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COLOR STABILITY OF NANOFILLED AND SUPRANANOFILLED RESIN COMPOSITES WITH DIFFERENT POLISHING TECHNIQUES AFTER IMMERSION IN COFFEE SOLUTION

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ABSTRACT

Objective: To determine color stability of nanofilled and suprananofilled resin composites subjected to different polishing systems after immersion in coffee solution.

Materials and Methods: Nanofilled (Filtek Z350 XT Universal) and suprananofilled (Palfique Omnichroma) resin composites were polished after polymerization, except for control groups, with three-step (Enamel Plus Shiny), two-step (Super-Snap X-treme) or one-step (OneGloss) polishing systems after polymerization. The baseline color was recorded using spectrophotometer (Vita Easyshade V), samples were stored in coffee solution for 14 days, then color change was calculated using (CIEDE2000) and statistically analyzed using two-way ANOVA test and post hoc pairwise comparisons.

Results: There was a significant interaction between type of composite resin and polishing protocol on color change. For the control samples, the discoloration in nanofilled samples was significantly higher than suprananofilled ones. For both types of composites, there was a significant difference between samples polished by different protocols.

Conclusions: 1. Unfinished and unpolished suprananofilled composite obtains less color change than the nanofilled composite 2. The three-step polishing provide the least color change in both types of resin composites, yet comparable with the two-step system in the nanofilled composite 3. Immersion of composite in coffee colorant showed apparent alterations in all tested groups.

KEYWORDS: Color stability, Nanofilled, Suprananofilled, Resin composite, Polishing, Staining, Omnichroma

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INTRODUCTION

Color stability of resin composite restorations is considered one of the most important physical properties of the material. The color alteration of restoration can affect the clinical longevity and the overall performance within the oral environment which is reported as a major cause of restorations replacement.[1–3] Surface staining and discoloration can negatively affect the patient's perception of the existing restoration which can be categorized as an esthetic failure.[4,5] Manufacturers have worked on making the color of resin composite material stable over time. Yet, several intrinsic and extrinsic factors can change the color of resin composite restoration dramatically during clinical service.[6] Extrinsic discoloration may occur as a result of improper oral hygiene habits, certain dietary chromogens, smoking, water sorption, and surface roughness of the restoration.[7–9] Different coloronts from dietary beverages such as coffee can be incorporated and adsorbed into the resin surface which subsequently cause substantial discoloration.[2,10,11] The decreased wettability of the surface of restoration and lower water sorption are considered valuable factors to reduce staining and plaque accumulation.[12,13]

In addition, proper finishing and polishing procedures ensures optimum esthetic outcome of the resin composite restorations.[14] A rough surface induces plaque accumulation, staining and unpleasant esthetic appearance.[15–17] The smoothest resin composite surface can be obtained against polyester Mylar strip.[18,19] Nevertheless, removal of the superficial resin-rich layer is crucial as it can compromise mechanical properties, biocompatibility and increasing staining susceptibility.[20] Moreover, many attempts have been made to select which polishing protocol provides the most color stable resin composite surface, and various techniques have been proposed without verifying which one is the best.[18,19,21]

Whereas intrinsic factors depend on the physio-mechanical reaction in the resin matrix, such as the type of resin matrix itself, that eventually affects the hydrophilicity or the hydrophobicity of the material.[22,23] It is essential to emphasise that the final matrix characteristics do not arise only from the properties of individual monomers, but also from the interactions within the monomer mixture and the resultant polymer network.[24–26] In addition to the degree of polymerization, alteration of the filler/matrix interface,[27,28] the type of photoinitiator used, the filler type, size and distribution are also key factors influencing composite dicoloration.[3,29,30] Moreover, the chemical discoloration is attributed to oxidation of the amine accelerator, the polymeric matrix composition, and the unreacted methacrylate groups.[31,32]

Currently, using the smart monochromatic shaded resin composites is rising and becoming a trend among dentists. This technology reduces chairside time, the need for a variety of composite shades supply, minimizes the waste of infrequently used shades of composite, eliminates the complexity of shade-matching procedures.[33] The suprananofill resin composite Omnichroma (Tokuyama Dental, Tokyo, Japan) is one of the smart monochromatic materials and claimed to obtain the shade of the surrounding tooth structure. Though, knowledge about the color stability of this material is still inadequate in the literature.[3,34,35] Therefore, it is useful to investigate the color stability of this type of suprananofill resin composite in comparison to one of the conventional nanofill composites.

