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Short and long term effects of additional post curing and polishing systems on the color change of dental nano-composites

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The purpose was to determine the short and long term effects of polishing systems and post-heat-light curing on the color stability of nano-composites. The disc shaped samples (ø=10 mm, h=3 mm) were prepared. Forty subgroups (n=5) were designed according to two different curing conditions and five different polishing methods. Color change measurements were performed on the day of specimen preparation (base) and repeated after 1 and 7 months of water storage. MANOVA and Tukey's post hoc tests were applied (p<0.05). While the color difference values of resin composites were ranked as EXP2>EXP1>CME≥XTE after 1 month (p<0.05), after 7 months the ranking was EXP2≥EXP1>XTE>CME (p<0.05). Type and compositions of nano-composites may be important on the long-term color stability of the restoration. Additional post-heat-light curing of nano-composites may produce higher color change than the hand-light curing protocol. Consequently, the polishing procedures should be applied to obtain more resistant composite surface to discoloration.

Keywords: Color stability, Nano-composites, Additional-post-curing, Polishing methods

INTRODUCTION

With the increasing demands for esthetic or metal-free restorations and the advent of adhesive cavity designs, research has focused on development of new tooth-colored materials and techniques[1]. Recently, several materials have been introduced to serve useful alternatives in restorative and prosthodontic applications. However, when considering the costs, resin composites are usually preferred to dental ceramics because of their esthetic results by offering shade match with the remaining tooth structure and adjacent teeth[2,3].

Color stability is an important issue for esthetic restorations and plays a role in the clinical success of restorative treatment. Color change of the dental restorations has been implied as a multifactorial phenomenon and can be caused by extrinsic and intrinsic factors[4]. The extrinsic factors include intensity and duration of polymerization, exposure to environmental factors, including ambient and UV irradiation, heat, water and food colorants[5]. Additionally, the intrinsic factors include composite photoinitiator system, polymer quality, type, and quantity of inorganic filler and type of accelerator added to the photoinitiator system[4].

Because of the optical properties of dental resin composites are influenced by surface changes during finishing and polishing[6], they are important clinical procedures that enhance both esthetics and longevity of the restorations[7]. A previous study by Morgan[8] demonstrated that the surface texture of dental materials has a major influence on plaque accumulation, discoloration, wear and the esthetic appearance of composite restorations. Moreover, the composition and volume of organic matrix, type and volume of filler particles, type of filler-matrix silanization, and polishing have been reported as the relevant factors in the composite susceptibility to staining[4]. Color stability of resin composites has been reported as being improved when materials showed low water sorption, high filler-resin ratio, reduced particle size and hardness and an optimal filler-matrix coupling system[9]. Previously, nano-composites have been claimed to combine the good mechanical strength of the hybrid-type composites[10,11] and the superior polish of the microfills[12]. Besides, these composites have been shown to have high wear resistance[13] improved optical characteristics[4] and reduced polymerization shrinkage[14]. A study about additional post-heat curing[15] demonstrated that application of heat above the glass transition temperature of the resin matrix can enhance the degree of monomer conversion and improve the mechanical properties of the composite. In addition, an increased biocompatibility of the resin composite after additional post-heat treatment was shown to occur[16,17]. For these reasons, post-curing might be efficient on the color stability of resin composites. Furthermore, the results could be clinically important due to the esthetic requirements of resin composites, which are intimately related to the optical interactions of light with matter.

Color and color difference are quantified using the CIE 1976 $L^*a^*b^*$color spaceand the associated ΔE^* respectively[18]. This system is a 3-dimensional color

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measurement system to describe color characteristics of an object based on three parameters: L^* refers to the lightness coordinate and its value ranges from 0 for perfect black to 100 for perfect white. a^* and b^* are chromaticity coordinates on the green-red ($-a^*$=green; $+a^*$=red) and blue-yellow ($-b^*$=blue; $+b^*$=yellow) axes[19]. Unacceptable color change values ($\Delta E^* \geq 2$[20], $\Delta E^* \geq 3.3$[21]) were reported previously. Also, the value $\Delta E^* \geq 3.3$ was reported as the threshold for clinical acceptability of color change[22].

The aim of this study was determine the short and long term effects of different polishing systems and post heat curing by heat on the color change of dental resin nano-composites. The following null hypotheses were tested: (1) The compositions of resin nano-composites do not affect the color stability. (2) Different polishing systems do not influence the color stability of the resin nano-composites. (3) Additional post-heat-light curing of the material does not influence the color difference of resin composites.

