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ORIGINAL ARTICLE

Influence of Artificial Aging on the Vickers Hardness of Milled and 3D Printed Provisional Materials

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Abstract

The purpose of this study was to assess the effect of artificial aging on the Vickers hardness of three resins for provisional dental restorations.

Materials and Methods: Three resins were tested: Evolux PMMA (milled resin), Cosmos Temp (3D-printed resin) and Structure 2 SC (bis-acrylic resin, as a control). Specimens were prepared in a disc shape (n = 9) and the Vickers hardness was measured under a load of 20 N for 10 s at two time points: 24h and 60 days after storage in distilled water at 37 °C in an incubator. The data were analysed using the Shapiro-Wilk test followed by the non-parametric Kruskal-Wallis test ($p < 0.05$).

Results: In periods of artificial aging for 24 h/60 d, there was no significant difference ($p > 0.05$) between the Vickers hardness of Structur 2 SC (33.37 VHN/33.96 VHN) and Evolux PMMA (32.34 VHN/29.11VHN); however, both materials were statistically superior to Cosmos Temp (10.90 VHN/15.40 VHN). The impact of artificial aging was only observed for 3D-printed resin ($p < 0.05$), with an increase in hardness after 60 days.

Conclusion: The milled and bis-acrylic resins were statistically superior to 3D-printed resin in both periods of artificial aging, which caused a significant increase of hardness only for 3D-printed resin.

Keywords

Artificial aging, Vickers hardness, Computer-aided design, Computer-aided manufacturing, Temporary dental restoration

Introduction

Temporary restorations represent an essential part of the oral rehabilitation procedure for fixed dental prostheses, to fulfil important roles, including the protection of pulpar and periodontal tissues, the evaluation of teeth preparations and aesthetic predictability [1]. For a long time, the resins based on polymethylmethacrylate (PMMA) were the material of choice; however, due to limitations such as high polymerization contraction, exothermic reactions and low colour stability, these have been replaced by bis-acryl resins more recently [2].

The bis-acryl resin, primarily comprised of bis-phenyl-glycidyl di-methacrylate (Bis-GMA) forming densely cross-linked structures, is considerably easy to handle, in auto- or dual-polymerization, is colour stable and has minimal shrinkage [3,4]. Due to these crucial characteristics, bis-acrylic resins are rapidly replacing resins based on polymethylmethacrylate (PMMA) for use in transitory restorations [5,6].

The development and application of CAD-CAM technology has also promoted important changes in the production of transitory restorations. Characterized by greater speed, efficiency and quality control the CAD/CAM fabrication process has been adopted using either subtractive manufacturing or additive manufacturing technologies [7].

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With results still being controversial, studies have shown superiority in the flexural resistance of PMMA resins in the form of CAD-CAM blocks, followed by bis-acryl and conventional PMMA [3,6,8,9]. Regarding additive manufacturing, 3D-printed resins have shown lower wear resistance, fracture resistance and hardness than those milled, made with bis-acrylic or conventionally fabricated [6,10].

Transitory restorations exposed to the oral environment suffer water absorption, which can lead to weakening due to degradation of the covalent bond of the polymer chain [11-13]. With the more homogeneous structure of CAD-CAM systems, greater stability of physical and mechanical properties can be expected, by overcoming the shortcomings of handmade PMMA systems such as excessive water sorption and residual monomer release [1]. However, studies regarding the behaviour of resins processed by the CAD-CAM system simulating aging are still scarce.

The purpose of this study was to assess the effect of artificial aging on the Vickers hardness of three resins for provisional dental restorations obtained with different techniques, including the CAD-CAM system. We postulated the following null hypotheses: ($H_0$1) in each aging period, there will be no significant difference in hardness among the three resins and ($H_0$2) that artificial aging will not cause a statistically significant difference in each resin individually.

Materials and Methods

Vickers hardness was assessed for three different materials for provisional restorations: bis-acrylic resin (BR), milled resin (MR) and 3D-printed resin (PR) (Table 1).

For BR, the specimens (n = 9) were fabricated with a disk shape (8 mm in diameter and 2 mm in thickness) according to ADA-ANSI specification #27 [14]. BR was mixed according to the manufacturer's instructions using a self mixing gun and injected into custom-fabricated 8 × 2 mm^3 silicone rubber moulds (Zetalabor, Zhermack, Badia Polesine, Italy). After 5 min, the blocks were retrieved from the moulds.

For MR, the specimens were virtually designed using a CAD software Ceramill Mind (Amann Girrbach, Koblach, Austria)to a dimension of 8 × 2 mm^3 and milled from a Evolux PMMA block (101 × 101 × 20 mm^3), using a milling machine Ceramill Motion 2 (Amann Girrbach,Koblach, Austria).

