Two-body wear and surface hardness of occlusal splint materials

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The aim of this in vitro study was to evaluate the wear and surface hardness of nine materials for conventional manufacturing, subtractive milling, and 3D printing of occlusal splints, as well as to evaluate the differences in wear and surface hardness between rigid and flexible 3D-printed occlusal splint materials. Two-body wear and Vickers hardness tests were performed. The vertical wear depth and Vickers hardness values were statistically analyzed. Vertical wear depth and surface hardness values were statistically significant among the investigated materials (p<0.05). The lowest vertical wear depth was observed for the heat-cured resin (27.5±2.4 μm), PMMA-based milled material (30.5±2.8 μm), and autopolymerizing resin (36.7±6.3 μm), with no statistical difference (p>0.05). Flexible 3D-printed and CAD-CAM milled polycarbonate-based splint materials displayed lower surface hardness and higher wear than the PMMA-based materials. PMMA-based splint materials displayed the most consistent surface hardness and wear resistance regardless of the manufacturing technology.

Keywords: Two-body wear, Occlusal splints, 3D printing, CAD/CAM, Surface hardness

INTRODUCTION

Temporomandibular joint and muscle disorders (TMJDs) are the second most frequent musculoskeletal diseases causing pain and impairment11. Disorders are associated with recurring or chronic pain and dysfunction of the jaw joint, as well as its related muscles and supporting structures. TMJDs affect between 5% and 12% of the population with a higher prevalence rate among young people and women2. Intraoral occlusal splints are one of the most prevalent treatments for symptomatic TMJDs, and are shown to be effective in 70–90% of cases3,4. The mechanism of action of occlusal splints can be explained by disrupting the cycle of neuromuscular reflex contraction and inducing muscle relaxation in patients with parafunctional habits. Additionally, they protect teeth from wear and mobility caused by clenching and grinding, distribute the occlusal force exerted on the teeth, and relocate the jaws and condyles into centric relation5.

Occlusal splints are often made of polymers. They can be conventionally fabricated by using traditionally heat-cured polymethyl methacrylate (PMMA), vacuum-formed thermoplastic resins, or autopolymerizing PMMA6–7. Alternatively, computer-aided design and computer-aided manufacturing (CAD-CAM) technologies can be used for the digital production of occlusal splints8. A variety of prefabricated standard blanks made of high-performance polymers are available on the market for milling precisely fitted splints, with superior or at least equivalent properties compared to conventional ones7,8. Furthermore, occlusal splints have been additively manufactured by three dimensional (3D) printing9–11, which was presented for the first time in 201312. Including ultra violet (UV) absorbers into the resin has made it possible to print transparent objects, which is especially useful for the production of occlusal devices, implant drilling guides, and clear surgical models7,13. Such technologies enable the rapid and simplified implementation of various treatment options while enhancing the quality control process and employing a wider range of materials10.

Hard and soft splints have been prescribed as a conservative treatment for TMJDs15,16. Soft splints, with their flexible and resilient material properties are able to readily disperse the heavy stresses experienced during parafunctional activities with a high level of patient tolerance16. Hard splints aim at reducing TMJDs symptoms by modifying the occlusal equilibrium, correcting the vertical dimension, and rectifying condylar position17. Previous studies15,16 reported that either a hard or soft orthopedic device can be successfully utilized to alleviate muscular pain in individuals with TMJDs. However, minimal occlusal changes have been associated with the use of soft occlusal splints18,19.

Splints are considered as orthotic devices since they are used to protect the dentation from parafunctional habits, such as diurnal or nocturnal bruxism2,20–22. Maximum occlusal forces in patients with bruxism range from 450 to 650 N22, with a mean of 380 N22. Therefore, wear resistance and surface hardness of these materials is of clinical importance. Materials with inadequate wear resistance may develop wear facets, which hinder occlusal contact stability and reduce the appliance lifespan20,25.

The aim of this in vitro study was to evaluate the

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wear and surface hardness of nine commercially available materials for conventional manufacturing, subtractive milling and 3D printing of occlusal splints, as well as to evaluate the differences in surface hardness and wear between rigid and flexible 3D-printed occlusal splint materials. The first research null hypothesis was that the different splint materials would have similar wear and surface hardness, while the second hypothesis was that there would be no differences in surface hardness and wear between rigid and flexible 3D-printed occlusal splint materials.