MATERIALS AND METHODS

Two brands of commercial and experimental light cured resin composites were used for the current study (Table 1).

Preparation of the experimental resin composites

Experimental resin matrix was prepared with 0.7 wt% photoinitiator (camphorquinone; Aldrich, USA) and 0.7 wt% photoactivator (2-(dimethylamino) ethyl methacrylate; Aldrich, USA) with resin mixtures containing 50 wt% Bis-GMA (Esstech, USA) and 50 wt% TEGDMA (Esstech, USA). Experimental composites were made with resin matrix added to the inorganic fillers, which were loaded with $BaAlSiO_2$ microfiller (average particle diameter 0.4 μm, SP345, Specialty Glass, USA). Resin matrix and constant filler fractions were 72.2 wt% for EXP1 and 73.6 wt% for EXP2. Also EXP2 was loaded with $BaAlSiO_2$ nanofiller (average grain size 175 nm, Schott, Germany). Nanofiller fraction was 4.9 wt% in EXP2 group. All inorganic filler particles were treated with methacryloxypropyltrimethoxysilane (MPS; Aldrich, USA). The experimental resin composites were weighted into a mixing cup and fillers were added into the cup in small portions. The composites were mixed with Speedmixer (DAC 150 FVZ-K, Hauschild, Germany) with a speed of 1,700 rpm for 30 s after the fillers were added.

Table 1 Resin composites and their compositions used in the study

Resin Composite	Code	Type	Composition	Batch Number	Manufacturer
Filtek™ Supreme XTE	XTE	Nanofilled	Matrix: bisphenol A polyethylene glycol dietherdimethacrylate (BisEMA), bisphenol A diglycidyl ether dimethacrylate (Bis-GMA), diurethane methacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA). Filler: aggregated silica/zirconia filler cluster filler of 0.6–10 μm with primary particle size of 4–20 nm and non-aggregated 20 nm silica/zirconia filler Filler loading: 63.3 vol% (78.5 wt%)	N221778	3M ESPE, St. Paul, MN, USA
Clearfil Majesty™ Esthetic	CME	Organic filled nanohybrid	Matrix: Bis-GMA, Hydrophobic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate Filler: silanated barium glass filler, pre-polymerized organic filler including nano filler of 0.2–100 μm with average particle size of 0.7 μm Filler loading: 66 vol% (78 wt%)	0021AA	Kuraray Medical Co., Tokyo, Japan
Experimental Composite 1	EXP 1	Nanohybrid	Matrix: Bis-GMA, DMAEMA, TEGDMA, camphorquinone (CQ) Filler: $BaAlSiO_2$microfiller (average particle diameter of 0.4 μm) Filler loading: 72.2 wt%		
Experimental Composite 2	EXP 2	Nanofilled	Matrix: Bis-GMA, DMAEMA, TEGDMA, camphorquinone (CQ) Filler: $BaAlSiO_2$microfiller (average particle diameter of 0.4 μm) and $BaAlSiO_2$nanofiller with average grain size of 175 nm Filler loading: 73.6 wt%		

Specimen preparation

All the resin composites were packed into a PTFE mold (10 mm in diameter and 3 mm in thickness) on a mylar sheet. After packing the composites, another mylar-formed surface was covered and a 1 mm glass microscope slide was pressed on the top of the specimen to exclude excessive resin material and to eliminate the air bubbles. All the specimens were then light cured for 20 s with a LED light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA). From each resin composite groups, two groups of twenty-five specimens each, two hundred specimens in total, were prepared at 22.0–22.5°C (room temperature) and relative humidity of 50%. Each group was divided into five subgroups of five specimens each. Fifty specimens were prepared for each composite. Hand-curing group was light-cured with LED light-curing unit and additional post curing group was light-cured with LED light-curing unit and received additional post-curing in a heat-light-curing device (Targis Power, Ivoclar AG, Schaan-FL, #TP1 1103509) in program P1, 95°C for 25 min. The same operator prepared all specimens.

