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Fracture resistance of additively or subtractively manufactured resin-based definitive crowns: Effect of restorative material, resin cement, and cyclic loading

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ABSTRACT

Objective: To evaluate how restorative material, resin cement, and cyclic loading affect the fracture resistance of resin-based crowns fabricated by using additive or subtractive manufacturing.
Methods: A right first molar crown standard tessellation language (STL) file was used to fabricate 120 crowns from one subtractively manufactured polymer-infiltrated ceramic network (SM) and two additively manufactured resin composites (AM-B and AM-S) ($N = 40$). These crowns were randomly divided into 4 groups within each material according to the dual-polymerizing resin cement to be used (RX and PN) and the aging condition ($n = 10$). After cementation, the crowns without cyclic loading were subjected to fracture testing, while the others were first cyclically loaded (1.7 Hz, 1.2 million cycles, and 49-N load) and then subjected to fracture testing. Data were analyzed with generalized linear model analysis ($\alpha = .05$).
Results: Fracture resistance of the crowns was affected by material, resin cement, and cyclic loading ($P \leq .030$). However, none of the interactions significantly affected fracture resistance of tested crowns ($P \geq .140$). Among tested materials, SM had the highest fracture resistance, whereas AM-B had the lowest ($P \leq .025$). RX led to higher fracture resistance, and cyclic loading decreased the fracture resistance ($P \leq .026$).
Significance: Tested materials can be considered reliable in terms of fracture resistance in short- or mid-term (5 years of intraoral simulation) when used for single molar crowns with 2 mm occlusal thickness. In the long term, polymer-infiltrated ceramic network crowns cemented with RelyX Universal may provide promising results and be less prone to complications considering higher fracture resistance values obtained.

1. Introduction

Additive manufacturing has become a feasible alternative to subtractive manufacturing, considering the ability to fabricate complex geometries with less material waste [1]. In addition, the advancements in computer-aided design and computer-aided manufacturing (CAD-CAM) technologies have diversified the materials that can be additively or subtractively manufactured [2–4] and be used monolithically [5]. Resin-based materials can be fabricated by both methods [6,7] and some of these resins are indicated for definitive fixed tooth- or implant-supported prostheses [2,8,9]. Additively manufactured resins are becoming increasingly popular as previous studies have reported results comparable to those of subtractively manufactured resins with similar chemical compositions [1,2,4]. Another advancement in CAD-CAM manufactured restorative materials is the introduction of hybrid materials for subtractive manufacturing [10,11]. Polymer-infiltrated ceramic network (Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany) is one such hybrid material and contains 86 wt% ceramic and 14 wt% acrylate polymer network [12–15]. This material is also indicated for both tooth- and implant-supported prostheses [16] and combines the advantages of resin composites and ceramics [17–19].

Fracture resistance, esthetic properties, and marginal adaptation of a restoration are essential for clinical success [14]. However, fracture is the main complication that is encountered with indirect restorations and

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it is related to different factors including the type of material and resin cement [20]. Resin cements are widely preferred for their mechanical properties [21] and can be categorized as light-polymerized, auto-polymerized, and dual-polymerized according to their polymerization method [22]. However, previous studies on different restorative materials recommended using dual-polymerizing resin cements for thick restorations to overcome the polymerization-related issues [22,23] as they comprise both chemical- and photo-initiators [24].

While the fracture resistance of additively manufactured resins has been investigated in previous studies [2,4,6,8,14,25–28], the effect of resin cement was not investigated in any of those studies. A recent study has reported higher shear bond strength for additively manufactured resins when cemented by using a dual-polymerizing self-adhesive resin cement (RelyX Universal, 3 M, St Paul, MN, USA) or a dual-polymerizing adhesive resin cement (Panavia V5, Kuraray Noritake, Tokyo, Japan) [29]. Studies on the fracture resistance of additively manufactured resins when cemented by using different resin cements could broaden the understanding of clinicians on the limitations and the applicability of these materials for definitive prostheses. In addition, knowledge on how aging affects the fracture resistance of additively manufactured resins cemented with different resin cements would elucidate their long-term mechanical properties and potential complications that might be encountered. Therefore, the present study aimed to evaluate the effect of restorative material, resin cement, and aging on the fracture resistance of two additively manufactured resins and a subtractively manufactured polymer-infiltrated ceramic network. The null hypothesis was that material type, resin cement type, and aging would not affect the fracture resistance of tested materials.

