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CHARACTERIZATION OF SLA-PRINTED CERAMIC COMPOSITES FOR DENTAL RESTORATIONS

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This study introduces a novel ceramic-composite resin specifically developed for stereolithography (SLA) 3D printing, aimed at enhancing dental restorations. The integration of advanced digital technologies in dentistry has shifted traditional methods towards more precise and efficient techniques such as computer-aided design/computer-aided manufacturing (CAD/CAM). However, these processes typically involve material waste due to their subtractive nature. Additive manufacturing, or 3D printing, particularly SLA, which uses ultraviolet light to cure photosensitive resins, presents a viable alternative with the potential for creating detailed, custom restorations with minimal waste. Our research focuses on formulating and evaluating a ceramic-composite resin that combines the benefits of light-cured materials with the mechanical robustness required for dental applications. We conducted comprehensive tests to assess the printability, mechanical properties and wear resistance of the developed material. The ceramic-composite resin demonstrated a tensile strength of approximately 73 MPa, significantly higher than the 42 MPa observed for traditional photopolymer resins. Additionally, the ceramic-composite resin showed ability to resist incidental friction and wear. This research could significantly impact the dental prosthetics field by providing a method for producing high-performance, patient-specific restorations efficiently and cost-effectively.

Keywords: dental restorations, additive manufacturing, stereolithography, ceramic composite, polymer

1. Introduction

The dental industry has witnessed a significant paradigm shift in recent years, with one of the most groundbreaking advancements being the adoption of 3D printing technology [1]. This cutting-edge method of fabricating dental restorations, such as dental implants, full dentures and partial dentures, is revolutionizing the practice of dentistry and dramatically improving patient outcomes [2]. Traditional dental restoration methods, such as porcelain casting [3], have served well for decades. Still, they are often labour-intensive, prone to human error, and limited in their ability to produce complex geometries [4].

The integration of advanced digital technologies in dentistry has shifted traditional methods towards more precise and efficient techniques such as computer-aided design/computer-aided manufacturing (CAD/CAM) [5]. However, these processes typically involve material waste due to their subtractive nature. Additive manufacturing (AM), particularly stereolithography (SLA), which uses ultraviolet light to cure photosensitive resins, presents a viable alternative with the potential for creating detailed, custom restorations with minimal waste [6].

Recent studies have demonstrated the potential of polymer 3D printing in dentistry, highlighting its ability to produce highly accurate and detailed dental restorations [7, 8]. The use of 3D printing technology has also been shown to reduce treatment time and costs, as well as promote a more patient-centric approach to dental care [9]. The study by

Chun et al. [10] compared the mechanical properties of dental restorative materials with those of enamel and dentin, highlighting the importance of understanding the mechanical properties of materials used in dentistry. Another study evaluated the mechanical properties of 3D-printed dental restorations, demonstrating their potential for use in clinical settings [11]. Polymeric materials are currently the most common option for 3D printing in dentistry, used for manufacturing of fixed and removable dentures, crowns, bridges, surgical guides, and custom trays [12].

This advancement in polymer-based 3D printing has paved the way for further research and development of novel materials, such as ceramic-composite resins, which represent a significant advancement in the field of 3D printing materials [13]. These innovative materials blend the strength and durability of ceramics with the flexibility and ease of processing of resins, resulting in products that exhibit enhanced mechanical properties and exceptional wear resistance [14, 15]. The incorporation of ceramic particles into the resin matrix enhances the overall strength and toughness of the printed objects, making them ideal for applications where high performance and longevity are crucial.

Several studies have focused on the development and application of ceramic-composite resins in dental restorations. For example, Ji et al. [16] explored the properties of multicolour 3D-printed 3Y-ZrO_2 sintered bodies by optimizing rheological properties of UV-curable high-content ceramic nanocomposites, finding that these materials exhibit superior mechanical properties and biocompatibility compared to traditional photopolymers. Another study by Mondal et al. [17] investigated the mSLA-based 3D printing of acrylated epoxidized soybean oil-nano-hydroxyapatite composites for bone repair, demonstrating that ceramic-composite resins could significantly reduce the wear rates of dental restorations, thereby extending their lifespan and improving patient satisfaction.

Moreover, advancements in SLA technology have further enhanced the applicability of ceramic composites in dental applications. Recent innovations in SLA printers, such as higher resolution and faster printing speeds, have enabled the production of more precise and intricate dental restorations. Additionally, the development of new photosensitive ceramic-composite resins that cure more efficiently under UV light has improved the mechanical properties and wear resistance of 3D-printed dental restorations.

Ceramic composites are particularly suited for dental restorations due to their unique combination of properties. Unlike traditional dental materials, ceramic composites offer a high compressive strength and fracture toughness, which are essential for withstanding the mechanical forces exerted during chewing and biting. Furthermore, ceramic composites exhibit an excellent biocompatibility, reducing the risk of adverse reactions in patients. Their superior wear resistance ensures that dental restorations remain functional and aesthetically pleasing for longer periods, minimising the need for frequent replacements.

