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Effect of Thermomechanical Fatigue on Shear Strength between a Conventional and an Experimental Polymer for Prosthetic Application

Efeito da Fadiga Termomecânica na Resistência ao Cisalhamento Entre um Polímero Convencional e um Polímero Experimental para Aplicação Protética

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Abstract

The incorporation of antimicrobial agents may influence the mechanical properties of acrylic resins. Thus, the use of these agents only in regions of dental prostheses subject to greater contamination may be an alternative. This study evaluates the effect of thermomechanical fatigue on the bond strength between a conventional and an experimental acrylic resin incorporated with nanostructured silver vanadate decorated with silver nanoparticles ($AgVO_3$). 60 specimens (Ø13mm x 23mm height) in self-curing resin were obtained and divided into groups according to the experimental resin incorporated with $AgVO_3$ (Ø4mm x 6mm height): G1–Conventional x Conventional, G2–Conventional x 2.5% of $AgVO_3$, G3–Conventional x 5% of $AgVO_3$. Ten samples of each group were subjected to bond strength analysis after manufacture, and 10 were previously submitted to 1.200.000 cycles with 98N load and 2Hz/second frequency and alternating baths of 5 ºC, 37ºC and 55 ºC. The fracture area was analyzed. The data were submitted to analysis of variance of two-factors with Bonferroni adjustment for *post hoc* comparisons (α=0.05) was used. The fatigue did not affect the bond strength (p=0.416), however, there was influence of the $AgVO_3$ concentration on the bond strength between the resins (p=0.013). Mixed failures with adhesive predominance were observed in samples without $AgVO_3$ and cohesive failures in samples with the nanomaterial. The use of $AgVO_3$ can improve or maintain the bond strength between resins with no thermomechanical fatigue influence.

Keywords: Acrylic Resins. Products with Antimicrobial Action. Nanotechnology. Thermomechanical fatigue

Resumo

A incorporação de agentes antimicrobianos pode influenciar nas propriedades mecânicas de resinas acrílicas. Desta forma, o uso destes agentes apenas em regiões das próteses dentárias sujeitas a maior contaminação pode ser uma alternativa. Este estudo avalia o efeito da fadiga termomecânica na resistência de união entre uma resina acrílica convencional e uma experimental incorporada com vanadato de prata nanoestruturado decorado com nanopartículas de prata ($AgVO_3$). Foram obtidos 60 espécimes (Ø13mm x 23mm de altura) em resina autopolimerizável, divididos em grupos de acordo com a resina experimental incorporada com $AgVO_3$ (Ø4mm x 6mm de altura): G1-Convencional x Convencional, G2-Convencional x 2,5% de $AgVO_3$, G3 -Convencional x 5% de $AgVO_3$. Dez amostras de cada grupo foram submetidas à análise de resistência à união após a confecção e 10 foram submetidas previamente a 1.200.000 ciclos com carga de 98 N e frequência de 2Hz/segundo e banhos alternados de 5 ºC, 37 ºC e 55 ºC. A área de fratura foi analisada. Os dados foram submetidos à análise de variância de dois fatores com ajuste de Bonferroni para comparações pos hoc (α = 0,05). A fadiga não afetou a força de união (p=0,416), no entanto, houve influência da concentração de $AgVO_3$ na resistência de união entre as resinas (p=0,013). Falhas mistas com predominância ‘ou manter a resistência da união entre as resinas sem influência da fadiga termomecânica.

Palavras-chave: *Resinas Acrílicas. Produtos com Ação Antimicrobiana. Nanotecnologia.*

1 Introduction

The loss of a dental element, especially in aesthetic areas, results in functional and psychological consequences to the patient. Therefore professionals have explored predictable and efficient therapeutic options for restoring the balance of soft and hard tissue[1-3].

The success of a treatment, conventional or *implant-supported*, depends largely on the quality of temporary prostheses that can remain for long periods of time in the oral cavity based on the planning that has been chosen, which must meet the biomechanical and aesthetic requirements[4,5].

