ARTIGO ORIGINAL

MECHANICAL STRENGTH ANALYSIS OF RESINS PRINTED WITH THE ADDITION OF NB_2O_5 NANOPARTICLES

Análise da resistência mecânica de resinas impressas com adição de nanopartículas de nb_2o_5

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Juary Rodrigues Pereira[1], Fabiano Perez[2], Caio Marques Martins[3], Francisco Fernando Massola Filho[4], Sérgio Cândido Dias[5], Geraldo Alberto Pinheiro de Carvalho[6]

ABSTRACT

Objective: To evaluate the strength of composite resins used for the fabrication of complete dentures after the addition of niobium pentoxide (Nb2O5). Methods: Different concentrations of Nb2O5 were added to the polymer matrix: 0% (control), 0.5%, 1.0%, 1.5%, and 2.0% per 100 mg of resin. In this context, nanoparticles were incorporated under ideal conditions, and the test specimens were designed using Dentsply Sirona's inLab MC XL software. After the addition of nanoparticles, samples were printed with dimensions of 25 mm in width, 2 mm in height, and 2 mm in thickness. The test specimens were fabricated through digital printing. Finally, the samples were subjected to mechanical testing for flexural strength and impact resistance. Results: The 0.5% and 1.0% groups showed higher flexural strength values (54.56 and 56.8 MPa), with statistical significance ($p<0.05$) compared to the 1.5% and 2.0% groups (45.26 and 40.65 MPa). Regarding the modulus of elasticity, the 1.0% group presented the highest value (1.15 GPa), while the 0.5% group obtained 0.98 GPa, showing significant statistical differences ($p<0.05$) between the groups. In terms of impact resistance, the 0.5% and 2.0% groups exhibited the highest resistance, differing significantly from the 1.0% and 1.5% groups ($p<0.05$). Conclusion: Niobium pentoxide improved the properties of PMMA in this study, proving to be a viable alternative for the development of denture bases.

Keywords: CAD-CAM; Nanoparticles; Polymethyl Methacrylate; Mechanical Properties; Flexural Strength.

RESUMO

Objetivo: avaliar a resistência de resinas compostas utilizadas para confecção de próteses totais após a adição de pentóxido de nióbio (Nb2O5). Métodos: foram adicionadas diferentes concentrações de Nb2O5 à matriz polimérica: 0% (controle), 0,5%, 1.0%, 1,5% e 2,0% para proporção de 100mg de resina. Nesse contexto, foram incorporadas nanopartículas em condições ideais e desenhados os corpos de prova no Software inLab MC XL da Dentsply Sirona. Após a adição de nanopartículas, foram impressas amostras de 25mm de largura por 2mm de altura e 2mm de espessura. A confecção dos corpos de prova se deu por meio de impressão digital. Para finalizar, as amostras foram submetidas ao ensaio mecânico de resistência a flexão e resistência ao impacto. Resultados: os grupos 0,5% e 1,0% apresentaram maiores valores de resistência a flexão (54,56 e 56,8 MPa), apresentando diferença estatística ($p<0,05$) em comparação os grupos 1,5% e 2,0% (45,26 e 40,65 MPa). Quanto ao módulo de elasticidade, o grupo 1,0% apresentou maior valor (1,15 GPa), enquanto o grupo 0,5% obteve 0,98 (Gpa), apresentando diferença estatística significativa ($p<0,05$) entre os grupos. Em se tratando da resistência ao impacto, os grupos 0,5% e 2,0% apresentaram maior resistência, diferindo significativamente dos grupos 1,0% e 1,5% ($p<0,05$). Conclusão: o pentóxido de nióbio melhorou as propriedades do PMMA no presente estudo, mostrando-se uma alternativa viável para desenvolvimento de base de próteses.

Palavras-chave: CAD-CAM; Nanopartículas; Polimetil Metacrilato; Propriedades Mecânicas; Resistência à Flexão.

