PONTIFÍCIA UNIVERSIDADE CATÓLICA DO PARANÁ
ESCOLA DE MEDICINA E CIÊNCIAS DA VIDA

PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA
ÁREA DE CONCENTRAÇÃO CLÍNICA ODONTOLÓGICA INTEGRADA

LUIZ FELIPE DE OLIVEIRA PEREIRA

RESISTÊNCIA DE FRATURA E DE UNIÃO AO MATERIAL DE RECOBRIMENTO DE FIBRA DE VIDRO EXPERIMENTAL CAD-CAM APLICADA EM INFRAESTRUTURA DE PRÓTESE FIXA IMPLANTO-SUPORTADA

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Tese apresentada ao Programa de Pós-Graduação em Odontologia da Pontifícia Universidade Católica do Paraná, como parte dos requisitos para obtenção do título de Doutor em Odontologia, Área de Concentração em Clínica Odontológica Integrada (Sub área Dentística).

Orientador: Prof. Dr. Rodrigo Nunes Rached

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SUMÁRIO

RESUMO

Próteses totais fixas implanto-suportadas são usualmente construídas à partir de uma infraestrutura metálica que é recoberta por uma base de resina acrílica e dentes de estoque. O metal apresenta como desvantagem estética desfavorável e adesão pobre ao material de cobertura, além de gosto ruim, biocompatibilidade questionável, e oxidação. Dessa forma a fibra de vidro pode ser proposta como material de infraestrutura por superar as desvantagens do metal. Ainda é necessário criar um protocolo de tratamento de superfície para a fibra de vidro investigada neste trabalho para otimizar a resistência de união ao material de cobertura. Além disso, é importante realizar estudos pré-clinicos, como o de resistência à fratura, para ajudar a prever o comportamento clínico da fibra como infraestrutura. No primeiro artigo, o objetivo do trabalho foi avaliar o efeito de diferentes tratamentos de superfície na resistência de união ao microcisalhamento de uma fibra de vidro experimental ao polimetil-metacrilato (PMMA). Foram confeccionados 18 discos de 25mm de diâmetro obtidos da fibra de vidro experimental e incluídos em tubos de PVC com resina acrílica. Os discos foram limpos em banhos ultrassônicos e divididos em 6 grupos de acordo com o tratamento de superfície. C: sem tratamento de superfície, S: aplicação de silano por 60s, A: jateamento com óxido de alumínio (Al_2O_3, 10s, 50µm, 2bar), M: imersão em monômero por 10s, PS: ácido fosfórico (37%, 30s) e silano (60s), APS: jateamento com óxido de alumínio, ácido fosfórico (37%, 30s) e silano (60s). Foram confeccionados 8 cilindros de resina acrílica na superfície da fibra imediatamente após o tratamento de superfície e estes foram submetidos ao teste de resistência ao microcisalhamento. A frequência dos padrões de falha foi obtida e classificada como adesiva, coesiva (na resina ou na fibra) ou mista. Os dados de resistência de união (MPa) foram analisados pelos testes ANOVA e Games-Howell (α=5%). Os resultados de resistência de união apresentaram diferenças estatisticamente significantes entre os grupos ($P<.001$). Valores de resistência de união dos grupos APS e PS foram significativamente maiores ($P<.001$), mas não apresentaram diferenças entre si ($P=.985$). O grupo S apresentou resultados significativamente maiores do que o grupo C ($P<.001$). Valores dos grupos A e C não apresentaram diferenças ($P=1.0$). Resultados do grupo M foram significativamente menores do que todos os demais grupos ($P<.008$). As falhas mais observadas foram falhas coesivas na resina no grupo APS (83,34%) seguido por PS (68,19%). Enquanto o grupo M apresentou 100% de falhas adesivas. O uso de ácido fosfórico e silano é indispensável como tratamento de superfície dessa fibra de vidro experimental. O alto índice de falhas coesiva na resina pode significar valores de resistência de união suficientemente adequados e boa resistência estrutural da fibra. No segundo artigo, o objetivo do trabalho foi avaliar a resistência a fratura após ciclos de fadiga de modelos de próteses parciais fixas fabricadas com infraestrutura de fibra de vidro usinada por CAD-CAM e compará-la com uma liga metálica pré-sinterizada de Co-Cr usinada em duas regiões de carregamento, pôntico intermediário e cantilever. Foram confeccionados 32 modelos pré-clínicos de prótese parcial fixa implanto-suportada por dois implantes em forma de barra e divididas em 4 grupos de acordo com o material de infraestrutura e local de carregamento (n=8). FP: infraestrutura de fibra de vidro e carregamento no pôntico intermediário, FC: infraestrutura de fibra de vidro e carregamento em região de cantilever, MP: infraestrutura de metal e carregamento no pôntico intermediário, MC: infraestrutura de metal e carregamento em região de cantilever. Todas as infraestruturas foram recobertas por resina acrílica termopolimerizável. Os espécimes foram submetidos a 100.000 ciclos de fadiga e ao ensaio de resistência à fratura. Valores de resistência à fratura (N) foram analisados por ANOVA à dois critérios e Tukey (α=5%). Comparando infraestruturas de um mesmo material, em regiões de carregamento diferentes, o pôntico intermediário sempre foi estatisticamente superior ($P<.001$). Em região de pôntico intermediário, a resistência à fratura do metal foi superior a da fibra ($P<.001$). Enquanto na região de cantilever os valores não apresentaram diferenças estatisticamente significantes entre o material da infraestrutura ($P=.633$). Nenhuma das infraestruturas fraturaram, assim, os padrões de falha observados nas infraestruturas de metal foram do material de cobertura que se desprendeu da peça protética. Na fibra, a fratura ocorreu também no material de cobertura, porém a resina manteve-se aderida à peça protética. Pode-se concluir que a fibra de vidro utilizada apresenta comportamento promissor, principalmente em região de cantilever. Os padrões de falha apresentados pela fibra facilitam o reparo das estruturas protéticas *in situ*.

