

Longevity of ceramic restoring materials in the confection of endocrowns restorations: an in vitro study on surface roughness and mechanical strength

Longevidade de materiais restauradores cerâmicos na confecção de restaurações endocrowns: um estudo in vitro sobre rugosidade superficial e resistência mecânica

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ABSTRACT

Objectives: The objective was to evaluate the longevity of ceramic restorative materials in the manufacture of endocrown restorations, through an in vitro study on surface roughness and mechanical strength. **Methods:** Three restorative materials were evaluated and assigned to experimental groups (n=10 disc specimens): Leucite Reinforced Ceramic, Lithium Disilicate and Nanoceramic Resin. These restorative materials were evaluated for surface before and after aging (n=02 specimens) under a stereomicroscope. The specimens were submitted to the average surface roughness test (Ra) (n=10) in a contact roughness meter before and after aging. As well as the biaxial flexural strength test (n=10), after aging, up to a test speed of 0.5 mm/min. Aging was carried out in a thermocycler, with 5,000 cycles with baths of 5 °C ± 1 ° and 55 °C ± 1 °. Fragments after fracture were observed under a stereomicroscope. The data obtained were tabulated and analyzed using the Minitab statistical program. **Results:** The surfaces of the specimens do not show changes between the restorative materials and also in relation to aging. For the roughness data, the restorative material factors (p=0.867) and aging (p=0.321) were not statistically significant. The DIS group presented the highest values of fracture resistance (p=0.000), in relation to the LEU and REN groups, which were statistically similar. The same statistical pattern was identified for post-fracture fragment data (p=0.030). **Conclusion:** The aging factors and restorative material do not interfere with the surface roughness performance. However, the mechanical performance and number of fragments after fracture is affected by the restorative material.

Indexing terms: Ceramics. Computer-aided design. Flexural strength.



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RESUMO

Objetivos: Objetivou-se avaliar a longevidade de materiais restauradores cerâmicos na confecção de restaurações endocrowns, através de um estudo *in vitro* sobre rugosidade superficial e resistência mecânica. **Métodos:** Três materiais restauradores foram avaliados e distribuídos em grupos experimentais ($n=10$ espécimes em disco): Cerâmica Reforçada por Leucita, Dissilicato de Lítio e Resina Nanocerâmica. Estes materiais restauradores foram avaliados quanto a superfície antes e após ao envelhecimento ($N=2$ espécimes) em estereomicroscópio. Os espécimes foram submetidos ao ensaio de rugosidade superficial média (R_a) ($N=10$) em rugosímetro de contato antes e após o envelhecimento. Como também, ao teste de resistência à flexão biaxial ($N=10$), após envelhecimento, em uma velocidade de ensaio de $0,5$ mm/min. O envelhecimento foi realizado uma termocicladora, sendo 5.000 ciclos com banhos de $5\text{ °C} \pm 1\text{ °}$ e $55\text{ °C} \pm 1\text{ °}$. Os fragmentos após fratura foram observados em estereomicroscópio. Os dados obtidos foram tabulados e analisados no programa estatístico Minitab. **Resultados:** As superfícies dos espécimes não mostram alterações entre os materiais restauradores e também em relação ao envelhecimento. Para os dados de rugosidade, os fatores materiais restauradores ($p=0,867$) e envelhecimento ($p=0,321$) não foram estatisticamente significativos. O grupo DIS apresentou os maiores valores de resistência à fratura ($p=0,000$), em relação aos grupos LEU e REN que foram estatisticamente semelhantes. O mesmo padrão estatístico foi identificado para os dados de fragmentos após a fratura ($p=0,030$). **Conclusão:** Os fatores envelhecimento e material restaurador não interferem quanto a performance da rugosidade superficial. No entanto, o desempenho mecânico e número de fragmentos após a fratura é afetado pelo material restaurador.

Termos de indexação: Cerâmica. Desenho assistido por computador. Resistência à flexão.

