

## Effect of Two Different Aging Protocols on The Color and Translucency of Two Dental Resin Composite Materials

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### Abstract:

**Aim:** This study was carried out to evaluate the effect of mechanical fatigue and chemical aging protocols on color stability and translucency of two dental resin composite materials. **Method:** Twenty disc-shaped specimens were prepared from two types of dental resin composite material; nanohybrid (Filtek™ Z250 XT) and nanofilled (Filtek™ Z350 XT). The 20 specimens were further equally divided into two groups according to aging procedure. Mechanically aged and chemically aged nano hybrid groups were designated with Gp 1A and Gp 2A, respectively. While those of nano filled specimens were designated with Gp 1B and Gp 2B for the same aging treatments, respectively. Baseline readings for color and translucency were recorded before any aging treatment using Reflective spectrophotometer. For mechanical fatigue, the specimens were subjected to 6000 cyclic loading at 23.5 N using chewing simulator, while chemical aging was done by storing the specimens in acidic storage media at pH of 3.6 for 8 days. Using Reflective spectrophotometer, the change in color ( $\Delta E$ ) was measured and the color differences on a standard black and white backgrounds were used to calculate the translucency parameter ( $\Delta TP$ ). **Results:** After mechanical fatigue aging protocol, The change in color ( $\Delta E$ ) of Gp 1B was significantly higher than of Gp 1A, while chemical aging protocol resulted in insignificant difference in  $\Delta E$  of Gps 2A & 2B. Regarding change in translucency ( $\Delta TP$ ), there was a significant increase in translucency after both aging protocols. Translucency of mechanically

aged groups Gps 1A & 1B increased with no significant difference between them. However, after chemical aging protocol, Gp 2A showed a significant increase in translucency than did Gp 2B. **Conclusions:** Within the limitations of this study, mechanical fatigue aging protocol could produce a significantly higher change in color of dental resin composite compared to chemical aging protocol. Mechanical fatigue aging protocol could produce similar effects in translucency of both types of dental resin composite. In contrast, chemical aging protocol caused a significant difference in translucency of the nano hybrid composite material.

**Key words:** Nanohybrid Composite, Nanofilled Composite, Mechanical Aging, Chemical Aging, color, and translucency.

## 1. Introduction:

Failure of dental materials is an inevitable consequence to loss of their original mechanical and physical qualities during service in different oral environmental conditions. During their intraoral service, dental materials are confronted with many challenges including fluctuation in temperature and pH from meal intake or acids from bacterial metabolism, ongoing moisture exposure, and masticatory forces [1, 2].

Resin composites are direct filling materials designed to mimic tooth color when replacing defective dental coronal tissues enhancing the aesthetic results of dental treatments [3, 4]. The fundamental element of a resin composite is its oligomer matrix system, such as Bis-GMA, which has been utilizing for many years, or urethane dimethacrylate (UDMA), which can be used alone in many commercial products or blended with Bis-GMA in others. Low molecular-weight diluent monomers are also added by the manufacturer to the organic matrix, such as triethylene glycol dimethacrylate (TEGDMA), helping to lower the viscosity of the composite. Inorganic fillers are the second fundamental component representing the dispersed phase in resin composite. They vary in shape, size, and chemistry. They have a major role in enhancing the strength and modulus of the

organic resin matrix and reducing its the thermal expansion coefficient, water sorption, and polymerization shrinkage. As well, they impart radiopacity and enhance the esthetic properties [5, 6].

The ongoing advancement of nanotechnology in restorative dentistry enables a production of relatively new resin composites with marked improvement in properties. The common types of current formulations having nano modified resin composites comprise are Nano-filled and nanohybrid resin ones [7]. Dental nano-composite has nano-fillers with dimensions of 5 to 100 nm. Nano-hybrid composites are a category of dental restorative materials combining nanometer particles and 0.2 to 1  $\mu\text{m}$  particles of in their dispersed phase system [8].

