THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Effect of supporting substrate on the failure behavior of a polymer-infiltrated ceramic network material

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ABSTRACT

Statement of problem. Restorative materials are cemented on different types of substrates, such as dentin, metal, and glass-fiber posts with composite resin cores.

Purpose. The purpose of this in vitro study was to evaluate the failure behavior after cycling fatigue of a polymer-infiltrated ceramic network material (PICN; VITA ENAMIC) cemented on different supporting substrates.

Material and methods. PICN plates (N=80) were obtained from computer-assisted design and computer-assisted manufacturing (CAD-CAM) blocks and cemented with a resin cement to 4 different supporting substrates (n=20): (1) human dentin (PICNDen); (2) dentin analog (PICNDenAn); (3) nickel-chromium alloy (PICNNiCr); and (4) composite resin plus fiberglass post (PICNRc). For comparison, the fracture behavior of a feldspathic ceramic (FelDenAn; VITABLOCS Mark II) and an indirect composite resin (ResDenAn; Opallis LAB Resin) cemented to the DenAn substrate was investigated (n=20). Thus, specimens were composed of the restorative material layer (1-mm thick) resin cemented (0.1-mm-thick layer) to a 2-mm-thick supporting substrate. All specimens were subjected to mechanical cycling (MC) using a pneumatic cycling machine (500 000 cycles, 2 Hz, 50 N). Specimens that did not fracture during cycling were tested under compression using a universal testing machine at a cross-head speed of 0.5 mm/min until the sound of the first crack was detected using an acoustic system. Failure data were statistically evaluated using Weibull distribution. Failures were classified as radial crack, cone crack, combined, and catastrophic fracture.

Results. All FelDenAn specimens were fractured during MC. Only 4 PICNRc specimens survived MC, so their fracture load data were not statistically analyzed. PICNNiCr showed the greatest characteristic load (L_0) value, followed by ResDenAn. Groups PICNDenAn and PICNDen showed lower and similar L_0 but statistically different Weibull modulus (*m*). There was a significant relationship between experimental group and failure mode ($P<.001$). FelDenAn and PICNRc had a higher frequency of radial cracks, whereas PICNNiCr failed from cone cracking.

Conclusions. The supporting substrate influenced the failure behavior of PICN. When the substrate had a higher elastic modulus than the restorative material, better mechanical behavior was observed. (J Prosthet Dent 2019;121:929-34)

Composite resins and ceramics are the most popular esthetic restorative materials. Despite the development of dental composite resins, their clinical performance is still inferior to that of ceramic restorations.[1-4] Ceramic restorations have better color stability and wear resistance than composite resin single-tooth restorations.[3] However, the brittle behavior and susceptibility to slow crack growth of ceramics may limit their clinical use.[5,6] Therefore, intending to optimize the clinical performance of indirect dental restorations, ceramic and composite resins have been combined into a restorative material.[7] This hybrid material named 'polymer-infiltrated ceramic-network' (PICN) has 2 interpenetrating phases, a sintered ceramic network (86 wt%) infiltrated by a resin matrix (14 wt%).[8-10]

Restorative materials are cemented on different types of

This work was supported by CAPES do Brasil; and CNPq and FAPERGS (Fundação de Amparo à Pesquisa do Estado do Rio Grande do Sul) for the ARD financial support (grant # 17/2551-0000843-0). 3M ESPE for provided the resin cement and adhesive systems.
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Clinical Implications

Polymer-infiltrated ceramic network material performed better when cemented on high elastic modulus supporting substrates. Polymer-content materials bonded to a dentin analog substrate are more resistant to cyclic loading than feldspathic ceramic.

substrates, including dentin, metal, and glass-fiber posts with composite resin cores, which could affect the mechanical behavior of the restoration. The fracture strength of a restoration is expected to increase as the elastic modulus of the substrate increases.[11-16] Glass-fiber posts are more esthetic and have a lower elastic modulus (E=17 to 40 GPa) than metal (E=89 to 205 GPa).[17-19] These commonly used substrates have been compared in a systematic review, which reported that both metal and glass-fiber posts are indicated to restore teeth with remaining coronal walls, and posts with high elastic moduli are more suitable for teeth with extensive destruction and without a ferrule.[20] Moreover, metal, such as titanium, is used as an abutment for implant-supported restorations.

