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Effect of artificial aging on mechanical and physical properties of CAD-CAM PMMA resins for occlusal splints

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PURPOSE. This study aimed to assess and compare the color stability, flexural strength (FS), and surface roughness of occlusal splints fabricated from heat-cured acrylic resin, milled polymethyl methacrylate (PMMA)-based resin, and 3D-printed (PMMA) based-resin. **MATERIALS AND METHODS.** Samples of each type of resin were obtained, and baseline measurements of color and surface roughness were recorded. The specimens were divided into three groups (n = 10) and subjected to distinct aging protocols: thermomechanical cycling (TMC), simulated brushing (SB), and control (without aging). Final assessments of color and surface roughness and three-point bending test (ODM100; Odeme) were conducted, and data were statistically analyzed (2-way ANOVA, Tukey, P <.05). **RESULTS.** Across all resin types, the most significant increase in surface roughness (Ra) was observed after TMC ($P < .05$), with the 3D-printed resin exhibiting the lowest Ra ($P < .05$). After brushing, milled resin displayed the highest Ra ($P < .05$) and greater color alteration (ΔE_{00}) compared to 3D-printed resin. The most substantial ΔE_{00} was recorded after brushing for all resins, except for heat-cured resin subjected to TMC. Regardless of aging, milled resin exhibited the highest FS ($P < .05$), except when compared to 3D-printed resin subjected to TMC. Heat-cured resin exposed to TMC demonstrated the lowest FS, different ($P < .05$) from the control. Under control conditions, milled resin exhibited the highest FS, different ($P < .05$) from the brushed group. 3D-printed resin subjected to TMC displayed the highest FS ($P < .05$). **CONCLUSION.** Among the tested resins, 3D-printed resin demonstrated superior longevity, characterized by minimal surface roughness and color alterations. Aging had a negligible impact on its mechanical properties. [J Adv Prosthodont 2023;15:227-37]

KEYWORDS

Computer-Aided Design; Occlusal splints; Color; Flexural strength; Polymethyl Metacrylate; Aging

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INTRODUCTION

Occlusal splints are used in the management of temporomandibular joint disorders (TMD) to alleviate patient symptoms.[1-3] These removable devices are typically made from heat-cured polymethylmethacrylate (PMMA) resin after obtaining dental impressions with irreversible hydrocolloid material and subsequently pouring with type III dental stone to create a diagnostic cast.[4,5]

PMMA resins are favored in dentistry for their cost-effectiveness, ease of handling, high biocompatibility, and favorable marginal adaptation.[6,7] Nevertheless, they exhibit notable polymerization shrinkage and limited wear resistance.[7-9]

To enhance the manufacturing process of occlusal splints, computer-aided design and manufacturing (CAD-CAM) technology has been proposed as an alternative to the conventional approach.[10] CAD-CAM technology enhances the efficiency and accuracy of the procedure, enabling precise replication of new devices when replacements are required.[11]

Digital impressions are acquired using either intraoral scanners or conventional impressions that are subsequently scanned in the dental laboratory. CAD software is employed to design the devices, which are fabricated using subtractive techniques, involving the milling of prefabricated PMMA blanks, or additive manufacturing (AM), such as stereolithography (SLA).[11]

With the advent of 3D printers and the availability of biocompatible resins suitable for 3D printing, AM has found applications in the dental field, experiencing a rapid increase in utilization.[11,12] Among the various AM processes, SLA stands out as one of the most commonly employed methods due to its ease of handling, exceptional precision, excellent surface finish, and efficient material utilization, thereby minimizing wastage.[13] In this process, dental devices are incrementally fabricated in layers using ultraviolet (UV)-curable liquid resins. The build platform of the printer is immersed in these resins, which are subsequently polymerized into the desired shapes through controlled exposure to ultraviolet laser radiation.[11,14] PMMA resins also rank as one of the most popular materials for 3D printing in dentistry, primarily owing to their high thermoplastic behavior, low cytotoxicity, and suitable mechanical and physicochemical properties for clinical application.[11,14,15]

Regardless of the chosen material and manufacturing process, occlusal splints must possess high strength characteristics to endure occlusal forces induced during teeth grinding and clenching, as well as to resist the aging process. Assessing the flexural strength of these splints allows for the evaluation of how dental materials behave under masticatory load. This test simulates clinical scenarios in which the devices are subjected to compressive, tensile, and shear forces.[16] Notably, as reported by Berli *et al.*,[17] the flexural strength of the material diminishes with aging, rendering the dental devices more susceptible to fracture.

