UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

CASSIANA KOCH SCOTTI

Mechanical, optical and surface properties of a composite to three-dimensional (3D) printing reinforced with nanofibers of niobium, bioactive glass and nanosilver

Caracterização mecânica, óptica e de superfície de uma resina para impressão tridimensional (3D) aditivada com nanofibras de nióbio, vidro bioativo e nanoprata

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Tese apresentada a Faculdade de Odontologia de Bauru da Universidade de São Paulo para obtenção do título de Doutor em Ciências no Programa de Ciências Odontológicas Aplicadas, na área de concentração Dentística.

Orientadora: Profa. Dra. Juliana Fraga Soares Bombonatti

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“A simplicidade é o último degrau da sabedoria.”

Khalil Gibran

ABSTRACT

Mechanical, optical and surface properties of a composite to three-dimensional (3D) printing reinforced with nanofibers of niobium, bioactive glass and nanosilver.

Rapid and automated prototyping (3D printing or additive manufacturing) have been hailed as a disruptive technology that tends to change the course of different production areas. The focus of our studies has been on technological strategy which allows to reduce the cost and chair time, boosting the work flow approach and comfort to the patient associated to the use of bioactive materials that promote tissue regeneration or repair in restorative dentistry. Aiming to understand and improve the properties of a composite for 3D printing with dental applications, this research was developed in 2 parts with specific objectives. In the first study, commercial 3D-printable resin for interim restorations (Next Dent C&B) was compared *in vitro* to two restorative materials (Filtek - Z350 and Protemp - 4) in relation to mechanical, optical and surface properties. The second study was conducted with the aim of synthesizing bioactive reinforcing nanofibers (niobium pentoxide, bioactive glass and nano-silver), incorporating them into a resin for 3D printing and to evaluate *in vitro* the effect on mechanical, optical and material surface properties. In the first study, the results showed properties suitable for use in interim restorations and highlighted its potential for a clinical approach in digital workflow. However, it also observed a concern about long-term color stability. The second study showed that the incorporation of nanofibers in the tested formulations modifies the mechanical, optical and surface properties of the resin for 3D printing at specific concentrations. In general, the studies support the use of 3D printing technology to interim restorations and highlighted the need for improvements regarding long-term color stability. In this gap, the reinforcement of niobium-based nanofibers, reveals to be a potential additive. These findings are of special interest to solidify the use of 3D technology and workflow approach in dentistry.

Key words: 3D printing. Composite. Nanofibers

RESUMO

Caracterização mecânica, óptica e de superfície de uma resina para impressão tridimensional (3D) aditivada com nanofibras de nióbio, vidro bioativo e nanoprata.

A prototipagem rápida e automatizada (impressão 3D ou manufatura aditiva) é saudada como uma tecnologia disruptiva que tende a mudar o rumo de diversas áreas de produção. O foco de nossos estudos tem sido estratégias tecnológicas que possibilitem a redução do custo e tempo operacional, aumento do fluxo de trabalho e maior conforto ao paciente, aliado a utilização de materiais bioativos que promovam a regeneração ou reparo tecidual em dentística restauradora. Objetivando o entendimento e a melhoria das propriedades de uma resina para impressão com aplicações odontológicas, essa pesquisa foi desenvolvida em 2 partes com objetivos específicos. No primeiro estudo, uma resina para impressão 3D comercial (Next Dent C&B) com indicação para restaurações provisórias foi comparada *in vitro* a dois materiais restauradores (Filtek Z350 e Protemp 4) em relação às propriedades mecânicas, ópticas e de superfície. O segundo estudo foi conduzido com o objetivo de sintetizar nanofibras bioativas de reforço (nanofibras de pentóxido de nióbio, vidro bioativo e nanoprata), incorporá-las a uma resina para impressão 3D (Next Dent C&B) e avaliar *in vitro* o efeito nas propriedades mecânicas, ópticas e de superfície do material. No primeiro estudo, os resultados destacam propriedades adequadas para o uso em restaurações provisórias enaltecendo o seu potencial para uma abordagem clínica em fluxo digital. Entretanto, deixa evidente a preocupação em relação à estabilidade de cor a longo prazo. O segundo estudo mostrou que a incorporação das nanofibras nas formulações testadas modifica as propriedades mecânicas, ópticas e de superfície da resina para impressão 3D em concentrações específicas. De modo geral, os estudos embasam a utilização da tecnologia de impressão 3D para confecção de restaurações provisórias e, enaltecem a necessidade de melhorias em relação à estabilidade de cor a longo prazo. Neste hiato, a incorporação de nanofibras de Nb_2O_5, revela ser um potencial aditivo. Tal conhecimento é de especial interesse para apresentar ferramentas que solidifiquem a utilização da tecnologia 3D na prática clínica em odontologia.

Palavras chave: Impressão 3D. Compósitos. Nanofibras.

SUMMARY

1 INTRODUCTION

1 INTRODUCTION

Although the evidence of minimal intervention and health promotion in dentistry, the loss or partial destruction of important structures of the stomatognathic system (i.e. the teeth) is still prevalent in the world wied and have been demonstrated a severe impact on the quality of life (FERNANDES et al., 2017; MARCENES et al., 2013; DYE et al., 2010; FRAZÃO et al., 2012).

Overall, the oral health care is still curative and has been attributed to highly complex procedures such as restorations, reconstruction of lost tissues, or even mutilating interventions. Estimated annual costs for treating dental diseases are $ 298 billion, corresponding to 4.6% of global health investments. This scenario has boosted the great scientific and technological development of Restorative Dentistry (LISTL S et al., 2015).

Therefore, technological strategies that enable the reduction of cost and chair time, increasing the workflow approach and patient comfort as soon as promoting tissue repair and optimizing the maintenance of health conditions are highly desirable (TURNER et al., 2014; VAN NOORT, 2012; CHENG, 2017). Rapid and automated prototyping (i.e. 3D printing or additive manufacturing) has been hailed as a disruptive technology that tends to change the course of different production areas. Used in the aerospace industry, engineering, robotics, art, and design, it is considered promising in particular in dentistry due to its communication and compatibility with the imaging and modeling technologies already used, such as computed tomography and intra-oral scanning. To date, 3D printing allows obtaining surgical guides, dental models, temporary unitary restorations, and dental or maxillofacial prostheses (VAN NOORT, 2012; TAHAYERI et al., 2018; MAI et al., 2017).

The main advantages of this system are high precision, low-cost technology (i.e. compared to subtractive manufacturing - CAM), creation of geometrically complex and customized structures layer by layer, reduction in building time, less waste of material, easy reproducibility due to the storage of digital data and better-quality control (REVILLA-LEÓN, 2018).

Currently, most computer-based manufacturing processes are based on subtractive manufacturing or milling. 3D printing was first developed in the 1980s, but with advances in computer-aided design (CAD) technology, these expensive and technologically limited 3D printers have improved. Nowadays, 3D printers are affordable, user-friendly, reliable, and capable of producing complex shapes. Many different printing technologies exist, each with their own advantages and disadvantages such as Light cured resin systems (stereolithography, photojet and digital light processing), Powder binder printers, Sintered powder (Selective laser sintering, Selective laser sintering and Electron beam melting), Thermoplastic systems (Fused deposition modelling).

Most used in dentistry 3D printers are based on stereolithography (SLA) or digital light processing (DLP). In SLA, voxels of monomers are polymerized by a laser light track directed by a Galvano mirror scanner to build 3D structures. In DLP, a digital projector screen flashes light through the entire layer to build the 3D structures. (DAWOOD et al., 2015; REVILLA-LEÓN, 2018; DEHURTEVEN et al., 2017; ASTUDILLO-RUBIO et al., 2018; TOPCU et al., 2009).

Despite a few shreds of evidence about the use of 3D-printed materials in dentistry, information related to their optical and surface properties, including their compatibility with the oral environment, surface roughness, and esthetics, is scarce and need to be investigated. Nonetheless, the improvement in the mechanical, optical, chemical, surface, biological, and antimicrobial properties of this class of materials is highly desirable. (REVILLA-LEÓN, 2018; REVILLA-LEÓN et al., 2017; KORTES et al., 2018; HOMSY et al., 2017).

The incorporation of nanostructures, mainly in the form of nanofibers, in restorative materials is considered a promising strategy (AL-EESA et al., 2018; WILLE et al., 2016;). Nanofibers mats have a high specific surface and high surface free energy, which is related to the increase in flexural strength of conventional composite resins (GAROUSHI et al., 2007; SUN et al., 2010). Zhang et al, 2012, reported about the ability of glass nanofibers to transfer stresses more efficiently than conventional filler because they are distributed in an aligned and in a uniform way into the resin. Thus, they act as bridges that limit the deflection of cracks, increasing the toughness and fracture of the material (ZHANG et al., 2012; WILLE et al., 2016; CHEN et al., 2011; GUO et al., 2012).

In the materials engineering area, the development of hybrid nanofibers (i.e. organic-inorganic) embedded with bioactive oxides, ceramics or metallic ions allows combining desirable properties of the inorganic phase, such as thermal stability and rigidity, with those of the organic phase, flexibility, processability, ductility, and biocompatibility (ANZAI et al., 2002; GAO Y et al., 2017; HOLOPAINEN et al., 2016; MEDEIROS et al., 2009; PAPKOV et al., 2013; TIAN et al., 2007; VIDOTTI et al., 2015; WILLE et al., 2016;).

In view of the above, the insertion of bioactive nanofibers in composites for additive manufacturing (3D printing) proposes the improvement of this class of materials, until now with limited applications in dentistry. Therefore, these may have their chemical, mechanical and antimicrobial properties improved and also can act in favor of tissue repair and regeneration. The synthesis of hybrid nanofibers (organic-inorganic), non-toxic, with unique mechanical properties, high surface energy, biocompatibility, and bioactive/antimicrobial properties is undoubtedly a nanotechnological challenge proposed by this research work.

In addition to the aforementioned, the response of this study will provide the basis for future interdisciplinary investigations of new dental materials for 3D printing applications which are more predictable and also potentially applicable to regenerative dentistry. We will also be able to infer the behavior of a nanofiber reinforced resin, which may open an arsenal of applications for doping other innovative composites with properties applicable in different areas of dentistry.

Main objective

To evaluate the properties of a 3D-printable resin for dental applications and to characterize mechanical, optical and surface properties of a 3D-printable resin with innovative nanotechnological potential.

Specific objectives

First study

1. To evaluate mechanical properties of a 3D-printed resin comparing to two restorative materials in flexure by 3-point bending (σ) and Knoop microhardness (H);

2. To evaluate the smoothness of a 3D-printed resin by roughness analysis (Ra) comparing with two restorative materials;

3. To evaluate the color stability after aging of a 3D-printed resin comparing with two restorative materials;

Second study

1. To synthesize three novel functional nanofibers of PDLLA embedded with orthorhombic niobium pentoxide (PDLLA/Nb_2O_5); PDLLA embedded with orthorhombic niobium pentoxide with network on bioactive glass (PDLLA/Nb_2O_5/SiO_2); and nanofibers doped with bioactive nano-silver glass (PLA-PEG/SiO_2/Ag^3).

2. To incorporate three novel functional nanofibers of PDLLA embedded with orthorhombic niobium pentoxide (PDLLA/Nb_2O_5); PDLLA embedded with orthorhombic niobium pentoxide with network on bioactive glass (PDLLA/Nb_2O_5/SiO_2); and nanofibers doped with bioactive nano-silver glass (PLA-PEG/SiO_2/Ag^3), in a 3D-printed resin.

3. To characterize mechanical properties of a 3D-printed resin reinforced with three novel functional nanofibers in flexure by 3-point bending (σ) and Knoop microhardness (H);

4. To evaluate the smoothness of a 3D-printed resin by roughness analysis (Ra) reinforced with three novel functional nanofibers;

5. To evaluate the color stability after aging of a 3D-printed resin reinforced with three novel functional nanofibers;

2 ARTICLES

2 ARTICLES

- ARTICLE 1 – Physical and surface properties of a 3D-printed composite resin for a digital workflow.
- ARTICLE 2 – Improved behavior of a novel 3D-printed reinforced by nanofibers of niobium, nanosilver and bioactive glass.

2.1 ARTICLE 1

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Physical and surface properties of a 3D-printed composite resin for a digital workflow

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ABSTRACT

Statement of problem. Information related to the optical and surface properties, including health compatibility, surface roughness, and esthetics, of 3D-printed dental materials is scarce.

Purpose. The purpose of this in vitro study was to compare the physical and surface properties of a 3D-printed resin with materials used for interim restorations.

Material and methods. A 3D-printed resin (PR) (NextDent C&B MFH; 3D Systems), an autopolymerizing interim material (BA) (Protemp 4; 3M ESPE), and a composite resin (Z350) (Filtek Z350XT; 3M ESPE) were tested for degree of color change (ΔE) (n=7) at different timepoints: 24 hours after polishing/baseline (P0), 8 days after polishing (P1), and after artificial aging in water at 60 ºC for 24 hours (P2) with a CIELab-based colorimeter; flexural strength (σ) (n=10) with a 3-point bend test; Knoop hardness (H) (n=8); and surface roughness (Ra) (n=7) with a profilometer. All specimens were polished 24 hours after polymerization, except for the additional group for surface roughness (BA) without polishing (BANP). Statistical analysis was performed by using 2-way repeated measures ANOVA followed by the Fischer test for ΔE and 1-way ANOVA followed by the Fisher test for microhardness and surface roughness(α=.05).

Results. The Z350 showed the highest values for σ and H, followed by PR and BA (*P*<.05). Considering roughness, the Z350 showed similar values to those of BA, but lower than PR; PR showed similar roughness when compared with BA. PR showed the highest color variation among the groups at all timepoints, followed by BA. The Z350 was the most color stable material at all timepoints.

Conclusions. The 3D-printed composite resin had adequate mechanical and surface properties for an interim restorative material. It has the potential to be a low-cost workflow in dentistry, although its color stability could be a concern for long-term use.

INTRODUCTION

Incorporating innovative technology has accelerated the adoption of a digital workflow in many dental practices, and computer-based methods of fabricating dental restorations are a growing field in dentistry.[1-4] The advantages of digital technologies include the capability to improve restorative treatment steps, including diagnostic casts, diagnostic waxing, preparation guides, trial restorations, and interim restorations, as well as the digital treatment plan, diagnosis, and fabrication of the restoration, resulting in a more efficient dental practice.[5-8]

Currently, most computer-based manufacturing processes are based on subtractive manufacturing or milling. 3D printing was first developed in the 1980s, but with advances in computer-aided design (CAD) technology, these expensive and technologically limited 3D printers have improved. Currently, 3D printers are affordable, user-friendly, reliable, and capable of producing complex shapes. Most 3D printers are based on stereolithography (SLA) or digital light processing (DLP). In SLA, voxels of monomers are polymerized by a laser light track directed by a Galvano mirror scanner to build 3D structures.[9,10] In DLP, a digital projector screen flashes light through the entire layer to build the 3D structures. Compared with milling, 3D printed appliances and restorations can improve patient acceptance, provide adequate marginal and internal fit, and reduce costs (1 liter of resin can print an average of 300 single crowns).[11-13] Nevertheless, information on the properties and clinical behavior of printed materials is still lacking.[8,14]

Interim restorations are fundamental to achieving predictable successful definitive restorations. They maintain soft tissue health, protect the pulpal tissue, achieve an adequate emergence profile, stabilize the 3D position of the prepared tooth, enhance the acceptance of the treatment plan and patient comfort, and promote better communication among clinicians, patients, and laboratory technicians.[15-20]

The most popular materials for interim restorations are polymethyl methacrylate (PMMA) and bis-acrylic resins. These materials are adequate and cost-effective for clinical use but have limitations (especially PMMA) as they cannot be directly integrated into a digital workflow and rely on free-hand fabrication.[10,15,21-32] Despite recent research on the use of 3D-printed materials in dentistry,[3,4,10,12,14,15,21-34] information related to their optical and surface properties, including their compatibility with the oral environment, surface roughness, and esthetics, is scarce.

Therefore, the purpose of this in vitro study was to compare the physical and surface properties of resins for 3D printing with those of resins for interim restorations. The null hypothesis tested were that no difference would be found in color stability, flexural strength, hardness, or roughness among the tested resin-based materials.

MATERIAL AND METHODS

This in vitro study involved 1 factor (resins) at 3 levels: an interim material based in bisacryl resin (BA; Protemp 4; 3M ESPE); a microfilled 3D-printable resin for stereolithography (SLA) printing (PR; NextDent C&B MFH; 3D Systems), and a conventional composite resin (Z350; Filtek Z350XT; 3M ESPE) with response variables of color stability (ΔE), hardness (H), surface roughness (Ra), and flexural strength (σ). Product specifications are shown in Table 1.