This study aims to characterize the ceramic-composite resin specifically developed for SLA 3D printing and compare it with the traditional photopolymer, aimed at enhancing dental restorations. The resin combines the benefits of light-cured materials with the mechanical robustness required for dental applications. The printability, mechanical properties and wear resistance of the selected material is comprehensively tested to assess its suitability for dental restoration.

2. Materials and methods

2.1. Printing materials

The ceramic-composite resin Liqcreate Composite-X is an extremely rigid and high-performance reinforced nano-micro composite resin for SLA, DLP (Digital Light Processing) and LCD/MSLA (Liquid Crystal Display/Masked Stereolithography) in a range of 385–420 nm. It is perfect for rapid tooling, wind tunnel testing, and industrial applications. Liqcreate Composite-X is one of the stiffest and strongest materials available in the market. It has a flexural modulus of over up to 8500 MPa and a tensile strength of up to 75 MPa. The material can be used after UV post-curing, or the properties can be boosted with a thermal cure. As an example of the traditional photopolymer, the resin Phrozen Aqua-Grey 8K-Resin was chosen to display 3D models in the highest resolution. Phrozen Aqua is designed for low shrinkage and high-precision printing. The low warpage and the good dimensional stability are particularly suitable for precise parts. The materials evaluated in this study are included in Table 1.

Table 1. Mechanical properties are given by manufacturers [18, 19].

	Liqcreate Composite-X	Phrozen Aqua 8K Series Resin
Tensile strength, MPa	50–75	35–46
Tensile modulus, MPa	7500–8500	2400–2800
Elongation at break, %	1	4.8–7.6
Density, g/cm^3	1.52	1.1

2.2. Stereolithography and specimen manufacturing

In our investigations, the dog-bone type specimens, 4 mm in height, 10 mm in width and 60 mm in length (Fig. 1), were prepared from Liqcreate Composite-X and Phrozen Aqua 8K Series Resin for the uniaxial tensile test and tribology measurements. The ANYCUBIC Photon Mono X, an LCD-based Stereolithographic 3D printer, was utilized for the fabrication of specimens. This printer is equipped with a 405 nm light source, capable of producing a resolution of 4K, and features a layer resolution ranging from 0.01 to 0.15 mm. The printing parameters for the polymer material included the first layer time of 35 s, another layer time of 2 s and the UV exposure of 60%. For the ceramic-composite material, the corresponding parameters were the first layer time of 180 s, the next layer time of 6 s and the UV exposure of 60%. Following the printing, the specimens were thoroughly cleaned with ethyl alcohol to remove any residual resin or debris.

2.3. Characterization

The mechanical properties of the specimens were obtained using a universal tensile testing machine, Labview version 2020 (*National Instruments*, USA) software, and the PXI system hardware (chassis NI PXIe-1073 and controller NITB-4330) and a tension dynamometer up to 5 kN were used for tensile tests. The tensile tests were carried out in the displacement control mode at room temperature in accordance with ISO 15733:2015 [18] and ISO 15490:2008 [19].

The macroscopic examination of tensile tested specimens was carried out using an Eclipse MA200 optical microscope (*Nikon*, Japan) with a Lumenera Infinity 2–2 video camera. The microscope was equipped with the motorized stage and imaging software, allowing for a detailed analysis of the fracture surfaces and failure mechanisms of the tested samples. High-resolution images were captured at various magnifications to study the microstructural features and identify any defects or irregularities that may have contributed to the observed mechanical behaviour.

The representative samples after tensile testing were gold-coated for scanning electron microscopy (SEM) using a dual beam system Helios NanoLab 650 (*FEI Company*) with an energy dispersion X-ray spectrometer INCAEnergy (*Oxford Instruments*). The system is supplied with a Schottky type field emission electron source and a focused Ga ions source.

A ball-on-disc (*Anton Paar*) tribometer in a ball-on-plate configuration was applied for the friction tests of samples using corundum balls of 6 mm diameter and 99.8% purity from *RGP International Srl* (Italy). The tribotesting parameters of 1 N or 10 N loads, 2 mm amplitude and 2 cm/s velocity were maintained in a linear reciprocal configuration with a track length of 4 mm and a total distance of 8 mm for one reciprocal friction cycle. The results were presented as coefficient of friction (COF) changes with progressing friction in terms of a number of cycles.

