Effect of 1- and 4-minute treatments of topical fluorides on a composite resin

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Abstract

The purpose of this in vitro study was to compare the effects of a 1-min immersion and of 4-min immersions in an acidulated phosphate fluoride (1.23% APF) foam, a 1.23% APF gel, a 2.0% sodium fluoride (NaF) gel, and water on surface topography and on weight of a composite resin (APH). Forty composite resin specimens were placed into eight groups (N = 5 each). For each treatment, a group of specimens was immersed for either 1 or 4 min (four 1-min immersions). Specimens were weighed before and after each immersion. The surface topography of two scanning electron micrographs of each specimen was scored visually by two investigators. Inter-rater reliability was r = 0.75 (intraclass correlation coefficient). There were no significant differences in the mean visual scores or weight among the 1-min immersion groups. Significantly greater surface changes and weight loss of this composite resin occurred following 4-min immersions in either 1.23% APF foam or gel as compared with those immersed in either 2.0% NaF gel or water (P 0.0001; one-way ANOVA, Tukey's Studentized Range Test). (Pediatr Dent 18:24–28, 1996)

Composite resin restorations are used to restore carious or hypoplastic posterior teeth in children and adolescents. Patients often receive topical fluoride treatments on a semiannual basis. However, there is concern that topical acidulated phosphate fluorides (APF) may cause deterioration of composite resins.[1-5]

In vitro studies show that dental materials such as porcelain, composite resins, sealants, and glass ionomer materials[1-14] are susceptible to surface change and weight loss when treated with some topically applied fluorides. These dental materials contain glass-like substances that can react at acidic pH levels. The presence of acids such as hydrofluoric acid and phosphoric acid in the APF agents may cause surface and weight changes of the dental materials.[2-4] Hydrofluoric acid is a well-known glass etchant. Phosphoric acid is used to etch glass-ionomer materials before placing a superficial layer of posterior composite resin.[15] The amount of weight loss and surface changes[2] is influ-

enced by the length of time that a composite resin is exposed to an APF agent. However, there is limited information[16] on the effect of 1-min immersion or 4-min immersions of composite resins in APF agents, in particular, an APF foam.

The purposes of this in vitro study were to compare surface topography and weight of a composite resin following 1-min immersion and following 4-min immersions in either 1.23% APF foam, 1.23% APF gel, 2.0% NaF gel, or water.

Methods and materials

Topical fluorides

The following topical fluoride agents were used:

1. 1.23% APF gel (Oral-B Minute-Gel™, Oral-B Co, Redwood City, CA), 1.23% F (w/w), pH 3.0–4.0, specific gravity 1.13–1.20

2. 2.0% neutral sodium fluoride gel (Neutra-Care™, Oral-B Co, Redwood City, CA), 0.9% F (w/w), pH 6.2–7.2, specific gravity 1.05–1.11

3. 1.23% APF foam (Minute-Foam™, Oral-B Co, Redwood City, CA), 1.23% F (w/w), pH 3.0–4.0, specific gravity 0.15

4. Deionized, distilled water.

The fluoride products were received labeled only with a reference number. The products were not identified until the end of the study. However, the physical consistency of the foam made identification evident when compared with the gels. Therefore, all specimens and micrographs were coded so that raters and the electron microscopist were masked as to the treatment allocations.

Specimen preparation

Forty specimens of a composite resin containing barium boroaluminosilicate glass and silica filler particles (Prism®APH®, Caulk Co, Milford, DE) were prepared by condensing the material into stainless steel dies (0.7 in. x 0.1 in.) placed on glass slabs. Each specimen was cured for 80 sec per side using an incandescent light source (Elipar, ESPE, West Germany). All

specimens were polished using 600-grit sandpaper (Belden Corp, Chicago, IL) and $1.0 \mu m$, $0.3 \mu m$, and 0.05 μm alumina polishes (Buehler Micropolishes, Buehler, Ltd, Evanston, IL) and stored in distilled water.

Specimen treatment

The specimens were divided into eight groups with five specimens per group. A group of specimens was immersed in each treatment for either 1 min or 4 min (four 1-min immersions). Specimens were weighed twice before and twice after each immersion. The precision of the weighing method (± 0.02 mg) was determined as described in Kula et al.[2]

Following treatment, all specimens were stored dry until they were examined using the scanning electron microscope.