The surface roughness of PMMA resins can have a significant impact on the durability of occlusal splints. There is an inverse relationship between the surface roughness and the flexural strength of dental materials. Consequently, rough surfaces may lead to a reduction in the flexural strength of PMMA resins.[18] Moreover, rough surfaces may disrupt the equilibrium of occlusal contacts, a critical factor in the success of splint therapy.[19] This interference can result in patient discomfort and contribute to biofilm accumulation.[20] Additionally, surface roughness can influence the coloration of PMMA resins with rough surfaces being more susceptible to staining due to pigment deposition from food and beverages.[21] This staining can lead to color alteration that may lead to the replacement of the splint. Over time, the aging process can induce changes in both the surface roughness and color of the material.[22,23]

Despite the aforementioned characteristics and advantages, there is no currently conclusive evidence regarding the physical and mechanical properties of CAD-CAM PMMA resins or their performance under aging conditions. Therefore, the aim of this study was to assess and compare the effects of artificial aging on the color stability, flexural strength, and surface roughness of PMMA-based resin used for occlusal splints, specifically those that are heat-cured, milled, and 3D-printed. The null hypothesis tested in this study is that there would be no differences in color stability, flexural strength, and surface roughness among the resins, regardless of the aging protocol.

MATERIALS AND METHODS

We determined the sample size based on mean values of flexural strength from a preliminary study, with a power of 80% and confidence interval of 95%, resulting in 7 samples per group. Considering the eventual loss of samples, 10 samples were obtained for each shape. Therefore, 30 samples were obtained for each type of resin, 10 samples of each shape for each of the groups (n = 10).

The samples were acquired from 3D-printed resin (3D - Cosmos Splint; Yller, Pelotas, RS, Brazil), milled resin blocks (CAD-CAM - Vipi Block; VIPI, Pirassununga, SP, Brazil), and heat-cured PMMA acrylic resin (HC group - Acrílico Termopolimerizável, Clássico, São Paulo, SP, Brazil). For color stability and surface roughness analysis, 3D-printed and heat-cured samples of 14 mm × 2 mm, and milled samples of 7 mm × 7 mm × 2 mm were employed. Thirty samples were prepared from each resin type for this purpose. For flexural strength test, thirty samples measuring 25 mm × 2 mm × 2 mm were fabricated from each PMMA resin.

For the CAD-CAM samples, sections were obtained from PMMA blocks through the utilization of a diamond disc, employing low-speed cutting under water-cooling conditions, using an Isomet 1000 cutting machine (Buehler, Lake Bluff, IL, USA). In contrast, for 3D-printed samples, a standard tessellation language (STL) file was created using CAD software Autodesk Meshmixer (Autodesk, Inc., San Francisco, CA, USA). These files were employed to produce the samples through a 3D printer (Moonray S; 3DC Med, São Paulo, Brazil). After the printing process, the samples were immersed in 99% isopropyl alcohol for 5 min to dissolve uncured or excess resin.

HC samples were obtained through the lost wax casting technique. Initially, a Teflon matrix (internal diameter of 14 mm and thickness of 2 mm) was lubricated with solid Vaseline. Then, molten casting wax (Cera para escultura; Polidental, Cotia, SP, Brazil) was introduced into the matrix. Upon cooling, any surplus wax was excised using a sharp instrument. The resultant wax molds were then extracted from the matrix and embedded in type IV dental stone (Dentsply Sirona, São Paulo, SP, Brazil) within a metallic flask. After the dental stone setting period, the wax was thoroughly eliminated using hot water. Subsequently, acrylic resin was manipulated according to the manufacturer's recommendation and deposited into the void left by the wax. The metallic flask containing the acrylic resin was securely sealed and subjected to a pneumatic press, exerting a pressure of 60 pounds. This assembly was further processed within a water bath until it reached a temperature of 120°C. After reaching this temperature, the press was deactivated and the system was allowed to cool down to 60°C. After reaching room temperature, the samples were extracted from the flasks.