Accordingly, the objective of this study was to evaluate color stability of nanofilled and suprananofilled resin composites subjected to three-step, two-step and one-step polishing systems after immersion in coffee solution. The null hypothesises tested were: 1. There was no difference in color stability between different resin composite materials, 2. There was no difference in color stability between both types of resin composites subjected to different polishing techniques.

MATERIALS AND METHODS

Specimen preparation:

The sample size was calculated using the analysis package program (G*Power 3.1.9.7 for Windows 10; Universität Düsseldorf), with 0.05 significance and at 80% power level. A total of 56 resin composite discs, divided into 2 main groups of 28 discs each of nanofilled Filtek Z350 XT Universal Restorative (3M ESPE Dental Products, USA) and suprananofilled Palfique Omnichroma (Tokuyama Dental, Japan) were prepared (Table 1). Each composite disc was prepared using split Teflon mold with a cylindrical cavity of (10 x 2mm). A microscope glass slide was placed under the mold and the composite material was inserted in a single increment into the mold using a smooth, round ended condenser with slight overfilling. A Mylar strip was placed over the resin composite surface and another microscope glass slide was gently pressed over the strip to flatten the surfaces and eliminate any excess. The composite was polymerized using Radii Plus LED Curing Light device (SDI, Basywater, Victoria, Australia) with 10 mm curing tip diameter and the intensity of 1400mW/cm^2 for 60s through Mylar strip and glass slide, with the curing tip touching and perpendicular to the glass slide. Additional polymerization was done on the experimental of the specimen for 20s after removing the strip and glass slide. The output intensity was measured every 5 specimens using a radiometer to ensure that the value is ≥1400mW/cm^2. The mold was disassembled, and each sample was checked to exclude any visible voids after removal from the mold and the bottom of the disc was labeled. The flash excess on the margins of the polymerized discs were trimmed carefully without any alteration to the Mylar-formed surface. The specimens were stabilized in acrylic mold with the experimental surface facing upward on the kitchen scale for polishing.

TABLE (1) Composition of the resin composite used

Material		Filtek Z350 XT Universal Restorative	Palfique Omnichroma
Filler		Non-agglomerated/non-aggregated 20 nm silica filler Non-agglomerated/non-aggregated 4 -11 nm zirconia filler Aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles). The Enamel shades average cluster particle size of 0.6 -10 μm	Uniformly sized supranano-spherical filler of SiO2 and ZrO2 plus a round-shaped composite filler all with a particle size of 260 nm
Filler Content	**wt%**	78.5	79.0
	vol%	63.3	68.0
Monomer		bis-GMA, UDMA, TEGDMA, bis-EMA(6) resins, and PEGDMA[1]	UDMA and TEGDMA
Shade		A2 Enamel shade	-
Manufacturer		3M ESPE Dental Products, St. Paul, MN, USA	Tokuyama Dental, Tokyo, Japan
LOT		NA02267	002E60
Code		FK	OM

[1]Bis-GMA: Bisphenol A diglycidimethacrylate: UDMA: Urethane dimethacrylate; EMA: ethylmethacrylate; TEGDMA: triethylene glycol dimethacrylates; PEGDMA: polyethylene glycol dimethacrylate

Finishing and polishing procedures:

Each group was randomly sub-divided into 4 groups of 7 each, a control group which received no surface treatment and 3 experimental groups. The surface of the composite disc cured against the mylar strip (except for the control groups) was manually ground for 30 seconds with wet 600-grit silicon carbide paper in a circular motion then rinsed and air-dried, representing the finishing step before polishing and used as the experimental surface.