Therefore, different supporting substrates should be used in laboratory simulations to predict the mechanical behavior of dental restorations more reliably. Laboratory simulations of clinical failure mechanisms and stress distribution for single crowns suggested the use of a fiber-reinforced epoxy resin material (NEMA G10) as a dentin analog.[14,21,22] As human teeth are difficult to standardize, the use of a dentin analog is indicated. Thus, the purpose of this in vitro study was to evaluate the failure behavior of a PICN material (VITA ENAMIC) cemented on different supporting substrates to test the hypotheses that the supporting substrate influences the failure behavior of PICN and that the restorative material also influences the failure behavior of the structure (restoration/cement/supporting substrate).

MATERIAL AND METHODS

The study was evaluated and approved by the local Human Research Ethics Committee and registered in the National Research Platform (no.: 49860715.9.0000.5342). Materials used in this study are presented in Table 1. Plate-shaped specimens (1 mm in thickness) of a restorative material were resin cemented (0.1 mm in thickness) to a supporting substrate (12 mm in diameter and 2 mm in thickness). Three restorative materials were investigated: a PICN material, a feldspathic porcelain (Fel), and a laboratory composite resin (Res). Four substrate materials were used: human dentin (Den), fiber-reinforced epoxy resin (DenAn), fiberglass post plus composite resin (Rc), and NiCr alloy (NiCr) (Table 1). Specimens of PICN were cemented onto all substrates (PICNDen, PICNDenAn, PICNRc, and PICNNiCr) (n=20). Fel and Res specimens were only cemented onto the dentin analog (FelDenAn and ResDenAn) (n=20).

Plates from PICN and Fel were obtained from CAD-CAM blocks using a diamond disk in a metallographic cutting machine (Minitom; Struers) under water cooling. Plates from Res were obtained by inserting the restorative material into a silicone mold (Zetalabor; Zhermack) with an incremental technique. Each 2-mm increment was light polymerized for 120 seconds (according to the manufacturer's instructions) using a light-emitting diode light-polymerization unit (1000 mW/cm^2, Radii-cal; SDI Victoria). The specimens were polished to the final thickness (1 mm) using a polishing machine (DP-10; Panambra-Struers) and SiC abrasive papers (#400, 600, 800, 1200 grit) with constant water irrigation.

NiCr substrate was produced using chemically activated acrylic resin (DuraLay; Reliance Dental Manufacturing) inserted into a metal mold (2 mm in thickness and 12 mm in diameter) and used as a matrix. Before casting, acrylic resin plates were polished to the final thickness (2 mm). The bonding surface of the NiCr specimens was abraded with ≤45 μm aluminum oxide particles (Polidental) at 0.25 MPa for 20 seconds.

Dentin substrate (Den) was obtained from 20 human third molar teeth, which had been stored in distilled water. Only the middle third of the crown was used. Each tooth was sectioned twice through the transversal axis using a diamond disk in a metallographic cutting machine under water cooling. The first cut (enamel removal) was located 2 mm below the cusp tips, and the second cut was located 2 mm above the cement-enamel junction (root removal). The coronal portion of the pulp chamber was cleaned and filled with composite resin (Filtek Z250 XT; 3M ESPE).

The Rc substrate was fabricated using fiberglass posts (Fib) and composite resin (Mic). The posts were cleaned with 37% phosphoric acid for 30 seconds, washed in water, and air-dried. A silane coupling agent (Prosil; FGM Dental Products) was applied for 60 seconds. Posts were placed at the center of a silicone mold (12 mm in diameter and 18 mm in thickness), and the mold was filled with composite resin (Mic) and light activated for 20 seconds (Radii-cal). The final Fib-Mic cylinder was cut into 2.2-mm-thick sections using a cutting machine.

DenAn blocks were obtained by cutting a NEMA G10 cylinder using a diamond disk in a metallographic cutting machine under water cooling.

