubated at 37°C, and the storage media were selected to be deionized water because it was more accessible and based on previous studies glass ionomers release more fluoride in deionized water compared with artificial saliva. Shafizadeh et al. in 2012 have indicated that fluoride releasing of different glass ionomers in artificial saliva is lower than distilled water.<sup>[12]</sup> Moreover, we used polyethylene vial instead of glass tubes because glass can absorb fluoride ions. Many factors such as PH, temperature, method of mixing and porosities in the structure of the material influence the amount of fluoride release. In this paper, all samples were prepared in the same procedure, and all the tests were conducted in the same conditions so we could assume that the aforementioned factors did not affect the veracity of our results.

Our results denoted that the EQUIA Forte glass ionomer had lower fluoride release in comparison with Fuji II glass ionomer happened because of resin coating used in EQUIA Forte that limited fluoride release. Jafari et al. in 2019 demonstrated that fluoride release in EQUIA Forte was lower than that in Fuji II LC, and they concluded that EQUIA Forte coat could reduce the amount of fluoride release.<sup>[9]</sup>

However, in another study Dasgupta et al. in 2018 represented that EQUIA Forte had highest fluoride release in comparison with composite, glass ionomer and resin –modified glass ionomer. They didn't use the coating layer in their study.<sup>[10]</sup> Brzović-Rajić et al. in 2018 also evaluated the amount fluoride release in EQUIA Forte with and without coating layer, and they

stated that fluoride release of EQUIA Forte reached its highest levels without coating. Although coating layer reduces the amount of fluoride release, it is favorable for preventing caries.<sup>[13]</sup> Previous studies have shown low concentration of fluoride still can enhance the deposition of hydroxyapatite in saliva and prevent caries.<sup>[14]</sup> In the current study, it was found that though EQUIA Forte had released lower fluoride than Fuji II, the difference between these two groups in first day was only 1.54ppm. Our findings demonstrated that EQUIA Forte glass ionomer had lower weight loss in all wear tests compared to Fuji II conventional glass ionomer.

In the present study, in order to reach maximum strength, all samples were incubated for 24 hours before performing the wear tests. It is evident that dehydration can decrease the wear resistance of glass ionomers and that is why immediately after mixing, the glass ionomers are weak and not prepared for wear test until they get to final setting which is estimated to take between one hour to 14 days.<sup>[1, 17, 18]</sup>

Water has an important role in primary setting of glass ionomer. Dehydration and contamination with water in primary setting stage can decrease the physical properties of glass ionomer. In 1990, ADA recommended use of varnish or light-cure resin as coating materials to protect glass ionomers. As a result, the dehydration would be controlled, craze and crack formation decreased and material strength increased. It has been reported that water contamination in setting stage caused surface roughness and decreased wear resistance and physical properties.<sup>[19-21]</sup>

As mentioned before, the EQUIA Forte glass ionomer showed more wear resistance compared to Fuji ii which could be due to coating the surface of glass ionomer with Equia. In addition, Ryu et al. in 2019 showed that EQUIA Forte coat had significantly increased wear resistance of Fuji IX and Fuji II LC.<sup>[20]</sup> However, the EQUIA Forte coat increased wear resistance of EQUIA Forte encapsulated glass ionomer, but it decreased the amount of fluoride release. Previous studies have confirmed that there is a negative relation between fluoride release and physical properties.<sup>[22]</sup> For instance, Xiaoming et al. in 2003 have represented that there is a negative linear relation between compressive strength and fluoride release.<sup>[23]</sup> In the current study, a P.D.B wear machine was used for wear test. The wear tests were performed with a chrome–cobalt bar because it had similar hardness to enamel. Furthermore, in order to simulate the chew cycles in oral cavity for six

months-one year, the wear tests were conducted to 120000 cycles.<sup>[19, 24]</sup>

Nevertheless, within the limitations of the study design (specimen size, storage media, etc.) definitive conclusion cannot be made, and further in vivo investigations are needed to evaluate fluoride release and wear resistance in the oral cavity. The clinical significance of the released fluoride is yet to be fully confirmed. The ultimate goal of correlating fluoride release with actual caries reduction is an objective that can only be met by completing controlled clinical studies on materials.

## Conclusion

The wear resistance and fluoride release of EQUIA Forte encapsulated glass ionomer were not significantly different from those of Fuji II glass ionomer. Therefore, due to good wear resistance and acceptable fluoride release, the encapsulated glass ionomer can be a useful material in people at high risk of caries, temporary restoration techniques, pediatric dentistry and root caries.

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**Conflict of interest:** We declare no conflict of interest

## Authors' Contribution

The study was designed by Effat khodadadi, Zahra Lari and Fariba Ezoji. The study data were collected by Zahra Lari. Analysis and interpretation of data were performed by Hemmat Gholinia. The article was written by Effat khodadadi and Zahra Lari. Study supervision was conducted by Effat Khodadadi..

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