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RESEARCH ARTICLE

MECHANICAL PERFORMANCE OF EXPERIMENTAL ZIRCONIA-REINFORCED POLYMER-INFILTRATED CERAMIC NETWORK MATERIAL VERSUS COMMERCIAL ENAMIC HYBRID CERAMICS

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ABSTRACT:

Background: Modern dental materials such as the porous three-dimensional structure of feldspathic ceramic infiltrated with acrylic resins, have revolutionized the field of dental ceramics for CAD-CAM technologies. This study aims to evaluate the mechanical properties of the experimental polymer-infiltrated ceramic network (PICN) and compare it to Vita Enamic, as the gold standard the commercial (PICN). **Materials and Methods**: In this study, two groups were investigated, the commercial PICN material (Vita Enamic) and experimental PZ10. Ten bar-shaped specimens were cut from each investigated material to assess the Vickers microhardness and flexural strength (FS) using a three-point bending test combined with elastic modulus. The resulting data were statistically analyzed. **Results**: Vita Enamic had a significantly higher (FS) of (158.10 MPa), elastic modulus (19.32 GPa) and hardness (206.29 VHN) in comparison to those values of exp. PZ10; 84.22 MPa, 6.55 GPa and 166.76 VHN, respectively. **Conclusion**: Vita Enamic had superior mechanical properties compared to the exp. PZ10. However, the exp. PZ10 could be used as a substitute for indirect restorations with an elastic modulus simulating dentin tooth structure thus reducing the chances of debonding.

Keywords: Indirect dental restoration, Polymer infiltrated ceramic network (PICN), Vita Enamic, Mechanical properties, flexural strength, elastic modulus, microhardness.

1. INTRODUCTION:

Modern dentistry has been revolutionized by CAD-CAM technology. High-performance materials were developed by the most recent advancements in CAD-CAM technologies. All-ceramic restorations have proved successful due to the low cost and enhanced clinical performance associated with increased precision and ease of use **Róth et al** (2022). Furthermore, the processing time of high-strength ceramics can be reduced by 90% **Munoz et al** (2023).

Three types of modern materials are available and they are compatible with CAD/CAM technology; CAD/CAM: glass ceramics, composite resins or hybrid ceramics, and polycrystalline alumina and zirconia. Hybrid ceramics are mainly used as indirect permanent restorations **Yin et al** (2019). All ceramic materials have the advantage of providing a perfect fit which allows for the best possible results for aesthetic restorations. All-ceramic systems, which include feldspathic, glass, and glass-reinforced ceramics, appear to be best suited for single crowns because of their low mechanical stability **Pjetursson et al** (2023). Different types of ceramics are available such as IPS e.max CAD (Ivoclar Vivadent AG), a lithium disilicate (Li2Si2O5) with 35 to 45 volume proportion of evenly dispersed 1-5 mm leucite crystals. On the other hand, blocks of Mark II (Vita Zahnfabrik) consist of a fine-particle (4 mm) feldspathic porcelain employing a sintering process at 1170 ^{o}C under vacuum that can generate a homogenous microstructure ceramic block for the milling process **Skorulska et al** (2021).

Recently, Vita Enamic (Vita Zahnfabrik) was approved as a polymer-infiltrated ceramic network (PICN) material **Coldea et al** (2013a), **Coldea et al** (2013b). This substance is considered a hybrid ceramic material because it comprises a resin-interpenetrated sintered ceramic network that forms a double-connecting network structure. The manufacturer does not disclose the manufacturing method, however, the mass proportion of the inorganic ceramic portion is 86 wt.%, and the organic polymer portion is 14 wt.% **Coldea et al** (2013b). The dominating ceramic network of a significant leucite-based phase of feldspar origin and a small crystalline phase of zirconia serves as a reinforcing component. Microstructurally, the ceramic network resembles filler particles from composites made of resin in certain ways **Della Bona et al** (2014). According to the manufacturer, elasticity is provided by the polymer network and stability is provided by the dominating fundamental ceramic network **Coldea et al** (2013b), **Della Bona et al** (2014). Additionally, this material demonstrated certain encouraging mechanical qualities similar to the dentin and enamel of human teeth; therefore, it has been recommended for veneers, onlays, and inlays **Della Bona et al** (2014).

