Can We Use Different Artificial Aging Protocols Interchangeably of Dental Resin Composite Flexure Strength and Surface Microhardness Model?

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Abstract:
Aim: This study was conducted to determine whether the mechanical fatigue and chemical aging protocols could be utilized interchangeably when flexural strength and surface microhardness of dental resin composites are to be evaluated. In other words, do their effects on testing parameters; flexure strength and microhardness; are comparable to induce the same effect or not? Two types of resin composite material models; nanohybrid (FiltekTm Z250 XT) and nanofilled (FiltekTm Z350 XT) were used in this study. **Method:** Sixty specimens were prepared from each composite type. Rectangular specimens (n= 30) and disc shaped specimens (n=30) were divided into four groups as followed; (Gp 1A) Control group of nano hybrid composite specimens, (Gp 2A) mechanically aged nano hybrid composite specimens, and (Gp 3A) chemically aged nano hybrid composite specimens. Nano filled composite specimens were designated as Gp 1B, Gp 2B and Gp 3B for the same aging treatments respectively. Each group contained 20 specimens; 10 rectangular and 10 disc shaped. For mechanical fatigue, the specimens were subjected to 6000 cyclic loading at 23.5 N, while chemical aging was done by storing the specimens in acidic artificial saliva at pH of 3.6 for 8 days. The rectangular specimens were used for 3-point flexure strength evaluation using a universal testing machine at a crosshead speed of 0.5 mm/minutes. The disc shaped specimens were used for surface microhardness testing. The surface microhardness was measured by using Digital Display Vickers Micro-hardness Tester. **Results:** Both aging protocols induced significant reduction in the two testing properties; the flexure strength and the microhardness. Regarding flexure strength, Gps 2A & 2B did not differ significantly from each other, but were significantly weaker than Gps 1A & 1B. In addition, Gps 3A & 3B did not differ significantly from each other, but were significantly weaker than all former groups. The highest decline in flexure strength was recorded with Gp 2B. Microhardness testing revealed insignificant difference among Gps 1A, 1B and 2A. Groups 2B & 3B were significantly softer than the previous groups, but they did not differ significantly from each other. The last group, Gp 3A was significantly softer than all other groups. **Conclusions:** With the limitations of this study, it might be accepted to use chemical aging protocol interchangeably with mechanical fatigue aging protocol, only if we consider flexure strength parameter. For surface microhardness assessment, mechanical fatigue aging protocols cannot be used interchangeably.

Key words: Nanohybrid Composite, Nanofilled Composite, Mechanical Aging, Artificial Aging, Flexural strength, and Microhardness.

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1. INTRODUCTION:

Failure of dental materials occurs due to loss of their initial mechanical and physical properties as a function of oral environmental stimuli. Fluctuation in mouth temperature, exposure to chemicals with different pH from the dietary intake or acids from bacterial metabolism, continuous exposure to moisture as well as mechanical stimuli are common factors affecting martials' properties [1] [2].

Resin composites are direct filling materials invented to mimic tooth color where replacement of lost dental structures is required and promote esthetically pleasing outcomes during dental treatments [9] [10]**.** They are made up three major elements: a highly cross-linked polymeric resin matrix reinforced by a dispersion of glass, silica, crystalline, metal oxide fillers which are combined with matrix with silane coupling agents [11]. Also they contain photoinitiators, accelerators, and pigments [7].

Dental nano-composite has nano-fillers with dimensions of 5 to 100 nm. Nano-hybrid composites are a category of dental restorative materials where in addition to nanometer particles,

particles of 0.2 to 1 μm in size are added to the composite resins[12].

Studying properties of the materials after long term use intraorally and under complex nature of the oral environment would be difficult and time consuming. The need for a reliable and fast method to simulate different oral conditions and their separate effects on different materials would be critical. Hence, different aging protocols have been immerged, as a way to stimulate dental materials under sever service conditions at shorter time periods. Furthermore, they would provide specific and separate kinds of stimuli allowing studying their particular effects on the materials [3], [4].

Cyclic fatigue testing more closely approximates the type of loading stress that is generated during mastication [5]. Due to cyclic forces, cracks propagate from existing flaws, and extend to form other localized damage until the material can no longer withstand the loading conditions [6] .

