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RESEARCH ARTICLE **BIOMATERIALS**

Comparison of Water Sorption and Solubility of CAD/CAM Milled PMMA, 3D-Printed PMMA, and 3D-Printed Resin Dental Polymers

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ABSTRACT

The objective of this study was to evaluate and compare the water sorption and solubility of three dental restorative polymers—CAD/CAM milled PMMA, 3D-printed PMMA, and 3D-printed resin—after aging for different time intervals. For this purpose, thirty specimens (n = 10/group) were fabricated from CAD/CAM milled PMMA (PMMA DISC), 3D-printed PMMA (Temporary Crown), and 3D-printed resin (SDC Resin). Specimens were dried in a ventilated oven at 45 °C for 24 hours, and then immersed in purified water at 23 ±1°C. The masses were measured after 7, 14, and 30 days. Water sorption (Wsp) and solubility (Wsl) were calculated in µg/mm³. Assumptions of normality, variance homogeneity, and sphericity were tested. Sorption was analyzed via two-way repeated measures ANOVA with Games-Howell post-hoc; solubility, which violated normality, via Kruskal-Wallis with Dunn's post-hoc ($\alpha = 0.05$). The achieved results showed that water sorption increased significantly with immersion time in all materials ($p < 0.001$). 3D-Resin demonstrated the highest sorption values, followed by 3D-PMMA, while Milled-PMMA exhibited the lowest values at all time intervals. At 30 days, mean sorption values were 16.77 ± 0.58 µg/mm³ (Milled-PMMA), 34.18 ± 3.68 µg/mm³ (3D-PMMA), and 47.68 ± 2.07 µg/mm³ (3D-Resin). Water solubility differed significantly among the tested materials ($p < 0.001$), with 3D-PMMA showing the highest solubility (5.39 ± 4.10 µg/mm³), followed by 3D-Resin (3.12 ± 1.63 µg/mm³), while Milled-PMMA exhibited negative values (−0.40 ± 0.26 µg/mm³). Consequently, it was concluded that material composition and immersion duration have a significant influence on water sorption and solubility of dental polymers. CAD/CAM milled PMMA exhibited higher hydrolytic stability, whereas 3D-printed resins demonstrated higher sorption and solubility, which makes them more susceptible to hydrolytic degradation. Clinically the materials selection for digitally fabricated restorations is critical, as the increased water sorption and solubility in 3D-printed resins may affect long-term clinical performance.

مقارنة امتصاص الماء والذوبان لبوليمرات الأسنان المصنعة بتقنية CAD/CAM، وPMMA المطبوع ثلاثي الأبعاد، والراتنج المطبوع ثلاثي الأبعاد

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الكلمات المفتاحية

امتصاص الماء
الذوبان في الماء
بولي ميثيل ميثاكريلات (PMMA) بتقنية CAD/CAM
بولي ميثيل ميثاكريلات (PMMA) مطبوع ثلاثي الأبعاد
راتنج مطبوع ثلاثي الأبعاد
بوليمرات الأسنان
الثبات المائي