INTRODUCTION

Rehabilitating devitalized posterior elements combining aesthetics, resistance, efficiency, speed and durability is one of the challenges in the daily clinic. Endocrown restorations emerge as an alternative to the classic way of using intraradicular retainers and crowns. Endocrowns are monoblock restorations, which do not require retention pins, reduce wear on the remnant and weakening of the root canal, in addition to reducing clinical time [1]. The study by El Ghoul et al. [2] showed that endocrowns made of lithium, zirconia-reinforced lithium silicate and nanoceramic resin showed higher fracture strength values compared to glass fiber post-supported lithium disilicate crowns.

To make an endocrown, it is necessary to use a resistant material to withstand the masticatory loads incident on the crown. Currently, due to the evolution of restorative materials, it is possible to make a restoration with the ideal characteristics [3]. Several studies have evaluated restoration materials to be indicated for the fabrication of endocrowns [4-15].

Laboratory and clinical studies are extremely important for the evolution of the technique and materials used in these restorations [16]. The mechanical behavior of ceramic materials varies with the structure and mechanical properties. The fracture strength and failure mode of endocrowns can be influenced by several factors, such as material type, occlusal load, aging, cervical termination, restoration design, restoration processing, and the cementation method [17,18]. Thus, further investigation is needed. are needed to explore the biomechanical behavior of recent materials when used to make endocrowns prior to clinical trials [8].

Based on the above, the objective was to evaluate the longevity of ceramic restorative materials in the manufacture of endocrown restorations, through an *in vitro* study on surface roughness and mechanical strength. The expected results for this research, based on the proposed objective, are: Null Hypothesis (H0) - There will be no statistically significant difference for surface roughness and mechanical strength between the materials tested; Alternative Hypothesis 1 (H1) - There will be a statistically significant difference for surface roughness and mechanical strength between the materials tested; Alternative Hypothesis 2 (H2) - There will be a statistically significant difference only for surface roughness and not for mechanical strength between the materials teste.

METHODS

Specimens manufacturim

Circular discs with approximately 12 mm in diameter and 1.2 mm in thickness were obtained from blocks for CAD-CAM of three indirect restorative materials, namely: Leucite-reinforced ceramic (IPS Empress CAD, Ivoclar Vivadent,

Switzerland), Disilicate Lithium (IPS Emax CAD, Ivoclar Vivadent, Switzerland) and Nanoceramic Resin (Lava Ultimate, 3M ESPE, Germany) (table 1).

Table 1. Characteristics of the restorative materials used in the study.*

Restorative Material (manufacturer)	Ceramic Classification	Chemical Composition	Modulus of elasticity
Lithium Disilicate (IPS Emax CAD - Ivocalr Vivadent)	Glass-matrix ceramics	58–80% SiO ₂ 11–19% Li ₂ O 0–13% K ₂ O 0–8% ZrO ₂ 0–5% Al ₂ O ₃	102.7 Gpa
Leucite-reinforced Ceramic (IPS Empress CAD - Ivocalr Vivadent)	Glass-matrix ceramics	60–65% SiO ₂ 16–20% Al ₂ O ₃ 10–14% K ₂ O 3.5–6.5% Na ₂ O 0.5–7% Others oxides 0.2–1% Pigments	65.5 Gpa
Resin Nanoceramic (Ultimate Lava -3M ESPE)	Resin-Matrix Ceramics	80% inorganic 69% SiO ₂ 31% ZrO ₂ 20% organic	12.7 Gpa

Note: *Data obtained in the literature [19,20].

First, slices were cut from the blocks for CAD-CAM in a cutting machine (Struers Accutom 100, Ballerup, Denmark) with a diamond disc at a speed of 250 rpm and water cooling. Then, with a truncated conical diamond tip in a high rotation pen and constant cooling, the slices were rounded to obtain a circular shape. Finally, the specimens were polished with SiC sandpaper of 300, 600 and 1200 grit. According to ISO/CD 6872, the specimens have final dimensions of 12 mm in diameter and 1.2 mm in thickness. The IPS Emax CAD specimens were cut prior to crystallization, which after polishing was performed according to the manufacturer's recommendations regarding oven use and temperature cycles.