In order to reduce variations, the same operator carried on all the polishing procedures with the same slow-speed handpiece (NSK FX25 1:1 Dental Low Speed Handpiece, Japan) at a rotational speed of 3000 rpm using a kitchen scale as a pressure guide maintaining light intermittent pressure 30-40 gm (approximately 0.3 newton) representing the light pressure needed for the polishing procedure.[36] Each stroke was applied in the same direction, and each polishing instrument was discarded following each use. The polishing procedures were performed as follows (Table 2):

Group 1: Three-step Enamel Plus Shiny polishing pastes (Micerium, Italy): The three polishing pastes used with a sequence of progressively finer polishing paste whereas Shiny A 3μm diamond paste was applied with a goat hair brush Shiny S each for 20 seconds. Then, Shiny B 1μm was applied with a different goat hair brush for 20 seconds, and finally, Shiny C aluminum-oxide paste was used with the disc felt Shiny FD for 20 seconds as a final polishing step. All were used under dry condition and after each step thorough rinsing with air-water spray for ten seconds then air drying for five seconds were done as recommended by the manufacturer.

Group 2: Two-step Super-Snap X-treme discs (Shofu, Japan): fine (green) and superfine (red) used sequentially each for 20 seconds under dry condition. After each step, all specimens were thoroughly rinsed with air-water spray for ten seconds and air dried for five seconds as recommended by the manufacturer.

Group 3: One-step OneGloss (Shofu, Japan): PS Silicone Polisher IC Inverted Cones Points were used in a light feather pressure (representing the polishing mode only) for 20 s with intermittent water spray as recommended by the manufacturer.

TABLE (2) Composition and list of the polishing systems used

Polishing system	Enamel Plus Shiny	Super-Snap X-treme	OneGloss
No of application steps	Three-step	Two-step	One-step
Matrix	Polyethyleneglykol	Base film (polyesther) Mounting core (PVC)	Synthetic rubber (Polyvinylsiloxiane)
Abrasives	Shiny A: diamond powder applied with a goat hair brush Shiny S Shiny B: diamond powder applied with a goat hair brush Shiny S Shiny C: Aluminium oxide powder applied with the disc felt Shiny FD	Aluminum oxide	Aluminum oxide Al2O3 and Slicone oxide SiO2 grains
Particle size	Shiny A: 3 μm Shiny B: 1 μm Shiny C: N/A	SS Green 20 μm SS Red 7 μm	Mean alumina particle size 85 μm
Manufacturer	Micerium, Italy	Shofu, Japan	Shofu, Japan

All specimens were thoroughly rinsed with air-water spray for 10 seconds then stored in 100% humidity separate labeled containers at room temperature at 37±2ºC for 24 hours before baseline color measurement and immersing in the coffee staining solution.

Staining procedure and color measurements:

The baseline color measurements of all composite samples were performed by a spectrophotometer (Vita Easyshade V, Vita Zahnfabrik, Bad Sackingen, Germany) using the Commission internationale de l'éclairage *L*a*b** system.[37] The device was calibrated according to the manufacturer's recommendations before each measurement. The tip of the spectrophotometer was placed perpendicular at the center of each sample and initial color values of all samples were measured against a standard black background.[38] Each measurement was repeated four times for each sample and the averaged shade measurement was recorded.

Coffee solution was prepared of 3.6 g coffee (Nescafe Classic, Nestle, Switzerland) dissolved in 300 ml boiled water, mixed for 10 min, and passed through solution filter paper.[22] Samples were stored in coffee solution at room temperature 37±2°C, while each sample was in a sealed labelled separate container. Solutions were replaced every 48 hours to prevent possible bacterial or yeast interactions.

After the completion of 14 days, the teeth were thoroughly rinsed and air dried for 10 seconds. Measurement of color alteration value was performed by the principal investigator, with the same device and the same protocol, as directed by the manufacturer. At last, the mean difference in the color alteration values (ΔE_{00}) of the samples before and after immersion in coffee solution was calculated using the following (CIEDE2000) color difference formula.[11]

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C}\right)\left(\frac{\Delta H'}{K_H S_H}\right)}$$

where ΔL', ΔC', and ΔH' are the differences in lightness, chroma, and hue, respectively, and R_T is a function that accounts for the interaction between chroma and hue differences in the blue region. Weighting functions, S_L , S_C , and S_H adjust the total color difference for variation in the location of the color difference pair in L', a', and b' coordinates and parametric factors, K_L, K_C, and K_H, are correction terms for experimental conditions.