الملخص

هدفت هذه الدراسة إلى تقييم ومقارنة امتصاص الماء والذوبانية لثلاث بوليمرات تعويضية للأسنان (PMMA المصنع بتقنية CAD/CAM، وPMMA المطبوع ثلاثي الأبعاد، والراتنج المطبوع ثلاثي الأبعاد) بعد غمرها في الماء لفترات زمنية مختلفة. ولهذا الغرض تم تصنيع ما مجموعه 30 عينة مستطيلة الشكل من المواد الثلاث، حيث العينات في فرن جيد التهوية عند درجة حرارة 45 مئوية لمدة 24 ساعة، ثم غُمرت في ماء نقي عند درجة حرارة الغرفة، وقيست كتل العينات بعد 7 و14 و30 يومًا لحساب امتصاص الماء (W_{sp}) والذوبانية (W_{sl}) بوحدة ميكروغرام/مم³. تم اختبار افتراضات التوزيع الطبيعي وتجانس التباين وSphericity. تم تحليل الامتصاص باستخدام تحليل التباين ثنائي الاتجاه للقياسات المتكررة مع اختبار Games-Howell اللاحق؛ أما الذوبانية، التي لم تكن ضمن التوزيع الطبيعي، فقد تم تحليلها باستخدام اختبار Kruskal-Wallis مع اختبار Dunn اللاحق ($\alpha = 0.05$). وقد أظهرت النتائج أن امتصاص الماء زاد بشكل ملحوظ مع زيادة مدة الغمر في جميع المواد ($p < 0.001$)؛ حيث أظهر الراتنج ثلاثي الأبعاد أعلى قيم امتصاص، يليه PMMA ثلاثي الأبعاد، بينما سجل PMMA المصنّع بتقنية CAD/CAM أقل القيم في جميع الفترات الزمنية. وبعد مضي 30 يومًا كانت متوسطات قيم الامتصاص 16.77 ± 0.58 ميكروغرام/مم³ (PMMA المصنع بتقنية CAD/CAM)، و34.18 ± 3.68 ميكروغرام/مم³ (PMMA المطبوع ثلاثي الأبعاد)، و47.68 ± 2.07 ميكروغرام/مم³ (الراتنج المطبوع ثلاثي الأبعاد). كما اختلفت قابلية الذوبان في الماء بشكل ملحوظ بين المواد المختبرة ($p < 0.001$)، حيث أظهر PMMA المطبوع ثلاثي الأبعاد أعلى نسبة ذوبان

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(5.39 ± 4.10 ميكروغرام/مم³)، يليه الراتنج المطبوع ثلاثي الأبعاد (3.12 ± 1.63 ميكروغرام/مم³)، بينما أظهر PMMA المصنّع بتقنية CAD/CAM قيمًا سالبة (−0.40 ± 0.26 ميكروغرام/مم³). بناء على ذلك استنتج أن لتركيب البوليمر ومدة الغمر تأثيرًا كبيرًا على امتصاص الماء والذوبانية، حيث أظهر **PMMA** المصنّع بتقنية **CAD/CAM** أعلى ثبات ضد التحلل المائي، بينما أظهرت البوليمرات المطبوعة ثلاثية الأبعاد (**PMMA** والراتنج) أعلى امتصاص وذوبانية، مما يجعلهما أكثر عرضة للتحلل المائي. من الناحية السريرية يعد اختيار مواد التعويضات السنية المصنّعة رقميًا أمرًا بالغ الأهمية، فقد تؤثر زيادة امتصاص الماء والذوبانية على الأداء السريري للبوليمرات المطبوعة ثلاثية الأبعاد على المدى الطويل.

Introduction

Significant improvements in the design and fabrication of dental restorations have been achieved due to the introduction of computer-aided design and computer-aided manufacturing (CAD-CAM) technologies [1]. These digital workflows include subtractive approaches (milling) and additive techniques which known as three-dimensional (3D) printing [2].

In CAD-CAM milling, PMMA blocks polymerized under high temperature and pressure (HTHP) are utilized, resulting in low-porosity prostheses, thereby improving mechanical properties [3]. In contrast, 3D printing technology is a method for fabricating complex objects by depositing powdered or thermoplastic materials layer by layer [4]. This technology offers advantages such as digital workflow integration [5], reduced material waste [6], and customization flexibility [7]. However, their polymerization mechanisms, layer-by-layer fabrication process, and potential for incomplete monomer conversion may influence their long-term physicochemical stability [8-10].

Water sorption and water solubility are considered critical determinants of the clinical longevity and patient comfort of the denture [11]. The molecules of absorbed water diffuse between the polymer chains of the denture base and act as a plasticizing agent. This phenomenon results in internal stresses and crack formation, thereby compromising mechanical properties over time [12]. Solubility refers to the elution of unreacted components, including residual monomers, plasticizers, and initiators [13], which can cause irritation, inflammation, or allergy [14].