Artificial saliva, acids or ethanol solutions have been used in studies to simulate chemical degradation. Saliva itself and enzymes may soften materials in addition to the chemical effects of various food items, such as fruit acids, fatty acids, and bacterial generated acids [7], [8] .

Douidar et al., 2022 defined hardness as “A material's resistance to persistent indentation or penetration when positioned against opposing tooth structure or materials”. It is used to demonstrate how resistant is a material to wear and abrasion. Due to its relationship to other physical properties, hardness is a mechanical property that should be taken into account while defining restorative materials [13].

Many researches judge the durability of different materials by subjecting their chemical, mechanical and/ or physical properties at basic parameters especially in-terms of temperature, pH, and mechanical loading to simulate clinical conditions.

The suitability of a particular aging method to mimic aggressive oral conditions on a specific dental material is usually the guiding key for researchers to select this aging method. Furthermore, the compatibility between the aging protocol and the expected properties to be affected by this simulated aggressive situation could be also another key. However, the ease of the aging protocols, their availability, their wide differences in simulation time as well as the need to complex and sophisticated equipment and consequently the expected cost are difficulties that might face the researchers during their studies. Hence, a question may emerge; is it possible to use easier, faster, and cheaper aging methods instead of more complex, time consuming and expensive methods to generate the same expected changes in the materials?

This study was conducted to answer the question; do mechanical fatigue and chemical aging protocols of dental resin composites can be used interchangeably when flexure strength and surface microhardness are to be evaluated?

The null hypothesis is that mechanical fatigue and chemical aging protocols cannot be used interchangeably to age resin composites before their flexural strength and microhardness evaluation.

2. Materials and methods:

2.1. Materials:

Two types of commercially available resin composite materials were used; nano hybrid composite (FiltekTm Z250 XT, 3M ESPE, USA) with a composition; 81.8% by wt. inorganic fillers and resins (bis-GMA, UDMA, TEGDMA, PEGDMA, and bis-EMA), and nano filled composite (FiltekTm Z350 XT, 3M ESPE, USA) with 78.5% by wt. inorganic fillers and organic matrix; (bis-GMA, UDMA, TEGDMA, PEGDMA, and bis-EMA).

2.2. Methods:

2.1.1. Mechanical tests:

2.1.1.1. Flexure strength test:

2.1.1.1.1. Preparation of samples:

Samples were prepared according to ethical committee No. 414, Faculty of Dentistry, Minia University. Thirty samples were from each composite type prepared in a specially constructed split stainless steel molds with dimensions of 25 ± 2mm in length, and a squared cross section with edge length of 2 ± 0.1 mm, 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[15]. 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 visible light with wavelength of 400-500 nm and an intensity of 1,000 mW/cm^2. 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). Packing of 5 mm increments in length was cured till full length of sample. Flashes at samples’ edges were removed using a sharp lancet No. 24. Finally, whole specimens were stored in deionized water for 24 hours.

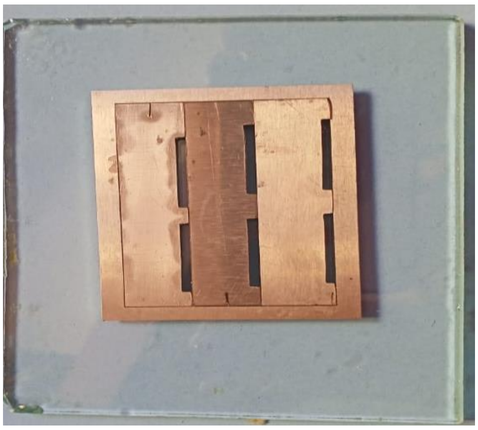

Figure 1: Split rectangular metallic mold.

2.1.1.1.2. Grouping of samples:

For each type of composite materials, samples were randomly divided into three groups according to the type of aging protocol; Control group (Gp 1): immediately tested after storage in deionized water for 24 hours, mechanically fatigued samples (Gp 2) and chemically aged samples (Gp 3). In each group, samples of nano hybrid composite were signed with letter (A) while samples of nano filled composite were signed as (B).