Scanning electron micrographs

Specimens were sputter coated (Polaron 5200 coating unit™; Polaron Instruments, Inc, Hatfield, PA) with palladium and gold and examined using the scanning electron microscope (ETEC Autoscan™; Hayward, CA). Each specimen was photographed twice, once in the center and once approximately 1–2 mm from the edge of the specimen.

Each micrograph (two per specimen) was coded. Two raters randomly evaluated each micrograph for surface topography. A micrograph of a composite resin showing the fewest surface voids was selected as a ref-

erence for a score of 1, and a micrograph of a specimen showing extensive surface voids was selected as a reference for a score of 4. Raters were asked to score the topography on each micrograph using the following criteria:

1. Surface smooth without voids; filler particles intact
2. Surface with small voids but not as many or as large as 3
3. Surface with more or larger voids than in 2, but not as many or as large as in 4
4. Surface with large or many voids.

One rater scored in 0.5 increments from 1 to 4. The sum of the four scores for each specimen (two raters times two micrographs per specimen) was calculated as the outcome measure. The lowest possible sum of visual scores was 4.0 (sum of four scores of 1) and the highest was 16.0 (four scores of 4). Ten micrographs were scored on two separate days by each rater to determine intrarater reliability.

Data analysis

The null hypotheses were that there were no significant differences among the treatment groups treated for 1 min or for 4 min. A one-way ANOVA and Tukey's Studentized Range Test was used to compare the average values among the treatment groups. The visual scores for all micrographs ($N = 80$) were tested for inter-rater reliability using an intraclass correlation coefficient. Paired t-tests were used to determine intrarater reliability. Statistically significant differences among data were accepted if the probability level was equal to or less than 0.01.

Results

There were no significant differences between the first and second scorings of either rater. Inter-rater reliability for scoring all micrographs was $r = 0.75$.

No significant differences in the mean visual scores (Table 1) were found among the 1-min treatment groups. Representative scanning electron micrographs of composite resin surfaces treated in the various
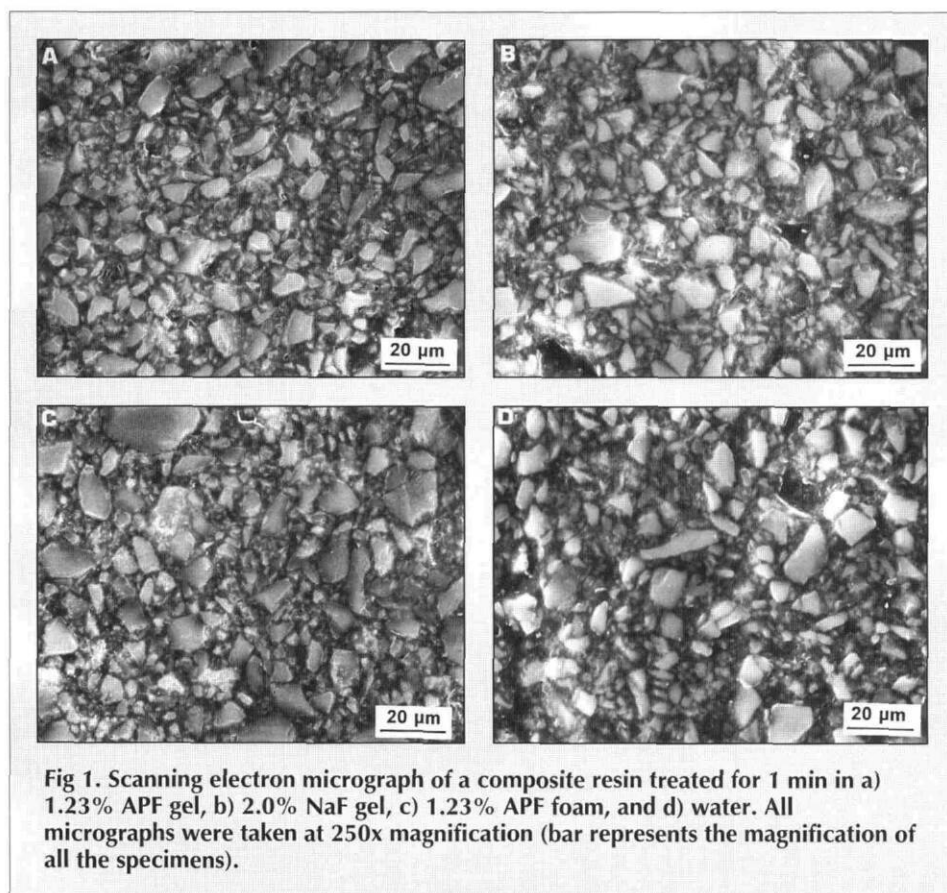