All the samples, regardless of the material and manufacturing process, were wet-polished with 600, 1200, and 2000-grit SiC paper, standardizing the thickness and surface roughness of the samples. Samples with surface roughness greater than 0.3 μm were discarded. A scheme of the study methodology can be visualized through the flowchart (Fig. 1).

Initial color was assessed using the CIE L*a*b* system with a spectrophotometer (PCB6807; Byk Gardner, Geretsried, Germany). To simulate natural daylight, the samples were placed within a neutral-gray enclosure (Munsell N-7) under D65 standard lighting conditions. Color measurements were performed against a white background (White Standard Sphere for 45°, 0° Reflectance). Three measurements were obtained for each sample, and the average values were employed as the baseline values.

Initial surface roughness measurements were obtained using a rugosimeter (Surfcorder SE 1700; Kosakalab, Tokyo, Japan) with 0.8 mm cut-off values and a scanning speed of 0.25 mm/sec. The measurements were carried out at three distinct locations: one at the central point of the sample and the other two, each positioned 1 mm away from the center on opposite sides. The average of these three readings was employed as the initial surface roughness value.

Following initial measurements of color and surface roughness, the samples were randomly separated into three groups (n = 10) according to the aging procedures to which they were submitted: control (no aging - stored in distilled water at 37°C for 30 days), simulated brushing, and thermomechanical cycling.

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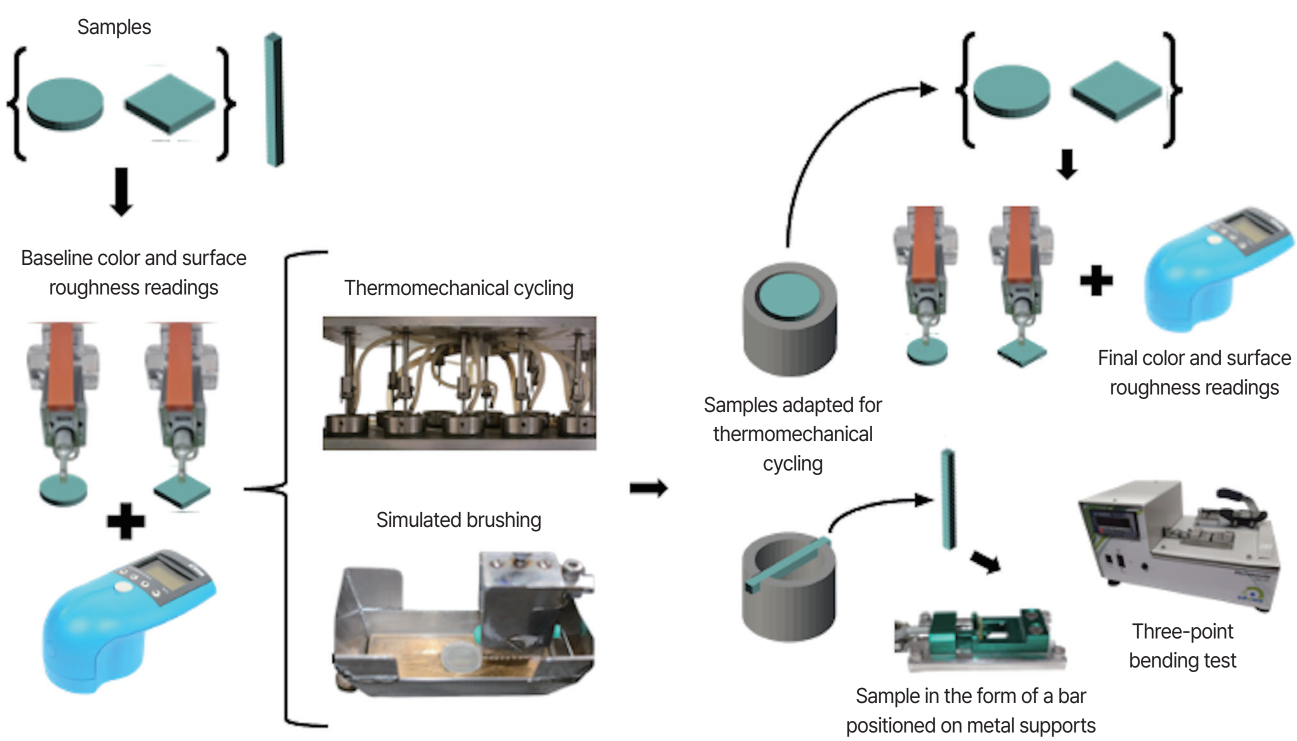