2.2.1.1. Mechanical fatigue aging protocol:

In this aging protocol, samples were subjected to 6000 cyclic loading under weight of 2.4kg (23.5 N) **[16]**which is equal to half of ultimate strength of the used materials as obtained from the testing of non-aged samples **[17] ,** using a programmable logical controlled equipment; chewing simulator (Model ACH-09075DC-T, AD-TECH TECHNOLOGY CO., LTD., GERMANY), figure 2. The prepared samples were mounted in a custom made chemically cured acrylic resin loading fixture, and the load was applied centrally onto the upper surface, figure 2.

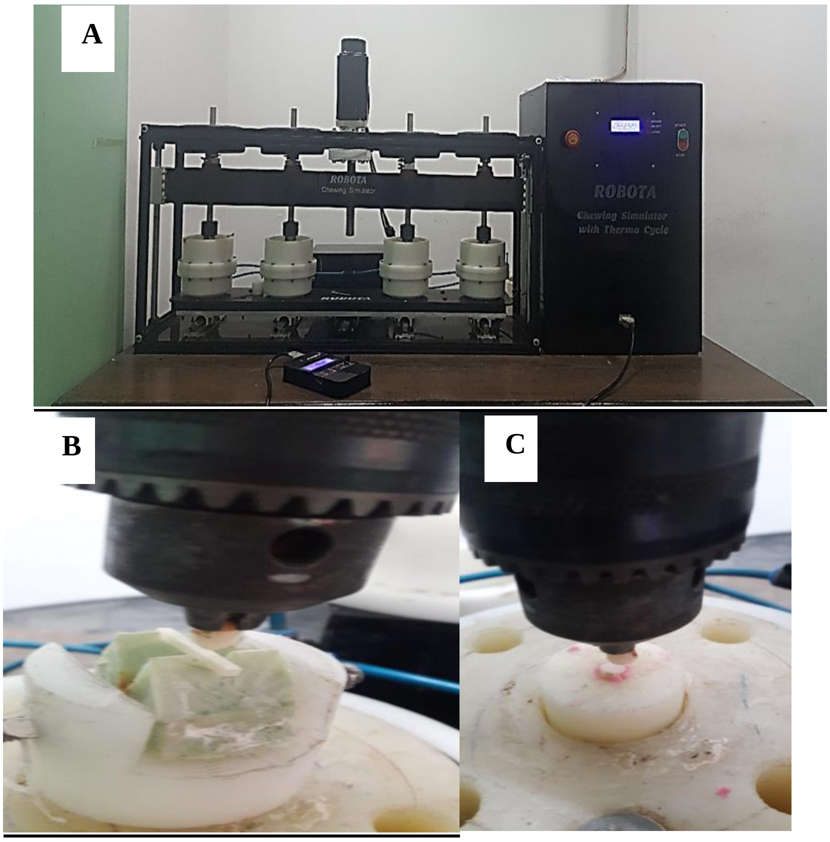

Figure 2: Chewing simulator device (A), a rectangular sample ready for mechanical fatigue aging (B), disc sample ready for mechanical aging (C).

2.1.1.1.3. Test procedure:

The test was conducted regarding to ISO No. 4049, 2000 [18]. The samples were subjected to 3 point bending test after their corresponding aging protocol. A specially constructed 3 point loading holder was fixed on the upper and lower jaws of the universal testing machine (Instron Industrial Products, USA (Norwood) moving at a crosshead speed of 0.5 mm/minutes. The load was applied to the samples at the center of distance between the 2 supporting points till fracture. The distance between the two supporting points was 20 mm. Flexural strength was calculated in MPa according the following equation:

$$\sigma = \frac{3FL}{2bd^2}$$

Where σ is the flexural strength, F is the maxim failure load in Newton, L is the distance between two supporting points, b and d are the width and the thickness of the sample, respectively.

2.1.1.2. Surface microhardness test:

2.1.1.2.1. Preparation of samples:

Thirty disc shaped samples from each type of composite were constructed using a cylindrical split stainless steel mold (5 mm in diameter and 2 mm in height). Packing and curing of composites were done as previously mentioned.

2.1.1.2.2. Grouping of samples:

Thirty samples from each resin composite material were randomly divided into three equal groups as described in flexure strength, 10 each.