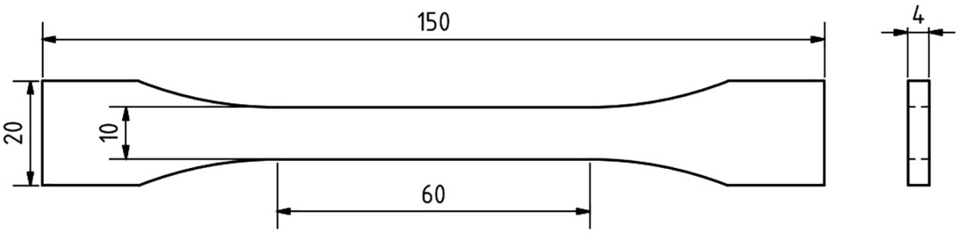

Fig. 1. Dog-bone type specimens for tensile and tribology tests.

3. Results and discussion

3.1. Mechanical properties

Mechanical properties such as tensile strength and modulus are crucial because these properties directly influence the performance, durability and longevity of the restorative materials. Dental restorations, like crowns, bridges and fillings, are subject to significant stresses due to biting and chewing. High tensile strength ensures that the materials can resist these forces without fracturing. Restorations with a high tensile strength are less likely to crack or break under the normal oral function, which is essential for their long-term success. The modulus of a material determines its ability to flex under load. Materials that are too rigid (high modulus) might transfer stress to the tooth structure, potentially leading to fractures or failure. Conversely, materials that are too flexible (low modulus) may deform excessively, affecting the function and aesthetics of the restoration.

The tensile test results of the ceramic-composite resin (Liqcreate Composite-X) and the traditional photopolymer resin (Phrozen Aqua 8K) are shown in Fig. 2. The graph illustrates the force-displacement curves for both materials and test results, derived from the average performance of five specimens for each material, showcasing their tensile failure behaviour. For the Liqcreate Composite-X, represented by the black curve, there is a sharp increase in force up to its ultimate tensile strength (UTS), reaching approximately 2600 N at a displacement of around 4 mm. After reaching the UTS, the material experiences an immediate failure, as evidenced by the abrupt drop in the force. On the other hand, the Phrozen Aqua 8K, represented by the red curve, shows a more gradual increase in force, with the UTS occurring at around 1500 N and a displacement of approximately 6 mm. Similar to the Liqcreate Composite-X, the Phrozen Aqua 8K also fails immediately after reaching its UTS, demonstrated by the sharp decline in force.

Overall, in accordance with ISO 15733:2015 [20] and ISO 15490:2008 [21], the maximum tensile strength was about 73 MPa for the Liqcreate samples and 42 MPa for the Phrozen samples (Table 2).

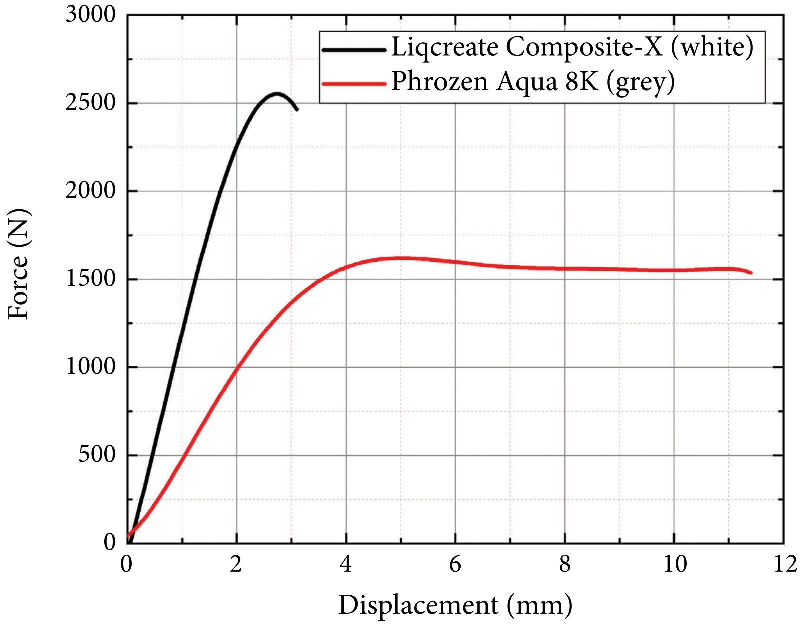

Fig. 2. The force–displacement diagram of Liqcreate Composite-X samples and Phrozen Aqua 8K Series Resin samples.

Table 2. Mechanical properties of tensile tested samples.

Specimen / Properties	1	2	3	4	5	6	7	8	9	10
F_m, [N]	2762	2764	2782	2325	2374	1631	1636	1683	1699	1493
R_m, [MPa]	73	69	70	65	66	43	43	42	42	41

The results obtained from the tensile testing confirm the mechanical properties of the materials as claimed by the manufacturers, which are given in Table 1. These properties include maximum force (F_m), tensile strength (R_m) and longitudinal deformation at the maximum tensile force (A_m), which are critical for assessing the material's performance under stress.