Den, Rc, and DenAn substrates were polished to the final thickness (2 mm) using SiC abrasive papers (#400, 600, and 800 grit) to obtain flat and parallel upper and lower surfaces. Before cementation, the bonding surface

Table 1. Brand name, description, and manufacturer information of materials used

Legend	Material	Description	Manufacturer
PICN	VITA ENAMIC	Polymer-infiltrated ceramic network	VITA Zahnfabrik
Fel	VITABLOCS Mark II	Feldspathic porcelain	VITA Zahnfabrik
Res	Opallis LAB	Laboratory composite resin	FGM Dental Products
DenAn	NEMA G10	Fiber-reinforced epoxy resin—dentin analog	International paper
Fib*	Fiberpost	Fiberglass post	FGM Dental Products
Mic*	Filtek Z250 XT	Micro-hybrid dental restorative composite resin	3M ESPE
NiCr	Fit Cast	N-iCr alloy	Talmax

*Association of Fib and Mic resulted in Rc substrate.

of Den and Rc substrates were etched with 37% phosphoric acid (Condac 37%; FGM Dental Products) for 15 seconds (Den) and 20 seconds (Rc). The surfaces were rinsed with water for 10 seconds and gently air-dried. For DenAn, 10% hydrofluoric acid (Condac porcelana 10%; FGM Dental Products) was applied to the bonding surface for 60 seconds, rinsed with water for 10 seconds, and air-dried. After surface treatment, for Den, Rc, and DenAn substrates, an adhesive (Single Bond Universal; 3M Dental Care) was applied for 20 seconds and light activated for 20 seconds (Radii-cal).

The bonding surface of the PICN, Fel, and Res specimens were etched with 5% hydrofluoric acid (Condac porcelana 5%; FGM Dental Products) for 60 seconds, rinsed with water for 10 seconds, and air-dried. A silane (Prosil; FGM Dental Products) was applied for 60 seconds and air-dried, and an adhesive (Single Bond Universal) was applied and light polymerized as described previously.

After the surface treatments, a dual-polymerizing adhesive resin cement (RelyX Ultimate; 3M Dental Care) was applied on the substrate treated surface, and the restorative material was placed at the center of the substrate. A special device was used to apply a uniform load of 7.4 N for 5 minutes, and excess cement was removed using a microbrush (FGM Dental Products). Resin cement was light activated (1000 mW/cm^2) on 4 sides for 40 seconds per side.

After 24 hours of storage in 37 °C distilled water, all specimens were subjected to mechanical cycling using a pneumatic machine (Biocycle; Biopdi) for 500 000 cycles (2 Hz, 50 N), with 37 °C distilled water. A G10 piston (DenAn) with a flat tip (3 mm in diameter) was used to apply the load to the center of the specimens. The G10 piston remained in contact with the restorative material surface to avoid impact damage. After fatigue, all specimens were analyzed using transillumination with blue light to detect cracks.[23,24]

Specimens that survived mechanical cycling were tested under a compressive load in a universal testing machine (DL 2000; EMIC) (Fig. 1). A compressive load was applied by a flat (3 mm in diameter) stainless steel

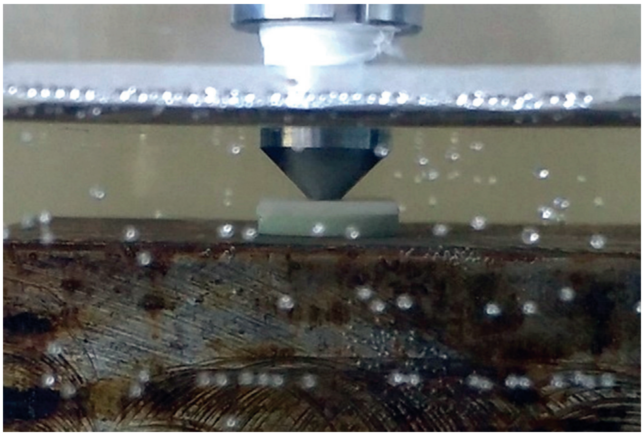

Figure 1. Monotonic compression test with metal piston performed in 37 °C distilled water.

piston with cross-head speed of 0.5 mm/min in 37 °C distilled water. When the sound of the first crack was detected by an acoustic system (Audacity Sound Editor; Free Software Foundation), the test was interrupted, and the failure load (in N) was recorded.

Failure load data were evaluated using Weibull analysis to estimate the characteristic load (L_0) and Weibull modulus (*m*) of the experimental groups (Minitab 14; Minitab). The maximum likelihood estimation approach was used.[25]

All specimens were analyzed using transillumination to identify fractographic features.[23,24] Failure modes were as follows: radial crack, crack originated from ceramic-cement (intaglio) interface (Fig. 2A); cone crack, surface cone crack (Hertzian) originated from the contact area with the piston (Fig. 2B, C); combined failure, when both radial crack (located in the intaglio surface) and cone crack (located in the restorative material surface) were found; and catastrophic fracture, complete failure of the specimen, with material detachment. The chi-square test was used to verify the relation between experimental group and failure mode (α=.05).

