

## Color Stability of Three Composite Resins following Accelerated Artificial Aging: An In-Vitro Study

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### Abstract

**Background and Aim:** Discoloration is among the most common problems of composite restorations. Color change over time compromises the main advantage of composite resins namely their high esthetics. In such cases, the restoration needs to be replaced. The aim of this in-vitro study was to evaluate the effect of accelerated artificial aging (AAA) on the color stability of three composite resins (Filtek Z250, Filtek Z250XT, and Filtek Supreme).

**Materials and Methods:** In this experimental study, 7 composite specimens with equal dimensions were fabricated of each composite resin. The initial color of specimens was measured using a spectroradiometer according to the CIE L\*a\*b\* system. The specimens were then submitted to AAA for 384h and underwent color assessment again. Before and after aging, the surface roughness of one specimen from each group was determined by Atomic Force Microscopy (AFM). The obtained color parameters were analyzed by one-way ANOVA and Tukey's test.

**Results:** The color change of Filtek Z250 was significantly lower than that of Filtek Z250XT and Filtek Supreme ( $p \leq 0.05$ ). No statistically significant differences were found between Z250XT and Supreme in this respect ( $p > 0.05$ ).

**Conclusion:** All composite resins showed color change above the clinically acceptable threshold ( $\Delta E \geq 3.3$ ). Z250 microhybrid composite was more color stable than nano-composites (Z250XT and Supreme). AAA increased the surface roughness in all groups but it was within the clinically acceptable range.

**Key Words:** Color stability, Composite, Aging

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### Introduction

In the recent years, application of composite restorations has increased due to higher patient demands for esthetic treatments, simplified bonding steps, improved properties of composite resins and advances in their manufacturing process [1]. One of the most important problems of composite restorations is their gradual color change and mismatch with the adjacent teeth over time [2]. More than 80% of patients complain of color mismatch between their composite restorations and adjacent teeth [3]. In such cases, the main advantage of

composites, their esthetic property, is lost and the restoration needs to be replaced [2, 4]. Composite restorations have several advantages namely less damage to the tooth structure and soft tissue and lower cost. However, their color match is extremely important [2].

Recent advances in the manufacturing of composite resins led to the production of nano-composites that are claimed to have superior mechanical and visual properties, higher esthetics, high wear resistance and low polymerization shrinkage [5-7]. The composition of composite



resins and their filler properties may directly influence the external stainability of the restoration. Moreover, resin composites are composed of different organic constituents that may be responsible for internal discoloration [4].

Surface roughness of composite restorations is a critical clinical parameter responsible for plaque accumulation, stainability and wear [5]. The acceptable threshold of surface roughness for restorative materials is  $0.2\mu$  [1]. Surface roughness higher than this threshold is associated with an increased risk of plaque accumulation, gingival inflammation, caries and staining [1, 3].

AAA is a suitable technique to assess the effect of aging on physical, chemical and optical properties of non-metal restorative materials like dental composites [2]. AAA simulates a destructive environment similar to the oral cavity and is used to predict the durability of materials via simulating chemical and physical reactions that occur in the oral environment. The role of saliva is simulated by 100% humidity and UV light is used for simulating daylight [8-9]. Considering all the above, this study sought to assess the effect of AAA on the color stability of 3 commonly used composite resins (a microhybrid, a nanofilled and a nanohybrid) under in-vitro conditions.

### Materials and Methods

In this experimental study, 7 disc-shaped specimens measuring 9mm in diameter and 2mm in thickness were fabricated of Z250 microhybrid (3M ESPE, USA), Supreme nanofilled (3M ESPE, USA) and Z250XT nanohybrid (3M ESPE, USA) composite resins.

A list of the 3 composite resins and their constituents is shown in Table 1. A metal mold was placed on a Mylar strip over a glass slab and filled with composite resin. A Mylar strip was placed over the mold and packed by another glass slab. Composite specimens were then light-cured by a QTH light-curing unit (Coltolux 2.5, Coltene, USA) with  $600\text{mW}/\text{cm}^2$  intensity using the overlapping technique each time for 40s to achieve uniform light distribution. Specimens were removed from the molds and polished with 360, 600 and 1200 grit abrasive paper discs (SofLex, 3M ESPE, USA), respectively.

Initial color of specimens was obtained using a radiospectrometer (CS-2000, Konica Minolta, USA) according to the CIE  $L^*a^*b^*$  system.  $L^*$  indicates lightness and its values run from black to white;  $a^*$  axis extends from green to red and  $b^*$  from yellow to blue. ( $A^*b^*$ ) indicates chroma.