Furthermore, it is asserted that this class of ceramics absorbs masticatory stresses and prevents crack formation **Coldea et al** (2013b). Materials typically develop cracks due to internal or surface flaws and/or voids, which subsequently spread to the grain-to-grain contact or weaker phases of the material. Surface imperfections in machinable materials may arise from the hydrofluoric acid etching or milling process. The heterogeneous phases of ceramics and resin found in vita enamic may be disturbed (phase separated) by mechanical stresses like flexure and compression. Furthermore, an efficient technique was created to stop the spread of cracks by adding crystals to interlock and reinforce phases **Coldea et al** (2013b), **Della Bona et al** (2014).

Due to its appealing qualities being closely linked to its crystalline polymorphs, zirconia has emerged as one of the most important ceramic materials in recent years. Tetragonal zirconia (t-ZrO2) causing a high degree of strength and fracture toughness is suitable for use in structural dental applications **Bekale et al** (2006). Furthermore, dental ceramics that are stronger, more translucent, and more resistant to deterioration at low temperatures are produced when partly stabilized zirconia (PSZ) is produced in nanosized grains **Shahmiri et al** (2018), **Zang et al** (2018). Therefore, tetragonal zirconia is a suitable component to arrest crack propagation in these hybrid ceramics. **Mohammed et al** (2021) and **Cui et al** (2020) found that the incorporation of biocompatible or tougher with different contents into experimental PICN material enhanced the mechanical properties without affecting its machinability.

The similarity of hybrid ceramics to tooth structure regarding mechanical properties has encouraged their fabrication. Therefore, the present study aimed to compare fabricated PICN containing 10% wt. of nanocrystalline t-ZrO2 to the most commercially popular hybrid ceramic in the dental market which is Enamic material in terms of flexural strength (FS), elastic modulus, and microhardness properties. The null hypothesis stated that there would be no difference in Flexural strength (FS), elastic modulus (E_f), and microhardness parameters between experimental polymer-infiltrated ceramics reinforced with 10wt% of nanocrystalline t-ZrO2 (PZ10) and Vita Enamic.

2. MATERIALS AND METHODS

Materials:

The materials used in this study are listed in Table 1. Two hybrid ceramics were investigated; experimental polymer infiltrated ceramic material containing 10wt.% of calcia stabilized tetragonal nanocrystalline zirconia (PZ10) and Enamic (Vita Zahnfabrik, Germany).

Table 1: Composition and structure of materials used in the study:

Component and Producer	**Category**	**Composition and Structure**
Enamic (Vita Zahnfabrik, Germany).	Hybrid ceramics	Porous structure-sintered ceramic matrix infiltrated with polymer material Inorganic ceramic 86 wt%: fine-structure feldspar ceramic enriched with aluminium oxide (silicon dioxide 58–63%, aluminium oxide 20–23%, sodium oxide 9–11%, potassium oxide 4–6%, boron trioxide 0.5–2%, zirconia <1%, calcium oxide <1% . Organic polymer 14 wt.% (urethane dimethacrylate, triethylene glycol dimethacrylate)
Experimental polymer-infiltrated ceramic material.	Hybrid ceramics	sodium aluminium silicate (Sigma-Aldrich, Germany) (82%SiO_2, 9.5%Al_2O_3, 8% Na_2O). 10% calcia stabilized zirconia. Organic copolymer TEGDMA (Triethylene glycol) dimethacrylate, Bis-GMA (glycerolate dimethacrylate).

Synthesis of experimental PICN material based on 10wt.% of t-ZrO2:

Nanosized 7- calcia stabilized zirconia (7-tCSZ) powder was prepared by modified coprecipitation method **Mohammed et al** (2021), **Foo et al** (2019). The previously prepared powder was incorporated in 10wt.% into aluminum silicate powder (82%SiO_2, 9.5%Al_2O_3, 8% Na_2O) and they were properly mixed. These different weights of aluminium silicate and nanosized 7-tCSZ powder were measured by a sensitive balance with an accuracy of 0.0001gm (Adam Lab PW 124 analytical Balance. England). The compositional ratio of the aluminium silicate/tetragonal zirconia (7-tCSZ) in the ceramic networks used was 90:10 (wt.%) respectively.