The presence of cross-links reduces the hole-free volume and the ability of polymer chains for swelling, thereby decreases the solvent permeability of polymer [15]. The increased water sorption and solubility is correlated with reduced degree of conversion (DC) [16].

Differences in manufacturing technologies can significantly influence water sorption and water solubility of denture base materials [17]. Materials used for CAD/CAM milling exhibit less porosity, low void content, and higher DC [12]. Water sorption and solubility of 3D-printed denture base materials are significantly affected by the printing angle [18].

Further investigations into water sorption and solubility of denture base materials are needed, due to the variations in reported findings across the literature [19]. Most available studies focus on mechanical properties or dimensional accuracy, while fewer investigations provide time-dependent gravimetric analysis across multiple early aging intervals.

According to ISO 20795-1:2013, the sorption should be less than 32 µg/mm3, while solubility should be less than 1.6 µg/mm3 [20]. Evaluating water sorption kinetics at 7, 14, and 30 days allows detection of diffusion patterns, equilibrium behavior, and early material instability. Such short-term hydrolytic assessment is clinically relevant, as the initial month following prosthesis delivery represents a critical adaptation period in the oral environment.

Therefore, systematic comparison of sorption and solubility among additively and subtractively manufactured dental restorative polymers under controlled immersion conditions is required to clarify whether emerging digital fabrication methods compromise hydrolytic stability.

The aim of this study was to evaluate and compare the time-dependent water sorption and solubility of three dental polymers (3D-printed resin, 3D-printed PMMA, and CAD/CAM milled PMMA) over a 30-day immersion period. The null hypotheses were that: (1) the type of dental polymer material would not significantly influence water sorption and solubility, and (2) that the duration of water storage would have no significant effect on these properties.

Materials and Methods

The experimental protocol for this study was adapted from ISO 20795-1:2013 for denture base polymers, with modifications in specimen geometry and immersion intervals to allow time-dependent analysis.

Materials

Three materials were used in this study; including a CAD/CAM milled PMMA (Milled-PMMA), 3D-printed PMMA (3D-PMMA) and 3D-printed resin (3D-Resin) (Table 1).

Specimen Preparation

The specimen dimensions specified in ISO 20795-1:2013 were modified because they were subsequently utilized to evaluate surface hardness, as a part of a broader research study. The selected thickness ensures adequate substrate support during hardness testing and prevents anvil effect, which can lead to inaccurate measurements.

30 (n = 10) rectangular specimens were prepared with dimensions of 30 × 15 × 3 mm. The CAD software (ExoCad, Chairside Cad 2.3 Matera, Germany) was used to design and produce standard tessellation language (STL) file, which was sent to the CAM software of the milling machine and the 3D-printer. Milling machine (SilaMill 5R; SILADENT) was utilized to mill PMMA block (DEPRAG (Zhengzhou) Dental Technology Co., Ltd.,China). Stereolithography (SLA) printer (MoonRay Model S; VertySystem) was used to fabricate the 3D-printed specimens. The printer operated at a wavelength of 405 nm and a layer thickness of 50 µm. PMMA liquid resin (iFUN, Shenzhen iFUN Technology Co., Ltd., China) was used for 3D-PMMA specimens, and SDC resin (SDC Technologies, Inc., USA) was used for 3D-Resin specimens. Specimens were printed in a horizontal (0°) orientation, removed from the build platform, and cleaned by sonication in isopropanol. Thereafter, they were immersed in a glycerol bath for 2 min and post-cured for 20 minutes in a lightbox (Moonlight; VertySystem) for final polymerization.

