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# Fluoride ion release from ultraviolet light-cured sealants containing sodium fluoride

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### Abstract

The ability of uv light-cured fissure sealants to release fluoride ion from different concentrations of sodium fluoride (NaF), over a 3-week period was studied. Two hundred samples of sealant were prepared in which 1 group contained no fluoride (control) and 4 others contained NaF at 0.05, 0.2, 0.5, and 2% concentrations, respectively. The fluoride ion released by each sample after 1 day, 3 days, 1 week, and 3 weeks was determined by a specific fluoride ion electrode. The study demonstrated that the quantity of fluoride ions released increases, in general, with time and concentration.

L he vulnerability of occlusal pits and fissures to dental decay<sup>1-3</sup> is a problem which has prompted the development of pit and fissure sealants.<sup>4-7</sup> Since topical applications of sodium fluoride (NaF) solutions and gels have been shown to be effective in reducing dental caries,<sup>8-11</sup> the introduction of NaF to a fissure sealant could enhance the reduction of pit and fissure decay. The concept of increasing the caries-reducing ability of pit and fissure sealants was investigated in studies which initially added fluoride to varnish.<sup>12-14</sup> Swartz et al.<sup>15</sup> conducted an in vivo study to test the feasibility of imparting an anti-cariogenic property to pit and fissure sealants by adding 2-5% NaF. These data indicated that fluoride added to a sealant resin material in the amount reported substantially increased the fluoride content of the enamel, reduced its solubility in acid, and provided an acceptable anticariogenic mechanism.

Since it has been found that the pretreatment of enamel with fluoride and the addition of fluoride to acid-etching solutions reduces bond strength,<sup>16-20</sup> the addition of fluoride to fissure sealants would appear to be worthy of study in the search for a better cariesreducing procedure.

The objectives of this study were to measure the ability of fissure sealant<sup>a</sup> to release fluoride ion from different concentrations of sodium fluoride<sup>b</sup> added to the sealant throughout a 3-week period as well as to determine the amount of fluoride ion released relative to the concentration of NaF at each designated time of analysis.

# **Methods and Materials**

The fissure sealant was combined with 4 different concentrations of NaF (0.05, 0.2, 0.5, 2.0%) by means of a mechanical mixer<sup>c</sup> to determine the ability of the sealant to release fluoride ion. These NaF concentrations were selected because of their use and effectiveness in mouth rinses (0.05, 0.2, 0.5%),<sup>10</sup> in dentifrices (0.2%)<sup>9</sup> and in topical application (2.0%).<sup>8</sup> Samples of the same sealant material without the addition of NaF served as controls. Standard sizes of the sealant samples were produced by using plastic molds<sup>d</sup> containing 10 compartments supplied for use with the sealant. New molds were used for the preparation of each group of sealant/NaF combination.

In order to find the best method of curing the sealant and sealant/fluoride mixture, 2 different uv light sources<sup>e,f</sup> were tried. No difference was found between 2 uv light sources. Because of the large sample size used in this study (N = 200) one uv light source

- <sup>a</sup> Nuva Seal LD Caulk Co: Milford, DE.
- <sup>b</sup> Sodium Fluoride (powder, Respond A209) Ward's Natural Science Establishment, Inc: Rochester, NY.
- <sup>c</sup> Vortex-Genie Scientific Industries: Springfield, DE.
- <sup>d</sup> Delton Sealant Johnson & Johnson: East Windsor, NJ.
- <sup>e</sup> Nuva Lite LD Caulk Co: Milford, DE.
- <sup>f</sup> Black Ray-Longwave Ultraviolet (Model X4) Ultraviolet Products, Inc: San Gabriel, Ca.

was used to cure the sealant and sealant/fluoride mixtures.<sup>f</sup>

#### Sample Preparation

The experimental groups consisted of the fissure sealant with: 0.05% NaF (40 samples); 0.2% NaF (40 samples); 0.5% NaF (40 samples); and 2.0% NaF (40 samples). The control group consisted of the sealant without fluoride (40 samples).

Prior to the experiment, the total amount of sealant required for each group was determined by adding enough drops of sealant from a volumetric pipette to fill 1 compartment of a plastic mold, thus forming 1 sample. Multiplication of this amount by the total number of samples required indicated that 35 cc of sealant was needed to make 40 samples.