As a binder, 3 weight percent of polyvinyl alcohol (PVA) was used in an aqueous solution (Oxford Lab Chem, Mumbai, India). Each of the powder mixtures received 1 ml of the PVA solution. Following thorough mixing, the combined powders known as "green bodies" were formed using a spherical stainless-steel mould and uniaxially compressed for 2 min at 158 MPa. They were packed using a uniaxial hydraulic press (SEIDNER, uniaxial hydraulic press, Hessen- Germany) into discs (25 mm in diameter and 4 mm thickness) followed by drying at 120 ºC for 24 hrs. The porous disc structure was sintered in an electric furnace at 700 ºC with a heating rate of 5 ºC/min until it reached 525 ºC. It was then soaked for 30 minutes. Finally, the temperature was raised to 700 ºC with the same heating rate and soaked for an hour. The resulting ceramic networks were expressed as Z10 after sintering.

Subsequently, the polymer matrix was prepared by regularly mixing A glycerolate dimethacrylate (Bis-GMA) and Tri(ethyleneglycol) dimethacrylate (TEGDMA) (Sigma Aldrich Chemical Co., USA) at a ratio of 1:1. As an initiator, use benzoyl peroxide (BPO) from Sigma Aldrich Chemical Co. in the USA. To achieve infiltration, the sintered ceramics that were previously constructed were submerged in the prepared monomer mixture for 24 hours.

The monomers were absorbed into ceramic discs through capillary action at room temperature. Ultimately, after the polymerization was triggered by heat treatment at 70 °C for two hours and 110 °C for an extra two hours under air pressure, the experimental PICN material comprising 10wt. percent of 7-CSZ (ex. PZ10) material was achieved. Then the experimental PICN material was prepared in the form of discs to be sectioned into the required dimensions for mechanical testing.

Samples preparation

Before the investigation, a power calculation based on the collected data was used to establish the number of samples needed in each group **Leung et al** (2015). Forty specimens (n=20) in each group, 10 specimens for each test (FS and microhardness) were found to provide 80% power for the independent samples T-test with a confidence interval of 95% and a level of 5 percent significance using G* Power 3.1.9.2 software **Faul et al** (2007). A total of forty specimens (n=40) were prepared in this study. Twenty specimens (n=20) for each investigated material (exp. PZ10 and Vita enamic materials) were constructed. For the flexural strength (FS) test, ten bar-shaped specimens (n=10) with dimensions of 2.2×2.2×15mm^3 were cut using a cutting machine (IsoMet 4000 Buehler Germany). For Vickers microhardness test, ten high gloss polished specimens (n=10) with dimensions 5×5×2 mm^3 were prepared.

Flexural strength (FS):

Three-point bending test was utilized to determine the FS. Wet polishing cloths and fine polishing paste (MetaDi Ultra1um Buehler Ltd., Illinois, USA) were used in sequence to polish each specimen until no apparent scratches remained on the surface. Using the Instron universal testing machine (Model 3345- England), the specimen was positioned centrally on a 3-point bending platform with a 12 mm supporting distance (**fig.** 1) until fracture, a load was delivered at a crosshead speed of 1 mm/min. The formula ($\sigma = 3Fl/2bh^2$) was used to calculate the flexural strength (σ) in MPa (mm), where F is the fracture (N), l is the distance between the two supporting rollers (mm), b is the width of the ceramic specimen (mm) and h is the height of the ceramic specimen. All bending bars were chamfered to reduce stress concentration from machining flaws.

Fig. (1): specimen of exp. PICN material for flexural strength testing mounted on universal testing machine.

Elastic modulus:

The elastic modulus; E_f was determined from the outcomes of three-point bending tests using the formula ($E_f = (F/m)\, l^3/4bh^3$) where; l is the distance of the roller span; b is the width and h is the height of the specimen, and (F/m) is the slope of the force-displacement curve **Cui et al** (2020).

Vickers microhardness:

The surface microhardness was determined using a hardness tester (TUCON1102 Wilson hardness tester, Buehler, Germany) on the previously prepared specimens of the tested materials (Fig 4). Three Vickers indentations were made using load 1.96 N. The indenter is held in place for a dwell time of 20 seconds. The mean hardness value was calculated for each specimen. The hardness was calculated using the formula ($H= 1.854 \times F/d^2$) where F is the load and d is the indentation diagonal length **Coldea et al** (2013b).

