



## IN VITRO STAINING EFFECTS OF STANNOUS FLUORIDE AND SODIUM FLUORIDE ON CERAMIC MATERIAL

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**Statement of problem.** Long-term fluoride application on the teeth of patients receiving radiation therapy for head and neck tumors results in excessive staining and roughening of ceramic restorations.

**Purpose.** The purpose of this in vitro study was to compare the staining effects of 2 fluoride treatments on ceramic disks by simulating 1 year of clinical exposure at 10 minutes per day. In addition, 2 different surface preparations were tested.

**Material and methods.** Eighty ceramic disks (IPS Empress), 20 x 2 mm, were fabricated. Half of the disks were glazed, and the remaining disks were polished. All disks were brushed for 3 minutes with a soft-bristle power toothbrush and mild dentifrice (baseline) and were immersed in 1 of the 2 fluoride products (0.4% SnF<sub>2</sub>, Gel-Kam Gel, or 1.1% NaF, Prevident 5000) for 10 days (n=20). Means and standard deviations of color change ( $\Delta E$ ), surface roughness (Ra,  $\mu m$ ), and surface gloss (GU) of the ceramic material were measured with a reflection spectrophotometer, a profilometer, and a gloss meter, respectively, at baseline and after fluoride treatment. Two- and 3-way ANOVA ( $\alpha=.05$ ), with surface preparation (polished vs. glazed) and fluoride treatment (0.4% SnF<sub>2</sub> or 1.1% NaF) as independent variables and condition (baseline vs. after fluoride treatment) as a repeated measure, was used to analyze the data. Fisher's PLSD intervals ( $\alpha=.05$ ) were calculated for comparisons among the means.

**Results.** The polished specimens had significantly higher  $\Delta E$  values, significantly higher surface gloss values, and significantly lower surface roughness values than the glazed specimens before fluoride treatment ( $P<.001$ ). After both fluoride treatments, ceramic disks exhibited significantly higher surface roughness values when polished and significantly lower surface gloss values when glazed or polished ( $P<.001$ ). The glazed specimens presented significantly higher surface roughness ( $P<.001$ ) and lower surface gloss values ( $P<.001$ ) when treated with 0.4% SnF<sub>2</sub> as compared to NaF. For the polished specimens, there was no significant difference in surface roughness and surface gloss values between the 2 fluoride treatments.

**Conclusions.** Use of 0.4% SnF<sub>2</sub> and 1.1% NaF gels, in vitro, caused significant color change in the polished IPS Empress ceramic disks. Polishing of the ceramic surface before immersion in either fluoride agent caused the ceramic tested to be more resistant to etching by the 2 solutions tested. The NaF caused less deterioration of the porcelain surface and was less stain inducing than SnF<sub>2</sub>. (J Prosthet Dent 2010;103:163-169)

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## CLINICAL IMPLICATIONS

To minimize the deteriorating effects of long-term fluoride treatment on dental porcelain, polishing of the ceramic surface before fluoride application is recommended. In addition, daily application of 0.4% SnF<sub>2</sub> could be alternated with courses of 1.1% NaF, which appears to be less damaging.

Several studies have shown that irradiation-induced xerostomia in patients with cancer of the head and neck is associated with changes in the oral microflora and a high risk for radiation-induced caries.<sup>1-6</sup> Radiation-induced caries is defined as a rampant dental breakdown that can progress rapidly, is difficult to control, and results from changes in salivary gland function due to therapeutic ionizing radiation therapy.<sup>7,8</sup> This aggressive form of dental caries can ultimately and irreversibly infect the dentinal pulpal tissue and may initiate osteoradionecrosis in the supporting irradiated bone, resulting in severe complications.<sup>9,10</sup>

The management strategy indicated in clinical studies in which almost complete caries prevention was achieved in irradiated patients includes meticulous oral hygiene and daily fluoride application.<sup>10,11-19</sup> The most common fluoride agents currently in use are 1.0% sodium fluoride (NaF) gel<sup>20-22</sup> and 0.4% stannous fluoride (SnF<sub>2</sub>) gel.<sup>11,23</sup> SnF<sub>2</sub>, at a 0.4% concentration, has a pH of 3.2, making it more acidic than 1.0% NaF. The soft tissue irritation experienced by patients during radiation therapy and during the postirradiation period has been problematic for up to 2% of patients.<sup>11</sup> The caries prevention protocol currently used at M. D. Anderson Cancer Center involves daily topical self-application of the 0.4% SnF<sub>2</sub> gel of choice by means of custom-fabricated polypropylene applicators that completely cover and extend slightly beyond the tooth surfaces.<sup>9,11</sup> Maxillary and mandibular fluoride carriers are positioned over the dentition for a 10-minute period every day following

oral hygiene procedures (flossing and brushing). After removing the fluoride carriers, the patient is instructed to swish the accumulated saliva and residual gel for 1 minute and then to expectorate the excess, and to refrain from rinsing, drinking, and brushing for 30 minutes to avoid disturbing the applied fluoride film.<sup>11</sup>