2. Materials and methods

2.1. Specimen preparation

Table 1 lists detailed information on the materials tested in the present study. A tactile scanner (Procera Piccolo, Nobel Biocare AB, Gothenburg, Sweden) was used to digitize a prepared typodont mandibular right first molar tooth with a 1-mm-wide chamfer finish line [30]. A mandibular right first molar crown with a 30-µm cement gap [1,30], 1.5-mm minimum axial and 2-mm minimum occlusal thickness was designed (Zirkonzahn.Modellier, Zirkonzahn GmbH, Gais, Italy) by using the standard tessellation language (STL) file of the prepared tooth. The master STL file was used to fabricate 120 crowns by using one subtractively manufactured polymer-infiltrated ceramic network (Vita Enamic [SM]; Vita Zahnfabrik) and two additively manufactured resin composites (VarseoSmile Crown Plus [AM-B], Bego and Crowntec [AM-S], Saremco) (N = 40).

To fabricate SM crowns, the master STL was imported into a nesting software program (PrograMill CAM V4.2; Ivoclar AG) and a 5-axis milling unit (PrograMill PM7; Ivoclar AG) was used for the subtractive manufacturing. After fabrication, the support structures were removed with a cut-off wheel (Keystone Cut-off Wheels; Keystone Industries). To fabricate AM-B and AM-S crowns, the master STL was imported into a nesting software program (Composer v1.3, ASIGA, Sydney, Australia) and positioned with its occlusal surface facing the build platform. Support structures were generated automatically and those at the critical regions such as margins or the intaglio surface of the crown were removed manually. This design was duplicated for standardization, and a digital light processing-based (DLP-based) 3-dimensional (3D) printer (MAX UV, ASIGA, Sydney, Australia) was used to additively manufacture both AM-B and AM-S crowns. After fabrication, AM-B crowns were cleaned in an ultrasonic bath containing reusable ethanol solution (95 % Ethanol Absolut, Grogg Chemie AG, Stettlen, Switzerland) followed by thorough cleaning in an ultrasonic bath containing fresh ethanol (95 % Ethanol Absolut, Grogg Chemie AG, Stettlen, Switzerland). After drying the crowns with an air syringe, support structures were removed by using the same cut-off wheel and the crowns were sandblasted with 50 µm glass beads (Rolloblast, Renfert, Hilzingen, Germany) at 1.5 bar to remove the whitish layer that appeared after cleaning. The AM-B crowns were then polymerized (2 ×1500 exposures) in a xenon polymerization device (Otoflash G171, NK Optik, Baierbrunn, Germany) under nitrogen oxide gas atmosphere [4,9]. AM-S crowns were cleaned with an alcohol-soaked (95 % Ethanol Absolut, Grogg Chemie AG, Stettlen, Switzerland) cloth to remove residual resin, dried with an air syringe, and post-polymerized by using the same xenon polymerization device under nitrogen oxide gas atmosphere (2 × 2000 exposures) [1,2,31]. After post-polymerization, external surfaces of AM-S crowns were sandblasted with 50 µm glass beads (Rolloblast, Renfert, Hilzingen, Germany) at 1.5 bar and the support structures were then removed with the same cut-off wheel [29]. The master STL file of the prepared molar tooth was also used to fabricate 120 fiberglass-reinforced epoxy resin abutments (G10, McMaster-Carr, Atlanta, GA, USA) [5] with the same 5-axis milling unit.

Table 1
List of materials tested in this study.