For the degree of color change (ΔE), Knoop microhardness (H), and surface roughness (Ra) tests, disk-shaped specimens were prepared using 2-part Teflon molds (10 mm diameter; 2 mm height), with 2 glass slides on the top and bottom. For the light-polymerizing materials, a wide spectrum polywave (395 to 480 nm) light-emitting diode (LED) light-polymerizing unit (Valo Grand; Ultradent Products, Inc) with 1000 mW/cm^2 was used for 40 seconds for each specimen. The irradiance from the polymerization light was checked with a radiometer (RD-7; Ecel Indústria e Comércio Ltda) before starting each group. The specimens were completely covered by the polymerization light tip (ø10 mm). For the BA resin, specimens were allowed to polymerize for 5 minutes before being removed from the matrix. The specimens were embedded in epoxy resin and polished with decreasing grit abrasive papers (600-, 1200-, and 2400-grit) (Buehler Ltd), followed by a polishing cloth with 0.5-µm diamond paste (Buehler Ltd) either immediately (ΔE) or 24 hours (H and Ra) after specimen preparation. Materials of similar color and opacity were chosen to standardize the conditions for all groups. To standardize specimen preparation, polishing procedures were performed by a single operator (K.C.S.) using an automated device.

For the ΔE test (n=7) the color change was assessed at different time points by using a CIELab-based colorimeter (Vita Easyshade V; Vita Zahnfabrik). Before measurements, the spectrophotometer was calibrated according to the manufacturer's instructions. An initial measurement (P0) was performed 24 hours after polishing; a second measurement (P1) 8 days after the polishing procedures (7 days after P0); and a third measurement after artificial aging (P2) consisting of 24 hours water storage at 60 °C.[16,34] Between P0 and P1, all specimens were dry stored at 37 °C in the absence of light. Three consecutive measurements were made in the center of each specimen until uniformity of values was observed.[18,19,21]

The ΔE was calculated based on the following equation: $\Delta E=\sqrt{(\Delta L^*)^2+(\Delta a^*)+(\Delta b^*)^2}$, where ΔL*, Δa*, and Δb* correspond to the color differences observed between the baseline (P0) and after the storage period (P1 and P2).

For the H test (n=8), 3 indentations separated by 100 μm (Knoop diamond, 0.49 N, 15 seconds) were made at the center of each specimen with a digital microhardness tester (HMV–2000; Shimadzu). The built-in software program calculated the Knoop hardness (KHN) values based on the dimensions of the longest diagonal by using the following formula: $KHN=\frac{14228c}{d^2}$, where *c* is the load in gram force, and *d* is the length of the longer diagonal in μm.

For Ra (n=7), including an additional group using BA without surface polishing (BA-WP), surface roughness was assessed with a roughness tester (Hommel-Etamic W10; PCE GmbH) following ISO 4287 guidelines[35] with a 0.80 mm cutoff, 0.0001 μm resolution (8 μm range), 0.5 mm/second speed, and total length of 4 mm. The average of the 3 measurements of the x- and y-axes was calculated and adopted as a result for each specimen.

For the flexural strength (σ), 10 bars with standard dimensions (10×2×2 mm) were fabricated for each material, stored at room temperature for 24 hours, and tested by using a 3-point bend test attached to a universal test machine (Instron 3342; Instron) with a cross-head speed of 0.5 mm/minute. A central load was applied between supporting rods (8-mm span length).

The mean σ values and the standard deviations were calculated for each of the materials using the following equation: σ= $\frac{3Fl}{2bd^2}$, where F is the loading force at the fracture point, l is the length of the support span (8 mm), b is the width (2 mm), and d is the thickness (2 mm).

The data were statistically analyzed using a statistical software program (Minitab 18; Minitab LLC). Normal distribution was checked for all variables using the Kolmogorov-Smirnov test. The σ, H, and Ra were analyzed by using 1-way ANOVA followed by the Fischer test, and ΔE was analyzed using 2-way repeated measurements ANOVA followed by the Fischer test (α=.05, for all tests).

RESULTS

The results for ΔE are shown in Table 2 and Figure 1. After 8 days in dry storage (7 days from P0 measurement), Z350 (positive control group) showed lower $\Delta E_{(P1\text{-}P0)}$ (1.19 ±0.26) than the PR (3.81 ±0.35) (*P*<.001) and BA (2.28 ±0.71)(*P*<.001) groups, which also showed significant differences among them (*P*<.001). Considering ΔE after artificial aging or $\Delta E_{(P2\text{-}P0)}$, the behavior was similar, with Z350 showing the lowest ΔE values (1.82 ±0.73), followed by BA (2.88 ±0.71) (*P*<.001). PR group showed the highest ΔE values (9.32 ±0.63) when compared with Z350 (*P*<.001) and BA (*P*<.001).

The σ and H means results are shown in Table 3. For both variables (σ and H), the Z350 showed the highest values followed by PR. BA showed the lowest values for both σ and H. The results for roughness analysis are shown in Table 4 and Figure 2. BA without polishing had the highest roughness values among all tested composite resins. No significant differences were observed between the BA and

Z350 groups (*P*>.05). The PR showed similar results when compared with BA but higher values when compared with Z350.

DISCUSSION

The potential increase in productivity and predictability, including the use of planning tools and 3D-printed interim restorations, has promoted the adoption of digital technology.[8] Nonetheless, the authors are unaware of studies on the surface properties, mechanical properties, and color stability of microfilled resins for 3D printing.

Based on data from the present study, all null hypotheses were rejected. The difference among the groups indicated a material-dependent effect. In general, the 3D-printed resin was less color stable after aging than the other tested materials. The color stability of resin-based materials is influenced by factors including the degree of conversion, polarity of monomers, amount of cross-linking, initiator system, particle size and distribution, water sorption, monomer conversion, and pigment stability.[18,19,23,24]

Aging may cause matrix softening, degradation, and increased staining in resin-based materials.[25] The PR and BA groups had greater mean color change than Z350 up to 8 days in dry storage or $\Delta E_{(P1-P0)}$. Such results indicate that Z350XT was almost completely polymerized after 24 hours, while PR and BA continued to polymerize for at least 8 days. After artificial aging ($\Delta E_{(P2-P0)}$), similar results were observed (PR>BA>Z350), with PR (ΔE values being clinically meaningful for both timepoints since they were higher than the clinical threshold of ΔE 3.3.[18,19,23] The greater color change presented by PR and BA when compared with Z350 might have occurred because of the lower filler content and higher organic matrix, which are also less hydrophobic than the resin matrix in conventional composite resins.[3,31]

The significant change in ΔE for BA can also be attributed to the presence of a higher number of polar molecules, resulting in greater affinity to water.[15] A systematic review reported that errors during preparation of bis-acrylic resins are common, resulting in poor homogenization, porosity, and compromised color stability, which might also help explain such results.[15] The PR resin showed the highest color alteration for both timepoints, which might be related to a lower initial degree of conversion since the authors did not perform the 1-hour postpolymerization using the recommended UV light box. This approach was adopted since postpolymerizing printed resins for such a long time is not compatible with a chairside workflow. In order to approach a more feasible clinical protocol, a wide spectrum polywave light polymerization unit (Valo Grand – 395-480 nm) was used to light polymerize the 3D-printed composite resins since it better approaches the UV light box reported wavelength (300-550 nm). Nonetheless, data for degree of conversion, polymer cross-linking density, and filler content is still uncertain, and further studies are needed.

The results of the present study suggest that the chemical composition of the interim material affects its mechanical properties, as previously reported.[14] The 3D-printed resin (PR) showed higher mechanical properties (hardness and flexural strength) when compared with the bis-acrylic resin (BA). The results for the bis-acrylic resin were similar to those reported in other studies,[29-32] although a recent study reported similar flexural resistance of bis-acrylic and an unfilled 3D printed resin.[3] Z350XT showed the highest values for mechanical testing, which might be explained by a higher amount of filler content and increased polymerization.[17]

Smooth interim restorations are essential to avoid biofilm accumulation and to maintain healthy periodontal tissues. The conventional nanofilled composite resin (Z350XT) had the smoothest surfaces, with significant difference when compared with PR and BA-WP, although similar to BA; PR was also similar to BA. Nonpolished bis-acrylic resin (BA-WP) showed the roughest surfaces. The manufacturers' instructions for the bis-acrylic resin do not indicate polishing procedures since it could weaken thin structures such as cervical areas. The present data support the fact that large size particles result in surface irregularities and increased staining.[26] In addition, larger filler particle content has been associated with increased roughness, consistent with the present study.[27,28]

3D-printed resins could be a promising material for clinical applications in which bis-acrylic resins have been used. Although the high ΔE could be of concern in an esthetic area, the surface roughness and mechanical properties can be considered more important in ensuring the maintenance of health and the position of the prepared tooth, especially when considering short-term interim restorations. Moreover, the use of the recommended light polymerization protocol could improve the color stability and should be studied. Such restorations might be used for a limited time, explaining why there is no need to use a regular composite resin as an interim restoration. Their superior physicomechanical properties are not usually justified by the additional costs and more time-consuming fabrication nor by their more complex clinical handling as a result of a higher elastic modulus.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. 3D-printed resin presented better mechanical and surface properties when compared with bis-acrylic resin, supporting its use for interim restorations.
2. However, its limited color stability is of concern for use in esthetic areas or when the interim restorations are to be used for an extended time.

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TABLES

Table 1. Products, manufacturers, and composition of materials tested

Resins	Brand/ Manufacturer	Shade	Lot No.	Composition
3D printable composite	Crown & Bridge - MFH (Next Dent)	A2	XK55N91	Methacrylic oligomers, Phosphine oxides, microfillers
Nanoparticle composite	Filtek Z350XT (3M ESPE)	A2	911689	Bis-GMA, Bis-EMA, UDMA, TEGDMA, Zirconia and silica fillers. Silane and pigments. Loading percentage by weight: 82% (5-20 nm non-agglomerated silica and 5-20 nm zirconium/ silica nanoagglomerate. 0.6-1.4μm agglomerated particles).
Bis-Acrylic Interim Composite Resin	ProTemp Plus (3M ESPE)	A2	3712273	Functionalized Dimethacrylate polymer; Bis-GMA, Zirconia and Silica fillers, Silane and pigments. Loading percentage by weight: 78%

Bis-GMA, bisphenol-A-diglycidyl methacrylate; Bis-EMA, bisphenol A ethoxylate dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

Table 2. Mean ±standard deviation of ΔE (n=7) for tested resins

Resins	$\Delta E_{(P0-P1)}$	$\Delta E_{(P2-P0)}$
Z350	1.19 ±0.26^{Aa}	1.82 ±0.73^{Aa}
BA	2.28 ±0.33^{Ba}	2.88 ±0.71^{Ba}
PR	3.81 ±0.35^{Ca}	9.32 ±0.63^{Cb}

Different uppercase letters indicate statistically significant differences between rows (inter-group) within same column (timepoint) (P<.05)

Different lowercase letters indicate statistically significant differences between columns (intra-group) within same row (P<.05)

Table 3. Mean ±standard deviation values of flexural strength (n=10) and hardness (n=8) for tested resins

Composite resins	Flexural strength	Hardness
Z350	105.10 ±9.80^{A}	61.70 ±5.70^{A}
BA	27.90 ±6.10^{C}	22.10 ±3.10^{C}
PR	67.15 ±11.70^{B}	35.00 ±2.50^{B}

Different uppercase letters indicate statistically significant differences between rows within same column (*P*<.05)

Table 4. Mean ±standard deviation values of surface roughness (n=7) for tested resins

Resins	Surface Roughness (Ra)
BA-WP	0.37 ±0.10^{A}
PR	0.16 ±0.02^{B}
BA	0.15 ±0.03^{BC}
Z350	0.07 ±0.01^{C}

Different uppercase letters indicate statistically significant differences between groups (P<.05)

FIGURES

Figure 1. Color difference comparison for tested resins.

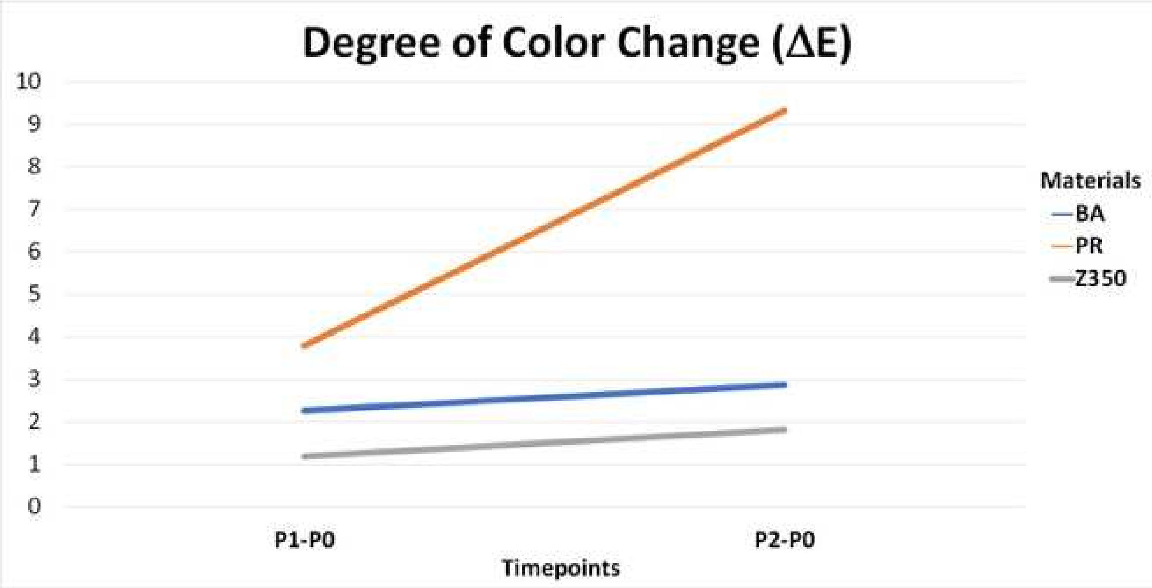
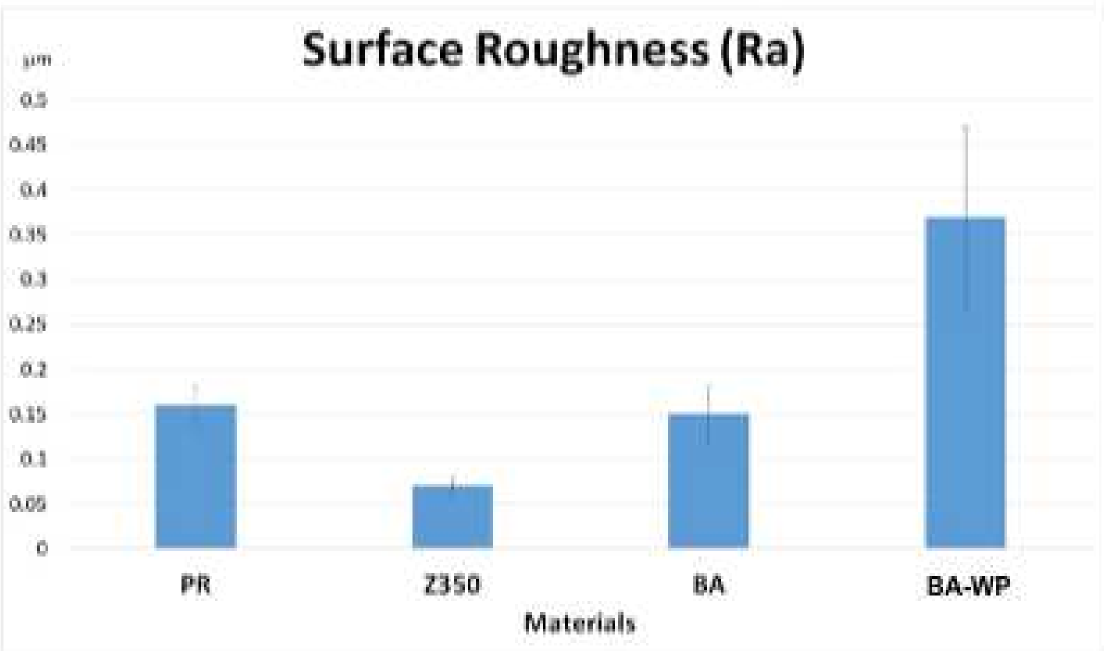


Figure 2. Surface roughness of tested resins.



2.2 ARTICLE 2

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Improvement behavior of a novel 3D-printed resin reinforced by nanofibers of niobium, nanosilver and bioactive glass.

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ABSTRACT

Objectives. In this study hybrid nanofibers embedded with niobium pentoxide, bioactive glass and nanosilver were synthesized, incorporated in a 3D printed resin, and their influence on mechanical, optical and surface properties was evaluated.

