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Surface Properties and Color Stability of Resin-Infiltrated Enamel Lesions

X Zhao • Y-F Ren

Clinical Relevance

Resin infiltration increases surface hardness of white spot lesions and remains stable under thermocycling challenges, but its surface polish and color stability may be of concern when used in the esthetic zone.

SUMMARY

Objectives: To examine the surface topographies, microhardness, and color stability of resin-infiltrated enamel lesions before and after aging challenges *in vitro* using three-dimensional laser scanning profilometry, surface microhardness testing, spectrophotometry, and scanning electron microscopy.

Methods: Forty human third molars were embedded in epoxy resin, and each tooth was prepared to have two white spot lesions and one sound enamel area. One white spot lesion received resin infiltration and the other was untreated. Ten specimens were subjected to thermocycling for 10,000 cycles, 10 specimens were immersed in coffee solutions, and 10 specimens were placed in water storage. Surface area roughness (Sa), Vickers microhardness (VHN), and CIE L*a*b* color values were measured on sound enamel, resin-infiltrated lesions, and untreated lesions before and after aging. The surface morphology of resin-infiltrated lesions was observed after aging under scanning electron microscopy and compared with 10 specimens that were not subjected to aging challenge.

Results: Resin infiltration increased the surface microhardness of the enamel lesions from 89.3 to 212.0 VHN. The surface microhardness of resin-infiltrated enamel lesions was not significantly affected by aging. The surface roughness of resin-infiltrated lesions (0.32–0.37 μm) was greater than that of sound enamel (0.05–0.06 μm) and untreated lesions (0.12–0.13 μm). Thermocycling and water storage further increased surface roughness of resin-infiltrated surfaces. Resin-infiltrated enamel lesions showed greater discoloration than sound enamel surfaces. Surface microfissures and microcracks were observed on resin-infiltrated enamel lesions after thermocycling.

Conclusions: Surface hardness of enamel lesions increased significantly after resin infiltration and remained stable following thermocycling. Surface roughness and color

Xiaoyi Zhao, DDS, PhD, Department of General Dentistry, Peking University School of Stomatology, Beijing, China; and Department of General Dentistry, University of Rochester Eastman Institute for Oral Health, Rochester, NY, USA

*Yan-Fang Ren, DDS, PhD, MPH, Department of General Dentistry, University of Rochester Eastman Institute for Oral Health, Rochester, NY, USA.

*Corresponding author: 625 Elmwood Ave, Rochester, NY 14620; e-mail: Yanfang_ren@urmc.rochester.edu

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stability of resin-infiltrated enamel lesions were less than ideal and might further deteriorate after aging in the oral environment.

INTRODUCTION

Although frequent application of fluoride is often recommended as the treatment of choice for initial enamel caries on smooth or proximal surfaces, the effectiveness of this approach depends strongly on the patient's oral hygiene practice and is therefore not suitable for noncompliant patients. An alternative therapy to arrest initial caries lesions is infiltration of the pores and microspaces within enamel lesions using a low-viscosity liquid resin.[1] It has been shown that artificial and natural caries lesions on smooth, interproximal, and occlusal surfaces can be successfully infiltrated using this microinvasive technique.[2-4]

In comparison to fluoride therapy, resin infiltration was found to result in greater surface hardness of enamel carious lesions.[5] Resin infiltration was effective, at least in the short-term, in arresting both the smooth surface and interproximal surface enamel lesions in randomized and controlled clinical trials,[6-9] and the procedure was well received by both the practitioners and the patients.[10] As this technique is relatively new, there is a lack of data on its long-term outcomes. In a recent study that followed 45 patients for a period of 12 months, the infiltrated surface showed excellent marginal adaptation but significant discoloration.[11] This finding was further substantiated by an experiment *in vitro*, which found that infiltration resin had the highest staining susceptibility as compared with several resin-based dental bonding and adhesive materials.[12]

As smooth surface white spot lesions are often present in the esthetic zone, color stability of resin-infiltrated surfaces is an important determinant for long-term success. It is generally accepted that all resin-based dental materials degrade to some extent in the oral environment. Water sorption and surface degradation are considered factors associated with discoloration of resin-based dental materials.[13,14] In addition, polishability and surface roughness contribute significantly to color stability and discoloration of this type of material.[15-17]

Although discoloration of infiltration resin has been reported in the aforementioned studies *in vivo* and *in vitro*, the mechanism underlying the color change remains unclear. In contrast to conventional resin-based restorative materials, currently available infiltration resin is composed mainly of hydrophilic triethyleneglycol-dimethacrylate (TEGDMA). Infiltration resin has two major differences from the other resin-based materials: it is an unfilled liquid resin composed of mostly TEGDMA and it does not have a polishing step after its application per the manufacturer's instructions. TEGDMA is important for maintaining the extremely low viscosity that allows penetration of the resin to the demineralized lesion. But it is well known that TEGDMA has a high water sorption rate and is prone to discoloration,[18,19] and a nonpolished surface may mean a rougher surface.[20] Aging challenges under thermal stress might further affect the physical property and color stability of resin-infiltrated enamel surfaces and compromise the long-term outcomes. The aim of the present study was to evaluate the influence of aging challenge on the physical properties and color stability of resin-infiltrated enamel surfaces in vitro.

