## **Future Dental Journal**

Volume 6 | Issue 1

Article 2

2020

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### **Recommended Citation**

Abdelhafeez, Bassma Reda; Niazy, Maha Ahmed; Naguib, essam Abdel Hafez; and Younis, Sara Hany (2020) "Fluoride Release of Conventional Glass Ionomer Modified with Flax Fibers and Ethanolic Extract of Propolis," *Future Dental Journal*: Vol. 6 : Iss. 1 , PP -. Available at: https://digitalcommons.aaru.edu.jo/fdj/vol6/iss1/2

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### **Cover Page Footnote**

I would like to express all my sincere gratitude and grateful appreciation To my mentor Dr. Essam Abd El Hafez Naguib, Professor of Operative dentistry, Faculty of Dental Medicine, Cairo University, for his continuous advice, super vision and valuable comments. also, I would like to express all my heart full thanks and sincere gratitude to Dr. Maha Ahmed Niazy, Professor of Operative Dentistry, Faculty of Dental Medicine, Al-Azhar University-Girls Branch, for her priceless and invaluable help. last bit not least, I would like to express my deep gratitude and thanks to Dr.Sara Mohamed Hany Aly, lecturer of conservative dentistry, Faculty of oral and dental medicine, Future University for her time, invaluable help, support and continuous cooperation throughout this study.

# Fluoride Release of Conventional Glass Ionomer Modified with Flax Fibers and Ethanolic Extract of Propolis

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### Abstract

Purpose: This study aimed to evaluate the fluoride release of conventional glass ionomer (GI) modified with flax fibers and/or ethanolic extract of propolis (EEP) with and without thermo-cycling. Materials and Methods: 80 total discs of GI were prepared, 20 discs per group: Group (GI) conventional glass ionomer (control), Group (F) glass ionomer incorporated with 5% flax fibers, Group (P) glass ionomer incorporated with 1% ethanolic extract of propolis (EEP), Group (FP) glass ionomer incorporated with 5% flax fibers and 1% ethanolic extract of propolis (EEP). Fluoride release testing was performed after the first day and after 7 days. Results: Conventional Fuji IX Gold Label (GI) revealed the lowest fluoride release mean values, Propolis modified Fuji IX Gold Label (P) showed higher mean values followed by Flax fibers and Propolis modified Fuji IX Gold Label (FP), while Flax fibers modified Fuji IX Gold Label (F) revealed the highest mean fluoride release value. Conclusion: Incorporation of 5% Flax fibers enhanced the initial fluoride release of conventional glass ionomer.

Keywords: Flax Fibers, Fluoride release, Glass Ionomer, Propolis

### 1. Introduction:

Glass ionomer material is greatly used in restorative dentistry because of its advantages over other restorative materials. Its unique properties like chemical adhesion, biocompatibility and remineralization of dental tissues were reported. The anticariogenic activity that is due to its fluoride release and its adhesive property make conventional glass ionomers greatly used with significant clinical results as filling, base and liner materials. Glass ionomer also have coefficient of thermal expansion similar to dental tissues [1].

To eliminate any concern about the biocompatibility issue of materials used in the oral cavity, the interest for natural products added to dental materials has increased [2]. As an example of biologically produced products, Flax fibers were commonly used in the biomedical field because of their antimicrobial action [3]. Flax fibers are lingo-cellulosic, natural multicellular fibers that has good physical, mechanical and chemical properties [4, 5].

Propolis is a resin exudate of plant that is collected by bees. Bees added wax and their secretions later to produce a complex product that is called Propolis. Propolis characterized by a lot of compounds depending on its type, but flavonoids, amino acids, different aromatic and phenolic compounds, minerals and vitamins are the essential chemical components of propolis. In dental field, Propolis has been used because of its antimicrobial effect. Also, it is used for dental caries prevention and treatment [6].

Fluoride release is the main advantage of glass ionomer. The antimicrobial and anticariogenic properties of glass ionomer are considered to be related to the fluoride release which is effective in caries prevention [7].

Therefore, the present work aimed to evaluate the fluoride release of conventional glass ionomer incorporated with flax fibers and/or ethanolic extract of propolis.

## 2. Materials and Methods:

### 2.1. Preparation of specimens:

80 discs of glass ionomer were prepared for fluoride release testing. Specimens were divided into four groups, each group of 20 specimens. In the first group, conventional glass ionomer (GC Fuji IX Gold Label) was used (GI), in the second group Flax fiber modified Fuji IX Gold Label was used (F), in the third group Propolis modified Fuji IX Gold Label was used (P), while in the fourth group Fuji IX Gold Label was modified with both Flax fibers and Propolis (FP). Each group was further subdivided into two subgroups (D), each subgroup of 10 discs, according to the assessment time, (D0) after one day, or after seven days (D1).