The schematic study design was demonstrated in Fig. 1. Five specimens in each group were served as control without any polishing procedures after removing the mylar strip. In two of the fifth polishing groups, the specimens were polished using an automated polishing machine (Struers, LaboPol-21, Copenhagen, Denmark) with #4000-or #320-grit SiC paper under water irrigation. The specimens were cleaned using an ultrasonic cleaner (Quantrex 90; L&R Ultrasonics Manufacturing Company, Kearny, NJ, USA) with deionized water for 2 min. In the fourth group, polishing was done with rubber-based silicon abrasives (Lot#3142679; Top Dent, DAB Dental, Upplands Väsby, Sweden) and in the fifth group, polishing was done with a three-step polishing system (SwissFlex, Coltène Whaledent AG, Altstätten, Switzerland). For SwissFlex and rubber-based silicon abrasive groups, a slow-speed handpiece was used with light pressure rotating at approximately 10,000 rpm.

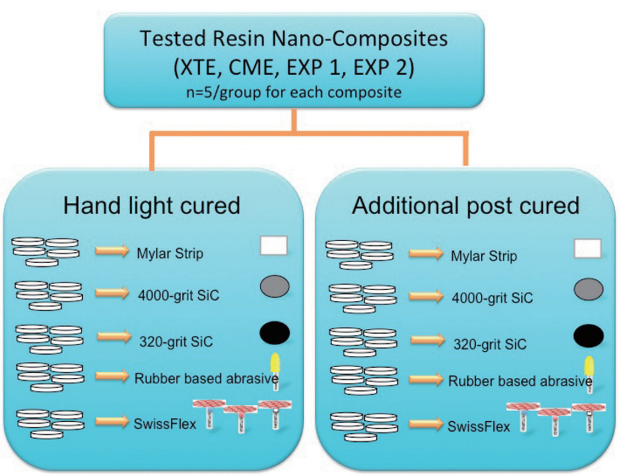


Fig. 1 Schematic study design.

The discs were used for 15 s for each polishing sequence as recommended by the manufacturer. Polishing discs were discarded after each use. However, their use has some limitations due to their geometry, being difficult to obtain completely flat surface on the specimens. In this study only flat surfaces were evaluated. The final thickness of all the specimens (2±0.1 mm) was measured with a digital calliper (Liaoning MEC Group, Dalian, China). Then the discs were stored in 30 mL deionized water in black containers for 1 and 7 months.

Color difference calculations

Color measurements of the specimens were performed on the day of specimen preparation (base) and repeated 1 and 7 months after deionized water storage.

ΔE^* values of specimens was measured according to the CIE 1976 L^* a^* b^* color scale relative to the CIE standard illuminant D_{65} (as defined by the International Commission on Illumination) which corresponds to "average" daylight (including ultraviolet wavelength region with a correlated color temperature of 6504 K) with a reflection spectrophotometer (CM-700d, Konica Minolta Sensing, INC. Tokyo, Japan) on the black backgrounds with SCI geometry. The measuring head of the spectrometer had a 3 mm diameter measuring area and used diffuse illumination and a viewing condition was CIE diffuse/8°geometry. Zero and white calibrations of the equipment were done immediately before each set of measurements using calibration plate, supplied by the manufacturer. Measurements were repeated three times for each specimen under each color measuring condition, and average values for the five specimens of the same group were calculated using the software was Spectra-Magic Version 2.11 (Konica Minolta, Tokyo, Japan).

Differences in color between base and 1 month, and base and 7 months storage were calculated by the equation as follows:

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

Scanning electron microscopy (SEM)

Two specimens from each polishing group in both curing subgroups were selected for SEM analysis. The specimens were sputtercoated (Bal-Tec SCD 050 Sputter Coater; Bal-Tec, Liechtenstein) with gold and observed with a scanning electron microscope (SEM, JSM-5500; JEOL, Tokyo, Japan).

Statistical analysis

Statistical analysis was performed by Statistical Package for Social Sciences (SPSS) 11.5 software (SPSS Inc., Chicago, IL, USA). Multivariate analysis of variance (MANOVA) taking such following factors as resin composites (4 levels –XTE, CME, EXP1, EXP2), curing methods (2 levels –Hand Curing, Additional Post Curing), polishing methods (5 levels, Mylar, #4000-, #320-grit SiC, rubber based silicone, SwissFlex) at each of storage time period (1 and 7 months) was performed for the evaluation of the data. Additionally, data were

analyzed by post hoc Tukey's tests when the effect of the main factors or the interaction reached significance. A p value less than 0.05 was considered statistically significant. But, for all possible multiple comparison tests, Bonferroni Adjustment was applied to control Type I error.