METHODS AND MATERIALS

A total of 40 freshly extracted permanent third molars were collected from oral surgery and general dentistry clinics following ethic guidelines from the authors' institution. The teeth were cleaned from soft tissues and stored in a refrigerator in 0.1% thymol solution for no more than two months before use.

Sample Preparation

The teeth were sectioned at the cemento-enamel junction using a high-speed handpiece (TF12, Mani, Inc., Tochigi, Japan) with water coolant. The crowns were placed in cylindrical plastic molds (20 mm in diameter and 20 mm in height) with cusps facing down on a flat surface and embedded in epoxy resin. The occlusal surfaces of the embedded crowns were ground flat with 400-grit waterproof SiC paper (Softflex, Matador GmbH, Remscheid, Germany) under water cooling until at least three flat areas of the enamel, each measuring at least 2 mm × 2 mm, were exposed. The exposed enamel areas were typically the two buccal cusps and one lingual cusp of the molar crown (Figure 1a,b). Exposed enamel surfaces were then polished in sequence with 800-, 1200-, 2400-, and 4000-grit waterproof SiC paper using running tap water as a coolant. The prepared specimens were examined under a stereomicroscope to verify that the enamel surfaces were exposed, with absence of cracks or other surface defects. After preparation, the specimens were stored in 0.1% thymol solution to avoid dehydration.

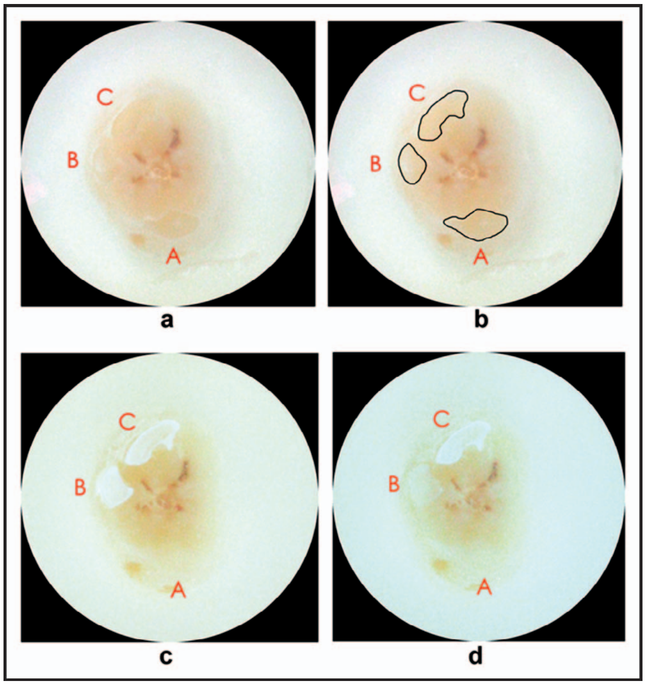

Figure 1. *Sample preparation. (a): Occlusal surface of human third molars were embedded in epoxy resin and ground flat to expose three areas (A, B, and C) of at least 2 mm × 2 mm enamel. (b): The exposed enamel areas were typically the two buccal cusps and one lingual cusp of the molar crown. (c): Two separate white spot lesions (A and B) were created. (d): One white spot lesion B was treated with the ICON infiltration resin and one lesion was untreated.*

Artificial Enamel Caries Lesions

Of the three exposed enamel surface areas, one area was covered with acid-resistant nail polish to serve as a sound enamel control. The other two areas were left exposed. Artificial lesions were created within the two exposed enamel areas by immersing each tooth into a 50-mL aliquot of a Ca/PO_4/acetate solution containing 2.0 mmol/L calcium, 2.0 mmol/L phosphate, and 0.075 mol/L acetate maintained at pH 4.5 and a temperature of 37°C for 48 hours. The artificial white spot lesions created presented depths between 100 and 150 μm and exhibited optical properties of early-stage caries lesions.[21]

The artificial caries model prepared this way has two separate artificial white spot lesions on the same specimen and one sound enamel area as internal control (Figure 1c). The white spot lesion was identified as an opaque and chalky white area on the enamel surface in contrast to the semi-translucent sound enamel.[22] Each specimen was inspected visually to ensure that the white spot lesions were successfully created on the enamel surfaces.

Resin Infiltration of Enamel Surface Lesions

Of the two lesions on the enamel surface of the specimen, one was randomly chosen to receive the ICON (ICON DMG, Hamburg, Germany) resin infiltration treatment, while the other served as the control. A computerized simple randomization scheme was used to select the lesion for treatment (see Supplementary Materials). The resin infiltration treatment followed a protocol described in detail in a previous report (Figure 1d).[23] Briefly, the selected lesion was etched with 37% phosphoric acid gel (Gluma Etch 35 Gel, Heraeus Kulzer GmbH, Hanau, Germany) for five seconds, rinsed with air-water spray for 30 seconds, and air dried for 10 seconds. Pure ethanol was then applied to the lesion surface for 10 seconds, followed by air drying for another 10 seconds to render surface desiccation. The ICON infiltration resin was applied to the enamel caries lesions for three minutes, and resin excess was removed with a cotton roll. The resin-infiltrated surface was then light-cured for 40 seconds. The infiltration resin was applied for a second time as above for one minute and light cured for another 40 seconds. After light curing, the resin surface was polished with 4000-grit aluminum oxide abrasive paper for 20 seconds.