Sorption and Solubility

The specimens were dried in a ventilated oven at 45 °C for 24 hours. Specimens were then allowed to cool in a sealed container at room temperature for 1 hour before weighing

Table 1: Polymer dental materials used in the study

Material	Commercial name	Manufacturer	Composition	Clinical indication
CAD/CAM Milled PMMA	PMMA DISC	DEPRAG (Zhengzhou) Dental Technology Co., Ltd.,China	PMMA (Polymethyl methacrylate)	Denture base
3D-Printed PMMA	Temporary Crown	iFUN (Shenzhen iFUN Technology Co., Ltd.), China	Methacrylate oligomers and monomers (Photopolymer resin)	Crown & Bridge
3D-Printed Resin	SDC Resin	SDC Technologies, Inc., USA	Photopolymer resin	Denture base

The specimens were then weighed using a digital analytical balance with an accuracy of 0.0001 g. To achieve a constant weight, the drying and weighing were repeated, until less than 0.2 mg was observed between successive readings. The stabilized mass obtained at this stage was recorded as the initial conditioned mass (m_1). The length, width, and thickness of each specimen were measured at three points using a digital caliper (accuracy of 0.01 mm). The mean values of volume (mm³) were calculated using the following equation:

$$V = L \times W \times H \quad (1)$$

Where:

- V = volume
- L = length
- W = width
- H = height (thickness)

Each specimen was immersed in a sealed container containing 20.0 mL of deionized water to ensure independent diffusion conditions and to prevent saturation effects. The specimens were immersed at room temperature (23 ± 1 °C) for 30 days. Water was renewed every 48 hours. At the storage intervals (7, 14, and 30 days), specimens were removed, gently blotted dry with absorbent paper for 30 seconds, and immediately weighed to obtain the mass after water immersion (m_2). Following the 30-day immersion period, the same drying method for m_1 was used to achieve the reconditioned mass (m_3). Sorption values were calculated at each immersion interval (7, 14, and 30 days), while solubility was determined after the final reconditioning step. According to ISO 20795-1, three mass measurements are required for the determination of water sorption and solubility: the initial conditioned mass (m_1), the mass after water immersion (m_2), and the reconditioned mass after redrying (m_3). The water sorption and solubility percentages were calculated using the following equations:

$$W_{sp} = m_2 - m_3 / v \quad (2)$$

$$W_{sl} = m_1 - m_3 / v \quad (3)$$

Where:

- m_1 = initial conditioned mass (µg)
- m_2 = mass after immersion (µg)
- m_3 = reconditioned mass after drying (µg)
- v = specimen volume (mm³)

Statistical Analysis

Before the inferential analysis, normality was assessed using the Shapiro-Wilk test, and homogeneity of variances using Levene's test. Mauchly's test was used to evaluate sphericity. Where Levene's test indicated unequal variances, Games-Howell post-hoc comparisons were used instead of Tukey's HSD. The non-parametric Kruskal-Wallis test with Dunn's post-hoc test was used in place of one-way ANOVA, where the normality assumption was violated (solubility).

Results

Water Sorption

Mean values and standard deviations of water sorption at 7, 14, and 30 days are presented in Table 2 and illustrated in **Error! Reference source not found.Error! Reference source not found.**. In all tested polymers, water sorption increased progressively with immersion time throughout the 30-day period.

3D-Resin showed the highest sorption values at all-time points, followed by 3D-PMMA, whereas Milled-PMMA demonstrated the lowest values. At 7 days, the mean values were 12.43 ± 0.63 µg/mm³, 26.36 ± 3.84 µg/mm³, and 32.03 ± 1.87 µg/mm³ for Milled-PMMA, 3D-PMMA, and 3D-Resin, respectively. At 14 days, the values increased to 14.96 ± 0.37 µg/mm³, 31.10 ± 3.79 µg/mm³, and 42.02 ± 1.80 µg/mm³, respectively. After 30 days, they further increased to 16.77 ± 0.58 µg/mm³, 34.18 ± 3.68 µg/mm³, and 47.68 ± 2.07 µg/mm³, respectively.