In each group, the surface roughness of one specimen was measured using AFM (DME, Dual Scope-C-26, Denmark). Specimens were then subjected to AAA in QUV/Spray (USA) under ASTM G 154-06 standard. In this machine, specimens were subjected to alternating cycles of UVA fluorescent lighting with 340 nm wavelength and  $0.89\text{ W}/\text{mm}^2$  intensity for 8h at  $60^\circ\text{C}$  followed by 4h in 100% moisture in the dark at  $50^\circ\text{C}$ . This process was repeated alternatively for 384h corresponding to one year of clinical service. Next, final color assessment was performed using a radiospectrometer (CS-2000, Konica Minolta, USA). Surface roughness was measured again by AFM (DME, Dual Scope-C-26, Denmark). Data were analyzed using one-way ANOVA and Tukey's test.

### Results

This study assessed the color stability of Filtek Z250XT, Filtek Z250 and Supreme composite resins after AAA. The color parameter values before and after AAA are shown in Tables 2 and 3, respectively. Changes in these values after AAA are demonstrated in Table 4.

One-way ANOVA and Tukey's test were used for data analysis. One-way ANOVA showed significant differences between groups ( $p < 0.05$ ).

Assessment of changes in color parameters after AAA by Tukey's test revealed that:  $\Delta L$  of Z250 was significantly higher than that of Supreme and Z250XT ( $p = 0.001$ ); no significant difference was found in this respect between Supreme and Z250XT ( $p = 0.94$ ).  $\Delta a$  in Z250 was significantly lower than in Supreme and Z250XT ( $p = 0.001$ ) but no significant difference was found between Supreme and Z250XT ( $p = 0.11$ ).  $\Delta b$  in Z250 was significantly lower than in Supreme and Z250XT ( $p = 0.001$ ) but no significant difference was found between Supreme and Z250XT ( $p = 0.38$ ).  $\Delta E$  in Z250 was significantly lower than that in Supreme and Z250XT ( $p = 0.001$ ). Although this parameter in Supreme was higher than in Z250XT, this dif



**Table 1.** Characteristics of the 3 composite resins

Material	Type	Manufacturing company	Color	Constituents	Filler content (w%)
Filtek Supreme	Nanofilled	3M ESPE	A <sub>2</sub> Body	Matrix: UDMA, Bis-EMA, Bis-GMA, PEGDMA, TEGDMA Filler: Silica nanofiller (5-75nm), zirconia silica nanocluster (0.6-1.4µm)	78.5%
Filtek Z250XT	Nanohybrid	3M ESPE	A <sub>2</sub>	Matrix: UDMA, Bis-EMA, Bis-GMA, PEGDMA, TEGDMA Filler: Silica nanofiller (20nm), zirconia silica (≤3µm)	82%
Filtek Z250	Microhybrid	3M ESPE	A <sub>2</sub>	Matrix: UDMA, Bis-EMA, Bis-GMA Filler: zirconia silica (0.01-3.5µm)	84.5%

**Table 2.** The minimum, maximum, mean and standard deviation of color parameters before aging in different composites

Before AAA Composite	Minimum			Maximum			Mean±SD			P value		
	b	a	L	b	a	L	b	a	L	b	a	L
<b>Z250</b>	20/36	-1/16	74/50	22/07	-0/29	80/25	21/02±0/49	-0/79±0/26	77/18±2/09			
<b>Supreme</b>	21/24	-0/01	78/55	22/13	0/66	82/26	21/72±0/32	0/28±0/19	80/52±1/27	≤0/001	≤0/001	≤0/001
<b>Z250XT</b>	18/32	0/50	77/85	22/46	1/09	80/83	20/88±1/25	0/83±0/19	79/51±0/90			

**Table 3.** The minimum, maximum, mean and standard deviation of color parameters after aging in different composites

Before AAA Composite	Minimum			Maximum			Mean±SD			P value		
	b	a	L	b	a	L	b	a	L	b	a	L
<b>Z250</b>	20/92	-0/59	65/37	21/82	0/19	70/53	21/27±0/29	-0/29±0/22	68/25±1/64			
<b>Supreme</b>	31/26	2/29	64/04	33/76	3/09	67/24	32/33±0/32	2/70±0/29	65/81±1/03	≤0/001	≤0/001	≤0/001
<b>Z250XT</b>	30/12	2/79	63/22	32/26	3/47	66/05	31/04±0/71	3/09±0/19	64/61±0/77			