Material	Chemical composition
Vita Enamic (SM) (Polymer-infiltrated ceramic)	14 wt% methacrylate polymer (urethane dimethacrylate and triethylene glycol dimethacrylate) and 86 wt% fine-structure feldspar ceramic network
VarseoSmile Crown Plus (AM-B) (Additively manufactured hybrid resin composite)	Esterification products of 4.4′-isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid, silanized dental glass, methyl benzoylformate, diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide. Total content of inorganic fillers: 30–50 wt %
Crowntec (AM-S) (Additively manufactured resin composite)	Esterification products of 4,4′-isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid, silanized dental glass, pyrogenic silica, initiators. Total content of inorganic fillers: 30–50 wt%
RelyX Universal (RX) (Dual-polymerizing self-adhesive resin cement)	Bisphenol A derivative free dimethacrylate monomers, phosphorylated dimethacrylate adhesion monomers, photoinitiators, amphiphilic redox initiators, radiopaque fillers, rheological additives, pigments
Panavia V5 (PN) (Dual-polymerizing adhesive resin cement)	Bisphenol A glycidyl methacrylate, triethylene glycol dimethacrylate, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica, silanated aluminum oxide filler, dl-camphorquinone, pigments
Scotchbond Universal Plus Adhesive (Universal adhesive)	10-methacryloyloxydecyl dihydrogen phosphate, 2-hydroxyethyl methacrylate, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane
Panavia V5 Tooth Primer (Self-etching primer)	10-methacryloyloxydecyl dihydrogen phosphate, 2-hydroxyethyl methacrylate, hydrophilic aliphatic dimethacrylate, accelerators, water
Clearfil Ceramic Primer Plus (Universal prosthetic primer)	3-methacryloxypropyl trimethoxysilane, 10-methacryloyloxydecyl dihydrogen phosphate, ethanol

2.2. Cementation, cyclic loading, and fracture resistance test

A software program (Excel, Microsoft Corp, Seattle, WA, USA) was used to randomly divide the crowns in each material group into four subgroups according to the resin cement to be used for cementation (RelyX Universal [RX], 3 M, Seefeld, Germany and Panavia V5 [PN], Kuraray Noritake, Tokyo, Japan) and aging condition (control and cyclically loaded) (n = 10). Intaglio surfaces of SM crowns were etched by using hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar AG, Schaan, Liechtenstein) for 60 s, steam-cleaned (Wasi-Steam Classic II, Wassermann Dental-maschinen GmbH, Hamburg, Germany) from a distance of 3 cm for 10 s, and air-dried with oil-free air. Then, a ceramic primer (Clearfil Ceramic Primer Plus, Kuraray Noritake, Tokyo, Japan) was applied by using a microbrush and was gently air-dried for 5 s. Intaglio surfaces of AM-B and AM-S crowns were sandblasted with 50 µm aluminum oxide (Cobra, Renfert, Hilzingen, Germany) from a distance of 10 mm for 10 s with 2-bar pressure, steam-cleaned from a distance of 10 cm for 5 s, and air-dried with oil-free air. Intaglio surfaces of AM-B and AM-S crowns to be cemented with PN were then treated with the same ceramic primer in the same fashion as the SM crowns, while AM-B and AM-S crowns to be cemented with RX received no further treatment. Epoxy resin dies were initially sandblasted with 110 µm Al_2O_3 (Cobra, Renfert, Hilzingen, Germany) from a distance of 10 mm for 10 s with 2-bar pressure, steam-cleaned from a distance of 10 cm for 5 s, and dried with oil-free air. Then, the dies were divided into 12 groups according to material type, resin cement, and aging condition. The dies to be cemented with RX were treated with an adhesive (Scotchbond Universal Plus Adhesive, 3 M, Seefeld, Germany) by using microbrushes, left to react for 20 s, and gently air-dried for 5 s, while those to be cemented with PN were initially treated with the same ceramic primer, which was air-dried for 5 s, and then treated with an adhesive (Panavia V5 Tooth Primer, Kuraray Noritake, Tokyo, Japan) by using microbrushes, left to react for 20 s, and gently air-dried for 5 s [29].

The cementation procedure was the same for all crowns, and refillable syringes of the respective manufacturers were used to apply resin cements. After resin cement application, the crown was seated on its respective die and placed under a brass holder that applies a constant load of 2 N [32]. A light emitting diode polymerization unit (Bluephase, Ivoclar AG, Schaan, Liechtenstein) was used to light-polymerize the resin cement for 3 s from each of 4 sides of the crown before removing the excess with a microbrush. The crown was then light-polymerized for 40 s (10 s from each of the 4 sides) and left under the brass holder until 10 min had passed after mixing the resin cement. After cementation (Fig. 1), all crowns were stored in tap water at 37 °C for 24 h before either being subjected to cyclic loading or fracture resistance test. A single operator (G.Ç.) performed all pretreatment and cementation procedures and the light intensity of the polymerization unit was verified periodically with a radiometer (Demetron LED Radiometer, Kerr, Middleton, WI, USA) to be at least 950 mW/cm^2 [29].

