

Topical Fluoride in Orthodontic Bonding

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Neutral and acidified sodium fluoride solutions were topically applied to enamel etched in a manner typical for orthodontic bonding procedures. Enamel fluoride levels were greater with the acidified solutions.

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White spot decalcifications, and even carious lesions, sometimes occur around the margins of fixed metal bands or bonded brackets(GORELICK ET AL. 1982, MIZRAHI 1982). The area of exposed etched enamel surrounding resin-bonded brackets lies in an area that is susceptible to formation of such lesions(ZACHRISSON 1977, GORELICK, ET AL. 1982).

It is known that the incorporation of fluoride into the enamel structure as fluorapatite ($\text{Ca}_5(\text{PO}_4)_3\text{F}$) can result in the remineralization of small decalcified or carious lesions, and also reduce the formation of new lesions(SILVERSTONE 1982, MELLBERG AND MALLON 1984). In addition, the regular use of topical applications of various neutral and acidulated sodium fluoride (NaF) formulations can reduce the incidence of new carious lesions in normal enamel(SHANNON 1976).

Use of a post-etch fluoride treatment in association with bracket bonding has been proposed as a preventive measure(ZACHRISSON 1977). Ideally, this would cause minimal interference with the application and subsequent adhesive strength of the resin bond, while providing maximal fluoride uptake for protection against the initiation of carious lesions.

Several investigations have shown that topical treatment with either neutral or acidified NaF preparations results in fluoride incorporation into intact enamel(WEFEL AND HARLESS 1981, DIJKMAN, ET AL. 1982). Other investigators have

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found that acid-etching the enamel before fluoride application increases fluoride uptake (MELLBERG AND LOERTSCHER 1973, BELSER, ET AL. 1975). However, the optimal fluoride concentration and pH for a topical NaF solution has not yet been determined.

The objective of this study was to determine how fluoride levels at specific enamel depths are affected by the concentration and acidity of a NaF solution applied topically to acid-etched enamel.

— Materials and Methods —

Twenty-five human teeth, extracted for orthodontic or other reasons and stored in distilled water, were selected for study. Only teeth exhibiting no cracks, hypoplastic areas or smooth surface caries were used. After thorough brushing with pumice, they were randomly assigned into five groups of five teeth each.

The teeth were then sectioned buccolingually into halves under a water spray. Using a cotton applicator, 50% phosphoric acid (H_3PO_4) was applied to all enamel surfaces for one minute to simulate the pre-bonding etch. Each half-tooth was then washed with water and dried with compressed air.

One half-tooth from each pair was randomly designated to serve as a control, and the other half was used as a treatment specimen. Control specimens were stored individually in vials of distilled water at 4°C.

Each treatment specimen received one of the following five treatments:

1. 1% NaF in distilled water (dH_2O)
2. 2% NaF in dH_2O
3. 4% NaF in dH_2O
4. 2% NaF in 0.1M H_3PO_4 , with pH adjusted to 3.0
5. 4% NaF in 0.1M H_3PO_4 , with pH adjusted to 3.0

Treatments were performed by spreading the fluoride solution onto the enamel surface with a cotton applicator and leaving it for one minute. The enamel surface was then thoroughly rinsed with water spray to minimize secondary fluoride interactions.

Treated specimens were stored under conditions similar to those described for control specimens. All vials, both control and treatment, were coded and randomized for blind evaluation in the laboratory.

Coded specimens were removed from their storage vials and air dried on paper toweling. An adhesive tape disc with a 2mm diameter hole was applied to the cervical enamel of each specimen, and the exposed enamel treated with three successive 12-second applications of 5μl of 2M perchloric acid ($HClO_4$). At the end of each 12-second etch, the remaining acid was absorbed into small circles of ashless filter paper in two successive wipes.

The two filter paper circles from each etch were placed in a vial containing 1ml of 50% total ionic strength buffer in distilled water and allowed to equilibrate for 24 hours prior to calcium and fluoride determinations. Calcium was determined using atomic absorption spectrophotometry at a wavelength of 4225 angstroms. Ionic fluoride was measured using the hanging drop fluoride electrode microprocedure (VENKATESWARLU 1975).

Calculations of enamel mass and depth of the analytical etch were performed using published assumptions and formulae (RETIEF ET AL. 1980, KOULOURIDES AND WALKER 1979).