Mauchly's test indicated a violation of sphericity ($W = 0.608$, $p = 0.002$); therefore, degrees of freedom were corrected using the Greenhouse-Geisser estimate ($\varepsilon = 0.718$). Sorption increased significantly with immersion time ($F(1.436, 38.779) = 789.785$, $p < 0.001$) and differed significantly among materials ($F(2, 27) = 308.426$, $p < 0.001$). In addition, it showed a significant time–material interaction ($F(2.873, 38.779) = 104.410$, $p < 0.001$), which indicates that the rate of increase depends on the polymer type.

Games-Howell post-hoc comparisons were used instead of Tukey's HSD because Levene's test indicated unequal variances among materials at all three intervals (7 days: $F(2,27) = 9.478$, $p = 0.001$; 14 days: $F(2,27) = 7.118$, $p = 0.003$; 30 days: $F(2,27) = 5.696$, $p = 0.009$). These confirmed significant differences between all three materials at every time point ($p < 0.003$), with a consistent order of sorption as follows: 3D-Resin > 3D-PMMA > Milled-PMMA.

Table 2: Means and standard deviations of water sorption (µg/mm³)

Material	7 Days		14 Days		30 Days	
	Mean	SD	Mean	SD	Mean	SD
Milled-PMMA	12.43	0.63	14.96	0.37	16.77	0.58
3D-PMMA	26.36	3.84	31.10	3.79	34.18	3.68
3D-Resin	32.03	1.87	42.02	1.80	47.68	2.07

Water Solubility

Mean values and standard deviations of water solubility are presented in Table 3 and illustrated in**Error! Reference source not found. Error! Reference source not found.**.

Table 3 Water solubility of tested materials (µg/mm³)

Material	Mean	SD
Milled-PMMA	-0.40	0.26
3D-PMMA	5.39	4.10
3D-Resin	3.12	1.63

3D-PMMA exhibited the highest mean solubility (5.39 ± 4.10 µg/mm³), followed by 3D-Resin (3.12 ± 1.63 µg/mm³),

whereas Milled-PMMA showed the lowest solubility mean with a negative value (−0.40 ± 0.26 µg/mm³).