**Table 4.** The mean and standard deviation of change in color parameters of different composites after AAA

After AAA Composite	Mean± SD				P value			
	ΔE	Δb	Δa	ΔL	ΔE	Δb	Δa	ΔL
<b>Z250</b>	8/95±2/23	0/24±0/36	0/50±0/22	-8/92±2/23				
<b>Supreme</b>	18/37±1/33	10/60±0/80	2/42±0/23	-14/71±2/01	≤0/001	≤0/001	≤0/001	≤0/001
<b>Z250XT</b>	18/25±0/96	10/15±1/66	2/25±0/31	-14/89±1/16				

ference was not significant ( $p=0.96$ ). Surface roughness of specimens before and after aging was evaluated using AFM with 50X magnification (Figure 1).

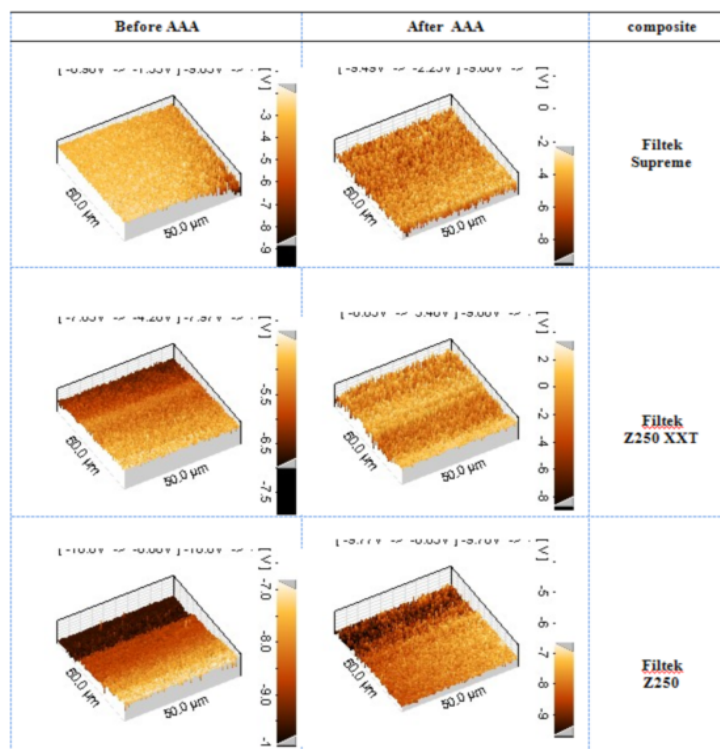
## Discussion

This study aimed to compare the color stability of Filtek Supreme nanofilled, Filtek Z250XT nanohybrid and Filtek Z250 hybrid composites after AAA. The results revealed that the highest color change occurred in nanofilled Filtek Supreme and

the lowest in Filtek Z250. The difference between Filtek Supreme and Filtek Z250XT in this respect was not significant. Also, the surface roughness of specimens increased after AAA.

Color change of composite restorations is influenced by intrinsic and extrinsic factors. The intrinsic factors involved include the resin matrix composition, type and percentage of filler particles, resin matrix-filler bond and the photo-initiator. The external factors include foods and physical stimuli such as the environment light [10, 11].





**Figure 1.** Surface roughness of specimens before and after AAA evaluated by AFM at 50X magnification

The standard AAA provides a combination of these factors and was used in our study to compare the color stability of 3 composite resins [10]. Based on the literature, AAA is suitable for the evaluation of changes in physical, chemical and optical properties of non-metal restorative materials like composite resins [2, 10, 12, 13]. Specimens were subjected to aging for 384h in QUV/Spray according to ASTM G 154-06 standard; which corresponds to one year of clinical service [12, 14]. However, some studies have shown that after 300h of AAA, changes in color parameters correspond to clinical changes in the oral environment after 2 years [13].

Silami et al. noticed that the  $b^*$  parameter increased in all groups indicating that the color of composite restorations turns yellow over time [15]. Pires-de-Souza et al. demonstrated that aging decreased  $\Delta L$  or brightness and increased chroma [16]. The results of afore-mentioned 2 studies are in accord with ours.

Internal factors namely the size, percentage and type of filler particles, type of initiator and its chemical composition affect the color stability of composites as well.