It would be challenging and time-consuming to study the properties of the materials under the complicated oral environment for prolonged intraoral use. It would be crucial to have an accurate and quick way to replicate various oral situations and their individual effects on different materials. Hence, different aging treatments have emerged as techniques stimulating short term aggressive conditions under which dental materials would service, which may represent longer periods of normal intraoral service. Furthermore, they would offer distinctive stimuli that would enable researchers to analyze their particular impacts on the materials [9, 10].

Testing for cyclic fatigue more closely simulates the nature of loading forces produced during mastication [11]. As a result of these cyclic forces, cracks spread from pre-existing surface faults to cause additional localized damage until the material is no longer able to tolerate the loading circumstances [12].

Also, chemical substances in ingested food and drinks dramatically affect restorative materials. Intermittent exposure takes place when eating and drinking

till the teeth are cleaned. Lactic acid generation results from bacterial debris breakdown, exposing the restorative materials permanently to chemicals. Diffusion of chemicals into microscopic fissures of restorative materials leads to deterioration [13].

Mechanical fatigue and chemical degradation could have negative effect on the optical qualities of polymeric restorative materials. Among these qualities, translucency and color stability, if affected, would alter the perception of observer's eye to changes in the anterior restorations and hence negatively affect their aesthetics [14]. Translucency is a feature of substances that allows light to pass through but scatters the light so that things cannot be seen clearly through the substance; as such, it could be characterized as a state halfway between perfect transparency and opacity [15]. The translucency of restorative materials is evaluated using the translucency parameter (TP). The CIELAB color formula has been used to quantify translucency in the majority of studies on the topic in the dental literature [16].

The color of the resin composite material and the natural tooth must match perfectly for a restoration to be aesthetically pleasing that the human eye is unable to detect the difference. The choice of resin composite for restorative treatments is based on the color appearance of the material, including its color compatibility, color stability, and color interactions [17]. Improved optical qualities and color stability are crucial for creating aesthetically pleasing dental restorations [18].

This study was aiming at investigating whether color and /or translucency of nano filled and nano hybrid resin composites would be affected after subjecting them to aggressive aging conditions of mechanical fatigue and immersion in acidic medium, simulating a longer time service conditions of restorations intra orally.

The null hypothesis is that mechanical fatigue and chemical aging protocols do not affect the color stability and translucency of nano filled and nano hybrid resin composites.

## **2. Materials and methods:**

### **2.1. Materials:**

Two types of commercially available resin composite materials were used; a nano hybrid composite (Filtek™ Z250 XT, 3M ESPE, USA) and a nano filled one (Filtek™ Z350 XT, 3M ESPE, USA). The inorganic filler contents of both materials in weight percentage were 81.8% and 78.5% respectively, while the organic matrix of both materials contained bis-GMA, UDMA, TEGDMA, PEGDMA, and bis-EMA.

### **2.2. Methods:**

#### **2.2.1. Preparation of specimens and grouping:**

Twenty specimens from each composite type were prepared in a specially constructed split stainless steel molds with dimensions of 5 mm in diameter, and 2 mm thickness, figure 1. The composite materials were packed in this mold, covered with celluloid strips onto upper and lower sides and secured between 2 glass slaps [19]. Materials were light cured from both sides for 10 seconds according to the manufacturer's instructions using a light curing device (Mini LED, Satelec, Acteon, France) emitting blue visible light with wavelength of 400-500 nm and an intensity of 1,000 mW/cm<sup>2</sup>. The light curing tip was kept touching the surface of the glass slap during each curing cycle. A spectroradiometer was used to evaluate the curing unit's lighting intensity on a regular basis (Demetron Research Corp. USA). Flashes at specimens' edges were removed using a sharp lancet No. 24. Finally, whole specimens were stored in deionized water for 24 hours.

For each type of composite materials, specimens were randomly divided into two groups (n=10) according to the type of aging protocol; mechanically fatigued specimens (Gp 1) and chemically aged specimens (Gp 2). Specimens of nano

hybrid composite were designated with letter (A) while specimens those of nano filled composite were designated as (B).

Baseline readings of materials' color and translucency were taken from specimens of each material before subjecting them to any aging procedure, then specimens were reevaluated for same parameters after both aging protocols.