Statistical analysis

Numerical data was represented as mean and standard deviation (SD) values. Shapiro- Wilk's test was used to test for normality. Homogeneity of variances was tested using Levene's test. Data showed parametric distribution and variance homogeneity and were analyzed using two-way ANOVA. Comparison of simple main effects was done utilizing the error term of the two-way model with p-values adjustment using Bonferroni correction. The significance level was set at $p<0.05$ within all tests. Statistical analysis was performed with R statistical analysis software version 4.1.3 for Windows (R Core Team (2022). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL https://www.R-project.org/).

RESULTS

Results of two-way ANOVA presented in Table (3), showed there was a significant interaction between type of composite resin and polishing protocol on color change ($p<0.001$). Comparison of simple main effects presented in Table (4) showed that for the control samples, the discoloration in nanofilled samples was significantly higher than supranofilled ones ($p<0.001$). However, for other protocols, the difference was not statistically significant ($p>0.05$).

For both types of composites, there was a significant difference between samples polished by different protocols ($p<0.001$). For the nanofilled

samples, post hoc pairwise comparisons were all statistically significant (p<0.001), except for (two and three-steps) (p=0.053). For the suprananofilled samples, post hoc pairwise comparisons were also all statistically significant (p<0.001). However, the difference between control and two-step samples was not statistically significant (p=1). Mean and standard deviation values for color change are presented in Figures (1) and (2).

TABLE (3) Two-way ANOVA test results

Parameter	Sum of squares	df	Mean square	f-value	p-value
Composite resin type	1.73	1	1.73	3.38	**0.072**
Polishing protocol	75.2	3	25.07	49.12	**<0.001***
Type * protocol	18.69	3	6.23	12.21	**<0.001***
Error	24.5	48	0.51		

**** Significant (p<0.05)***

TABLE (4) Comparisons of simple main effects

Polishing protocol	Color change (ΔE_{00}) (Mean±SD)		p-value
	Nanofilled	Suprananofilled	
Control	6.07±1.04^{A}	3.80±0.58^{B}	**<0.001***
One-step	4.92±1.13^{B}	5.36±0.85^{A}	**0.262**
Two-step	3.40±0.45^{C}	4.08±0.57^{B}	**0.082**
Three-step	2.36±0.22^{C}	2.11±0.33^{C}	**0.512**
p-value	**<0.001***	**<0.001***	

Means with different superscript letters within the same vertical column are significantly different *significant (p<0.05)

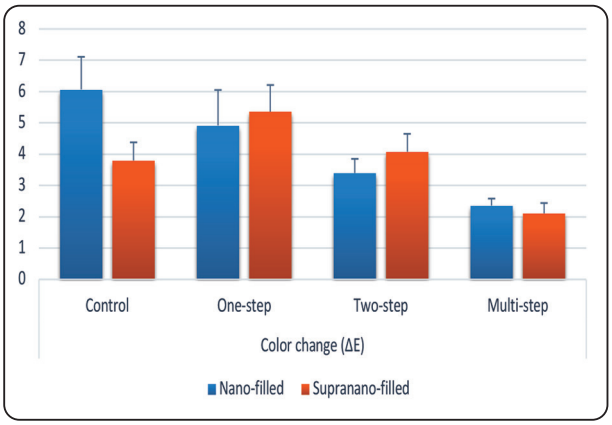

Fig. (1) Bar chart showing mean and standard deviation values of color change (ΔE_{00}) values in different types of composite resin

