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Effect of different restorative materials for use in CAD-CAM in biaxial bending resistance after accelerated aging

Efeito de diferentes materiais restauradores para uso em CAD-CAM na resistência à flexão biaxial após envelhecimento acelerado

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ABSTRACT

Objective: The aim of this study was to evaluate the effect of different restorative materials for use in CAD-CAM on biaxial flexural strength after accelerated mechanical and thermal aging. **Methods**: Samples were fabricated and divided into two groups: CL (leucite-reinforced ceramic: IPS Empress CAD) and NR (nanoceramic resin: Lava Ultimate). Morphological analysis of the surface was performed using Scanning

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Electron Microscopy, and chemical analysis was conducted using Energy Dispersive X-ray Spectroscopy on one sample from each group. All samples underwent accelerated aging, initially through fatigue testing using a mechanical cyclic loading machine (200,000 cycles, 15N force, 1Hz rotation speed) in distilled water at room temperature (37°C). Subsequently, thermal aging was carried out in a thermocycler (5,000 cycles, baths at 5°C and 55°C). **Results**: The CL group exhibited a more diffuse surface, and chemically, it showed the presence of Silicon, Oxygen, Aluminum, Sodium, Potassium, and Calcium. The NR group had a more compact surface and contained the following chemical elements: Oxygen, Silicon, Carbon, Zirconium, Nitrogen, Aluminum, and Sodium. The CL (193.1MPa) showed a higher mechanical strength value than the NR (52.45MPa), which was statistically significant. However, the NR (2.3) exhibited a lower average number of fragments after fracture compared to the CL (5.6). **Conclusion**: Restorative materials exhibited distinct morphology and chemical composition. These restorative materials had a statistically significant effect on biaxial flexural strength, with leucite-reinforced ceramics being mechanically superior to nanoceramic resin. And the ceramic matrix resin family showed a lower quantity of fragments after fracture compared to the material representing the glass-ceramic family.

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

RESUMO

Objetivo: Avaliar o efeito de diferentes materiais restauradores para uso em CAD-CAM na resistência à flexão biaxial após envelhecimento mecânico e térmico acelerado. **Métodos**: Foram fabricadas amostras (n=10) por grupo: CL (cerâmica reforçada com leucita: IPS Empress CAD) e NR (resina nanocerâmica: Lava Ultimate). A análise morfológica da superfície foi realizada por Microscopia Eletrônica de Varredura, e a análise química foi realizada por Espectroscopia de Raios X por Dispersão de Energia em uma amostra de cada grupo. Todas as amostras foram submetidas a envelhecimento acelerado, inicialmente através de ensaio de fadiga utilizando máquina mecânica de carregamento cíclico (200.000 ciclos, força 15N, 1Hz) em água destilada à temperatura (37°C). O envelhecimento térmico foi realizado em termociclador (5.000 ciclos, banhos de 5°C-55°C). **Resultados**: A análise estatística 1 Fator Anova ($p< 0{,}05$) O grupo CL apresentou superfície mais difusa e quimicamente apresentou presença de Silício, Oxigênio, Alumínio, Sódio, Potássio e Cálcio. O grupo NR apresentava superfície mais compacta e continha os seguintes elementos químicos: Oxigênio, Silício, Carbono, Zircônio, Nitrogênio, Alumínio e Sódio. O CL (193,1MPa) apresentou valor de resistência mecânica superior ao NR (52,45MPa), o que foi estatisticamente significativo ($p=0{,}000$). Porém, o NR (2,3) apresentou menor média de fragmentos após fratura em relação ao CL (5,6). **Conclusão**: Os materiais restauradores exibiram morfologia e composição química distintas. Esses materiais restauradores tiveram um efeito estatisticamente significativo na resistência à flexão biaxial, sendo a cerâmica reforçada com leucita mecanicamente superior à resina nanocerâmica. E a família de resinas de matriz cerâmica apresentou menor quantidade de fragmentos após fratura em comparação ao material representativo da família vitrocerâmica.