## 2.2. Preparation of Flax fibers:

Short flax fibers were cut using sharp ended scissors and measured by digital caliper, Fibers which were more than 1 mm in length were excluded. To confirm the fibers length, fibers were scanned using Scanning electron microscope (Type XL30; Philips Electron Optics, Eindhoven, SEM Unit, Main Defense Chemical Laboratory). Flax fibers were collected and accurately weighed using a sensitive digital balance to obtain a 5 wt.%, then the Flax powder was added and properly mixed with the powder bottle of glass ionomer.

# 2.3. Preparation of 1% Ethanolic Extract of Propolis:

20% Propolis has been dissolved in 60% ethanol. Before being dried, the sticky product

was filtered. To gain the 1% EEP concentration, 0.08ml of EEP was measured using measuring pipette and added to the liquid bottle of glass ionomer.

## 2.4. For fluoride release testing:

20 discs of each glass ionomer material were prepared using a split teflon mold of 3mm thickness and 4mm inner diameter. The mold base was formed by fixing a mylar strip on a glass plate. Each group material was mixed according to the manufacturer's instructions and packed in the mold, then covered using another mylar strip, on which a second glass plate was placed. A weight of 500 g was then put for one minute on the top. After that, excess material was removed (55). Each disc was removed from the teflon molds carefully and put in firmly closed plastic container that contain 5ml of de-ionized water and maintained at 37°C in a laboratory incubator till the assessment time. De-ionized water was renewed every 24 hours for one week. 1 ml of the de-ionized water from each container was taken after 1:1 dilution with Total Ionic Strength Adjustment Buffer (TISAB) to evaluate the fluoride release. Fluoride release was measured twice, after the  $1^{st}$  day and after 7 days using Ion selective electrode.

## 2.5. Statistical analysis:

Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Fluoride release data showed normal (parametric) distribution. Data were presented as mean and standard deviation (SD) values. Repeated measures ANOVA test was used to study the effect of material type, time and the interaction of the two variables on amount of Fluoride release. Bonferroni's post-hoc test was used for pair-wise comparisons. The significance level was set at  $P \leq 0.05$ . Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 22.0. Armonk, NY: IBM Corp.

### 3. Results:

### 3.1. Effect of material type regardless of time:

Pair-wise comparisons between the materials showed that (F) material showed the statistically significantly highest mean fluoride release. FP showed statistically significantly lower mean value. There was no statistically significant difference between (GI) and (P) groups; both showed the statistically significantly lowest mean Fluoride release values.

Table 1: 1: The mean, standard deviation (SD) values and results of repeated measures ANOVA test for comparison between Fluoride release of different materials regardless of time

GI	F	FP	-	P-	11000
Mean SI	) Mean SI	) Mean SD M	fean SD	value	size
16.5C14	.323.6A20	.518.5B16.2	16.8C15	o<0.001*	0.865

\*: Significant at P  $\leq$  0.05, Different superscripts are statistically significantly different

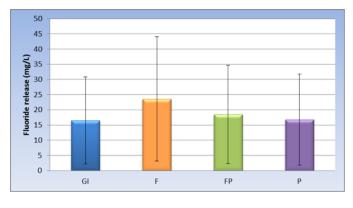


Figure 1:

Chart-1: Bar chart representing mean and standard deviation values for fluoride release of different materials regardless of time.

### 3.2. Effect of time regardless of material type:

There was a statistically significant decrease in mean values of fluoride release after 7 days.

\*: Significant at  $P \le 0.05$ 

Chart-2: Bar chart representing mean and standard deviation values for Fluoride release at different times regardless of material type.

Table 2: 2: The mean, standard deviation (SD) values and results of repeated measures ANOVA test for comparison between Fluoride release at different times regardless of material type.

$1  \mathrm{day}$		7 days		P-	Effect
Mean	SD	Mean	SD	value	size
34.9	5.8	2.8	0.6	< 0.001*	0.995

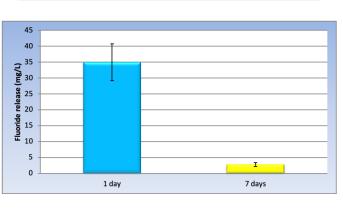


Figure 2:

# 3.3. Effect of different interactions on Fluoride release

Pair-wise comparisons between the materials revealed that Flax fibers modified Fuji IX Gold Label (F) material showed the highest statistically significant mean values of fluoride release after the  $1^{st}$  day and after 7 days followed by Flax fibers and Propolis modified Fuji IX Gold Label (FP) group. While there was no statistically significant difference between conventional GI (GI) and Propolis modified Fuji IX Gold Label (P); both showed the lowest statistically significant mean Fluoride release values after the first day and after 7 days.