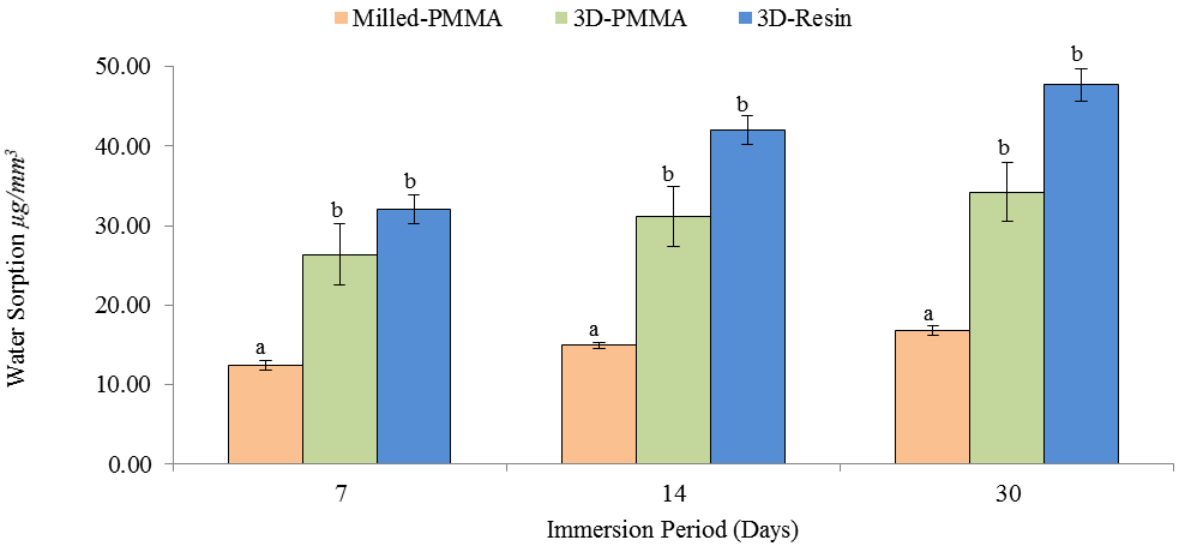

Figure 1: Water sorption of tested polymers
Different lowercase letters indicate statistically significant differences between the tested polymers ($p < 0.001$)

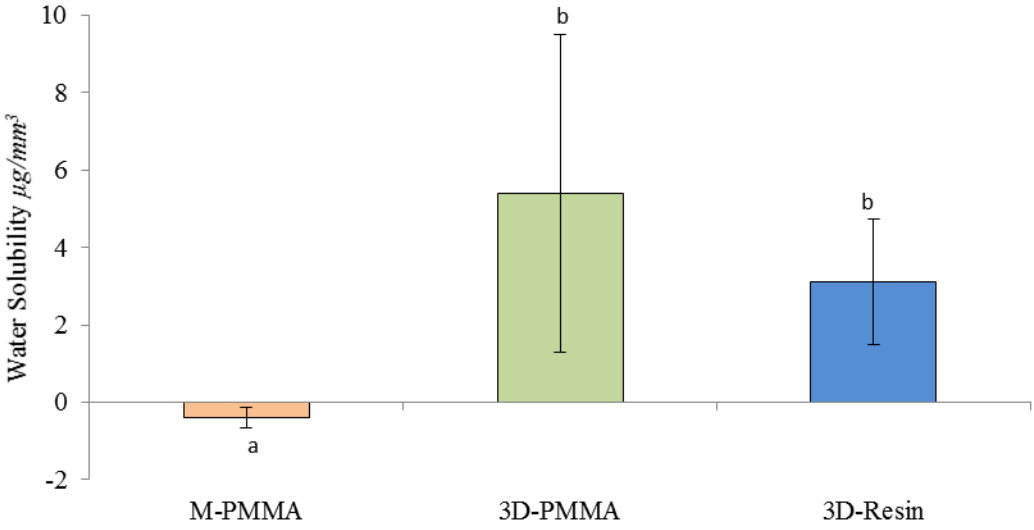

Figure 2: Water solubility of tested polymers
Different lowercase letters indicate statistically significant differences between the tested polymers ($p < 0.001$)

The Kruskal-Wallis test was performed ($H(2)=15.30$, $p<0.001$) because Shapiro-Wilk indicated non-normal distribution for 3D-Resin solubility ($W=0.812$, $p=0.020$), and Levene's indicated unequal variances ($F(2,27)=7.33$, $p=0.003$). Dunn's post-hoc (Holm-corrected) confirmed 3D-PMMA and 3D-Resin differed significantly from Milled-PMMA ($p=0.009$ and $p<0.001$, respectively), with no significant difference between 3D-PMMA and 3D-Resin ($p=0.212$).

The negative values recorded for Milled-PMMA indicated minimal mass gain after re-drying.

Discussion

According to the present results, the null hypotheses must be rejected because significant statistical differences were observed. The three tested polymers demonstrate distinct sorption and solubility behaviors. In addition, there was a significant influence of storage time on water sorption, where the values showed a progressive increase from 7 to 30 days.

In the current study, three dental polymers (Milled-PMMA, 3D-PMMA and 3D-Resin) were evaluated for water sorption and solubility after immersion in deionized water for 7, 14, and 30 days. The observations revealed that, in all tested materials water sorption increased gradually with storage time. This finding confirms that water sorption increases until equilibrium is approached [21-23]. A similar trend has been reported in previous studies [12, 24], where prolonged immersion caused mass and volume change due to diffusion-controlled water sorption. This finding is consistent with the hygroscopic nature of these polymers, where the small size of

water molecules enables them to diffuse into spaces between the polymer molecules [25].