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

INTRODUCTION

The digital flow in dentistry adopted in recent decades has provided the ease of execution of clinical and laboratory procedures, expanded use of new materials for CAD-CAM (*computer-aided design - computer-aided manufacturing*) and impacted scientific development [1,2]. With the Covid-19 Pandemic, digital dentistry was only reaffirmed in the restorative clinical scenario through measures implemented in dental practices to reduce cross-contamination in the dental team and with the prosthodontic laboratory, such as the use of digital impression taking, expanding the indication of monolithic materials for CAD-CAM, and applying digital manufacturing for making indirect restorations [3,4]. However, achieving the natural appearance of the tooth with sufficient mechanical strength is one of the most challenging issues of computer-aided design and fabrication materials in CAD-CAM. As well, the available evidence is limited on their optical and mechanical properties for the selection of the appropriate restorative material by the dentist [5].

The expansion of digital dentistry has promoted the development of new restorative materials for use in CAD-CAM, thus Gracis [6] presented a new classification for ceramic materials into ceramics with glass matrix (vitroceramics), polycrystalline ceramics and ceramics with resin matrix. The last one presents itself with a modulus of elasticity that approaches dentin, easier to milling and repair in comparison to polycrystalline and vitroceramic materials. Nanoceramic resins are a CAD-CAM material of the ceramic with resinous matrix family, which has a resin matrix with 80% nanoceramic particles, and has shown ample clinical indication for crowns and partial restorations [6-8]. This restorative material was developed to overcome the adverse properties of ceramics with glass matrix and composite resins [9]. Therefore, nanoceramic resin presents itself as a good alternative material for CAD-CAM, because it combines the benefits of glass resins and ceramics such as low wear of the simulated opposing dentition, favoring it to be adopted as a material for bruxist patients [7,8,10]. The recent development of these restorative materials for use in CAD-CAM and the reduced availability of studies on such materials, makes it pertinent to evaluate their properties, identify potentials and constraints for clinical application [8].

Studies that compared nanoceramic resins with other clinically consolidated ceramic materials, such as reinforced glass-matrix ceramics, show data limitations regarding mechanical aging [9,11,12]. Chemical and mechanical degradation play a key role in the durability of dental restorative materials. Therefore, the prediction of their long-term performance in the oral environment should also be based on fatigue [13]. There are few studies in the literature evaluating the mechanical and thermal aging of these materials, which makes it impossible to extrapolate data to daily clinical practice, as the parameters have not yet been established. Based on the above, this study aimed to evaluate the effect of different restorative materials for use in CAD-CAM on the biaxial flexural strength after accelerated mechanical and thermal aging. The expected results for this research, based on the proposed objective, are: Null Hypothesis (H0): There will be no statistically significant differences between nanoceramic resin and leucite reinforced ceramics in mechanical fracture resistance; Alternative Hypothesis 1 (H1): There will be statistically significant differences between nanoceramic resin and leucite reinforced ceramics in mechanical fracture resistance.

METHODS

Fabrication of specimens

Disc-shaped specimens (n=10) were obtained using two indirect restorative materials on CAD-CAM blocks, leucite reinforced ceramics (IPS Empress CAD, Ivoclar Vivadent, Switzerland) and nanoceramic resin (Lava Ultimate, 3M ESPE, Germany). The CAD-CAM blocks were sectioned in a cutting machine (Struers Accutom 100, Ballerup, Denmark), with 1.3 mm thick dimensions, with a diamond disk at a speed of 250 rpm and water cooling. Next, the fragments of the restorative materials were rounded into 12-mm discs using a diamond bur and high-spin handpiece with water cooling. Subsequently, polishing of the samples with Silicon Carbide (SiC) sandpapers of 600 and 1200 grain size was performed. According to ISO/CD 6872, the specimens had final dimensions of 12 mm in diameter and 1.2 mm in thickness.