Provisional prostheses are most often made of acrylic resin, in which the main component is methyl polymethacrylate (PMMA)[6]. Local factors such as roughness and porosity of the resin, coupled to problems of hygiene and loss of tissue integrity associated with systemic factors such as malnutrition, diabetes mellitus, infection by human immunodeficiency virus and xerostomia contribute to the proliferation and adherence of microorganisms[7,8].

In rehabilitations using immediate-loading, a temporary prosthesis is manufactured shortly after installing the implant[9]. However, the control of bacterial contamination at the bone/implant/crown interface is critical for the longevity of the treatment, since the accumulation of microorganisms can have consequences that range from the installation of peri-implant mucositis to peri-implantitis, characterized by loss of supporting bone tissue, which although easily diagnosed, may result in treatments that include resection surgeries[10,11].

The problems caused by contamination by microorganisms are also frequent in treatments with conventional prostheses, which can basically affect teeth dentures by caries and periodontal disease and soft tissue by prosthetic stomatitis[12-14]. Within this context, there is currently a growing interest by researchers to develop new dental materials with antimicrobial activity, in order to prevent contamination and formation of oral biofilm[15-17].

Silver is known to have satisfactory antimicrobial properties, especially when prepared in the form of nanoparticles[18]. In dentistry, silver nanoparticles (AgNPs) have been incorporated in several materials, such as acrylic resins and denture-based materials[19,20]. However, although AgNPs have different applications, they have disadvantages, mostly associated with agglomeration[21].

A promising antimicrobial agent, composed of vanadium and silver on nano-scale, the nanostructured silver vanadate decorated with AgNPs ($AgVO_3$) has been described[21,22]. The minimum inhibitory concentration (MIC) results were 100 times lower than those reported by an antibiotic used as a reference, Oxacillin, and included high activity against *Staphylococcus aureus* methicillin resistant, and *Enterococcus*, vancomycin resistant. The authors suggest this nanomaterial as an additive for water based paints for application in domestic and hospital environments with a high degree of bacterial contamination. Recent studies have also demonstrated good antimicrobial results of acrylic resins incorporated with $AgVO_3$, against *Staphylococcus aureus*, *Streptococcus mutans*, *Pseudomonas aeruginosa* and *Candida albicans*. However, adverse effects were observed in the mechanical properties of the resin, and this combination may increase the possibility of fracture of the prosthetic device[23-25].

Therefore, considering that the use of $AgVO_3$ only in regions subjected to higher microbial contamination, as in the internal base of conventional or implant-supported temporary prosthesis, could reduce the risks caused by microbial contamination without mechanical failure problems, the objective of this study is to evaluate the bond strength by means of a shear test between a conventional acrylic resin and an experimental resin with $AgVO_3$, before and after thermomechanical fatigue, and also classify the type of fracture. The null hypothesis is that the incorporation of $AgVO_3$ or the fatigue process has no affect the bond strength between the acrylic resins.

2 Material and Method

2.1 Synthesis and characterization of nanostructured silver vanadate decorated with AgNPs

The nanostructured silver vanadate decorated with silver nanoparticles was synthesized by a precipitation reaction between silver nitrate ($AgNO_3$, Merck 99.8%) and ammonium metavanadate (NH_4VO_3, Merck 99%)[21,22] and characterized by scanning transmission electron microscopy (STEM) (STEM), using a JEOL JEM-100CX II microscope.

2.2 Preparation of samples

The colorless and pink self-cured acrylic resin (Clássico Artigos Odontológicos®) were used in the preparation of the specimens. The first phase was to obtain the base sample of the specimen, in cylindrical format of Ø 13 mm x 23 mm in height. The second phase for the preparation of the opposing sample of Ø 4 mm x 6 mm in height with different concentrations of the nanomaterial (2.5% and 5%) by weight. In the first phase, the specimens were manufactured using the inclusion of matrices (Ø 13 mm x 23 mm in height) in a conventional metallic muffle (OGP, Produtos Odontológicos Ltda., São Paulo, SP, Brazil). The colorless acrylic resin was treated according to the manufacturer's recommendations and during the plastic phase it was accommodated in the molds previously prepared in the muffles. The muffles were placed in hydraulic presses (Protecni Equip. Med., Araraquara, SP, Brazil) with a load of 1000 Kgf for 60 minutes, and after the disinclusion, the excesses were removed with a Maxi-cut cutter (Malleifer AS, Ballaiguer, Switzerland) and specimen was sanded using Norton 180 and 400 sandpapers. The specimens were taken to an ultrasonic tank for 3 minutes to remove the residues and were then kept in an oven at 37 °C for 24 hours. Subsequently, the specimens were adapted to an aluminum-based teflon matrix and three groups were obtained, according to the experimental resin with different percentages of $AgVO_3$ injected on them (Ø 4 mm x 6 mm in height): G1 – Conventional x Conventional, G2 – Conventional x 2.5% of $AgVO_3$, G3 – Conventional x 5% of $AgVO_3$). For this, homogenization of the pink polymer resin and $AgVO_3$ was performed and them, the monomer was added in the amount specified by the manufacturer. A syringe was used to insert the material into the central hole (Ø 4 mm x 6 mm in height) of the matrix.