Fig. 1. Study Flowchart.

For the simulated brushing procedure, a brushing machine (Pepsodent; MAVTEC - Com. Peças, Acess. E Serv. Ltda. ME, Ribeirão Preto, SP, Brazil) was employed. A toothbrush with soft bristles (Tek; Johnson & Johnson Ind. Com. Ltd., São José dos Campos, SP, Brazil) was allocated to each sample. The samples were affixed to acrylic resin plates (Plexiglass; Acrilpress Artefatos de acrylic Ltda, Ribeirão Preto, SP, Brazil) using hot glue to immobilize them during brushing.

A slurry of toothpaste (Colgate Total 12 Clean Mint; Colgate, São Paulo, SP, Brazil) was prepared by diluting it with distilled water in a 1:1 ratio to ensure a consistent and uniform texture. Then, 10 mL of this solution was dispensed onto each sample using a plastic syringe. The samples underwent 73,000 brushing cycles, simulating a period equivalent to 5 years of brushing by a healthy individual.[24]

Samples were subjected to thermomechanical cycling (ER System; Erios, São Paulo, SP, Brazil) designed to mimic masticatory load and temperature fluctuations. Samples were enclosed within 20-mm diameter PVC rings and embedded with self-curing acrylic resin (Acrílico Auto Polimerizante JET; Clássico, São Paulo, SP, Brazil) and light-body condensation silicone (Orange Wash Zhermack; Badia Polesine, RO, Italy) to simulate periodontal ligament.

Samples were submitted to 1,200,000 cycles (equivalent to 5 years of masticatory force)[25,26] applied at a frequency of 2 Hz with a load of 1.4kg/f. The temperature conditions during cycling were 5°C, 37°C and 55°C (± 2°C), each maintained for 30 s, with an intermediate pause of 12 s.

After the aging procedures, samples obtained for the flexural strength test underwent a three-point bending test (Microtensile OM100; Odeme, Luzerna, SC, Brazil) at a constant speed of 0.5 mm/min. Samples were positioned on two metal supports maintaining a 20 mm separation between them, and a force was steadily applied at the midpoint of the sample until its fracture occurred.

Flexural strength was calculated according to the formula that follows ISO4049:

$$\text{Flexural strength} = 6.x.l/b.d.2;$$

where x is the load at the fracture point (kg), d is the thickness of the specimen (cm), l is the length of the support span (cm), and b is the width of the specimen (cm).

Final color readings were conducted, as previously described, and color stability (ΔE_{00}) was calculated by the following formula:[27]

$$\Delta E_{00} = (\Delta L/K_L \cdot S_L)^2 + (\Delta C/K_C \cdot S_C)^2 + (\Delta H/K_H \cdot S_H)^2 + RT \cdot (\Delta C/K_C \cdot S_C) \times (\Delta H/K_H \cdot S_H)^{0.5};$$

where ΔL^*, ΔC^* and ΔH^* are the differences in brightness, chroma, and hue between the final and baseline color values, and RT (rotation function) is a function that explains the interaction between chroma and hue differences in the blue region. SL, SC, and SH are the weighting functions for the luminance, chroma, and hue components, respectively. KL, KC, and KH are the parametric factors according to different visualization parameters that were defined as 1.