Numerous in vitro studies have shown that topically applied fluoride agents may cause surface changes and volume loss in dental materials, including ceramic, composite resins, sealants, and glass ionomer materials.<sup>24-33</sup> Fluoride agents with a low pH and a high fluoride concentration have been shown to cause more surface damage to dental porcelain; therefore, a neutral fluoride agent with a low fluoride concentration is recommended for use, especially for daily in-home fluoride treatment.<sup>26-28</sup> In a study by Copps et al,<sup>26</sup> both 0.4% SnF<sub>2</sub> fluoride and 0.5% and 1.1% neutral fluoride preparations did not affect dental porcelains after 20 minutes of exposure. However, Wunderlich and Yaman<sup>28</sup> reported that neither NaF solutions of 0.05%, 0.2%, and 2% concentrations, nor 0.4% SnF<sub>2</sub> gel, had a significant effect on the surface roughness of porcelain surfaces after exposure for 4, 10, 15, and 20 minutes, or 5 days. Demirhanoglu and Sahin<sup>31</sup> reported that 0.4% SnF<sub>2</sub> gel had no roughening effect on porcelain surfaces after 6 and 12 hours of application, whereas Kula and Kula<sup>32</sup> showed that there were no significant differences in visual scores when the color change in porcelain restorations following 4-minute treatment with 2.0% NaF was compared to the control group. However, the authors

could identify no studies in which the application time for the fluoride regimen replicated the significant clinical exposure as suggested by an effective daily topical self-application protocol.<sup>4,9-11,23,34</sup>

Color can be defined by the CIE L\*a\*b\* color system. The CIE L\*a\*b\* system is derived from the Commission Internationale d'Eclairage (CIE)<sup>35</sup> standard color table, and involves transforming the original 3 colorimetric coordinates (color values) x, y, and z into 3 new reference values: L\*, a\*, and b\*. The objective of this transformation is a color space to aid the numerical classification of color differences. These values are calculated from the X, Y, and Z tristimulus values of the CIE L\*a\*b\* color system. The L\* parameter corresponds to the degree of lightness or darkness in the Munsell system. The a\* and b\* coordinates correspond to the chromaticity of the specimen; the a\* coordinate is the red-green component, and the b\* coordinate is the blue-yellow component. A positive a\* value indicates the amount of red, and a negative a\* value relates to the amount of green. A positive b\* value relates to the amount of yellow in the specimen, and a negative b\* value corresponds to the amount of blue. As in the Munsell system, chroma increases with the distance from the L\* axis on both the a\* and b\* coordinates. The CIE color difference, ΔE, is the total color change occurring in all 3 variables and is calculated with the equation<sup>35</sup>:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

Goldstein and Schmitt<sup>36</sup> proposed that a ΔE value of greater than 0.4 is detectable by the highly trained human eye. O'Brien et al<sup>37</sup> classified the

different values of  $\Delta E$  on the basis of clinical observations, with 1 as excellent, 2 as clinically acceptable, and 3.7 as a poor match. A  $\Delta E$  value between 1 and 2 was classified as the lowest color change the human eye could perceive.<sup>38</sup>

The purpose of this *in vitro* study was to determine the staining effect of 2 different fluoride treatments on a ceramic material, simulating 1 year of clinical exposure for 10 minutes per day. In addition, 2 ceramic surface preparations were tested. The null hypothesis was that there would be no difference between the effects of the 2 different fluoride treatments on color change, surface roughness, and surface gloss of a ceramic material with 2 surface preparations.