When comparing the technology of SLA-printed ceramic composites for dental restorations with other existing options for dental restorations, such as composite resin core without a post, composites with metal posts, composites with glass fibre posts and composites with carbon fibre posts, the SLA printing technology appears much more effective. This is also confirmed by tensile strength tests. The maximum tensile strength for SLA-printed ceramic-composites was at the level of 73 MPa, while for conventional materials, the maximum tensile strength did not exceed 51.65 MPa [22].

There is a difference between monolithic ceramics and ceramic composites for uniaxial tensile testing. In general, monolithic ceramics that are not reinforced with fibres or other particles do not exhibit plastic zones but show a typical brittle fracture behaviour leading to fracture immediately after passing through the elastic zones. Meanwhile, ceramic matrix composites reinforced with continuous ceramic fibres show the same elastic deformation as monolithic ceramics until the first matrix cracking occurs. Afterward, they show the shape of the plastic zone that can be seen in the metallic materials through further processes such as further matrix failure, crack deflection at the interface, and more interfacial interactions. This is not a plastic deformation actually occurring, but rather the fracture toughness is increased due to the behaviour that increases the time to fracture in the processes of the formation of cracks and fibre pullout, showing the deformation behaviour of this type. After continuing to reach ultimate tensile strength (UTS), they undergo a process such as fibre pullout, indicating the so-called graceful failure, and finally, the failure occurs [23]. Some 3D-printed polymer and ceramic-composite samples for tensile tests are shown in Figs. 3 and 4.

Both tensile strength and modulus are critical for dental restorations because they ensure that the materials can withstand the mechanical stresses of the oral environment while maintaining their integrity and functionality. High tensile strength prevents fractures and de-bonding, while an appropriate modulus ensures that the restoration can distribute loads effectively and match the mechanical behaviour of natural tooth structures. Together, these properties help in creating durable, reliable and long-lasting dental restorations.

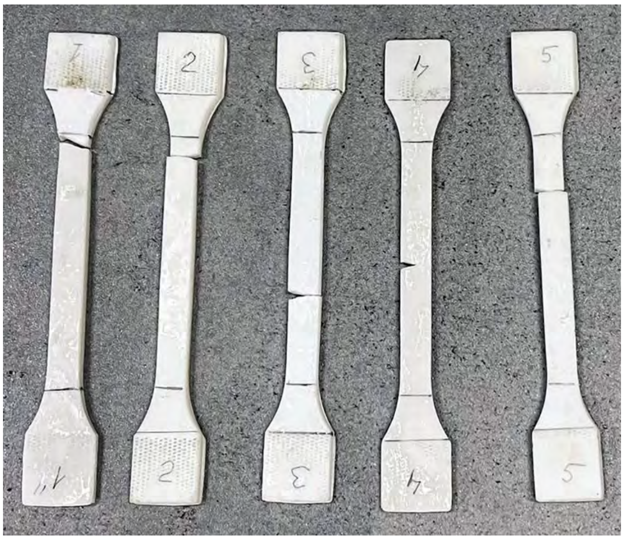

Fig. 3. The tensile tested Liqcreate Composite-X samples (marked from 1 to 5 left to right).

Fig. 4. The tensile tested Phrozen Aqua 8K Series Resin samples (marked from 6 to 10 left to right).

3.2. Specimen microstructure

The macroscopic examination results of the tensile tested samples, shown in Fig. 5, reveal the fracture characteristics of two different materials: Liqcreate Composite-X and Phrozen Aqua 8K Series