Comphorquinone/amine (CQ/amine) is the most commonly used photo-initiator incorporated into different composite resins in an amount of 0.1%-1%. The yellow color of CQ adds yellow to the composite shade and adversely affects the appearance and esthetics of composite restorations [9, 15-17]. On the other hand, decreasing its percentage reduces the degree of polymerization leading to a reduction in optimal mechanical properties, higher water sorption and less color stability. In order to control the unfavorable effects of CQ, amine accelerator is incorporated into composites; however, it can also lead to the darkening of composite restorations over time [15].

Another constituent affecting the color stability is TEGDMA resin monomer [12]. It has higher degree of polymerization but greater hydrophilicity than Bis-GMA and UDMA [18] leading to higher water sorption [16].

Studies have demonstrated that water sorption and penetration of fluids and solvents into the matrix or matrix-filler interface cause hydrolytic degradation and form additional voids in the polymer that per se increase the penetration of water molecules and other solvents leading to further destruction. On



the other hand, the described process can change light emission [10, 13, 14]. The difference in color stability of Z250 and nano-filled Supreme and Z250XT may be due to the presence of TEGDMA and PEGDMA monomers in nanofilled composites and their absence in Z250 [12]. The volume of resin also influences the color stability of composite restorations [18]. Supreme, with the greatest color change in our study, has higher content of resin and hydrophilic monomers.

Z250XT has higher filler content than Z250 (68v% versus 60 v%); but showed greater color change than Z250. Pires-de-Souza et al, [10] also showed that Tetric nanohybrid composites despite having more filler content (78 v%) undergo greater color change compared to Z250 (60v%) [10]. Higher volume of particles in composites decreases the degree of polymerization [14]. Thus, Z250XT due to higher volume of particles (68v% versus 60v%) may have lower degree of polymerization; which may be responsible for greater discoloration. Therefore, increased volume of particles by reducing the resin volume improves color stability and at the same time by decreasing the degree of polymerization negatively affects color stability.

Degree of polymerization depends on the type and intensity of the light curing unit, duration of polymerization, color, translucency and opacity of composite and its thickness [13, 16]. In Bis-GMA-based composites, degree of polymerization varies between 45-85%. In Bis-GMA and UDMA-based composites, degree of polymerization is 20% lower than other monomers [15]. Low degree of polymerization makes the material more susceptible to degradation. Consequently, color stability decreases and release of products such as methacrylic acid and formaldehyde may intensify the color change [2, 15].

In a study by Pires-de-Souza et al, Tetric nanohybrid composite after the first 8h cycle of AAA showed color change over the clinically acceptable threshold. Z250 after four 8h cycles of AAA demonstrated a color change over the clinically acceptable threshold [10]. Thus, higher  $\Delta E$  values obtained in our study may be attributed to the 384h aging (including 48 8h cycles). In a study by Schulze et al, obtained  $\Delta E$  values were lower than our rates; but duration of aging was 122h in their study and 384h in ours [17]. In another study Pires-

de-Souza et al. compared the color stability of silorane-based and methacrylate-based (Z250 and Z350) composites after AAA and all three showed  $\Delta E$  values over the clinically acceptable threshold [12].

In our study, surface roughness of specimens was evaluated using AFM. Aging increased surface roughness in all groups. However, the rates were still within the clinically acceptable range of 0.2 $\mu$ . In other words, despite an increase in surface roughness, it was still clinically acceptable [1]. Furthermore, it was found that composite discs may have microcracks without any clinical manifestation. Aging increased surface roughness by exposing the fillers and increasing the width and length of microcracks. This phenomenon may structurally weaken the composites and made them more susceptible to future discolorations. Our results are in accord with the findings of many previous studies evaluating the surface texture of composites and change in their optical properties after AAA [14, 17-19].

AAA exaggerates thermal, humidity and UV radiation conditions causing irreversible endogenous changes in the materials. As reported by Santos et al, [13] these conditions are exaggerated and do not exactly correspond to oral conditions. Considering the fact that AAA conditions are much more exaggerated than oral conditions, AAA protocol needs to be moderated to some extent to better simulate the oral environment. Moreover, as mentioned earlier, many other factors such as the type of light-curing unit directly and indirectly affect the color stability of composites and color change is the result of a combination of all these factors. Thus, future studies are recommended to assess the effect of type of light-curing unit on the color stability of composite resins.

## Conclusion

Within the limitations of this study, the following conclusions may be drawn:

1. 384h of AAA led to the color change of composite resins over the clinically acceptable threshold ( $\Delta E \geq 3.3$ )
2. Z250 microhybrid composite had greater color stability than nano-composites.



3.384h of AAA increased the surface roughness of understudy composites within the clinically acceptable range.

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