**MATERIALS AND METHODS**

**Preparation of specimens**
A total of nine different occlusal splint materials were evaluated, which were either heat-cured, autopolymerized, milled, or 3D-printed (Table 1). One of the tested 3D-printed materials is intended for the fabrication of rigid splints, while the rest are used for

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**Table 1** Occlusal splint materials included in the study identified by material commercial name, manufacturer, manufacturing technique, and composition as per the manufacturer's data sheet

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Resin type based on manufacturing technique</th>
<th>Chemical composition from the manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paladon 65</td>
<td>Kulzer, Hanau, Germany</td>
<td>Heat-cured resin</td>
<td>Liquid: methylmethacrylate (&gt;90%); tetramethylene dimethacrylate (≥1–≤5%); p-Mentha-1,4-diene (&lt;0.25%)&lt;br&gt;Powder: (based on methacrylate copolymers) methylmethacrylate (≥1–≤5%); dibenzoyl peroxide (≥0.25–&lt;1%)</td>
</tr>
<tr>
<td>Palapress</td>
<td>Kulzer</td>
<td>Autopolymerizing resin</td>
<td>Liquid: methylmethacrylate (&gt;90%); tetramethylene dimethacrylate (≥1–≤5%); maleic acid (&lt;0.1%); 2-Hydroxy-4-methoxy benzophenone (&lt;0.25%); mequinol (&lt;1%); Quaternary ammonium compounds, tri-C8-10-alkylmethyl, chlorides (≥0.025–&lt;0.25%)&lt;br&gt;Powder: (based on methacrylate copolymers) dibenzoyl peroxide (≥1–&lt;2.5%); methyl methacrylate (≥1–≤5%); 1-Benzyl-5-phenylbarbitursäure (≥0–≤5%)</td>
</tr>
<tr>
<td>Cast</td>
<td>Degos Dental, Regenstauf, Germany</td>
<td>CAD-CAM milled resin discs</td>
<td>PMMA</td>
</tr>
<tr>
<td>Aqua</td>
<td>Degos Dental</td>
<td>CAD-CAM milled resin discs</td>
<td>PMMA</td>
</tr>
<tr>
<td>Temp Premium</td>
<td>Zirkonzahn, South Tyrol, Italy</td>
<td>CAD-CAM milled resin discs</td>
<td>Polycarbonate</td>
</tr>
<tr>
<td>Flexible Transpa</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>IMPRIMO LC Splint</td>
<td>Scheu-Dental, Iserlohn, Germany</td>
<td>Light curing resin for 3D printing of splints with relative flexibility (rigid splints)</td>
<td>(Methacrylate-based resin) esterification products of 4,4'-isopropylidenediphenol, ethoxylated and 2-methylprop-2-enoic acid (&gt;95%); Diphenyl-(2,4,6-trimethylbenzoyl)phosphine oxide (&lt;2%)</td>
</tr>
<tr>
<td>IMPRIMO LC Splint flex</td>
<td>Scheu-Dental</td>
<td>Light curing resin with high flexibility and thermo-active memory effect for 3D printing of transparent occlusal splints as well as for bruxism and lower jaw protrusion splints</td>
<td>(Methacrylate-based resin) methacrylate monomer 1(&lt;60%); methacrylate monomer 2 (&lt;40%); methacrylate monomer 3 (&lt;2%), photo initiator (2%)</td>
</tr>
<tr>
<td>KeySplint Soft</td>
<td>Keystone, Singen, Germany</td>
<td>Light curing resin for 3D printing of flexible dental splints and night guards</td>
<td>Proprietary Ingredient</td>
</tr>
<tr>
<td>V-Print splint comfort</td>
<td>VOCA, Cuxhaven, Germany</td>
<td>Light-curing resin for the generative production of thermoflexible dental, therapeutic splints</td>
<td>Aliphatic acrylate (25–50%); triethylene glycol dimethacrylate 5-(10%); Diphenyl-(2,4,6-trimethylbenzoyl)phosphine oxide (&lt;2.5%)</td>
</tr>
</tbody>
</table>
the fabrication of occlusal splints with high flexibility.