The experimental groups are defined by CL (leucite-reinforced ceramics - n=10 specimens) and NR (nanoceramic resin - n=10 specimens). The sample value of this study was calculated with the aid of the statistical program Minitab (version 17 for windows, Pennsylvania USA), based on the standard deviation of similar research described by Porto [11] for flexural strength, thus n=10 presented a sample power of 80.0% in relation to maximum differences.

Surface analysis

One specimen of each material was analyzed for surface morphology and chemical characterization. As for morphology, the specimens by Scanning Electron Microscopy (SEM) (TESCAN MIRA3 FEG, Australia) using magnification of 5,000X and 15,000X. The chemical characterization and surface mapping by X-ray dispersive spectroscopy (Electron Dispersive Spectroscopy - EDS) method (Oxford Instruments, X act, UK). The EDS system with AZtec program, has an EDS detector coupled to the SEM. Spot readings and mapping were performed on a single area of the specimen. The elemental concentration was determined after averaging the weight percentages of the chemical elements at each point. The surface mapping images were generated by the software itself to locate the highest concentration of each chemical element.

Aging

Specimens from both experimental groups were subjected to accelerated aging, initially to mechanical fatigue in a mechanical cycler (Biopidi, São Paulo, Brazil) at 200,000 cycles with a force of 15 N at a rotation speed of 1 Hz in distilled water at ambient temperature (37°C) [10]. Then, the specimens were subjected to thermal aging in a thermocycler (Nova Ética, São Paulo, Brazil) for 5,000 cycles with baths of 5°C ± 1° and 55°C ± 1°. The immersion time in each bath was 30 seconds and the transfer time between the two baths was 2 seconds [12].

Biaxial flexural strength

For the biaxial flexural strength test, the specimens remained positioned on a circular metal base with three 3.2 mm diameter spheres in distilled water, equidistant from each other, forming a plane (ISO 6872). A 1.6-mm diameter blunt tip was attached to a testing machine (Emic DL-1000, Emic, São José dos Pinhais, PR, Brazil), and the load was applied. During the biaxial flexion test, the specimen was covered with a tape on the compression side in order to prevent contact with the load application tip from producing defects and to keep the fragments in position. The test was conducted with a speed of 0.5 mm/min and a load cell of 100 Kgf.

The calculation of the biaxial flexural strength (σ) (MPa) of the discs was obtained according to the description of the ISO 6872 standard (Formula 1): where P is the load in kgf, X and Y are parameters related to the elastic properties of the material (Poisson’s Ratio in Elastic Modulus) and b is the specimen thickness at the origin of fracture in mm. The article by Wendler14 was adopted for the reference values X and Y.

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

Calculation of the biaxial flexural strength.

Fracture analysis

The fractured specimens were analyzed under a stereomicroscope (Discovery V20, CarlZeiss, Germany) to determine the fracture characteristics.

Analysis of results

The results obtained were tabulated and analyzed in Minitab statistical software (version 17 for windows, Pennsylvania, USA), with a significance level of 5%. The biaxial flexural strength data were submitted to the 1 Factor Anova statistical test ($p< 0.05$), to evaluate the effect of the material. Previously, the Normality Test Komolgorov Smirnov Test was applied to the data and showed a significance level higher than 1% between the experimental groups. The data obtained by surface analysis and fractography were evaluated qualitatively.

RESULTS

The surfaces of the ceramic materials (figure 1), according to morphological and chemical analysis, were different from each other. The leucite-reinforced ceramics were similar to the glass-ceramic family in having a more diffuse surface, and chemically the presence of Silicon (Si) in larger quantities, followed