After 60 minutes, polymerization time, the assembly (base + opposing sample) was removed using a metal piston to minimize applying force on the interface. Twenty specimens were obtained from each group, which were then sub-divided into two groups (n=10), where one was submitted to thermomechanical fatigue.

2.3 Thermomechanical fatigue

Thermomechanical equipment was used (GERVIC, Thermomechanical wear system GTF 100) for the mechanical and thermal fatigue test. The specimens were subjected to 1.200.000 cycles, with a load of 98N at a frequency of 2Hz/second, corresponding to five years of clinical use[26]. At the same time, the samples were subjected to thermal cycles at temperatures of 5 °C, 37 °C and 55 °C.

2.4 Shear strength test

The shear test used a metal device with a central hole where the samples were affixed to a chisel. After fitting the base onto the EMIC DL1000 universal test machine (EMIC, São José dos Pinhais, PR), the specimen was positioned in the hole of the support, and then the chisel, connected to a 100 kg load cell, was positioned in the interface of the materials. The test was performed with a cross head speed of 1 mm/min until the rupture of the interface, and the tensile strength was recorded in Megapascal (MPa).

2.5 Fractographic analysis

After the shear test, three samples of each group were examined under a Leica S8AP0 stereomicroscope coupled to Leica DFC295 camera (Leica Microsystems, Heidelberg, Germany) with 16 x magnification that enabled to obtain clear and sufficient images to distinguish the type of fracture caused in the shear strength test.

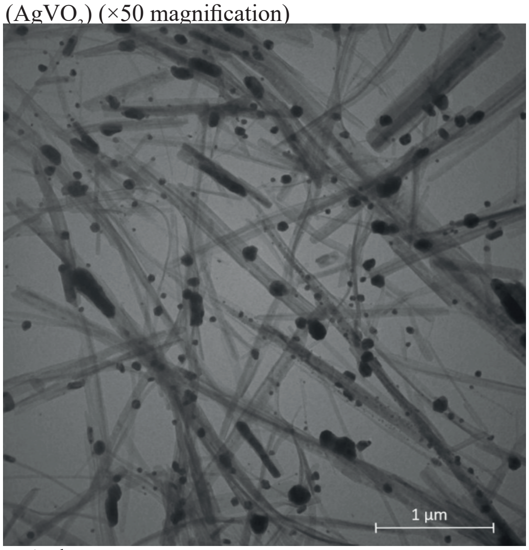
2.6 Statistical analysis

Once the data normality and homogeneity was verified, 2-way ANOVA with Bonferroni adjustments for *post hoc* comparisons (α=0.05) was used. Statistical analysis was performed using the software (SPSS v22.0, SPSS Inc.).

3 Results and Discussion

Transmission/Scanning electron microscopy showed that silver vanadate wires have an acicular pattern and are decorated with silver nanoparticles (Figure 1).

Figure 1 - Scanning transmission electron microscopy image of silver vanadate decorated with silver nanoparticles ($AgVO_3$) (×50 magnification)



Source: Authors.

Table 1 shows the bond strength values obtained by each group before and after thermomechanical fatigue.