Following the above procedures, one treatment specimen and its respective control were randomly selected from each of the five groups. These ten specimens were mounted, coated with palladium, and examined at 20 kV by scanning electron microscopy (SEM). Resultant coded

photomicrographs were judged by the investigators for qualitative differences between the surfaces of the control and treatment specimens.

Differences in mean analytical etch depths between control and treatment specimens for each of the three etches were tested using Student's t-test (α level 0.01). Linear regressions and least squares statistical analyses were used to compare fluoride masses at standardized depths of 0-10, 10-20 and 20-30 μm (SADOWSKY, ET AL. 1981).

Overall difference in mean fluoride mass between control and treatment specimens at each standardized depth was also tested with Student's t-test (α level 0.01).

The paired data Student's t-test was used to test whether mean fluoride levels in the treatment specimens differed significantly from control specimens at each standardized depth for each of the five treatment groups.

Differences in fluoride levels across groups before and after fluoride treatments were evaluated with a one-way ANOVA test (α level 0.01). At depths where the ANOVA was significant, the Tukey's Honest Significant Difference (HSD) statistic (α level of 0.05) was used to evaluate all possible comparisons for differences in the mean difference fluoride levels (WINER 1971).

— Results —

Mean analytical etch depth was determined for all specimens in control and treatment groups (Table 1). It should be noted that these values represent the *mean* depth of each etch, not the total etch depth. The mean depth of etch increased with each successive etch for both control and treatment specimens, and for all three etches the controls showed slightly deeper etching than the treatment specimens. The difference

between the control and treatment etches at each depth was not statistically significant.

A greater analytical etch depth and lower fluoride mass were found at successive standardized etch depths for all control and treatment specimens (Table 2). Although the mean fluoride masses at standardized depths were found to be slightly greater in the treatment specimens than in the controls, this tendency was not statistically significant at any of the three standardized depths.

The mean difference and standard deviation between fluoride masses of all control and treatment specimens for the three standardized depths are shown in

Table 1

Mean Depth for Each Perchloric Acid Etch Mean of 25 observations on 25 specimens		
Etch Sequence	Control Depth (μm)	Treatment Depth (μm)
1st	11.13 \pm 1.77	9.99 \pm 1.66
2nd	11.95 \pm 2.28	10.65 \pm 1.70
3rd	12.51 \pm 1.74	11.52 \pm 1.54
t-test not significant at $p < 0.01$.		

Table 2

Mean Fluoride Mass at Standardized Depths Mean of 25 observations on 25 specimens		
Standardized Etch (μm)	Control Mass (μg)	Treatment Mass (μg)
0-10	0.163 \pm 0.057	0.248 \pm 0.060
10-20	0.116 \pm 0.045	0.149 \pm 0.049
20-30	0.102 \pm 0.043	0.129 \pm 0.048
t-test not significant at $p < 0.01$.		

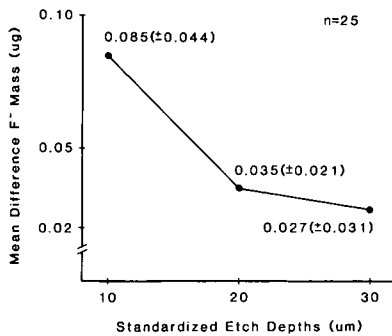


Fig. 1 Mean fluoride mass difference between control and treatment specimens was greatest at shallow depths.

Figure 1. It is apparent that the difference in fluoride level was greatest from the first 10 μ m to the second 10 μ m, and tended to level off at greater depths.

Table 3 shows the mean and standard deviation of the fluoride mass for each group, and their differences at each of the three standardized depths. Mean fluoride masses for the control and treatment specimens showed statistically significant differences for all five groups at 0–10 μ m. However, at 10–20 μ m, only group 2 (2% NaF in dH₂O) and group 5 (4% NaF in 0.1M H₃PO₄ at pH 3.0) differed with statistical significance. At 20–30 μ m, only group 5 demonstrated a significant difference between the mean fluoride mass in the control and treatment specimens.

The ANOVA performed at each of the three standardized depths to examine whether or not the mean fluoride mass difference varied across the five treatment groups showed significant differences at the first and second etch depths. At those depths, the mean fluoride mass difference of at least one group differed from the remaining groups. At the third etch depth there was no significant difference across the five groups. Tukey's

HSD statistic showed significant differences in mean fluoride mass difference only between group 2 and 5 and between 3 and 5 at the first etch depth, and between group 5 and groups 1, 3 and 4 at the second etch depth.