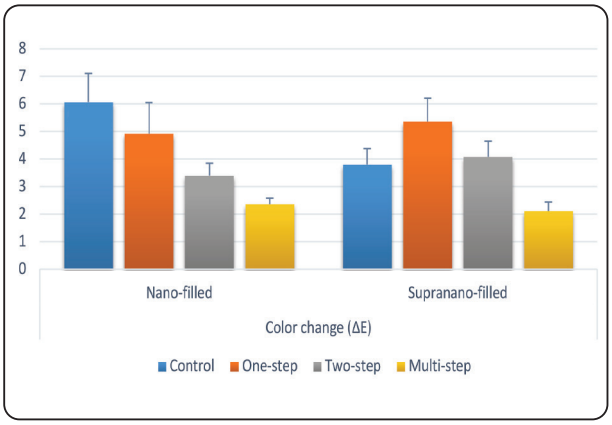

Fig. (2) Bar chart showing mean and standard deviation values of color change (ΔE_{00}) values in different polishing protocols

DISCUSSION

Long-term color stability of resin composite is crucial to the acceptability of restoration during their intraoral functional life, which is related to frequent replacement of dental restorations.[1,2] Color change of resin composites is due to inherent factors related to the characteristics of the material itself as well as extrinsic factors such as adsorption of exogenous pigments and surface roughness of the restoration related to the applied finishing/polishing protocol.[4,14] Suprananofilled resin composite material used in the study was selected, as studies on the color change of this material specially in

comparison with conventional nanofilled resin composite polished with different polishing techniques, have not yet been encountered sufficiently.[3,34,35]

The first null hypothesis of this study that there would be no difference in color stability between different resin composite materials, was partially rejected. As for the control samples, the discoloration in nanofilled samples was significantly higher than suprananofilled ones ($p<0.001$). However, for other polishing protocols, the difference was not statistically significant ($p>0.05$). These results are in agreement with another study that showed higher staining resistance of Omnichroma than Filtek Z350 XT, after immersion in coffee solution.[33] This could be due to the difference in material composition between the two types.

The second null hypothesis that there would be no difference in color stability between both resin composites when subjected to different polishing techniques, was rejected. As for both types of composites, there was a significant difference between samples polished by different protocols ($p<0.001$). This implicate that simply some abrasive instruments perform better on a matching resin composite material, depending on their specific characteristics.

In this study the pre-polishing was done with a wet 600-grit silicon carbide paper (equivalent to the yellow-coded finishing stone) to insure even initial surface topography.[15] All polishing procedures were applied by the same operator according to their corresponding manufacturers recommendations to achieve predictable results. Moreover, the speed, pressure, motion used during the polishing were fixed, guided by the range of their manufacturers, to reduce the variability for all polishing protocols.[16,17]

In the current study, coffee was used as a coloring solution for its common intake in daily life, which can cause yellow-brown stains on the surfaces of the resin composite restorations.[32] While the initial surface lustre and smoothness depends mainly on the filler characteristics, color stability and staining resistance after polishing are a function of hydrophobic resin monomer structure, coloring agents and adequate polymerization of the material.[22,27] Low degree of conversion of the resin monomers additionally results in inferior mechanical properties and more color alteration.[28] The filler particles do not absorb water into the bulk of the material but can adsorb water onto their surface. The degree of water sorption depends on the chemistry of the polymer matrix, crosslinking density, and the degree of hydrophilicity. Consequently, this has a plasticization effect on the polymer structure of resin composite, leads to the chemical instability and hydrolytic deterioration of resin-filler interface which accordingly reduce polishing retention and color stability over time.[29]

Regarding the resin composite type in this study, the difference found in color stability between the control groups of nanofilled and suprananofilled composites can be a result of extreme tight contact between the various fillers and the resin base in OM due to the its higher filler content of OM (68% by volume) than that of the FK (63.3% by volume).[20] Moreover, the hardness of resin matrix rise from the characteristics of particular monomers in addition to the properties of the resultant polymer network from the monomer mixture.[23] The UDMA/TEGDMA matrices can obtain higher hardness values than Bis-GMA based matrices. This could be explained as the UDMA has low viscosity and subsequently more flexible than Bis-GMA, which increase its degree of conversion and form denser polymer network.[24,25] In addition, the resin hardness values increase with the existing of aromatic rings and urethane bonds.[26] It is possible that the higher relative hardness of UDMA based matrix of OM have worked in favour of a more stain resistant surface than the Bis-GMA based matrix of FK.[24,25]