1 Master's Student in Oral Rehabilitation - SL Mandic College
2 Health Department, Professor of the Master's Program in Prosthetics - SL Mandic College, Campinas SP Unit
3 Health Department, Professor of the Master's Program in Prosthetics - SL Mandic College, Campinas SP Unit
4 Health Department, Professor of the Master's Program in Prosthetics - SL Mandic College, Campinas SP Unit
5 Health Department, Professor of the Master's Program in Prosthetics - SL Mandic College, Campinas SP Unit
6 Health Department, Coordinator of the Master's Program in Prosthetics - SL Mandic College, Campinas SP Unit

Autor de correspondência

Juary Rodrigues Pereira

juary.rodriguespereira@gmail.com

INTRODUCTION

Currently, various types of nanoparticles are being studied. Several oxides are used to improve the properties of different types of materials. Niobium pentoxide (Nb2O5) stands out as one of the most important, exhibiting characteristics such as air stability and insolubility in water. Additionally, Brazil holds the largest niobium reserves in the world [(1)]. Nanoparticles have the ability to increase the compaction of materials, consequently improving their mechanical properties [(2)].

Over the past few years, nanomaterials have been increasingly utilized due to their unique properties. The concept of nanomaterials began in the 1980s, referring to materials with dimensions close to zero, approximately 100 nm. Nanomaterials can be divided into five categories: nanotubes, nanopowders, nanofibers, nanomembranes, and nanoblocks. Among these, nanopowders have been the most extensively studied [(3)].

Studies show that materials based on niobium for dental use demonstrate satisfactory results in terms of biocompatibility and improved physical and optical properties. Additionally, they can be used for high-value applications in equally strategic areas [(4)]. Moreover, the addition of niobium to biological materials has been considered due to studies showing that niobium improves the properties of these materials [(5)].

Niobium (Nb) is a metal that has shown potential to enhance the properties of different materials, even when applied in small quantities. Its addition to composite resin, resin cement, and adhesives increases the strength of these materials, and it also exhibits high biocompatibility [(6)].

Given the properties of niobium pentoxide and the significant availability of this nanomaterial in Brazil, its study is entirely valid and justified. Therefore, this study aims to evaluate the behavior of PMMA fabricated using the CAD/CAM system with the addition of Nb2O5 nanoparticles, focusing on mechanical testing for flexural strength, impact resistance, and measuring the modulus of elasticity.

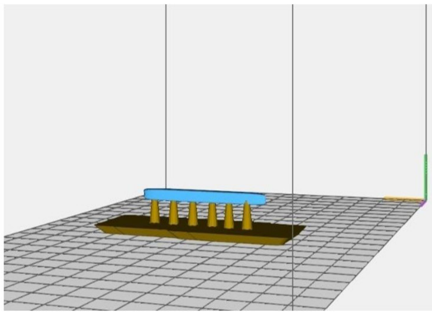
OBJECTIVE

To evaluate, in vitro, the flexural strength, impact resistance, and modulus of elasticity of PMMA used in the fabrication of complete denture bases with the addition of Nb2O5 nanoparticles.

METHODS

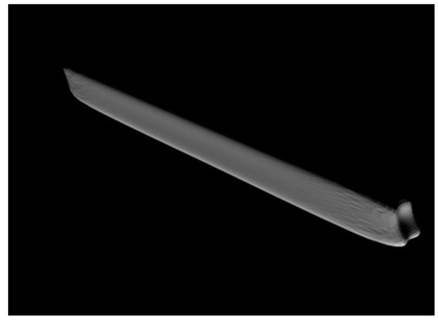
The study consisted of 5 sample groups (NB-0%, control group, NB-0.5%, NB-1.0%, NB-1.5%, and NB-2.0%). Each group had 12 test specimens for testing Flexural Strength (FS), Modulus of Elasticity (ME), and Impact Resistance (IR). This is an experimental analysis of PMMA, a polymeric material used in the CAD-CAM system for the fabrication of printed complete dentures. Niobium Pentoxide Nb2O5 nanoparticles (Sigma-Aldrich Germany USA 99.9%) were added to the polymer matrix (PMMA Yller Cosmos Denture), a 3D printing resin for dentures. The material was planned and designed for printing according to the projects shown in the figures:

Figure 1. 3D Project



Source: Own authorship

Figure 2. File in STL



Source: Own authorship

The Nb2O5 (Sigma-Aldrich, USA, 99.9%) used in this study was washed (99.5% anhydrous ethanol, Decon Labs) and centrifuged at 10,000 rpm for 5 minutes. Then, the Nb2O5 was added to a 2M HCl solution, where it remained in a water bath (50°C/60min). After this process, the particle was washed again with ethanol and centrifuged 3 times, obtaining a final Nb2O5 solution with an approximate concentration of 25 mg/mL. Different concentrations of Nb2O5 (0, 0.5, 1.0, 1.5, and 2.0 wt%) were incorporated into PMMA 3D Yller Cosmos Denture 3D printing resin. The PMMA was homogenized using a specific mixer (Speed Mixer, DAC Iso 1.FVZ, FlackTek, Inc.) at 3500 rpm for 2 minutes. PMMA was handled in the absence of light. The planning was carried out in CAD using inLab MC XL software (Dentsply Sirona, USA 2019), specifically used to design the pieces that were subsequently printed. The file with the appropriate proportions was selected, consisting of rectangular samples (25 mm in width x 2 mm in height x 2 mm in thickness), according to ISO 4049. Initially, a calibration piece with the same proportions was printed, following the parameters provided by the manufacturer as shown in the table below:

Table 1. Manufacturer parameters followed for printing the rectangular PMMA piece.

Parameter	**Resin Cosmos**
Normal exposure time	2.5
Bottom exposure time	50
Off Time	5
Bottom layers	4
Layer thickness	0.05 mm

Source: Own authorship

After printing, the piece was immersed in a liquid (Isopropyl Alcohol TogMax) for 10 minutes. After drying the piece, post-curing was performed in a 72W UV chamber for 10 minutes. The dimensions were within the appropriate proportions after the curing process. There were 12 rectangular samples (25 mm in width x 2 mm in height x 2 mm in thickness) printed on the 3D printer (Anycubic 1-014 Photon Mono 4k). PMMA (Yller Cosmos Denture 3D printing resin) was used for the denture.

The study design was conducted as shown in the table below:

Table 2. Experimental Design

Experimental Units	1. PMMA 3D Test Specimens	
Factors Under Study	1) (wt%) Niobium Pentoxide (Nb)	**1. Nb_0% (control)** **2. Nb_0.5%** **3. Nb_1.0%** **4. Nb_1.5%** **5. Nb_2.0%**
Response Variables	• Flexural Strength (FS - MPa) (n=12) • Modulus of Elasticity (ME - GPa) (n=12) • Impact Resistance (kJ/mm²) (n=12)	

Source: Own authorship

To evaluate the flexural strength (FS) and modulus of elasticity (ME), test specimens with dimensions of 25 × 2 × 2 mm were fabricated according to ISO 4049 and subjected to three-point bending in a universal testing machine (Instron 4411, Canton) with a span length of 20 mm and a speed of 0.5 mm/min (n=12). The following formula was used to determine the flexural strength values:

$$E = \frac{C \times L^3}{4 \times b \times h^3 \times d} x10^{-3} \quad \sigma$$

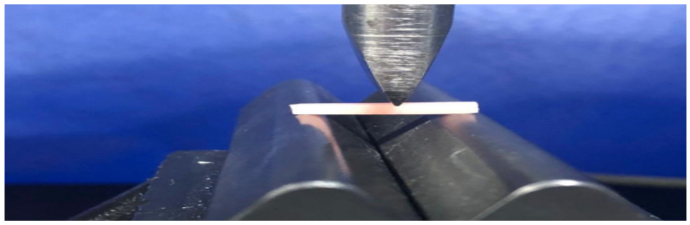
Where: E is the flexural modulus of elasticity (GPa); σ is the flexural stress (MPa); δ is the flexural strain (mm/mm); L is the span length (mm); b is the width of the specimen (mm); h is the height of the specimen (mm).

The values for the modulus of elasticity were determined according to the equation:

$$\sigma = \frac{3 \times F \times L}{2 \times b \times h^2}$$

Where: E is the flexural modulus of elasticity (GPa); C is the load recorded during elastic deformation (N); L is the span length (mm); b is the width of the specimen (mm); h is the height of the specimen (mm); d is the corresponding deflection at C. The flexural strength test was performed as shown in the figure below:

Figure 3. Flexural Strength Test



Regarding impact resistance (IR), the test specimens (n=12) were subjected to the impact resistance test on a Wolpert-Werke machine using the Charpy system, with an impact load of 40 kJ/ mm^2. The impact value obtained at the moment of fracture was converted into impact resistance (kJ/mm^2) using the formula ($Ri = Ci / b \times h$), where Ri = impact resistance; Ci = impact load applied (kJ/mm); b = width of the test specimen (mm); and h = height of the test specimen (mm).