The plate-on-foundation theory was used to predict the critical load for radial cracking (L_R) using the following equation[13,16]:

$$L_R = \frac{B\sigma d^2}{\log\left(\frac{CE}{E^*}\right)},$$

where σ and E are the flexural strength and elastic modulus of the restorative material, respectively, B (1.35) and C (1) are dimensionless constants, and E^* is the effective modulus of cement/substrate layers according to the following equation:

$$E^* = E_C\left(\frac{E_s}{E_c}\right)^L,$$

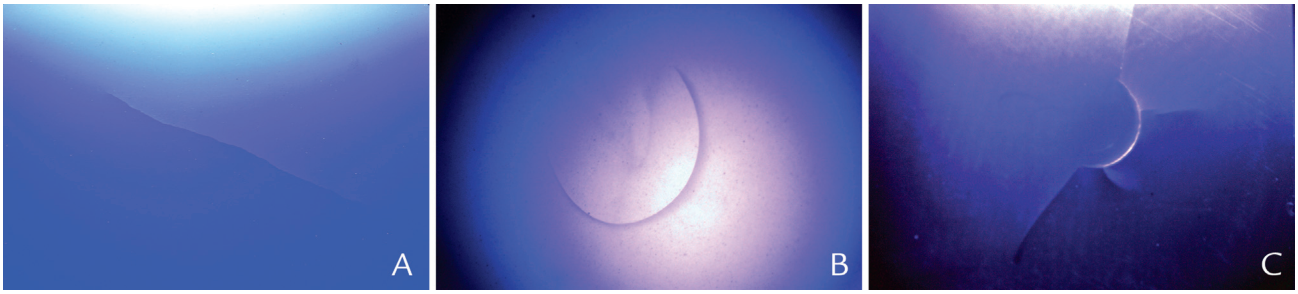

Figure 2. Representative images of failure modes verified with transillumination (*blue light*) (original magnification ×10). A, Radial crack. B, Cone crack. C, Cone crack with radial propagation.

where E_c is the elastic modulus of cement, and E_s is the elastic modulus substrate. L is a dimensionless function:

$$L=\exp\left\{-\left[\alpha+\beta\log\left(\frac{h}{d}\right)\right]^{\gamma}\right\},$$

where α is equal to 1.18, β is equal to 0.33, and γ is equal to 3.13; d is the thicknesses of the restorative material, and h is the thicknesses of the cement layer.

RESULTS

All specimens (100%) from group FelDenAn failed during mechanical cycling. Most specimens (80%) from the group PICNRc failed during fatigue, and the surviving specimens (4) were tested in compression (Table 2); but, data were not included in the statistical analysis. As more than 10 specimens survived mechanical cycling in other groups, data were statistically analyzed.

Groups PICNDenAn and PICNDen showed similar characteristic load (L_0) but statistically different Weibull modulus (*m*), suggesting a greater reliability for DenAn than for Den as a substrate material. PICNNiCr showed the greatest characteristic load (L_o) and the lowest number of failures under fatigue. The L_0 of ResDenAn was statistically greater than PICNDenAn and PICNDen, as their confidence intervals did not overlap (Table 2).

A significant relationship was detected between the experimental group and failure mode (P<.001, chi-square test with 15 degrees of freedom) (Table 3). Groups PICNRc and FelDenAn, which showed the highest number of failures during fatigue, had a higher frequency of radial cracks. Most specimens from group PICNNiCr failed from cone cracking. When tested in compression, radial cracks emanating from the cone cracks at the PICN surface could also be observed (Fig. 2C).

The L_R was calculated for groups PICNDen, PICNDenAn, PICNRc, and FelDenAn, which met the requirements of the theory of plates on elastic foundations (the substrate has lower E than the restorative material). Data used for the calculation were obtained from the literature and are presented in Table 4. L_R was lower for groups FelDenAn and PICNRc.