The control group which received no finishing or polishing had the highest significant color change value among the FK groups, also it did not obtain the lowest discoloration value among the OM

groups. Several studies found that the smoothest surface obtained on resin composite restoration, is that produced by a properly applied Mylar strip.[18,19] Compression of resin composites against polyester strip leads to slide lower-order (less dense) particles, creating resin-rich surface layer followed by subsurface layer of filler particles interspersed with organic matrix yielding this smooth surface.[21] However, even though the outcrop of organic matrix can generate a smooth resin composite surface, free of any oxygen-inhibited layer, it produces a more hydrophilic surface which can attain more staining.[1,9,19] Contrariwise, even if polishing may relatively increase surface roughness, it increase hydrophobicity as well, probably due to the reduction of the quantity of superficial organic matrix.[21] This is favourable to limit water sorption and adhesion of colorants.[12] Apparently, frictional confinement and the load induced by the applied Mylar strip hinder the free reorganization of molecules of the contracting resin composite, consequently cause strain which activates the surface atoms and prompotes accumulation of the colorants and dental plaque.[13] In addition, even with a careful use of Mylar strip, the surface may contain imperfections such as air inclusions, folds or at least sections of flash excess near margins, making finishing/polishing inevitable.

The three-step polishing system obtained the lowest color change values (ΔE_{00}) with a significant difference among the OM groups, and non-significant difference with the two-step group among the Filtek groups. Among the polishing systems used, the three-step polishing paste was the only system containing diamond, which is a very hard element that can promote deep wear.[17] The abrasive particles that initiate deep wear cause exposure of deeper sub-subsurface layer rich in higher percentage filler particles in OM along with large clusters present in FK, which is positively related to the composite color stability.[30] Whereas the one-step and two-step polishing systems, containing aluminium oxide only or combined with silicone oxide, did not produce the same wear and exposed only the subsurface layer rich in smaller nanosized filler particles in FK, or suprananofiller filler particles in OM along with higher percentage of resin matrix. Accordingly, discoloration of resin composite clinically is highly correlated to chemical changes within the resin matrix, such as oxidation of unpolymerized monomers, organic pigments and the incorporated fillers (particle size, percentage and distribution).[6]

In the present study, color measurements were assessed using a spectrophotometer that eliminated the possibility of human errors, since color perception is a psychophysical process that is prone to extreme variations between different individuals or the same individual at different times. A study showed that with CIEDE2000 formula, 50:50% acceptability threshold was 1.8 (1.23–2.37). In this study, it was found that all groups were above the acceptability threshold after 14 days immersion in coffee solution, however, three-step polishing groups showed the least color change nearest to the clinically acceptable values (2.36±0.22 in FK group and 2.11±0.33 in OM group). However, further inherent factors, such as the purity of the monomers, initiators, inhibitors, activator (type and concentration) and filler loading, have a considerable impact as previously discussed.[10] In addition, presence of inorganic pigments as in FK or their absence as in case of OM, should also be considered.

The limitations of this in-vitro study should be considered when analysing the results: the resin composites were not constantly exposed to varied equilibrated diet, exposed to saliva, any brushing or oral hygiene measurements as in the natural oral environment. The examined materials were continuously immersed in the coffee solution for 14 days. Twenty-four hours of continuous in-vitro immersion has been reported to simulate about 1 month of coffee consumption in vivo.[11] Additional in-vitro studies including variant other finishing/polishing systems, different staining solutions and in-vivo studies researching these variables are necessary to confirm the results obtained here and for more precise evaluations.

CONCLUSIONS

Within the limitations of this current study, it was concluded that:

1. The monochromatic suprananofilled resin composite set against Mylar strip without any finishing and polishing obtains less color change over time than the nanofilled resin composite.
2. The three-step polishing pastes system provides the least color change in both types of resin composites, yet it is comparable with the two-step disc system in the nanofilled resin composite.
3. The immersion of composite in coffee colorant showed apparent alterations in the color with different degrees of both suprananofilled and nanofilled resin composites with different polishing techniques. Hence, the procedure of polishing should be selected wisely, depending on the resin composite type.

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