EGYPTIAN
DENTAL JOURNAL
www.eda-egypt.org

Vol. 69, 705:713, January, 2023

Print ISSN 0070-9484 • Online ISSN 2090-2360

Conservative Dentistry and Endodontics

Submit Date : 19-09-2022 • Accept Date : 19-11-2022 • Available online: 1-1-2023 • DOI : 10.21608/edj.2022.162786.2263

# CORRELATION BETWEEN SURFACE ROUGHNESS AND COLOR STABILITY OF NANO- AND MICRO-HYBRID RESIN COMPOSITES USING DIFFERENT SURFACE TREATMENT PROTOCOLS

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## *ABSTRACT*

**Objectives:** To compare in vitro the color stability of one nanohybrid and one microhybrid composite restorative materials and correlate with surface roughness following different surface treat**ment protocols.**

**Materials and Methods:** 150 specimens of each composite type were fabricated. The specimens were divided into five groups according to finishing/polishing technique, 30 specimen each. Surface roughness values (Ra) of each specimen were measured five times, and mean Ra values were determined. Baseline color measurements were made with a Vita Easy shadeV. Each group was then divided into three subgroups of 10 specimens each according to storage media, specimens of each subgroup were stored for 7 days in distilled water, black coffee, or black tea respectively. The specimens were then rinsed with distilled water and dried before the second color measurement.

**Results:** No significant difference in surface roughness among the two types of composite resins regardless the method of finishing & polishing used. Additionally, no statistically significant correlation between the surface roughness and the color difference values.

**Conclusion:** Intra-orally, composite surface discoloration might happen with extremely smooth surface and not always related to the degree of surface roughness. Proper finishing and polishing of composite restorations will reduce but not totally prevent the color change caused by coloring beverages.

**KEYWORDS:** Color stability, Finishing and Polishing, coloring beverages, surface roughness.

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## INTRODUCTION

Achievement of typical color and appearance of natural teeth is the greatest exciting challenge that the dentist usually face. The restorative materials should copy the shape and color of the natural teeth. Color match and its stability overtime are the keys for success of esthetic restorations [1]. Color mismatch is a primary cause for composite restoration failure [2]. One of the greatest complications of composite restorations is their gradual color change and mismatch with the adjacent teeth over time [3].

Color changes could be classified into: first, intrinsic discoloration factors which are inherent to the material itself due to the internal physical/ mechanical inside the material [4] and second, extrinsic discoloration as a result of surface roughness, water absorption, diet, and smoking [5]. Drinking of certain beverages especially coffee/tea can influence the appearance and characteristics of composite restorative materials [5].

All composite restorative materials composed of two principal components; filler particles and resin matrix. Different hardness ratios of filler particles can result in discrepancy in polishing effectiveness. This discrepancy can lead to modifications in surface roughness [6].

Additionally, particle size of the composite has a principal effect as larger particles are often associated with an important detachment of the fillers, and therefore to a higher porosity of the restoration [7]. That is the reason most operators use micro-filled or nano-filled composites to replace the enamel layer, these materials initially provide a relatively good surface smoothness and higher shine.

Appropriate finishing/polishing methods are challenging clinical step improving appearance and enhance durability of restorations. Polishing diminishes scratches caused by finishing instruments [8] [9]. Smooth surface is very helpful not only in minimizing plaque accumulation and gingival irritation, but also to control the esthetics and durability of the composite resin restorations [10, 11].

A number of studies confirmed that resin composite reached its smoothest surface using a clear matrix covering the restoration surface during hardening [12]. However, further contouring is usually needed, so it is helpful to determine a suitable finishing technique resulting in ideal smooth surface during clinical service [13].

Among the widely used polishing devices are silicon carbide-coated and aluminum oxide-coated abrasive discs. Surface sealants also could be used to fill the irregularities on composite surfaces aiming to enhance the luster and smoothness of the surface [14, 15]. Using of these sealants affects stain absorption and color stability of such esthetic restorations [16].

Hypothesis: There will be positive relationship between surface roughness of polished composite and its color change.

The aim of the current study was to compare in vitro the color stability of two resin composite restorative materials and its correlation with surface roughness following different surface finishing/ polishing procedures.