DISCUSSION

The first study hypothesis was accepted as the supporting substrate influenced the failure behavior of PICN. The substrate with the highest elastic modulus (NiCr) resulted in the greatest L_0 after mechanical cycling. In addition, PICNNiCr had the greatest number of specimens that survived mechanical cycling. These findings agree with those of a previous report where loads 2.2 times higher were required to create a radial crack on the porcelain cemented to NiCr compared with porcelain cemented on composite resin.[14]

When the supporting substrate has a higher elastic modulus than the restorative material, tensile stresses at the material undersurface are suppressed, and the structure is more likely to fail from cone cracking (or quasiplasticity) at the top surface, as observed for group PICNNiCr (90% of cone cracks).[12] This stress distribution favors the mechanical behavior of the layered structure resulting in higher fracture load values. Therefore, good mechanical behavior could be expected for PICN restorations cemented onto metal posts or onto metal implant abutments.

However, if the restorative material is bonded to a less rigid supporting substrate, its flexure induces high tensile stresses at the intaglio surface, generating radial cracks. In this situation, critical loads for radial cracking (L_R) can be estimated using the theory of plates on elastic foundations.[13,16] The lowest L_R values were estimated for groups FelDenAn and PICNRc, which had the highest frequency of failure during mechanical cycling, most originating from radial cracks. These groups also showed a larger mismatch between the elastic modulus of the restorative material and the substrate, which has been associated with a decrease in the L_R of layered structures.[13,16] Moreover, the heterogeneity of the fiberglass post and composite resin substrate may also contribute to the inferior mechanical behavior of PICNRc.

Table 2. Number of specimens (n) that failed during mechanical cycling (fatigue) and under compressive load with mean ±standard deviation (SD) values of compressive failure load, Weibull modulus (*m*), characteristic load (L_0), and respective 95% confidence intervals (95% CIs) for experimental groups

			Compressive Load (N)			
Restorative Material	Groups	Fatigue Failure (n)	n	Mean ±SD	*m* (95% CI)	L_0 (95% CI) (N)
PICN	PICNNiCr	2	18	3322 ±679	5.7 (4-8.2)[ab]	3588 (3296-3906)[a]
	PICNRc*	16	4	1512 ±1099	–	–
	PICNDen	5	15	2026 ±742	3.2 (2-4.8)[b]	2251 (1905-2660)[c]
	PICNDenAn	9	11	2300 ±280	9 (5.7-14.1)[a]	2424 (2260-2598)[c]
Composite resin	ResDenAn	6	14	2753 ±689	5.7 (3.6-8.9)[ab]	2986 (2714-3286)[b]
Porcelain	FelDenAn*	20	0	–	–	–

PICN, Polymer-infiltrated ceramic network. Values followed by similar letters in same column statistically similar (Weibull analysis, maximum likelihood estimation approach). *No quantitative statistical analysis performed for these groups.

Table 3. Frequency of each failure mode for experimental groups tested in fatigue (F) and compressive load (CL)

	Failure Modes							
	Radial		Cone		Combined		Catastrophic	
Groups (n=20)	F	CL	F	CL	F	CL	F	CL
PICNNiCr	1 (5%)	0 (0%)	1 (5%)	17 (85%)	0 (0%)	0 (0%)	0 (0%)	1 (5%)
PICNRc	15 (75%)	2 (10%)	0 (0%)	2 (10%)	0 (0%)	0 (0%)	1 (5%)	0 (0%)
PICNDen	4 (20%)	0 (0%)	1 (5%)	7 (35%)	0 (0%)	6 (30%)	0 (0%)	2 (10%)
PICNDenAn	7 (35%)	0 (0%)	1 (5%)	10 (50%)	1 (5%)	1 (5%)	0 (0%)	0 (0%)
ResDenAn	6 (30%)	4 (20%)	0 (0%)	10 (50%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)
FelDenAn	14 (70%)	0 (0%)	1 (5%)	0 (0%)	5 (25%)	0 (0%)	0 (0%)	0 (0%)
Total	47	6	4	46	6	7	1	3

Table 4. Values of strength (σ), elastic modulus (E), and thickness (d) of restorative materials; elastic modulus (E_c) and thickness of cement layer (h); elastic modulus of substrate (E_s); and critical load for radial cracking (L_R)