Fig 1. Scanning electron micrograph of a composite resin treated for 1 min in a) 1.23% APF gel, b) 2.0% NaF gel, c) 1.23% APF foam, and d) water. All micrographs were taken at 250x magnification (bar represents the magnification of all the specimens).

TABLE 1. MEAN VISUAL SCORES (x ± SD) OF COMPOSITE RESIN SURFACES				
	Treatment Groups[•]			
No. Immer-sions	1.23% APF Gel	2.0% NaF Gel	1.23% APF Foam	Water
1[†]	10.3 ± 0.6	7.8 ± 1.8	10.2 ± 1.8	8.8 ± 2.4
4[‡]	15.2 ± 0.3	8.8 ± 1.9	13.9 ± 2.3[‡]	8.3 ± 2.6

[•] N = 5 per group.

[†] No significant differences (One-way ANOVA; df = 3; f = 2.33; $P = 0.11$).

[‡] One-way ANOVA; df = 3; f = 29.7; $P = 0.0001$; Tukey's Studentized Range Test (1.23% APF gel = 1.23% APF foam > 2.0% NaF = water).

TABLE 2. MEAN (x ± SD) WEIGHT LOSS (MG) OF COMPOSITE RESIN				
	Treatment Groups[•]			
No. Immer-sions	1.23% APF Gel	2.0% NaF Gel	1.23% APF Foam	Water
1[†]	0.06 ± 0.07	0.02 ± 0.02	0.10 ± 0.03	0.03 ± 0.01
4[‡]	0.38 ± 0.17	0.01 ± 0.02	0.41 ± 0.07	0.01 ± 0.02

[•] N = 5 per group.

[†] No significant differences (One-way ANOVA; df = 3; f = 3.97; $P > 0.01$).

[‡] One-way ANOVA; df = 3; f = 29.7; P 0.0001; Tukey's Studentized Range Test (1.23% APF gel = 1.23% APF foam > 2.0% NaF = water).

agents for 1 min are shown in Figs 1a-d. Voids are seen on all micrographs. The configuration of some voids may be interpreted as areas where large particles were removed from the surface (Figs 1b and d), possibly during the polishing procedure.

Significantly higher mean visual scores (Table 1) were found for the specimens immersed for 4 min in 1.23% APF gel and in 1.23% APF foam compared with specimens immersed in 2.0% NaF gel or in water. There was no significant difference between the mean visual scores of specimens immersed in 2.0% NaF gel or in water. Representative micrographs of each 4-min immersions group are shown in Fig 2a-d.

The surfaces of the specimens immersed in 1.23% APF gel or in 1.23% APF foam have noticeably more voids compared with the other groups. The surfaces of the specimens immersed in 1.23% APF gel (Fig 2a) show more particle loss than the specimens immersed in 1.23% APF foam (Fig 2c). Some particles remaining in the surface of the 1.23% APF-immersed speci-mens show partial loss of structure similar to a cleavage (Fig 2a). The specimens treated with 1.23% APF foam (Fig 2c) show primarily a loss of material at the interface of larger particles with loss of many small filler particles.