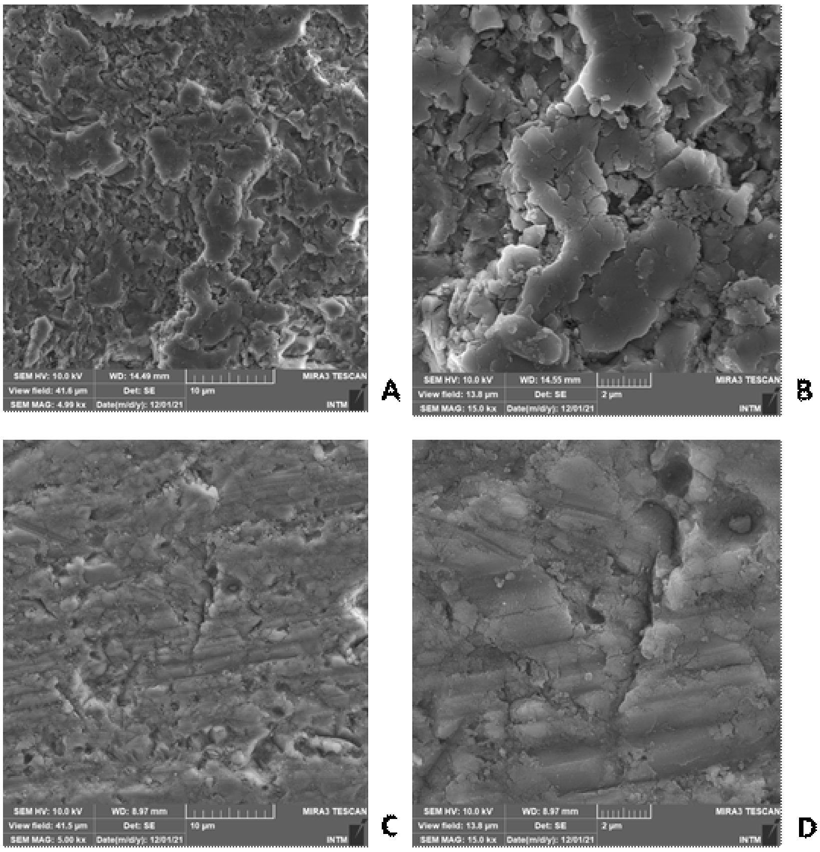

Figure 1. SEM image of the surface of the leucite-reinforced ceramic, magnification 5,000X (A) and 15000X (B). SEM image of the nanoceramic resin surface, magnification 5,000X (C) and 15000X (D).

by Oxygen (O), Aluminum (Al), Sodium (Na), Potassium (K) and Calcium (Ca) (figure 2). Whereas, the nanoceramic resin, from the family of ceramics with resin matrix, shows a more compact surface, and the presence of the following chemical elements Oxygen (O), Silicon (Si), Carbon (C), Zirconium (Zr), Nitrogen (N), Aluminum (Al) and Sodium (Na) (figure 3).