Thermocycling, Staining Challenge, and Water Storage

Following resin infiltration treatments, the specimens were randomly divided into the following four groups using a random list generator: 10 specimens for thermocylcing challenges, 10 specimens for staining challenges in coffee at 37°C, 10 specimens for water storage at 37°C as a control, and 10 specimens for scanning electron microscopy (SEM) to obtain baseline surface morphology data on resin-infiltrated enamel lesions.

For the thermocycling group, the specimens were placed in a thermocycling machine programmed to perform 10,000 cycles in 180 hours (7 days 12 hours) between two water baths at temperatures of 5°C and 55°C, respectively, with a dwell time of 30 seconds at each bath temperature.

For the staining challenge group, the specimens were immersed in coffee solution prepared with 75 g instant coffee (Nescafé, Nestlé, Vevey, Switzerland) in 750 mL boiling water and stored at 37°C for 180 hours. The coffee solution was refreshed every day for seven days.

For the water storage group, the specimens were stored in distilled water at 37°C for 180 hours, the same duration as the thermocycling and the staining challenge group.

3D Laser Scanning Microscopy

Specimens in the thermocycling and water storage group were subjected to surface roughness testing using a three-dimensional (3D) laser scanning microscope (VK-X100/X200, Keyence, Osaka, Japan) at 3000× magnification before and after the aging challenges. For each area on each specimen, three different locations (95.7993 × 71.8495 μm^2 in size) were randomly chosen and scanned for 3D surface area profiling. Surface roughness was measured in average arithmetic roughness (Sa) values. The mean values of the three measurements for each area were used as the Sa value.

Surface Microhardness Testing

Surface microhardness of sound enamel, resin-infiltrated lesions, and untreated lesions on each specimen in the thermocycling and water storage group was determined using a Shimadzu microhardness tester (HMV-2T, Shimadzu Corporation, Kyoto, Japan) with a Vickers diamond indenter. Three microhardness indentations were performed with 25-g load and 10 seconds dwell time on each area. The Vickers microhardness (VHN) value for each specimen was measured before and after thermocycling or water storage. The mean values of the three indentations for each area were used as the VHN value.

Spectrophotometry

Specimens in the staining challenge group were subjected to color measurement using an Olympus CrystalEye Spectrophotometer (Olympus, Tokyo, Japan) before and after coffee storage. For each area on the specimen, three different sites were measured according to the CIE L*a*b* system, and the mean values were used as the color of the area.

Scanning Electron Microscopy

To qualitatively assess the surface morphology of resin-infiltrated lesions before aging challenges, 10 specimens were examined with an SEM (BCPCAS4800, JEOL, Tokyo, Japan) immediately following infiltration resin treatment. All specimens were coated with a gold layer approximately 10-nm thick before examination, and the scanning was operated at 4000× and 20,000× magnifications with an accelerating voltage of 1.5 kV. All 20 specimens in the thermocycling and the water storage groups were also scanned in the same manner to qualitatively assess the surface morphology of the resin-infiltrated lesions after the 180-hour aging challenges.

Statistical Analysis

The primary outcome measures of the present study were the changes in the surface roughness of the resin-infiltrated enamel surfaces following thermocycling challenges. Our pilot testing showed that the surface roughness of enamel lesions treated with the ICON infiltrant resin was approximately 0.4 with a standard deviation of approximately 0.05. We considered that a 20% difference in surface roughness was clinically significant as it correlated to an increase of ΔE from 2.9 to 3.5.[24] It has been shown that a ΔE smaller than 3.3 is not clinically significant.[25,26] Based on the effect size of 20% difference in surface roughness, we needed nine samples in each group to achieve 90% power at an alpha level of 0.05. We decided to use 10 specimens in each group. Surface roughness, microhardness, and coffee staining data were compared among the three areas (untreated lesions, resin-infiltrated lesions, and sound enamel) using analysis of variance and the post hoc Fisher PLSD test. The surface roughness and microhardness data were compared before and after thermocycling or water storage using paired t tests with Bonferroni corrections. Data were analyzed using StatView (SAS Institute Inc., Cary, NC, USA) for two-tailed tests, and a P value smaller than 0.05 was considered statistically significant.

RESULTS

Surface Roughness

Representative 3D laser scanning images of sound enamel, resin-infiltrated lesions, and untreated lesions are shown in Figure 2. Surface roughness data before and after thermocycling or water storage are listed in Tables 1 and 2. Before aging challenges, the surface roughness of the untreated enamel lesions was greater than that of the sound enamel but less than that of resin-infiltrated lesions ($p<0.01$; Figure 2; Tables 1 and 2), indicating resin infiltration increased the surface roughness of the enamel lesions. Thermocycling and water storage caused a further increase in surface roughness of the resin-infiltrated surfaces ($p<0.01$) but did not have a significant impact on the sound enamel and untreated lesion surfaces ($p>0.05$; Figure 2; Tables 1 and 2). The average surface roughness (Sa) of the resin-infiltrated surface increased from 0.373 µm to 0.621