Color stability values were compared to the limits of perceptibility (0.8) and acceptability (1.8).[28]

Final surface roughness measurements were obtained, and roughness alteration was calculated using the following formula:

$$\Delta Ra = Ra_F - Ra_I,$$

where Ra_F represents the final roughness mean value and Ra_I, the initial one.

Shapiro-Wilk test was used to verify the normality of the data, which showed normal distribution. Thus, data were analyzed by 2-way ANOVA (variation factors: aging and types of resin) and post hoc Tukey's test ($P < .05$).

RESULTS

The comparison of mean flexural strength values is observed in Figure 2. Regardless of the aging protocol, the CAD-CAM resin exhibited the highest flexural strength, which was significantly different ($P < .0001$) from the other resins, except for the 3D/thermomechanical cycling (TMC) resin.

Comparing the aging protocols within the same resin, the HC/TMC revealed the lowest flexural strength, significantly different ($P = .0073$) from the control group but similar ($P = .765$) to the brushing group. The HC resin showed no significant difference after brushing under control conditions ($P = .6393$). The 3D/TMC demonstrated the highest flexural strength, different from the other aging protocols ($P < .0001$) that presented no significant difference ($P > .7671$) between them. CAD-CAM resin under control conditions exhibited the highest flexural strength, different ($P = .0440$) from the brushed one but similar ($P = .3382$) to the one submitted to TMC. Between the CAD-CAM submitted to TMC and the brushed one, there was no difference ($P = .5629$).

The comparison of color alteration is shown in Figure 3. Without aging, the resins demonstrated no significant difference. After brushing, CAD-CAM showed higher color alteration ($P = .0011$) than the 3D. The HC

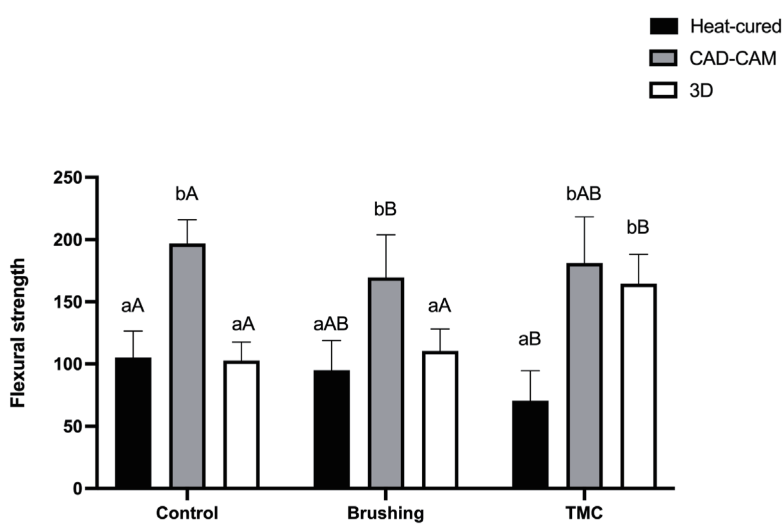

Fig. 2. Comparison of flexural strength. Different letters, lowercase among the resins within the same aging protocol and uppercase among the aging protocols within the same resin, indicate significant difference ($P < .05$).

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resin presented similar results to the other resins (P =.2555 and P =.0948). After TMC, HC resin showed the highest color alteration, different (P = .0001; P = .0003) from the other resins that showed no significant difference between them (P = .9563).

Comparing aging protocols within the same material, regardless of the resin type, the highest color alterations occurred after brushing, differing from the other aging protocols, except for HC/TMC (P = .7664). HC resin also presented greater color alteration after TMC than under control conditions (P = .0492). Among the other groups, there was no significant difference.

All resins displayed color alterations exceeding the perceptibility threshold (0.8) and acceptability threshold (1.8),[28] regardless of the aging protocol. Furthermore, after brushing, all resins exhibited color alterations exceeding the acceptability threshold. The heat-cured resin also demonstrated color changes above the acceptability threshold, regardless of the aging protocol.