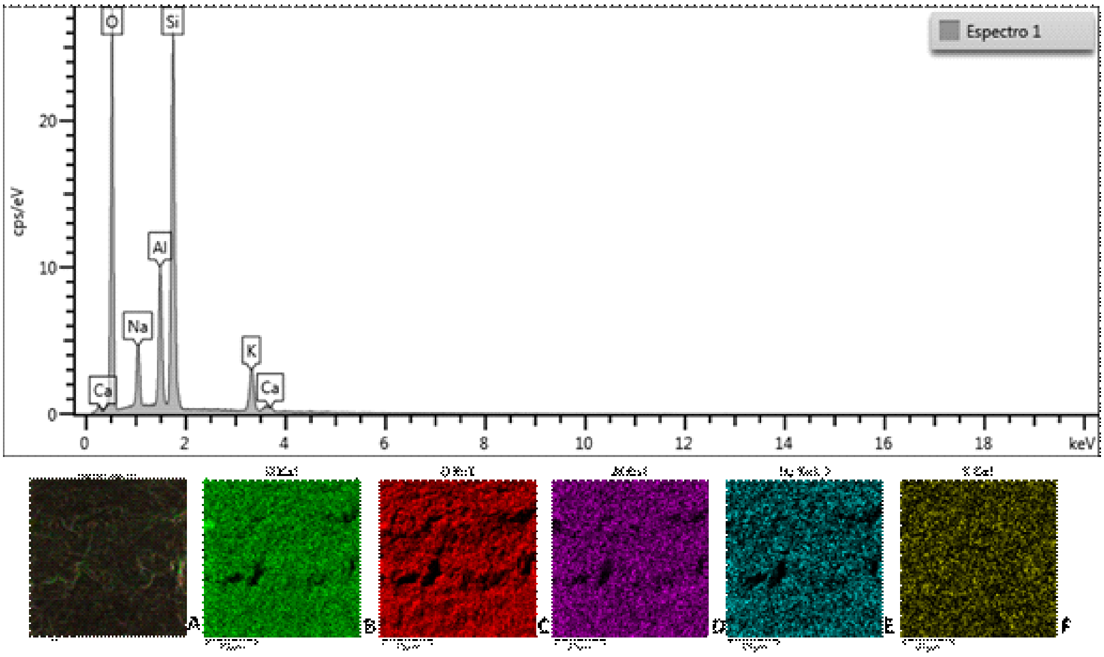

Figure 2. Identification and mapping of chemical elements present on the surface of the leucite-reinforced ceramic.

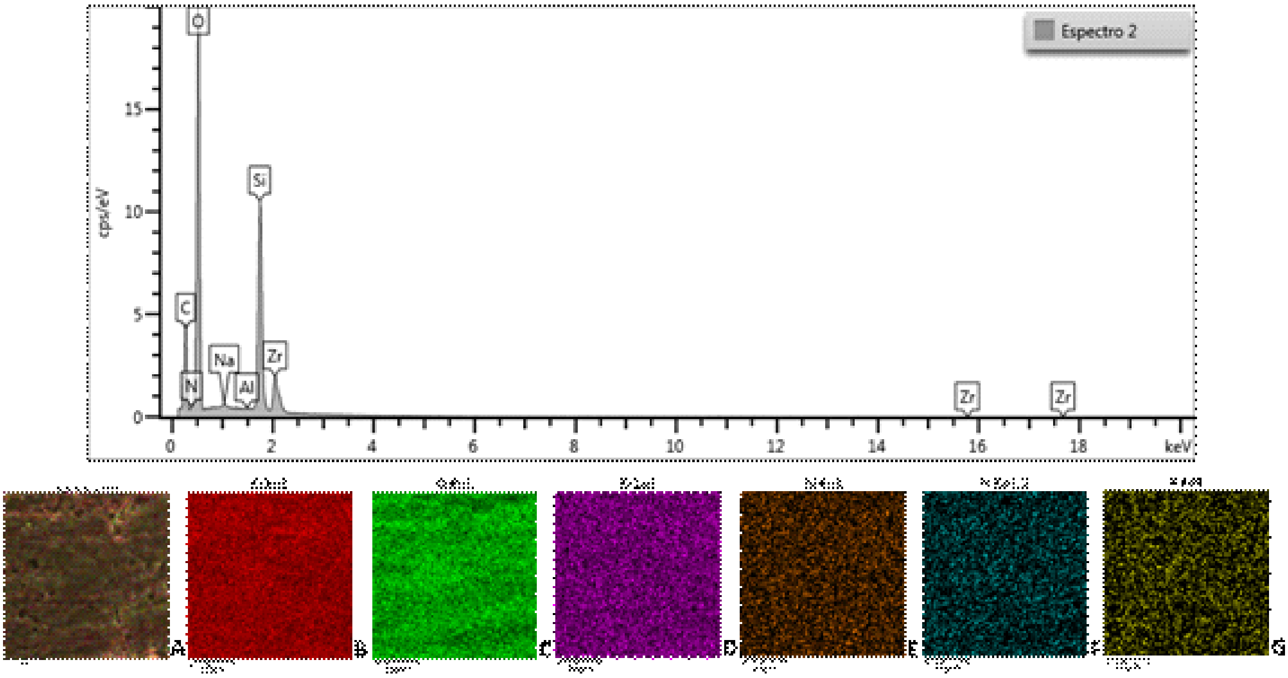

Figure 3. Identification and mapping of chemical elements present on the surface of the nanoceramic resin.