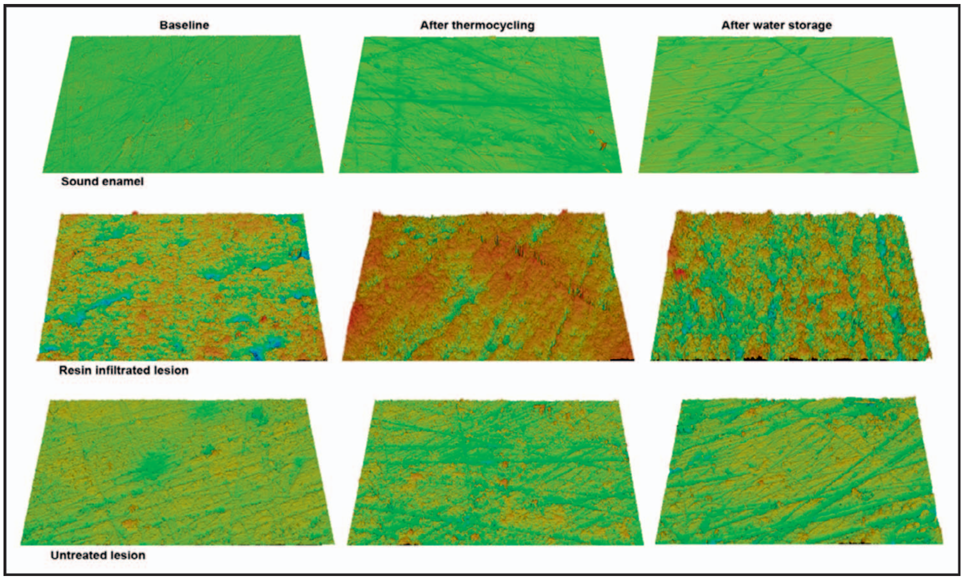

Figure 2. *Three-dimensional laser scanning images of sound enamel (top row), resin-infiltrated lesions (middle row), and untreated lesions (bottom row) at baseline (left column), after thermocycling (center column), and after water storage (right column). Surface roughness of resin-infiltrated lesions are greater than sound enamel and untreated lesions and further increased after thermocycling and water storage.*

μm after thermocycling for 180 hours. Water storage had a similar effect on the resin-infiltrated surfaces but was smaller in magnitude (from 0.317 μm to 0.472 μm on average).

Surface Microhardness

Surface microhardness measured in VHN before and after thermocycling or water storage is listed in Tables 3 and 4. Compared with that of sound enamel, the mean surface microhardness of the artificial carious lesion was reduced by more than threefold. Resin infiltration of the enamel lesions increased the surface microhardness by more than twofold ($p<0.05$; Tables 3 and 4). Surface microhardness of resin-infiltrated enamel lesions decreased slightly after thermocycling and water storage, but this change did not reach the *a priori* level of statistical significance ($p>0.05$).

Surface Staining

The overall color change of the specimens after coffee storage for all experimental sites is shown in Table 5. Color changes happened to all experimental sites (sound enamel, resin-infiltrated lesions, and untreated lesions). Resin-infiltrated surfaces showed significantly higher color alteration ($\Delta E=12.7\pm4.7$) than sound enamel ($\Delta E=4.3\pm0.8$) but much less than untreated lesions ($\Delta E=31.1\pm4.4$; $p<0.05$). Analysis of L*a*b* values showed that color change of resin-infiltrated lesions was largely due to decreased lightness (ΔL; $p<0.05$), while changes in Δa and Δb were not statistically significant as compared with the sound enamel ($p>0.05$). Untreated lesions showed significant increases in Δa and Δb values ($p<0.05$) in addition to decreased lightness.

Surface Morphology Under SEM

As shown in Figure 3, resin-infiltrated surfaces were largely intact and uniform before aging challenges (Figure 3a). Surface microcracks and microfissures appeared on resin-infiltrated lesions following thermocycling challenges (Figure 3b). Minor changes could also be observed following water storage but were much less distinct than those after thermocycling (Figure 3c). At high magnification (20,000×), hydroxyapatite crystals were shown to be embedded in the resin matrix to form a relatively uniform and intact surface (Figure 3d). After aging challenges, microfissures and microcracks could be observed on

Table 1: *Effect of Thermocycling on Surface Roughness (Sa) of Resin-Infiltrated Enamel Lesions (Mean ± SD)*

	Before Thermocycling[a]	After Thermocycling[a]	Paired *t* Test
Sound enamel	0.059±0.004a	0.063±0.006a	NS
Resin-infiltrated lesion	0.373±0.022b	0.621±0.035b	<0.01
Untreated lesion	0.129±0.041c	0.142±0.037c	NS
Analysis of variance	<0.01	<0.01	

[a] *Different letters in the same column denote statistically significant differences between each other with analysis of variance post hoc tests. NS, not significant.*

Table 2: *Effect of Water Storage on Surface Roughness (Sa) of Resin-Infiltrated Enamel Lesions (Mean ± SD)*[a]

	Before Water Storage	After Water Storage	Paired *t* Test
Sound enamel	0.049±0.008a	0.053±0.011a	NS
Resin infiltrated lesion	0.317±0.042b	0.472±0.114b	<0.01
Untreated lesion	0.122±0.039c	0.134±0.031c	NS
Analysis of variance	<0.01	<0.01	

[a] *Different letters in the same column denote statistically significant differences between each other with analysis of variance post hoc tests. NS, not significant.*

the surfaces, especially after thermocycling (Figure 3e,f).