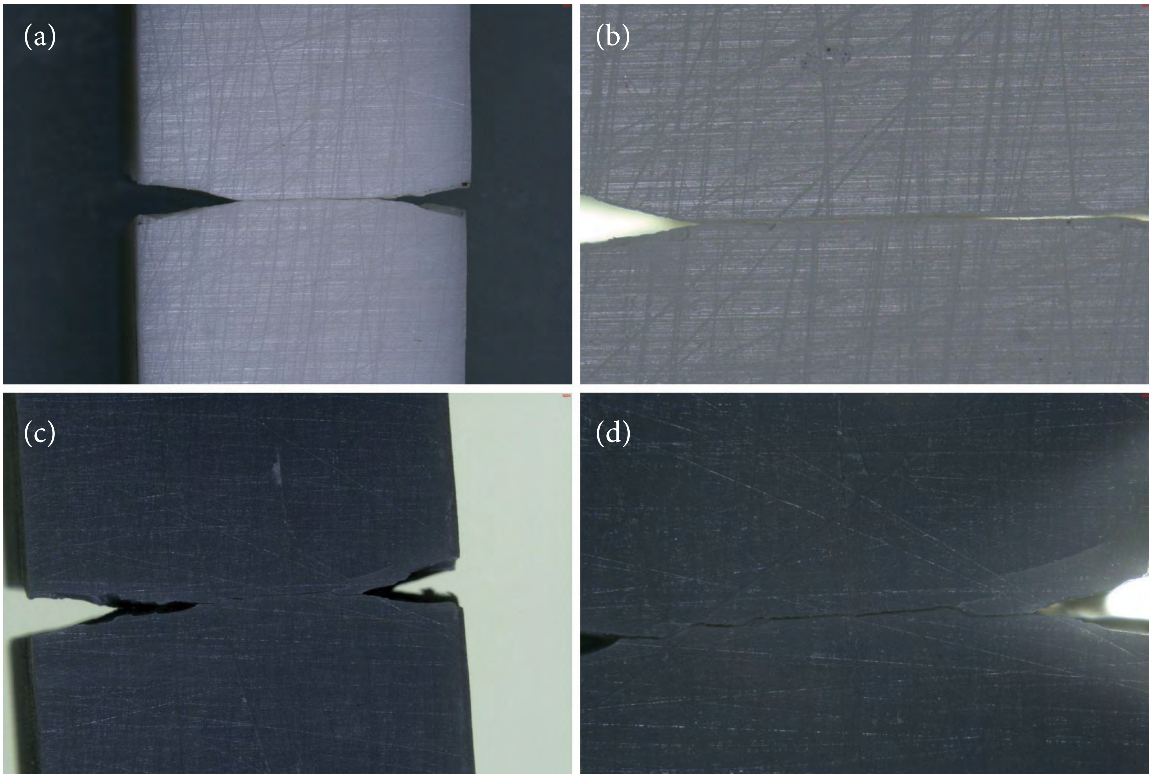

Fig. 5. Macroscopic examination of the tensile tested samples: Liqcreate Composite-X (a, b); Phrozen Aqua 8K Series Resin (c, d).

Resin. In Figs. 5(a, b), representing the Liqcreate Composite-X, the fracture surfaces are observed to be relatively clean and straight, indicating a brittle failure mode with a minimal plastic deformation prior to breaking. This aligns with a sharp drop in force observed immediately after the ultimate tensile strength (UTS) in the tensile test graph. In contrast, Fig. 5(c, d), representing the Phrozen Aqua 8K Series Resin, show a slightly rougher fracture surface with visible stress lines and some signs of plastic deformation, suggesting a more ductile failure mode. These observations support the tensile test results, where the Phrozen Aqua 8K displayed a gradual increase in force and a greater displacement before failure, indicative of its more ductile nature compared to the brittle Liqcreate Composite-X.

Figure 6 shows the SEM images of the 3D printed Liqcreate Composite-X and Phrozen Aqua 8K Series Resin composite samples. The microstructure of the Liqcreate Composite-X sample surface is shown in Fig. 6(a), while Fig. 6(c) shows the microstructure of the Phrozen Aqua 8K Series Resin composite sample surfaces. Figures 6(b, d) demonstrate that the grains are clearly defined and allocated closer to each other in Fig. 6(d) for Phrozen Aqua 8K Series Resin compared to Fig. 6(b) for Liqcreate Composite-X. The SEM microscopy and the grain arrangement confirm that the Phrozen Aqua 8K Series Resin has a more brittle structure because the grains are closely packed and lack the ability to shift significantly compared to the grains in the Liqcreate Composite-X material, which is also supported by tensile strength tests.

3.3. Wear resistance

For ceramic materials, wear resistance is closely linked to the grain size. The tribological characteristics of ceramics can be influenced by their mechanical properties, which are determined by their microstructures. According to the adhesion-deformation theory of friction, frictional forces arise from breaking adhesive bonds and the deformation of surface layers on solids. The adhesive aspect of friction is minimized under conditions of elastic deformation, meaning that harder materials tend to exhibit lower coefficients of friction, assuming that all other factors are constant.