## MATERIAL AND METHODS

Eighty specimens were modeled in casting wax (Kerr Corp, Orange, Calif). The matrix-formed cylindrical disks were 20 mm in diameter and 2 mm thick, as recommended in American Dental Association specification No. 69.<sup>39</sup> The wax patterns were invested (IPS PressVEST; Ivoclar Vivadent, Inc, Amherst, NY) using the system's cylindrical muffle formers. After investing, the muffle was preheated to 800°C to burn out the wax pattern. The ceramic ingot and the aluminum oxide plunger were placed in the cylindrical opening. The readied muffle was then placed in the furnace (EP 600 Combi; Ivoclar Vivadent, Inc) to initiate the program. After the necessary temperature was reached (1150°C) and a holding time of 20 minutes had elapsed, the pressing procedure was fully automated. The softened ceramic was slowly pressed into the mold under vacuum. After pressing, specimens were divested using airborne-particle abrasion (Glass Beads; Shofu, Inc, Kyoto, Japan).

Half ( $n=40$ ) of the ceramic disks were polished to a definitive finish of 15  $\mu\text{m}$  using 600-grit silicon carbide paper (3M ESPE, St. Paul, Minn), and the other half were glazed with

glazing paste (IPS Empress Universal Glaze Paste; Ivoclar Vivadent, Inc). The ceramic disks were brushed for 3 minutes with a soft-bristle power toothbrush (Sonicare; Optiva Corp, Bellevue, Wash) and mild dentifrice (Colgate Total; Colgate-Palmolive Co, New York, NY), as baseline (control group), and were immersed in 1 of the 2 fluoride products (0.4%  $\text{SnF}_2$  in Colgate Gel-Kam Gel or 1.1% NaF in Colgate Prevident 5000; Colgate-Oral Pharmaceuticals, Inc, New York, NY) ( $n=20$ ) for 10 days to replicate 1 year of clinical exposure, 10 minutes per day. This protocol was intended to simulate the 10 minutes that the patient applies the fluoride regimen, plus the following 30 minutes that the patient is instructed to refrain from rinsing, drinking, and brushing. Specimen preparation, as well as the color, surface roughness, and surface gloss readings, were performed by the same operator.

The color of the ceramic disks was measured according to the CIE  $L^*a^*b^*$  color scale relative to the standard illuminant D65, and over a white standard tile (CIE  $L^*=82.4$ ,  $a^*=20.1$ , and  $b^*=20.6$ ) on a reflection spectrophotometer (ColorEye 7000A; X-Rite, Grand Rapids, Mich) with specular component excluded (SCE) geometry. A UV filter was positioned to include 100% UV. The aperture size was 25 mm, and the illuminating and viewing configuration was CIE diffuse/8-degree geometry. The CIE  $L^*a^*b^*$  system was used to determine the changes in the color of the specimens through measurement of the  $L^*a^*b^*$  values and computation of the  $\Delta E$  at baseline and after fluoride treatment. Average surface roughness (Ra) of each disk at baseline and after fluoride treatment was measured with a profilometer (Form Talysurf Plus; Taylor Hobson Ltd, Leicester, UK), with a cutoff value of 0.8 mm, a transverse length of 4.5 mm, and a measuring speed of 0.25 mm/s. Three tracings per specimen were performed, at a 120-degree angle in relation to each other, in the

middle of the specimen.

Surface gloss of each ceramic disk at baseline and after fluoride treatment was measured with a small-area glossmeter (Novo-Curve; Rhopoint Instruments, East Sussex, UK) with a square measurement area of 2 x 2 mm and 60-degree geometry. Gloss was expressed as gloss unit (GU). Means and standard deviations of the color change ( $\Delta E$  following fluoride treatment minus baseline), surface roughness (Ra), and surface gloss (GU) of the ceramic material were calculated at baseline and after fluoride treatment. A 2-way analysis of variance (ANOVA,  $\alpha=.05$ ) (StatView; SAS Institute, Inc, Cary, NC), with surface preparation (polished vs. glazed) and fluoride treatment (0.4%  $\text{SnF}_2$  vs. 1.1% NaF) as independent variables, was performed for  $\Delta E$ . A 3-way ANOVA ( $\alpha=.05$ ), with surface preparation (polished vs. glazed) and fluoride treatment (0.4%  $\text{SnF}_2$  vs. 1.1% NaF) as independent variables, and condition (baseline and after fluoride treatment) as a repeated measure, was performed for surface roughness and gloss. Fisher's PLSD intervals ( $\alpha=.05$ ) were calculated for comparisons among the mean values.

## RESULTS

Means and standard deviations for  $\Delta E$  are presented in Table I. Two-way ANOVA results for  $\Delta E$  are shown in Table II. Significant main effects and interactions were observed for surface preparation and fluoride treatment. Analysis of the data in Table I showed that with both fluoride treatments (0.4%  $\text{SnF}_2$  and 1.1% NaF), the polished disks had significantly higher  $\Delta E$  values than the glazed ones. For the glazed specimens, there was no significant difference in  $\Delta E$  values between the 2 fluoride treatments. The polished specimens presented significantly higher  $\Delta E$  values when treated with 0.4%  $\text{SnF}_2$ .