Statistical analysis was performed using SPSS for Windows version 23 (IBM SPSS Inc, Chicago, IL, USA) with one-way analysis of variance (ANOVA) and Tukey post-hoc test, with normality and homoscedasticity confirmed for all variables by the Levene and Shapiro-Wilk tests. The Dunnett's test compared the control with the other experimental groups with the significance level set at 5%.

RESULTS

Table 3 summarizes the mechanical performance results of 3D PMMA denture base modified by niobium pentoxide, including their respective means and standard deviations, as well as the statistical analysis performed.

Table 1. Mean ± Standard Deviation of Flexural Strength, Modulus of Elasticity, and Impact Resistance of 3D PMMA modified by Nb_2O_5

Groups	RF (MPa)	ME (GPa)	RI (kJ/mm^2)
Nb_0%	$28.7 \pm 3.01^{\#}$	$0.57 \pm 0.07^{\#}$	$4.43 \pm 2.43^{\#}$
Nb_0,5%	54.56 ± 4.83^{A}	0.98 ± 0.14^{B}	7.8 ± 2.55^{A}
Nb_1%	56.8 ± 7.06^{A}	1.15 ± 0.23^{A}	$2.74 \pm 1.09^{B\#}$
Nb_1,5%	45.26 ± 3.49^{B}	1.07 ± 0.12^{AB}	$3.09 \pm 0.59^{B\#}$
Nb_2%	40.65 ± 7.16^{B}	1.04 ± 0.14^{AB}	7.88 ± 4.2^{A}

Different letters indicate statistical differences between groups in the same column ($p<0.05$). Groups with hashtag (#) are statistically similar to the control group (Nb_0%).

Source: Own authorship

In terms of flexural strength, it was significantly higher in the Nb 0.5%, 1.0%, 1.5%, and 2.0% groups compared to the control group (Nb_0%) ($p<0.05$). Among the experimental groups, Nb 0.5% and Nb 1.0% achieved the highest performance in this parameter (54.56 MPa and 56.8 MPa), respectively, differing statistically from the Nb 1.5% and 2.0% groups ($p>0.05$).

On the other hand, Nb 0.5% and 1.0% were not statistically different from each other, as well as Nb 1.5% and 2.0% ($p>0.05$).

Regarding the modulus of elasticity (ME), the Nb 0.5%, 1%, 1.5%, and 2.0% groups showed significantly higher ME than the control group (Nb_0%) ($p<0.01$). Nb_1% obtained the highest ME values (1.15 GPa); however, it only differed statistically from the Nb_0.5% group (0.98 GPa) ($p<0.05$). The evaluated groups showed mean ME values ranging from 0.57 GPa (Nb_0%) to 1.15 GPa (Nb_1%).

Regarding the impact resistance (IR) of the evaluated groups, it was possible to observe a similarity, with no statistical differences ($p>0.05$) between the control group (Nb_0%) and the Nb_1% and 1.5% groups, showing values of 4.43 kJ/mm² (Nb_0%), 2.74 kJ/mm² (Nb_1%), and 3.09 kJ/mm² (Nb_1.5%). The Nb_0.5% and 2% groups obtained the highest IR values (7.8 kJ/mm² and 7.88 kJ/mm², respectively) with statistical differences from the control group and the Nb_1% and 1.5% groups ($p<0.05$). However, Nb_0.5% and 2.0% were not different from each other ($p>0.05$).

DISCUSSION

This study aimed to evaluate in vitro the flexural strength, impact resistance, and modulus of elasticity of PMMA used in the fabrication of complete denture bases with the addition of Nb_2O_5 nanoparticles. When evaluating flexural strength (FS), the results showed that the addition of niobium pentoxide to PMMA significantly increased this property ($P<0.05$). Compared to the control group, which did not have the nanomaterial added and showed a mean FS of 28.7 MPa, the NB 0.5% group obtained a mean of 54.56 MPa, the NB 1% group 56.8 MPa, the NB 1.5% group 45.26 MPa, and the NB 2.0% group 40.65 MPa. These values indicate that the 1% concentration achieved the best performance, with a tendency for flexural strength to decrease as the proportion of the nanomaterial exceeded this value.