To obtain heat-cured and autopolymerizing resin specimens, the respective components were mixed according to the manufacturer’s instructions and placed in Teflon molds, that were then transferred to a pressure pot (Ivomat IP3, Ivoclar Vivadent, Schaan, Liechtenstein) for curing. Autopolymerizing resin specimens were cured for 15 min in water bath at a temperature of 55°C and a pressure of 200 kPa, while heat-cured specimens were cured in a water bath at 90°C for 30 min and then allowed to cool slowly in the water bath. For the CAD-CAM milled groups, a puck of the material was cut to the proposed dimensions at low speed using a water-cooled diamond saw (Secotom 50, Struers, Ballerup, Denmark). For 3D-printed splint materials, an STL file with specimen dimensions was created in AutoCAD (Autodesk, San Rafael, CA, USA) and printed in a 3D printer (Asiga MAX™, Asiga, Sydney, Australia) at 90° orientation to the build platform and with a layer thickness of 100 μm, followed by postcuring (Form cure, Formlabs, Berlin, Germany) according to the manufacturer’s instructions. Wet polishing of the specimens was performed with a polishing machine (LabPol-21, Struers) using 800 and 1200 grit FEPA silicon carbide abrasive papers (Buehler, NY, USA).

Wear test

Similar to prior studies, four specimens of each splint material (thickness: 2 mm, length: 10 mm, width: 15 mm) were tested for wear. Each specimen was mounted to an acrylic resin block and polished sequentially on a rotary machine using silicon carbide sheets with grain sizes up to 4000 grit FEPA. Specimens were stored in water at 37°C for 24 h before testing. A chewing simulator machine (CS-4.2, SD Mechatronik, Feldkirchen-Westerham, Germany) was utilized for conducting the two-body wear test, which comprises two chambers simulating vertical and horizontal movements in the presence of water at the same time. Each chamber has a lower plastic sample holder for inserting the specimen, as well as an upper antagonist for retaining the loading tip. The manufacturer’s standard loading tips (Steatite ball, 6 mm) were embedded in autopolymerizing acrylic resin material within a plastic ring, and then fixed in the upper antagonist with a fastening screw. A chewing simulation was performed at 1.5 Hz with vertical weight of 2 kg, yielding 20 N of chewing force. Each specimen was exposed to 15,000 loading cycles. Wear patterns were scanned with a 3D optical microscope (Bruker Nano, Berlin, Germany), and then analyzed with Vision64 Map software for material loss measurements. Total wear depth values (μm) were obtained from various sites, according to the average of the deepest points of all profile scans.

Additionally, after evaluating the vertical wear depth, representative specimens of each material were examined by scanning electron microscopy (SEM; JSM 5500, JEOL, Tokyo, Japan) to analyze the wear facets and surface features. Before SEM examination, the specimens were gold sputtered with a gold layer in a vacuum evaporator using a sputter coater (BALTec SCD 050 Sputter Coater, BAL-TEC, Balzers, Liechtenstein).

Surface hardness measurement

The Vickers surface hardness (VHN) of dry and water-stored (30 days at 37°C) bar specimens (4×10×10 mm²) was measured using a microhardness testing device (Duramin-5, Struers) with a load of 1.96 N applied for 15 s. The specimens were cleaned in an ultrasonic cleaning device (Quantrex 90, L&R Ultrasonics, Kearny, NJ, USA) in deionized water for 10 min before testing. An average of three measurements was calculated from each specimen (n=16/material). The imprints’ diagonal lengths were measured with the aid of the eyepiece operator of the testing machines and then used in the calculation of VHN values.

Statistical analysis

Statistical software was used to conduct the analysis (SPSS V25, IBM, Armonk, NY, USA). Vertical wear depth and surface hardness data were analyzed by using one-way ANOVA while applying Dunnett’s T3 from multiple comparison tests to determine the differences between materials. To compare the values of surface hardness of dry and water-stored specimens for each group of materials separately, the independent samples t test was used. The results of the analysis were presented as means and standard deviations, and a p value of 0.05 was considered significant. A Pearson correlation coefficient was made to evaluate the relationship between the vertical wear depth and dry surface hardness values of the investigated splint materials.