DISCUSSION

The findings of the present study indicate that resin infiltration significantly increases the surface microhardness of enamel lesions and remains stable after thermocycling challenges. However, surface properties of resin-infiltrated enamel lesions may deteriorate with time in the oral environment and result in an increase in surface roughness and discoloration. Microcracks may appear on the resin-infiltrated surfaces after thermocycling challenges, which may further render the surface vulnerable to staining and discoloration.

Water sorption and surface degradation caused by thermocycling may affect the mechanical properties of resin-based dental materials. Surface microhardness of many resin composites decreased following thermocycling challenges.[27,28] Plasticization of the resin matrix by water sorption and hydrolytic breakdown of the resin–filler interface were considered to be the causes of the reduced surface hardness of resin composite materials.[28] However, the surface microhardness of resin-infiltrated enamel lesions was not significantly altered following thermocycling in the present study. Although the TEGDMA-based infiltration resin is unfilled and prone to water sorption and matrix degradation, the microhardness of the resin-infiltrated surfaces found in the present study and other studies ranged from 150 to 240 VHN (median 185 VHN)[5,20,29] which is significantly higher than most of the highly filled resin composites with a range from 40 to 150 VHN and a median of 72 VHN.[28,30] Such high surface hardness is obviously not a function of the resin matrix as polymerized TEGDMA is the softest (26 VHN) among the resin polymers used in dental restorative materials.[31] The infiltration resin was designed to penetrate the porous lesions left after acid etching and to fill the voids and spaces of the demineralized zone in a white spot lesion, thus preventing further demineralization and lesion progression.[32,33] It appears that the infiltration resin was able to encapsulate the hydroxyapatite crystals in the white spot lesion and form a relatively uniform resin-hydroxyapatite complex (Figure 3a,d) that exhibits high surface hardness. Although microcracks and microfissures may occur on the surface of the resin–hydroxyapatite complex, its surface hardness remained stable following thermocycling challenges. A stable resin–hydroxyapatite complex may be the foundation for clinical success of the resin infiltration technology.

Surface roughness of resin-infiltrated carious lesions was reported to be as high as 6.9 μm on average using the ICON infiltration resin,[20] which is considerably higher than the 0.2 μm threshold generally regarded as acceptable for a restorative material to resist plaque accumulation.[34-36] We found that the average surface roughness of resin-infiltrated areas was approximately 0.32 μm to 0.37 μm immediately after treatments, which was generally in agreement with that of Mueller and others.[36] After thermocycling challenge at temperatures between 5°C and 55°C for 10,000 cycles, simulating one-year of clinical service,[37] the surface roughness of resin-infiltrated lesions further deteriorated to 0.62 μm on average (Table 2; Figure 2), signifying a 70% increase compared with baseline. Repeated temperature fluctuations in the oral cavity may

Table 3: *Effect of Thermocycling on Surface Microhardness (VHN) of Resin-Infiltrated Enamel Lesions (Mean ± SD)*[a]

	Before Thermocycling	After Thermocycling	Paired *t* Test
Sound enamel	315.2±31.9a	320.2±30.7a	NS
Resin-infiltrated lesion	212.0±45.6b	202.9±55.2b	NS
Untreated lesion	89.3±24.1c	93.4±29.5c	NS
Analysis of variance	<0.01	<0.01	

[a] *Different letters in the same column denote statistically significant differences between each other with analysis of variance post hoc tests. NS, not significant.*

Table 4: *Effect of Water Storage on Surface Microhardness (VHN) of Resin-Infiltrated Enamel Lesions (mean ± SD)*[a]

	Before Water Storage	After Water Storage	Paired *t* Test
Sound enamel	313.2±25.5a	312.3±17.5a	NS
Resin-infiltrated lesion	219.1±25.2b	209.6±35.6b	NS
Untreated lesion	93.8±25.6c	90.2±23.0c	NS
Analysis of variance	<0.01	<0.01	

[a] *Different letters in the same column denote statistically significant differences between each other with analysis of variance post hoc tests. NS, not significant.*

induce degradation of resin-hydoxyapatite bonds due to differences in thermal expansion coefficients between enamel hydroxyapatite and the infiltration resin.[38,39] Thermal stress may also affect the surface integrity of resin-infiltrated enamel lesions as indicated by the presence of surface microcracks and microfissures after thermocycling challenges (Figure 3). These changes in surface properties may contribute to staining and discoloration of resin-infiltrated surfaces. The findings of the present study are in agreement with recent reports that ICON resin-infiltrated carious lesions were prone to discoloration under staining challenges.[40,41]