## MATERIALS AND METHODS

Materials used in the current study are listed in Table I

An A1 color shade was used for both composite materials (Microhybrid composite A, Nanohybrid Composite B).

Thirty disks from each group were prepared using a Teflon mold (total 150 specimens), 9-mm in diameter and 2-mm in depth. The materials were managed according to the manufacturers' instructions.

A nylon thread was fixed into the specimen to be used in specimen's suspension in the solutions. The mold with the composite resin was held between two glass slides, each covered with a transparent

TABLE (I) Materials used in the study.

| Materials | Lot Number | Manufacturer | Composition |
|---|---|---|---|
| Microhybrid Composite resin | 6647356 | Herculite Classic Kerr Italia | 79 % inorganic filler (by weight) 59% by volume -trimethyl-dioxo-dioxa- -diazahexadecane-Bismethacrylate -hexanediyl bismethacrylate -ethylenedioxydiethyl dimethacrylate -hexamethylene diacrylate -trimethoxysilylpropyl methacrylate |
| Nanohybrid Composite resin | 6119059 | Herculite XRV Ultra Kerr Italia | 71% fillers Fillers types: prepolymerized filler (PPF), Barium glass (0.4 micron). silica nanofillers (20 – 50 nm), trimethyl-dioxo-dioxa -diazahexadecane- -diylbismethacrylate -bis (acryloyloxymethyl) butyl acrylate -trimethoxysilylpropyl methacrylate |
| Polishing Paste | | Dental Town® Mansoura Egypt | Aluminum Oxide 2.5 gm |
| Polishing Discs | 01616112 | Tor W Ltd Moscow Russia | No 1.071 Diameter 14 mm |
| Sealer | 1802221 | GC corporation Tokyo, Japan | Equia Coat Nanofilled self- adhesive light- cured protective coating |

polyester strip (Mylar; Henry Schein, Melville, NY), the slides were gently pressed together to remove excess material. Specimens were polymerized using a conventional halogen light polymerizing unit (ESPE Elipar Trilight; 3M ESPE, St. Paul, Minn) with light intensity of 450 mW/cm$^2$, using 40 seconds of exposure to both surfaces with the tip of the light curing unit contacting the glass surface during light-curing process. The specimens were removed out of the mold and kept in distilled water at 37$^o$C for 24 hours.

Specimens from each material group were divided randomly into five groups, 15 specimens each.

Group I (control group), specimens not subjected to any finishing/polishing procedure after removal of Mylar strip.

In group II, the surface of each sample was polished with medium, fine then superfine polishing discs for 30 seconds each on a rotary low speed handpiece with maximum of 15,000 rpm (Strong 204 Saeshin Precision Co, Daegu, Korea) with light hand pressure.

In group III, specimens were polished with medium, fine and superfine abrasive disks then aluminum oxide paste was used for 30 seconds.

In group IV, specimens were sequentially polished as described for group II, then protected by sealer (Equia Coat, GC) and light cured for 20 seconds.

In group V, specimens were polished as described for group III, then protected by sealer and light cured for 20 seconds.

A new polishing disk was used for each specimen and discarded after each use. Sealer was placed and cured following manufacturers' recommendations.

TABLE (2) The mean, standard deviation (SD) values and results of three-way ANOVA test for comparison between ΔE values of different interactions of variables