Experimental groups

The experimental groups are defined by LEU (Leucite-reinforced ceramic), DIS (Lithium disilicate) and REN (Nanoceramic Resin). The sample value of this study was calculated using the Minitab statistical program (version 17 for windows, Pennsylvania USA), based on the standard deviation (0.668) of the Skalskyi et al. [21] study, which also evaluated the performance of ceramic materials with specimens in disk. Thus, N=10 presented a sampling power of 80.0% in relation to the maximum differences and this value of N will be adopted for both analyses, surface roughness and mechanical strength.

Surface roughness

The specimens were evaluated for mean surface roughness (Ra - μm) using a Taylor Hobson contact roughness meter, which is connected to a computerized unit with a computer program, Tayle Profile Gold. In each specimen, three roughness values were obtained, the readings were performed parallel to each other and in a horizontal direction. Finally, the average of the Ra values was calculated, representing the average roughness value of the specimen.

Aging

Thermal aging was performed through thermal cycling in a thermocycler (Nova Ethics, São Paulo, Brazil). 5,000 cycles were performed with baths of $5\text{ }^{\circ}\text{C} \pm 1^{\circ}$ and $55\text{ }^{\circ}\text{C} \pm 1^{\circ}$, as adopted in endocrown studies [6,8,12,15] The immersion time in each bath was 30 seconds and the transfer time between the two baths took place at 2-second interval.

Biaxial bending resistance

The biaxial flexural strength test was performed on a testing machine (Emic DL-1000, Emic, São José dos Pinhais, PR, Brazil) with a blunt tip of 1.6 mm in diameter. Initially, the specimens were positioned on a metallic circular base with three spheres of 3.2 mm in diameter, forming a plane and equally distant from each other, according to ISO 6872, and received the load applied by the testing machine. During the receipt of the load, the specimen was covered with an adhesive tape to prevent contact with the blunt tip in order to keep the fragments in position [22].

The test was conducted with a speed of 0.5 mm/min and a load cell of 100 kgf. The calculation of biaxial flexural strength (σ) (MPa) of the discs was obtained according to the description of ISO 6872 (Formula 1): where P is the load in kgf, X and Y are parameters related to the elastic properties of the material (Poisson Ratio in Elastic Modulus) and b is the thickness of the specimen at the origin of the fracture in mm. [22] The reference values X and Y were obtained from the study by Wendler et al. [20].

$$\sigma = \frac{(-0,2387P(X-Y))}{b^2}$$

Formula 1: Calculation of biaxial flexural strength

Fracture analysis

Disc fracture characteristics were analyzed with the aid of a stereomicroscope (Discovery V20, CarlZeiss, Germany). After that, specimens were selected to represent each of the experimental groups.

Analysis of results

The results were tabulated and analyzed in Minitab (version 17 for windows, Pennsylvania, USA), with a significance level of 5%. The surface roughness data were submitted to the Anova 2 Factor statistical test ($p < 0.05$), to evaluate the effect of the material and aging. Meanwhile, the Anova 1 Factor statistical test ($p < 0.05$) was adopted to evaluate the effect of the material in relation to mechanical strength and number of fragments after fracture. Fisher's test was adopted to identify differences between groups for the analysis of fracture resistance and number of fragments after fracture.

RESULTS

The research results are presented according to the analyzes performed.

Surface roughness

The observation of the surfaces of the specimens in a stereomicroscope did not show surface changes between the restorative materials, also before and after aging. For the roughness data, the restorative material factors ($p = 0.867$)

and aging ($p=0.321$) were not statistically significant, in addition to showing no interaction between the variables ($p=0.774$). Descriptive data regarding the experimental groups are seen in table 2.

Table 2. Ra (μm) data before and after aging.

Experimental Group (Aging)	Average	Standard deviation	Maximum Ra	Minimum Ra
LEU (no Aging)	0.123860	0.0828657	0.2693	0.0343
LEU (yes Aging)	0.0942333	0.0479892	0.1880	0.0330
DIS (no Aging)	0.123697	0.106841	0.3513	0.0402
DIS (yes Aging)	0.123543	0.113305	0.4060	0.0393
REN (no Aging)	0.134050	0.0841469	0.2937	0.0692
REN (yes Aging)	0.0966633	0.0664320	0.2257	0.0418

Mechanical strength and fracture analysis

The mechanical performance of restorative materials after aging showed a statistically significant difference ($p=0.000$) and differences between experimental groups (table 3). The same statistical pattern was identified for the data on fragments after fracture ($P=0.030$) (table 4). Representative stereomicroscope images of the fractures for the experimental groups are seen in figures 1, 2, 3.