Discoloration of resin-based restorative materials may arrive from intrinsic and/or extrinsic stains. Intrinsic stain is associated with the properties of the polymeric networks such as water sorption and the presence of unreacted methacrylate in the resin matrix, while extrinsic stain is caused by external colorants such as those in beverages and foods.[42-44] The ICON infiltration resin is primarily a TEGDMA-based polymer with high penetration efficiencies.[45,46] Compared with other resin polymers commonly used in dental materials, such as UDMA and Bis-GMA, TEGDMA has the highest degree of water sorption owing to the presence of hydrophilic ether linkages.[19,42,47] A high degree of water sorption has long been linked to color stability issues and discoloration of resin-based dental materials.[13,14,18] On the other hand, surface roughness was recognized as the most important extrinsic factor for discoloration of resin-based dental materials.[15-17] Most modern resin composite materials for esthetic restorations could achieve a high glossy finish with surface roughness below the acceptable threshold of 0.2 μm after finishing and polishing.[35] Such a high degree of polishability appears to be difficult to achieve with the infiltration resin, as its surface roughness was not improved even after polishing with the Sof-Lex finishing and polishing system.[36] Therefore, the mechanisms underlying the discoloration of infiltration resin are likely twofold: one is intrinsically associated with its primary constituent TEGDMA, which has a high degree of water sorption, and the other is extrinsically related to a less than ideal surface polish that deteriorates with time in the oral cavity. To ensure long-term success, further research is warranted to improve surface polish and esthetic outcomes of resin infiltration, especially when smooth surface white spot lesions are involved in the esthetic zone.

As TEGDMA is prone to water sorption and may absorb twice as much water as compared with Bis-GMA, TEGDMA-based materials may be more susceptible to degradation than Bis-GMA- or UDMA-based materials.[42,48,49] The presence of water in the resin matrix may increase internal stress, leading to microcracking.[50] It was also shown that water sorption by TEGDMA increases with the elevation of temperatures,[49] which in combination with the thermal expansion and contraction effects of temperature fluctuation may further affect the integrity of the TEGDMA-based infiltration resin. The surface microcracks and microfissures observed on the resin-infiltrated lesions in the present study may be a result of such internal and thermal stresses due to water sorption and thermal expansion and contraction.

Table 5: *Effect of Staining Challenge on Surface Color Change of Resin-Infiltrated Enamel Lesions (Mean±SD)*

	ΔL	Δa	Δb	ΔE
Sound enamel	−3.0±0.8a	0.7±0.4a	2.9±0.7a	4.3±0.8a
Resin-infiltrated lesion	−9.7±3.5b	1.7±1.7a	6.4±5.8a	12.7±4.7b
Untreated lesion	−25.3±4.9c	5.5±2.6c	16.2±5.1c	31.1±4.4c
Analysis of variance	<0.01	<0.01	<0.01	<0.01

[a] *Different letters in the same column denote statistically significant differences with analysis of variance post hoc tests.*

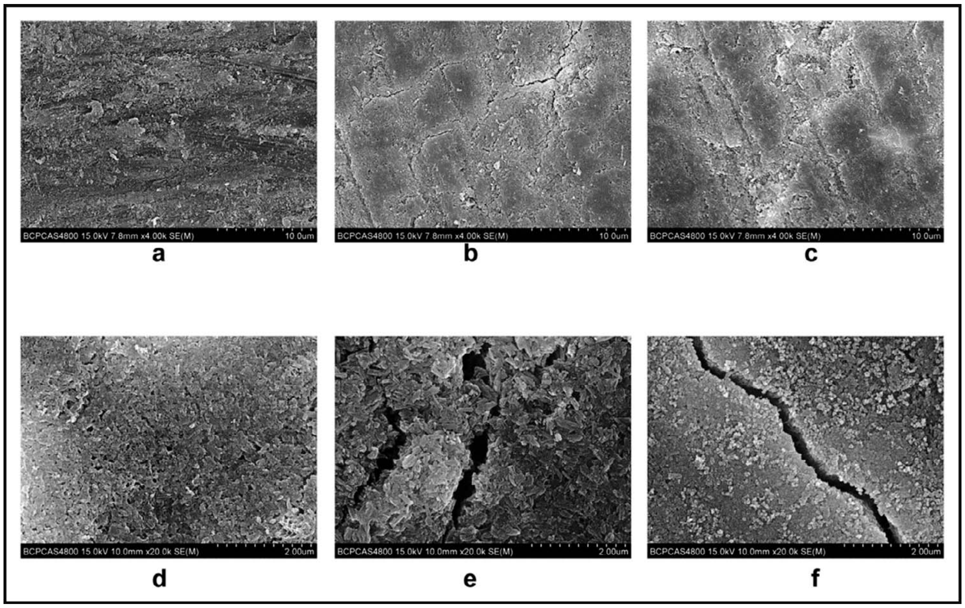

Figure 3. *Scanning electron microscopy images of resin-infiltrated lesions. Top row, at 4000×: (a): immediately after resin infiltration; (b): after thermocycling; (c): after water storage. Bottom row, at 20,000×: (d): immediately after resin infiltration; e: microfissures after thermocycling; f: microcracks after thermocycling.*