#### **Color difference ( $\Delta E$ ) measurement:**

The specimens' colors were measured using a Reflective Spectrophotometer (X-Rite, model RM200QC, Neu-Isenburg, Germany). The aperture size was set to 4 mm and the specimens were aligned in 45/0 configuration to measure reflection spectra. The white background was selected and measurements were made according to the CIE  $L^*a^*b^*$  color space relative to the CIE standard illuminant D65; which is the primary reference illuminant that emits an average amount of daylight including the UV wavelength area. After different aging tests, the color of discs was measured again and the color difference ( $\Delta E$ ) of the specimens was evaluated using the following formula:

$$\Delta E_{\text{CIELAB}} = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$$

Where:  $L^*$  = lightness (0-100),  $a^*$  = (change the color of the axis red/green) and  $b^*$  = (color variation axis yellow/blue) [20].

#### **Translucency parameter (TP) measurement:**

The translucency parameter (TP) values were obtained by calculating the color difference of the disc shaped specimens over standard black and white backgrounds by using the spectrophotometer according to the following equation:

$$\text{TP} = [(L_b^* - L_w^*)^2 + (a_b^* - a_w^*)^2 + (b_b^* - b_w^*)^2]^{1/2}$$

Where letters “b” and “w” refer to color coordinates over the black and white backgrounds, respectively. The  $L^*$  values of 0 to 100 represent a black and a reference white, respectively [21].

The translucency of the specimens was measured before and after different aging methods.

### **2.2.2. Mechanical fatigue aging protocol:**

In this aging protocol, specimens were subjected to 6000 cyclic loading under weight of 2.4kg (23.5 N); which is equal to half the ultimate strength of these samples as obtained from a pilot study [22]. The load was applied using a programmable logical controlled equipment; chewing simulator (Model ACH-09075DC-T, AD-TECH TECHNOLOGY CO., LTD., GERMANY), figure 2 (A). The prepared specimens were mounted in a cylindrical Teflon holder on the lower part of the simulator, and the load was applied centrally onto the upper surface of specimen with vertical and horizontal movements, figure 2 (B).

### **2.2.3. Chemical aging protocol:**

An acidic storage medium was specially prepared in the laboratory of Pharmaceutics Department, Faculty of Pharmacy, Minia University) according to **Mariano, N.A. et al., 2009** [23] and **Alzaid et al. 2023** [24]. The medium was composed of 1.68g sodium carbonate ( $\text{NaHCO}_3$ ), 0.426g disodium hydrogen phosphate ( $\text{Na}_2\text{HPO}_4$ ), 0.147g anhydrous calcium chloride ( $\text{CaCl}_2$ ) and 800 ml of water ( $\text{H}_2\text{O}$ ). The acidity of the solution was adjusted to be 3.6 pH by addition of 28 ml of lactic acid to the medium. Each two specimens were hanged inside a closed tube filled with 40 ml of the solution with dental floss to avoid touching walls and expose all sides to the solution evenly for 8 days without changing the solution [25].

### 2.3. Statistical analysis:

The collected data were coded, tabulated, and statistically analyzed using SPSS program (Statistical Package for Social Sciences) software version 25 Windows (SPSS Inc., Chicago, IL, USA). Statistically significant level was considered when calculated probability (p value) was  $\leq 0.05$ .

## 3. Results:

### 3.1. Color change of tested composite materials after mechanical fatigue and chemical aging protocols:

Generally, there was a change in the color ( $\Delta E$ ) of the two composites after both aging protocols. Chemically aged group showed a significant lower color change than mechanical fatigue aging one with mean values of  $\Delta E$  5 ( $\pm 1.3$ ) and 10.1 ( $\pm 1.9$ ) respectively, **figure 3**.

After subjecting specimens to mechanical fatigue aging protocol, the color change of the nanohybrid composite was significantly lower than that of the nanofilled composite with values of 8.9 ( $\pm 2.1$ ) and 11.2 ( $\pm 0.7$ ), respectively, with *p-value* of 0.048. Chemical aging protocol caused a lower change in  $\Delta E$  than mechanical fatigue aging; with values of 4.9 ( $\pm 1.3$ ) for nanohybrid composite and 5.1 ( $\pm 1.5$ ) for nanofilled composite with *p-value* of 0.860 indicating insignificant difference between them, **figure 4**.