Methods. PDLLA and PLA-PEG nanofibers with niobium, bioactive glass or nanosilver were formulated and spun into submicron fibers by solution blow spinning and air brushing technique. Three formulations of nanofiber (PDLLA/Nb_2O_5, PDLLA/Nb_2O_5/SiO and PLA-PEG/SiO/Ag) were combined with a 3D-printable resin for interim restoration (Next Dent C&B) in five mass fractions: 0.3wt%, 0.5wt%, 1wt%, 2.5wt%, 5wt%. Mechanical, optical and surface properties were assessed in flexure by 3-point bending (σ), Knoop hardness (H), roughness (Ra), scanning electron microscopy (SEM) and color stability after aging (ΔE).

Results. There were significant differences among formulations and concentrations for ΔE, σ, H and Ra ($P<0.05$). PDLLA/Nb_2O_5 2.5 wt.%, provided the highest mechanical, surface and optical properties among all materials tested. Ra and H was affected for all groups, except for PDLLA/Nb_2O_5/SiO and lower concentration (0.3wt% and 0.5wt) of PLA-PEG/SiO/Ag.

Significance. Hybrid reinforcement nanofibers are promising as filler for dental materials on 3D printing field. The resin with PDLLA/Nb2O5 nanofibers presented superior performance than the control group for all concentrations tested.

Keywords: Niobium pentoxide; 3D printing; Nanofibers; Nanosilver;

INTRODUCTION

The emerging incorporation of technology has given a digital workflow approach in dentistry. It is evidenced by a predicted increase in the marked size by 2020 to around $5 billion dollars [1]. However, the challenge for the dental materials research community is to link the technology with materials that are suitable for use in dentistry. This can potentially take dental materials research in a totally different direction [2,3].

Recent researches highlight the advantages of rapid and automated prototyping of dental restorations and crowns using a precise computer-aided design (CAD) software and additive manufacturing (3D printing) [4,5,6]. The most widely used method in dentistry is stereolithography (SLA) and the related digital light processing (DLP) technique. Overall, both methods produce less material waste than subtractive manufacturing (CAD-CAM), can reproduce complex geometries, material gradients can theoretically be generated, and have been reported as energy efficient method with relatively low-cost due to passivity of the build process. In this way, Dentistry is widely acknowledged as one of the fields that can greatly benefit from these 3D printing technologies to obtain clinical and restorative tools such as surgical guides, diagnostic models, occlusal splints, provisional restorations and prosthesis. However, to date there are no commercial resins whose properties allow their indication to permanent restorations [7,8].

In addition, questions pertaining to parameters defining the properties of 3D printed restorative dental materials is unknow and it seems like there is still strikingly inferiority compared to subtractive manufacturing [2,9,10,11]. Therefore, development of strategies to improve 3D printed materials properties would be highly favorable [7,12,13].

Resin-based materials have a dentin-like Young modulus (12.8 GPa), allows minimally invasive restorative approaches and has been demonstrated some advantages to ceramics (95 – 210GPa) manly if we consider the equal distribution of masticatory load. Therefore, strong efforts have been made to improve resin-based dental materials with submicron and nanostructures aiming to achieve enhanced long-term clinical performance [14,15,16].

In the field of composite resins, adhesives and resin cements, recent studies have been showed promising results on physical-mechanical properties and biological behavior when reinforced by silica, hydroxyapatite, titanium dioxide and silver [17, 18,19, 20]. The incorporation of nanofibers has been highlighted due to its remarkable physicochemical properties, mainly due to the effects of fiber bridging and pullout, which were able to transfer load more efficiently than particular fillers. Nanofibers are considerably more ductile than inorganic fillers and their extreme reduced diameter result in significant increase in strength [21, 22, 23]. Besides that, can provide a large ratio of surface area to volume, which can enhance the intermolecular hydrogen bonding between the nanofibrous filler and the resin matrix, providing good load transfer between them [24,25]. Also, in some other reports, filler was thought able to reduce polymerization shrinkage benefiting from the overlapping of fibers, and to improve wear resistance [24].

The method to synthetize nanofiber determines their configuration which includes: 3D-scaffolds, foams, fibermats and cotton wool-like. Once inorganic-organic nanofibers can act as a platform for therapeutic ion release, their use in dental materials could be explored to promote a tissue response or strategic environmental changes [25,26]. Currently, the reinforcement of biomaterials with niobium pentoxide (Nb_2O_5) has been highlighted due to its remarkable physicochemical properties and high biocompatibility [27]. It is considered a metal oxide, which presents bioactivity and promoter the growth of hydroxyapatite crystals when in contact with saliva. It also presents high mechanical stability, insoluble in water and similar optical properties to the dental structure [19,28]. Nanofibers of Nb_2O_5 present chemical and mechanical properties highly advantageous, such as mechanical strength, similar optical properties to dental structure, high light conductivity, presents photocatalytic properties and, therefore, high energy absorption. However, its application in Dentistry is still scarce [29,30].

A range of bioactive ions can be explored as a dopant in nanofibers intended to reinforce resin-based materials. The use of bioactive glass (BAG) has been considerate an important strategy in modern regenerative dentistry due to stimulate tissue mineralization [31,32,33,34]. Bioactive glass presents high bioactivity index (I_B = 12.5) and when embedded in nanofibers could promote deposition of hydroxyapatite, enamel and dentin remineralization, act as antimicrobial agent as well as reduce MMP activity when BAG particles was incorporated into resin composites [35,36,37,38].

On the other hand, when nanofibers are doped with nanosilver plays an important role in mechanical properties besides presenting a stronger antibacterial effect against both gram-positive and gram-negative bacteria and may also improve the biological properties of the material [39,40]. Antibacterial activity of materials to provisional or permanent restorations was helpful in controlling secondary caries adjacent to the filling, which was reported the main cause for numerous restoration failures [40].

In order to boost the development of materials applicable to 3D printing with improved mechanical and bioactive properties, the purpose of this study was to synthesize three novel functional nanofibers of (1) PDLLA embedded with orthorhombic niobium pentoxide (PDLLA/Nb_2O_5); (2) PDLLA embedded with orthorhombic niobium pentoxide with network on bioactive glass (PDLLA/Nb_2O_5/SiO_2); and (3) nanofibers doped with bioactive nanosilver glass (PLA-PEG/SiO_2/Ag^3). Then, we evaluated the effects of their incorporation on the overall mechanical and surface properties, and color stability after aging of a dental 3D printable composite for fast prototyping. The null hypothesis was the incorporation of hybrid nanofibers would not influence the Knoop microhardness, flexural strength, color stability and smoothness of a 3D printed resin.

MATERIALS AND METHODS

2.1. Study Design

This study presents three variation factors: (1) Resin (one level – Next Dent™ C&B), (2) nanofibers (three levels: nanofibers of PDLLA embedded with orthorhombic niobium pentoxide; PDLLA embedded with orthorhombic niobium pentoxide with network on bioactive glass; and nanofibers of PLA-PEG doped with bioactive nano-silver glass, and (3) the mass fraction of each formulation (six levels – 0 wt.%; 0,1wt.%; 0,5wt.%; 1wt.%; 2,5wt.% e 5wt.%).

The response variables were: color stability (ΔE), flexural strength (σ), hardness (H), scanning electron microscopy (SEM) and surface roughness (Ra).

2.2 Materials

A commercial 3D printable resin (Crown & Bridge – MFH Next Dent, shade A2) composed of methacrylic oligomers, phosphine oxides and microfillers, was used in this study (Lot Nº XK55N91).

The fibers of PDLLA/Nb_2O_5 and PDLLA/Nb_2O_5/SiO_2 were produced by solution blow technique (Figure 1) according set-up described in previous studies (Zhou P, et al., 2017). To synthetize PDLLA based nanofibers, the parameters for spinning were: 2 mm nozzle protrusion, a work distance of 20 cm at 40ºC, 45 psi pressure, flow rate 7.2 mL/h, internal (1.6mm) and external (3mm) nozzle diameter, and the hood remained with controlled temperature. Cotton-woll-like fiber with three-dimensional Poly (D,L-lactide, PDLLA) (Jamplas Inc., MO, USA – pellet form) were obtained by a rotation collector at ~19 m/s. Nanofibers were formulated and spun into submicron fiber via SBS. Composite fibers were produced with precursor solutions of niobium-filled PDLLA fibers.

A silica precursor was used to produce the inorganic-organic hybrid sol-gel-based fibers using formulations, in the stoichiometric ratio of 1:2:2:0.01 (TEOS, ethanol, H_2O. AND 1 M HCl – Sigma-Aldrich, Germany), respectively in order to hydrolyze. Under constant stirring for 24 h at room temperature the solution was prepared and a second polymer solution with 20% in mass was prepared to contain initially 10 wt.% of niobium oxide powder (99% orthorhombic, Nb_2O_5 – Ogramac Metalização, São Paulo - Brasil) added in 10 mL of Dimethyl carbonate (DMC > 99%, Sigma-Aldrich, Germany) and sonification was performed for 3 h at 8 min intervals every hour.

After, under stirring for 6h at 80ºC, the mass of PDLLA (molecular weight of 120kg/mol) was added to the initial solution. Next, 3.7 mL of the primer solution was slowing dripped onto 7.3 mL of the second solution (30 wt.% of silica) and constant stirring at room temperature per 3 h to result in the organic-inorganic hybrid solution.

To synthetize PLA bioactive nano-silver glass nanofibers (PLA-PEG/SiO_3/Ag^3), the sol-gel inorganic method was used. A solution containing TEOS, ETOH, deionized water in standard 1N HCl solution, in the proportions 1:3:3 (mol/L) were stirring during 6 hours, at room temperature, according to previous studies (Poologasundarampillai G, et al., 2011; Greenhalgh RD, et al., 2017). The hydrolyzes TEOS obtained and the reduce silver solution were mixed in the proportions 30:70, 50:50 and 70:30 (w/v). The 50:50 solution was the one that shown visible homogeneity and there was no phase separation. The solution of the hydrolyzed TEOS + reduced silver was taken to the oven and kept at 600ºC about 4 hours. This process was necessary to avoid organic residues or toxic reagents of the composition, also suggest that the silver ions may have fused the SiO_3 particles and entered the interconnect silica network. Developing a product in the solid phase of bioactive nano-silver glass powder (BNSG).

A polymeric blend solution of 15 wt.% containing poly lactic acid (PLA) and poly ethylene glycol (PEG) in 80:20 w/v (blend proportions) were solubilized in chloroform, constant stirring at a temperature of 55ºC for ~2h. The polymeric blend solution (PLA-PEG) was added bioactive nano-silver glass powder up to solubility limit of 10% and kept under the same conditions for 1 hour. An ultrasonic equipment was used to disperse particles in blend polymer solution. The fibers were produced by air blowing, which uses a simple and low-cost equipment, a professional airbrush cup fixed, 0.3 mm, double action (Model BC 61 – 7cc Reservoir) to produce fibers layer-by-layer until creating the architecture of a no-woven fibermats. The parameters for spinning fibermats layer by layer were: 0.3 mm nozzle standard, a work distance of 40 cm at temperature of 18ºC, 60 psi pressure. Subsequently, 2 ml of PLA-PEG + Bioactive glass solution was poured into the airbrush reservoir and rotated for 40 minutes. The ultrafine nanofibers arranged in layers were successfully obtained according Figure 1.

The nanofibers were measured with open source Image J software based on a minimum of 70 fibers per each sample according previous studies of our research team[16]. The image was obtained by Scanning Electron Microscopy (SEM) (CM 200, Phillps, Netherlands) with electrons acceleration of 200kV.

The samples were prepared by mixing the fibers into the composite. Different mass fractions (0.3, 0.5, 1, 2.5, 5 wt.%) of three formulations of nanofibers were weight with the value corresponding to the composite mass. These percentages were determined in previous pilot studies varying the fibers concentration in order to obtain a sufficient fluidity composite with improved mechanical properties. Fiber were manually added to the composite and mixed for 10s. All samples were light-activated with a third generation LED device (Valo, Ultradent) operating at 1000mW cm^{-2} for 20 s at 1 mm distance. The irradiance was measured previously.

For all tests, samples were prepared by dispensing materials in bi-part teflon molds (10mm diameter: 2mm height), covered with two glass slides on the top and bottom. For the light-curing materials, a third generation polywave LED-LCU (Valo Cordless, Ultradent) with 1000 mW/cm^2 was used during 40s for each sample. The irradiance from the curing light was checked using a radiometer (RD-7, Ecel Indústria e Comércio Ltda) before starting each group.

2.3 Color stability evaluation (ΔE)

Immediately after preparation of the specimens (n = 10), the color of all was recorded with a spectrophotometer *(*Easyshade Vita spectrophotometer, Vita-Zanhnfabrik). First, the spectrophotometer was calibrated according to manufactures instructions. The color change was assessed at different time points using a *CIEL*a*b*-based colorimeter*. The same examiner evaluated the color of all specimens and three consecutive color measurements were made for each specimen in the same area (center of the specimen), until uniformity of the values was observed.

The first measurements were considered the baseline in the evaluation of the color change caused by post-irradiation conversion. The color change caused post-irradiation period was evaluated after 24 hours and 8 days. After 8-day dry storage, the specimens were submitted to artificial aging, and the color stability was evaluated. All color readings were conducted after positioning specimens over a standard white background.

After the baseline color measurement, the specimens were dry-stored at 37^{o}C for 24h water storage in dark canisters, ensuring that the environment was fully protected from light[22]. The color parameters were measured, and the specimens were stored in the same canisters for 7 more days and measurements were performed again. This 8-day color measurement was considered the baseline color in the second phase of the study, in which the color stability after artificial aging was evaluated. For the artificial aging, the specimens were immersed in deionized water and stored at 60ºC for 48h. The specimens were blot-dried and color parameters were measure again.

The ΔE was calculated based on the following equation: $\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*) + (\Delta b^*)^2}$ where ΔL*, Δa*, and Δb* correspond to the color differences observed between the baseline and after the storage period, considering the respective coordinates.

Three color variations were obtained: ΔE obtained after 24h of storage in dry dark canisters (ΔE_{24h}); ΔE after 8 days of storage in dry dark canisters (ΔE_{8days}), and ΔE after artificial aging (ΔE_{aging}). The ΔE_{24h}, ΔE_{8days} and ΔE_{aging} the L*, the a* and b* values obtained immediately after the light activation of the specimens were used to calculated the ΔL*, Δa*, and Δb* respectively. ΔE values equal of higher that 3.3 unit were considered clinically significant and highly noticeable.

2.4 Flexural strength (σ)

The flexural strength (σ) values were determined using a 3-point bending test attached to a Universal Test Machine (Instron 3342, Ilinois Tool Works) with cross-head speed of 0.5 mm min^{-1}. A central load was applied between supporting rods (6-mm span length). The mean σ values and the standard deviations were calculated for each of the materials using the following equation: $\sigma = \frac{3Fl}{2bd^2}$, where F is the loading force at the fracture point, l is the length of the support span (6 mm), b is the width (2mm), and d is the thickness (2 mm).

Ten bars with standard dimensions (8 mm × 2 mm × 2 mm) were fabricated for each material/group and stored at room temperature for 24 hours. The length, width and thickness of all bars were measured following ISO 4049 with adaptations in sample's length in order to standardize the light curing protocol, avoiding overexposure or uncured resin areas.

2.5 Knoop Microhardness (H)

Twenty-four hours after preparation, 90 disk-shaped specimens (n = 6 / per group) were polished using decreasing grit size abrasive papers (600, 1200, 2400 grit, Buehler Ltd). A 0.5 μm diamond paste (Buehler Ltd) was used with a polishing cloth to obtain flat and smooth surfaces. Three indentations were made at the center of each specimen separated by 100μm (Knoop diamond, 50g, 15s), using a digital microhardness tester (HMV–2000, Shimadzu), (Micromet II, Buehler). The built-in software calculated the KHN values based on the dimensions of longest diagonal and on the following formula: KHN = $\frac{14228c}{d^2}$, where *c* is the load in grams and *d* is the length of the longer diagonal in μm.

2.6 Surface roughness (Ra)

Surface roughness was assessed following ISO 4287 guidelines with 0.80-mm cutoff, 0.0001-μm resolution (8-μm range), 0.5 mm/s speed, and total length of 4mm, using a roughness tester (Hommel-Etamic W10 GmbH Nr 3947). The average of three measurements of the x- and y-axes was calculated and adopted as result for each specimen.

2.7 Scanning electron microscopy (SEM)

Fractured samples of flexure strength test were mounted on stubs and analyzed by variable pressure SEM (Aspex Express; FEI Europe, Eindhoven, Holland) at voltage of 15-20 kV and at 1.000 and 3.500 X magnification. Images were captured, recorded, and analyzed. The SEM images were evaluated as a qualitative analysis.

2.8 Statistical Analysis

Normal distribution and equality of variance were checked for all the variables using Shapiro-Wilk test, results for microhardness, roughness and flexural strength were assessed using one-way ANOVA followed by Tukey test. For color stability, data were analyzed using 2-way repeated measurements ANOVA followed by Tukey test. All statistical analysis was performed adopting 5% significance level.