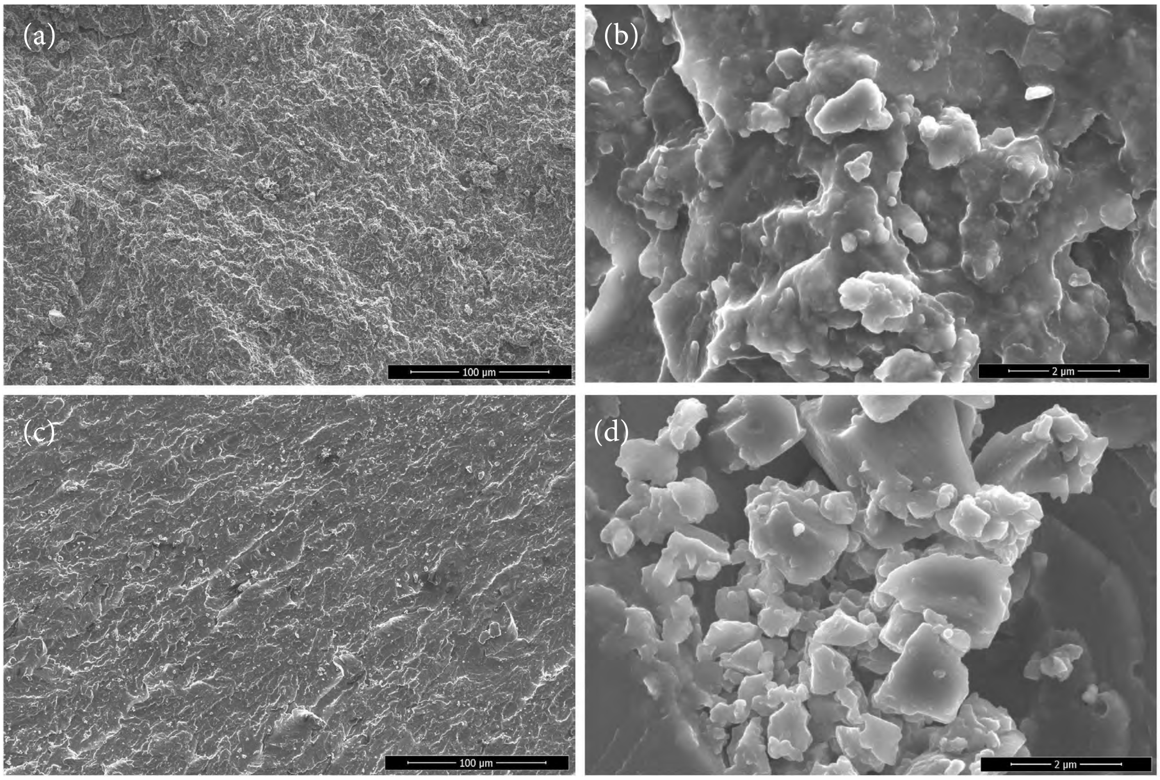

Fig. 6. SEM images of 3D printed composite samples: Liqcreate Composite-X: (a) magnification 500×, (b) magnification 20000×; Phrozen Aqua 8K Series Resin: (c) magnification 500×, (d) magnification 20000×.

The friction of Liqcreate Composite-X and Phrozen Aqua Grey 8K Resin was tested against a chemically inert corundum ball to eliminate the influence of unnecessary reactions. The tribological tests demonstrated that both materials exhibit a low friction with the initial coefficient of friction (COF) around 0.2 for the Liqcreate Composite-X and 0.1 for Phrozen Aqua Grey 8K Resin following a gradual friction increase after 100 friction cycles under 1 N load. Both samples showed a high reproducibility. However, the Liqcreate Composite-X demonstrated a tendency to increase COF during friction as a result of wear particle accumulation in the wear track zone leading to some variations in later friction stages under 1 N load. Higher loads revealed significant differences in friction reduction between the tested composites with clearly distinct inflection points, after which the COF values increased sharply. While the Liqcreate samples sustained up to only 40 friction cycles, the Phrozen samples maintained a low friction of at least 5000 cycles under 10 N load. This shows that the friction and wear resistance of composite materials can be significantly improved using advanced 3D printing technology (Figs. 7, 8).

The high stiffness and strength of ceramic-composite resin suggest a favourable mechanical resistance against incidental friction and wear under both low and high loads. On the other hand, hard nanoparticles in composite resin might act as abrasives and increase surface friction and wear. Alternative resin materials suggest tribological improvements with a low COF and an extremely high wear resistance suitable for dental applications. The friction of tested samples (Liqcreate and Phrozen) was compared to titanium and ceramic materials, which are often recommended for dental implants [24–27]. The comparison results are illustrated in Fig. 9.

Ti alloy BT1 of 96.32% purity (1.76 wt% Mn; 1.75 wt% Al; 0.11 wt% Fe; 0.06 wt% Si) demonstrated steady-state friction with COF ~0.5, which is considered as high, leading to surface damage and wear after several friction cycles under dry friction conditions. Nanoporous ceramic alumina coatings produced by electrochemical oxidation (anodization) of Al alloys in acidic electrolytes