Throughout all storage intervals, 3D-Resin displayed the highest sorption values, followed by 3D-PMMA, whereas Milled-PMMA recorded the lowest. This trend can be primarily attributed to variations in the chemical compositions of 3D-printed resins and PMMA acrylics, as the polymer type significantly influences the level of water sorption and solubility [26, 27]. This finding aligns with previous studies reporting a significant higher water sorption in 3D-printed resins compared with conventional or milled PMMA [12, 26, 28, 29]. The four studies confirmed that 3D-printed resins showed greater susceptibility to water uptake than milled or conventionally processed PMMA.

The increased sorption of 3D-printed resins can be attributed to the additive manufacturing technique, which induces internal porosities [30] and an anisotropic nature characterized by stronger intralayer than interlayer bonds [31]. Additionally, water uptake is facilitated by incomplete polymerization and residual monomer release [32], as well as the free volume and micro-voids within the cross-linked chains [33, 34].

Milled-PMMA group exhibited the lowest values at all immersion periods, indicating superior hydrolytic stability. This finding may be attributed to the HTHP polymerization utilized during fabrication of pre-polymerized PMMA pucks [35]. This process leads to the formation of longer polymer chains and resulting in a higher degree of conversion with minimal residual monomer content [36].

Regarding water solubility, significant differences were observed among the tested materials. The 3D-PMMA group demonstrated the highest solubility values, followed by the 3D-Resin, whereas the Milled-PMMA showed the lowest solubility and even negative values in several specimens. The Increased solubility in 3D-printed resins has been attributed to the leaching of residual monomers and soluble additives [37, 38]. These observations align with the findings of Sagen et al. [39] who reported that total monomer leaching from 3D-printed resins was significantly higher than that of milled resins. Similarly, Wedekind et al. [40] demonstrated that 3D-printed resins released the highest concentration of leachable components, whereas milled and conventional resins exhibited no detectable elution. The negative solubility values in Milled-PMMA group indicating minimal degradation and material dissolution [10].

Most water sorption values were below the maximum threshold of 32 µg/mm³ specified by ISO 20795-1. However, 3D-Resin exceeded this limit at later time points. This result indicates that Milled-PMMA and 3D-PMMA remained within the acceptable limits, while the 3D-Resin concerns regarding long-term hydrolytic stability.

Increased water sorption and solubility may adversely influence mechanical properties, dimensional and color stability [41-43]. Water absorbed acted as a plasticizer, lowering the intermolecular forces of the polymer chains, leading to a reduced stiffness [44], as well as promoting microcrack formation and resin degradation [45]. In addition, leaching of non-converted monomers, oligomers, catalysts, silane and other by-products can increase surface roughness [46], which facilitates microbial adhesion [47-50].

Using one commercial product per material category in addition to immersing the specimens in conditions did not fully simulate the biochemical complexity of the oral cavity are limitations of this study. Moreover, the immersion period was limited to 30 days, which does not reflect the long-term clinical performance of the tested polymers.

Conclusion

Water sorption showed a progressive increase in all the three investigated dental polymers, indicating time-dependent water uptake. Milled-PMMA exhibited the lowest water sorption and solubility values at all immersion intervals. In contrast, the 3D-Resin demonstrated the highest water sorption, whereas 3D-PMMA yielded the highest solubility. These observations suggest that manufacturing technique and material composition have a significant influence on the hydrolytic stability of dental polymers.

Recommendations

Future studies should investigate the long-term hydrolytic degradation of these polymers. Studies evaluate the correlation between water sorption/solubility and the mechanical properties (e.g., fracture toughness, flexural strength, and impact strength) of the tested polymers are also recommended.

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