RESULTS

Color stability results

Mean ΔE^* values between base and 1 month, and base and 7 months storage and standard deviations (SD) are shown in Figs. 2 (a) and (b). Statistical analysis revealed that there was a significant difference amongst the resin composites ($p<0.05$), curing protocols ($p<0.05$), polishing methods ($p<0.05$), and storage time ($p<0.05$) after 1 month. The interactions between resin composites×polishing method ($p<0.0025$), resin composite×curing method ($p<0.00625$) have a statistically significant effect on ΔE^*. However, no statistically significant effect of polishing method×curing method ($p=0.063$) or the triple interaction between resin composite×polishing method×curing method ($p=0.083$) was shown. MANOVA demonstrated that color difference values were ranked in resin nano-composites as follows: EXP2>EXP1>CME≥XTE. Additionally, the specimens subjected to additional post curing showed significantly higher color change than the hand-light cured ones. According to the statistical analysis, the ranking in polishing method groups were as follows: Mylar>#320≥#4000≥Rubber≥SwissFlex.

Furthermore, after 7 months, a significant difference was observed amongst the water storage of resin composites ($p<0.05$), curing protocols ($p<0.05$), polishing methods ($p<0.05$), and storage time ($p<0.05$). The interactions between resin composites×polishing method ($p<0.0025$), resin composite×curing method ($p<0.00625$), polishing method×curing method ($p<0.005$) were statistically significant. No statistically significant effect of the triple interaction between resin

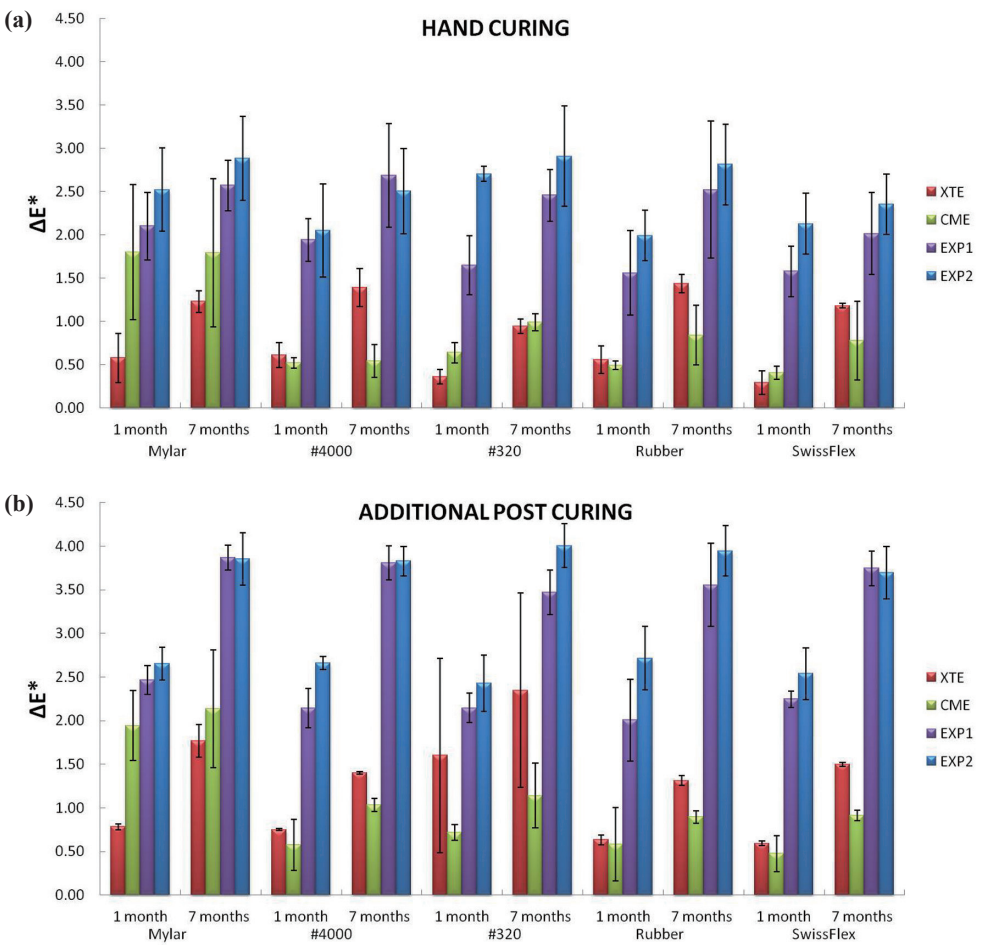


Fig. 2 (a) Color difference (ΔE^*) results of hand cured resin composites after the application of different curing protocols and polishing methods in storage times.
(b) Color difference (ΔE^*) results of additional post cured resin composites after the application of different curing protocols and polishing methods in storage times.