A study evaluated flexural strength (FS) with the addition of (TiO_2 + ZnO) at different concentrations (0%, control group, 1%, 2%, 3%, 4%, and 5%) in the PMMA matrix. The authors found the best results at the 4% concentration (71.53 MPa). At 1% and 2% concentrations, the values were 56.78 MPa and 54.42 MPa, respectively. At 3% and 5% concentrations, there was a reduction in FS (34.09 MPa and 37.98 MPa, respectively), with the control group (0%) achieving 51.90 MPa. At these concentrations, there was a weakening of the resin, with a 34.2% and 26.83% decrease in FS for the 3% and 5% concentrations, respectively. In the other groups, flexural strength increased by 9.5% with the addition of 1%, 4.48% with the addition of 2%, and 36.08% with the addition of 4% (7). The absence of studies using niobium pentoxide as a nanomaterial incorporated into the PMMA matrix made it impossible to compare works with similar methodologies.

It is important to remember that in recent decades, niobium-based compounds have received special attention due to their use in high-tech industries. The use of niobium oxides, as well as their combination with other oxides, presents unique properties, especially when used as catalysts in chemical reactions [8]. Another important factor for the use of these oxides is that niobium pentoxide (Nb2O5) stands out as one of the most important, exhibiting characteristics such as air stability and water insolubility. Additionally, Brazil has the largest niobium reserves in the world [1]. These facts support the idea that this is a material that needs to be studied and utilized to its fullest potential.

Nanoparticles have the ability to increase the compaction of materials and improve their mechanical properties [2]. Various nanoscale materials, such as silica, calcium carbonate, and metal oxides, have the ability to improve the physical properties of the PMMA matrix when added [9]. This justifies conducting studies with various nanoparticles until the desired material is found.

Scientists are seeking a technique capable of eliminating or reducing the disadvantages observed with the conventional denture fabrication technique. This technique has been in use for over eight decades, and it is viable to find ways to contribute to the improvement of the mechanical, physical, and optical properties of polymethyl methacrylate (PMMA) material [10].

Currently, great attention has been given to the use of nanometric fillers, as the development of nanotechnology and nanophase materials to reinforce denture base resins is quite prevalent. As a result, polymer nanocomposites with improved physical and mechanical properties have been produced compared to resins filled with microscale particles [11].

In this context, a study added zinc oxide nanoparticles to PMMA at concentrations of 0, control group, 0.4, 0.6, 0.8, 1.2, and 1.4 percent. After the analyses, the authors concluded that the flexural strength of the PMMA denture base increased with the addition of ZnO nanoparticles [12].

The incorporation of nanometric reinforcements into a polymer matrix aims to improve properties such as tensile strength, impact resistance, modulus of elasticity, thermal and electrical conductivity, thermal stability, and flame resistance [13]. Although different nanoparticles and methodologies were used, both studies corroborate the improvement of PMMA matrix properties after the addition of nanoparticles.

Regarding the modulus of elasticity (ME), in the present study, all groups showed significantly higher averages than the control group ($p<0.01$). Nb_1% obtained the highest ME values (1.15 GPa). In another study, when evaluating the modulus of elasticity, it was found that the addition of 2% (TiO2 + ZnO) increased the modulus of elasticity of the material by 0.570

GPa, while the addition of nano (TiO_2 + ZnO) at concentrations higher and lower than 2% decreased the modulus of elasticity of the material (7). This demonstrates that studies should be conducted to test all possibilities until a material with satisfactory properties is achieved. In the present study, the 1% niobium pentoxide concentration showed the best result in terms of the modulus of elasticity.

Another study aimed to measure surface hardness, modulus of elasticity, and surface roughness of 5 different photo-polymerized enamel materials for polymethyl methacrylate denture base materials. The authors concluded that surface coatings containing silica dioxide nanoparticles produced the highest surface hardness and modulus of elasticity (14).

Regarding impact resistance (IR), the present study shows that the NB-0.5% and NB-2.0% groups had the best results, while the 1% and 1.5% concentrations showed a reduction in impact resistance. In another study, while the addition of 1% and 4% (TiO_2 + ZnO) increased the impact resistance of the material, other concentrations reduced this property (7). This is justified as high-proportion fillers lead to large stresses within the polymer near the filler edges (15). Stress concentrations also occur in regions where nano-additives contact the area of decreased interfacial bonding between the nano-additives and the matrix. This stress is caused by clustered nano-additives and voids that act as stress concentration points in the polymer matrix (16).

