THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

A study of the flexural strength and surface hardness of different materials and technologies for occlusal device fabrication

Vladimir Prpic, DMD,[a] Ivan Slacanin, MME,[b] Zdravko Schauperl, BSME,[c] Amir Catic, DMD,[d] Niksa Dulcic, DMD,[e] and Samir Cimic, DMD[f]

ABSTRACT

Statement of problem. With the emergence of digital technologies, new materials have become available for occlusal devices. However, data are scarce about these different materials and technologies and their mechanical properties.

Purpose. The purpose of this in vitro study was to investigate the flexural strength and surface hardness of different materials using different technologies for occlusal device fabrication, with an emphasis on the digital technologies of computer-aided design and computer-aided manufacturing (CAD-CAM) and 3D printing.

Material and methods. A total of 140 rectangular specimens were fabricated from two 3D-printed (VarseoWax Splint and Ortho Rigid), 2 CAD-CAM–produced (Ceramill Splintec and CopraDur), and 3 conventional autopolymerizing occlusal device materials (ProBase Cold, Resilit S, and Orthocryl) according to ISO 20795-1:2013. Flexural strength and surface hardness were determined for 10 specimens of each tested material using the 3-point bend test and the Brinell method. The data were analyzed using descriptive statistics and 1-way ANOVA with Bonferroni corrections (α=.05).

Results. Surface hardness values ranged from 28.5 ±2.5 MPa to 116.2 ±1.6 MPa. During flexural testing, neither the CopraDur nor the VarseoWax Splint specimens fractured during loading within the end limits of the penetrant's possible movement. Flexural strength values for other groups ranged from 75.0 ±12.0 MPa to 104.9 ±6.2 MPa. Statistical analysis determined significant differences among the tested materials for flexural strength and surface hardness.

Conclusions. Mechanical properties among different occlusal device materials were significantly different. Acrylic resins were less flexible than polyamide and nonacrylic occlusal device materials for 3D printing but had higher and more consistent values of surface hardness. Clinicians should consider the different mechanical properties of the available materials when choosing occlusal device materials. (J Prosthet Dent 2019;121:955-9)

Temporomandibular disorder (TMD) is a collective term that involves several clinical problems affecting the masticatory muscles, temporomandibular joints, and associated structures.[1] Bruxism is defined as a diurnal or nocturnal parafunctional activity that includes unconsciously clenching, grinding, or bracing the teeth.[2] The incidence of TMD is over 10% in the general population,[3] whereas some studies confirm an overall 8% incidence of bruxism, although this differs with age.[4,5] Occlusal devices are often used to manage TMD symptoms and prevent the negative effects of bruxism on the stomatognathic system.

Occlusal devices are usually made of poly(methyl methacrylate) (PMMA)-based polymers, whose mechanical properties and ease of use represent the gold standard for occlusal device material. Recently, although the most common technique of occlusal device fabrication remains vacuum-thermoforming foil and autopolymerizing PMMA,[6] occlusal devices can be made by using computer-aided design and computer-aided manufacturing (CAD-CAM)

This study was supported, in part, by University of Zagreb scientific support "Diagnostic and therapy of craniomandibular dysfunctions."
[a]Doctoral student, School of Dental Medicine, University of Zagreb, Zagreb, Croatia.
[b]Postgraduate student, Faculty of Mechanical Engineering and Naval Architecture, University of Zagreb, Zagreb, Croatia.
[c]Associate Professor, Department for Materials, Faculty of Mechanical Engineering and Naval Architecture, University of Zagreb, Zagreb, Croatia.
[d]Associate Professor, Department of Prosthodontics, School of Dental Medicine, University of Zagreb, Zagreb, Croatia.
[e]Associate Professor, Department of Prosthodontics, School of Dental Medicine, University of Zagreb, Zagreb, Croatia.
[f]Assistant Professor, Department of Prosthodontics, School of Dental Medicine, University of Zagreb, Zagreb, Croatia.