Statistical analysis:

Version 23.0 of the statistical program for social sciences was used for statistical analysis (SPSS Inc., Chicago, Illinois, USA). When the distribution of the quantitative data was parametric, they were displayed as ranges and as mean± standard deviation. Using the Shapiro-Wilk and Kolmogorov-Smirnov tests, data were examined for normality. Mann Whitney U test was performed for two-group comparisons in nonparametric data at a 5% margin of error and 95% confidence interval. Thus, the following p-value was deemed significant: A probability P-value of 0.05 was regarded as negligible.

3. RESULTS

Tables (2) displayed the mean values and standard deviations for the tested groups' flexural strength and elastic modulus.

Flexural strength:

The results of FS showed a statistically significant difference ($p<0.001$) between the two groups. The Enamic group's flexural strength (158.10±10.81MPa) was substantially greater than that of the experimental PZ10 group's (84.22±8.91MPa) Table (2) and **Fig.** (2).

Elastic modulus:

The elastic modulus of Enamic (19.32±0.62 GPa) was statistically significantly higher than that of experimental PZ10 (6.55±1.38 GPa) ($P<0.001$) Table (2) and **fig.** (3).

Table (2): The mean ±SD obtained for the flexural strength (MPa) and elastic modulus (GPa) of the tested groups.

Flexural strength and elastic modulus / **Materials**	**Mean**	**SD**	**Test value**	***P* value**
Vita Group (n=10)	158.10 19.32	10.81 0.62	16.682 26.771	<0.001* <0.001*
Exp.PZ$_{10}$ Group (n=10)	84.22 6.55	8.91 1.38		

Mean ±SD obtained through t-independent Sample t-test; p >0.05 is insignificant, p <0.05 is significant.

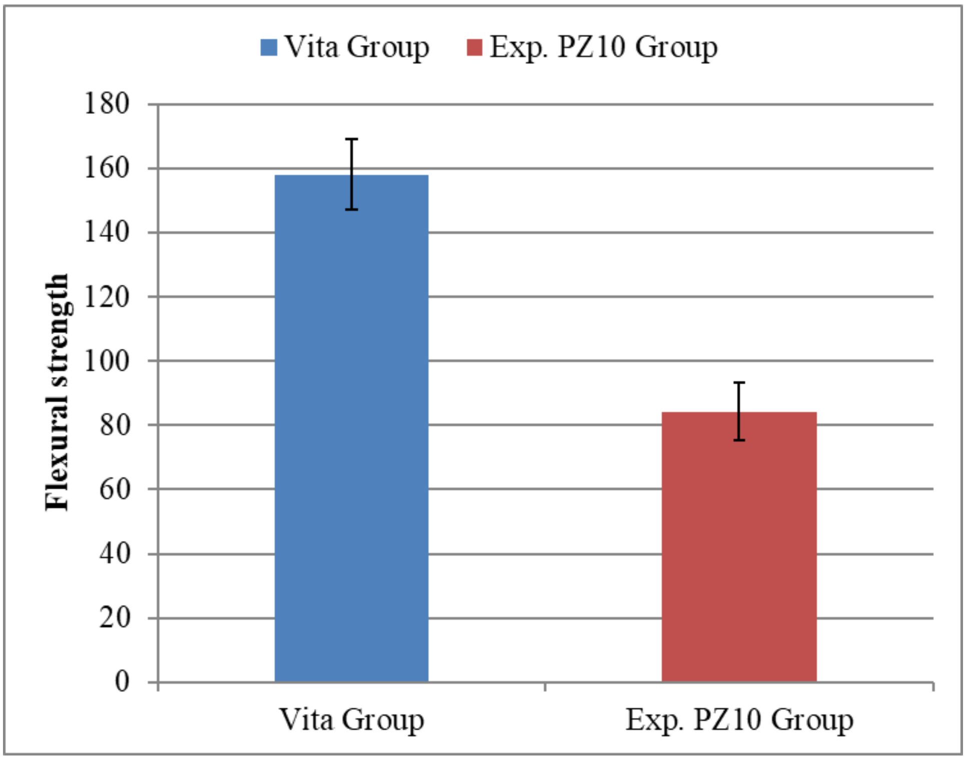

Figure (2): Bar chart of flexural strength of Vita and exp. PZ10 groups.

