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Fluoride Release from Dental Adhesive Reinforced with Fluorapatite or Calcium Fluoride

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ABSTRACT

Objective: *This study was done to compare the fluoride release from dental adhesive systems (total etch Tetric N-bond and self-etch Tetric N-bond) with the addition of fluorapatite or calcium fluoride in percentages that didn't affect its original shear bond strength for 89 days in vitro.* ***Materials and methods:*** *From each material, 10 cylindrical disk-shaped specimens (diameter: 8.0 mm, height: 1.0 mm) were prepared and immersed individually in 10 ml of deionized water in plastic containers. After 1, 2, 4, 7, 14, 21 and 28 days and after 1 month and 2 months from the last measurement the samples were transferred into new plastic containers that contained 10 ml of deionized water to measure the amount of fluoride in it. The fluoride content was determined with Fluoride Ion Selective Meter HI 96729.* ***Results:*** *The mean fluoride ion release from each material over the tested periods was recorded and the data were statistically analyzed with ANOVA and LSD tests. Self-etch Tetric N-bond with 7% of its weight of calcium fluoride addition showed the greatest fluoride release over the tested periods among the tested material.* ***Conclusion:*** *All the tested materials with the addition of fluorapatite or calcium fluoride showed highest fluoride release during the first two days.*

Keywords: Fluoride release, Dental adhesive, Fluorapatite, Calcium fluoride, Ion-selective meter HI 96729

INTRODUCTION

The dental filling materials with different marginal imperfections are commonly used [1]. Secondary caries in the clinical studies was responsible for 72% and 43% of the overall number of substitutes which are necessary for amalgam and composite restorations respectively. The capability of the restorative material to resist secondary caries attack and microleakage at its margin determines whether the restoration will fail or succeed [2]. Therefore, the addition of an inhibitory effect on the restorative materials against secondary caries is important because of the difficulty of having a perfect seal at the cervical margins [3]. In latest years, resin composite has been prepared to release fluoride. A small amount of fluoride released slowly from composite resin would be beneficial with more than a high concentration of fluoride applications periodically [4]. In some studies, it has been concluded that secondary caries formation inhibition may be influenced by the fluoride containing adhesive systems because the fluoride ions released in the hybrid layer penetrates into the dentin [5].

Dijkman, et al., indicated that 200-300 $\mu g/cm^2$ of fluoride cumulatively released per month is sufficient for the enamel demineralization inhibition [6]. Long and short-term restorative materials which release fluoride is directly related to their matrices, the content of fluoride, the fluoride setting mechanism and the fluoride nature that is incorporated into the resin-based materials, and also dependent on some environmental conditions [7]. Studies have indicated that the caries formation in the dentin can be prevented even if an insufficient amount of fluoride is released from the dental adhesives. The acid resistance of the cavity margins can be increased in the presence of fluoride and therefore play a role in the cavity walls integrity [8,9].

PATIENTS AND METHODS

Debonding preliminary studies were done to choose the best ratio of addition of fluorapatite or calcium fluoride to

Tetric® N-Bond and Tetric® N-Bond self-etch adhesive which was 3% of fluorapatite and 5% of calcium fluoride for Tetric® N-Bond adhesive, while the best ratio addition for Tetric® N-Bond self-etch adhesive were 1% of fluorapatite and 7% of calcium fluoride.

Custom-Made silicone molds with an inner diameter of 8 mm and a depth of 1.0 mm were used to prepare cylindrical disk-shaped specimens (10 specimens of each of the 6 tested materials which were TE, TE+FA 3%, TE+CaF_2 5%, SE, SE+FA 1% and SE+CaF_2 7%). One end of a 10 cm piece of the threat of suture was incorporated into each of the disc specimen space by inserting it through the needle of the silk suture before the material had poured. The adhesive solution was placed in a silicone mold with a glass slide to cover the top surface, then light-cured for 40 sec from the top side using a visible light curing unit (SDI, Japan) [10]. Each specimen was removed from its mold and additionally light-cured for 20 sec from the bottom side.