Clinical Implications

With the development of digital technologies, new materials for occlusal device fabrication have become available, and their mechanical properties differ from those of the conventional autopolymerizing acrylic materials. Polyamide and nonacrylic occlusal device materials for 3D printing are more flexible and have a lower hardness than acrylic resin (regardless of acrylic resin fabrication technology). Clinicians should consider these differences when choosing a material for occlusal device fabrication.

or by additive manufacturing.[6-8] Occlusal device production using the CAD-CAM technology has 3 main requirements: data acquired directly through intraoral scanners or indirectly through a dental stone cast, software to design a virtual restoration, and a computerized milling device.[9]

Stereolithography is the type of additive technology most frequently used for 3D printing occlusal devices. Stereolithography photopolymers are polymerized from liquid to solid under ultraviolet light. The light-polymerizing resin is polymerized layer by layer until the final size of the object (occlusal device) according to the standard tessellation language file is achieved. New technologies imply the adaptation of existing materials or the development of new ones, depending on the technology and the purpose of the material.[10] The materials used for occlusal device fabrication with CAD-CAM or 3D printing are acrylics, polyamides, or other resins. These materials must have appropriate biomechanical properties.[11]

Unlike denture materials, which have been frequently investigated,[12-23] published data are scarce on the different materials and technologies used for occlusal device fabrication and their resulting mechanical properties. The purpose of this in vitro study was to investigate the flexural strength and surface hardness of different materials using different technologies for occlusal device fabrication, with an emphasis on the digital technologies of CAD-CAM and 3D printing. The null hypothesis was that different materials would have similar flexural strength and surface hardness.

MATERIAL AND METHODS

Two types of CAD-CAM materials, 2 types of 3D printing materials, and 3 conventional materials (autopolymerizing acrylic resins) for occlusal device fabrication were selected. A list of the materials, manufacturers, types, and occlusal device fabrication techniques is shown in Table 1. Most of the materials used were acrylic resins (ProBase Cold [PRC], Orthocryl [ORT], Resilit S [RES], Ceramill Splintec [CSP], and Ortho Rigid [ORR]); 1 was a polyamide resin (CopraDur [COP]), and VarseoWax Splint (VWS) was a poly(oxy-1,2-ethandiyl), alpha, alpha′-[(1-methylethyliden)di-4 1-phenylene]bis [omega-[(2-methyl-1-oxo-2-propenyl)oxy]-based material.

Table 1. Materials, types, manufacturers, and occlusal device fabrication techniques

Material	Abbreviation	Type	Manufacturer	Occlusal Device Fabrication Technique
ProBase Cold	PRC	PMMA	Ivoclar Vivadent AG	Conventional; autopolymerizing
Orthocryl	ORT	PMMA	Dentaurum KG	Conventional; autopolymerizing
Resilit S	RES	PMMA	Erkodent Erich Kopp	Conventional; autopolymerizing
Ceramill Splintec	CSP	PMMA	Amann Girrbach AG	CAD-CAM
CopraDur	COP	Cross-linked polyamide	Whitepeaks Dental Solutions KG	CAD-CAM
VarseoWax Splint	VWS	Nonacrylic light-polymerizing resin	Bego KG	3D printing
Ortho Rigid	ORR	Acrylic light-polymerizing resin	Next Dent B.V.	3D printing

CAD-CAM, computer-aided design and computer-aided manufacturing; PMMA, poly(methyl methacrylate).

Power analysis to estimate the appropriate sample size was based on the results of the study by Ayaz et al,[15] who found mean flexural strengths of 89.1 ±7.5 MPa and 69.6 ±4.1 MPa for PMMA and polyamide denture base materials, respectively. The effect size was hypothesized to be 1.4. Accordingly, with α=.05 and β=.95, the projected sample size needed was n=10 (GPower 3.1), as in similar studies.[6,20,22,23]

A total of 140 rectangular specimens (64.0×10.0×3.3 ±0.2 mm) were fabricated for flexural strength and surface hardness testing. All the testing procedures were performed according to ISO standards (ISO 20795-1:2013[24]; ISO 2039-1:2001[25]). A specimen plate (block) each of ORT, RES, and PRC (conventional autopolymerizing resins) was prepared from a silicone mold (Elite HD; Zhermack SpA) according to the manufacturer's instructions. After the monomer and polymer were mixed in an appropriate ratio and the silicone mold was filled, the mold was placed into a pressure-polymerizing unit (Ivomat IP3; Ivoclar Vivadent AG) at 0.22 MPa for 15 minutes. A VWS and an ORR specimen plate were prepared according to the standard tessellation language (STL) file in an appropriate light-polymerizing unit (VWS: Varseo 3D-Printer [Bego KG]; ORR: DentalFab [Microlay]) following postpolymerization (VWS: Bego Otoflash [Bego KG]; ORR: LC-3DPrint Box [NextDent B.V.]). Polymerization