There was no statisti-cally significant weight change among the 1-min specimens (Table 2). How-ever, significantly more weight loss occurred in speci-mens following 4-min immersions in either 1.23% APF gel or foam than in specimens immersed in 2.0% NaF gel or water. There were no significant differences in weight loss between specimens immersed in 1.23% APF gel or 1.23% APF foam or between specimens im-mersed in 2.0% NaF or water following 4-min immer-sions. Specimens immersed in either 1.23% APF gel or foam showed progressively greater weight loss with each additional immersion (Fig 3).
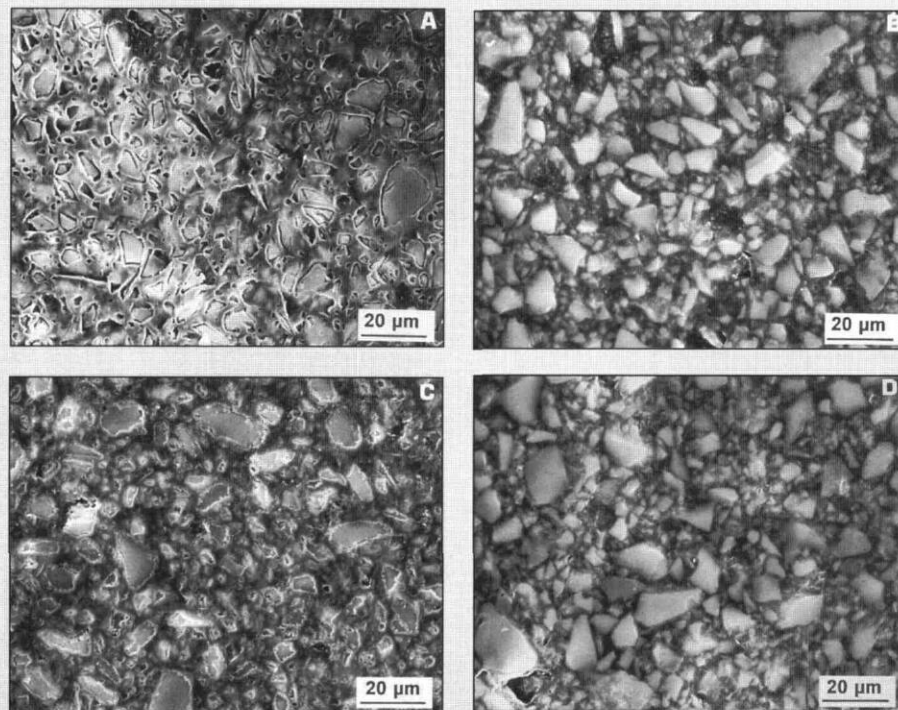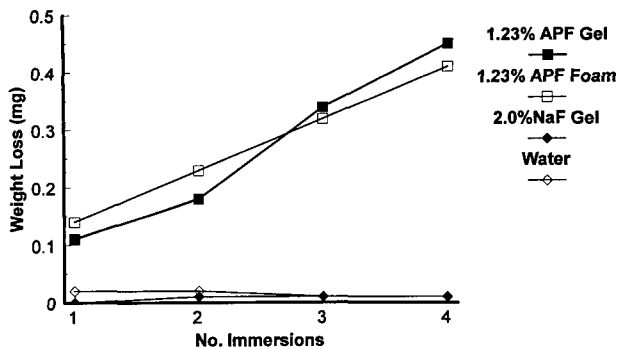

Fig 2. Scanning electron micrograph of a composite resin treated for 4 min in a) 1.23% APF gel, b) 2.0% NaF gel, c) 1.23% APF foam, and d) water. All micrographs were taken at 250x magnification (the bar represents the magnification of all the specimens).

3. Cumulative weight loss of composite resin following immersions.

Discussion

Our study shows that a 1-min treatment with either a 1.23% APF gel or foam does not result in significant visual changes or weight losses in the surface of a composite resin with barium boroaluminosilicate glass and silica filler particles. However, visual scores and weight loss both are significantly higher in composite resins treated with either the 1.23% gel or foam for 4 min. Weight losses also were reported[2] in a strontium-glass-filled composite resin treated 4-min with 1.23% APF gel, as compared with the group treated with water, although the differences were not evaluated statistically. The series of 4-min immersions was selected so that the total time would be similar to a standard 4-min treatment. A series of 1-min immersions was more appropriate than one 4-min immersion to simulate a clinical situation where these APF agents are used as originally formulated and marketed. In addition, the 4-min immersions allowed a longitudinal series of weight changes on the same set of specimens.

The visual scores show that changes in composite resin surfaces increase with increased exposure to APF agents. In addition, increased weight loss occurs with increased immersion in APF gel or foam (Fig 3). These findings are consistent with those of Kula et al.[19] who report increases in weight loss of another posterior composite resin containing barium boroaluminosilicate glass and silicate filler particles with increased exposure to APF products. However, those authors reported weight changes occurring over 10 immersions of 6 min each. Our study shows that APF agents can cause increasing changes over the first 4 min of immersion.