The crowns to be immediately subjected to fracture resistance testing were mounted in a universal testing machine (Instron 5942, Instron Corp, Norwood, MA, USA). The force was applied with a Ø12-mm stainless steel sphere at a crosshead speed of 1 mm/min and the maximum fracture strength values were recorded. The other half of the crowns were initially subjected to cyclic loading at 1.7 Hz for 1.2 million cycles under 49-N load, to simulate 5 years of functional loading [26, 27], by using an in-house built chewing simulator [33]. During cyclic loading, the crowns were immersed in distilled water [5] and the force was applied by using Ø12-mm polyoxymethylene spheres [34] (Delrin, Dupont, Wilmington, DE, USA) that were secured on the crowns with the metal jigs of the chewing simulator (Fig. 2). Polyoxymethylene spheres were deliberately used to evenly distribute the occlusal loads and to prevent direct contact related fractures. After cyclic loading, each crown was evaluated for any failure and given that none of the crowns failed, all were subjected to fracture testing as described above.

Fig. 1. Representative images of one crown from each material subgroup after cementation. AM-B; VarseoSmile Crown Plus. AM-S; Crowntec. SM; Vita Enamic.

2.3. *Statistical analysis*

Normality of data was analyzed by using Kolmogorov-Smirnov test. Due to normal distribution, generalized linear model analysis was used to evaluate the effect of material, resin cement, and cyclic loading on fracture resistance, and of every possible interaction. All statistical analyses were performed by using a software (Minitab Software V.17, Minitab Inc, State College, PA, USA) at a significance level of $\alpha = .05$.

3. Results

The fracture resistance results are shown in Table 2, while a representative crown from each material after fracture testing is presented in Fig. 3. The generalized linear model analysis revealed a significant effect of material ($P < .001$), resin cement ($P = .030$), and cyclic loading ($P = .010$) on the fracture resistance of the tested crowns. However, neither the interactions between any two main factors ($P \geq .140$) nor the interaction among all main factors ($P = .630$) were statistically significant.

SM had the highest ($P < .001$) and AM-B ($P \leq .025$) had the lowest fracture resistance. When resin cements were considered, RX resulted in higher fracture resistance than PN ($P = .026$). Cyclic loading significantly reduced the fracture resistance ($P = .006$).

4. Discussion

The present study found that type of material, resin cement, and aging condition all affected the fracture resistance of additively and subtractively manufactured tooth-borne molar crowns. Therefore, the null hypothesis was rejected.

Similar to the results of previous studies on the fracture resistance of

Fig. 2. Cyclic loading setup used in this study.

Table 2
Mean ± standard deviation fracture resistance (N) of each material-resin cement combinations according to aging condition.

Resin cement	Aging condition	Materials			Total
		SM	AM-B	AM-S	
RX	Control	5593 ± 528	3947 ± 482	4179 ± 358	4399 ± 921^{a}
	Cyclic loaded	5231 ± 901	3548 ± 464	3895 ± 418	
PN	Control	5151 ± 577	3695 ± 339	3983 ± 268	4177 ± 747^{b}
	Cyclic loaded	4675 ± 893	3609 ± 349	3953 ± 424	
Total	Control	5372 ± 584	3821 ± 426	4081 ± 324	4425 ± 818*
	Cyclic loaded	4953 ± 918	3578 ± 401	3924 ± 411	4152 ± 851$^{\wedge}$
	Total	5162 ± 789^{A}	3700 ± 426^{C}	4003 ± 374^{B}	

Different superscript uppercase letters indicate significant differences among materials, while different superscript lowercase letters indicate significant differences between resin cements. Different superscript symbols indicate significant differences between aging conditions. Total values are derived from pooled data ($P < .05$).

additively and subtractively manufactured resin-based crowns [4,6,14], SM had the highest fracture resistance, regardless of the resin cement and aging condition used. This favorable result may be related to the chemical composition of SM as well as the fabrication method. AM-B and AM-S have similar chemical compositions, which comprise a silanized glass matrix of up to 50 wt%. In contrast, SM has a ceramic content of 86 wt%. Even though lower inorganic content results in a more flowable material, necessary for adequate fabrication of additively manufactured resins [7], this might explain the lower fracture resistance of AM-B and AM-S. AM-B and AM-S crowns were fabricated in line with manufacturers' recommendations; however, SM crowns were milled from prefabricated CAD-CAM blocks, which are fabricated under standardized pressure and temperature conditions [4]. These standardized conditions may have also attributed to the fracture resistance of SM with increased degree of conversion and less residual monomers [6]. SM was deliberately chosen as the control group of the present study, given that it has already been used in previous studies on the mechanical properties of additively manufactured crowns [4,7,27] and has generally been well-studied [35]. Nevertheless, SM is not the only available subtractively manufactured material with resin and ceramic composition [18], and the results of this study should be substantiated with different subtractively manufactured materials.