For PR, the specimens were also virtually designed using the Ceramill Mind software (Amann Girrbach, Koblach, Austria) and printed using a stereolithography (SLA) printer (D30, Rapid Shape, Heimsheim, Germany). After printing, the specimens were cleaned with 90% isopropyl alcohol for 5 min according to the manufacturer's specifications and post-polymerized with 3000 flashes of ultraviolet light (385 nm) in a UV-A type 3 post-polymerization lightbox (Flashlight plus, Shera Material Technology, Lemforde, Germany). All specimens were polished by grinding on wet silicon carbide papers (200, 400 and 800 grit) and then stored in a water bath at 37 ± 1 °C prior to testing.

The Vickers hardness test was performed after 24h and 60 days using a micro-Vickers hardness tester (HMV-G20, Shimadzu, Tokyo, Japan), with a 20N load applied for 10s. Five readings were taken from the top and bottom of the test specimens, respectively, and the final mean was averaged for each sample.

The data were checked for normal distribution using the Shapiro-Wilk test. As the data were not normally distributed, statistical significance was tested with the non-parametric Kruskal-Wallis test to compare the three resins and Mann-Whitney test to compare the artificial aging 24h and 60d in each resin ($\alpha = 0.05$). Analyses were performed using the SPSS software (Version 25.0, Chicago, IL, USA). The level of significance was set at 0.05.

Results

The values obtained for Vickers hardness (VHN) after aging for 24h and 60d of the three resins were recorded and are listed in Table 2.

In two periods of artificial aging for 24h/60d, there was no significant difference ($p > 0.05$) between the Vickers hardness of bis-acrylic resin Structure 2 SC (33.37 VHN/33.96 VHN) and milled resin Evolux PMMA (29.11 VHN/32.34 VHN); however, both materials were statistically superior to printed resin Cosmos Temp (10.90 VHN/15.40 VHN). The impact of artificial aging was only observed for 3D-printed resin ($p < 0.05$), with an increased hardness after 60d (Table 3).

Discussion

This study proposed, through the mechanical property of Vickers hardness surface characteristics, to evaluate the effects of immersion in water on provisional resins processed by the CAD/CAM system, milled and printed, using a bis-acrylic resin as a control group. In both immersion periods, 24h and 60 days, the

Table 1: Type, code, resin, and manufacturer of tested resin materials for provisional restorations

Type	Code	Resin	Compositions	Manufacturer
Bis-acrylic	BR	Structure 2 SC	Bis-Acryl methacrylate	Voco, Porto Alegre, Brazil
Milled	MR	Evolux PMMA	PMMA Polymer	Blue Dent, Pirassununga, Brazil
3D-printed	PR	Cosmos Temp	Methylmethacrylate	Yller, Pelotas, Brazil

Table 2: Mean vickers hardness of the three resins.

		Structure 2 SC (BR)	Evolux PMMA (MR)	Cosmos Temp (PR)
Vickers Hardness (VHN)	24h	32.27[aA]	32.34[aA]	10.90[Bb]
	60d	33.96[aA]	29.84[aA]	15.40[bC]

*Different lowercase letter indicates statistically significant difference between materials; Different uppercase letters indicate statistically significant difference between aging conditions.

Table 3: Kruskal-Wallis test after artificial aging 24H and 60D.

		Compared factors	Post hoc	Post Dif.	Calculated Z	Critical Z
Vickers Hardness	24h	PR-MR	10.4	2.7914	2.394	p < 0.05
		PR-BR	16.5	4.4247		
		MR-BR	6.1	1.6333		ns
	60d	PR-MR	11.2	2.9993	2.394	p < 0.05
		PR-BR	15.7	4.2168		
		MR-BR	4.5	1.2175		ns

null hypothesis ($H_0 1$), that there would be no significant difference between the three resins, was rejected. The alternative hypothesis (H1) was adopted, given that the milled (Evolux PMMA) and bis-acrylic resins (Structure 2 SC) were statistically similar, but with a higher Vickers hardness than the 3D-printed resins (Cosmos Temp) (Table 2).

Regarding the effect of immersion time on each resin, the null hypothesis ($H_0 2$) was accepted for the milled (Evolux PMMA) and bis-acrylic (Structure 2 SC) resins, as the 60-day immersion period did not causes significant changes in the Vickers hardness values compared to the 24h period. On the other hand, in the 3D-printed resin (Cosmos Temp) there was a significant increase in hardness after 60 days of immersion, thus opting for the alternative hypothesis (H1) (Table 2).