| Composite type | Surface treatment | Distilled water | | Coffee | | Black tea | | P-value (Between media) | Effect size (Partial eta squared) |
|---|---|---|---|---|---|---|---|---|---|
| | | Mean | SD | Mean | SD | Mean | SD | | |
| Micro-hybrid | Mylar strip | 1.38 $^{C}$ | 0.26 | 7.63 $^{AD}$ | 0.36 | 5.74 $^{BE}$ | 0.73 | <0.001* | 0.710 |
| | Successive discs | 1.56 $^{B}$ | 0.29 | 6.01 $^{AE}$ | 0.65 | 5.77 $^{AE}$ | 0.37 | <0.001* | 0.600 |
| | Successive discs + Polishing paste | 1.34 $^{B}$ | 0.47 | 6.53 $^{AE}$ | 0.13 | 5.82 $^{AE}$ | 1.63 | <0.001* | 0.654 |
| | Successive discs + Sealer | 2.13 $^{C}$ | 0.69 | 6.35 $^{BE}$ | 0.33 | 7.42 $^{AD}$ | 0.68 | <0.001* | 0.651 |
| | Successive discs + Polishing paste+ Sealer | 2.44 $^{B}$ | 0.42 | 7.14 $^{AD}$ | 0.26 | 7.08 $^{AD}$ | 0.23 | <0.001* | 0.634 |
| | P-value (Between surface treatments) | 0.160 | | 0.025* | | 0.002* | | | |
| | Effect size (Partial eta squared) | 0.102 | | 0.167 | | 0.241 | | | |
| Nano-hybrid | Mylar strip | 2.13 $^{C}$ | 0.24 | 4.67 $^{BE}$ | 1.55 | 8.28 $^{AD}$ | 1.11 | <0.001* | 0.695 |
| | Successive discs | 2.44 $^{B}$ | 0.22 | 5.6 $^{AE}$ | 0.66 | 6.61 $^{AE}$ | 0.12 | <0.001* | 0.531 |
| | Successive discs + Polishing paste | 1.17 $^{C}$ | 0.36 | 7.02 $^{AD}$ | 0.41 | 5.07 $^{BF}$ | 0.61 | <0.001* | 0.679 |
| | Successive discs + Sealer | 1.14 $^{B}$ | 0.33 | 7.9 $^{AD}$ | 0.14 | 8.73 $^{AD}$ | 1.28 | <0.001* | 0.805 |
| | Successive discs + Polishing paste+ Sealer | 1.75 $^{C}$ | 0.26 | 5.41 $^{BE}$ | 0.51 | 6.73 $^{AE}$ | 0.15 | <0.001* | 0.614 |
| | P-value (Between surface treatments) | 0.062 | | <0.001* | | <0.001* | | | |
| | Effect size (Partial eta squared) | 0.137 | | 0.450 | | 0.505 | | | |
| [P-value for Effect of composite type, (Effect size)] | | Distilled water | | Coffee | | Black tea | | | |
| Mylar strip | | 0.165 (0.032) | | <0.001* (0.344) | | <0.001* (0.277) | | | |
| Successive discs | | 0.100 (0.044) | | 0.441 (0.010) | | 0.117 (0.040) | | | |
| Successive discs + Polishing paste | | 0.749 (0.002) | | 0.354 (0.014) | | 0.163 (0.032) | | | |
| Successive discs + Sealer | | 0.066 (0.055) | | 0.005* (0.125) | | 0.016* (0.093) | | | |
| Successive discs + Polishing paste + Sealer | | 0.195 (0.028) | | 0.002* (0.151) | | 0.510 (0.007) | | | |

***: Significant at P ≤ 0.05***

***A,B,C superscripts in each row represent statistically significant difference among immersion media***

***D,E,F superscripts in each column represent statistically significant difference among surface treatments***

### Surface roughness test

The polished specimens were washed and stored for 7 days in distilled water. Ra values of each specimen were measured five times, and mean Ra values were determined with a cut-off value of 0.8 mm, and a stylus speed of 0.5 mm/s near the center of each specimen using a surface profilometer (Mitutoyo Surftest SJ-210 Surface Roughness Tester, Japan). Calibration of profilometer done against a standard specimen before starting the next measuring process [17].

### Base line color testing

Composite resin specimens were air-dried and measured with a Vita Easy shadeV (Vita Zahnfabrik, D-79713 Bad Säckingen, Germany) using CIE LAB color space relative to CIE standard illuminant D55 at baseline, and following staining. The color differences ($\Delta E_{ab}$*) between the 2 measurements were calculated as follows:

$\Delta E_{ab} = [(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2)]^{1/2}$ where L* is lightness, a* is green-red (-a*=green; +a*= red), and b* is blue-yellow (-b*=blue; +b*=yellow).

A perceptible discoloration that is $\Delta E_{ab}$ * 1.0 will be referred to as acceptable up to the value $\Delta E_{ab}$ *=3.3 in subjective visual evaluations made in vitro under optimal lighting conditions [18,2].