A visual comparison of the SEM photomicrographs indicated that the HClO₄ etched areas were well circumscribed in all specimens. No discernible difference was seen in the quality of the enamel surface between control and treatment specimens in the non-HClO₄-etched areas. A comparison after three HClO₄ etches also indicated no difference in the quality of the surfaces between control and treatment specimens.

— Discussion —

Our findings of increased depth of etch and decreased fluoride mass upon successive etchings corroborate previously published work demonstrating that successive acid etches remove an increasingly greater mass of enamel (DIJKMAN AND ARENDS 1982). Our findings also indicate that each of the three analytical etches of fluoride-treated specimens, on average, was not as deep as its respective etch on the control specimens. This suggests higher levels of incorporated fluoride in the treatment specimens and supports the observation of LEHMAN AND DAVIDSON (1981) that topical fluoride treatment of enamel produces a more acid-resistant outer layer. Increased fluoride mass in enamel plays an important role in decreased enamel solubility (DRIESSENS 1973, SHANNON 1980).

The large variability in etch depth and fluoride mass seen in both treatment and control specimens may be explained by the systemic fluoride history of the teeth. Although the precise history of each tooth was not known, it is possible that some of the teeth were from individuals

who used fluoridated drinking water as children, and some from individuals who dit not.

The teeth were taken from dental patients seen at the Mayo Clinic in Rochester, Minnesota, a community that has had a fluoridated water supply since 1960. However, because Rochester is relatively isolated from other large communities, the Mayo Clinic also serves a large surrounding rural area, much of which is without a fluoridated water supply.

Our demonstration of the greatest difference in fluoride mass between treatment and control specimens at 0–10µm etch depth, and lesser differences at 10–20µm and 20–30µm etch depths, lends support to those who have concluded that

topical fluoride is most effective in increasing the fluoride content of the surface layer of enamel (LEHMAN AND DAVIDSON 1981, DIJKMAN, ET AL. 1982).

The amount of fluoride actually incorporated into enamel as $\text{Ca}_5(\text{PO}_4)_3\text{F}$, in comparison with the amount of fluoride persisting on the surface as CaF_2 , remains unknown. However, we believe that there was little CaF_2 remaining on the fluoride-treated surfaces. This conclusion is based on the low fluoride levels found with the application of 4% NaF in dH_2O , a treatment which is likely to produce large amounts of CaF_2 (McCANN 1968, RAMSEY, ET AL. 1973). Further, the thorough rinsing and storage in distilled water would have removed much of any

Table 3 Mean Fluoride Masses at Standardized Depths Grouped by Fluoride Treatment Means of 5 observations on 5 specimens					
Group	Standardized Depth (µm)	Control Mass (µg)	Treatment Mass (µg)	Difference Mass (µg)	Paired Diff t-test (p<0.01)
1 1% NaF in dH ₂ O	0-10	0.168±0.046	0.246±0.050	0.078±0.038	sig.
	10-20	0.122±0.022	0.148±0.042	0.026±0.022	n.s.
	20-30	0.106±0.021	0.128±0.041	0.022±0.028	n.s.
2 2% NaF in dH ₂ O	0-10	0.135±0.058	0.214±0.059	0.079±0.032	sig.
	10-20	0.107±0.044	0.146±0.034	0.039±0.014	sig.
	20-30	0.102±0.052	0.130±0.031	0.028±0.024	n.s.
3 4% NaF in H ₂ O	0-10	0.189±0.067	0.242±0.074	0.053±0.027	sig.
	10-20	0.133±0.054	0.164±0.069	0.031±0.027	n.s.
	20-30	0.112±0.052	0.142±0.065	0.030±0.037	n.s.
4 2% NaF in H ₃ PO ₄	0-10	0.184±0.069	0.262±0.071	0.078±0.042	sig.
	10-20	0.132±0.058	0.150±0.077	0.018±0.025	n.s.
	20-30	0.118±0.052	0.126±0.078	0.008±0.040	n.s.
5 4% NaF in H ₃ PO ₄	0-10	0.136±0.039	0.278±0.043	0.142±0.026	sig.
	10-20	0.084±0.036	0.138±0.027	0.054±0.017	sig.
	20-30	0.072±0.030	0.118±0.022	0.046±0.021	sig.