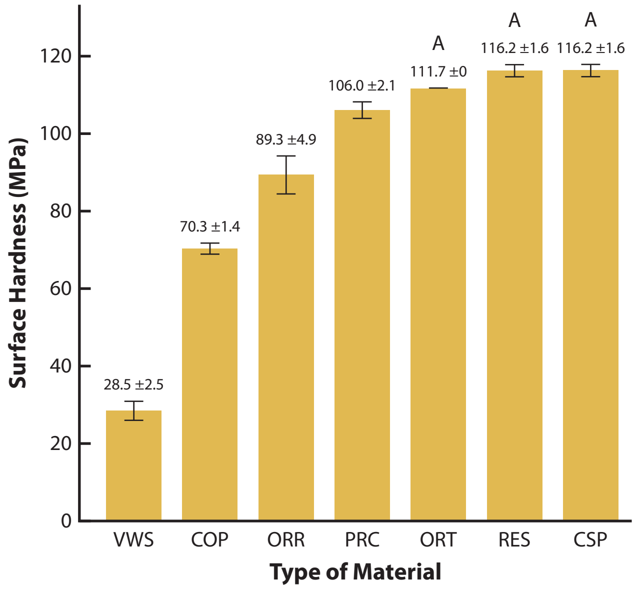

Figure 1. Means and standard deviations of surface hardness for groups. Similar uppercase letters denote no significant differences among groups (Bonferroni post hoc test, *P*>.05). COP, CopraDur; CSP, Ceramill Splintec; ORR, Ortho Rigid; ORT, Orthocryl; PRC, ProBase Cold; RES, Resilit S; VWS, VarseoWax Splint.

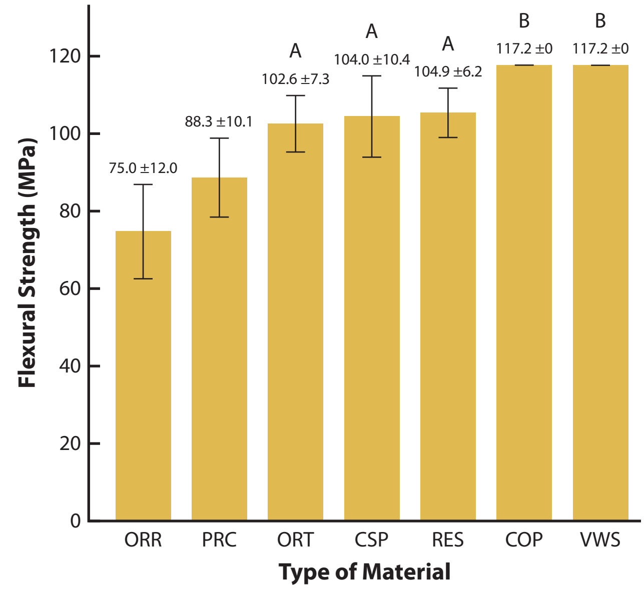

Figure 2. Means and standard deviations of flexural strength for groups. Similar uppercase letters denote no significant differences among groups (Bonferroni post hoc test, *P*>.05). For groups COP and VWS, neither specimen fractured during loading within end limits of possible movement of penetrant. For inclusion of these 2 groups in statistical analysis, arbitrarily high number was assigned (maximum flexural strength value obtained in other groups plus 1). COP, CopraDur; CSP, Ceramill Splintec; ORR, Ortho Rigid; ORT, Orthocryl; PRC, ProBase Cold; RES, Resilit S; VWS, VarseoWax Splint.

and postpolymerization were conducted according to the manufacturer's instructions.

Slightly oversized specimens were cut of conventional (ORT, RES, and PRC) and 3D-printed specimen plates (VWS and ORR) and CAD-CAM blocks (CSP, and COP) using a water-cooled milling machine (IsoMet 1000; Buehler). All sides of the specimens were wet-ground using standard metallographic grinding papers (P500, P1000, and P1200) to the required width, height, and surface smoothness. Flexural strength was measured according to ISO 20795-1:2013[24] in a 3-point bend test using a universal testing machine (VEB Werkstoffprüfmaschinen). Ten specimens of each material were tested. Specimens were immersed in a water bath for 50 ±1 hours at 37°C before testing. After random removal from the water (randomization chart was made using Excel; Microsoft), each specimen was immediately laid symmetrically with the flat surface on the supports of the flexural test rig. The force on the loading plunger was increased uniformly from zero by using a constant displacement rate of 5 ±1 mm/min until the specimen fractured. The flexural strength of each specimen was calculated in megapascals (MPa) according to the following formula: $FS = 3FL/2bh^2$, where FS is flexural strength, F is the maximum load exerted on the specimen in newtons (N), L is the distance between the supports (mm), b is the width of the specimen (mm), and h is the height of the specimen (mm).