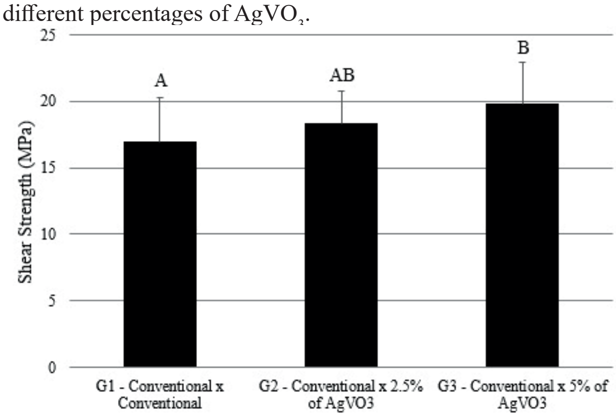
Table 1 - Mean (standard deviation) of shear strength between acrylic resins before and after thermomechanical fatigue (MPa).

Group	Before Fatigue	After Fatigue
G1 - Conventional x Conventional	16.76 (2.88)	17.11 (3.82)
G2 - Conventional x 2.5% of $AgVO_3$	17.52 (2.14)	19.18 (2.47)
G3 - Conventional x 5% of $AgVO_3$	19.83 (2.34)	19.74 (3.93)

Source: Research Data.

The bond strength between the resins increased when a concentration of 5% was added to the experimental group (p=0.016), which was statistically different from G1 - Conventional x Conventional (p=0.013). The incorporation of 2.5% promoted intermediate results (p>0.05) (Figure 2).

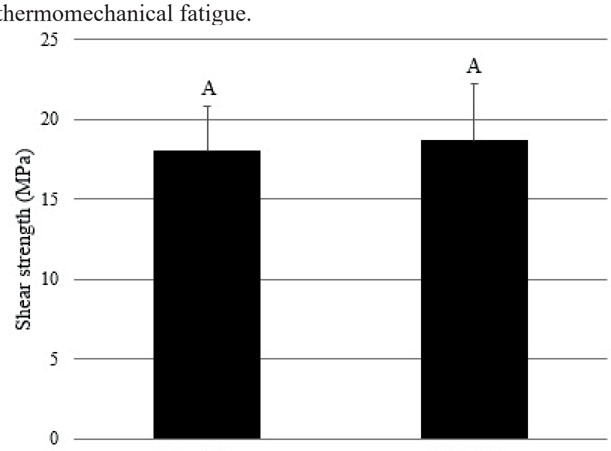
Figure 2 - Shear strength (MPa) and standard deviation: same letters indicate the statistical equality between groups with different percentages of $AgVO_3$.



Source: Research Data.

The thermomechanical fatigue process did not affect the bond strength between the resins (p=0.416) (Figure 3).

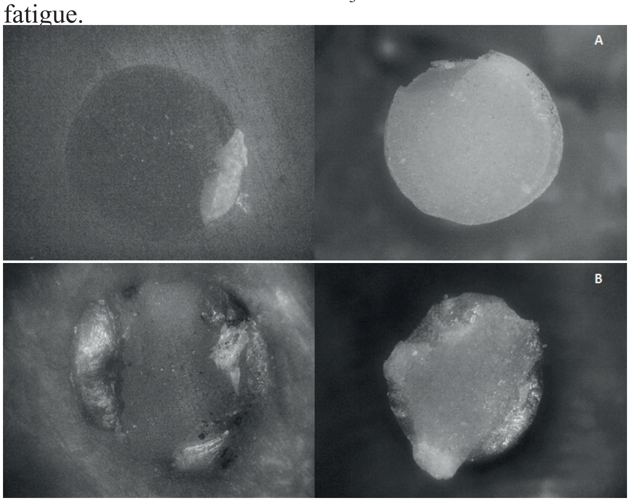
Figure 3 - Shear strength (MPa) and standard deviation: same letters indicate the statistical equality before and after thermomechanical fatigue.



Source: Research Data.

All groups presented mixed failures, both before and after the fatigue process, however, for G1- Conventional x Conventional there was a predominance of adhesive failures. Moreover, the incorporation of $AgVO_3$ favored the occurrence of failure with the highest cohesive prevalence (Figure 4).

Figure 4 - Fractographic analysis. A. Conventional x Conventional before thermomechanical fatigue; B. Conventional x 5% of $AgVO_3$ after thermomechanical fatigue.