Table 3. Mechanical Strength (Mpa) data after aging.*

Experimental Group	Average	Standard deviation	Maximum value	Minimum value
LEU	200.3 ^B	41.3	118.9	262.9
DIS	681.0 ^A	400.0	100.0	1.216.0
REN	53.19 ^B	24.63	25.87	109.29

Note: *Different letters mean difference between groups.

Table 4. Data on the number of fragments after fracture.*

Experimental Group	Average	Standard deviation	Maximum value	Minimum value
LEU	4,3 ^{AB}	1,494	06	02
DIS	4,5 ^A	1,434	08	03
REN	2,9 ^B	1,197	06	02

Note: *Different letters mean difference between groups.

DISCUSSION

Based on the results of this research, alternative hypothesis 3 (H3) was accepted and the other tested hypotheses were rejected. That is, there was a statistically significant difference only for mechanical strength and not for surface

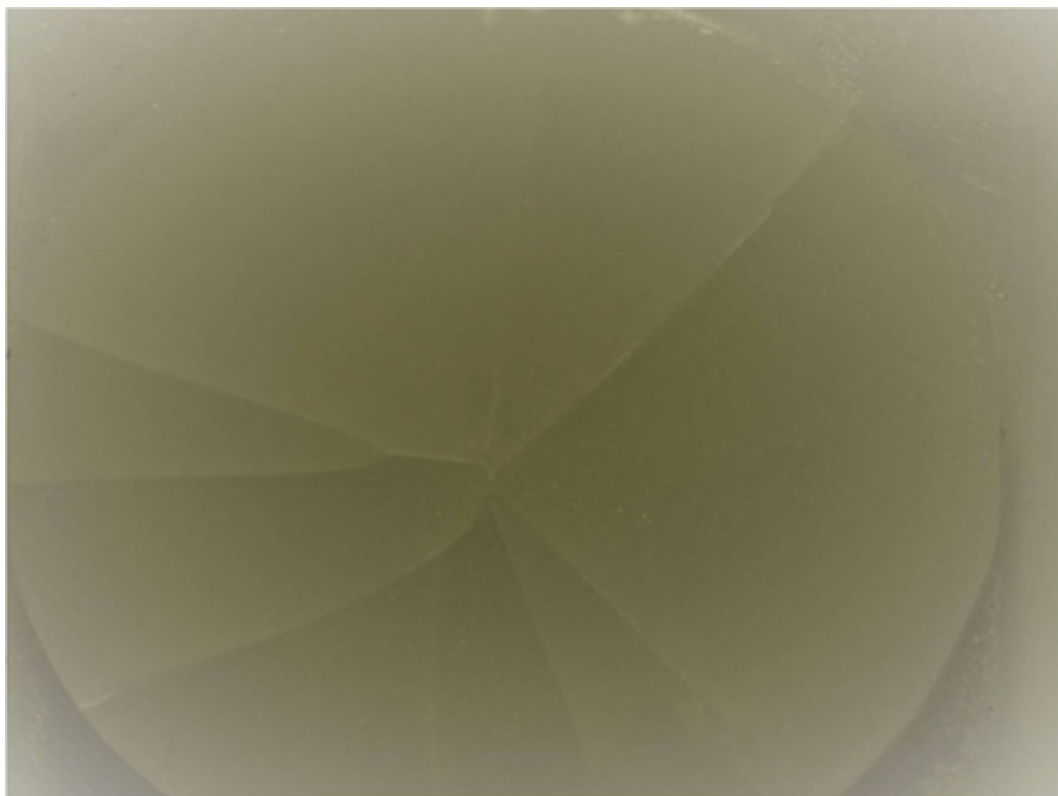


Figure 1. DIS Group - Specimen 1, 07 fragments after fracture. 0.65x magnification.