A dry, clear, premeasured dispenser<sup>g</sup> was used to dispense the required amount of sealant to the respective concentrations of NaF. The sealant/NaF mixture was agitated vigorously on the mechanical mixer to assure homogeneity and then transferred immediately to the plastic mold using the drop method. The sealant was cured in layers by the uv light for 60 sec. Hardness was checked with a dental explorer to verify complete polymerization prior to adding a new layer of sealant/fluoride. To assure uniformity in the size of the samples being produced, the same number of drops were placed in each plastic mold, which then was coded (the samples were numbered).

Each group of 40 samples then was divided randomly, using a table of random numbers into subgroups of 10 samples for evaluation of fluoride ion release during each of the experimental time periods (1 day, 3 days, 1 week, 3 weeks).

#### Sample Treatment

Following preparation, each sample was placed into a disposable microbeaker<sup>h</sup> containing 5 ml of deionized distilled water. Each beaker was covered,<sup>i</sup> secured well and stored at room temperature for the experimental time periods before being analyzed for the release of fluoride ion into the bathing solution. A small pilot study in which the volume of water in the beaker was measured did not reveal any loss of liquid by evaporation over a period equal to the experimental time. A total of 40 samples were employed for each of the 4 fluoride concentrations and the control group.

#### Sample Analysis

An ion-specific electrode<sup>j</sup> was used to determine the amount of fluoride ion released from each sample. At the designated time for analysis, each sample was removed from a beaker and 5 ml of buffer<sup>i</sup> was added to the beaker contents. With the meter adjusted to room temperature and the meter electrode rinsed with distilled, deionized water, the electrode was immersed into the solution until a steady response was noted. A stirrer bar<sup>k</sup> in the beaker, which was positioned on a magnetic stirrer, provided constant agitation. Readings in mV were recorded for each prepared sample. These readings then were used to determine the amount of fluoride ion released by each sample by protracting the readings on a standard calibration curve. A standard curve was constructed by plotting mV readings of solutions containing known amounts of fluoride ion (according to the manufacturer's specifications) on semilog graph paper to make a straight-line graph. Daily adjustments were made to the standard graph by recording mV readings of a solution containing a known amount of fluoride ion (1 ppm).

#### **Statistical Analysis**

A 4  $\times$  4, analysis of variance (ANOVA) was used. The dependent variable was the amount of fluoride ion released. The independent variables were: (1) the concentrations of NaF used, and (2) the time during which the fluoride ion was released.

#### Results

Measurable fluoride ions were released by all of the experimental groups at every analytical period. As expected, the control group samples released no fluoride ion throughout the study and therefore were not included in the statistical analysis.

The mean fluoride ion released from the 4 experimental groups over the 4 time periods is shown in Table 1. For the 0.05% NaF/sealant mixture, the mean fluoride ion released ranged from 2.2  $\pm$  1.0 to 5.3  $\pm$  3.9 ppm after 1 day and 3 weeks, respectively. The range of mean fluoride ion released by the three mixtures were: 0.2% NaF/sealant — 17.6  $\pm$  6.1 (1 day) to 22.2  $\pm$  11.8 ppm (3 weeks); 0.5% NaF/sealant — 51.6  $\pm$  36.0 (1 day) to 72.8  $\pm$  36.0 (3 weeks); and 2.0% NaF/sealant — 168.1  $\pm$  58.1 (1 day) to 369.0  $\pm$  177.9 (3 weeks).

A 4  $\times$  4 ANOVA was performed to determine the statistical significance of the results of this study. The ANOVA summary for the concentration of NaF added to the sealant, time during which fluoride ion was

<sup>&</sup>lt;sup>f</sup> Black Ray-Longwave Ultraviolet (Model X4) — Ultraviolet Products, Inc: San Gabriel, Ca.

<sup>&</sup>lt;sup>8</sup> Lange's jet acrylic — Lange Dental Mfg Co: Chicago, IL.

<sup>&</sup>lt;sup>h</sup> Disposable Microbeakers — Fisher Scientific Co: Cincinnati, OH.

<sup>&</sup>lt;sup>i</sup> Parafilm "M" — Laboratory Div, American Can Co: Greenwich, CT.