The comparison of mean values regarding surface roughness alteration is depicted in Fig. 4. When comparing the resins submitted to the same aging protocol, there was no difference under control conditions. After brushing, CAD-CAM resin displayed the greatest surface roughness alteration, significantly different (P = .0049) from the 3D resin, which exhibited the smallest change. The HC resin yielded results similar to the other resins. After TMC, 3D resin demonstrated the smallest surface roughness alteration, significantly different (P < .0001) from the other resins, which exhibited no significant differences between them (P = .3086).

Comparing aging protocols within the same resin, the greatest surface roughness alteration occurred after TMC, significantly different from the other aging protocols (P < .0001) in all resins except for CAD-CAM, which showed differences (P = .0077) after all aging protocols.

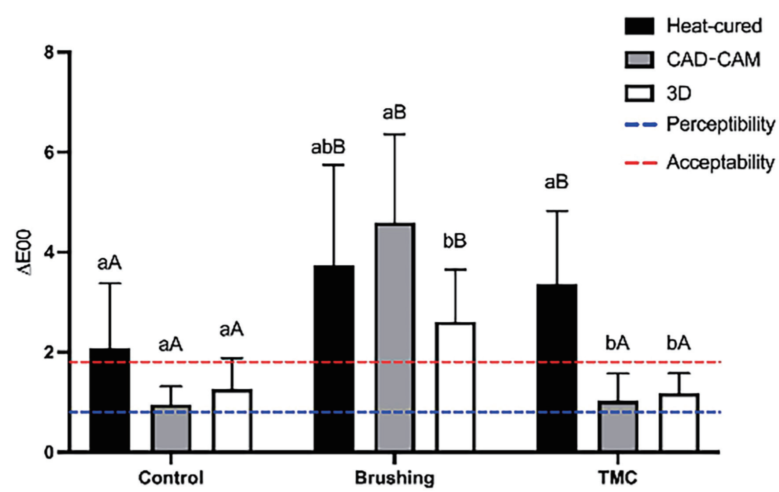

Fig. 3. Comparison of color alteration. Different letters, lowercase among the resins within the same aging protocol and uppercase among the aging protocols within the same resin, indicate a significant difference (P < .05).

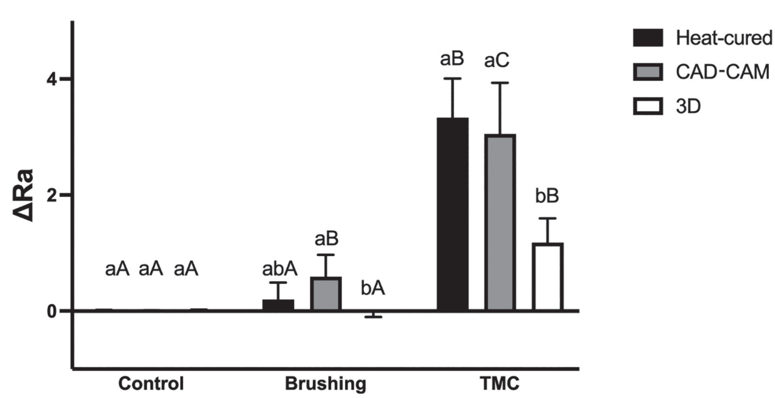

Fig. 4. Comparison of surface roughness alteration (ΔRa). Different letters, lowercase among the resins within the same aging protocol and uppercase among the aging protocols within the same resin, indicate a significant difference (P < .05).

DISCUSSION

This study aimed to investigate the impact of artificial aging on the color stability, flexural strength (FS), and surface roughness of three types of resins used in the fabrication of occlusal splints: HC resins, CAD-CAM resins, and 3D resins. The null hypothesis tested was that there would be no alterations in the properties of the analyzed resins, regardless of the resin type employed and the aging protocols applied. Based on the obtained results, the null hypothesis was rejected. This rejection is substantiated by the presence of significant differences among the resins across all analyzed properties following the aging process.