Means and standard deviations for surface roughness are presented in Table III, and the 3-way ANOVA

**TABLE I.** Means (SD) for  $\Delta E$ 

		Fluoride Treatment - Baseline
Glazed	SnF <sub>2</sub>	2.0 (0.7) <sup>a</sup>
	NaF	1.8 (0.6) <sup>a</sup>
Polished	SnF <sub>2</sub>	5.0 (0.6) <sup>c</sup>
	NaF	3.5 (0.8) <sup>d</sup>

Means with same superscripted lowercase letters are not statistically different ( $P > .05$ ). Fisher's PLSD intervals for comparisons of means between polished vs. glazed and between SnF<sub>2</sub> and NaF were 0.4 and 0.4, respectively.

**TABLE II.** Two-way ANOVA for  $\Delta E$ 

	<i>df</i>	Sum of Squares	Mean Square	F	<i>P</i>
Surface preparation (prep)	1	56.16	56.16	116.9	<.001
Fluoride treatment (fl)	1	6.95	6.95	14.5	<.001
Prep x fl	1	4.05	4.05	8.4	.006
Residual	36	17.3	0.48		

**TABLE III.** Means (SDs) for surface roughness (Ra)

		Baseline	After Fluoride
Glazed	SnF <sub>2</sub>	0.43 (0.11) <sup>ag</sup>	0.46 (0.12) <sup>cg</sup>
	NaF	0.36 (0.16) <sup>bh</sup>	0.36 (0.15) <sup>dh</sup>
Polished	SnF <sub>2</sub>	0.04 (0.01) <sup>e</sup>	0.11 (0.06) <sup>f</sup>
	NaF	0.04 (0.01) <sup>e</sup>	0.15 (0.09) <sup>f</sup>

Means with same superscripted lowercase letters are not statistically different ( $P > .05$ ). Fisher's PLSD intervals for comparisons of means between polished vs. glazed, between SnF<sub>2</sub> and NaF, and between baseline and after fluoride were 0.05, 0.05, and 0.05  $\mu\text{m}$ , respectively.

results are presented in Table IV. Significant main effects and interactions were observed for surface preparation, condition, and fluoride treatment. Data analysis in Table III showed that in both conditions (baseline and after fluoride), and with both fluoride treatments (0.4% SnF<sub>2</sub> and 1.1% NaF), the glazed disks had significantly higher surface roughness values than the polished ones. Follow-

ing both types of fluoride treatment, surface roughness values were significantly higher for the polished disks, whereas for the glazed specimens, surface roughness values remained unaffected as compared to baseline. The glazed specimens presented significantly higher surface roughness values when treated with 0.4% SnF<sub>2</sub> as compared to NaF treatment. For the polished specimens, there was no sig-

nificant difference in surface roughness values between the 2 fluoride treatments.

Means and standard deviations for surface gloss are presented in Table V, and the 3-way ANOVA results are shown in Table VI. Significant main effects but no interactions were observed for surface preparation and condition. In both conditions (baseline and after fluoride), and with both

**TABLE IV.** Three-way ANOVA for surface roughness (Ra)

	<i>df</i>	Sum of Squares	Mean Square	F	<i>P</i>
Surface preparation (prep)	1	2.022	2.022	179.991	<.0001
Condition (cond)	1	0.050	0.050	4.470	.0380
Fluoride treatment (fl)	1	0.023	0.023	2.047	.1569
Prep × cond	1	0.030	0.030	2.663	.1071
Prep × fl	1	0.056	0.056	5.016	.0282
Cond × fl	1	$6.2 \times 10^{-5}$	$6.2 \times 10^{-5}$	0.006	.9409
Prep × cond × fl	1	0.006	0.006	0.542	.4642
Residual	72	0.809	0.011		

**TABLE V.** Means (SDs) for surface gloss (GU)

		Baseline	After Fluoride
Glazed	SnF <sub>2</sub>	41 (7) <sup>a</sup>	32 (8) <sup>d</sup>
	NaF	47 (12) <sup>b</sup>	38 (12) <sup>eg</sup>
Polished	SnF <sub>2</sub>	57 (10) <sup>c</sup>	43 (10) <sup>f</sup>
	NaF	59 (12) <sup>c</sup>	40 (7) <sup>fg</sup>

Means with same superscripted lowercase letters are not statistically different ( $P > .05$ ). Fisher's PLSD intervals for comparisons of means between polished vs. glazed, between SnF<sub>2</sub> and NaF, and between baseline and after fluoride were 4, 4, and 4 GU, respectively.