In terms of impact resistance, a study was conducted to evaluate and compare the impact resistance of high-impact acrylic resin reinforced with zirconia (ZrO_2) with conventional high-impact acrylic resin without reinforcement. The authors prepared 40 test specimens, 20 of each type of resin. The test specimens were then subjected to impact resistance testing. The authors concluded that there was a significant increase in the impact resistance of the group reinforced with zirconia nanoparticles compared to the control group, despite the addition of only 1% nanoparticles to the PMMA (17). The incorporation of zirconia in various dental materials has been studied, showing biocompatibility and significant beneficial effects on the mechanical properties of PMMA (18).

Another study evaluated the mechanical properties of PMMA after the addition of nanoparticles, incorporating four different types: fly ash, dust, zirconia, and aluminum. These nanoparticles were added at different volume fraction proportions (1%, 2%, and 3%) to polymethyl methacrylate (PMMA), cold-cured resin (Castavaria), and the new fluid resin (pour type) as the matrix. In this work, the nanocomposite and hybrid nanocomposite for denture specimens were prepared using the hand lay-up method, divided into six groups: the first three groups consisted of PMMA resin reinforced with fly ash, dust, and ZrO_2 nanoparticles, respectively. The second three groups consisted of three types of hybrid nanocomposites, which included (PMMA: X% nF.A) + (1% Al + 3% ZrO_2), (PMMA: X% nD.A) + (1% Al + 3% ZrO_2), and (PMMA - X% $nZrO_2$) + (1% FA + 3% FD) respectively for fly ash, dust, and zirconium oxide. The authors concluded that mechanical properties such as hardness and flexural modulus increased with the increase in volume fractions of nanoparticles (fly ash, dust, and zirconium oxide) in PMMA nanocomposites and hybrid PMMA nanocomposites [(19)].

Mechanical properties such as flexural strength and maximum shear stress increased with the addition of low concentrations of nanoparticles (fly ash, dust, and zirconium oxide) and decreased with the addition of high concentrations above 2% of all nanoparticles to PMMA nanocomposites and hybrid PMMA nanocomposites [(19)].

A study was conducted by adding Nb_2O_5 nanoparticles and hydroxyapatite to dental composite resins. Nanoparticles in powder form were used in proportions of 0.02g and 0.01g for niobium pentoxide and hydroxyapatite in proportions of 0.02g and 0.04g. In this study, the results showed that the composite resin formed by 0.5g of nanohybrid resin with 0.02g Nb_2O_5 and 0.04g HAp did not interfere with the mechanical properties and appearance of the samples but contributed to increased corrosion resistance, making it a favorable non-toxic compound for use in the dental field [(20)].

In another study, an experimental flow composite incorporating niobium pentoxide (Nb_2O_5), with or without titanium dioxide co-doped with fluorine and nitrogen (NF_TiO_2), was developed and its mechanical and antibacterial properties were evaluated. It was concluded that the addition of Nb_2O_5 particles at concentrations of 0.5%, 1%, 1.5%, and 2%, and NF_TiO_2 particles at concentrations of 1%, 1.5%, and 2%, as well as the combination of both (Nb_2O_5 + NF_TiO_2) at a concentration of 1% and a ratio of 1:1, showed significant antibacterial effects [(21)]. The incorporation of niobium pentoxide into biomaterials also stands out due to its remarkable physicochemical properties and high biocompatibility, demonstrating potential in biomedical applications [(22)].

CONCLUSION

Based on the findings of the present study, it can be concluded that:

Niobium pentoxide improved the properties of PMMA, proving to be a viable alternative for the development of denture bases with enhanced properties.

The concentrations of 0.5% and 1.0% added to PMMA showed the highest values for the modulus of elasticity and flexural strength.

The 0.5% concentration exhibited the highest impact resistance.

It is suggested that further studies be conducted to test niobium pentoxide in combination with other materials that also improve the mechanical properties of PMMA.

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Observação: os/(as) autores/(as) declaram não existir conflitos de interesses de qualquer natureza.