Source: Authors.

Acrylic resins are widely used in the manufacture of conventional and implant-supported temporary prostheses[27]. However, porosity and surface roughness favor microbial adhesion and consequent oral and systemic diseases[28]. In the case of implant-supported prostheses, the pathogenic biofilm is a factor that may contribute to early and late implant loss[11].

The development of antimicrobial dental materials is an objective of dental surgeons and patients who need complementary treatment to oral conditions. This demand has developed a need in the scientific community to formulate alternatives capable of reducing the amount of pathogenic microorganisms in the oral environment[29]. However, none of them has yet been able to obtain sufficient stability and antimicrobial gains without negatively influencing the mechanical properties and/or biocompatibility of the materials[30].

Nanodentistry is an emerging area in dentistry and uses nanostructured materials to diagnose, treat and prevent oral diseases, and exhibit improved properties compared to conventional ones[31]. In particular, there is much expectation regarding the use of $AgVO_3$. Their use in controlled amounts may be able to maintain the mechanical properties of acrylic resins while promoting antimicrobial activity. Castro et al., 2016[25] found that only the 10% concentration of this nanomaterial promoted results below those required by ISO 20795-1:2008 in terms of flexural strength, when incorporated into dental polymers. Reduction in impact strength were also observed[24]. For this reason, the study presented herein proposed the use of $AgVO_3$ only in regions that are critical to the contamination of provisional and conventional implant-supported prostheses adjacent to prosthetic components and supporting tissues. Therefore, adequate bond strength between the acrylic resin used in the manufacture of the prosthesis body is required, with the acrylic resin incorporated in the antimicrobial agent.

Several mechanical tests can be used to evaluate the bonding of dental materials. Of these tests, the most widely found in the literature are the tensile[32,33] and shear tests[34,35]. In the shear test the bond is disrupted by a force applied parallel to the interface between the two materials by a tip coupled to a universal testing machine[36]. When it comes to bond strength, another important factor to be evaluated is the type of fracture that occurs. When the bond between two materials is deficient there is a greater chance for the occurrence of adhesive failures at the interface under relatively low stresses. In effective bonds, in which there are usually cohesive failures during fracture, less infiltration at the interface can be observed, reducing staining and microorganism proliferation[37].

During the clinical practice, temporary prostheses are subject to thermal challenges, due to food intake, and mechanical stress, induced during chewing[38]. The temperature variation in aqueous media can degrade the polymer, since water acting as a plasticizing agent can diffuse through it, contributing to the deterioration of the surface as well as the bonding capacity to other materials. The application of cyclic loading can produce expansion and contraction of the material, causing stress in the bonding area[39]. However, despite these evidences, in this study, the thermomechanical fatigue did not affect the bond strength of the resins assessed.

However, the concentration of $AgVO_3$ affected the bond strength. Higher bond strength values were observed between the conventional resin and the experimental resin with 5% of $AgVO_3$ (G3). This group is statistically different from the control group (G1) that evaluated the bond strength between acrylic resins without the nanomaterial. The group modified with 2.5% of $AgVO_3$ (G2) exhibited intermediate values. These results corroborate with those obtained by the fractographic analysis, which demonstrate that the incorporation of the nanomaterial favored the occurrence of mixed failures with greater cohesive predominance, suggesting that the bond was more effective. This demonstrates that nanotechnology was able to improve the dynamic behavior of polymers incorporated with $AgVO_3$, which can be explained by the formation of a microstructure produced by the nanomaterial inside the polymer matrix, capable of promoting a more efficient mechanical interaction at the interface between the two materials.

Considering these results, it can be concluded that the bond strength between conventional resin and resin incorporated with $AgVO_3$ shows to be satisfactory for this proposal to be applied in critical contamination regions without the influence of the fatigue process, and may be an alternative to eliminate the disadvantages of the use of the nanomaterial in terms of mechanical properties, in order to contribute to the hygiene and general health of patients who need provisional dental prostheses.

4 Conclusion

It is concluded that the use of $AgVO_3$ can improve or maintain the bond strength between the resins with no thermomechanical fatigue effect.

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