Ten specimens of each material were measured for surface hardness (Brinell method, ISO 2039-1:2001[25]). Brinell hardness was calculated from the equation: $HB = F/\pi Dh_k$, where HB is Brinell hardness (MPa), F is load applied to the specimen (N), D is diameter of the indenter (mm), and h_k is penetration depth (mm). Brinell hardness was determined for each specimen using a Zwick apparatus (Zwick Roell Group). A 358-N load was applied through the indenter with a dwell time of 60 seconds (for 1 tested material, VWS, a 132-N load was used because of that material's lower hardness). Brinell hardness was measured at 5 locations on each specimen, and mean hardness was determined for each specimen.

Descriptive statistics were calculated for all study groups. One-way ANOVA and Bonferroni post hoc tests were performed using a statistical software program (SPSS Statistics v17.0; SPSS Inc) to compare the tested groups of materials (α=.05).

RESULTS

Descriptive statistics for the obtained values of surface hardness and flexural strength are presented in Figures 1 and 2. All specimens in the COP and VWS groups were loaded to the end limits of the possible movement of the penetrant. Because neither specimen fractured, flexural strength for the COP and VWS groups could not be measured. To include these 2 groups in statistical analyses, an arbitrarily high value was assigned (maximum flexural strength value obtained in other groups plus 1). One-way ANOVA showed statistically significant differences among the tested groups for surface hardness (F=879.9, $P<.05$) and flexural strength (F=36.2, $P<.05$). Results of the Bonferroni post hoc test for flexural strength and surface hardness are presented in Figures 1 and 2. One-way ANOVA showed statistically significant differences among different fabrication techniques in flexural strength (F=5.1, $P<.05$) and surface hardness values (F=17.7, $P<.05$). The Bonferroni post hoc test determined higher flexural strength values in the CAD-CAM materials than in the autopolymerized materials ($P=.042$) and 3D-printed materials ($P=.011$), and lower surface hardness values were found for the 3D printing materials than for the autopolymerizing ($P<.001$) and CAD-CAM materials ($P=.004$).

DISCUSSION

The flexural strength and surface hardness of different occlusal device materials produced with different techniques, especially digital technologies, were investigated. Because statistical analysis demonstrated between-material differences in flexural strength and surface hardness, the null hypothesis was rejected.

Surface hardness describes the density of a material and its resistance to wear and/or scratching, and it affects prosthetic restorations during function and cleaning.[12] Because occlusal loads in functional or parafunctional activity, especially bruxism, can be higher than 785 N,[26] the wear resistance of occlusal device materials is important. In the present study, PMMA (3 conventional autopolymerizing materials and 1 CAD-CAM occlusal device material) had the most consistent results for surface hardness, followed by the acrylic resin for additive manufacturing (Fig. 1). Despite the difference between the PMMA and the acrylic resin for additive manufacturing, the polyamide material (CAD-CAM) and the nonacrylic light-polymerizing resin for additive manufacturing showed the lowest surface hardness values.

Nguyen et al[13] tested 2 polyamide materials that were used as denture bases and reported statistically significantly lower values of surface hardness than PMMA. Hamanaka et al[14] and Ayaz et al[15] reported similar results. Although other authors[13-15] investigated denture base materials and not occlusal device materials, their results were comparable with those of the present study (Fig. 1). It is safe to conclude that polyamide materials used for occlusal devices have lower surface hardness than PMMA. To the authors' knowledge, the only comparable study of a light-polymerizing resin for additive manufacturing of occlusal devices was carried out by Huettig et al.[6] The authors examined the polishability and wear resistance of different occlusal device materials for oral appliances. A nonacrylic light-polymerizing resin for additive manufacturing showed lower wear values than a conventional acrylic resin, which seems contrary to the results of the present study (Fig. 1). Opposite results can most easily be explained by the different properties investigated (wear resistance compared with surface hardness). Although surface hardness describes resistance to wear[12] and several studies have found a correlation between hardness and wear resistance,[16-18] some authors disagree with this correlation.[14] For a more thorough comparison of surface hardness and intraoral wear resistance of different digitally processed occlusal device materials, further investigations are necessary, especially clinical studies.