RESULTS

Overall, the incorporation of nanofibers into 3D printed resin resulted in significant changes in H, σ, ΔE and Ra. The results for H, σ and Ra analysis were showed in Table 1. The resin without reinforcement of nanofiber (control group) displayed average flexural strength and hardness as 70.8 MPa and 36.22 KHN, respectively. The main effects plot for H, σ (material and wt.%) could be observed in figure 2.

All the resins with the addition of PDLLA/Nb_2O_5 nanofibers achieved higher H values than the neat resin (control) (*P*>.05). When the mass fraction of PDLLA/Nb2O5 nanofibers increased to 5 wt.%, the hardness property of the composite improved significantly (*P*>.05). There were no difference to PLA-PEG/SiO^2/Ag wt.1%, PLA-PEG/SiO^2/Ag wt.2.5% and PLA-PEG/SiO^2/Ag wt.5% compared to control, although higher values were obtained by mass fraction of PDLLA/Nb_2O_5 1wt.%, 2.5wt.% and 5wt.%, compared to all formulations (*P*>.05). On the contrary, the additional of all formulations of PDLLA/Nb_2O_5/SiO^2(0.3-5wt.%) and, PLA-PEG/SiO^2/Ag 0.3 and 0.5wt.%, did not differ from the control group without nanofiber (*P*>.05).

Higher values were observed by mass fraction of PDLLA/Nb_2O_5 1wt.% and 5wt.% compared to all formulations (*P*>.05) although without significant differences to PDLLA/Nb_2O_5 2.5wt% (figure 2).

The results of roughness analysis showed there is no differences between control and all formulations tested of PDLLA/Nb_2O_5/SiO^2 (*P*>.05). There is no difference between control and mass fraction of PLA-PEG/SiO^2/Ag 0.3wt.%, 0.5wt.% and 1wt.% (*P*>.05). In contrast, the mass fraction of PLA-PEG/SiO^2/Ag 5wt.% presented the higher value of Ra significantly different of control group (*P*>.05) (figure 3).

While the roughness began to descend when the mass fraction of PDLLA/Nb_2O_5 nanofibers were 2.5%. The lowest value of roughness was observed to mass fraction of PDLLA/Nb_2O_5 wt. 2.5% presenting differences between control group and all formulation of PDLLA/Nb_2O_5/SiO^2 and PLA-PEG/SiO^2/Ag (*P*>.05).

For the color change occurring after the post-irradiation storage, all ∆E values were higher than 3.3 units and therefore were considered clinically significant and highly visible. Figure 4 show the influence of formulations over ∆E values for different concentrations tested. At both evaluation periods (24h versus baseline and 24 h ageing versus baseline) the lowest ∆E was observed to PDLLA/NB_2O_5 wt. 2.5%. Although, at 8 days versus baseline, the lowest ∆E was observed to PLA-PEG/SiO_2/Ag wt. 1%, differing to the control group (P>.05).

In overall perspective, the incorporation of PDLLA/Nb_2O_5 provided the highest mechanical, optical properties tested among all mass fraction (wt.) tested, principally to mass fraction of 2.5% with reduction of roughness.

In the figure 5, the fractured surfaces of 3D-printed resin were displayed by SEM observation. In overall, until the range of wt. 5%, PDLLA nanofibers were seen disperses evenly throughout the resin matrix with no hint of aggregation. The presence of PDDLA and PLA on surface layer promotes an excellent interfacial adhesion between the nanofibers and the resin matrix. However, bundles of nanofibers could be detected as the addition mass fraction (2.5 wt.%) of PLA-PEG nanofibers as showed in Figure 5. The occurring of aggregation at high mass fractions of PLA-PEG/SiO_2/Ag nanofibers was responsible for the decreasing surface property of the corresponding 3D-printed resin. To reinforced fractured samples, it is possible to observe that even around the nanofibers is broken, while nanofiber still links two layers they are inserted.

DISCUSSION:

Inorganic-organic nanofibers had been reported as promising fillers for composites with dental applications to improve their performance including mechanical properties, polymerization shrinkage, and wear resistance [14,16,41,42]. Indeed, there is no finding in the literature that report the reinforcement of 3D-printed resins with nanostructures/nanofibers so far. Thus, this is the first exploratory study to understand the series of events relating to incorporation of nanofibers into 3D-printed resin with innovative nanotechnological potential. According the results of this study, the null hypothesis tested were rejected.

One of the procedures that can benefit the most from the recent developments in 3D printing technologies is the fabrication of interim restorations. It increases productivity in the clinic and allows for a more practical way (as little as 15 min) of making interim restorations in the same patient visit of tooth prepare and scan [4,5]. On the other hand, some recent studies have been reported a concern about color stability and smoothness of modern provisional materials (bisacryl resin and 3D-printable resin) used in esthetic restorative procedures. Even if temporarily, a step of this kind of treatment compromising the esthetics it is considered unacceptable to the patient [4,11,]. In general, in degradation process of dental resins, the absorption of aqueous solvents is accompanied by a loss of non-reacted components, erosion of the filler-matrix interface, and plasticization with a reduction in hardness, stiffness, wear resistance, and flexural strength, which may compromise the clinical results of resin materials [43].

From this perspective, according to the results of this study, the addition of PDLLA/Nb2O5 nanofibers at wt. 2.5% and wt. 5% can represent an innovative and promising strategy, owing to the fact that showed improvement of all properties tested. Improvements in mechanical properties shown by present results could be explained by physicochemical properties of Nb_2O_5, such as high wear, corrosion resistance, photocatalytic properties, which improves the light absorption region of the light spectrum and together make it a stable material. Velo et al., 2019, reported an improvement in the degree of conversion and also mechanical properties of a resin cement reinforced by PDLLA/Nb_2O_5. As Nb_2O_5 is a semiconductor oxide and presents high-energy absorption, a higher degree of conversion was expected in the samples with Nb_2O_5 and Nb_2O_5/SiO_2 considering that it improves the extent of the reaction of monomer into polymer [14, 19, 28, 44].

An increase of hardness is expected in the presence of anisotropic fibers considering the polymer network formed although the relationship between the degree of monomer conversion and hardness is not always straightforward. In the present study the changing of the hardness seems depends of the mass fraction (wt. %), the surface area and orientation of fibers [45,46].

Contrarily, the authors believe that the reduction of Nb_2O_5 proportion to allow the addition of bioactive glass in PDLLA/Nb_2O_5/SiO_2 nanofiber was enough to prevent higher improvements in material properties as observed in the PDLLA/Nb_2O_5 groups.

The filler increases the viscosity of the resin reducing the mobility of monomers, which tends to decrease mechanical properties at higher concentrations [47]. However, it was not observed in this study. The worst behavior of high concentrations of PLA-PEG/SiO/Ag (2.5% and 5%) to roughness analysis can be associated as PLA-PEG has a simpler structure than PDLLA which has a semi-crystalline structure, i.e., monomeric chains of organized interatomic spaces. It promotes a network of chains, considering high energy surface of nanofillers reducing their aggregation and improving also mechanical and physical properties as observed [48]. Besides that, silver nanoparticles tend to localize on the outermost layers of the fiber changing its surface and making it rougher. In the higher mass fraction, it can be expressive and contribute to the formation of nanofiber-agglomerations.

The lower flexural strength of PLA-PEG/SiO/Ag and PDLLA/Nb_2O_5 comparing to PDLLA/Nb_2O_5 can be attributed to the presence of silver and silica compounds, which modifies the polymerization behavior, due to the refractive index difference which can influence the reflection and refraction effects that lead to turbidity or opacity [49]. Although the incorporation of all nanofibers tested differ of the control group.

The authors attribute flexural strength improvement due to using fiber instead of fillers as reinforcement in composites allows better load transferability since the fiber produces the bridging effects and can promote higher fracture aspect ratios. A nanofibrous mat interleaved between two layers of a laminate can bridge the two plies even when the matrix is broken, carrying on additional loads [50]. In flexural strength testing, flexural forces are generated to simulate clinical situations where materials need to withstand flexing, especially in the posterior region. Although not confirmed clinically, high flexural strength is desired for these materials that might experience cracking under occlusal stress [51].

The fractured surfaces after flexural strength test showed the nanofibers inside resin matrix and seems were rough. According to Tian et al., 2007, a rough surface suggests the presence of nanofibers effectively deflected crack propagation [25]. The rougher surface fracture suggests energy consumption during fracture, and consequently, enhanced fracture resistance. Indeed, were observed that even though the sample had been fractured, the nanofibers remained intact as observed in

Fig 5, a fact also responsible for minimizing the dispersion of tension and increasing the strength of the material. Even when the matrix around the nanofibers is broken, the nanofibers still link the two layers between which they are inserted. It happens due to nanofibers provide good load transfer between them. It does not occur in the sample of the control group ensuring that the load is transferred to the stronger fiber and this is how the fiber actually works as a reinforcement [44, 47, 48].

Nonetheless, smooth interim restorations are essential to avoid biofilm accumulation and maintain healthy periodontal tissues. Previous studies have shown the 3D-printed resin roughness similar to findings of this study. Scotti et al., 2020 reported an improved behavior of 3D-printed surface properties than bisacryl resin [11]. In general, the incorporation of nanofiber into 3D-printed resin did not affect the roughness, an important finding considering the manufactures indication of interim restorations. The roughness did not reach unacceptable thresholds (0.3μm), which suggests that these differences in smoothness caused by nanofibers incorporation may not have clinical relevance and is according to the expected to provisional materials. A tendency was observed to roughness and color stability in the present study considering the concentration of each nanofiber formulation tested. Its finding corroborates Barakah and Taher (2014), that reported improvement in the color stability of resin composites to the smoothness surfaces [52].

In contrast, Chen et al. (2014) found that the mechanical properties of dental composites could be improved with the incorporation of only a small mass fraction of hydroxyapatite nanofibers. However, it is important to emphasize that resin composition and nanofiber compounds tested differ from the present study [53].

The diameter of the fibers plays an important role in the mechanical properties of composite resin materials. Nanofibers of lower diameter present an average failure strain stayed over 50%, due to this reason, they are considered more ductile [54, 55] PDLLA/Nb_2O_5/SiO and PLA-PEG/SiO_2/Ag present higher diameters (300nm) than the PDLLA/NB_2O_5 (250nm)[16]. The higher diameter can make light penetration difficult since obstructs the passage of light, resulting in lower mechanical properties and higher ΔE compared to PDLLA/Nb_2O_5.

A tendency of color change was observed to the same group at different timepoint evaluations except to PLA-PEG/SiO/Ag which increased ΔE substantially at 24 h aging versus baseline assessment. Although all groups present higher color change than were considered clinically significant and highly visible, it cannot be disregarded that some reinforced groups had ΔE values statistically lower than the control group. According to the results, we can speculate that PLA-PEG/SiO/Ag is more susceptible to hydrothermal degradation than the other groups.

The polymer degradation promotes chemical and atomic structural/organization changes which can be simulated by artificial aging methods [56]. It is considered multifactorial and causes clinical problems such as discoloration, polish loss, opacity increase, loss of adhesion, as well as the appearance of fractures. The color alteration after aging is the one of methods to predict the hydrothermal degradation of resins which is mediated by sorption and solubility, phenomena that produce chemical changes with deleterious effects on the mechanical properties of polymeric materials [57].

A volumetric expansion due to diffusion of solvents into the polymer network leads to a separation of polymeric chains and depends on the composition and microstructure of the materials [56, 57, 58]. Higher Young's modulus and flexural strength of composite resins have been associated to a higher amount of inorganic filler. While a greater organic content has been commonly associated with increased hydrothermal degradation and greater polymerization shrinkage. In addition, defects and porosities may work as stress raisers, frequently associated with failure origin [50].

From a procedural point of view, 3D printing resins have specific viscosity requirements which distinguish them from conventional composites. 3D printer manufacturers face certain challenges: After each exposure cycle, the build platform is raised to ensure that the resin can flow between the gap and the platform and vat. When the viscosity is increased by amount of fillers, gravity and surface tension will no longer be capable to produce a smooth surface, causing the object to be printed incompletely [5,8].

Therefore, a resin with low viscosity is therefore required, while at the same time fillers must be incorporated in order to improve the mechanical properties and achieve radiopacity. Compared to conventional composites, the total volume fraction of fillers must be much smaller to keep a flowable material. According observed to calibrate operator, the viscosity kept low to all groups. However, other studies were necessary to investigate the effect of nanofiber reinforcement of 3D-printed resin in this property specifically.

In contrast to our results, nano-silver and bioactive glass-containing resin composites have been shown improved mechanical properties to conventional composite, and antibacterial activities [53, 59]. Therefore, other studies investigating antibacterial and biological properties of 3D-printed resin reinforced by PDLLA/NB_2O_5/Si and PLA-PEG/SiO/Ag nanofibers are recommended. Besides that, owing to the knowledge that niobium oxide promotes crystal growth and biomimetic mineralization of adjacent tissues, further studies will address it to extend the applications of 3D-printed resin to permanent restorations in clinical practice. Among the limitations of this study is non-processing of specimens on a 3D printer.

Considering the promising results, we present, and with the knowledge that hybrid nanofibers have been successfully processed and offer a promising reinforcement alternative to organic fibers or fillers, the present outcomes encourage more research with hybrid nanofibers in the field of dental materials.

Conclusion

The incorporation of inorganic-organic hybrid fiber embedded with niobium pentoxide, specially to 5 wt.%, provided the highest mechanical, optical and surface properties among all materials tested, which makes them a potential reinforcing agent for 3D-printed resin. In overall, none mass fraction of PDLLA/ Nb_2O_5, PLA-PEG/SiO_2/Ag and PDLLA/Nb_2O_5/SiO worsened the properties compared to 3D-printed resin without reinforcing.

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Disclosure Statement

There are no conflicts of interest with respect to the authorship and/or publication of this work.

TABLES

Table 1. Mean and standard deviation values of 3D-printed resin control and reinforced with nanofibers.

	Microhardness (KHN) (n = 6)	**Flexural Resistence (MPa) (n = 10)**	**Roughness (n = 10)**
Control	36.82 (0.89)^{CD}	70.8 (3.99)^{H}	0.18 (0.05)^{BC}
PDLLA/Nb_2O_5/$SiO^2$0.3%	36.22 (1.36)^{G}	80.16 (9)^{G}	0.15 (0.02)^{CDE}
PDLLA/Nb_2O_5/$SiO^2$0.5%	37.14 (2.91)^{G}	80.22 (8.75)^{G}	0.16 (0.02)^{CD}
PDLLA/Nb_2O_5/$SiO^2$1%	36.93 (2.53)^{G}	80.52 (8.12)^{G}	0.17 (0.01)^{C}
PDLLA/Nb_2O_5/$SiO^2$2.5%	39.14 (5.74)^{G}	98.43 (16.96)^{F}	0.16 (0.01)^{CD}
PDLLA/Nb_2O_5/$SiO^2$5%	39.7 (4.29)^{FG}	118.68 (10.59)^{CDE}	0.17 (0.01)^{C}
PLA-PEG/SiO^2/Ag0.3%	37.37 (2.84)^{G}	115.71 (4.04)^{DE}	0.16 (0.02)^{C}
PLA-PEG/SiO^2/Ag0.5%	37.94 (2.87)^{G}	112.34 (4.54)^{E}	0.16 (0.01)^{C}
PLA-PEG/SiO^2/Ag1%	42.96 (3.79)^{EF}	121.33 (4.98)^{CD}	0.19 (0.05)^{BC}
PLA-PEG/SiO^2/Ag2.5%	46.44 (2.35)^{DE}	124.54 (6.39)^{C}	0.26 (0.08)^{A}
PLA-PEG/SiO^2/Ag5%	43.51 (2.76)^{E}	115.41 (3.88)^{DE}	0.21 (0.05)^{B}
PDLLA/$Nb_2O_5$0.3%	50.11 (3.44)^{CD}	119.07 (6.65)^{CDE}	0.12 (0.03)^{DEF}
PDLLA/$Nb_2O_5$0.5%	50.67 (3.87)^{BC}	123.23 (9.15)^{C}	0.12 (0.05)^{EF}
PDLLA/$Nb_2O_5$1%	54.43 (3.11)^{B}	133.41 (8.45)^{B}	0.1 (0.05)^{FG}
PDLLA/$Nb_2O_5$2.5%	54.29 (2.94)^{B}	131.92 (5.84)^{B}	0.08 (0.02)^{G}
PDLLA/$Nb_2O_5$5%	58.56 (4.07)^{A}	144.9 (10.94)^{A}	0.11 (0.07)^{FG}

Distinct capital letters indicate difference statistically significant ($p \leq 0.05$) among the lines.