composite×polishing method×curing method (p=0.371) was demonstrated. Aside from 1 month results, the color difference values were ranked in resin nano-composites as EXP2≥EXP1>XTE>CME. Additional post curing resulted in significantly higher color change than the hand-light curing protocol. Furthermore, the ranking in polishing method groups were as follows: Mylar≥ #320≥#4000≥Rubber≥SwissFlex. After 1 month, overall ΔE^* values of resin nano-composites were 0.677±0.064 for XTE, 0.816±0.058 for CME, 1.984±0.052 EXP1 and 2.440±0.052 for EXP2 whereas, the values were 1.451±0.076 for XTE, 1.106±0.069 for CME, 3.070±0.062 for EXP1 and 3.279±0.062 for EXP2 after 7 months.

SEM observation

SEM micrographs showed different surface characteristics in mylar and Swissflex groups (Figs. 3, 4). Furthermore, different compositions and filler content of the tested resin composites were observed (Fig. 5). XTE contained nanofillers and mostly spherical nanoclusters. Homogeneous distribution of fine-milled submicron fillers with good integration into the surrounding resin matrix was seen. Additionally, CME contained different fillers in very different sizes. Inhomogeneous filler distribution with relatively large fillers than that of XTE, EXP1 and EXP2 was identified by SEM observations. Experimental groups (EXP1 and EXP2) demonstrated homogeneous fine-milled submicron filler composition. Voids within the matrix were observed (Fig. 5).

DISCUSSION

In this study, the color differences of resin nano-composites subjected to different curing protocols and polishing systems were measured at 1 and 7 months water storage by using a spectrophotometer with SCI geometry. A previous study by Lee *et al.*[23] demonstrated that color differences caused by the difference in the illuminant varied depending on the measuring geometry. However similar trends regardless of measuring geometries (SCI and SCE) had been shown[23].

In the current study, four resin composites were chosen. Half of the specimens were cured by hand light-curing and other half received additional heat-light curing. After curing protocols, all of the specimens were roughened with identical procedures. According to

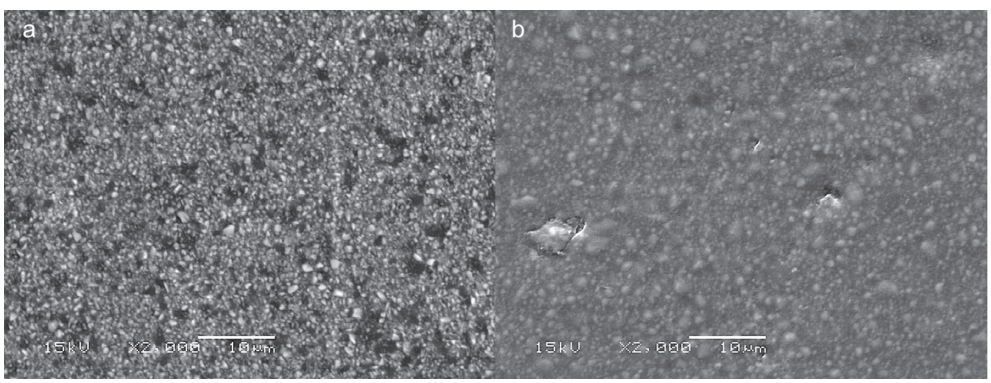


Fig. 3 SEM-photomicrographs of the commercial nano-composites after hand-light curing protocol in Mylar group (original magnification ×2000, bar=10 µm).
(a) Filtek™ Supreme XTE (XTE), (b) Clearfil Majesty™ Esthetic (CME)