2.1.1.3. 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** [19] and **Alzaid et al. 2023** [20] **.** The medium was composed of 1.68g sodium carbonate ($NaHCO_3$), 0.426g disodium hydrogen phosphate (Na_2HPO_4), 0.147g anhydrous calcium chloride ($CaCl_2$) and 800 ml of water (H_2O). The acidity of the solution was adjusted to 3.6 pH by addition of 28 ml of lactic acid. 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 [21].

2.1.1.3.1. Test procedures:

Surface Micro-hardness was determined using Digital Display Vickers Micro-hardness tester (Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd. China), figure 3. A Vickers diamond indenter was applied under a load of 100 g for 20 seconds. For each disc sample, the mean of 3 indentations was calculated. These 3 indentations were equally placed over a circumference of a circle 1.81 mm in radius so that they were apart from each other by at least 0.5mm. The diagonals lengths of the indentations were measured by a built in scaled microscope at magnification of 20X and Vickers values were converted into micro-hardness values, figure 3. Micro-hardness was obtained using the following equation:

$$HV = 1.854\frac{P}{d^2}$$

Where, **HV** is the Vickers hardness in Kgf/mm^2, **P** is the load in Kgf and **d** is the length of the diagonals in mm [22].

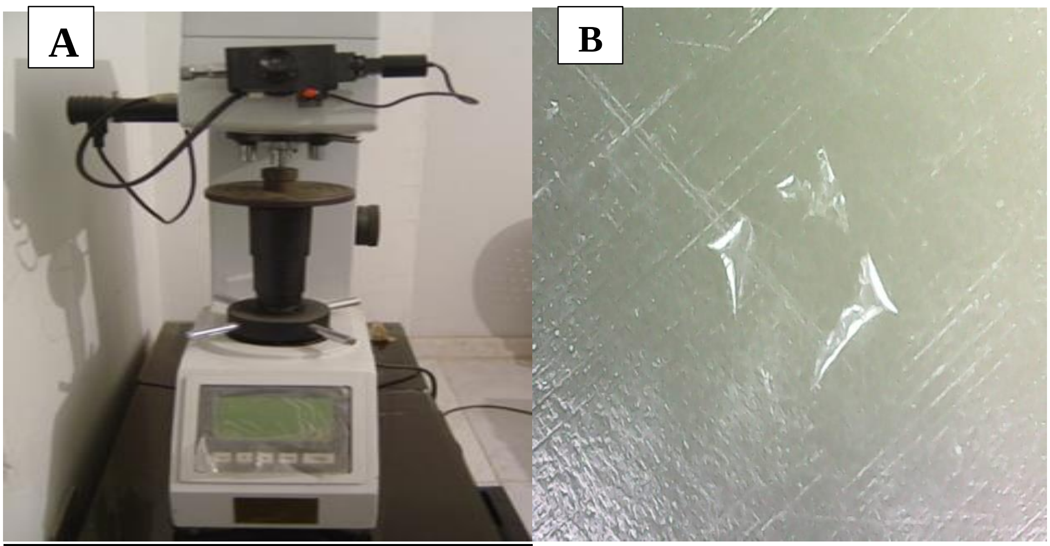

Figure 3: Vickers micro-hardness tester (A), indentations on the surface of sample at 20X (B)

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 ≤ 0.05.

3. Results:

3.1. Flexural strength of tested composite materials after mechanical fatigue and chemical aging protocols:

The control group (Gp 1) showed average flexural strength of 180.9 (±22.1) MPa. After subjecting samples to either aging protocol, there was significant reduction in their flexural strength. Samples subjected to mechanical fatigue (Gp 2) showed average flexural strength values of 137.7 (±14) MPa, while those subjected to chemical aging (Gp 3) recorded average values of 137.5 (±15.6) MPa. The difference among the later groups was statistically insignificant; Figure 4.