After accelerated, mechanical and thermal aging, all samples survived. The CL group showed higher mean values for mechanical strength than the RN group, and this condition was statistically significant (p=0.000). Regarding the number of fragments after fracture, the RN group showed a lower average compared to the CL group (table 1) (figure 4).

Table 1. Descriptive statistical data of the survey.

Experimental group	Average mechanical strength (Mpa)	Standard Deviation	Minimum strength value (Mpa)	Maximum strength value (Mpa)	Average number of fragments after fracture
CL	193.1	54.3	155.1	340.3	5.6
RN	52.45	7.17	40.02	64.54	2.3

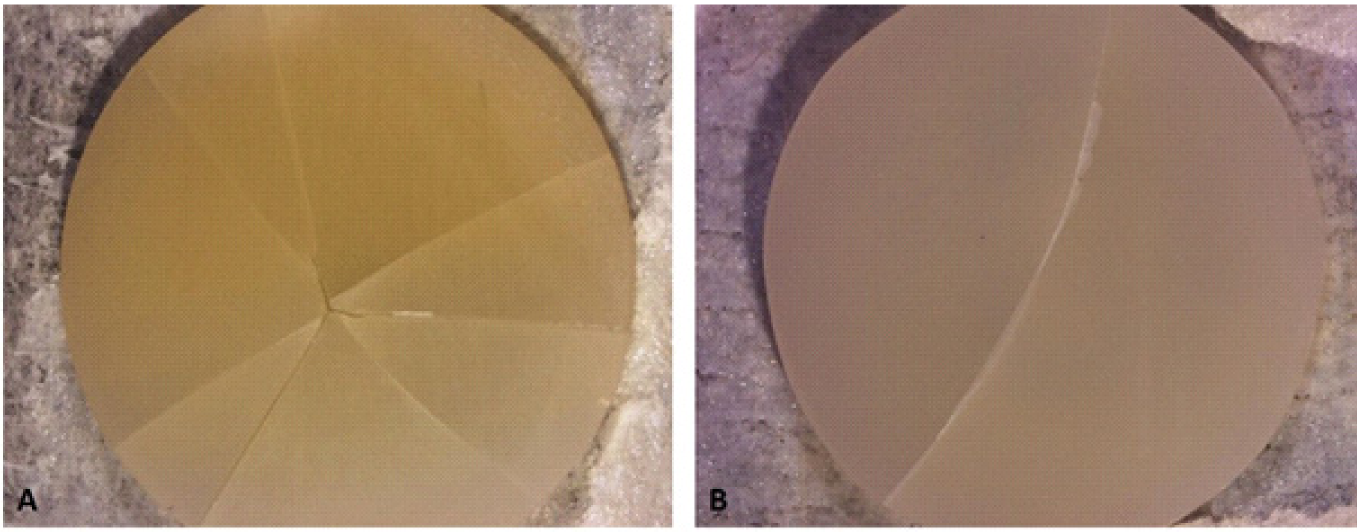

Figure 4. A: Image of the CL group specimen after the fracture strength test under a stereomicroscope, magnification 0.65X; B: Image of the NR group specimen after the fracture strength test under a stereomicroscope, magnification 0.65X.

DISCUSSION

According to the results presented by the statistical analysis of this study, the alternative hypothesis (H1) was accepted and the null hypothesis (H0) was rejected. That is, there was a statistically significant difference between the materials under study, the nanoceramic resin and the leucite-reinforced ceramic, in the mechanical fracture strength after accelerated aging.