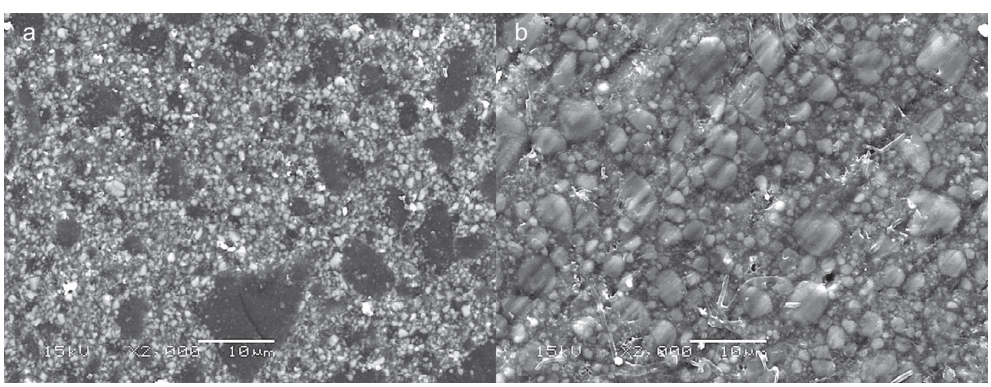


Fig. 4 SEM-photomicrographs of the commercial nano-composites after hand-light curing protocol in SwissFlex group (original magnification ×2000, bar=10 µm).
(a) Filtek™ Supreme XTE (XTE), (b) Clearfil Majesty™ Esthetic (CME)

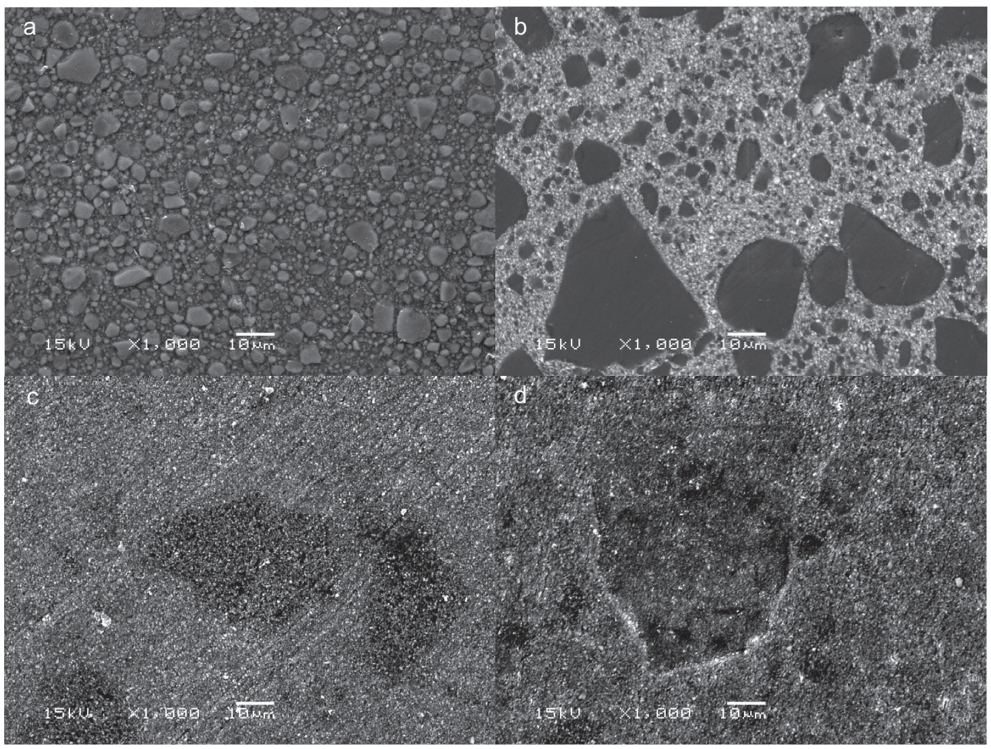


Fig. 5 SEM-photomicrographs of the tested resin composites after hand-light curing protocol in 4000- SiC paper group (original magnification ×1000, bar=10 μm).
(a) Filtek™ Supreme XTE (XTE), (b) Clearfil Majesty™ Esthetic (CME),
(c) Experimental composite 1 (EXP1), (d) Experimental composite 2 (EXP2)

the both 1 and 7 months results of the current study, additional post-curing increased the color change of composite specimens significantly. A previous study by Rueggeberg *et al.*[24] reported that additional post-curing caused color changes on Bis-GMA-based and Bis-GMA/urethane based composites. They concluded that the degree of color change was influenced by the amount of resin content in the composite systems rather than the particular resin composition. On the contrary, a previous study by Eldiwany *et al.*[25] reported that once the composites were light-cured, additional post-curing caused no further perceptible changes in their shade. In the present study, 1 month color change results of tested composites cured by both hand and additional post curing, after polishing procedures were in the range of 0.16 to 3.01, which were considered imperceptible clinically (ΔE^*<3.3). On the contrary, after 7 months, ΔE^* results of additional post curing, varied by the resin composites (from 0.27 to 4.26) were significantly different.