FIGURES

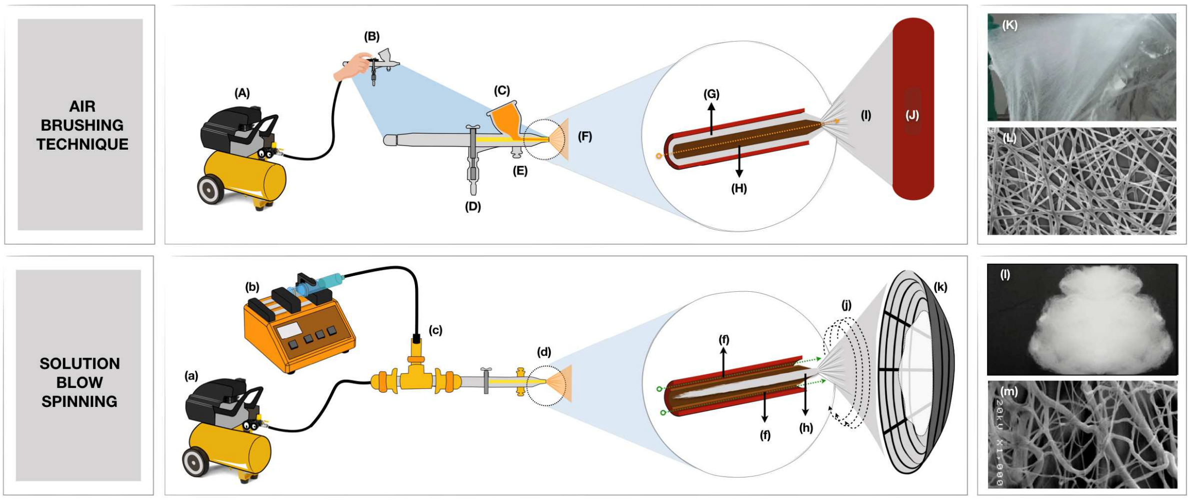

Figure 1. Schematic representation of nanofibers production. Air brushing technique: (A) Air compressor; (B) Airbrush double action trigger; (C) Polymer solution reservoir; (D) Air flow; (E) Internal pressure regulator; (F) Jet outlet; (G) Gas flow; (H) Polymer solution; (I) Jet outlet; (J) Collector; (K) No-woven fibermats layer by layer. Solution Blow Spinning technique: (a) Air compressor; (b) Injection pump; (c) Nozzle; (d) Jet outlet; (f) Gas flow; (h) Polymer solution; (j) Working distance; (k) Collector; (l) cotton wool-like fibers; (m) SEM image of nanofibers.

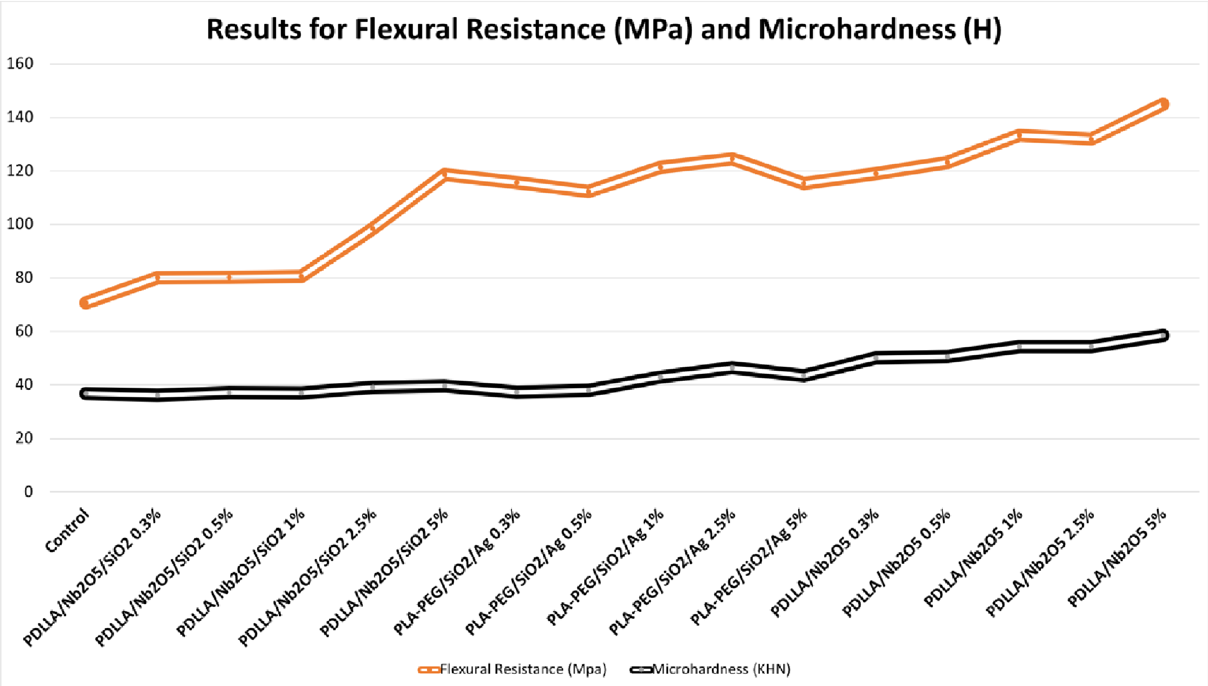

Figure 2. Main effects plot for H and σ of material (3D printed resin + nanofiber formulation) and wt.%

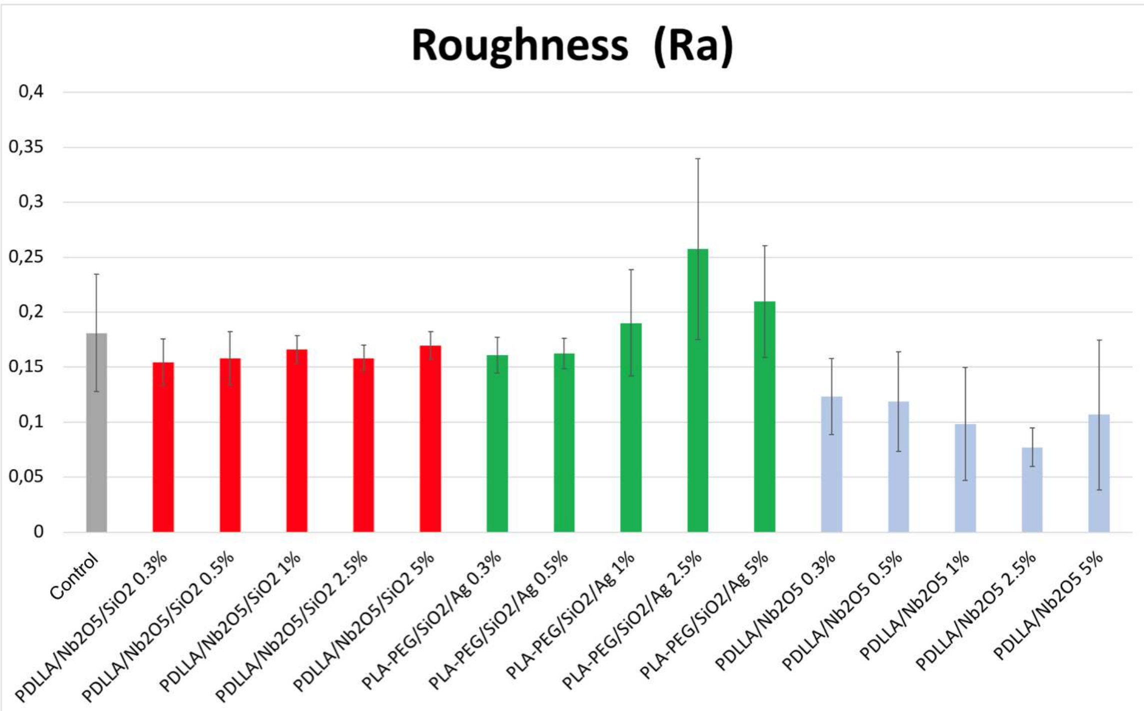

Figure 3. Surface roughness of tested formulations

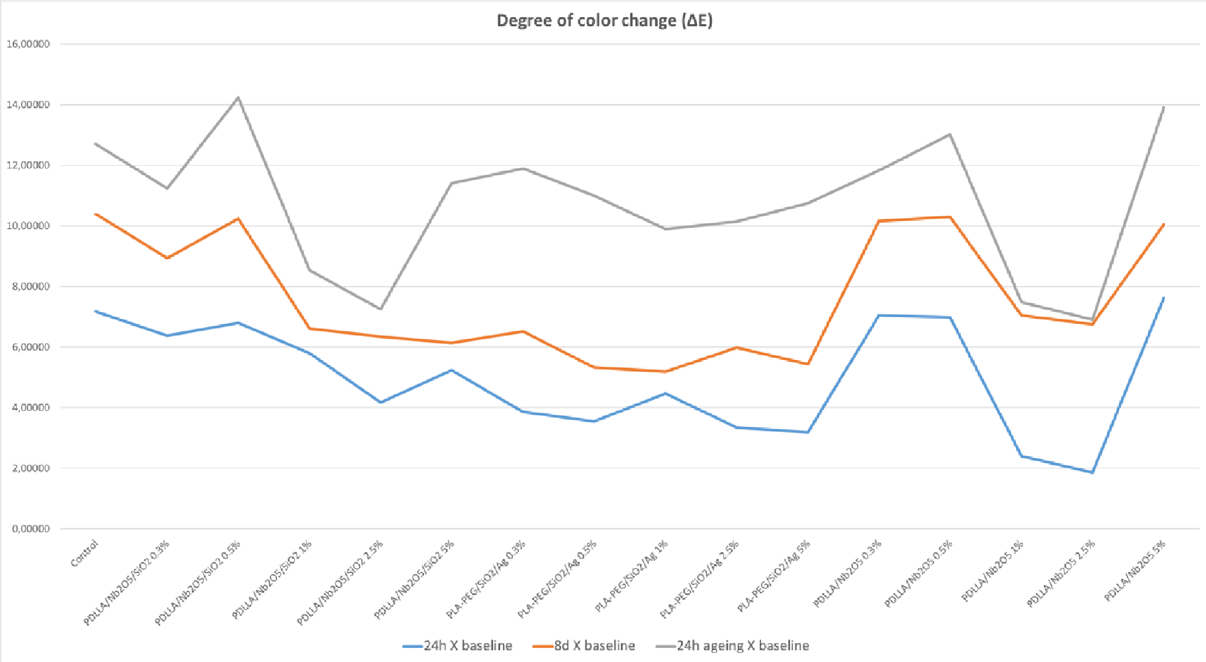

Figure 4. Color difference comparison for tested resins in each evaluation period.

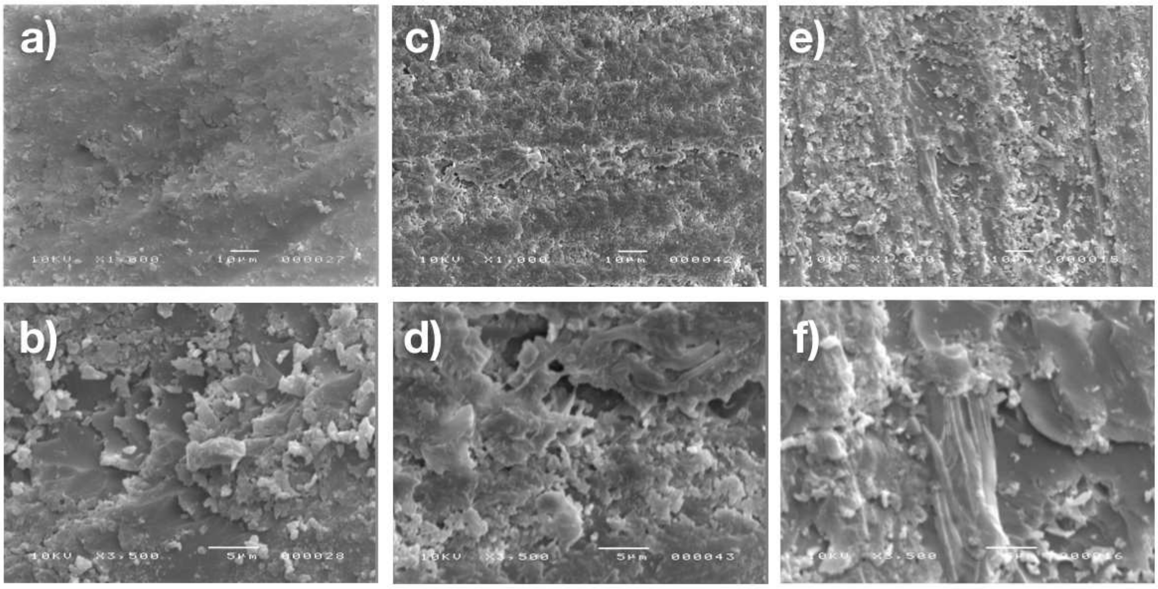

Figure 5. SEM images of fractured surfaces of flexural strength test samples of a 3D-printed reinforced. a) Planar fracture pattern b) Unreinforced 3D-printed resin presenting visible fillers and voids c) Rougher surface fracture pattern of reinforced 3D-printed resin d) Reinforced 3D-printed resin with matrix around nanofiber is broken and nanofiber liking two layers of resin e) Rougher surface of reinforced samples a 2.5 wt. % PLA-PEG/SiO/Ag f) bundles of nanofibers in 2.5 wt. % PLA-PEG/SiO/Ag sample.

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3 Discussion

3 DISCUSSION

The potential increase in productivity and predictability, including the use of planning tools and 3D-printed interim restorations, has promoted the adoption of digital technology (DAWOOD et al., 2015). 3D-printed resins could be a promising material for clinical applications in which bis-acrylic resins have been used.

Based on data from the first study, although the high ΔE could be of concern in an esthetic area, the surface roughness and mechanical properties can be considered more important in ensuring the maintenance of health and the position of the prepared tooth, especially when considering short-term interim restorations (MONDELLI et al., 2018; MONDELLI et al., 2018; SALAR et al., 2018; FALKEMANNER et al., 2013; REVILLA-LEÓN et al., 2019; SINGH et al., 2016; RIZZANTE et al., 2019; TAHAYERI et al., 2018).

The difference among the groups indicated a material-dependent effect. In general, the 3D-printed resin was less color stable after aging than the other tested materials. The color stability of resin-based materials is influenced by factors including the degree of conversion, polarity of monomers, amount of cross-linking, initiator system, particle size and distribution, water sorption, monomer conversion, and pigment stability (MONDELLI et al., 2018; MONDELLI et al., 2018; SALAR et al., 2018; FALKEMANNER et al., 2013).

Nonetheless, data for degree of conversion, polymer cross-linking density, and filler content is still uncertain, and further studies are needed.

The results of the present study suggest that the chemical composition of the interim material affects its mechanical properties, as previously reported (REVILLA-LEÓN et al., 2019). The 3D-printed resin (PR) showed higher mechanical properties (hardness and flexural strength) when compared with the bis-acrylic resin (BA). The results for the bis-acrylic resin were similar to those reported in other studies, although a recent study reported similar flexural resistance of bis-acrylic and an unfilled 3D printed resin (SINGH et al., 2016). Z350XT showed the highest values for mechanical testing, which might be explained by a higher amount of filler content and increased polymerization.

Moreover, the use of the recommended light polymerization protocol could improve the color stability and should be studied. Such restorations might be used for a limited time, explaining why there is no need to use a regular composite resin as an interim restoration. Their superior physicomechanical properties are not usually justified by the additional costs and more time-consuming fabrication nor by their more complex clinical handling as a result of a higher elastic modulus.

On the other hand, considering the results of second study, the addition of PDLLA/Nb2O5 nanofibers at wt. 2.5% can represent an innovative and promising strategy, owing to the fact that showed improvement of all properties tested. Improvements in mechanical properties shown by present results could be explained by physicochemical properties of Nb_2O_5, such as high wear, corrosion resistance, photocatalytic properties, which improves the light absorption region of the light spectrum and together make it a stable material (ALTMANN et al., 2017; MARINS et al., 2019; LEITUNE et al., 2013). Velo et al., 2019 reported an improvement in the degree of conversion and also mechanical properties of a resin cement reinforced by PDLLA/Nb_2O_5. As Nb_2O_5 is a semiconductor oxide and presents high-energy absorption, a higher degree of conversion was expected in the samples with Nb_2O_5 and Nb_2O_5/SiO_2 considering that it improves the extent of the reaction of monomer into polymer.

The worst behavior of high concentrations of PLA-PEG/SiO/Ag (2.5% and 5%) to roughness analysis can be associated as PLA-PEG has a simpler structure than PDLLA which has a semi-crystalline structure, i.e., monomeric chains of organized interatomic spaces. It promotes a network of chains, considering high energy surface of nanofillers reducing their aggregation and improving also mechanical and physical properties as observed (BEHRENS et al., 2016). Besides that, silver nanoparticles tend to localize on the outermost layers of the fiber changing its surface and making it rougher. In the higher mass fraction, it can be expressive and contribute to the formation of nanofiber-agglomerations as highlighted in figure 5.