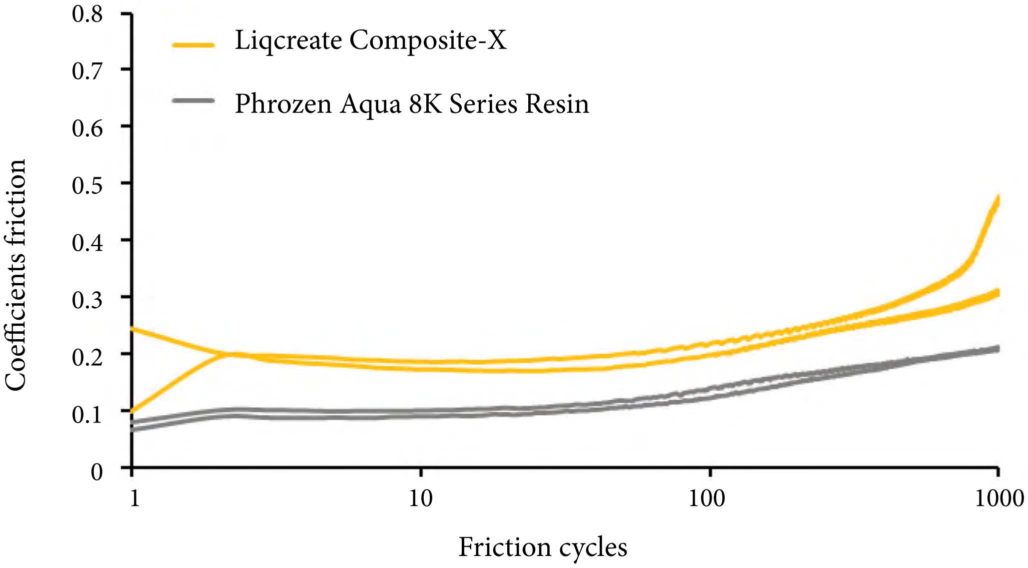

Fig. 7. Friction coefficients of the samples made under the conditions: 1 N load, 2 cm/s velocity, 2 mm amplitude, 4 mm length, 6 mm corundum ball.

offer improved hardness and corrosion resistance with coating thickness sometimes exceeding 100 μm [25]. Ceramic coatings with porous structures also increase cell adhesion and biocompatibility suitable for biomedical applications [24, 26]. Despite its hardness, friction, and wear of ceramic coatings remain one of the major problems leading to even more intensive friction than that of Ti alloy BT1. Sputtered Ti nanolayers of 75 nm in thickness showed a significant improvement in reducing friction on nanoporous ceramic alumina with COF below 0.2 for at least 10 friction cycles, after which the COF values increased sharply. The hypothesis of the quasi-fluid behaviour of Ti is more closely affiliated with the wear mechanisms in thin films or layers during friction [27]. However, neither Ti nor nanostructured alumina coatings could improve tribological effectiveness like ceramic-composite resin or other resin samples.

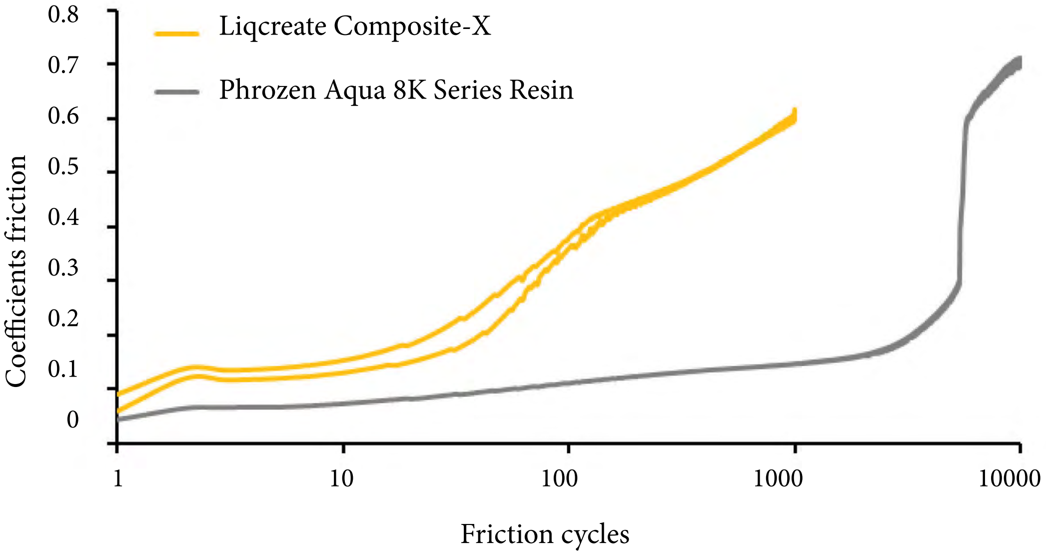

Fig. 8. Friction coefficients of the samples made under the conditions: 10 N load, 2 cm/s velocity, 2 mm amplitude, 4 mm length, 6 mm corundum ball.