Table 3: 3: The mean, standard deviation (SD) values and results of repeated measures ANOVA test for comparison between Fluoride release values of the four materials at each time.

				FP Mean SD		Effect size
1day	<sup>7</sup> 30.3C 3	43.6A 1	.1 31.3C 3	.4 34.3B 0.7	< 0.001*	0.848
7 days	, 2.6B 0.5	3.6 A 0.	4 2.4B 0.	3 2.8B 0.5	< 0.001*	0.566

\*: Significant at P  $\leq$  0.05, Different superscripts in the same row are statistically significantly different.

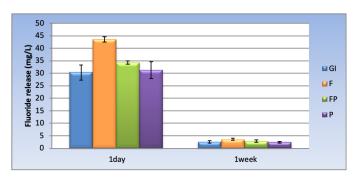


Figure 3:

Chart-3: Bar chart representing mean and standard deviation values for Fluoride release of the four materials at each time.

### 4. Discussion:

This work was done to evaluate the effect of Flax fibers and /or

Propolis incorporation on the fluoride release of glass ionomer restorative material. Fuji IX gold label glass ionomer was used in this study because it is a high strength strontium-based and fluoride releasing material [8].

Flax fiber is considered one of the most used bio-fibers recently because of its significant mechanical properties. Modified flax fibers had been used for infections treatment as dressings for wounds due to their antibacterial effect [9, 10].

Propolis is a natural non-poisonous resinous material that has antimicrobial action [11]. Propolis cannot be used directly because of its complex structure. So, solvents were used to remove the inactive material and maintain the desired ones [12]. The common forms of Propolis are lyophilized and ethanolic extract. Lyophilized Propolis led to difficult manipulation of glass ionomer while ethanolic extract of Propolis which is the richest source of phenolic acids, flavonoids and proanthocyanidins (PA) verified the opposite [13]. It was stated that 1% EEP enhanced the fluoride release of glass ionomer [14]. So it was used in this study. It was proved that the initiation and progression of recurrent caries is significantly decreased when GI restorative materials were used, because of the advantage of fluoride release. Moreover, the extent of antimicrobial and caries prevention properties of GI restorative materials are related to the amount of fluoride release [15]. That is why it was crucial to measure the amount of fluoride release following glass ionomer modification with different natural agents.

Flax fibers modified Fuji IX Gold Label specimens showed the highest fluoride release values after one and 7 days. This was because Flax fibers contain hydroxyl group and so the moisture absorption is high (rapid material dissolution) and this led to the "cleaning effect" caused by water on the surface of the material with more fluoride release [16]. Moreover, the reason may be due to the presence of Flax fibers within glass ionomer matrix system which was expected to be the weakest area of the formulation (Flax fiber-matrix interface) that may have created a pathway for easier water diffusion into the material and more release of fluoride ions [17].

Again, a synergistic effect on fluoride release occurred when GI was modified with both Flax fibers and Propolis after one and 7 days. Flax fibers created pathways for fluoride release but Propolis filled those pathways.

There was no significant difference between conventional GI and Propolis modified Fuji IX Gold Label specimens. Since the fluoride release from GI depends on various intrinsic factors such as its formulation, solubility and porosity [18]. Due to the intermolecular interaction that occurred in the Propolis modified Fuji IX Gold Label specimens, it behaved similar to the conventional GI regarding all these intrinsic factors.

All the groups showed a significant release of fluoride after the first day evaluation that significantly decreased after 7 days. The diffusion and concentration in the particles and the matrix had a great effect on the amount of fluoride release of glass ionomer. Fluoride ions become part of the reaction following the acid base reaction of GI. After that the glass particles start to dissolute slowly by time through the restoration pores (bulk fluoride release followed by a maturation period) [1].

### 5. Conclusions:

This study showed that:

1-Incorporation of 5% Flax fibers enhanced the initial fluoride release of conventional glass ionomer.

2-Glass ionomer modified with natural biological products is a promising restorative material to be used in the dental field.

### 6. Recommendations:

Further investigations of fluoride release over a longer period of time should be evaluated.

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