RESULTS

Figure 1 and Table 2 demonstrate descriptive statistics for the obtained values of vertical wear depth and surface hardness. There was a statistically significant difference among the investigated materials in the vertical wear depth (p<0.001) and surface hardness of dry (p<0.001) and water-stored (p<0.001) specimens.

The lowest vertical wear depth was observed in Paladon 65 (27.5±2.4 μm), Cast (30.5±2.8 μm), and Palapress (36.7±6.3 μm), with no statistical difference (p>0.05). Meanwhile, Temp Premium Flexible Transpa, V-Print splint comfort, and IMPRIMO LC Split flex had the highest vertical wear depth values, with a statistically non-significant difference from each other (p=1.000, p=0.708, p=0.999). Interestingly, the flexible 3D-printed splint material, KeySplint Soft was not statistically different from Palapress (p=0.991), Cast (p=0.999), and Aqua (p=1.000), while being significantly lower than the rigid 3D-printed splint material, IMPRIMO LC Split (p=0.037). The other two flexible 3D-printed splint materials, V-Print splint comfort and IMPRIMO LC Split flex were significantly higher than IMPRIMO LC Split (p<0.05).

Figure 2 described SEM images of the tested materials after chewing simulation. Materials
specimens displayed visual signs of wear in the form of pits, valleys, scratches, cracks and irregularities. There were differences in wear trends and facet size among the investigated materials. All materials showed grooves oriented parallel with the sliding directions. Paladon 65, Palapress, Aqua, Cast, and IMPRIMO LC Splint materials showed uniform wear facets. Plastic deformation was noticed in the wear facets of IMPRIMO LC Splint flex, V-Print splint comfort, and KeySplint Soft. Paladon 65 and Cast showed a smoother surface at high magnification.

As seen in Table 2, water storage tended to cause a significant reduction in surface hardness for all of the materials except KeySplint Soft ($p=0.737$). The milled PMMA-based resins, Aqua and Cast, respectively displayed the highest surface hardness after water storage. In addition, Paladon 65 was not significantly different from Cast ($p=0.163$). While the lowest surface
hardness was displayed by the three flexible additively manufactured splint materials, KeySplint Soft, IMPRIMO LC Splint flex, and V-Print splint comfort ($p>0.05$). A relatively moderate correlation was detected between the vertical wear depth and the surface hardness ($r^2=−0.596, p<0.001$) for the investigated materials.

**DISCUSSION**

In this *in vitro* study, the surface hardness and material surface wear of various conventionally manufactured, milled, or 3D-printed occlusal splint materials with variable degrees of flexibility were investigated. While clinical studies have significant limitations, such as complex procedures, high cost, time constraints, and metrological and analytical difficulties, laboratory studies are commonly used to evaluate the wear resistance of a material. Such studies are useful for understanding the basic wear mechanisms. Statistical analysis revealed significant differences in the evaluated aspects between materials. At the same time, significant differences were found between rigid and flexible 3D-printed splint materials. Consequently, the null hypotheses of the study were rejected.

Wear can be defined as the amount of material removed. Unlike restorative materials, occlusal devices are not exposed to abrasive intermediate substrates such as food. Therefore, a 2-body wear test was performed using a chewing simulator that complies with wear test guidelines. The test load was set at 20 N, however, load values of 50 N and 5 N have been documented in the literature. Considering the anisotropicity of additively manufactured splints, all the tested specimens were printed with a layer thickness of 100 μm while the printing angle was 90°.