Each specimen was immersed and stored in individual plastic containers with 10 ml of deionized water at room temperature. At the time of fluoride measurement, each specimen was removed from its container and the storage solution was decanted for analysis. The specimen was returned to a new container with fresh 10 ml deionized water, and storage was continued. At each solution change, the disc was rinsed with fresh deionized water. The measurement of fluoride release was made by using the Fluoride Ion Selective Meter at 1, 2, 4, 7, 14, 21 and 28 days and after 1 month and 2 months from the last measurement.

Fluoride release was measured using Fluoride Ion Selective Meter HI 96729. A mixture of 10 ml of storage solution and 2 ml of HI 93729-0 fluoride reagent was utilized and fluoride concentrations were measured in ppm. The outcomes were calculated as the amount of fluoride released was per unit surface area of samples (µg /cm^2). [11]. The final results detailed were fluoride release rate (µg/cm^2/day) and cumulative fluoride release (µg/ cm^2) which accounts the surface area and solution volume of each sample.

RESULTS

The mean fluoride ion released from each material over the tested periods is represented in Table 1 (Figure 1).

Table 1 Mean fluoride ion released (µg/cm^2) from tested materials for the tested periods

Tested Period	TE	TE+FA 3%	TE+CaF_2 5%	SE	SE+FA 1%	SE+CaF_2 7%
After 1st day	0	11.104	5.336	0	12.408	12.800
After 2nd day	0	12.000	4.720	0	11.416	15.328
After 4th day	0	6.960	3.120	0	6.800	15.152
After 7th day	0	6.480	5.360	0	4.320	15.880
After 14th day	0	7.920	9.200	0	5.200	16.000
After 21st day	0	6.080	7.600	0	1.632	14.944
After 28th day	0	5.520	7.440	0	2.000	15.568
After 58th day	0	4.720	13.120	0	2.640	15.968
After 88th day	0	3.920	12.000	0	2.800	16.000