In the study by Ayman,[19] conventional heat-polymerized PMMA had lower surface hardness values than CAD-CAM PMMA. In the present study, PMMA processed conventionally and with CAD-CAM had similar surface hardness values, whereas 2 light-polymerized resins (acrylic and nonacrylic) processed with additive manufacturing and 2 resins (polyamide and PMMA) processed with CAD-CAM differed significantly (Fig. 1). Between-material differences were mainly due to their chemical compositions rather than the technology, but differences due to different technologies are not excluded.[19]

As in the study by Ucar et al,[20] neither a specimen of a polyamide-based material nor one of a nonacrylic light-polymerizing resin for additive manufacturing fractured during flexural testing. Several studies have investigated polyamide materials for removable partial and complete denture bases,[13-15,20-23] although not for occlusal device fabrication. Three of these studies established that polyamide materials are more flexible than acrylic resins,[21-23] similar to the results of the present study (Fig. 2). Although flexibility is important for absorbing energy when a patient drops an appliance,[20] nonflexible occlusal devices are considered a better option for a patient with bruxism.[27-29] The clinical implications of the flexibility of occlusal device materials produced with digital technologies are not yet clear. For a better comparison with acrylic resin occlusal devices, clinical studies with newly developed materials having different mechanical properties and influence on the stomatognathic system are necessary.

Unlike the polyamide material and the nonacrylic light-polymerizing resin for additive manufacturing, all specimens of the different acrylic resin materials fractured. Two conventional occlusal device materials and one CAD-CAM PMMA material showed similar values for flexural strength (Fig. 2), contrary to the results of Alp et al.[30] Despite the statistically significant differences found among the tested materials (Fig. 2), all the tested materials met the ISO requirements for flexural strength $\geq$65 MPa.[24]

CONCLUSIONS

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

1. The mechanical properties of occlusal devices depend more on the material than on the particular technology.
2. Acrylic resin has the most consistent values of surface hardness regardless of the technology.
3. Polyamide resins and nonacrylic light-polymerizing resins for additive manufacturing have lower surface hardness, but their flexural strength is higher than that of acrylic resin.
4. No specimen of either polyamide or nonacrylic light-polymerizing resin for additive manufacturing fractured during flexural strength measurement.