<sup>&</sup>lt;sup>j</sup> Total Ionic Strength Adjustment Buffer — Orion Research, Inc: Cambridge, MA.

<sup>&</sup>lt;sup>k</sup> Magnetic Stirrer — Fisher Scientific Co: Cincinnati, OH.

Time of	Experimental Time of Fluoride Conce			
Analysis	0.05%	0.2%	0.5%	2.0%
One Day	$2.2^* \pm 1.0^+$	17.6 ± 6.1	$51.6 \pm 36.0$	168.1 ± 58.1
Three days	$3.3 \pm 0.9$	$16.2 \pm 9.6$	$50.8 \pm 22.6$	$194.2 \pm 51.0$
One week	$2.4 \pm 1.0$	17.5 ± 11.7	$58.6 \pm 21.2$	$254.0 \pm 132.8$
Three weeks	$5.3 \pm 3.9$	$22.2 \pm 11.8$	$72.8 \pm 36.0$	$369.0 \pm 177.9$

TABLE 1. Mean and Standard Deviation of Fluoride Ion Released

\* Mean fluoride ion released. † Standard deviation.

released, and their interaction are presented in Table 2. The main effects for both concentration of NaF and time were statistically significant (p<0.05). The statistical significance of the above-mentioned variables should, however, be interpreted in light of their significant interaction (p<0.05). Tests for simple main effects to ascertain the underlying interaction revealed the following:

- 1. Comparisons among the concentrations of NaF at each time point indicated that, irrespective of time, the amount of fluoride released increased as the concentration of NaF increased (Table 1, Fig 1).
- 2. Comparison for the effect of time at each concentration of NaF revealed that only at the 2.0% concentration was there any significant effect of

TABLE 2. Analysis of Variance Summary

Source of Variation	Sum of Squares	Degrees of Freedom	Mean/ Square	Р
Time	79805.5	3	26601.8	7.2*
Concentration	1515666	3	505222	136.4*
Interaction	164037.1	9	18226.3	4.9*
Error	533406.9	144	3704.2	

\* P<0.05.



FIG. 1. Relation between fluoride ion released (in ppm) and times of analyses (1 and 3 days, 1 and 3 weeks).

time, particularly at the 3-week interval. The increase in fluoride ion released across time for both the 0.05 and 0.2% concentrations is negligible at 1 day through 3 weeks. While the fluoride ion released after 3 weeks for the 0.5% concentration is greater than that of the other 3 time points, it is not of a sufficient magnitude to affect statistical significance.

Table 3 indicates the calculated percentage of fluoride ion released from the experimental sample using an estimated average of 0.8 ml volume. The maximum fluoride ion released occurred with 2.0% NaF concentration after 3 weeks when nearly 25% of available fluoride ion was released.

# Discussion

The addition of NaF to the sealant produced a fluoride ion release from the experimental groups that increased generally with all levels of fluoride concentration and time, but specifically with the 2.0% group at 3 weeks. Examination of Figure 1 indicates that low concentrations of NaF produced less fluoride ion release when compared to that obtained from higher concentrations (0.5 and 2.0%). Throughout the study the low concentration groups (0.05 and 0.2%) released most of their fluoride ion during the first day. Based on the mean fluoride ion released by the 2.0% NaF group after 1 week (254.0  $\pm$  132.8 ppm fluoride) and 3 weeks (369.0  $\pm$  177.9 ppm fluoride), it can be anticipated that the fluoride ion released might be increased if the study were extended for a longer time period. This study would suggest, in addition, that low concentrations of NaF should not be used if a high fluoride ion release is desired from the fluoridecontaining sealant. The fluoride ion released should

TABLE 3. Percentage of Fluoride Released by Experimental Group

Time of	Experimental Group Fluoride Concentrations			
Analysis	0.05%	0.2%	0.5%	2.0%
One day	6.3	12.2	14.3	11.7
Three days	9.2	11.3	14.1	13.5
One week	7.0	12.2	16.3	17.6
Three weeks	9.3	15.4	20.2	25.6

be balanced with fluoride available from other sources within the local community.