### 3.2. Translucency parameter change ( $\Delta TP$ ) after mechanical fatigue and chemical aging protocols:

Generally, there was an increase in translucency compared to baseline values. Mechanically and chemically aged groups (Gps 1 & 2) recorded significant

increase in translucency with values of  $30.5(\pm 1.2)$  &  $30.6(\pm 7.3)$ , respectively, compared to baseline values  $25.3(\pm 2.4)$  *p*-value 0.959 indicating insignificant difference between aging protocols, **figure 5**.

Mechanically and chemically aged specimens of nanohybrid composite (Gps 1A & 2A) showed significantly higher translucency values of  $30.6(\pm 1.6)$  &  $36.2(\pm 4.9)$  with *p*-value of 0.015, respectively, than that of  $24.4(\pm 2.4)$  baseline value, **figure 6**.

Translucency of nanofilled composite increased significantly with *p*-value of 0.022 after mechanical fatigue aging protocol (Gps 1B) recording  $30.4(\pm 0.9)$ , while chemical aging protocol (Gp 2B) resulted in an insignificant decrease in translucency with value of  $25(\pm 4.4)$  with *p*-value of 0.551, compared to baseline value  $26.1(\pm 2.4)$ , **figure 6**. The differences in translucency parameter ( $\Delta TP$ ) values were calculated as “the value after aging – the value before aging”. Therefore, the changes in translucency ( $\Delta TP$ ) were 5.2 and 5.3 after mechanical and chemical aging protocols, respectively.

#### **4. Discussion:**

This study was conducted to find out whether mechanical fatigue and chemical aging protocols can change color and translucency of two types of dental resin composites; nanohybrid and nanofilled. The wide popularity and wide clinical use of these materials was the trigger to test them [26]. The tested materials were aged by mechanical fatigue and by immersion in reduced pH medium to evaluate color stability by measuring  $\Delta E$  and change in translucency by measuring  $\Delta TP$ . Color stability and change in translucency in this study were assessed because they directly correspond to the esthetical clinical success of aesthetic restorative materials [27].

Artificial aging is a procedure to mimic intraoral circumstances, where the material is exposed to mechanical and chemical stimuli, thereby undergoes a process of aging [10]. Several artificial aging procedures, including storage in artificial saliva, water, or different chemicals at different times and temperatures, and mechanical loading have been used in several research studies [9]. In both short- and long-term analyses, artificial ageing can be used to simulate materials' degradation and affect the materials' optical properties. The aging method can lead to changes in the interior chemical composition and/or influence surface integrity of the aged materials [28].

Aging protocols followed in this study were mechanical fatigue and chemical aging. Mechanical fatigue (cyclic loading) is a recommended aging protocol by many studies as it nearly simulates the nature of loading stresses that are produced during service [29]. According to **ÖZCAN M. et al., 2018**, tests on the fatigue behavior of dental restorations have been conducted using cycles with very wide range of loading cycles where no specific number of cycles is agreed upon [30]. Therefore, the number of 6000 cycles for the mechanical aging protocol was chosen according to previous studies [31, 32] where they are claimed to be equivalent to two weeks of intraoral service [33]. The directions of applied load were vertical and horizontal movements as it resembles the physiological chewing movements [34]. The applied load was equal to half of ultimate strength of the used materials as obtained from a pilot study for testing of non-aged specimens [30].

Regarding chemical aging, it was conducted as it is fast, easy and economic aging protocol. Moreover, in last decades, global statistics indicated that the consumption of carbonated soft drinks has increased dramatically [35]. These drinks are known of their high acidity with pH value range between 2.62 and 4.26 [36]. High acidic beverages intake has marked detrimental effect on resin composite restorative materials, and an acid attack may cause the matrix/filler interface to be destroyed [37]. **Korać et al., 2022**, found that 8 days immersion of dental composites in an acidic medium would be sufficient to produce the greatest changes; a significant portion of the unreacted monomers elute from the composite, with liquid filling residual free spaces [38] and softening and degradation of the resin matrix components such as the UDMA, TEGDMA, AND Bis-GMA [39]; in the aged materials, especially in their color.