The better mechanical behaviour of bis-acrylic and milled resins compared to printed resin seen in the present study is in accordance with the results of previous studies [5,6,15-17]. The structure of bis-acrylic resins, dimethacrylates, which exhibit a rigid cross linked network due to the presence of an organic resin matrix, inorganic fillers and functional groups of monomers (Bis-GMA and TEGDMA), provide the durable structure to withstand breaking and aging-stress [1,11,18-21].

The favourable combination of composition/structure of bis-acryl resins may explain the absence of a drastic influence in the Vickers hardness of artificial aging process. The water sorption might progressively degrade the mechanical properties of polymers due to softening of the matrix (water molecules penetrate the spaces between polymer chains and separate them and, by acting as a plasticizer, the polymer chains become more mobile and weaken) and the release of monomers and degradation products [1,8,11].

The industrial production process of CAD-CAM PMMA blocks, under high pressure and temperature, makes it possible to also obtain a more homogeneous structure, with less free monomer and lower porosity, in addition to lower water absorption and less solubility [3,7-9,22]. All of these factors might reduce the plasticizing effects of milled resins.

On the other hand, a study by Ellakany, et al. [23] showed no significant difference in hardness between resins milled and printed with stereolithography (SLA) technology, similar to that used in the present study. Additionally, other studies reported higher hardness for 3D-printed resins than conventional and milled resins [15,24]. These results would be due to the presence of cross-linked monomers and inorganic fillers, which increased the abrasion resistance [15,20,24]. The divergence of the results of mechanical behaviour of the 3D-printed resin is related to several factors, including printing technology, light intensity and wavelength, CAD design, printing orientation, layer thickness, post-processing procedures and material characteristics [25-30].

The orientation angle (0°, 45° and 90°) has shown a strong influence on the mechanical properties of the 3D-printed resins [25,30-33]. In this study, an SLA printer was used and samples were obtained with an orientation angle of 0°. A probable limitation of this study was that other orientation angles were not considered, suggesting that additional tests are needed in the future.

The hardness results demonstrated that only the 3D-printed resin was affected by the aging process, with a significant increase in values after 60 days of immersion in water. This shows that for the conditions adopted, including the resin brand, orientation angle, printer technology (SLA), post-processing method and water immersion process, did not cause the expected effects on hardness, which involve: The softening of the matrix, the release of monomers and degradation products [1,8,11].

In the evaluation of fracture resistance and flexural resistance, the effects of artificial aging have been contradictory. In the study by Stawarczyk, et al. [34], higher values of fracture resistance for 3D-printed resins were observed after 7 days of immersion in water, remaining constant for up to 28 days. In contrast, no increase in fracture load was observed, but rather a decrease, in a similar study with immersion in water for a period of 21 days [31]. It was suggested that post-polymerization occurred to justify the increase in fracture load after artificial aging [34].

However, the increase in mechanical properties of 3D-printed temporary resins after aging remains unclear. As the present study was limited to a single 3D-printed temporary resin, comparison with other resin brands, with amounts and types of integrated filler [35], can contribute to a better understanding of these results.

As this is only a surface characteristic, hardness alone is not an indicator of overall rigidity and strength and cannot be used to predict the clinical behaviour of long-span prostheses [36]. This limitation shows the need for association with other mechanical tests such as flexural strength, which is generally considered to be the main indicator of the mechanical response of a restorative material [30]. Additionally, considering that the oral environment has presented a detrimental effect on temporary polymeric prostheses properties, complementing the artificial aging evaluation with thermomechanical aging tests will allow an extensive characterization of the temporary resins obtained by the CAD/CAM system.

Despite the advantages of additive manufacturing include material savings, usually lower costs of equipment and materials, the production of complex geometries and the possibility of combinations of materials [37], the present study corroborates the current literature which states that it raises the needs of enhanced control and improvements in the processing and post-processing phases particular to 3D printers.

Conclusion

Within the limitations of this current study, it was concluded that:

1. In two artificial aging periods, 24h and 60d, the bis-acrylic and milled resins showed similar behaviours of Vickers hardness which were statistically superior to the 3D-printed resin.
2. Sixty days of aging in water does not affect the hardness of bis-acrylic and milled resins, while the 3D-printed resin presented a significant increase in values.

Author Contributions

Conceptualization: Jorge Luiz OC Filho, Sicknan S Rocha; Methodology: Ana Luiza C Souza, Jorge Luiz OC Filho, Sicknan S Rocha; Validation, Formal Analysis, Investigation: Sicknan S Rocha; Resources: Ana Luiza C Souza, Jorge Luiz OC Filho; Data Curation, Writing-Original Draft Preparation, Writing- Review & Editing, Visualization, Supervision: Sicknan S Rocha.

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