The leucite-reinforced ceramic (IPS Empess CAD) showed higher mechanical strength values than the nanoceramic resin (Lava Ultimate) after accelerated aging. Similar research has found that thermal aging significantly reduces the mechanical strength values of nanoceramic resin only, but no statistical difference was found when evaluating the strength values between the already thermocycled materials, and leucite-reinforced ceramics did not change their mechanical performance after aging [9,11,12]. Other research findings contradicted these, highlighting the better mechanical performance of the nanoceramic resin [7,8,18].

It can be seen that the simulated aging process affects the ceramic materials with resin matrix. Thermocycling can cause the assimilation of water into the resin structure, promoting the widening of the network and reducing the strength of the polymeric chains, while water absorption is not observed in vitroceramics. Scanning electron microscopy (SEM) images support these findings, as the surface of Lava Ultimate shows deterioration and the presence of some micro-cracks after thermocycling, while there are no noticeable differences in SEM images before and after thermocycling for IPS Empress CAD [9]. The presence of the micro-cracks on the Lava Ultimate after thermocycling favor the breakdown of cohesion and the reduction of strength due to hydrolysis of the silane coupling agent [15]. When the microstructure is not well controlled and the reinforcement particles are not well dispersed or not well bonded to the material matrix, the reinforcement particles act as a limiting factor on tenacity [15].

Artificial aging affects materials with CAD/CAM use differently, as some materials tested are susceptible to aging. As observed, Martens hardness and indentation modulus decreased after thermocycling for Lava Ultimate, due to mechanically induced microcracks or increased surface roughness [16]. The fracture toughness of resin-bonded materials is also negatively affected, whereas leucite-reinforced ceramics and lithium disilicate were stable [15]. The decrease in fracture strength values can be translated by the definition of modulus of resilience, which is the ability of materials to absorb energy while being elastically deformed. The absorbed energy must be released during loading [11].

Research that presented contrary results may be due to the type of specimen, for example crowns or bars, which present a different mechanical test conduction than the research in question, corroborating with literature reports [5,7,8,18]. As well as, research that does not evaluate the long-term mechanical performance of specimens [8]. When Lava Ultimate was mechanically tested in the absence of aging, the flexural strength values were statistically superior to vitroceramics [7,8]. Perhaps due to the organic content absorbing the forces from chewing and thus increasing the flexural strength; possibly suggesting a hardening mechanism created by the resin matrix in the microstructure [5,9].

Mechanical fatigue has been adopted mainly to evaluate the wear of the restorative material for CAD/CAM and its antagonist [7,17-20]. Ultrathin occlusal veneers of Lava Ultimate showed as little cracking as IPS e.max CAD and were therefore superior to IPS Empress CAD in terms of fatigue loading, the viscoelastic properties of the composite material should be further investigated [20]. Lava Ultimate has shown an affinity for the antagonist, promoting lower wear ratios and adequate wear resistance to withstand the loads on the restorations, while more rigid materials have been shown to be more abrasive [7,10,17]. However, the roughness and friction coefficient of some materials may change during the wear process, as Lava Ultimate showed the highest values of roughness [10].

The SEM images, which were only taken prior to aging, are in accordance with the reports in the literature, homogeneous surfaces with characteristic of glass-ceramics for the leucite-reinforced ceramic and the presence of organic components between the inorganic surface for the nanoceramic resin [5,9,17]. Lava Ultimate has a resin matrix and ceramic loading structure that includes zirconia nanoparticles and silica [5,19]. IPS Empress CAD, on the other hand, has high proportions of SiO2, Al2O3 and other metal oxides [9]. The EDS results of the present study corroborate the findings in the literature, Zirconium peaks were also identified during the EDS analysis of the resin matrix ceramics, but to a lesser extent than the Silicon and Carbon peaks [5,9]. In other words, the ceramic matrix is composed of elements from feldspathic ceramics (Si, O, Al, Na, K) and the polymer is composed primarily of C and O [17]. However, the possible formation of ZrO2 did not promote maintenance of mechanical strength and hardness a for Lava Ultimate before and after aging in the study by Sonmez et al. [9] nor superior mechanical performance to IPS Empress CAD in this research.