The color was changed after both aging protocols. As mentioned by **Subramanya and Muttagi, 2011** [40], color change cannot be visually detected when values of  $\Delta E < 1$ , while values of  $1 < \Delta E < 2.3$  are identified by skilled individuals but accepted clinically, whereas values of  $\Delta E > 2.3$  are detectable by non-skilled observers, making this change not accepted clinically. As a result, in this study, both aging protocols caused clinically unaccepted color change.

The change in  $\Delta E$  after mechanical fatigue may be attributed to loss of filler from the surface due to generated microcracks at filler/ matrix interface [41]. This may lead to discontinuity in the surface. According to **Çobanoğlu et al., 2022** [42], instrumental color coordinates are affected by a sample's surface discontinuity. Surfaces irregularities of resin composites make them appear lighter and less chromatic than smooth surfaces do causing diffuse reflection conditions assessed by a spectrophotometer.

A low  $\Delta E$  after chemical aging protocol (Gp 2) may be attributed to chemical degradation which leads to softening and degradation of the outer surface layers of resins [14]. Mechanical aging protocol resulted in a higher color change in nanofilled composite specimens. This may be related to its higher amount of resin matrix which exhibited more deterioration, affecting the surface continuity [43]. Mechanical fatigue has more destructive effect compared to chemical aging on the bulk of the composite rather than solely surface discontinuity [44].

Translucency is one of the main governing criteria and a key aspect in choosing of materials in terms of aesthetics.

According to **lee YK. 2016** [45], clinical change in translucency can be evaluated by visual perceptibility threshold in translucency parameter difference ( $\Delta TP$ ) which is 2. Consequently, mechanically and chemically aged groups (Gps 1&2) caused perceivable increase in translucency. In this study, both aging protocols resulted in perceivable change in translucency in nanohybrid composite. While only mechanical fatigue aging protocol of nanofilled composite resulted in perceivable change in translucency. This may be attributed to the debonding of fillers due to mechanical aging [46] leading to change in refractive index of the composite [47]. Translucency change after chemical aging protocol might be attributed to the degradation of resin monomers and fillers debonding [48]. Nanohybrid composite has a higher amount of fillers [49]. Therefore, its refractive index was influenced by mechanical fatigue aging and chemical aging protocols.

Therefore, the null hypothesis has been rejected.

## **5. Conclusions:**

According to the limitations of this study, it can be concluded that mechanical fatigue for 6000 cycles (23.5 N) could produce more significant

change in color of the tested resin composite materials than did chemical aging for 8 days in media with a pH 3.6. In contrast, chemical aging could affect translucency of nanohybrid dental resin composite significantly more than mechanical fatigue.

#### **6. Recommendation:**

- It is recommended that patients who have their teeth filled with resin composite materials should be instructed to reduce their consumption of acidic drinks.
- Further investigations on the same types of composite materials with various immersion times, more numbers of cyclic loading, and long-term clinical follow-up are recommended.