TMDs have a multifactorial etiology, affecting various components, including the masticatory musculature, temporomandibular joint, and associate structures. Occlusal splints represent a non-invasive and reversible therapeutic approach that has demonstrated efficacy in pain reduction and occlusion stabilization for TMD patients. These splints have been crafted from HC resins using conventional laboratory techniques. However, the growing popularity of CAD-CAM technology in recent decades necessitates the evaluation and comparison of properties associated with these newer resin materials.[10]

In the current study, the highest color changes occurred after brushing, regardless of the resin type. This phenomenon is likely due to the abrasive particles present in the toothpaste, which may have caused surface abrasion on the resins, resulting in surface roughness alterations.[29,30] Changes in the surface roughness have the potential to influence light reflection properties, consequently affecting the color appearance of the material.[22,31] Since the spectrophotometer used in the present study measures the amount of light reflected from a sample, any surface alteration that changes the light reflection will lead to different color values.[22,23,32-34]

The assessment of whether these color alterations are perceptible or deemed acceptable holds significant clinical importance. In our study, these color alterations exceeded the acceptability threshold, indicating that brushing significantly affected the color stability of all the resin materials. Over time, this brushing-induced discoloration becomes clinically unacceptable, which can lead to discomfort and dissatisfaction in patients, ultimately necessitating the replacement of the splints.[28,35]

Among the resins, CAD-CAM resin exhibited the most pronounced color alteration after brushing. This resin is pre-polymerized and no additional processing is required before its use, resulting in an initially smooth surface, as evident in the control conditions (Table 1). However, after brushing, its surface roughness increases significantly, thereby inducing alterations in its color.

Furthermore, variations in water sorption and temperature changes can also impact the coloration of the PMMA resins.[35] TMC simulates the transformations occurring in the oral environment. During this process, samples undergo immersion in water at different temperatures, which may lead to thermal stress and subsequent surface degradation.[35,36] Conventional materials exhibit a higher propensity to absorb water in comparison to CAD-CAM materials.[37] After TMC, the HC resin presented the most substantial color alteration, distinguishing it from the other resins. This observation aligns with findings from a previous study by Alfouzan *et al*.[38] which suggested that color stability hinges on the composition of the material. The 3D PMMA resins, according to the same study, exhibit superior stability.[38] This enhanced stability can also be attributed to CAD-CAM resins, which

Table 1. Comparison of surface roughness alteration (mean values and standard deviation) among the groups

	Heat-cured resin	Milled resin	3D-printed resin
Control	0.01 (0.01) aA	0.00 (0.01) aA	0.02 (0.01) aA
Brushing	0.2 (0.29) abA	0.59 (0.38) aB	-0.03 (0.08) bA
TMC	3.33 (0.68) aB	3.05 (0.88) aC	1.12 (0.42) bB

* Different letters, lowercase in the line and uppercase in the column, indicate significant difference (P < .05).
TMC: thermomechanical cycling.

undergo prepolymerization under elevated temperature and pressure conditions. This process minimizes the release of residual monomers and effectively manages volumetric and shrinkage distortions, thus contributing to greater color stability.[35]

Nevertheless, our findings contrast with a prior research that reported higher water sorption and greater discoloration in 3D resins compared to HC and CAD-CAM materials.[35,39] The lower wear resistance of 3D resins can be attributed to their reduced filler content, which is necessary to assure their low viscosity and facilitated material flow during the manufacturing process. This also contributes to the achievement of smooth finished surfaces, as confirmed in our current study.[39] The 3D resin under control conditions presented a smooth surface, but after undergoing TMC, its surface roughness increased (Table 1). However, unlike those studies,[35,39] the 3D resin displayed less color alteration than HC. Beyond material composition, factors such as 3D printing parameters can impact the surface quality of the resin.[40]