The flexural strength of both types of control unaged composite materials was statistically insignificantly different with average values of 184.3 (±20. 2) and 177.6 (±25.8) MPa for nano hybrid (Gp 1A) and nano filled (Gp 1B), respectively. After either mechanical fatigue or chemical aging, nano filled composite groups (Gps 2B & 3B) were significantly weaker than nano hybrid composite groups (Gps 2A & 3A). After mechanical and chemical aging, nano hybrid composite samples (Gps 2A & 3A) recorded average values of 148.4 (±3.6) and 144.5 (±10.1) MPa, respectively, while the average values of nano filled composite samples (Gps 2B & 3B) were 127 (±11.9) and 130.5 (±18.1) MPa, respectively. The flexural strength of each material was not affected significantly by the type aging protocol; Figure 5.

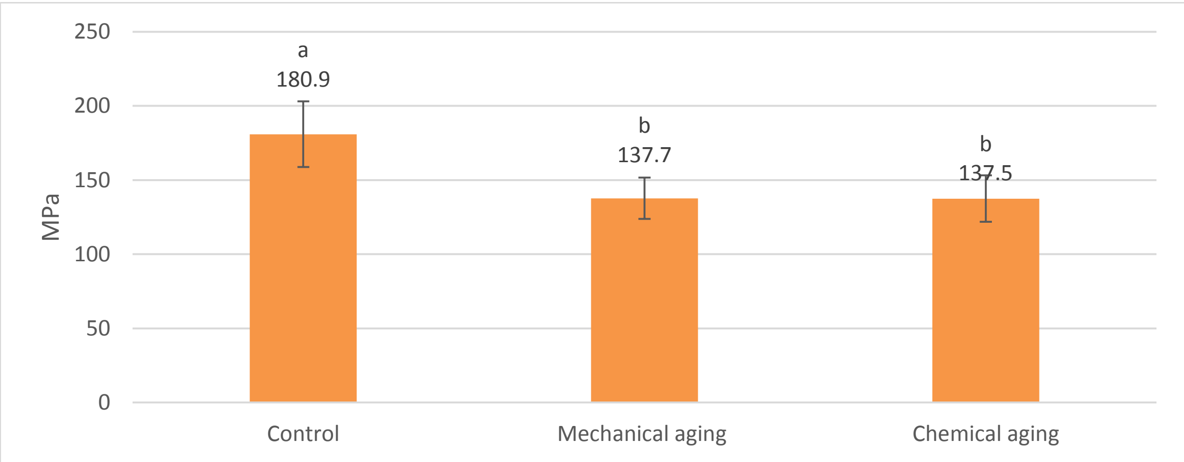

Figure 4: A bar chart showing mean values of the flexure strength after different aging protocols of the tested composite materials. Bars sharing same letters have statistically insignificant differences.