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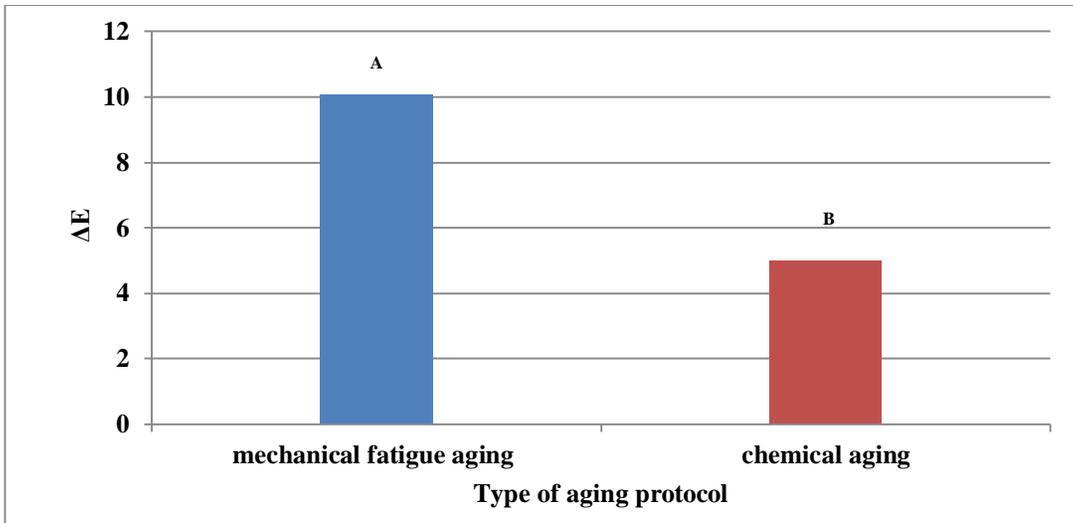
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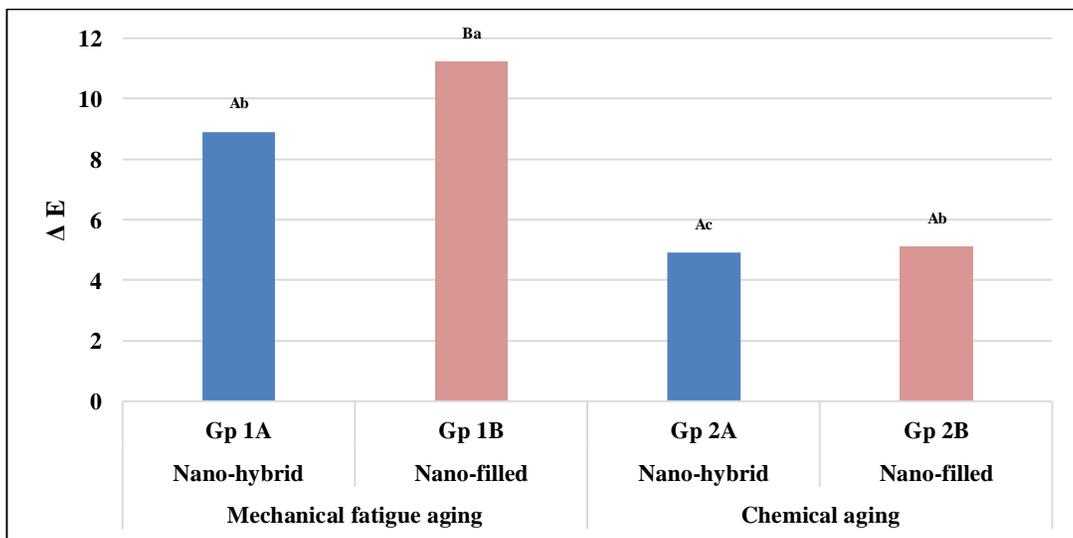
Figure 1: Split mold for disc sample preparation



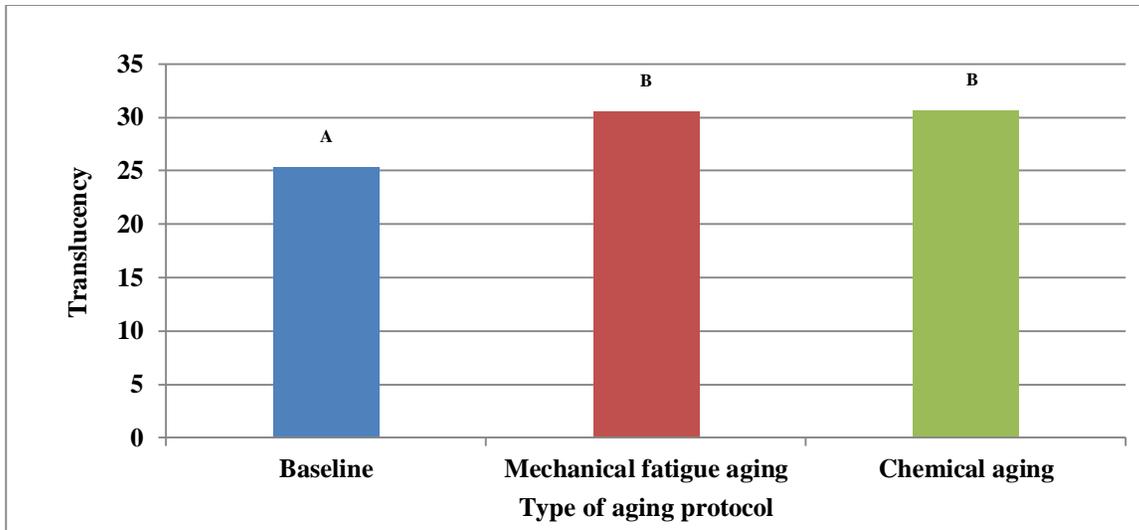
Figure 2: Chewing simulator device (A), disc sample ready for mechanical aging (B).