Previous studies that reported results similar to those of the present study have also used SM, AM-B, and AM-S [4,6,14]. Suksuphan et al. [14] compared AM-B with SM and a resin nanoceramic in varying occlusal thicknesses (0.8 mm, 1 mm, and 1.5 mm). The authors [14] concluded that regardless of the thickness, SM crowns resisted loads higher than 2000 N, and that AM-B had lower fracture resistance than SM as well as the tested resin nanoceramic. In another study, screw-retained implant-supported crowns in SM, AM-B, AM-S, and a reinforced resin composite were investigated for their fracture resistance after thermomechanical aging [4]. SM was found to have the highest fracture resistance, while AM-S had similar fracture resistance to that of the reinforced resin composite [4]. Rosentritt et al. [6] showed that AM-B had lower fracture resistance than a resin composite and a nanoceramic hybrid material. However, the mean fracture resistance values in those previous studies ranged between 536 N and 2601 N [4,6,14]. These values are considerably lower than the lowest mean fracture resistance value measured in the present study (3548 N). The design of tested crowns may be one reason for this difference as the study of Diken Türksayar et al. [4] was conducted on implant-supported premolar crowns with a screw access channel. Possible wedging effect of titanium base abutments and their elastic modulus may also have led to fracture resistance values lower than those recorded in the present study. The occlusal thickness of the previously tested crowns ranged from 0.8 mm to 1.5 mm [4,6,14], which may also have contributed to the lower fracture resistance values found in those studies. In fact, one of the studies found increased occlusal thickness to be associated with increased fracture resistance [14].

The effect of resin cement on the fracture resistance of resin-based materials has been shown previously [36], and in the present study, the crowns cemented with RX had higher fracture resistance. However, it should be emphasized that the maximum mean difference between RX and PN for AM-B and AM-S crowns was approximately 252 N, whereas this difference was approximately 556 N for SM crowns. Given that 252 N is quite low considering the mean fracture resistance values measured in the present study, the authors believe that the statistically higher fracture resistance values of crowns cemented with RX were caused by the fracture resistance data of SM crowns, and the difference between RX and PN may be clinically negligible for AM-B and AM-S crowns. It should also be mentioned that even though RX is a dual-polymerizing self-adhesive resin cement, it can also be used as an adhesive resin cement when combined with the adhesive (Scotchbond Universal Plus Adhesive) as in the present study. In either situation, the components, and in the situation of adhesive resin cement, the steps required for cementation with RX are fewer than those required with PN, which may be another advantage along with higher fracture resistance. In addition, a recent study reported that RX led to shear bond strength values that were either similar to or higher than PN when the restorative materials tested in the present study were used [29]. However,

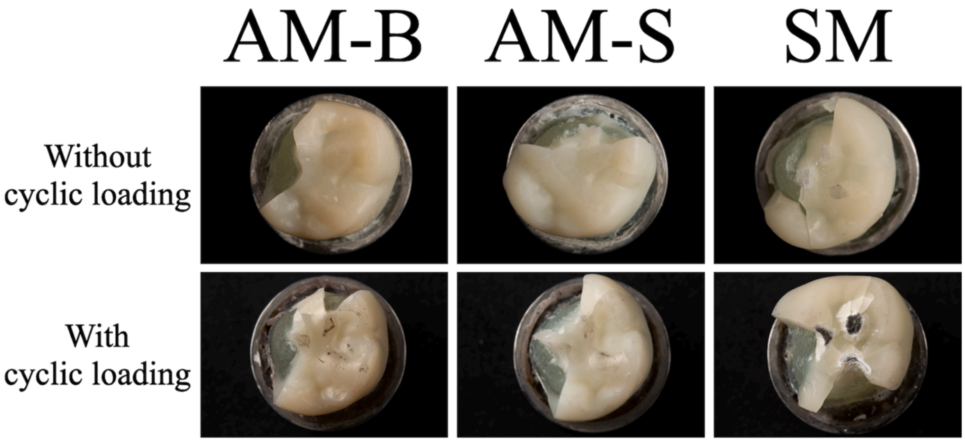

Fig. 3. Representative crowns from each material subgroup after fracture testing. AM-B; VarseoSmile Crown Plus. AM-S; Crowntec. SM; Vita Enamic.