REFERENCES

1. Alajbeg IZ, Gikic M, Valentic-Peruzovic M. Changes in pain intensity and oral health-related quality of life in patients with temporomandibular disorders during stabilization splint therapy – a pilot study. Acta Clin Croat 2014;53:7-16.
2. Quadri MF, Mahnashi A, Al Almutahhir A, Tubayqi H, Hakami A, Arishi M, et al. Association of awake bruxism with khat, coffee, tobacco, and stress among Jazan University Students. Int J Dent 2015;2015:842096.
3. Ferendiuk E, Zajdel K, Pihut M. Incidence of otolaryngological symptoms in patients with temporomandibular joint dysfunctions. Biomed Res Int 2014;2014:824684.
4. Kato T, Velly AM, Nakane T, Masuda Y, Maki S. Age is associated with self-reported sleep bruxism, independently of tooth loss. Sleep Breath 2012;16:1159-65.
5. Ohayon MM, Li KK, Guilleminault C. Risk factors for sleep bruxism in the general population. Chest 2001;119:53-61.
6. Huettig F, Kustermann A, Kuscu E, Geis-Gerstorfer J, Spintzyk S. Polishability and wear resistance of splint material for oral appliances produced with conventional, subtractive, and additive manufacturing. J Mech Behav Biomed Mater 2017;75:175-9.
7. Vayrynen VO, Tanner J, Vallittu PK. The anisotropicity of the flexural properties of an occlusal device material processed by stereolithography. J Prosthet Dent 2016;116:811-7.
8. Kim JE, Kwon JH, Kim JH, Shim JS. Recording the trajectory of mouth opening and closing for the fabrication of an occlusal splint. J Prosthet Dent 2017;117:597-600.
9. Alghazzawi TF. Advancements in CAD/CAM technology: options for practical implementation. J Prosthodont Res 2016;60:72-84.
10. Stansbury JW, Idacavage MJ. 3D printing with polymers: challenges among expanding options and opportunities. Dent Mater 2016;32:54-64.
11. Xu X, He L, Zhu B, Li J, Li J. Advances in polymeric materials for dental applications. Polymer Chemistry 2017;8:807-23.
12. Murakami N, Wakabayashi N, Matsushima R, Kishida A, Igarashi Y. Effect of high-pressure polymerization on mechanical properties of PMMA denture base resin. J Mech Behav Biomed Mater 2013;20:98-104.
13. Nguyen LG, Kopperud HM, Oilo M. Water sorption and solubility of polyamide denture base materials. Acta Biomater Odontol Scand 2017;3:47-52.
14. Hamanaka I, Iwamoto M, Lassila LVJ, Vallittu PK, Takahashi Y. Wear resistance of injection-molded thermoplastic denture base resins. Acta Biomater Odontol Scand 2016;2:31-7.
15. Ayaz EA, Bagis B, Turgut S. Effects of thermal cycling on surface roughness, hardness and flexural strength of polymethylmethacrylate and polyamide denture base resins. J Appl Biomater Funct Mater 2015;13:e280-6.
16. Suwannaroop P, Chaijareenont P, Koottathape N, Takahashi H, Arksornnukit M. In vitro wear resistance, hardness and elastic modulus of artificial denture teeth. Dent Mater J 2011;30:461-8.
17. Suzuki S. In vitro wear of nano-composite denture teeth. J Prosthodont 2004;13:238-43.
18. Zeng J, Sato Y, Ohkubo C, Hosoi T. In vitro wear resistance of three types of composite resin denture teeth. J Prosthet Dent 2005;94:453-7.
19. Ayman AD. The residual monomer content and mechanical properties of CAD\CAM resins used in the fabrication of complete dentures as compared to heat cured resins. Electron Physician 2017;9:4766-72.
20. Ucar Y, Akova T, Aysan I. Mechanical properties of polyamide versus different PMMA denture base materials. J Prosthodont 2012;21:173-6.
21. Hamanaka I, Takahashi Y, Shimizu H. Mechanical properties of injection-molded thermoplastic denture base resins. Acta Odontol Scand 2011;69:75-9.
22. Takahashi Y, Hamanaka I, Shimizu H. Effect of thermal shock on mechanical properties of injection-molded thermoplastic denture base resins. Acta Odontol Scand 2012;70:297-302.
23. Sasaki H, Hamanaka I, Takahashi Y, Kawaguchi T. Effect of reinforcement on the flexural properties of injection-molded thermoplastic denture base resins. J Prosthodont 2017;26:302-8.
24. International Organization for Standardization. ISO 20795-1:2013. Dentistry – base polymers. Part 1: denture base polymers. Geneva: ISO; 2015. Available at: https://www.iso.org/standard/62277.html.
25. International Organization for Standardization. ISO 2039-1:2001. Plastics – determination of hardness. Part 1: ball indentation method. Geneva: ISO; 2001. Available at: https://www.iso.org/standard/31264.html.
26. Nishigawa K, Bando E, Nakano M. Quantitative study of bite force during sleep associated bruxism. J Oral Rehabil 2001;28:485-91.
27. Cruz-Reyes RA, Martinez-Aragon I, Guerrero-Arias RE, Garcia-Zura DA, Gonzalez-Sanchez LE. Influence of occlusal stabilization splints and soft occlusal splints on the electromyographic pattern, in basal state and at the end of six weeks treatment in patients with bruxism. Acta Odontol Latinoam 2011;24:66-74.
28. Amin A, Meshramkar R, Lekha K. Comparative evaluation of clinical performance of different kind of occlusal splint in management of myofascial pain. J Indian Prosthodont Soc 2016;16:176-81.
29. al-Quran FA, Lyons MF. The immediate effect of hard and soft splints on the EMG activity of the masseter and temporalis muscles. J Oral Rehabil 1999;26:559-63.
30. Alp G, Murat S, Yilmaz B. Comparison of flexural strength of different CAD/CAM PMMA-based polymers. J Prosthodont 2019;28:e491-5.

Corresponding author:
Dr Samir Cimic
Department of Prosthodontics
School of Dental Medicine
University of Zagreb
Gundulićeva 5, 10000, Zagreb
CROATIA
Email: scimic@sfzg.hr

https://doi.org/10.1016/j.prosdent.2018.09.022