CaF₂ that might have formed (MELLBERG, ET AL. 1966). Finally, there were no clear visual differences, as viewed by SEM, in the surfaces of fluoride-treated and control tooth halves.

The higher levels of fluoride found at the second and third etch depths of treated specimens approximately followed NaF concentrations. Exceptions were with 4% NaF in dH₂O (above) and 2% NaF in 0.1M H₃PO₄ (below). We feel that it is reasonable to conclude that 4% NaF in 0.1M H₃PO₄ was the most effective topical fluoride treatment of those used in this study. This is in agreement with other authors who have found that acidified NaF is more readily incorporated into enamel than is neutral NaF (MELLBERG AND LOERTSCHER 1972, KIRKEGAARD 1977). The extremely low levels of fluoride seen at the second and third etch depths following 2% NaF in 0.1M H₃PO₄ treatment cannot be readily explained.

Assuming that the practitioner wants to use a topical fluoride treatment to increase enamel fluoride levels, the next question is when it should be applied in relation to the bonding procedure. It could be applied either immediately after acid etching but before resin placement, or after the entire bonding procedure has been completed.

Studies using SEM to examine the effect of various topical fluoride treatments on acid-etched enamel surfaces have demonstrated the formation of a globular reaction product (possibly CaF₂) on the etched surface following topical fluoride treatment (GWINNETT, ET AL. 1972, KOCHAVI, ET AL. 1975). The authors speculate that this reaction product could cause a reduction in resin bond strength.

However, studies in which bond strength has been measured indicate that some topical fluoride treatments (eg. acidified NaF, stannous fluoride, and basic phosphate fluoride) did not cause signifi-

cantly lower bond strength (LOW, ET AL. 1977, HIRCE, ET AL. 1980).

Thorough rinsing following topical fluoride application may be important to remove residual fluoride products that could interfere with bond strength (MELLBERG, ET AL. 1966, GEDALIA, ET AL. 1979).

The best time to apply a topical fluoride treatment during the orthodontic bracket bonding procedure remains an open question.

— Conclusions —

Treatment of acid-etched enamel with 4% NaF in an acidified vehicle produced the highest levels of incorporated fluoride of any of the treatments used in these experiments. In general, topical application of acidified NaF appears to be more effective than neutral NaF treatments.

Although this study did not specifically address resin bond strength following fluoride treatment, some studies have indicated that this type of post-etch fluoride treatment does not alter bond strength. Before it can be recommended that acidified NaF solutions be used prior to resin placement, further resin bond strength tests need to be made on etched enamel surfaces treated in the manner described here.

However, it can be recommended at this time that acidified topical NaF treatments rather than neutral NaF be used, after resin placement, to incorporate maximum fluoride into the exposed etched enamel periphery surrounding bonded orthodontic brackets and other resin procedures.