The lower flexural strength of PLA-PEG/SiO/Ag and PDLLA/Nb_2O_5 comparing to PDLLA/Nb_2O_5 can be attributed to the presence of silver and silica compounds, which modifies the polymerization behavior, due to the refractive index difference which can influence the reflection and refraction effects that lead to turbidity or opacity (HABID E et al., 2017). Although the incorporation of all nanofibers tested differ of the control group.

The authors attribute flexural strength improvement due to using fiber materials instead of fillers as reinforcement in composites is their better load transferability due to the fiber bridging effects and the higher fracture aspect ratios. A nanofibrous mat interleaved between two layers of a laminate can bridge the two plies even when the matrix is broken, carrying on additional loads (YANCEY et al., 2019). In flexural strength testing, flexural forces are generated to simulate clinical situations where materials need to withstand flexing, especially in the posterior region. Although not confirmed clinically, high flexural strength is desired for these materials that might experience cracking under occlusal stress (ILIE et al., 2017).

Nonetheless, smooth interim restorations are essential to avoid biofilm accumulation and maintain healthy periodontal tissues. Previews studies have shown the 3D-printed resin roughness similar to findings of this study. Scotti et al., 2020 reported an improved behavior of 3D-printed surface properties than bisacryl composites. In general, the incorporation of nanofiber into 3D-printed resin did not affect the roughness, an important finding considering the manufactures indication of interim restorations. The roughness did not reach unacceptable thresholds (0.3μm), which suggests that these differences in smoothness caused by nanofibers incorporation may not have clinical relevance and is according to the expected to provisional materials. A tendency was observed to roughness and color stability in the present study considering the concentration of each nanofiber formulation tested. Its finding corroborates Barakah and Taher (2014), that reported improvement in the color stability of resin composites to the smoothness surfaces (BARAKAH AND TAHER, 2014).

In contrast to our results, nano-silver and bioactive glass-containing resin composites have been shown improved mechanical properties to conventional composite, and antibacterial activities (CHENG et al., 2012; MIAO et al., 2017). Therefore, other studies investigating antibacterial and biological properties of 3D-printed resin reinforced by PDLLA/NB_2O_5/Si and PLA-PEG/SiO/Ag nanofibers are recommended. Besides that, owing to the knowledge that niobium oxide promotes crystal growth and biomimetic mineralization of adjacent tissues, further studies will address it to extend the applications of 3D-printed resin to permanent restorations in clinical practice.

Considering the promising results, we present, and with the knowledge that hybrid nanofibers have been successfully processed and offer a promising reinforcement alternative to organic fibers or fillers, the present outcomes encourage more research with hybrid nanofibers in the field of dental materials.

4 Final Considerations

4 FINAL CONSIDERATIONS

The present results focused on 3D-printable resin properties and the effects of their reinforcement with bioactive nanofibers. The main idea was to understand and improved 3D-printable resin to boost workflow approach in dentistry. It was showed *in vitro* that 3D-printed resin had adequate mechanical and surface properties for interim restorative indications. It has the potential to be a low-cost workflow in dentistry, although its color stability can represent a concern for long-term use. Second, it was observed that the incorporation of inorganic-organic hybrid fiber embedded with niobium pentoxide, provided the highest mechanical, surface and optical properties among all materials tested, which makes them a potential reinforcing agent for 3D-printed resin. Therefore, this material can present other behavior from other important properties, thus further studies are necessary.

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ANNEXES

ANNEXE 1

De: em.jpd.0.69aacd.cc699606@editorialmanager.com <em.jpd.0.69aacd.cc699606@editorialmanager.com> em nome de The Journal of Prosthetic Dentistry <em@editorialmanager.com>
Enviado: segunda-feira, 2 de março de 2020 11:14
Para: Fabio A Rizzante
Assunto: Your Submission to The Journal of Prosthetic Dentistry

Mar 02, 2020

Re: Manuscript # JPD-D-19-00233R4

Dear Rizzante:

I am happy to report that the manuscript #JPD-D-19-00233R4, entitled "Physical and surface properties of a 3D-printing-composite for digital workflow.," has been accepted for publication as an online article, pending your responses to the following final requests:

Please download and review the edited manuscript to ensure that your meaning has not been inadvertently altered. Please address the comments.

%ATTACH_FOR_REVIEWER_DEEP_LINK INSTRUCTIONS%

Please revise your manuscript and return it by Apr 13, 2020 so that we can proceed with the review process. We ask that you work from the attached manuscript file, as we wish to preserve certain formatting changes made to the originally submitted document.

Online articles in The Journal of Prosthetic Dentistry are fully indexed for the MEDLINE database maintained by the National Library of Medicine and abstracts are freely accessible via the PubMed online platform. Online articles are also indexed by Thomson Scientific for their Web of Science database. In addition, the Title and Abstract will be printed and indexed in the print journal.

Please make appropriate revisions to the manuscript (see link below) and provide a detailed response to the comments. Distinguish your changes in the manuscript document by putting them in bright blue font. Work directly from the manuscript sent back to you. Please do not otherwise change the format of the manuscript document in any way except to remove Track Changes.

Check the Title Page in the File Inventory to ensure that information is accurate.

The manuscript will go through a final editing process before being formatted for online publishing. If the manuscript editor has any additional questions you will be contacted. Otherwise, you will receive an email when the file has been forwarded to our compositor for layout into proofs.

Please be aware that we have a backlog of print manuscripts. However, online articles are published shortly after the proofs have been corrected. This will help if you have a pressing need for publication.

To submit your revision, please do the following:

Best regards,

Stephen F Rosenstiel, BDS, MSD
Editor
The Journal of Prosthetic Dentistry
http://www.thejpd.org

Online submission and review system:
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The Journal of Prosthetic Dentistry

Physical and surface properties of a 3D-printed composite resin for a digital workflow

--Manuscript Draft--

Manuscript Number:	JPD-D-19-00233R5
Article Type:	Research and Education
Keywords:	3D printing, Digital dentistry, Esthetic.
Corresponding Author:	Fabio A Rizzante, MSc, PhD Case Western Reserve University School of Dental Medicine Cleveland, Ohio UNITED STATES
First Author:	Cassiana Koch Scotti, Ms
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	Marilia MA C Velo, PhD
	Fabio A Rizzante, MSc, PhD
	Tatiana R Nascimento, Ms
	Rafael F Mondelli, Professor, PhD
	Juliana F S Bombonatti, Professor, PhD
Abstract:	Statement of problem. Despite recent researches suggesting the use of 3D printing materials in dentistry, information related to their optical and surface properties, including health compatibility, biofilm accumulation (surface roughness for example), and esthetics is scarce. Purpose. This study aimed to compare the physical and surface properties of 3D printing resin with the properties of different materials used for interim restorations. Material and Methods. A 3D printed resin/PR (NextDent C&B MFH; 3D Systems), a self-polymerizing interim material/BA (Protemp TM 4; 3M ESPE), and a composite resin /Z350 (Filtek Z350XT; 3M ESPE) were tested for: degree of color change/DE (n=7 per group), at different timepoints (24 hours after polishing/baseline/P0, 8 days after polishing/P1, and after artificial aging in water at 60 ºC for 24 hours/P2) using a CIE L*a*b based colorimeter; flexural strength (σ, n=10 per group) using a 3-point bending test; Knoop hardness (H, n=8 per group) using a Knoop indenter; and surface roughness (Ra, n=7) using a profilometer. All specimens were polished 24 hours after polymerization, except for the additional group for surface roughness (BA without polishing/BANP). Statistical analysis were performed using 2-way repeated measures ANOVA followed by Fischer test for DE, and 1-way ANOVA followed Fisher test (P =.05) for microhardness and surface roughness Results. The Z350 showed the highest values for σ and H, followed by PR. BA showed the lowest results for both tests (P <.05). Considering roughness, the Z350 showed similar values to BA, but lower than PR; PR showed similar roughness when compared with BA. PR showed the highest color variation among the groups for all timepoints, followed by BA. The Z350 was the most color stable material for all timepoints. Conclusions. The 3D printing composite presented sufficient mechanical and surface properties to be applied as an interim restorative material. It presents high potential for low-cost workflow in dentistry, although its color stability could be a concern in long-term setting.

ANNEXE 2

DECLARATION OF EXCLUSIVE OF THE ARTICLE IN DISSERTATION/THESIS

We hereby declare that we are aware of the article (Physical and surface properties of a 3D-printing-composite for digital workflow.) will be included in (Thesis) of the student (Cassiana Koch Scotti) was not used and may not be used in other works of Graduate Programs at the Bauru School of Dentistry, University of São Paulo.

Bauru, 10 de Junho de 2020.

Cassiana Koch Scotti | Marília Mattar de Amoêdo Campos Velo | Fabio Antonio Piola Rizzante

Tatiana Rita de Lima Nascimento | Rafael Francisco Lia Mondelli | Juliana Fraga Soares Bombonatti

ANNEXE 3

DECLARATION OF EXCLUSIVE OF THE ARTICLE IN DISSERTATION/THESIS

We hereby declare that we are aware of the article [Improvement behavior of a novel 3D-printed resin reinforced by nanofibers of niobium, nanosilver and bioactive glass] will be included in (Thesis) of the student (Cassiana Koch Scotti) was not used and may not be used in other works of Graduate Programs at the Bauru School of Dentistry, University of São Paulo.

Bauru, 10 de Junho de 2020.

Cassiana Koch Scotti — Marília Mattar de Amoêdo Campos Velo — Fabio Antonio Piola Rizzante

Tatiana Rita de Lima Nascimento — Rafael Francisco Lia Mondelli — Lúcio Roberto Cançado Castellano

Juliana Fraga Soares Bombonatti

ANNEXE 4 – Guide for authors of The Journal of Prosthetic Dentistry

The Journal of Prosthetic Dentistry- 4

Orientações de Submissão

Obrigado pelo seu interesse em escrever um artigo para o *Journal of Prosthetic Dentistry*. No processo de publicação, como em odontologia, procedimentos precisos são essenciais. Sua atenção e complacência com as seguintes políticas ajudará a garantir o processamento atempado da sua submissão.

Comprimento de Manuscritos
Comprimento do manuscrito depende do tipo. Artigos de pesquisa e ciência clínicos gerais não deve exceder 10 a 12 páginas, escritos em espaço duplo (excluindo referências, legendas e tabelas). Relatórios Clínicos e Técnicas Dentárias não deve exceder 4 a 5 páginas, e conselhos dos nossos leitores não deve exceder 1 a 2 páginas. O comprimento varia de revisões sistemáticas.

Número de Autores
O número de autores é limitado a 4, inclusão *de mais de 4 deve ser justificada* na carta de submissão. (Contribuição de cada autor deve ser anotado) Caso contrário, autores acima de 4 serão listados nos agradecimentos.

Formatação Geral
Todas as submissões devem ser enviadas através do sistema de EES em Microsoft Word ou num formato compatível com Microsoft Word usando páginas de 8.5 X 11 polegadas em tamanho. As seguintes especificações deve ser seguido:

- Times Roman, 12 pt
- Espaço duplo
- Justificado à esquerda
- Margens de 1 polegada (2,5cm) em todos os lados da página
- Tabulação de meia polegada (1,25cm)
- Cabeçalhos/rodapés deve ser livre de números de páginas ou qualquer outra informação
- Referências; não deve ser numerados automaticamente (formatado).
- Defina a linguagem em MS Word para Inglês (EUA).

Orientações de Submissão

Obrigado pelo seu interesse em escrever um artigo para o *Journal of Prosthetic Dentistry*. No processo de publicação, como em odontologia, procedimentos precisos são essenciais. Sua atenção e complacência com as seguintes políticas ajudará a garantir o processamento atempado da sua submissão.

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- Times Roman, 12 pt
- Espaço duplo
- Justificado à esquerda
- Margens de 1 polegada (2,5cm) em todos os lados da página
- Tabulação de meia polegada (1,25cm)
- Cabeçalhos/rodapés deve ser livre de números de páginas ou qualquer outra informação
- Referências; não deve ser numerados automaticamente (formatado).
- Defina a linguagem em MS Word para Inglês (EUA).

Tipos de Artigos

Os artigos são classificados da seguinte maneira: Relatório de Pesquisa/Casos Clínicos, Relatório Clínico, Técnica Dentária, Revisão Sistemática, ou Concelhos dos Nossos Leitores. Seções necessárias para cada tipo de artigo são listados na ordem em que devem ser apresentados.

RELATÓRIO DE PESQUISA/ESTUDO CLÍNICO

O relatório da pesquisa não deve ser mais de 10-12 páginas digitadas em espaço duplo e deve ser acompanhado por não mais de 12 ilustrações de alta qualidade. Evite o uso de forma de esboço (ou seja enumerações e/ou frases ou parágrafos com marcadores). O texto deve ser escrito em frases completes e em forma de parágrafo.

- ***Abstract* (Abstrato)**: (aproximadamente 250 palavras): Crie um resumo estruturado com os seguintes subseções: *Statement of the Problem* (Declaração do Problema), *Objective* (Objetivo), *Materials and Methods* (Métodos e Materiais), *Results* (Resultados) e *Conclusions* (Conclusões). O abstrato deve conter detalhes suficientes para descrever o experimento e os variáveis do projeto. O tamanho da amostra, os controles, o método de medição, estandardização, confiabilidade examinador, e método estatístico utilizado com nível de significância associado deve ser descritos na seção de Materiais e Métodos. Valores reais devem ser fornecido na seção de Resultados.
- ***Clinical Implications* (Implicações Clínicas)**: Em 2-4 frases, descreva o impacto dos resultados do estudo sobre prática clínica.
- ***Introduction* (Introdução)**: Explique o problema completamente com precisão. Resuma a literatura relevante, e identifique qualquer viés em estudos anteriores. Declare claramente o objetivo do estudo e a hipótese da pesquisa no final da introdução. Observe que, numa profunda revisão da literatura, a maioria das referências (se não todas) devem ser citadas na seção Materiais e Métodos e/ou na Introdução.
- ***Materials and Methods* (Materiais e Métodos)**: No parágrafo inicial, forneça uma visão geral do experimento. Forneça informações completas de todos os produtos de fabricação e instrumentos utilizados, entre parênteses ou em uma tabela. Descreva o que foi medido, como foi medido, e as unidades de medida utilizadas. Liste os critérios para julgamento quantitativo. Descreva o designo experimental e variáveis, incluindo critérios definidos para controlar variáveis, estandardizar os testes, a alocação de espécimes/sujeitos a grupos (método de randomização), o tamanho total da amostra, controles, calibração dos examinadores, e confiabilidade de instrumentos e examinadores. Descreva como o tamanho das amostras foi determinada (por exemplo, com a análise de força (*power analysis*)). Evite o uso de números para identificar grupos. Em vez, use abreviações ou códigos que claramente indicaram as características do grupo e assim, os grupos serão mais significativo para o leitor. Os testes estatísticos e níveis de significância associado devem ser descrito no final desta seção.
- ***Results* (Resultados)**: Descreva com precisão e brevemente, na mesma ordem que os testes foram descritos na seção de Materiais e Métodos. Para uma listagem extensa, os dados poderão ser apresentados em forma tabular ou forma gráfica para ajudar o leitor. Para *1-way ANOVA* apresente *df*, e valores de F e P nas áreas apropriada no texto. Para todas as outras *ANOVA*s, de acordo com as orientações, forneça a tabela ANOVA. Descreva os resultados e as tendências mais significativas. Texto, tabelas e figuras não devem repetir ao outro. Resultados notados como significativos devem ser validados por dados atuais e valores P.

- ***Discussion* (Discussão)**: Discuta os resultados do estudo, em relação à hipótese e a relevante literatura. A discussão deve começar por explicar se sim ou não há suporte a rejeitar a hipótese nula. Se os resultados não concordam com outros estudos e/ou com opiniões aceites, declare como, e porquê os resultados são diferentes. Resultados concordantes com outros estudos também devem ser declarados. Identifique as limitações do seu estudo e sugere pesquisas futuras.
- ***Conclusion* (Conclusão)**: Liste concisamente conclusões da pesquisa que possam ser retiradas do seu estudo, não simplesmente reafirmar os resultados. As conclusões devem ser pertinentes aos objetivos e justificado pelos dados. Na maioria das situações, as conclusões são só verdade para a população do experimento. Todas as conclusões devem ser acompanhadas por análises estatísticas
- ***References* (Referências)**: Consulte a página 9 para obter mais orientações, página 22 para amostras.
- **Tables (Tabelas)**: Construir tabelas de acordo com as orientações na página 11.
- ***Legends for Illustrations* (Legendas para as Ilustrações)**: Descreva de forma concisa cada ilustração sem diretamente duplicar o texto. Consulte a página 13 para obter mais orientações; página 23 para a página de amostra de legendas.