CONCLUSION

Within the limitations of this study, we conclude that the surface hardness of resin-infiltrated enamel lesions was high and remained stable following the thermocycling challenges. Surface roughness and color stability of resin-infiltrated enamel lesions was less than ideal and might further deteriorate after aging in the oral environment. Surface microcracks and microfissures could occur on the surface of the resin-hydroxyapatite complex after aging challenges. These changes may render the resin-infiltrated areas susceptible to discoloration.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Rochester.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Paris S, Meyer-Lueckel H, Colfen H, & Kielbassa AM (2007) Resin infiltration of artificial enamel caries lesions with experimental light curing resins *Dental Materials Journal* **26(4)** 582-588.
2. Ekstrand KR, Bakhshandeh A, & Martignon S (2010) Treatment of proximal superficial caries lesions on primary molar teeth with resin infiltration and fluoride varnish versus fluoride varnish only: efficacy after 1 year *Caries Research* **44(1)** 41-46.
3. Meyer-Lueckel H. & Paris S (2008) Progression of artificial enamel caries lesions after infiltration with experimental light curing resins. *Caries Research* **42(2)** 117-124.
4. Lausch J, Paris S, Selje T, Dorfer CE, & Meyer-Lueckel H (2015) Resin infiltration of fissure caries with various techniques of pretreatment in vitro *Caries Research* **49(1)** 50-55.
5. Torres CR, Rosa PC, Ferreira NS, & Borges AB (2012) Effect of caries infiltration technique and fluoride therapy on microhardness of enamel carious lesions *Operative Dentistry* **37(4)** 363-369.
6. Senestraro SV, Crowe JJ, Wang M, Vo A, Huang G, Ferracane J, & Covell DA Jr (2013) Minimally invasive resin infiltration of arrested white-spot lesions: a randomized clinical trial *Journal of the Americal Dental Association* **144(9)** 997-1005.
7. Martignon S, Ekstrand KR, Gomez J, Lara JS, & Cortes A (2012) Infiltrating/sealing proximal caries lesions: a 3-year randomized clinical trial *Journal of Dental Research* **91(3)** 288-292.
8. Paris S, Bitter K, Naumann M, Dorfer CE, & Meyer-Lueckel H (2011) Resin infiltration of proximal caries lesions differing in ICDAS codes *European Journal of Oral Sciences* **119(2)** 182-186.
9. Paris S, Hopfenmuller W, & Meyer-Lueckel H (2010) Resin infiltration of caries lesions: an efficacy randomized trial *Journal of Dental Research* **89(8)** 823-826.

10. Altarabulsi MB, Alkilzy M, & Splieth CH (2013) Clinical applicability of resin infiltration for proximal caries *Quintessence International* **44(2)** 97-104.

11. Altarabulsi MB, Alkilzy M, Petrou MA, & Splieth C (2014) Clinical safety, quality and effect of resin infiltration for proximal caries *European Journal of Paediatric Dentistry* **15(1)** 39-44.

12. Rey N, Benbachir N, Bortolotto T, & Krejci I (2014) Evaluation of the staining potential of a caries infiltrant in comparison to other products *Dental Materials Journal* **33(1)** 86-91.

13. Arocha MA, Mayoral JR, Lefever D, Mercade M, Basilio J, & Roig M (2013) Color stability of siloranes versus methacrylate-based composites after immersion in staining solutions *Clinical Oral Investigations* **17(6)** 1481-1487.

14. Nasim I, Neelakantan P, Sujeer R, & Subbarao CV (2010) Color stability of microfilled, microhybrid and nanocomposite resins—an in vitro study *Journal of Dentistry* **38(Supplement 2)** e137-e142.

15. Festuccia MS, Garcia Lda F, Cruvinel DR, & Pires-De-Souza Fde C (2012) Color stability, surface roughness and microhardness of composites submitted to mouthrinsing action *Journal of Applied Oral Science* **20(2)** 200-205.

16. Gonulol N, & Yilmaz F (2012) The effects of finishing and polishing techniques on surface roughness and color stability of nanocomposites *Journal of Dentistry* **40(Supplement 2**) e64-e70.

17. Sirin Karaarslan E, Bulbul M, Yildiz E, Secilmis A, Sari F, & Usumez A (2013) Effects of different polishing methods on color stability of resin composites after accelerated aging *Dental Materials Journal* **32(1)** 58-67.

18. Shintani H, Satou N, Yukihiro A, Satou J, Yamane I, Kouzai T, Andou T, Kai M, Hayashihara H, & Inoue T (1985) Water sorption, solubility and staining properties of microfilled resins polished by various methods *Dental Materials Journal* **4(1)** 54-62.

19. Sideridou I, Tserki V, Papanastasiou G (2003) Study of water sorption, solubility and modulus of elasticity of light-cured dimethacrylate-based dental resins *Biomaterials* **24(4)** 655-665.

20. Taher NM, Alkhamis HA, & Dowaidi SM (2012) The influence of resin infiltration system on enamel microhardness and surface roughness: an *in vitro* study *Saudi Dental Journal* **24(2)** 79-84.

21. Wu J, & Fried D (2009) High contrast near-infrared polarized reflectance images of demineralization on tooth buccal and occlusal surfaces at lambda = 1310-nm *Lasers in Surgery and Medicine* **41(3)** 208-213.

22. Bishara SE, & Ostby AW (2008) White spot lesions: formation, prevention, and treatment *Seminars in Orthodontics* **14(3)** 174-182.

23. Neuhaus KW, Schlafer S, Lussi A, & Nyvad B (2013) Infiltration of natural caries lesions in relation to their activity status and acid pretreatment *in vitro Caries Research* **47(3)** 203-210.

24. Gönülol N, & Yılmaz F (2012) The effects of finishing and polishing techniques on surface roughness and color stability of nanocomposites *Journal of Dentistry* **40(Supplement 2)** e64-e70.