The most obvious area of deterioration following APF immersion is the interface between the filler particle and the resin as seen in the micrographs. After 4-min immersions in APF foam, the smaller particles appear missing as compared with the specimens immersed in water or 2.0% NaF gel. However, nonuniform loss of areas on some of the larger filler particles in specimens immersed in 1.23% APF gel suggest that the particles may have distinct, susceptible phases within a nonhomogeneous glass. Variation in the extent and appearance of surface loss of filler particles with differ-

ent compositions was reported by Kula et al.[3] The loss of filler particles would help explain the weight loss by specimens immersed in APF agents. The weight loss and apparent microscopic voids in this composite resin are consistent with other reports.[1-5]

Sposetti et al.[12] suggested that silicon dioxide which is a component of glass is susceptible to hydrofluoric acid and indicated that the reaction may occur as follows:

$$12HF + 3SiO_2 = 2H_2SiF_6 + Si(OH)_4 + 2H_2O$$
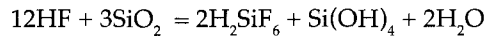
However, the resin-particle interface also may be damaged by fluoride ions. Bowen and Cleek[17] suggest that fluoride may cause depolymerization of the matrix-particle interface. However, no studies have addressed this issue. The micrographs of the specimens treated with APF foam show surface loss at the resin-particle interface. However, the treatment agents were not tested to determine the materials released from the composite resins.

The Council of Dental Materials, Instruments, and Equipment and the Council on Dental Therapeutics[16] recommended that nonacidic fluoride preparations effective in limiting caries be considered as alternatives for patients with composite restorations when fluoride treatment is indicated. Only a few reports exist[4, 5] on topical neutral sodium fluorides causing changes in surface or weight of dental materials.

The lower specific gravity of the APF foam compared with the gel means that the same unit volume of 1.23% APF foam contains less hydrogen ion and fluoride ion than the 1.23% APF gel. Since the visual scores and weight loss are similar between the two agents, it appears that the interaction of these ions with the composite resin surface is not rate-limiting in the foam. Recent studies[18, 19] show fluoride uptake from APF foam into enamel equals or surpasses fluoride uptake from APF gel. Thus, the fluoride ion concentration required for enamel uptake or caries reduction may be lower for a foam. A lower fluoride concentration in the foam may minimize the effect on the composite resins, although 0.5% APF gel, which is less acidic and has a lower concentration of fluoride than 1.23% APF gel also is reported to cause surface damage to dental restorative materials.[4, 5]

Although many composite resins[1-5] are susceptible to weight or surface loss following APF treatment, the amount of visual and weight changes found in this study cannot be extrapolated to all composite resins. Kula et al.[3] report that visual scores of composite resins following immersion in 1.23% APF vary depending on the type of filler particles present in the composite resin. In addition, the pattern of surface loss varies among the composite resins.[3] Some products show complete loss of filler particles after immersion in 1.23% APF gel, whereas others show various patterns of partial surface loss of the filler particle suggesting nonhomogeneous glass phases. The role of the interface between the resin and filler particle in surface loss is not known because of the

extensive loss of filler particles in composite resins with barium glass filler particles.

The preponderance of in vitro studies suggests that 1.23% APF agents should be used with caution with patients who have composite resin restorations, although in vivo studies are needed to determine the effects of 1.23% APF foam and 1.23% APF gel on restorative materials.[20] Composite resin surface loss increases with increased exposure to APF agents. Additional studies also are required to determine whether the type of filler particle or variations in the interface between the filler particle and the resin cause differences in susceptibility of the composite resins to APF agents.

Conclusions

1. There were no significant differences in the mean visual scores or weight changes among composite resin specimens with barium boroaluminosilicate glass and silicate filler particles immersed 1 min in either 1.23% APF foam, 1.23% APF gel, 2.0% NaF gel, or water.

2. Composite resins treated for 4-min immersions in either 1.23% APF gel or foam had significantly greater mean visual scores and weight loss than did specimens treated in 2.0% NaF gel or water.

3. Weight loss of composite resins with barium boroaluminosilicate glass and silicate filler particles is progressive with exposure time.

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