**TABLE VI.** Three-way ANOVA for surface gloss

	<i>df</i>	Sum of Squares	Mean Square	F	<i>P</i>
Surface preparation (prep)	1	1992	1992	18.95	<.001
Condition (cond)	1	3406	3406	32.39	<.001
Fluoride treatment (fl)	1	150	150	1.43	.236
Prep × cond	1	288	288	2.74	.102
Prep × fl	1	183	183	1.74	.191
Cond × fl	1	35	35	0.329	.568
Prep × cond × fl	1	27	27	0.256	.615
Residual	72	7571	105		

fluoride treatments (0.4% SnF<sub>2</sub> and 1.1% NaF), the polished disks had significantly higher surface gloss values than the glazed ones. Surface gloss values were always significantly higher at baseline than after fluoride treatment for both surface preparations (polished and glazed) and both fluoride treatments (0.4% SnF<sub>2</sub> and 1.1% NaF). The glazed specimens presented significantly higher surface gloss values when treated with 0.4% SnF<sub>2</sub>. For the polished specimens, there was no significant difference in surface gloss values between the 2 fluoride treatments.

## DISCUSSION

The results of this study support rejection of the null hypothesis that there would be no significant differences between the effects of the 2 different fluoride treatments on color change, surface roughness, and surface gloss of a ceramic material with 2 surface preparations. In the present study, all of the specimens experienced color change after immersion in the fluoride solution, with the polished ceramic disks always recording higher  $\Delta E$  values than the glazed disks. A possible reason for this finding could be the resultant increase in roughness of the polished specimens after treatment with the fluoride.

When white light is cast on the surface of a solid, some of the light is reflected from the surface and remains white light.<sup>40</sup> This light, when mixed with the light reflected from the object itself, dilutes the color of the object. Therefore, a rougher surface appears lighter than a smooth surface of the same material.<sup>40</sup> Furthermore, the nonglazed polished surface provides for the seepage of staining materials through the porosities and surface defects of the porcelain. Both fluorides had similar effects on glazed specimens, whereas the polished specimens treated with SnF<sub>2</sub> demonstrated higher color changes than those immersed in NaF. This could be related to the increased acidity of the SnF<sub>2</sub>

agent (pH=4) when compared to the neutral NaF (pH=7).

It is apparent from the present study that fluoride treatment contributed to significant roughening of the polished ceramic specimens. However, the polished ceramic disks always appeared to be smoother than the glazed specimens. A possible explanation is that glazing can produce an undulating and rough surface that has irregularities. These results are in agreement with earlier studies in which polishing of dental porcelain reduced the effects of immersion in fluoride solutions.<sup>26,33</sup>

Surface gloss changes relate to alterations in specular reflectance, as measured at a 45-degree angle. Overall, the surface gloss of all of the specimens following fluoride treatment was reduced when both regimens were used. The polished specimens had higher surface gloss than the glazed specimens, which could be explained by the relatively increased surface roughness recorded by the glazed specimens. Both fluoride agents had the same effect on the polished specimens, whereas the glazed specimens demonstrated higher gloss values when NaF was used. This, again, could be related to the acidity of the applied fluoride product.

Limitations of this study include the possible effect of dietary factors and more frequent oral hygiene procedures. These could result in further abrasion of the porcelain surfaces and an increase in the staining and roughening effects of the fluoride regimen, and were not evaluated. Furthermore, the study used disk-shaped specimens, which are dissimilar in shape to dental restorations, and this may have influenced the propensity for discoloration and surface roughening. In an effort to reproduce tooth brushing as a component of regular oral hygiene procedures, the ceramic specimens were brushed before immersion in the fluoride product using a soft-bristle toothbrush and mild dentifrice. Further research is indicated to evaluate the long-term in vivo effects of fluoride on dental porcelain.

## CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn:

1. Both fluoride gels tested in vitro caused significant color change to the polished IPS Empress ceramic disks.
2. Both fluoride regimens increased the surface roughness of the polished disks, and decreased the surface gloss for both glazed and polished ceramic specimens.
3. Polishing of the ceramic surface before immersion in either fluoride agent caused the ceramic tested to be more resistant to etching by the 2 solutions. The NaF (1.1%) agent caused less deterioration to the porcelain surface than 0.4% SnF<sub>2</sub>.

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