RELATÓRIO CLÍNICO

O relatório clínico descreve os métodos do autor para cumprir um tratamento difícil dum paciente; não deve ser mais de 4 a 5 páginas, espaço duplo, e deve ser acompanhado por não mais do que 8 ilustrações de alta qualidade . Em algumas situações, o editor pode aprovar a publicação de figuras adicionais se contribuírem significativamente ao manuscrito.

- ***Abstract* (Abstrato)**: Forneça um curto abstrato, sem estrutura, num parágrafo que brevemente resume o problema encontrado e tratamento administrado.
- ***Introduction* (Introdução)**: Resuma a literatura relevante para o problema encontrado, incluindo referências de tratamentos e protocolos padrão. Por favor note que a maioria das referências, se não todas, devem ser citadas na introdução e/ou na seção Relatório Clínico.
- ***Clinical Report* (Relatório Clínico)**: Descreva o paciente, o problema com o qual ele/ela apresentou, e qualquer história médica ou odontológica relevante. Descreva as várias opções de tratamento e as razões para tratamento escolhido. Descreva completamente o tratamento, a duração do período de acompanhamento, e melhorias notáveis resultado do tratamento. Esta seção deve ser escrito no passado e em forma de parágrafo.
- **Discussion (Discussão)**: Comente sobre as vantagens e desvantagens do tratamento escolhido e descreva qualquer contraindicações do tratamento. Se o texto torna repetitivo, omita a discussão.
- ***Summary* (Sumário)**: Resume brevemente o tratamento do paciente.
- ***References* (Referências)**: Selecione e escreva referências bibliográficas de acordo com as orientações da página 10.
- ***Legends for illustrations* (Legendas para as ilustrações)**: Descreva de forma concisa cada ilustração sem diretamente duplicar o texto principal.

TÉCNICA DENTÁRIA

Um artigo sobre uma técnica dentária deve ser apresentada num formato de passo-a-passo, um procedimento único, útil para profissionais de odontologia. Não deve ser mais de 4 a 5 páginas digitadas, em espaço duplo, e ser acompanhado por não mais de 8 ilustrações de alta qualidade. Em algumas

CONSELHOS DOS NOSSO LEITORES

Conselhos dos nossos leitores são breves relatórios sobre procedimentos úteis ou que economizam tempo. Devem ser limitado a 2 autores, não mais do que 250 palavras, e incluem não mais de 2 ilustrações de alta qualidade. Descreva o procedimento num formato numerado de passo-a-passo, escreva o texto em forma ativa, em vez de forma passiva (por exemplo, "Survey the diagnostic cast" em vés de "The diagnostic cast was surveyed.")

Instruções de Formato

ARRANJO da PRIMEIRA PÁGINA – Página Título (*Title Page)*

Por favor, veja o exemplo da página título no Apêndice I (página 19).

- **Título**: O título deve definir a ideia do estudo, o conteúdo do estudo, e significado clínico. Utilize letra maiúscula apenas na primeira letra da primeira palavra. Não sublinhar o título. Abreviaturas ou nomes comerciais não deve ser usado no título. Palavras como '*new*', '*novel*', ou '*simple*' não são recomendados para o título.
- **Autores**: Diretamente sobre o título, escreva os nomes e títulos dos autores. Liste somente os graus académicos. Por favor não use denominações de associações.
- **Instituições**: Diretamente sobre os nomes dos autores, escreva a afiliação institucional e as cidades, estados ou países (se não os Estados Unidos) em que estas instituições são localizadas. Se necessário, inclua a tradução do nome da instituição. Se os autores não são afiliados com uma instituição, por favor, liste a cidade, estado ou país (se não os Estados Unidos), em que os autores vivem.
- **Apresentação/informações de suporte financeiro e títulos**: Se a pesquisa foi apresentada antes numa reunião, escreva o nome da organização, o local, e data da reunião. Se o trabalho foi apoiado por uma bolsa de estudo ou qualquer outro tipo de financiamento, forneça o nome da organização de suporte e o número de concessão. Liste os títulos acadêmicos (por exemplo, *Assistant Professor*) e afiliações departamental de todos os autores.
- **Informações de contato:** Liste o endereço para correspondência, telefone comercial, número de fax, e e-mail do autor onde receberá a correspondência.

ABSTRATO

- O abstrato deve ser escrito numa página separada do texto principal.
- O abstrato não deve incluir abreviaturas ou informações de fabricação.

TEXTO PRINCIPAL

Cabeçalhos

- Os cabeçalhos devem contribuir a clareza do artigo e mudança de uma seção para outra (por exemplo, da discussão para conclusões).
- O uso de subtítulos podem ser apropriados para seção de Materiais e Métodos, mas é geralmente desencorajado nos Resultados e Discussão.
- Todos os cabeçalhos devem ser alinhados com a margem esquerda. Cabeçalhos principais (por exemplo, "*MATERIALS AND METHODS*") devem ser escrito em letras maiúsculas, subtítulos (por exemplo, "Specimen preparation" deve ser escrito com a primeira letra maiúscula e o restante da frase em letras minúsculas.)

Informações de identificação de produto e sua manufatura

- Descreva produtos em termos genéricos. Imediatamente após a palavra, forneça as seguintes informações em parênteses: nome do produto e do fabricante; por exemplo: *"The impression was poured in Type IV stone (Denstone; Heraeus Kulzer) and related to each other with a fastsetting vinyl polysiloxane occlusal registration material (Correct VPS Bite Registration; Jeneric/Pentron, Inc)."* Por favor, note que há um ponto e vírgula após o nome do produto. Nós já não exigemos a cidade e estado/País para cada fabricante que esta informação muda com tempo e é facil de encontrar na rede.
- Não use símbolos de marca registrada, não são consistentes com estilo do Jornal.
- Use nomes de medicamentos genéricos; os nomes comerciais podem ser mencionados em parênteses na primeira menção.

Abreviaturas

- Se abreviaturas foram utilizadas, forneça a forma expandida na primeira menção e abreviar daí em diante, por exemplo, "*fixed dental prosthesis* (FDP)".

Referências

Referências aceitáveis e a sua colocação no documento

- A maioria das referências, se não todas, devem ser citada na introdução e/ou na seção de Materiais e Métodos. Apenas aquelas referências que foram citadas anteriormente ou que se relacionam diretamente aos resultados do estudo podem ser citados na discussão.
- Só os artigos publicados que foram revisados por pares podem ser usado como referência. Manuscritos em preparação, manuscritos submetidos para consideração e teses não publicadas não são referências aceitáveis.
- Os abstratos são considerados observações não publicadas e não são permitidos como referência a não ser que estudos de acompanhamento foram publicados em revistas revisadas por pares.
- **A referência de publicações em língua estrangeira devem ser mantidas a um mínimo (não mais que 3). Estas referências são permitidas apenas quando o artigo original foi traduzido para Inglês.** O título traduzido deve ser citado e a língua original deve ser mencionada entre parênteses na citação ao final.
- Referências de livros didáticos devem ser mantidas a um mínimo; livros didáticos muitas vezes refletem as opiniões dos seus autores e/ou editores. Quando necessário, as edições mais recentes

dos livros didáticos devem ser utilizadas de preferência. Periódicos baseados em evidência científica são preferidos.

Formatação de Referências

- As referências devem ser identificadas no corpo do artigo, com números arábicos sobrescritos. O número da referência deve ser posto após o período no final da frase.
- A lista das referências completa deve ser em espaço duplo e em ordem numérica, deve seguir a seção de conclusões mas começar numa página separada. Apenas as referências citadas no texto devem aparecer na lista das referências.
- Formatação das referências devem acordar com o estilo **Vancouver**, conforme estabelecido no "Uniform Requirements for Manuscripts Submitted to Biomedical Journals" (Ann Intern Med 1997;126:36-47).
- As referências devem ser numeradas manualmente.
- Liste até seis autores. Se houver sete ou mais, após o sexto nome, adicione *et al.*
- Nome do jornal será abreviado de acordo com **Cumulative Index Medicus**. Uma lista completa de abreviaturas está disponível através do site do PubMed: http://www.ncbi.nlm.nih.gov/nlmcatalog/journals
- Formato para artigos: forneça os sobrenomes e iniciais de todos os autores, o título do artigo, o nome do periódico; e, o ano, volume e números das página de publicação. Não utilize itálico, letras realçadas ou sublinhadas para qualquer parte da referência. Coloque um período após os iniciais do último autor, após o título do artigo, e no final da referência. Coloque um ponto e virgule após o ano de publicação e uma vírgula após o volume. Números de emissão não são usado em estilo **Vancouver**.

Exemplo: Jones ER, Smith IM, Doe JQ. Uses of acrylic resin. J Prosthet Dent 1985; 53:120-9.

- Referências dos livros: A edição mais atual deve ser citada. Forneça os nomes e iniciais de todos os autores/editores, o título do livro, a cidade de publicação, a editora, o ano de publicação e os números das página consultadas. Não use itálico, letras realçadas ou sublinhadas para qualquer parte da referência.

Exemplo: Zarb GA, Carlsson GE, Bolender CL. Boucher's prosthodontic treatment for edentulous patients. 11th ed. St. Louis: Mosby; 1997. p. 112-23.

*Um exemplo duma página de referências pode ser encontrado na página 21.

IMPORTANTE

As referências não devem ser submetidas em Endnote ou de qualquer outro software bibliográfico. Essa formatação não pode ser editado pela Oficina Editorial ou revisores, e devem ser suprimidos ou removidos do manuscrito antes de sua submissão. As referências nem devem ser numerados automaticamente.

TABELAS

- As tabelas devem complementar, e não duplicar, o texto.

- Todas as tabelas devem ser postas no final do manuscrito, após a lista de referências e antes das Legendas. Deve haver apenas uma tabela por página. Omita linhas horizontais e verticais. Omita qualquer sombreado ou cor.
- Não liste as tabelas em partes (por exemplo, Tables Ia, Ib, *etc.*) Cada tabela deve ter o seu próprio número. Numerar cada tabela na ordem em que são mencionadas no texto.
- Forneça uma legenda concisa que descreve o conteúdo da tabela. Crie nomes para cabeçalhos e coluna descritivos. Dentro de colunas, alinhar os dados de tal forma que os pontos decimais estão numa linha reta. Use pontos decimais (períodos), e não vírgulas, para marcar lugares passado o número inteiro (por exemplo, 3.5 em vez de 3,5).
- Numa linha de baixo da tabela, defina qualquer abreviaturas utilizadas na tabela.
- Se uma tabela (ou qualquer dado dentro dela) foi publicado anteriormente; dê todo o crédito ao autor original no rodapé. Se necessário, obtenha permissão para reimprimir a tabela do autor /editor.
- As tabelas devem ser submetidas em *Microsoft Word* ou formato compatível. *Microsoft Word* é preferido. Se uma tabela foi criada em *Excel*, deve ser importados para um dos formatos referidos acima antes de submissão.

SUBMISSÃO DE IMAGENS ELECTRÔNICAS

Tipo de Arquivo

Todas as figuras devem ser enviadas arquivadas em *Tagged Image File Format (TIFF)*. As figuras não devem ser submetidos com *Microsoft Word, Corel Draw, Harvard Graphics, PowerPoint*, ou outros formatos de software de apresentação. Desenhos ou outros trabalhos de arte são melhores submetidos no formato original como *EPS (Encapsulated PostScript), Adobe Illustrator, InDesign, etc.* **Antes de submissão, deve ser guardado como um .TIFF.**

Especificações do Arquivo da Imagem

Dimensões da figura deve ser ao mínimo de 4 × 6 polegadas (10 X 15 cm).

Todas as figuras devem ser do mesmo tamanho (o mesmo tamanho físico), a não ser que o tipo da imagem proíbe ser do mesmo tamanho das outras figuras dentro do manuscrito, como no caso duma radiografia panorâmica ou radiografias peri-apical, imagens SEM, ou gráficos e capturas de tela. Não marque nos rostos das figuras com letras ou números para indicar a ordem em que as figuras devem aparecer; tais legendas serão postas durante o processo de publicação.

Resolução

As fotos devem ser de qualidade profissional e de alta resolução. A seguir estão as orientações de resolução:

- Fotografias em preto-e-branco ou a cores devem ser criados e guardados no mínimo de 300 pontos por polegada (dpi). (Note: Uma imagem de 4X6 polegadas com uma resolução de 300 dpi será aproximadamente 6 megabytes. Uma figura de menos de 300 dpi não deve ser aumentada artificialmente a 300 dpi, a qualidade e resolução resultante será pobre.

- Desenhos de linhas devem ser criados e guardado em 1200 dpi.
- Um trabalho artístico em combinação (uma ilustração que contem ambas fotografias e desenho de linha) deve ser criado e guardado em 600-1000 dpi.
- Claridade, contraste, e a qualidade deve ser uniforme entre as partes de uma figura multiparte, e entre todas as figuras dentro do manuscrito.
- Figuras compostas (várias imagens combinadas em um único composição) não são aceitáveis. Cada parte da imagem deve ser 4 × 6 polegadas, com 300 dpi.
- O fundo da imagem deve ser uniforme, sem textura, azul médio quando possível.

Texto dentro de imagens

Se texto é para aparecer dentro duma figura, versões marcadas e não marcadas devem ser fornecida. O texto que aparece nas versões marcadas devem ser em **fonte Ariel e ao mínimo 10 pt em tamanho**. O texto deve ser dimensionado para facilitar legibilidade, se a figura é reduzida para produção no Jornal. As letras devem ser em proporção com desenho, gráfico ou fotografia. O tamanho de fonte deve ser consistente entre cada figura, e para todas as figuras. Note que os títulos e subtítulos não devem aparecer no arquivo de figura, mas serão fornecidas no texto manuscrito (ver Legendas de Figuras, abaixo).

Se uma chave para uma ilustração requer obras de arte (linhas de tela, pontos, símbolos especiais), a chave deve ser incorporada no desenho, em vez de ser incluída na legenda. Todos os símbolos devam ser feitos profissionalmente, devem ser visível contra o fundo da imagem, e ser de proporção legível se a ilustração é reduzida para publicação.

Todas as fotografias de imagens de microscópicas devem ter uma barra de medida e unidade de medida na imagem.

Figuras em Cor

Ilustrações coloridas podem ser submetidas quando o seu uso aumenta consideravelmente o valor do manuscrito. **O editor tem a autoridade final para determinar se as ilustrações coloridas fornecem uma apresentação mais eficaz.** Geralmente, um máximo de 8 figuras são aceites para um relatório clínico e artigos de técnica dentária, e 2 figuras são aceites para conselhos para nosso leitores. Mas, o Editor pode aprovar a publicação de figuras adicionais, se elas contribuem significativamente para o manuscrito.

Figuras clínicas devem ser de cor equilibrada. Imagens coloridas devem ser em CMYK (Ciano/Magenta/Amarelo/Preto) formato de cor invés de RGB formato de cor (vermelho/verde/azul).

Gráficos

Os gráficos devem ser numerados como figuras e o enchimento nos gráficos de barras deve ser distinto e sólidos; sombreamento e desenhos devem ser evitada. Linhas grossas e sólidas devem ser usadas e em letras realçadas e sólidas. **Fonte Times New Roman é o preferido**. Coloque letras num fundo branco e evite o reverso (letras brancas sobre um fundo escuro). **Imagens de 1200 dpi devem ser fornecidas, se forem preto e branco.**

A Jornal reserva o direito de uniformizar o formato dos gráficos e tabelas.

Nomeação de Arquivos

Cada figura deve ser numerados de acordo com a sua posição no texto (Figure 1, Figure 2, e assim), usando algarismos arábicos. Os arquivos das imagens electrónicas devem ser nomeados de modo que o número da figura e formato pode ser facilmente identificado. Por exemplo, figura 1 no formato TIFF deve ser nomeado *fig1.tif*. Figuras com várias componentes devem ser claramente identificáveis pelos nomes de arquivo: Figura 1A, Fig 1B, Fig 1C, *etc.*

No artigo, referência claramente cada ilustração, incluindo o seu número entre parênteses no final da frase apropriada antes de fechar pontuação. Por exemplo: " The sutures were removed after 3 weeks (Fig. 4)."

Legendas de Figuras

As legendas das figuras devem aparecer no texto do manuscrito numa página separada após as Referências e Tabelas e referências devem aparecer sobre o título "Legends". O estilo do Jornal requer que os artigos (*a, an, e the*) são omitidos nas legendas de figuras e tabelas.

Se uma ilustração é tirada de material já publicado, a legenda deve dar todo o crédito a autor original (consulte Permissões).

Os autores são obrigados a revelar se ilustrações foram modificados em qualquer forma.