Groups	σ (MPa)	E (GPa)	d (mm)	E_c (GPa)	h (mm)	E_s (GPa)	L_R (N)	E*/E[a]
PICNNiCr[b]	180[c]	37[e]	1.0	5[d]	0.01	200[d]	–	–
PICNRc	180[c]	37[e]	1.0	5[d]	0.01	10[d]	402	4.0
PICNDen	180[c]	37[e]	1.0	5[d]	0.01	18[g]	639	2.4
PICNDenAn	180[c]	37[e]	1.0	5[d]	0.01	15[h]	540	2.8
ResDenAn[b]	110[d]	9[d]	1.0	5[d]	0.01	15[h]	–	–
FelDenAn	138[c]	70[f]	1.0	5[d]	0.01	15[h]	256	5.3

[a]E* effective modulus of cement/substrate layers. [b]Did not meet requirements for L_R calculation. [c]Albero et al[9] (2015). [d]Values obtained from manufacturer. [e]Della Bona et al[7] (2014). [f]Borba et al.[6] (2011). [g]Kelly et al[14] (2010). [h]Yi and Kelly[22] (2008).

Specimens cemented onto dentin analog (NEMA G10) and onto dentin showed similar L_0 and L_R values as both substrates have similar elastic moduli.[14] Yet, the DenAn substrate resulted in greater *m* value, which may be explained by the structural variability of the dentin substrate, which is not found in the DenAn. The mechanical and physical characteristics of dentin depend on the structural composition and dentin type (dentinal tubules, peritubular dentin, and intertubular dentin), which varies between teeth and changes with age or/and from external stimulus.[17,26] In addition, the Den substrate was composed not only of dentin but also of the surrounding enamel layer. Therefore, during compressive loading, a nonuniform stress distribution was induced in PICN due to the mismatch between the elastic properties of dentin and enamel and resulted in secondary surface cracks located in PICN periphery (close to dentin-enamel junction). Thus, the more homogeneous behavior of the DenAn substrate supports its indication as a replacement material for dentin in mechanical tests, corroborating previous studies that showed its elastic and adhesive properties to be similar to those of dentin.[14,21]

The different restorative materials bonded to the same supporting substrate resulted in different failure behavior of the structures, leading to acceptance of the second study hypothesis. The structure can be reinforced if the supporting substrate has a higher elastic modulus than the restorative material. Therefore, even though the indirect composite resin has lower flexural strength than PICN and feldspathic porcelain (Table 4), it showed better mechanical behavior as the substrate DenAn is stiffer than the composite resin investigated.

The same rationale applies for the FelDenAn group. A combination of a less rigid substrate and a high elastic modulus mismatch between materials resulted in failure of all the FelDenAn specimens during cycling. In addition, ceramics are susceptible to fatigue, and radial cracks may be expected to undergo slow crack growth if water gains access to the flaws in the ceramic undersurface.[6,12] Additional effects from deformation in less rigid substrates and cement degradation can be expected to affect the mechanical behavior of the structures.[12] Nevertheless, studies have reported that PICN has higher damage tolerance and resistance to fatigue than feldspathic porcelain, which is consistent with the findings of the present study.[9,10]

Mechanical cycling was performed based on clinical parameters such as occlusal load (50 N) and frequency (2 Hz), the humidity, and human body temperature (37 °C).[14,24] Radial cracks originating at the intaglio surface were observed for 47 (81%) of all specimens that failed in fatigue. Cone cracks and combined failure modes were commonly found in specimens subjected to the compressive load test. This could be explained by the fact that most specimens that survived mechanical cycling were bonded to stiffer substrates (PICNNiCr and ResDenAn) that are more prone to cone cracking.[12] Moreover, the test configuration in which the compressive load was applied by a metal piston with a high elastic modulus may increase the stress concentration in the specimen surface. Nevertheless, although radial cracking at the restorative material intaglio surface may be a principal source of failure, cone cracking at the surface remains a competing mode.[12]

Extrapolation to the clinical situation should consider limitations such as the simplified geometry of tested specimens, which neglects the effect of restoration geometry on stress distribution, and the number of specimens (11-18) used for Weibull analysis.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. The supporting substrate influenced the fracture load and failure mode of PICN.
2. The substrate with higher elastic modulus resulted in higher fracture load values of PICN.
3. When a less rigid substrate was used, a small mismatch between the elastic modulus of the different materials of the structure is desired.
4. PICN cemented on the dentin analog resulted in more reliable values than when cemented on human dentin.
5. Polymer-content materials were more resistant to mechanical cycling than the feldspathic porcelain.

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https://doi.org/10.1016/j.prosdent.2018.08.008