Previously, Powers *et al.*[26] reported that the color stability of restorative resins have been attributed to the some causes such as water sorption, chemical degradation, oxidation of the unreacted carbon double bonds, and surface roughness. Color change after additional post heat- and light curing may be correlated with chemical degradation. Besides, water sorption may also enhance the color stability of additional cured composites negatively, since the composites are also capable of absorbing other fluids with pigments, resulting in discoloration[27]. Moreover, it is assumed that water acts as a conductor for the pigment and stain penetration into the resin matrix[27,28]. Excessive water sorption may lead to the expansion and plasticization of the resin component, and cause micro-crack formation[29]. As a result, the micro-cracks or interfacial gaps at the interface between the filler and matrix allow stain penetration and discoloration[27,30]. The present study was intended to evaluate color changes associated with factors inherent to the material composition, polymerization protocols, different finishing and polishing methods and water sorption rather than the result of extrinsic staining factors on the composites. A controlled environment such as distilled water was used as storage media for this purpose.

The present overall results demonstrated that the changes in color were significant among the type of nano-composites. EXP2 showed the highest color change whereas XTE and CME exhibited the lowest after both storage times. This may be related to the difference in material composition, especially filler content and distribution. Furthermore, XTE showed lower color change than that of CME, after 7 month whereas the color changes of both resin composites were similar after

1 month. These results are likely associated with the inherent microstructural differences between these two composites. Nano-composites are formulated using both nanoparticle and nanocluster fillers. Nanoparticles are discrete nonagglomerated and nonaggregated particles of 20 nm in size. Nanocluster fillers are loosely bound agglomerates of nano-sized particles. The primary particles, not the clusters themselves, can be worn away during the polishing[10]. XTE contains 20 nm silica particle and predominantly 600–1,000 nm zirconia-silica nanoclusters (composed of 4–20 nm silica and zirconia nano particles). CME contains 70 nm silanated barium glass fillers and pre-polymerized organic filler including nano filler of 0.2–100 μm with average particle size of 0.7 μm. Therefore, the difference in color change between XTE and CME may be due to not only the surface conditioning technique but also to the filler type, filler size and the amount of the clusters in each resin composite. This finding is in line with the results of a study[31] about the effects of specular component on color differences of different filler type resin composites.

A previous study reported that the accelerator material also influenced the color stability of restorative resins[32]. In most current light-cured resin composites, camphorquinone (CQ) is a typically used compound as a visible light-activated free-radical photoinitiator. It absorbs blue light before curing and generates free radicals. Moreover, the concentration of CQ limits the degree of polymerization[33]. The cause of higher color change in ΔE^*values of experimental composites of the present study might be related to their CQ contents.

The results of the current study also re-confirmed by the previous reports[34,35] that demonstrates the finishing and polishing procedures should be applied to obtain more resistant composite surface to discoloration. All Mylar-finished specimens showed significant color change when compared to the polished specimens in 1 month storage time. Also, in 7 months storage time, mylar and #320 groups gave the highest color change results. Ergucu *et al.*[34] reported that the use of a Mylar strip not only resulted in a smooth surface finish, but it also eliminated the presence of an uncured layer on the surface. However, the surface beneath the strip was speculated not to have the same degree of polymerization as the bulk of the resin composite[34]. Furthermore, Gordan *et al.*[36] demonstrated that, compared with other finishing treatments, Mylar strip finishing results in surfaces with the lowest hardness, which is evidence of a lower degree of polymerization on the surface. In this study, experimental groups (EXP1 and EXP2) demonstrated the highest color change in mylar group due to their higher filler concentration. This could be related with the porous structures of the experimental groups.

In the context of the present study, its results suggested that further long-term clinical investigations are necessary to investigate the short and long term effect of additional post curing and polishing systems on degree of conversion and polymerization characteristics of resin composites.

CONCLUSIONS

Within the limitations of this *in-vitro* study, following conclusions could be drawn:

1. Type, composition, curing protocols and polishing methods of resin composites may influence the color characteristics of the restorations.
2. Additional post-heat-curing of dental nano composites may produce higher color change than the hand-light curing protocol.
3. The finishing and polishing procedures should be applied to obtain more resistant composite surface for its color stability.

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