The data also suggest that only a small portion of the available fluoride is released from the samples tested (Table 3). No definite conclusions can be reached as to the total amount of fluoride ion that would be released if the study had been conducted beyond 3 weeks. Based on the trends shown with the higher concentrations of fluoride-containing samples of the sealants, there is some indication that, over a prolonged period, more fluoride would be released. The data also suggest that a uniform release would be attained based on the indicated leveling off at lower concentrations.

That fluoride ion is released in this study is apparent; the mechanism of the release remains speculative. Release could occur from the insoluble Bis-GMA resin as a result of resin porocity or because the fluoride ion is not tightly bound to the resin molecules. In addition, release may be due to fluoride deposition on the surface of the resin in spite of constant mixing efforts. Additional studies to resolve these speculations are indicated.

Fluoride ion could be released in this in vitro system from the unpolymerized air-inhibited layer on the surface of the test samples. The size and surface areas of the sealant undoubtedly would influence the quantity of release. Unpolymerized resin probably would not be of benefit to the enamel since it contacts the enamel only minimally and also would be worn away almost immediately after sealant placement.

Results of this study are similar to patterns of release obtained with acid-etching solutions to which NaF was added<sup>18,20,21</sup> and with varnishes containing NaF.<sup>12-14</sup>

Maintaining uniform mixtures of fluoride within the sealant vehicle to ensure a uniform release of the fluoride ion was an important variable involving 2 factors: the amount of NaF powder added to each 35 cc sealant specimen and the viscosity of the sealant itself. Although mechanical mixing was used and the experimental samples were divided randomly, the problem of maintaining a uniform dispersion of fluoride salt in the sealant remained. Fluoride ion release from the sealant specimen containing a 2.0% NaF concentration illustrates the need for attention to this factor (Table 4). Fluoride ion release ranged from 11.8 to 600.0 ppm fluoride for the 3-week samples of the fourth experimental group (sealant + 2.0% NaF).

The results of this study, which show a significant (p<0.05) fluoride ion release when NaF is added to the fissure sealant, could imply the feasibility of an interesting alternative in the prevention of pit and fissure caries, especially when one considers the following points:

TABLE 4. Fluoride Release for Experimental Group Four\*

0	One Day	Three Days	Analysis Time One Week	Three Weeks
	209.0	124.0	268.0	11.8
ride	129.0	178.0	115.0	440.0
	182.0	185.0	536.0	548.0
	120.0	290.0	194.0	340.0
0	159.0	172.0	290.0	136.0
<u> </u>	172.0	172.0	290.0	390.0
Ρ	258.0	178.0	102.0	600.0
٩	246.0	280.0	268.0	342.0
	129.0	182.0	365.0	474.0
	74.0	178.0	120.0	362.0

\* Sealant +2.0% NaF.

1. The incidence of caries in children is highest on occlusal surfaces<sup>1-3</sup>

2. Fissure sealants have been found to be effective in reducing occlusal caries<sup>4-7</sup>

3. The effect of topical NaF is an effective dental caries reducing agent<sup>8</sup>

4. Fluoride in dentifrices and mouth washes has been shown to be beneficial.<sup>9,10</sup>

Certainly any mechanism that can help reduce the incidence of dental caries on occlusal surfaces is a potential component of an effective public health program.

In addition to determining the fluoride ion release from sealants in vitro, long-term in vivo studies should be conducted which evaluate the factors of concentration, time, rate, and duration of release. Also, the fluoride uptake and solubility of enamel in acid could be measured following application of a sealant containing NaF in concentrations similar to those used in this study (which are lower than other reported concentrations).<sup>15</sup> Studies also are indicated on: (1) the ability of other commercial sealants to accept and release fluoride ions and (2) the effects, if any, on how the addition of NaF may affect sealant properties. Finally, further study is indicated on the anticaries effect of sealants containing NaF.

# Conclusions

Based on the findings of this study, it can be concluded that:

- 1. Nuva Seal<sup>®</sup> fissure sealant has the ability to release fluoride ion over a 3-week period when NaF (0.05, 0.2, 0.5, 2.0% concentrations) was added to the sealant.
- 2. At the lower concentrations (0.05 and 0.2% NaF), there is relatively little increase in fluoride ion release over time while at the higher concentrations (0.5 and 2.0% NaF), fluoride ion release is increased markedly after 3 weeks.

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