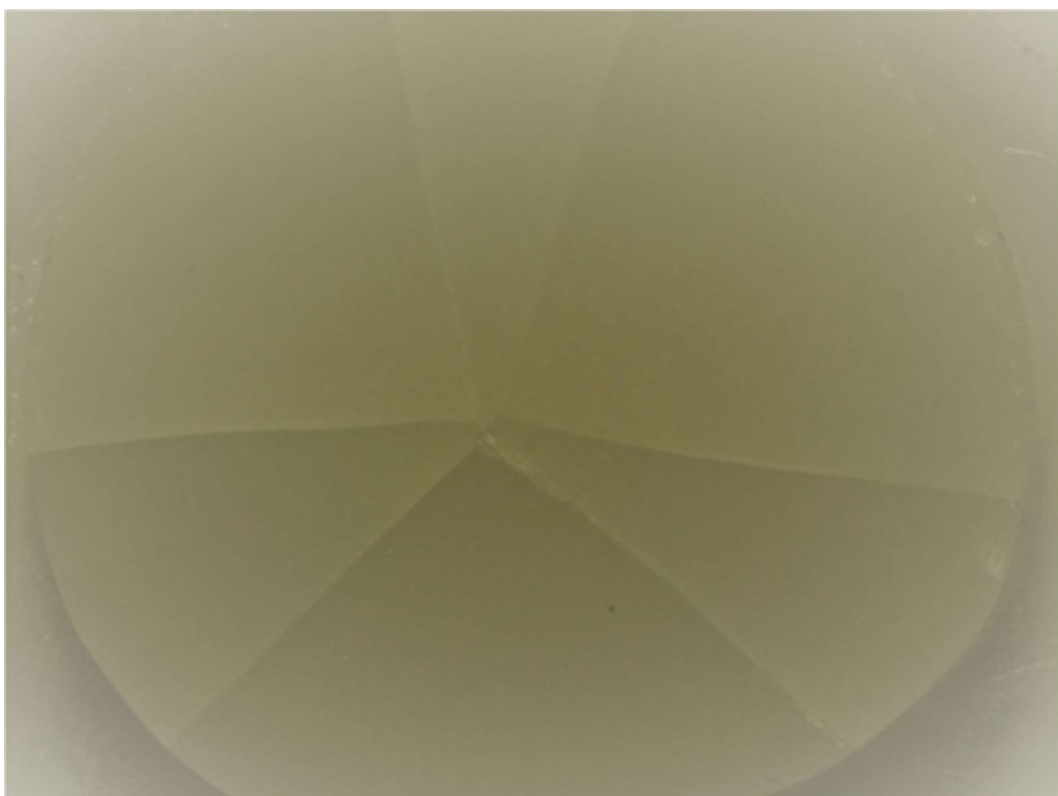


Figure 2. LEU Group- Specimen 2, 06 fragments after fracture. 0.65x magnification.