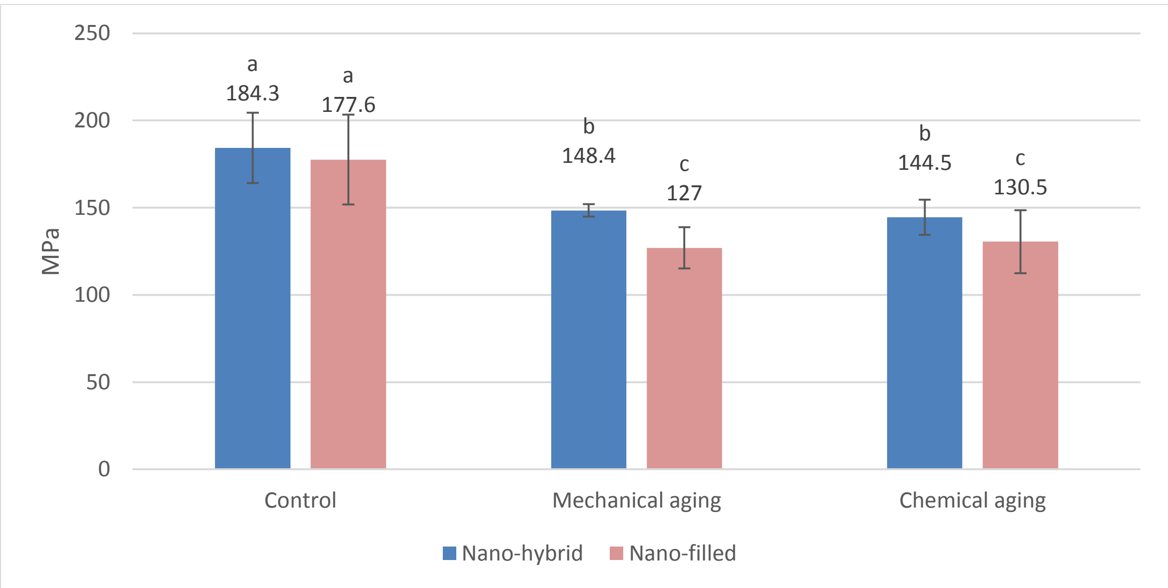

Figure 5: A bar chart showing the mean values of flexure strength between both composite types after each aging protocol. Bars sharing same letters have statistically insignificant differences.

3.2. Microhardness after mechanical fatigue and chemical aging protocols:

Average surface microhardness for the control group (Gp 1) was 101(±1.3) kgf/mm^2. Both aging protocols resulted in a considerable improve in the surface smoothness with significant decrease in the surface microhardness of the samples. Surface microhardness values for samples subjected to mechanical fatigue (Gp 2) averaged 94.9 (±4.7) kgf/mm^2, whereas values for samples subjected to chemical aging (Gp 3) were 90.6 (±3.3) kgf/mm^2. All differences among the groups were statistically significant; Figure 6.

With average values of 100.6 (±1.7) and 101.4 (±0.5) kgf/mm^2 for nano hybrid (Gp 1A) and nano filled (Gp 1B), respectively. This difference was statistically insignificant.

The surface microhardness values of nanohybrid composite didn't change significantly after mechanical aging (Gp 2A) than control group (Gp 1A).

After mechanical and chemical aging, nano hybrid composite samples (Gps 2A & 3A) showed significantly different mean values of 97.5(±2.5) and 90.1(±4.5) kgf/mm^2, respectively. On the other hand, nano filled composite samples (Gps 2B & 3B) showed insignificantly different mean values of 92.3(±5.2) and 91.2(±1.9) kgf/mm^2, respectively, figure 7.

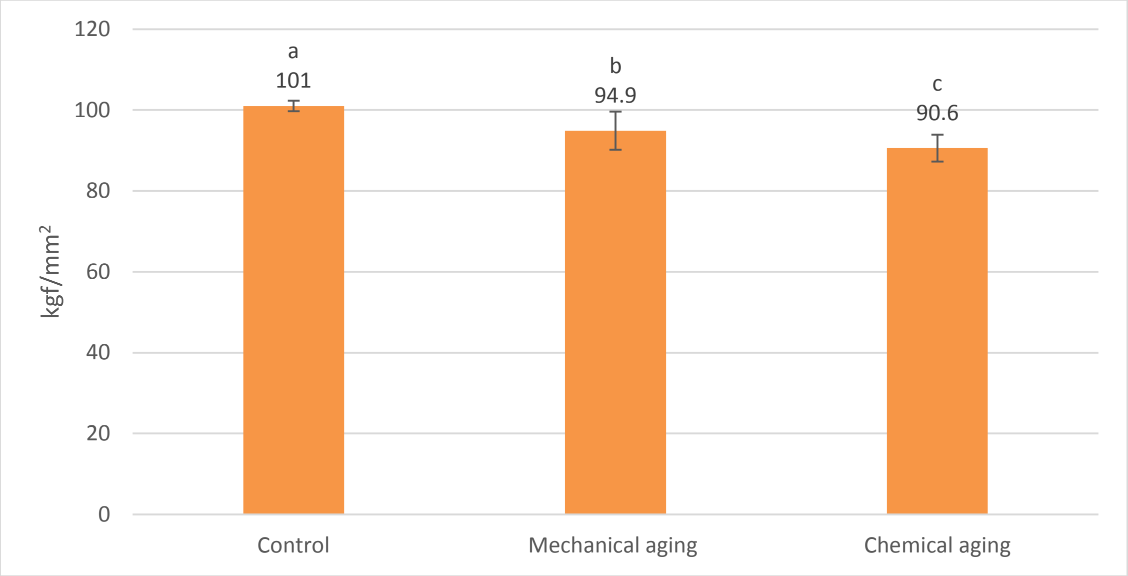

Figure 6: A bar chart showing mean values of surface microhardness after different aging protocols of the tested composite materials. Bars sharing same letters have statistically insignificant differences.