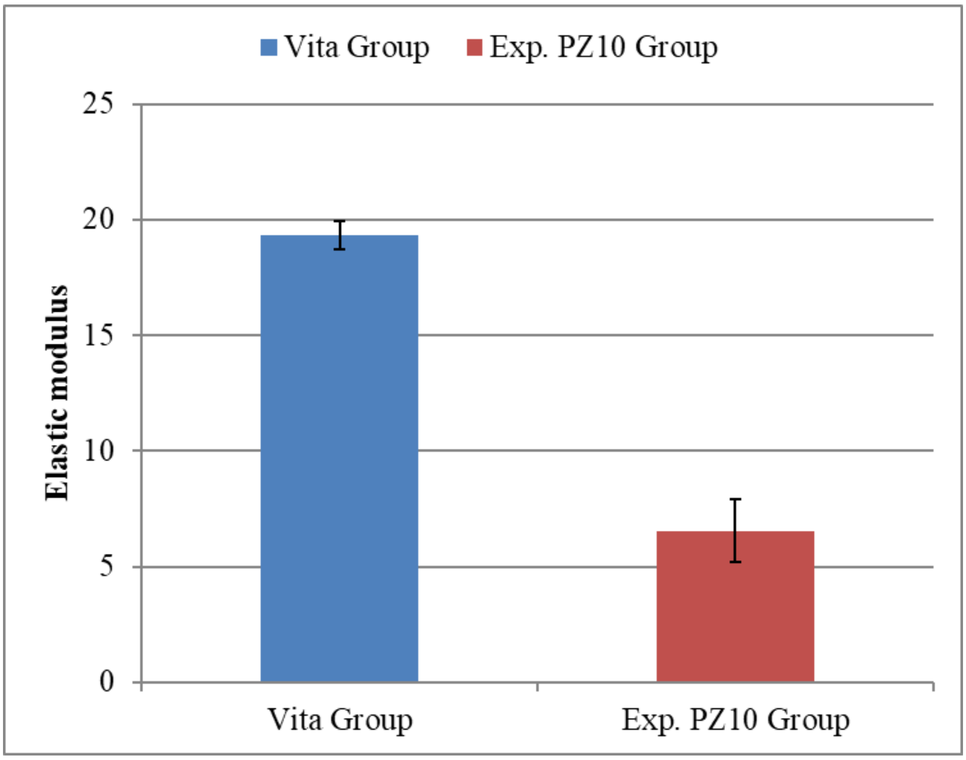

Figure (3): Bar chart of elastic modulus of Vita and exp. PZ10 groups.

Vickers microhardness

Moreover, a noteworthy distinction in Vickers microhardness was observed between the two cohorts (P<.001). Vita enamic was found to be significantly (206.29±43.85 VHN) higher than that of exp. PZ10 group (166.76±4.58 VHN). **Table** (3) and **fig.**(4) present the average values and standard deviations for the tested groups' flexural strength, elastic modulus, and Vickers microhardness.

Table (3): The mean ±SD obtained for Vickers microhardness (VHN) of the tested groups.

Microhardness / **Materials**	**Mean**	**SD**	**Test value**	***P* value**
Vita Group (n=10)	206.29	43.85	2.835	0.011*
Exp.PZ$_{10}$ Group (n=10)	166.76	4.58		

Mean ±SD obtained through t-Independent Sample t-test; p >0.05 is insignificant, p <0.05 is significant.