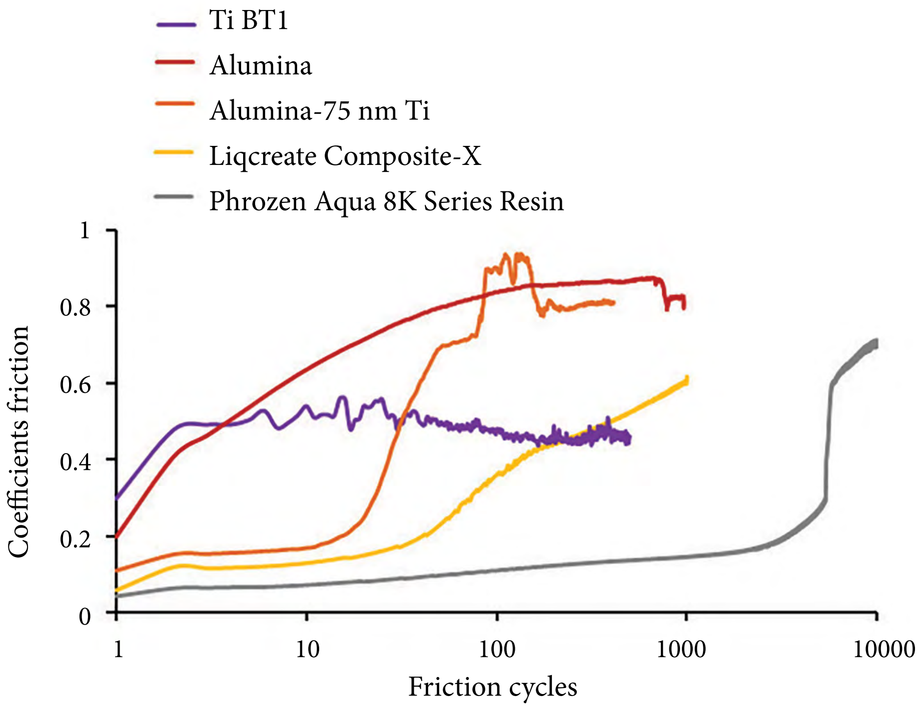

Fig. 9. Comparison of the friction tendencies of the tested samples vs Ti BT1 alloy and nanostructured alumina under 10 N load, 2 cm/s velocity, 2 mm amplitude and 4 mm length, against 6 mm corundum ball adapted from Ref. [23].

4. Conclusions

1. The ceramic-composite resin was successfully printed using SLA technology, demonstrating a good printability and the ability to produce detailed and complex geometries necessary for dental restorations.

2. The Liqcreate Composite-X resin exhibited superior mechanical properties compared to traditional photopolymer resins such as Phrozen Aqua 8K. Specifically, the tensile strength of the Liqcreate Composite-X reached approximately 73 MPa, significantly higher than 42 MPa observed for the Phrozen Aqua 8K resin. The high tensile modulus of the ceramic-composite resin, ranging between 75 to 85 MPa, indicates a robust material capable of withstanding a significant stress, making it highly suitable for dental applications that require durability and resilience.

3. Tribological tests highlighted the excellent wear resistance of the ceramic-composite and other resin materials compared to titanium and alumina. Under higher loads, the Phrozen Aqua 8K resin maintained a low coefficient of friction (COF) over a greater number of cycles compared to the Liqcreate Composite-X. This indicates the material's potential for longer-lasting dental restorations with reduced wear over time. The ceramic particles within the composite resin suggest the ability to resist incidental friction and wear by enhancing the material's hardness.

4. The novel ceramic-composite resin holds a significant promise for improving the quality and longevity of dental restorations. Its enhanced mechanical and tribological properties could lead to more durable and reliable dental prosthetics, ultimately benefitting patient outcomes. By reducing material waste and enabling the production of patient-specific restorations with a high precision, this resin can contribute to more efficient and cost-effective dental manufacturing processes.

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STEREOLITOGRAFIJA ATSPAUSDINTŲ KERAMINIŲ KOMPOZITŲ, SKIRTŲ DANTIMS RESTAURUOTI, CHARAKTERIZAVIMAS

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Santrauka

Šiame tyrime pristatoma nauja keraminė kompozitinė derva, specialiai sukurta stereolitografijos (SLA) 3D spausdinimui, kuria siekiama patobulinti dantų restauravimą. Pažangių skaitmeninių technologijų integravimas odontologijoje pakeitė tradicinius metodus, pereinant prie tikslesnių ir efektyvesnių metodų, pavyzdžiui, kompiuterizuoto projektavimo ir kompiuterizuotos gamybos (CAD/CAM). Tačiau šie procesai dėl savo veikimo pobūdžio medžiagas yra linkę švaistyti. Pridėtinė gamyba, arba 3D spausdinimas, ypač SLA, yra perspektyvi alternatyva, galinti kurti tikslias, individualias ir lengvai pritaikomas dantų koregavimo ir atkūrimo priemones. Šiame straipsnyje pagrindinis dėmesys skiriamas keramikos kompozito dervos, pasižyminčios mechaniniu tvirtumu, reikalingu dantims restauruoti, spausdinimo galimybių įvertinimui. Atlikti išsamūs mechaninių savybių ir atsparumo dilimui bandymai parodė kompozitinės dervos 73 MPa stiprumo ribą, kuri yra gerokai didesnė nei tradicinės fotopolimerinės dervos (42 MPa). Taip pat nustatyta, jog keraminė kompozitinė derva pasižymi atsparumu atsitiktinei trinčiai ir dilimui. Šis tyrimas turi praktinės reikšmės dantų protezų gamybai, nes suteikia galimybę efektyviai ir pigiai gaminti aukštos kokybės, pacientui pritaikytus dantims restauruoti skirtus gaminius.