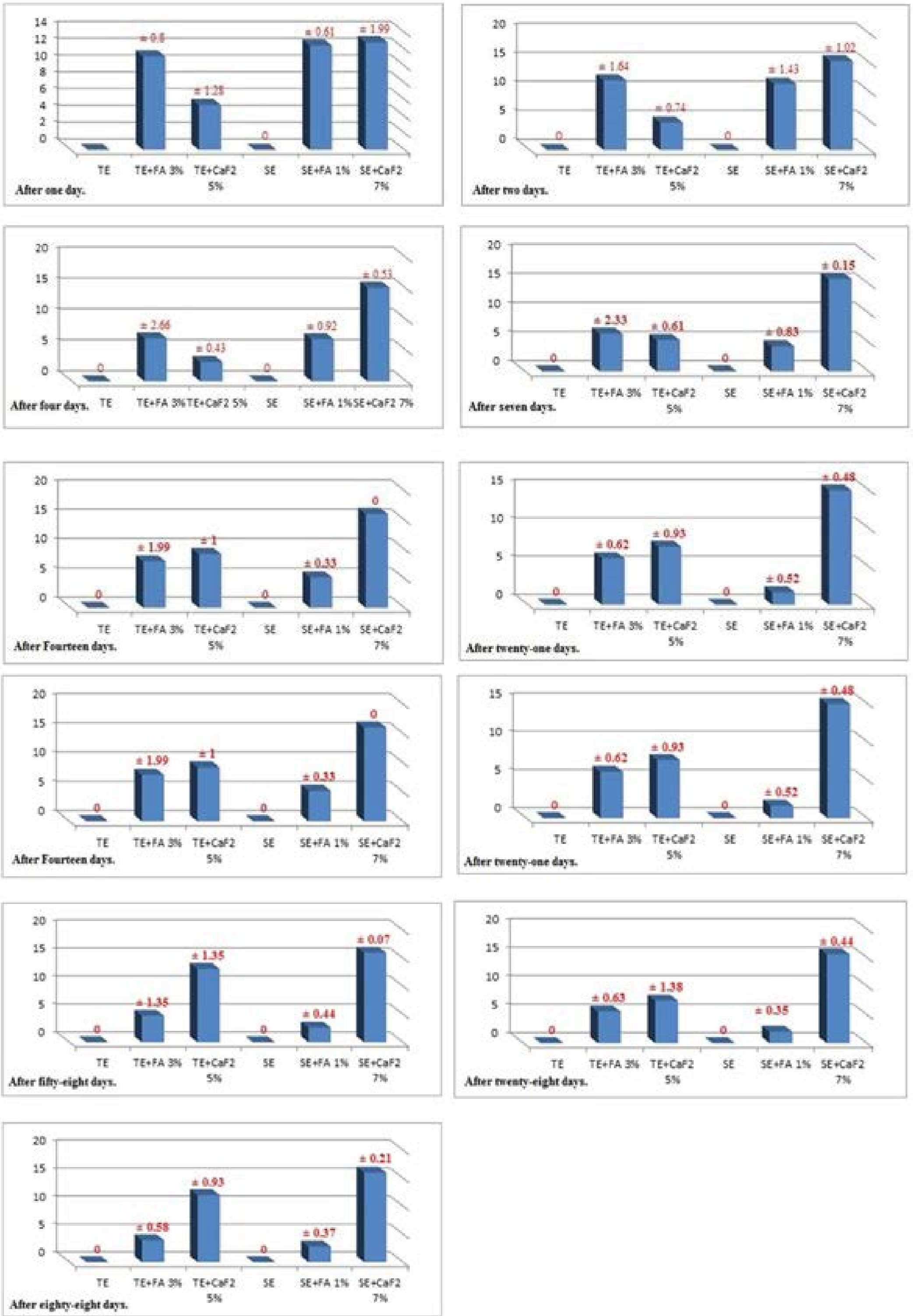

Figure 1 Mean fluoride ion release (µg/cm^2) from tested materials

The mean fluoride ion released from each material over the tested periods for total etch groups shows that the maximum fluoride released from TE+FA 3% were in the first two days, then decreased for the remaining period unless for the 14th day when it increased slightly, while for the TE+CaF_2 5% the peak level of fluoride ion released was in the 58th day as shown in Figure 2 [10].

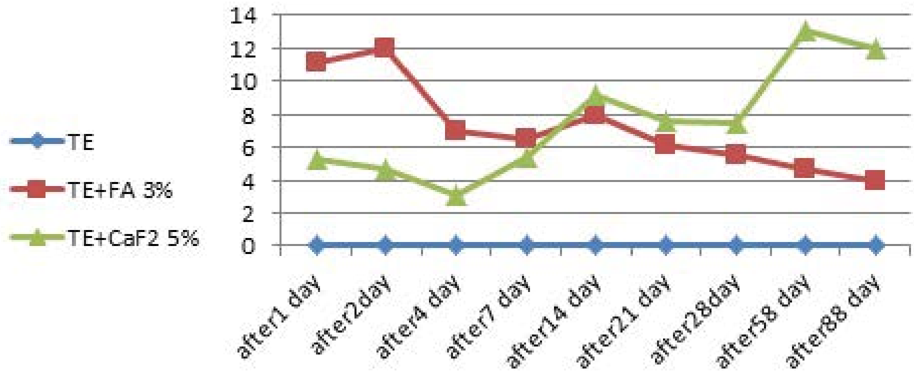

Figure 2 The mean fluoride ion release of the total etch groups over the tested periods in µg/cm²

For the self-etch groups, the mean fluoride ion released from each material over the tested periods shows that the maximum fluoride release from SE+FA 1% were in the first two days then decreased nearly in descending order till the 21st day, the curve became nearly steady for the remaining periods, while the SE+CaF_2 7% presented higher fluoride ion release for all tested periods as shown in Figure 3 [11].