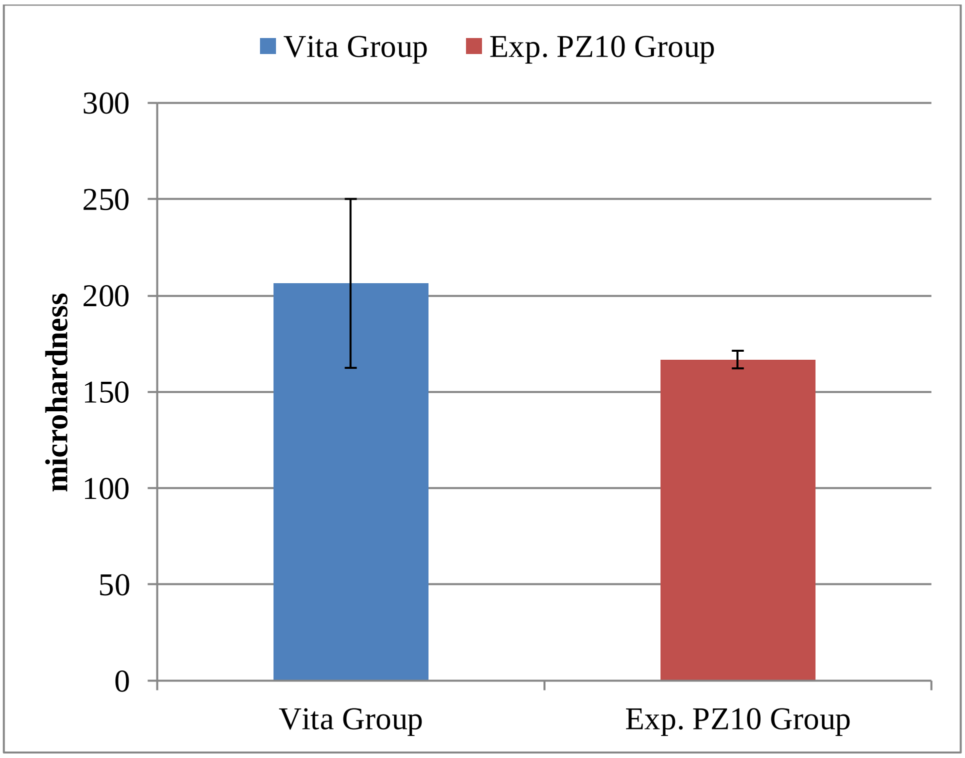

Figure (4): Bar chart of Vickers microhardness of Vita and exp. PZ10 groups.

4. DISCUSSION:

This study's main objective was to evaluate the mechanical characteristics of an experimental polymer-infiltrated ceramic material (exp. PZ 10) with Vita enamic, the most widely used hybrid CAD/CAM material on the market. Schlenz et al (2019) and Kruzic et al (2018) found that resin-infiltrated ceramics exhibited favorable biomechanical behavior due to their low elastic modulus and high polymeric content. This led to enhanced damage tolerance and occlusal force reduction. Replacing missing tooth structures with a material whose physical characteristics and structure are like natural teeth is one of the fundamental goals of restorative dentistry. For this reason, CAD-CAM resin-infused ceramics are quickly gaining traction since they offer dentin's rigidity and long-term attractiveness **Coldea et al** (2013b). It was concluded that the mechanical properties of these materials resemble those of natural dentin and may be similar to enamel. This has been the goal of restorative materials over the years Bottino et al (2015), He and Swain (2011).

When assessing brittle materials like ceramics, FS is a crucial mechanical criterion. A common test for the determination of flexural strength is the 3-point bending test Della Bona et al (2014). The FS of the current experimental PZ10 was (84.22±8.91 MPa), which significantly exceeded the value reported by an earlier study (52.61±8.82 MPa) by **Mohammed et al** (2021). This might be attributed to the variation in the distribution of flaws among the specimens which might lead to a change in the FS values. On the other hand, the results of FS of Enamic (158.10 ±10.81 MPa) were consistent with the previous study by **Albero et al** (2015). Additionally, the result of the FS of Enamic material in the current study was comparable with data from the manufacturer (150-160MPa).

However, the value of the strength of vita enamic in the current study was higher than that of exp. PZ10. This might be due to the higher density of Veta Enamic which may be attributed to the differences in composition and sintering temperature **Coldea et al** (2013a), **Coldea et al** (2013b). Feldspar ceramic enriched with aluminium oxide is the main component of the ceramic matrix of Enamic which has greater strength and density than unreinforced silica of exp. PZ10. Furthermore, **Della Bona et al** reported that the presence of a fewer flaws at polymeric and ceramic network boundaries was caused by the silanization of the ceramic network of Enamic **Della Bona et al** (2014).