The current study showed that the differences in the investigated properties between materials were not only related to the different manufacturing technologies, but also to their chemical composition. PMMA-based (conventionally manufactured and CAD-CAM milled) occlusal splint materials had the lowest surface wear. While both CAD-CAM milled polycarbonate and additively manufactured flexible splint materials, excluding KeySplint Soft, displayed the highest wear. Furthermore, significant differences in wear behavior and surface hardness were observed between rigid and flexible 3D-printed splint materials. The latter displayed plastic deformation after wear testing, as shown by SEM examination (Figs. 2G, H, I). The flexible resins used in 3D printing are UV-curable elastomers characterized by a low modulus of elasticity and low Shore hardness, but with higher flexibility, elongation at break, and elastic rebound. The mechanical behavior of photocurable resins used for additive manufacturing can be controlled by modifying their composition which is based on liquid monomers, oligomers, and photoinitiators. Patel *et al.* were able to increase the stretchability of a printable elastomer up to 1,100% by mixing a monofunctional monomer of epoxy aliphatic acrylate (EAA) with aliphatic urethane diacrylate (AUD) oligomer at different ratios.
Unfortunately, the reported compositions for the 3D printing resin products are quite general, vague, and provide extremely limited information, as seen in Table 1, making it difficult to explain the results based on compositions.

Due to the variation in wear test parameters such as wetting medium, indenter material and design, number of chewing cycles, applied movements/rotations, and assessment techniques, an absolute comparison of results between published studies was not applicable. Grymak et al.\textsuperscript{39} found that 3D-printed splint materials exhibited lower wear resistance than the heat-cured and CAD-CAM milled PMMA materials. In contrast, in the study by Wesemann et al.\textsuperscript{31}, conventional, milled, and additionally manufactured occlusal splints displayed similar wear resistance after 200,000 loading cycles at 20 N and 50 N, respectively.

Hardness is particularly significant since it shows the ability of a material to resist scratches and applied occlusal loads\textsuperscript{40}. Although some studies showed a correlation between surface hardness and wear resistance\textsuperscript{29,43}, others\textsuperscript{42} differed. Since occlusal devices are susceptible to intraoral moisture, surface hardness was measured both in dry specimens and after 30 days of water storage at 37°C. Water storage led to a significant decrease in surface hardness for the majority of the investigated materials, especially the additively manufactured ones, which recorded a decrease between 27.5% and 36.4%. This could be explained by the previously reported higher water absorption for stereolithographically processed photopolymers due to microscopic voids between resin layers, which adversely affect their mechanical properties\textsuperscript{31,33,43}. While CAD-CAM milled and conventional PMMA-based splint materials were the hardest, the 3D-printed materials, especially the flexible ones, displayed the lowest surface hardness values. In a study conducted by Prpic et al.\textsuperscript{44}, milled and conventional acrylic-based resin materials were harder than 3D-printed materials. The manufacturing processes of CAD-CAM milled blocks result in dense packing of the PMMA material creating a homogeneous porosity-free structure\textsuperscript{34,35}. On the other hand, in 3D printing, layers are deposited parallel to the loading direction, resulting in low mechanical properties, while the adhesion between successive layers is lower than the strength within the layer itself\textsuperscript{40}. Moreover, acrylic ester-based monomers used in 3D printing have comparatively low double bond conversion compared to standard acrylic resins\textsuperscript{40}.

Although thermoplastic resins such as polycarbonates are known for their high impact strength, fracture resistance, and flexibility\textsuperscript{47}, they exhibited lower wear resistance and surface hardness than PMMA-based polymers, which was consistent with previous studies\textsuperscript{38,49}.

Despite flexible splints may be superior in terms of patient tolerance\textsuperscript{38} rigid PMMA-based ones would be more convenient for bruxism patients and long-term treatment due to their higher wear resistance and surface hardness. The results of the study suggest differences between materials fabricated by different techniques, which should be taken in consideration when selecting a material for the fabrication of occlusal splints depending on the patient situation and needs.

A limitation of the present study was the lack of wear testing after prolonged water storage. In addition, the influence of the printing direction as well as the layer thickness of the 3D-printed splint materials was not investigated.

**CONCLUSIONS**

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The wear and surface hardness of occlusal splints depend more on the material composition rather than the manufacturing technology.
2. PMMA-based materials had the most consistent surface hardness and wear resistance regardless of the manufacturing technology.
3. Flexible 3D-printed and CAD-CAM milled polycarbonate-based resins displayed lower surface hardness than the PMMA-based splint materials.
4. With the exception of KeySplint Soft, the flexible 3D-printed and milled polycarbonate-based splint materials exhibited the greatest wear among the investigated materials, potentially limiting their use for bruxism patients and long-term treatments.

**REFERENCES**