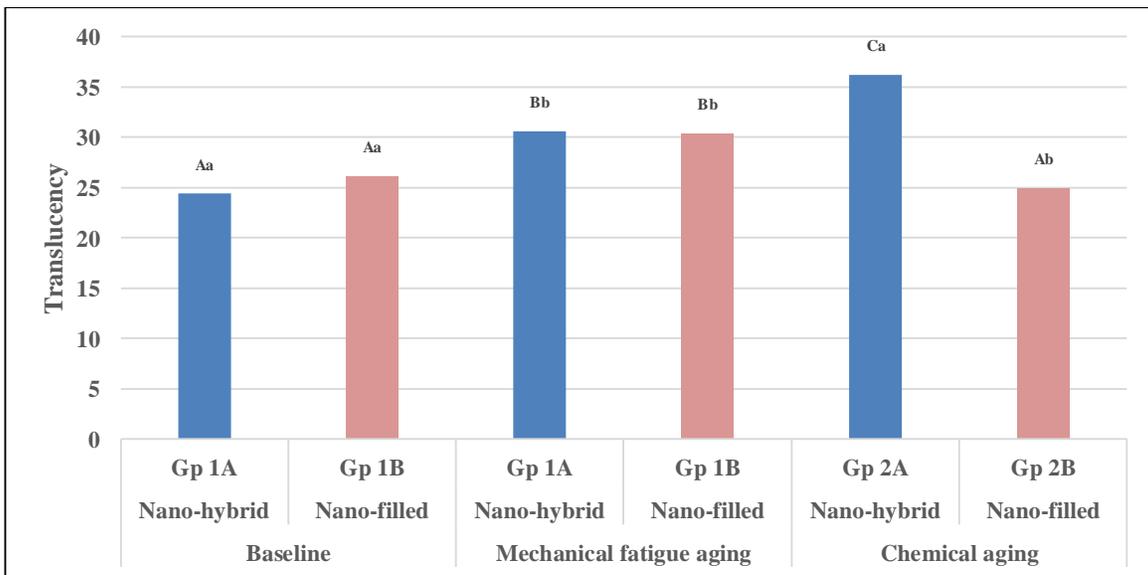
**Figure 3: A bar chart showing mean values of  $\Delta E$  after different aging protocols of the tested composite materials. Bars sharing same capital letters have statistically insignificant differences regarding aging protocol.**



**Figure 4: A bar chart showing mean values of  $\Delta E$  of different composite types in each aging protocol. Bars sharing same capital letters have statistically insignificant differences regarding aging protocol. Bars sharing same small letters have statistically insignificant differences regarding tested materials within each aging protocol.**



**Figure 5:** A bar chart showing mean values of translucency after different aging protocols of the tested composite materials. Bars sharing same capital letters have statistically insignificant differences regarding aging protocol.



**Figure 6:** A bar chart showing the mean values of translucency of different composite types in each aging protocol. Bars sharing same capital letters have statistically insignificant differences regarding aging protocol. Bars sharing same small letters have statistically insignificant differences regarding tested materials within each aging protocol.