achieving the best possible result from adhesive cementation depends on the composition of the restorative material, its thickness, and the resin cement [20]. Therefore, the higher fracture resistance obtained with RX in the present study should not be generalized, and general superiority of one type of resin cement over the other has not yet been established [21]. Moreover, a recent study has shown that the type of resin cement did not affect the biaxial flexural strength and compressive load of SM [10]; thus, the results of the present study should be substantiated with studies investigating other mechanical properties of restorative materials and resin cements.

Regardless of the restorative material and resin cement, cyclic loading decreased the fracture resistance. The cyclic loading methodology used in the present study simulates approximately 5 years intraorally and has been used in previous studies on the fracture resistance of additively and subtractively manufactured resin-based restorations [4,8, 25–27]. The effect of mechanical aging on the fracture resistance of the tested materials has not been widely studied as only 3 studies focused on the fracture resistance of SM after thermomechanical aging [11,15,19]. While 2 of those studies [11,15] concluded that thermomechanical aging did not affect the fracture resistance of SM, the third reported similar results as those of the present study [19]. However, a direct comparison between the present and those previous studies [11,15,19] might be misleading, given the differences in aging protocols, design of the crowns, abutment materials, and resin cements used. To the authors' knowledge, the present study was the first on the effect of cyclic loading on the fracture resistance of AM-B and AM-S; thus, comparisons with previous studies were not possible. However, a recent study on the fracture resistance of additively manufactured zirconia also found that cyclic loading significantly decreased the fracture resistance [37]. Regardless of aging or the cement used, the fracture resistance values measured in the present study are much higher than the maximum masticatory forces of approximately 800 N generated by patients with bruxism [38]. Therefore, all 3 tested materials and 2 cements may be suitable for medium- to long-term rehabilitation of molar teeth in need of crowns with a minimum occlusal thickness of 2 mm. However, these hypotheses should be supported by in vivo studies with long-term follow-ups.

To the authors' knowledge, the present study was the first on the combined effect of restorative material, resin cement, and cyclic loading on the fracture resistance of additively manufactured definitive resin composites. Therefore, a priori power analysis could not be performed. However, post-hoc power analyses showed that the sample size was adequate for a minimum of 65% power with a minimum effect size of 0.21 and $\alpha = .05$. Another limitation of the present study was that only 2 additively manufactured and one subtractively manufactured resin-based materials, and 2 dual-polymerizing resin cements were tested. In addition, no glazing or polishing was performed to evaluate the inherent properties of tested restorative materials. Cyclic loading did not involve saliva or temperature changes that are frequent intraorally, and human enamel was not used as an antagonist during cyclic loading. Nevertheless, the occlusal morphology of the crowns was designed to have the polyoxymethylene spheres contact all cusp slopes to generate shear forces on these cusps during the cyclic loading and also when loading to fracture as the steel spheres had the same shape and diameter. The abutment dies were fabricated from a fiberglass-reinforced epoxy resin that has similar elastic modulus to that of dentin [5] for standardization. However, considering that the abutment material may affect the fracture resistance of restorative materials, the results of this study should be substantiated with future studies that involve human dentin. All crowns were fabricated by using a standardized crown design with adequate occlusal thickness. Nevertheless, the effect of material thickness on the fracture resistance of additively manufactured resins [8, 14,27] and polymer-infiltrated ceramic network [3] has been shown. Future studies should investigate how different prosthetic designs with increased number of units or coverage areas affect the fracture resistance and other mechanical or optical properties of the tested materials cemented by using different resin cements and aged with a setup that involves thermal fluctuations to broaden the knowledge on the limitations of these materials, particularly those manufactured additively.

5. Conclusion

Within the limitations of the present study, the following conclusions were drawn:

1. Crowns fabricated from subtractively manufactured polymer-infiltrated ceramic network had the highest and crowns fabricated from additively manufactured hybrid resin had the lowest fracture resistance.
2. Regardless of the restorative material, the dual-polymerizing self-adhesive resin cement led to higher fracture resistance of the crowns.
3. Cyclic loading significantly decreased the fracture resistance of the crowns. However, all crowns had fracture resistance values higher than reported masticatory forces of the molar region.

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