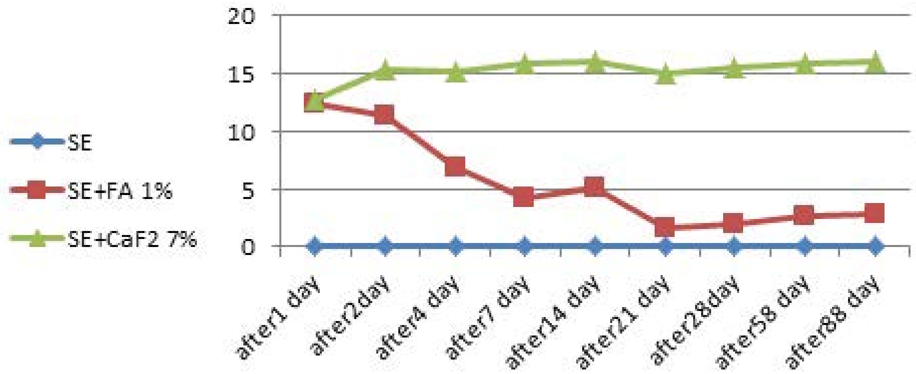

Figure 3 The mean Fluoride ion release of the Self- Etch groups over the tested periods in µg/cm²

Analyzing the data using ANOVA test showed a highly significant difference (p<0.001) in fluoride release between total etch groups for all tested periods as shown in Table 2, and a highly significant difference (p<0.001) in fluoride release between self-etch groups for all tested periods as shown in Table 3.

Table 2 ANOVA test of fluoride ion release means between total etch groups for all tested periods

Tested Period	ANOVA F-test	p-value	Sig
After 1st day	404.300	<0.001	HS
After 2nd day	337.900	<0.001	HS
After 4th day	50.052	<0.001	HS
After 7th day	61.830	<0.001	HS
After 14th day	149.200	<0.001	HS
After 21st day	385.400	<0.001	HS
After 28th day	149.100	<0.001	HS
After 58th day	359.500	<0.001	HS
After 88th day	931.500	<0.001	HS

Table 3 ANOVA test of fluoride ion release means between self-etch groups for all tested periods

Tested Period	ANOVA F-test	p-value	Sig
After 1st day	365.40	<0.001	HS
After 2nd day	612.80	<0.001	HS
After 4th day	149.50	<0.001	HS

After 7th day	281.90	<0.001	HS
After 14th day	175.60	<0.001	HS
After 21st day	398.10	<0.001	HS
After 28th day	651.80	<0.001	HS
After 58th day	107.90	<0.001	HS
After 88th day	115.07	<0.001	HS

The amounts of fluoride release from materials were statistically analyzed using the LSD test for comparison among the materials. For total etch groups at 7th day there was non-significant difference between TE+FA 3% and TE+CaF_2 5% at ($p<0.05$) and at the 14th day there was a significant difference between them at ($p<0.05$), while the comparison between materials shows a highly significant difference at ($p<0.001$) for all the remaining tested periods.

LSD test for self-etch groups shows that there was a highly significant difference at ($p<0.001$) for all the tested periods between materials unless in the first tested day there was a non-significant difference at ($p<0.05$) between SE+FA 1% and SE+CaF_2 7%.

DISCUSSION AND CONCLUSION

One of the fluoride measurement methods is the colorimetric method in which the sample that contains the fluoride reacts with the reagent which is red in color and the interaction between fluoride and reagent leads to decrease the red color of the reagent and as the fluoride concentration increases the reagent color will be clearer which is then detected by Silicon photodetector. The device has a locking system that helps to insert the cuvette in the same position every time used for measurement. Also, it has an indicator for the cooling lamp to allow the component of the sample to be cooled before each measurement for highest accuracy reading.

The shortcoming of this device is that it can determine the concentration of the fluoride between 0.0-2 mg/L (PPM) and the amount of fluoride above 2 mg/L cannot determine, it just gives an indication that it has exceeded 2 mg/L.

The fluoride releasing composite resin materials differs from GIC or RMGI in which it is placed over a bonding resin which leads to restricting the possibility of fluoride-releasing, and this leads to thinking about adhesive systems with fluoride releasing, and in this study reinforcement of dentin adhesive system with fluorapatite or calcium fluoride helps in releasing of the fluoride.

All the fluoridated adhesives demonstrated a continuous release of fluoride into the deionized water for the tested periods (88 days). According to the following equation, the fluoride release depends on the exposed surface area and not on the weight [12,13]:

mgF/cm^2=ppm (μgF/mL) mL (storage media volume at unit time) 1/surface area of each tested sample material which was 1.25 cm^2.