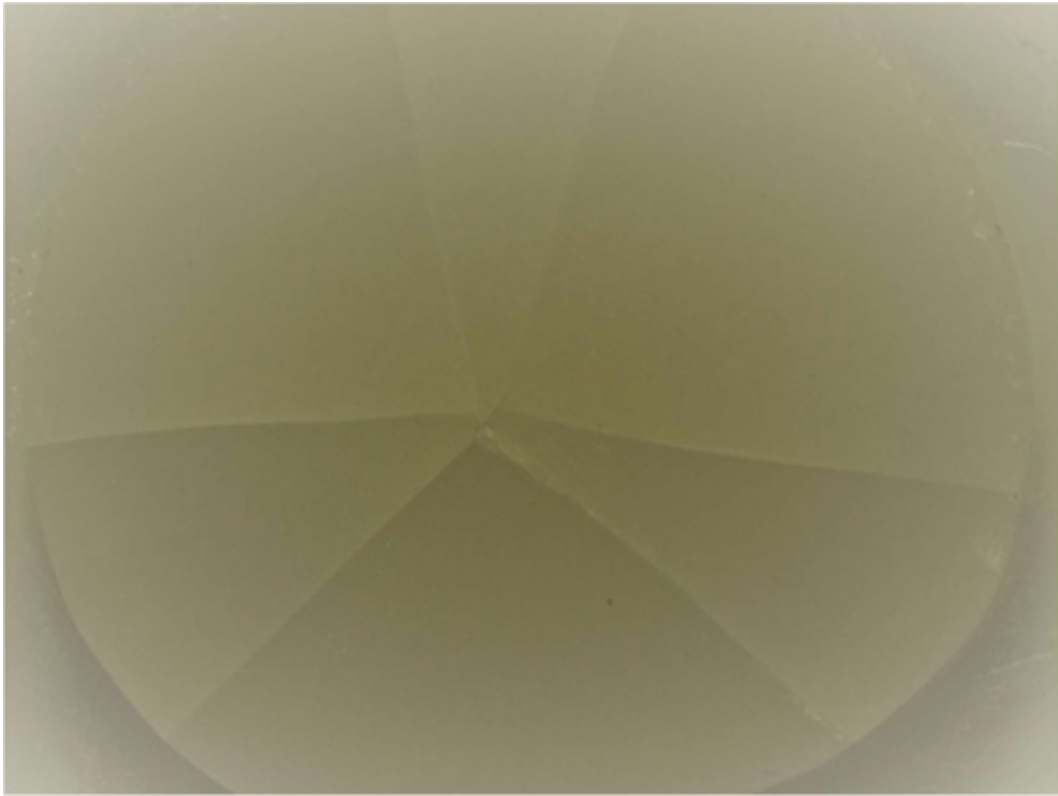


Figure 3. Group REN- Specimen 1, 03 fragments after fracture. 0.65x magnification.

roughness between the materials tested. The findings of this research corroborate the study by El Ghouli et al. [2] and Acar and Kalyoncuoğlu [12] for fracture resistance under endocrown restorations and are in agreement with other published studies [13,15].

Lithium disilicate showed superior mechanical performance compared to Leucite Reinforced Ceramics and Nanoceramic Resin after thermal aging. As observed in the study by El Ghouli et al. [2], for axial load Lithium Disilicate proved to be more resistant to fracture than Lithium Silicate reinforced by Zirconia and similar to Nanoceramic Resin after thermomechanical aging. Acar and Kalyoncuoğlu [12] also observed that Lithium Disilicate was the material that supports greater lateral load until fracture, in relation to hybrid materials such as Nanoceramic Resin. Lithium disilicate has been a suitable material for making endocrown [11], due to its excellent mechanical properties, such as high flexural strength [18,19] and hardness after thermal aging [23].

Articles that disagree with the findings of this research, choose zirconia as the most fracture resistant material. However, this ceramic material had a higher rate of non-restorable failures compared to endocrown restorations [13,15]. Zirconia is a ceramic of the polycrystalline group, which among all-ceramic materials has greater mechanical strength [3]. This characteristic makes this restorative material be limited to endocrown restorations, as it does not favor restorative biomedical principles and promotes catastrophic fractures to the dental structure [24].

Ceramics Reinforced by Leucite and Resin Nanoceramics statistically presented equivalent mechanical performance after thermal aging, which was also observed in the study by Sonmez et al. [24] However, the study by Porto et al. [18] showed that there was mechanical superiority of Ceramics Reinforced by Leucite and aging negatively affected only the Nanoceramic Resin. The same is repeated with Lava Ultimate in a study with endocrown restorations, that is, lower values of fracture resistance after thermomechanical aging, when this material was compared to Hybrid Ceramic, Hybrid Composite and Lithium Disilicate. research in question, are due to the methodology adopted regarding the specimen,

aging and fracture resistance. Making discs for the biaxial bending test is complex, CAM are rectangular. Factor that makes it difficult to standardize specimens [24]. Lava Ultimate has been affected by aging, according to the literature, because the organic matrix undergoes thermal degradation [19]. And different mechanical tests can influence the values of mechanical strength. Therefore, the results that corroborate the article by Sonmez et al. [19] should be close to the number of thermal cycles with the research in question, while Porto et al. [18] have specimens in bar and three-point bending test, finally Acar and Kalyoncuoğlu [12] edocrows restorations and compression test.