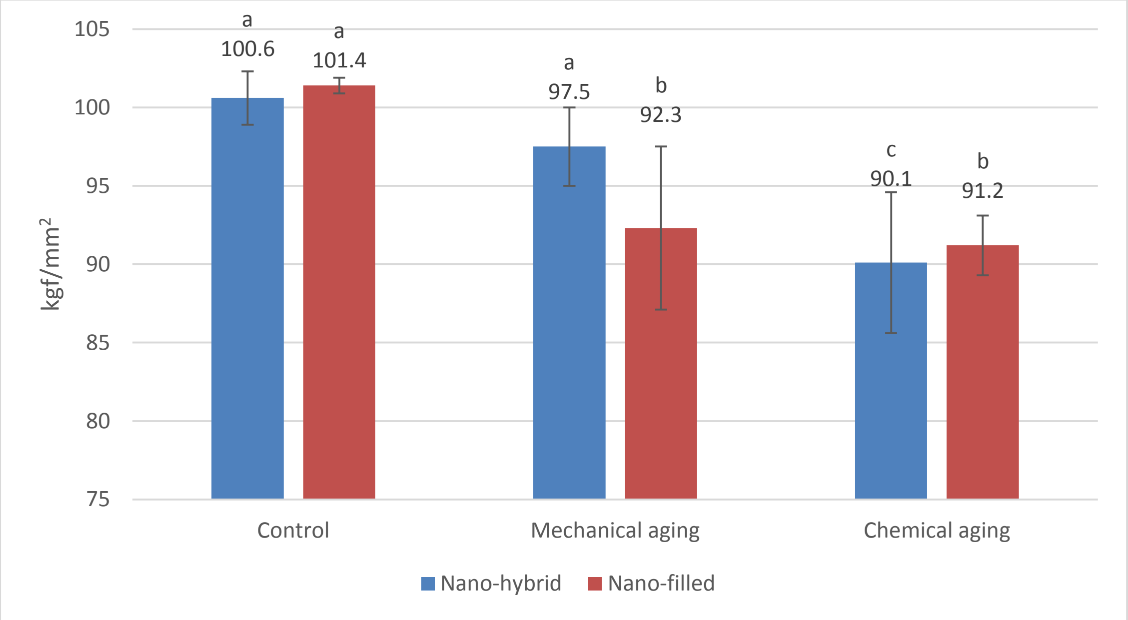

Figure 7: A bar chart showing the mean values of microhardness of different composite types in each aging protocol. Bars sharing same letters have statistically insignificant differences.

4. Discussion:

This study was conducted to find out whether mechanical fatigue and chemical aging protocols can provide comparable effects and can be used interchangeably considering flexure strength and surface microhardness as aging techniques of two types of dental resin composites. Two types of dental resin composites were used; nanohybrid and nanofilled. The wide popularity and wide clinical use of these materials was the trigger to test them [23]. Aging (deterioration) parameters were mechanical fatigue and reduced pH immersion. The aged materials were tested by 3 point loading as a challenge to their bulk properties, and their resistance to indentation was also tested by microhardness test as a challenge to their surface quality. Flexure strength is considered an important parameter for characterizing brittle materials, and this type of test generates complex stresses that combine tensile, compressive, and shear stresses when specimen is loaded[12] **.** One of the essential characteristics of resin composites that ensures their surface finish and smoothness is surface microhardness. This property enables the material to withstand any surface damage brought on by compressive pressures [24].

Artificial aging is a procedure to mimic intraoral circumstances, where the material is exposed to mechanical, chemical, and thermal stress and thereby undergoes a process of aging [4]. Several artificial aging procedures, including storage in artificial saliva, water, or different chemicals at different times and temperatures, thermocycling, and mechanical loading have been used in studies [3]. In both short- and long-term analyses, artificial ageing can be used to simulate material degradation and affect the material's mechanical and optical properties. The ageing is influenced by a variety of factors and can lead to changes in the interior chemical composition and surface microstructure of the aged materials [25].