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REFERENCES

- Belser, U.; Spörri, S.; and Mühlemann, H. R. 1975. Uptake and retention of fluoride by intact and etched enamel. *Helv. Odontol. Acta* 19(2):69-71.
- Dijkman, A. G. and Arends, J. 1982. Thickness of enamel layers removed by HClO_4 etching. *Caries Res.* 16:129-37.
- Dijkman, A. G. and Arends, J. 1982. Fluoride deposited by topical applications in enamel: KOH-soluble and acquired fluoride. *Caries Res.* 16:147-55.
- Driessens, F. C. M. 1973. Fluoride incorporation and apatite solubility. *Caries Res.* 7:297-314.
- Gedalia, I.; Grajower, R.; Baharev, H.; Glick, A.; and Kochavi, D. 1979. Treatment of enamel with 50% phosphoric acid containing 0.5% and 2% NaF. *J. Dent. Res.* 58(B):1004-05.
- Gorelick, L.; Geiger, A.M.; and Gwinnett, J.A. 1982. Incidence of white spot formation after bonding and banding. *Am. J. Orthod.* 81(2):93-98.
- Gwinnett, A. J.; Buonocore, M. G.; and Sheykholeslam, Z. 1972. Effect of fluoride on etched human and bovine tooth enamel surfaces as demonstrated by scanning electron microscopy. *Arch. Oral Biol.* 17:271-78.
- Hirce, J. D.; Sather, A. H.; and Chao, E. Y. S. 1980. The effect of topical fluorides, after acid etching of enamel, on the bond strength of directly bond orthodontic brackets. *Am. J. Orthod.* 78(4):444-52.
- Kirkegaard, E. 1977. In vitro fluoride uptake in human dental enamel from various fluoride solutions. *Caries Res.* 11:16-23.
- Kochavi, D.; Gedalia, I.; and Anaise, J. 1975. Effect of conditioning with fluoride and phosphoric acid on enamel surfaces as evaluated by scanning electron microscopy and fluoride incorporation. *J. Dent. Res.* 54(2):304-09.
- Koulourides, N.; and Walker, A. 1979. Fluoride distribution in the facial surfaces of human maxillary central incisors. *J. Oral Pathol.* 8:179-83.
- Lehman, R.; and Davidson, C. L. 1981. Loss of surface enamel after acid etching procedures and its relation to fluoride content. *Am. J. Orthod.* 80(1):73-82.
- Low, T.; von Fraunhofer, J. A.; and Winter, G. B. 1977. Influence of the topical application of fluoride on the in vitro adhesion of fissure sealants. *J. Dent. Res.* 56(1):17-20.
- McCann, H. G. 1968. The solubility of fluorapatite and its relationship to that of calcium fluoride. *Arch. Oral Biol.* 13:987-1001.
- Mellberg, J. R.; Laakso, P. V.; and Nicholson, C. R. 1966. The acquisition and loss of fluoride by topically fluoridated human tooth enamel. *Arch. Oral Biol.* 11:1213-20, 1966.
- Mellberg, J. R.; and Loertscher, K. L. 1972. Fluoride acquisition in vitro by sound human tooth enamel from sodium fluoride and ammonium silicofluoride-phosphate solutions. *Arch. Oral Biol.* 17:1107-16.
- Mellberg, J. R.; and Loertscher, K. L. 1973. Fluoride acquisition in vitro by etched enamel from acidulated phosphate-fluoride preparations. *J. Dent. Res.* 53(3):447-50.
- Mellberg, J. R.; and Mallon, D. E. 1984. Acceleration of remineralization in vitro by sodium monofluorophosphate and sodium fluoride. *J. Dent. Res.* 63(9):1130-35.
- Mizrahi, E. 1982. Enamel demineralization following orthodontic treatment. *Am. J. Orthod.* 82(1):62-7.
- Ramsey, A. C.; Duff, E. J.; Paterson, L.; and Stuart, J. L. 1973. The uptake of F- by hydroxyapatite at varying pH. *Caries Res.* 7:231-44.
- Retief, D. H.; Sorvas, P. G.; Bradley, E. L.; Taylor R. E.; and Walker, A. K. 1980. In vitro fluoride uptake, distribution and retention by human enamel after 1- and 24-hour application of various topical fluoride agents. *J. Dent. Res.* 59(3):573-82.
- Sadowsky, P. L.; Retief, D. H.; and Bradley, E. L. 1981. Enamel fluoride uptake from orthodontic cements and its effect on demineralization. *Am. J. Orthod.* 79(5):523-34.
- Shannon, I. L. 1976. Mouthrinsing with fluoride. A review of clinical studies. *Dent. Hyg.* 50:448-54.
- Shannon, I. L. 1980. Responses of enamel, dentin, and root surfaces to mouthrinse concentrations of sodium fluoride and stannous fluoride. *J. Dent. Child.* 47:17-20.
- Silverstone, L. M. 1982. The effect of fluoride in the remineralization of enamel caries and caries-like lesions in vitro. *J. Pub. Health Dent.* 42(1):42-53.
- U.S. Department of Health and Human Services, Public Health Service. June 1984. *Fluoridation census 1980*, Atlanta, Georgia.
- Venkateswarlu, P. 1975. A micro method for direct determination of ionic fluoride in body fluids with the hanging drop fluoride electrode. *Clin. Chim. Acta* 59:277-82.
- Wefel, J. S.; and Harless, J. D. 1981. The effect of topical fluoride agents on fluoride uptake and surface morphology. *J. Dent. Res.* 60(11):1842-48.
- Winer, B. J. 1971. *Statistical principles in experimental design*. 2nd ed. McGraw-Hill New York.
- Zachrisson, B. U. 1977. A post-treatment evaluation of direct bonding in orthodontics. *Am. J. Orthod.* 71:173-89.