PERMISSÕES

- Todo o material citado deve ser claramente marcado com aspas e uma referência numérica. Se mais de 5 linhas são citados, uma carta de autorização deve ser obtida do autor e editor do material citado.
- Todos os manuscritos são submetidos para um software que identifica semelhanças entre o manuscrito submetidos e trabalhos anteriormente publicados.
- Se as citações são mais do que um parágrafo de comprimento, abra aspas no início de cada parágrafo e fecha aspas perto apenas no último parágrafo.
- Escreva todo o material citado exatamente como aparece na publicação original, sem alterações em ortografia ou pontuação. Indique o material omitido duma citação com reticências (três pontos) para omissão de material dentro de uma frase, 4 pontos para o material omitido após o fim duma frase.
- As fotografias que incluem os olhos dum paciente, o paciente deve assinar um consentimento autorizando o uso de seu/sua foto no Jornal. Se tal permissão não foi obtido, os olhos serão bloqueados com barras pretas na publicação.
- As ilustrações que são reimpressas ou emprestadas de outros artigos ou livros publicados não podem ser utilizados sem a permissão do autor original e editor. O autor do manuscrito deve garantir essa permissão e enviá-la para revisão. Na legenda da ilustração, forneça a citação completa da fonte original entre parênteses.

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INTERESSE COMERCIAL EM EMPRESAS E/OU PRODUTOS

- Autores não podem diretamente ou indiretamente fazer reclame aos equipamentos, instrumentos ou produtos em que eles têm um investimento pessoal.
- Declarações e opiniões expressadas nos manuscritos são as dos autores e não necessariamente aqueles dos editores. Os editores não assumem qualquer responsabilidade por tais materiais. Os editores não garantem ou endossam qualquer produto ou serviço anunciado no jornal; os editores não garantem qualquer alegação feita pelo fabricante sobre esse produto ou serviço.
- Autores devem divulgar qualquer interesse financeiro que eles podem ter nos produtos mencionados no artigo. Esta divulgação deve ser mencionada após a seção das conclusões.

Orientações de Escrita

REGRAS GERAIS E SUGESTÕES

- Autores que sua língua materna não é inglês devem obter a assistência dum especialista em escrita científica e inglês antes de submeter seu manuscrito. Manuscritos que não contem os padrões de linguagem básica serão retornados antes de revisão.
- Jornal não usa linguagem na primeira pessoa (*I, we, us, our, etc.*). "*We conducted the study*" pode facilmente alterado para "*The study was conducted.*"
- Evite o uso de termos subjetivos, tais como "*extremely*", "*innovative*" *etc.*
- O Jornal utiliza a vírgula serial, uma vírgula que é posta antecede da conjunção antes do último artigo numa lista de três ou mais: "*The tooth was prepared with a diamond rotary instrument, carbide bur, and carbide finishing bur.*"
- Preferimos a forma não possessiva de epônimos: "*The Tukey Test*" em invés de "*Tukey's Test*", "*Down Syndrome*" em vez de "*Down's Syndrome*" assim por diante.
- Descreva os procedimentos experimentais, tratamentos, e resultados no tempo passivo. Tudo o resto deve ser escrito numa voz ativa.
- Descreva os dentes pelo seu nome (por exemplo, Maxillary right first molar), não seu número.
- Hífens não são usados para sufixos e prefixos comuns, a não ser que o seu uso é fundamental para compreender a palavra. Alguns prefixos com os quais nós não usamos hifens incluem: *pre-, non-, anti-, multi-, auto-, inter-, intra-, peri-.*
- Elimina o uso de *i.e* .ou *e.g*; eles não são consistentes com o estilo do Jornal.
- É geralmente melhor paráfrase a informação duma publicação em vez de usar citações diretas. Parafraseando economiza espaço. A exceção é uma citação direta que é invulgarmente pontiagudo e concisa.
- As palavras compridas com abreviaturas padrões (como em *TMJ* para *temporomandibular joint*) são usadas frequentemente, use a palavra completa e forneça a abreviatura entre parênteses. Use a abreviatura de lá em frente. Acrônimos comuns devem ser definidos na primeira menção.
- Nós não usamos itálico para palavras estrangeiras como "*in vivo*", "*in vitro*"
- Abreviar unidades de medida sem um ponto no texto e nas tabelas (*9 mm*). Por favor, introduza um espaço não separável entre todos os números e suas unidades (*100mm, 25MPa*) exceto antes **%** e **°C**. Nunca deve haver um hífen entre o número e a abreviatura ou símbolo, exceto quando em forma adjetiva (100-mm span).

- Escreva a palavra completa "*degree*" quando fala sobre anglos. Use o símbolo de grau somente para temperatura.
- Para os resultados estatísticos comuns P, α, β omita o zero antes do ponto decimal como não pode ser maior que 1.
- Nomes proprietários funcionam como adjetivos. Substantivo devem ser fornecido após o uso como em *Vaseline petroleum jelly*. Sempre quando possível, use apenas o termo genérico.

ALGUNS ELEMENTOS DO ESTILO DE ESCRITA EFICAZ

- *Palavras curtas*. Palavras curtas são preferíveis as palavras longas se a mais curta é igualmente precisa.
- *Palavras conhecidas*. Os leitores querem informações que eles podem compreender facilmente e rapidamente. Palavras simples, familiares fornecem clareza e impacto.
- *Palavras específicas, em invés de palavras gerais*. Termos específicos identificam o significado e criam "palavras fotos"; termos gerais podem ser difusas e aberta a interpretações variadas.
- *Abertura concisa*. Mergulhe no seu assunto no primeiro parágrafo do artigo.
- *Uso limitada de modificação de palavras e frases.* Verifique seus adjetivos, advérbios, e frases preposicionais. Se eles não são necessários, removê-los.
- *Repetição desnecessária.* Uma ideia pode ser repetida para dar ênfase — contanto que a repetição é eficaz.
- *Comprimento de frases*. Vinte palavras ou menos são recomendado. Frases sem coerência ou cheia de orações subordinadas e outros modificadores são difíceis de ler e podem causar que os leitores perdão sua linha de raciocínio. Frases curtas devem, no entanto, ser equilibradas com aquelas pouco maiores para evitar a monotonia.
- *Parágrafos*. Separar seções longas em parágrafos, mas evite parágrafos de uma única frase.
- *Coibição*. Escritores que usam palavras extravagantes ou exageram sua proposição ou conclusões desacreditam de si mesmos. Os fatos falam por si.
- *Declare claramente as conclusões*. Se não sabe algo, diga.

TERMOS CENSURÁVEIS

A seguir são termos selecionados censuráveis e seus substitutos adequados. Para obter uma lista completa de terminologia prostodônticas aprovadas, consulta a oitava edição do Glossary of Prosthodontic Terms (J Prosthet Dent 2005; 94:10-92).

Ou visite JPD http://www.prosdent.org e clique em Collections/Glossary of Prosthodontic Terms.

Incorreto	Correto
Alginate	Irreversible hydrocolloid
Bite	Occlusion
Bridge	Partial fixed dental prosthesis
Case	Patient, situation, or treatment as appropriate
Cure	Polymerize
Final	Definitive
Freeway space	Interocclusal distance
Full denture	Complete denture
Lower (teeth, arch)	Mandibular
Model	Cast
Modeling compound	Modeling plastic impression compound
Muscle trimming	Border molding

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Overbite, overjet	Vertical overlap, horizontal overlap
Periphery	Border
Post dam, postpalatal seal	Posterior palatal seal
Prematurity	Interceptive occlusal contact
Saddle	Denture base
Study model	Diagnostic cast
Upper (teeth, arch)	Maxillary
X-ray, roentgenogram	Radiograph

Além disso, a palavra "*specimen*" deve ser usado em invés de "*sample*" quando se refere a um exemplo considerado típico de sua classe.

Orientações Adicionais de Terminologia

Acrylic
Uma forma adjetivo que requer um substantivo, como em *acrylic resin.*

Affect, Effect
Affect é um verbo; *effect* é um substantivo.

African American
É preferido sobre *Negro* ou *Black* em ambos formas adjetiva (*African American patients)* e substantivo *(... of whom 20% were African American).*

Average, mean, median
Mean e *average* são sinônimos. *Median* refere-se ao ponto médio dum interval de itens; o ponto médio tem muitos itens acima como abaixo.

Basic
Como *fundamental*, esta palavra é muitas vezes desnecessário. Um exemplo de uso desnecessário: *Dental Implants consist of two basic types: Subperiosteal and endosteal.*

Between, among
Use *between* quando duas coisas são envolvidas e *among* quando há mais de dois.

Biopsy
Esse substantivo não deve ser usado como um verbo. *A biopsy was performed on the tissue,* em vez de: *The tissue was biopsied.*

Centric
Um adjetivo que requer um substantivo, com em *centric relation.*

Currently, now, at present, etc.
Essas expressões são muitas vezes desnecessárias, como em: *This technique is currently being used.*

Data
Use forma plural, como em: *The data were...*

Employ
Não deve tornar-se numa variação de *use;* como em *This method is employed...*

Ensure
Preferido sobre *insure* no senso de ter certeza.

Fewer, less
Use *fewer* com substantivos que podem ser contados (*fewer patients were seen)* e *less* com substantivos que não podem ser contados *(less material was used).*

Following
After é preferido.

Imply, infer
O falador *implies;* o ouvinte *infers.*

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Abreviaturas Aprovadas para Jornais Geralmente Citadas

Porque *The Journal of Prosthetic Dentistry* é publicada não só em forma escrita, mas também on-line, os autores devem usar as abreviaturas de PubMed padrão para títulos de periódicos. Se uma alternativa ou uma abreviações não é usada, as referências não serão ligadas na publicação on-line. Uma lista completa de abreviaturas padrões está disponível através do PubMed-site:
http://www.ncbi.nlm.nih.gov/nlmcatalog/journals.

Acta Odontologica Scandinavica .... Acta Odontol Scand
American Journal of Orthodontics .... Am J Orthod
Angle Orthodontist .... Angle Orthod
British Dental Journal .... Br Dent J
Cleft Palate Journal .... Cleft Palate J
Dental Clinics of North America .... Dent Clin North Am
Dental Digest .... Dent Dig
Dental Practitioner and Dental Record .... Dent Pract Dent Rec
Dental Progress .... Dent Prog
Dental Survey .... Dent Surv
International Dental Journal .... Int Dent J
International Journal of Oral and Maxillofacial Implants .... Int J Oral Maxillofac Implants
International Journal of Periodontics and Restorative Dentistry ....Int J Periodontics Restorative Dent
International Journal of Prosthodontics ....Int J Prosthodont
Journal of the American College of Dentists .... J Am Coll Dent
Journal of the American Dental Association .... J Am Dent Assoc
Journal of Dentistry for Children .... J Dent Child
Journal of Dental Education .... J Dent Educ
Journal of Dental Research .... J Dent Res
Journal of Endodontics .... J Endod
Journal of Oral Rehabilitation .... J Oral Rehabil
Journal of Oral Surgery .... J Oral Surg
Journal of Periodontology .... J Periodontol
Journal of Prosthetic Dentistry .... J Prosthet Dent
Journal of Prosthodontics .... J Prosthodont
Oral Surgery, Oral Medicine, and Oral Pathology .... Oral Surg Oral Med Oral Pathol
Quintessence International .... Quintessence Int

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Incidence
O número de casos de doença que ocorre num determinado tempo; muitas vezes é confundida com *prevalence* (o número total de casos duma doença numa determinada região).

Majority
Significa mais de metade, use *most* quando quer dizer quais todos.

Male, female
Para humanos adultos, use *men* e *women. Para meninos,* use *boys* e *girls.*

Must, should
Must significa que o curso de ação é essencial. *Should* é menos forte e significa que um curso de ação é recomendado.

Numbers
Soletrar números usados em títulos ou cabeçalhos e para os números no início duma frase. A versão escrita também pode ser preferível numa série de números consecutivos que podem confundir o leitor (por exemplo, 2 3.5-*inch disks* deve ser escrito *two 3.5-inch disks).* Em todos os outros casos, use algarismos árabes.

Orient
Forma própria: evite *orientate.*

Pathologic
Use em vez de *pathological.* Outras palavras em que o suffixo *–al* foi descontinuado incluem *biologic, histologic,* e *physiologic.*
Pathology
O estudo de doença; muitas vezes confundido com *pathosis (*o estado de doença).

Percent
Use o sinal de percentagem no texto, como em *The distribution of scores was as follows: adequate, 8%; oversized, 23%; and undersized, 69%.* Mas soletrar para quando a percentagem abre uma frase, como em *Twenty percent of the castings…*

Prior to
Before é preferido.

Rare, infrequent, often not, etc.
Sempre que possível, esses termos vagos devem ser acompanhada por um número especifico.

Rather
Como *very,* esta palavra deve ser evitada.

Regimen
Refer-se a um programa planejado para tomar medicação, dieta, exercício, etc. Não deve ser confundido com *regime,* ou seja, um system de governo ou gestão.

Symptomatology
A ciência ou o estudo dos sintomas; esta palavra não é um sinônimo para a palavra *symptoms.*

Technique
Preferida sobre *technic.*

Using
Evite o pendendo modificador em frases tais como *The impression was made using vinyl polysiloxane impression material.* Escreva em vez *with* ou *by using.*

Utilize
Use é preferido.

Vertical
O adjetivo que precisa um substantivo, como em *vertical relation.*

Via
Use *through, with,* ou *by means of.*

White
Preferido sobre *Caucasian.* Isso só é verdade se o paciente for da região do Cáucaso da Europa do leste. Se não, use o termo *white* para descrever o paciente.

Abreviaturas Aprovadas para Jornais Geralmente Citadas

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Quintessence International .... Quintessence Int

ANNEXE 5 – Guide for authors of Dental Materials Journal

ELSEVIER

DENTAL MATERIALS

Official Publication of the Academy of Dental Materials

AUTHOR INFORMATION PACK

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ISSN: 0109-5641

DESCRIPTION

Online submission and editorial system now available at http://ees.elsevier.com/dema

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The principal aim of *Dental Materials* is to promote rapid communication of scientific information between academia, industry, and the dental practitioner. Original Manuscripts on clinical and laboratory research of basic and applied character which focus on the **properties** or **performance** of **dental materials** or the **reaction** of host tissues to materials are given priority publication. Other acceptable topics include application technology in **clinical dentistry** and dental laboratory technology.

Comprehensive reviews and editorial commentaries on pertinent subjects will be considered.

AUDIENCE

Dental research scientists, materials scientists, clinicians, students of dentistry, dental materials and equipment manufacturers.

IMPACT FACTOR

2018: 4.440 © Clarivate Analytics Journal Citation Reports 2019

GUIDE FOR AUTHORS

INTRODUCTION

Authors are requested to submit their original manuscript and figures via the online submission and editorial system for *Dental Materials*. Using this online system, authors may submit manuscripts and track their progress through the system to publication. Reviewers can download manuscripts and submit their opinions to the editor. Editors can manage the whole submission/review/revise/publish process. Please register at: https://www.editorialmanager.com/dentma/default.aspx.
Dental Materials now only accepts online submissions.
The Artwork Quality Control Tool is now available to users of the online submission system. To help authors submit high-quality artwork early in the process, this tool checks the submitted artwork and other file types against the artwork requirements outlined in the Artwork Instructions to Authors on https://www.elsevier.com/artworkinstructions. The Artwork Quality Control Tool automatically checks all artwork files when they are first uploaded. Each figure/file is checked only once, so further along in the process only new uploaded files will be checked.

Manuscripts
.**Submitted manuscripts must relate directly to both Materials Science and Dentistry**. The journal is principally for publication of **Original Research Reports**, which should preferably investigate a defined hypothesis. Maximum length 6 journal pages (approximately 20 double-spaced typescript pages) including illustrations and tables.

Systematic Reviews will however be considered.**Prior approval must be sought from the Editor before submission of Review Manuscripts**. Authors should send the Editor a structured abstract of the proposed review topic.Intending authors should communicate with the Editor beforehand, by email, outlining the proposed scope of the review. Maximum length 10 journal pages (approximately 33 double-spaced typescript pages) including figures and tables.

Three copies of the manuscript should be submitted: each accompanied by a set of illustrations. The requirements for submission are in accordance with the "Uniform Requirements for Manuscripts Submitted to Biomedical Journals", Annals of Internal Medicine, 1997,126, 36-47. All manuscripts must be written in American English. Authors are urged to write as concisely as possible.

The Editor and Publisher reserve the right to make minimal literary corrections for the sake of clarity. Authors for whom English is not the first language should have their manuscripts read by colleagues fluent in English. If extensive English corrections are needed, authors may be charged for the cost of editing. For additional reference, consult issues of *Dental Materials* published after January 1999 or the Council of Biology Editors Style Manual (1995 ed.).

All manuscripts should be accompanied by a **letter of transmittal**, signed by each author, and stating that the manuscript is not concurrently under consideration for publication in another journal, that all of the named authors were involved in the work leading to the publication of the paper, and that all the named authors have read the paper before it is submitted for publication.

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