Although the flexural strength of exp. PZ10 showed a lower value, it might be considered higher than the ceramic network alone (30MPa) as found in the previous study[7]. When compared to earlier single ceramic components, the current two-phase experimental material's improved FS in this investigation may indicate a polymeric phase reinforcement process. The highest mean FS value of 84.22±8.91 MPa was observed in the Exp.PZ10 group. The results presented herein suggest that this experimental PICN material may be suitable for anterior crowns, and adhesively cemented laminate veneers, inlays and onlays. This was in accordance with Albero et al who suggested that FS ranging 50-100 MPa is required for such restoration. However, its use as a posterior crown material may increase the risk of fracture as a minimum of 100 MPa is required in the posterior area **Albero et al** (2015).

The influence of introducing nanoparticles into the material influences microhardness. Although the hardness of exp. PZ10 (166.76 ±4.58 VHN) was lower than that of Vita enamic (206.29 ±43.85 VHN), the former was comparable to the hardness of dentin (102 VHN). Nevertheless, neither of the tested materials' hardness levels was as higher as that of enamel (611 VHN) **Xu et al** (1998), **Cuy et al** (2002), **Mahoney et al** (2000). The experimental material (exp. PZ10) appeared less destructive towards opposing tooth structures. Enamic and exp. PZ10 have a porous ceramic structure infiltrated by resin. It was easy to remove the resin which has less hardness from porous ceramic nature. This is consistent with the findings of Ruizhi Yin et al who reported that Enamic had lower hardness than other commercial CAD/CAM materials **Yin et al** (2019). The current study estimated that the hardness value was less than that reported by Leung et al, **Leung et al** (2015). This disagreement is anticipated, due to the differences in the dwell time applied to the tested materials in both studies. Furthermore, the outcome of the Enamic material in this investigation was similar to values obtained from the manufacturer (254 VHN).

A material's resistance to elastic deformation is a measurable quantity can be expressed as its elastic modulus. While the ceramic and polymeric phases of the two investigated PICN materials are identical, the experimental material exhibits a significantly lower elastic modulus. It was clarified that the ceramic matrix phase of both materials primarily influences and determines the elastic constants hence, the internal structure of both materials differs **Belli et al** (2017). The result of the elastic modulus of Enamic (19.32±0.62 GPa) was higher than that of exp. PZ10 (6.55 ±1.38 GPa). The cause of higher elastic modulus as well as the hardness of Enamic rather than that of exp. PZ10 might be due to the difference in ceramic fraction and precursor between both materials **Coldea et al** (2013a), **Coldea et al** (2013b).

Although the elastic modulus of exp. PZ10 (6.55 ±1.38 GPa) was lower than that of Vita enamic (19.32±0.62 GPa), it highly resembles the elastic modulus of adhesive luting cement (6.8-10.8 GPa) as reported by Song et al and Ausiello et al Song et al (2018) and Ausiello et al (2004). Also, it was found that the elastic moduli of both Vita enamic and exp. PZ10 resembles that of dentin (8.7-25GPa) as recorded by Kinney et al and Ziskind et al (Kinney et al 1996 and Ziskind et al 2011). This elastic modulus similarity between the dentin from one side and luting cement materials from the other allows stability under an appropriate, uniform stress distribution, hence reducing the likelihood of debonding Swain et al (2016).

As seen from the current study, the mechanical properties were greatly affected by ceramic network precursors and fractions. However, because the tests were conducted in vitro and there were no clinical efficacy observations, this study has certain limitations. To fully understand the mechanical characteristics of these restorative materials in an oral environment, more investigation is needed. Additionally, more research needs to be done to improve the exp PZ10 by varying the percentage and employing various ceramic precursors.

In addition, mechanical characteristics should be considered while selecting a restorative material in clinical prosthodontics. Under physiological settings, the best restorative material for a crown should possess qualities like those of enamel. Therefore, based on the findings of this study, we recommend using Exp PZ10 for anterior crowns and adhesively cemented laminate veneers, inlays, and onlays where debonding is not a risk. Further improvements should be performed to allow for the use of this material as a posterior crown or FPD.

5. CONCLUSION:

Taking into consideration the limitations of this investigation, it appears that Vita enamic's mechanical properties are superior to those of the exp. PZ10. However, the exp. PZ10 could be considered a valid material for indirect restorations with an elastic modulus simulating dentin tooth structure, thereby reducing the chances of debonding.

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