There is various number of aging protocols to dental resin composite materials. Therefore, Aging protocols followed in this study were mechanical fatigue and chemical aging. Mechanical fatigue (cyclic loading) is a recommended aging protocol by many researchers as it nearly simulates the loading stresses that are produced during service [26]. According to **Zankuli et al., 2015**, tests on the fatigue behavior of dental restorations have been conducted using cycles ranging from 1000 to 1,000,000 cycles [27]. Therefore, the number of cycles in the mechanical aging protocol was chosen as it was the highest frequency of fatigue test during fatigue testing [28].

Regarding chemical aging, it was conducted as it is recommended by many authors [29-31]. This is because 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 [8]. These drinks are known of their high acidity with pH value range between 2.62 and 4.26 [32]. 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 [33]. **Korać et al., 2022**, found that 8 days immersion of dental materials in an acidic medium would be sufficient to produce the greatest changes in the aged materials [34].

The significant reduction in flexural strength of tested resin composites after mechanical fatigue aging protocol could be attributed to discontinuities at the filler-matrix interface which produced by cyclic loading could be a composite's weak point [35]. Consequently, based on the quality of micro-crack interface, a minute crack front may occur under cyclic loading. These micro cracks gradually spread by repeated loading throughout the material causing secondary crack generation at the filler matrix interface[36], leading to weakening of the material under three-point loading [37].Regarding the decline after chemical aging (Gp 3), acid-related hydrolysis of ester radicals found in dimethacrylate monomers including bisphenol A glycidyl methacrylate (Bis-GMA), ethoxylated bisphenol A dimethacrylate (Bis-EMA), urethane dimethacrylate(UDMA),and triethylene glycol dimethacrylate (TGDMA) may be the cause of resin composite deterioration[38, 39] . In addition, the covalent bond formed between coupling agent and inorganic filler is more vulnerable to hydrolysis reaction, which is accelerated by presence of acid, than the bond between matrix and silane coupling agent[40].Flexure strength is a bulk property which depends on many factors such as filler content, bond between inorganic fillers and organic matrix, degree of conversion, surface treatment and surface topography [41] [42]. Resin composite is a polymeric material and its properties depend on degree of conversion, filler loading, nature of bond at filler/matrix interface and silane bonding [43]. Composite properties affected by mechanical fatigue and chemical aging, resulting in degradation in the matrix and filler/matrix interface. Therefore, in this study, there is non-statistically significant difference between mechanical fatigue and chemical aging protocols results.

The significant weakening in flexural strength of nanofilled composite compared to nanohybrid composite may be attributed to the amount of filler loading and amount of organic matrix. Nanohybrid composite contains 81.8% fillers by weight and less amount of organic matrix, while nanofilled composite contains only 78.5% by weight and higher amount of polymeric matrix [44].

Regarding microhardness, and irrespective to the type of composite, the lowest mean value was recorded with samples subjected to chemical aging protocol (Gp 3). According to **Abouelmagd, D.M. et al. 2022 and Branco, A., et al. 2019**, this may be attributed to acid penetration into the resin matrix, softening the Bis-GMA and enabling the release of unreacted monomers. Urethane dimethacrylate (UDMA), TEGDMA, and Bis-GMA have high susceptibility to absorption of water and many other fluids such as acids resulting in high solubility, which may cause the resin matrix to soften and degrade [39, 45]. Nanohybrid composite microhardness was more resistant to mechanical fatigue and chemical aging than nano filled one. This may be attributed to the high amount of fillers that incorporated to resin matrix of nanohybrid composite, decreasing the total amount of organic matrix which is responsible for the absorption of water and other substances like acids that cause the surfaces of composite materials to soften [46].

Therefore, the null hypothesis is accepted only for microhardness evaluation of the tested resin composite.

5. Conclusions:

According to the limitations of this study, it can be concluded that mechanical fatigue for 6000 cycles (23.5 N) could produce the same effect of chemical aging for 8 days in media with a pH 3.6, if the flexure strength of dental resin composite is to be tested. Therefore, they could be used interchangeably for assessment of this parameter. However, if microhardness is to be considered, these two aging protocols could not be utilized interchangeably.

6. Recommendation:

Further studies are recommended on the same types of composite with different time intervals of immersion and a higher number of cyclic loading together long-term clinical follow up.

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