### Color stability

After baseline color measurements, specimens will be allocated into subgroups including 5 specimens, each subgroup was stored for 7 days in 200 mL of distilled water (subgroup i), black coffee (subgroup ii), or black tea (subgroup iii) in a separate polypropylene container.

Preparation of Coffee solution was done by mixing (15 g) of instant coffee powder (Grandos, café Gold, Hamburg, Germany) with 200 mL boiled water as recommended by the manufacturer. Preparation of Tea solution was done by immersing black tea packet 2g (Ahmed Tea, Hampshire, England) in 200 ml boiling water as recommended by the manufacturer. Every day the solution was refreshed [19].

After 7 days of immersion, the specimens were washed with distilled water for 5 min and dried with absorbent paper. The second color measurement reading was then carried out.

### Statistical Analysis:

Surface roughness (Ra) data showed non-parametric distribution while color change ($\Delta E$) data showed parametric distribution. Data were shown as mean and standard deviation (SD) values. Three-way Analysis of Variance (ANOVA) test was used to study the effect of composite type, surface treatment, immersion medium and their interaction on ($\Delta E$) (table 2). Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$.

## RESULTS

***Average Roughness between both composite types:***

When using Mylar strip for finishing composite, no statistically significant difference was found between Micro- and Nano-hybrid composites. Following immersion in distilled water and coffee; Micro-hybrid composite had statistically significantly lower mean (Ra) than Nano-hybrid composite. Following immersion in black tea; no statistically significant difference found in mean (Ra) compared to Nano-hybrid composite. After immersion in distilled water and in coffee; no statistically significant difference noticed between Micro- and Nano-hybrid composites. After immersion in black tea; Micro-hybrid composite had statistically significantly lower mean (Ra) than Nano-hybrid composite.

When using successive disks with polishing paste for polishing composite, after immersion in distilled water or black tea; no statistically significant difference was found between Micro- and Nano-hybrid composites. After immersion in coffee; Micro-hybrid had statistically significantly lower mean (Ra) than Nano-hybrid composite.

When using successive disks then applying sealer for polishing composite, after immersion in distilled water or black tea; no significant difference was found between Micro- and Nano-hybrid composites (Ra). After immersion in coffee; Micro-hybrid had statistically significantly lower mean (Ra) than Nano-hybrid composite.

When using successive disks with polishing paste then applying sealer for polishing composite, following immersion in distilled water**;** no statistically significant difference was noticed between Micro- and Nano-hybrid composites. After immersion in coffee or black tea; Micro-hybrid composite had statistically significantly higher mean (Ra) than Nano-hybrid composite.

**Color measurements results;**

In Micro-hybrid composite: Using Mylar strip; coffee had the highest statistically significant difference in mean ΔE followed by Black tea, while the lowest is the distilled water. Using successive discs, successive discs + polishing paste as well as successive discs + polishing paste + sealer; no statistically significant difference observed between coffee and black tea; while distilled water showed the lowest mean ΔE. Using successive discs + sealer; black tea showed the statistically significant highest mean ΔE. Coffee showed statistically significantly lower mean value. Distilled water showed the lowest mean ΔE.

In Nano-hybrid composite: Using Mylar strip as well as successive discs + polishing paste + sealer; black tea represented the statistically significant highest mean ΔE followed by Coffee. Distilled water showed the lowest mean ΔE. Using successive discs as well as successive discs + sealer; no statistically significant difference was noticed between coffee and black tea. Distilled water showed the lowest mean ΔE. Using successive discs + polishing paste; coffee showed the statistically significant highest mean ΔE followed by Black tea. Distilled water showed the lowest mean ΔE.

## DISCUSSION

Discoloration of resin composite surface is a multifaceted action involves numerous factors [20]. In the present study we tried to correlate surface roughness as one of the contributing factors in composite discoloration process. Surface roughness is usually correlated to the filler size of the resin composites and pattern of finishing and polishing of their surfaces[1,21] .

Two types of composite resins were used in this study representing the most commonly clinically used esthetic direct restorations; micro-hybrid and nano-hybrid types.