Surface area of cylinder=$2\pi r (r+h)$ [13].

In the first two days, all fluoridated adhesive showed the highest amount of fluoride released and this is in agreement with Atsushi, et al., and Han, et al., as the initial burst of the fluoride releases from the surface [14,15].

On the 4th day, all the fluoridated groups decrease in fluoride release unless the SE+CaF_2 7% and this may be due to the higher percentage of fluoride content which leads to the high availability of fluoride to be transmitted to the surface of the specimens. The fluoride released is decreased after two days for the other groups due to diffusion of a small amount of fluoride to the surrounding medium and this is in agreement with Itota, et al., Wiegand, et al., and Moreau, et al., [7,6,17].

The fluoridated adhesive with fluorapatite continue to decrease in the fluoride release while the TE+CaF_2 5% increases in the mid time and this may be due to high fluoride contents and also fluoride from CaF_2 is easily liberated in the presence of water while the SE+CaF_2 7% liberates the highest amount of fluoride for the tested period.

One of the important reasons for replacement of restoration is secondary caries which is usually associated with the microleakage between the tooth-restoration interfaces. The presence of fluoride helps to inhibit demineralization and even helps in remineralization by binding with dissolved calcium and phosphate and formation of fluorohydroxyapatite.

Dijkman, et al., stated that 200-300 µg/cm^2 accumulative fluoride released per month is enough to inhibit enamel demineralization completely. In the present study, fluoridated adhesive with CaF_2 filler reinforcement release accumulative fluoride above 200 µg/cm^2 per month, while fluoridated adhesives reinforced with fluorapatite filler release fluoride less than 200 µg/cm^2 per month except for TE+FA 3% which release more than 200 µg/cm^2 in the first month [6].

Even a small amount of fluoride is released continuously surrounding the tooth will decrease demineralization of the tissue of the tooth [16], and this means that all fluoridated adhesives in this study will help in preventing demineralization and enhance the remineralization because they continued to release fluoride for all the tested periods, and this free fluoride ions is very important to increase the resistance of the tooth structure because it will be used in the transformation of hydroxyapatite to fluorapatite in addition to its reaction with the dissolved calcium and phosphate during acidogenic attack.

Itota, et al., Wiegand, et al., and Moreau, et al., stated that the fluoride released from different fluoridated restorative materials enhanced the severe acidic condition below pH 4.0, this means that in this study if acidic medium was used instead of deionized water more fluoride may be released, also this indicates that the fluoride will be released more in bacterial acidogenic attack and this will help to prevent demineralization by binding the released fluoride with the dissolved calcium and phosphate and transformation of hydroxyapatite to fluorapatite [7,16,17].

In this study, all the fluoridated adhesives continues to release fluoride, this is important because mostly there are residual bacteria left during cavity preparation and also the continuous release of fluoride during acidogenic attack is able to absorb the apatite crystals which leads to inhibit demineralization and convert hydroxyapatite to fluorapatite and rendering the enamel which is more resistance to future acidic challenges, or fluoride precipitated onto tooth surface as CaF_2, and this will serve as a fluoride reservoir when the PH drops.

The initial high fluoride release from FA reinforced adhesive may be due to the initial superficial rinsing effect and in the subsequent day due to the ability of fluoride to diffuse to the surface, while for CaF_2 the release of high amount of fluoride relatively to FA adhesive groups may be due to the dissolution of inorganic fluoride by the high-water sorption of the matrix region in addition to the high percentage of the fillers.

Statistically, for total-etch adhesive groups on the 7th day, there were non-significant differences in fluoride release between TE+FA 3% and TE+CaF_2 5% in which there was a comparable amount of fluoride released in this period.

For the self-etch groups, only on the first day, there was the non-significant difference in fluoride release between SE+FA 1% and SE+CaF_2 7% in which there was a high amount of fluoride release. For all other periods, there was the highly significant difference between CaF_2 groups and FA groups.

DECLARATIONS

Conflict of Interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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