Regardless of the aging process, the color alteration of HC resin exceeded the acceptable threshold, necessitating replacement for aesthetic reasons after a certain period.[28] The other resins subjected to TMC and under control conditions, exhibited color changes surpassing the perceptibility threshold but remaining below the acceptability threshold, indicating clinically acceptable color alterations.[28] Besides its influence on color stability, surface roughness plays a significant role in patient comfort, susceptibility to staining, and biofilm accumulation.[19-21] Therefore, evaluating the surface roughness of PMMA resins after aging is essential to determine the longevity of these materials. In our study, TMC led to a significant increase in surface roughness for all the resins. Masticatory load and temperature fluctuations may cause thermal expansion and shrinkage of polymers, such as PMMA resins, which accelerate their wear.[19] In addition, immersion in water during TMC may cause material swelling, further contributing to increase its surface roughness.[41]

Among the resins, the 3D resin presented the lowest surface roughness alteration, indicating greater resistance to surface degradation when compared to the other resin types. Studies investigating the effect of aging on the surface roughness of 3D resins are scarce. In a previous study, Nam *et al*. compared the surface roughness of 3D resins and CAD-CAM resins after hydrothermal aging and found similar results between them.[42] Our findings, however, diverged presumably due to disparities in resin composition, printing parameters and/or aging procedures,[43] relative to those made by Nam *et al*..[42] Notably, we observed a reduction in surface roughness for the 3D resin after abrasive brushing, denoted by a negative value, indicative of a smoother surface. This effect is likely attributable to the low filler content within the 3D resin.[39]

The analysis of flexural strength determines the resistance of the material against deformation. This is especially relevant for the occlusal splints that are constantly subjected to masticatory load.[43] In our current study, CAD-CAM resin revealed the highest flexural strength, both under control conditions and after brushing. When submitted to TMC, this resin showed similar performance to the 3D resin. Our findings are in agreement with previous studies.[44-46] The high flexural strength of CAD-CAM resin is explained by its manufacturing process. As previously mentioned, this resin undergoes prepolymerization under carefully controlled conditions of high pressure and temperature, leading to increased degree of conversion, reduced residual monomer content, and diminished microporosities. Therefore, its mechanical properties are improved.[47] After TMC, the flexural strength of the 3D resin was similar to that of the CAD-CAM resin, which is in accordance with previous studies.[10,19,28,43] According to Chung *et al*., 3D printing technology employing resins provides sufficient fracture resistance for dental application.[48] Occlusal splints are fabricated via CAM, wherein each resin layer is polymerized using UV radiation, reducing its polymerization shrinkage.[49] In addition, post-printing, unpolymerized resin is removed with solvents, and the splints undergo further curing process in a polymerization device equipped with an ultraviolet source to achieve final polymerization. This meticulous process results in a high degree of conversion, preventing the release of residual monomers and enhances the mechanical properties of the material.[49]

The low flexural strength values of CAD-CAM resin can also be justified by its processing technique, which relies on operator skill. HC resins are composed of powder and liquid components that are manually mixed and processed according to instructions of the manufacturer. Inadequate processing can lead to excessive residual monomers, low degree of conversion, the presence of voids or bubbles, and material inhomogeneity, all of which adversely impact the mechanical and physical properties of the resin.[22]

CAD-CAM technology reduces manufacturing time and laboratory technique sensitivity. Current CAD-CAM PMMA resins show similar or even better physical and mechanical properties compared to conventional resins. However, their long-term clinical performance may depend on the manufacturing process. Further studies comparing other resin materials and processing methods are warranted.

Moreover, it is essential to exercise prudence when interpreting the results obtained from our present study. *In vitro* experiments endeavor to mimic the oral environment, yet this milieu is inherently intricate, characterized by continual pH fluctuations, the buildup of plaque, varying temperatures, and dynamic mechanical loads. Consequently, we advocate for the implementation of *in vivo* studies to provide a more comprehensive assessment of the performance of these materials in a clinical context.

CONCLUSION

Based on the results obtained, it can be concluded that the 3D resin exhibits superior longevity compared to the other tested resins, demonstrating reduced surface roughness and minimal color alterations. Moreover, aging had no significant impact on its properties, including flexural strength. Consequently, 3D resin may be considered the preferred material for occlusal splints.

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