25. Turkun LS, & Turkun M (2004) Effect of bleaching and repolishing procedures on coffee and tea stain removal from three anterior composite veneering materials *Journal of Esthetic & Restorative Dentistry* **16(5)** 290-301.

26. Um CM, & Ruyter IE (1991) Staining of resin-based veneering materials with coffee and tea *Quintessence International* **22(5)** 377-386.

27. Baek CJ, Hyun SH, Lee SK, Seol HJ, Kim HI, & Kwon YH (2008) The effects of light intensity and light-curing time on the degree of polymerization of dental composite resins *Dental Materials Journal* **27(4)** 523-533.

28. Tuncer S, Demirci M, Tiryaki M, Unlu N, & Uysal O (2013) The effect of a modeling resin and thermocycling on the surface hardness, roughness, and color of different resin composites *Journal of Esthetic & Restorative Dentistry* **25(6)** 404-419.

29. Paris S, Schwendicke F, Seddig S, Muller WD, Dorfer C, & Meyer-Lueckel H (2013) Micro-hardness and mineral loss of enamel lesions after infiltration with various resins: influence of infiltrant composition and application frequency *in vitro Journal of Dentistry* **41(6)** 543-548.

30. Choudhary S, & Suprabha B (2013) Effectiveness of light emitting diode and halogen light curing units for curing microhybrid and nanocomposites *Journal of Conservative Dentistry* **16(3)** 233-237.

31. Venter SAdS, Fávaro SL, Radovanovic E, & Girotto EM (2013) Hardness and degree of conversion of dental restorative composites based on an organic-inorganic hybrid. *Materials Research* **16** 898-902.

32. Paris S, & Meyer-Lueckel H (20096) Masking of labial enamel white spot lesions by resin infiltration—a clinical report *Quintessence International* **40(9)** 713-718.

33. Paris S, Meyer-Lueckel H, Colfen H, & Kielbassa AM (2007) Penetration coefficients of commercially available and experimental composites intended to infiltrate enamel carious lesions *Dental Materials* **23(6)** 742-748.

34. Bollen CM, Lambrechts P, & Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. *Dental Materials* **13(4)** 258-269.

35. Janus J, Fauxpoint G, Arntz Y, Pelletier H, & Etienne O (2010) Surface roughness and morphology of three nanocomposites after two different polishing treatments by a multitechnique approach *Dental Materials* **26(5)** 416-425.

36. Mueller J, Yang F, Neumann K, & Kielbassa AM (2011) Surface tridimensional topography analysis of materials and finishing procedures after resinous infiltration of subsurface bovine enamel lesions *Quintessence International* **42(2)** 135-147.

37. Gale MS, & Darvell BW (1999) Thermal cycling procedures for laboratory testing of dental restorations *Journal of Dentistry* **27(2)** 89-99.

38. Hahnel S, Henrich A, Burgers R, Handel G, & Rosentritt M (2010) Investigation of mechanical properties of

modern dental composites after artificial aging for one year *Operative Dentistry* **35(4)** 412-419.

39. Borges AF, Santos Jde S, Ramos CM, Ishikiriama SK, & Shinohara MS (2012) Effect of thermo-mechanical load cycling on silorane-based composite restorations. *Dental Materials Journal* **31(6)** 1054-1059.

40. Araujo G, Naufel FS, Alonso R, Lima D, & Puppin-Rontani RM (2015) Influence of staining solution and bleaching on color stability of resin used for caries infiltration *Operative Dentistry* **40(6)** E250-E256.

41. Borges A, Caneppele T, Luz M, Pucci C, & Torres C (2014) Color stability of resin used for caries infiltration after exposure to different staining solutions *Operative Dentistry* **39(4)** 433-440.

42. Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22(3)** 211-222.

43. Barutcigil C, & Yildiz M (2012) Intrinsic and extrinsic discoloration of dimethacrylate and silorane based composites *Journal of Dentistry* **40(Supplement 1)** e57-e63.

44. Ren YF, Feng L, Serban D, & Malmstrom HS (2012) Effects of common beverage colorants on color stability of dental composite resins: the utility of a thermocycling stain challenge model in vitro *Journal of Dentistry* **40(Supplement 1)** e48-e56.

45. Meyer-Lueckel H, Chatzidakis A, Naumann M, Dorfer CE, & Paris S (2011) Influence of application time on penetration of an infiltrant into natural enamel caries *Journal of Dentistry* **39(7)** 465-469.

46. Meyer-Lueckel H, & Paris S (2010) Infiltration of natural caries lesions with experimental resins differing in penetration coefficients and ethanol addition *Caries Research* **44(4)** 408-414.

47. Venz S, & Dickens B (1991) NIR-spectroscopic investigation of water sorption characteristics of dental resins and composites *Journal of Biomedical Materials Research* **25(10)** 1231-1248.

48. Bagheri R, Tyas MJ, & Burrow MF (2007) Subsurface degradation of resin-based composites *Dental Materials* **23(8)** 944-951.

49. Kalachandra S, & Turner DT (1987) Water sorption of polymethacrylate networks: bis-GMA/TEGDM copolymers. *Journal of Biomedical Materials Research* **21(3)** 329-338.

50. Mair LH (1989) Surface permeability and degradation of dental composites resulting from oral temperature changes *Dental Materials* **5(4)** 247-255.