Finishing and polishing procedures affect surface smoothness, that may be associated with early surface discoloration. Rough surfaces retain stains more than smooth surfaces [22,23]. A variety of polishing systems are now available, but none of them assure perfect surface smoothness as obtained by mylar strip. Polishing protocol followed in this study is the widely clinically used successive aluminum discs [24,25]. Use of polishing paste is also suggested to increase surface smoothness [26,27], however some scratches and micro gaps could be left on the polished surfaces of composite resin restoration.

Use of a thin layer of a low-viscosity resin was studied to overcome this problem. The surface-penetrating sealant should have the ability to fill the structural micro defects and micro fissures formed during finishing/polishing process. The sealant is expected to give more homogenous and smoother surface [28,29]. Doray et al, stated that surface sealants could enhance staining resistance of resin composite restorative materials [16].

However, in the present study, the surface sealant applied on composite surface was not efficient on preventing color alteration. This in accordance with Fernanda Valentini et al, who stated that use of sealant causing more staining of the restorations kept in coffee. This could be attributed to that, in contrary to the restorative composite, there are no filler particles present in the resin sealant. The glass filler particles are generally inert and has no affinity to absorb fluids. Rate of water/dye uptake being is proportional to the resin percentage of restorative composites. Hydrophilic comonomers in the resin sealant could also cause color instability [30] .

Another factor related to composite discoloration is the adsorption and absorption of pigments into the organic phase of resin-based materials [31]. Difference in polarity between resin polymers and dietary pigments, causing absorption of yellow pigments by the organic content of composite materials [32]. Coffee and tea were used in the study as coloring agents because of their frequent consumption in daily life. 7 days immersion period was chosen as it is appropriate to cause color modifications of resin composite [33,34]. 24 hours *in vitro* mimic about one month *in vivo*, that is considered to be sufficient for a long-term color stability assessment [35]. According to Guler, et al. (2005), 15 min is the average time for drinking of 1 cup of coffee, 3-2 cups is the average consumption per day among coffee consumers. Thus, seven days of storage simulated more than six months of drink consumption [27] .

In the present study significant differences in ΔE values were found among micro- and nano-filled composite materials for all storage media. Coffee media caused the highest color changes in the specimens than the other medias that is could be attributed to the great amounts of gallic acids incorporated in coffee.[31], however tannic acid is the responsible on staining caused by tea [5]. Regarding surface roughness, all groups represented higher Ra values compared to the mylar strip group, and no significant difference was found in surface roughness between microhybrid and nanohybrid composites resin materials cured against the Mylar strip.

Visual assessment of minimal color change is not a precise quantitative evaluation, in addition to lack of reproducibility. Continuous development of electronic optics put the electronic techniques for color selection to be more adequate for daily usage [13,36]. In the present study, Vita Easy shadeV( Vita Zahnfabrik,D-79713 Bad Säckingen, Germany) was used for standardized evaluation. Both types of resin composites showed decrease in Δ*E** values in relation to the finishing/polishing techniques. The highest values were noticed with sealer coated, followed by mylar strip and finally the successive discs polishing.

Regarding Δ*E** value, the use of mylar strip, both of microhybrid and nanohybrid composites resulted in more staining. This could be explained by high percentage of matrix. Resin matrices have a propensity for water absorption and then susceptibility to discoloration by dye infiltration [13,36,37]. This finding is in accordance with Gönülol and Yılmaz [38]. Same results are also reached by de Costa *et al.* [39] who stated that adsorption of stains could be the cause of rough surfaces discoloration, even though absence of direct relationship between rough surface and staining [20].

## CONCLUSION

1. No significant difference in surface roughness was observed among nanohybrid and microhybrid resin composites with the use of different surface treatment methods used.
2. No statistically significant correlation noticed between the surface roughness and the color difference values for both composite types.
3. Intra-orally, composite surface discoloration might happen with extremely smooth surface and not always related to the degree of surface roughness.
4. Proper finishing and polishing of composite restorations is very important step to get a clinically apparent smooth surface. However, it will not grantee inhibition of color change caused by coloring beverages.

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