As for the surface roughness findings, the restorative materials and thermal aging were not statistically significant. As well, no research was identified that investigated the surface wear of endocrown restorations, which limits the discussion of the results. In a study for polished specimens without aging, it showed that the roughness visibly increased as the surface treatment was more aggressive and this surface change does not influence the flexural strength of Lava Ultimate, VITA ENAMIC, Shofu Block HC and IPS Empress CAD, which in a way it corroborates the research in question [18]. For Ludovichetti et al. [25] who studied the wear resistance and abrasiveness of monolithic materials for CAD-CAM in the absence of aging, Vita Enamic and Lava Ultimate showed the highest surface roughness values, while IPS e.max CAD and Vita Suprinity showed the lowest mean values, these results were statistically significant and are in line with the research presented. In the study by Awada et al. [24], there was a statistically significant difference in the edge of restorations between Nanoceramic Resin and Leucite-Reinforced Ceramic in the absence of aging, with the second material having higher surface roughness. The lack of statistical difference between the results of Ra of the research in question, can be explained by the specimens being evaluated only polished. As for the Nanoceramic Resin not having been superficially affected after thermal aging, Lava Ultimate belongs to the Ceramics with resin matrix group, which is characterized by an organic matrix with inorganic particles on the surface such as SiO₂ and ZrO₂, which may make the surface of this material more resistant to thermal action [3].

Regarding the number of fragments after fracture, the Leucite-Reinforced Ceramic was statistically similar to Lithium Disilicate and Nanoceramic Resin. IPS Emax CAD being the material that obtained the highest number of fragments after fracture. Lithium disilicate endocrown restorations had a higher frequency of irreparable failures [2,12,13,15]. Leucite-reinforced Nanoceramic and Ceramic Resins showed a more repairable fracture scenario [3,12,13]. The unfavorable results of Lithium Disilicate are due to the high modulus of elasticity, thus promoting greater fracture load and tensions in the dental structure [2]. While the Nanoceramic Resin has a modulus of elasticity that approximates the dental structure, favoring the indication of indirect restorations according to biomimetic perspectives [24]. The greater number of fragments after Lithium Disilicate fracture is due to the material presenting greater mechanical strength, thus absorbing a greater amount of energy until fracture [20,22]. The nanoceramic resin, due to its lower mechanical performance, supports lower stresses until fracture. Finally, Leucite Reinforced Ceramics proved to be an intermediate material, perhaps due to its chemical composition not having oxides that would guarantee greater mechanical strength.

If it were possible to extrapolate the data from this research to the daily clinic, which is not because it is an in vitro study. Leucite Reinforced Ceramics would be a restorative material, for use in CAD-CAM, with a favorable indication for the fabrication of an endocrown restoration. Because this material, showed resistance to fracture and fracture fragments in intermediate values to Lithium Disilicate and Nanoceramic Resin. In addition to the excellent aesthetic performance and scientific knowledge that this ceramic has in the dental environment, it is a more predictable material for single-unit restorations.

The limitation of this research is due to the non-use of specimens that represent the endocrown restoration, making the comparison between the results more difficult. The complexity in the standardization of disk specimens, especially Lithium Disilicate for being more resistant. Not having performed mechanical aging, which would be important to bring the findings closer to the clinical condition. Having an experimental group with zirconia, since this restorative material is evaluated in most studies on endocrown restorations. New studies should look at the surface roughness after thermomechanical aging of restorative materials for CAD-CAM, research on the wear of endocrown restoration and its antagonist is necessary in the long term so that it is possible to understand mechanical behavior. Finally, randomized clinical studies that evaluate the performance of restorative materials in patients over the years with several variables under observation.

CONCLUSION

Based on the limitations of this study, the following conclusions were presented:

1. The aging factors and restorative material do not interfere with the surface roughness performance;
2. Mechanical performance and number of fragments after fracture are affected by the restorative material.

Collaborators

CML Silva carried out the literature search, made the specimens, carried out the tests, wrote the article. AGCM Raimundo, TFS Souza and AJ Torres Neto made the specimens. MTV Grangeiro came up with the idea for the article, carried out the aging. VMG de Figueiredo came up with the idea for the article, carried out the literature search, analysis of data, wrote the article.

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