The higher number of fragments after fracture of IPS Empress CAD in relation to Lava Ultimate is due to the fact that ceramic materials with a resin matrix are more flexible, so they tend to break less by absorbing stresses during loading. In contrast, vitroceramic materials exhibit relatively high flexural strength and flexural modulus, this combination translates into a lower capacity to undergo deformation to absorb the stress of increased loading. This difference in elastic properties between polymer-based materials and ceramic materials can be attributed to the resin component, which helps to reduce fragility [8]. Another report shows, that the number of fragments the disc fractures for the biaxial strength test correlates with the strength of the material. That is, the more fragments generated after fracture means that more energy was required for fracture. Investigating the fracture of a restorative material is as important as defining its mechanical properties and there are few studies that perform fractographic analysis [11].

Due to the reduced dimensions of CAD-CAM blocks, ISO 6872 has been the international standard adopted for biaxial flexural strength testing of ceramics, through modification of ISO 687230. The application of the protocol for the biaxial flexural test requires the preparation of disk-shaped specimens, thus it is a challenge because the blocks are rectangular and need to be made into cylinders with a diameter of no less than 12 mm to be tested on the standard 10 mm support extension [8]. The biaxial flexural test is one of the main methods used to investigate the fracture strength and long-term clinical performance of dental materials before they can be It is widely adopted in the literature [5,8]. Also, aging by thermocycling reflects the clinical situation better than isothermal storage and is widely used to simulate the long-term clinical behavior of a restorative material. So much so that temperatures of 5-55 °C are considered the closest to the physiological situation [15].

Should Lava Ultimate be adopted as an indirect restorative material in daily practice? Based on the results of this research, even though it is an in vitro study, there is no recommendation for extrapolation of the data to clinical practice. The disk format of the specimens adopted in the research is restricted to extend the indication of a restorative material. It is known that the properties of materials can change according to their geometry and analysis adopted [8,11]. These differences should be taken into consideration when selecting a restorative material for prosthetic treatments [16]. The dentist should consider the mechanical performance of the CAD-CAM material when deciding on the treatment plan for clinical situations. Research studies investigating the long-term use of Lava Ultimate are still limited [7,9,11,12,15-20]. Considering that surface changes and water absorption are not the only factors that age restorative materials, therefore studies that evaluate ceramics with resin matrix to simulate the clinical situation are recommended [9]. Therefore, more research on the optical and mechanical properties of monolithic restorative materials is needed, especially by simulating the variables of the intraoral environment to make definitive clinical recommendations. In vivo studies that evaluate the clinical complications, biocompatibility, wear, microleakage, color stability and survival rate are essential to validate the use of a material [5,7]. The action of polishing on these CAD/CAM materials should also be investigated, since it is a factor that may interfere with clinical performance, since polishing reduces surface roughness and consequently the fracture of the restoration [19].

The limitation of this research is due to using geometric specimens, restricting the reproduction of the characteristics of an indirect restoration. Only two families of ceramic materials were addressed and the origin of the fracture between the specimens was not evaluated. Therefore, new studies should be proposed to conduct research that compares the ceramic families; vitroceramic, polycrystalline and resin matrix ceramics, adopting specimens that represent the clinical reality and the origin of the fracture is to be investigated. In vitro studies are needed to reveal the long-term performance of resin matrix ceramics. Finally, clinical studies with a high degree of scientific evidence should be possible to extend the indications of these ceramic materials into daily practice.

CONCLUSIONS

Within the limitations of this study, the following conclusions were presented:

1. Restorative materials for use in CAD-CAM, IPS Empress CAD and Lava Ultimate, exhibit distinct morphology and chemical composition.

2. After accelerated mechanical and thermal aging, the restorative materials show a statistically significant effect on biaxial flexural strength, with leucite-reinforced ceramic being mechanically superior than nanoceramic resin.

3. The restorative material representing the ceramic family with resin matrix showed less fragment amount after fracture than the material representing the vitroceramic family.

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Collaborators

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