Palavras-chave: Resistência à fratura, Resistência ao cisalhamento, Silano, Fibra de vidro, Prótese dentária.

INTRODUÇÃO GERAL

Fibras sintéticas tais como polietileno, poliaramida e vidro tem sido amplamente utilizadas na odontologia como reforços de estruturas protéticas. (1) Propriedades estéticas e adesão à matrizes poliméricas são vantagens notáveis dessas fibras sobre outros materiais utilizados para a mesma finalidade. (2) Desses materiais, a fibra de vidro destaca-se por apresentar boas propriedades mecânicas e potencial de interromper a propagação de trincas. (3–5)

Próteses totais fixas implanto-suportadas (PTFIS) são usualmente metalo-plásticas, construídas na combinação de infraestrutura em metais fundidos ou usinados por CAD-CAM, tais como ligas de CrCo, titânio ou metais nobres, e cobertura com dentes de estoque e base em polimetilmetacrilato (PMMA). (6,7) Outras opções de fabricação podem ser metalo-cerâmica, com infraestrutura em metal e cobertura em cerâmica, ou ceramo-cerâmica com infraestrutura usinada em zircônia e cobertura em cerâmica translúcida. (8,9)

Próteses implanto-suportadas não apresentam propriocepção, presente em próteses dento-suportadas e fornecida pelo ligamento periodontal. (10) Essa condição favorece o uso de materiais de baixo módulo de elasticidade, tais como resinas. (11) Materiais de alto módulo de elasticidade, como cerâmicas, são friáveis e favorecem fraturas e lascamentos em situações de estresse com cargas estáticas ou dinâmicas à longo prazo. (12)

Usualmente PTFIS são construídas com infraestrutura em metal. A inclusão de metal nas bases acrílicas fornece resistência e rigidez, porém apresenta adesão pobre ao PMMA, o que pode comprometer o sucesso do tratamento reabilitador. (3,13) A fibra de vidro foi proposta como infraestrutura de PTFIS, apresentando os benefícios de reforço mecânico, com boas propriedades estéticas e adesão à matrizes poliméricas. (14–16) Alguns tipos de PTFIS construídas por fluxo convencional/analógico de trabalho reforçadas com fibras de vidro experimentais trançadas foram reportadas como boas próteses provisórias de longa duração. (15,16)

A adesão entre material de infraestrutura e de cobertura pode ser determinante no sucesso da reabilitação protética. (5) Além disso, os padrões de falha presentes em próteses com infraestruturas metálicas normalmente são fraturas catastróficas na base acrílica que se desprendem da prótese e dificultam o reparo. (2) Já as estruturas reforçadas com fibra, apresentam fraturas que ficam aprisionadas pela adesão do material resinoso à fibra, o que facilita o reparo. (17)

Apesar da questionável relação entre testes de resistência de união in vitro e a performance clínica de determinados materiais, ambas informações guiaram a tomada de decisões do clínico ao passar dos anos. (18) Testes laboratoriais constituem etapa fundamental na pesquisa e desenvolvimento de materiais odontológicos, previamente aos testes clínicos e lançamento do produto ao mercado. (19) Outros autores acreditam que os resultados clínicos podem ser estimados com base nos testes laboratoriais de resistência de união. (20) Uma das metodologias mais utilizadas para definir a resistência de união de materiais dentários é o teste de microcisalhamento. (21) Esse teste é realizado em espécimes com pequenas áreas aderidas, normalmente abaixo de $2mm^2$, com o intuito de eliminar ruídos e gerar resultados mais precisos. (22)

Fatores como resistência à fratura não estão ligados a técnica utilizada para a construção das próteses, mas sim à propriedades mecânicas básicas inerentes aos materiais. (23) A fratura de uma estrutura protética está ligada às condições de cargas aplicadas, mas está principalmente relacionada à resistência do material utilizado. (24) Por isso é importante conhecer a resistência à fratura dos materiais utilizados para a construção desse tipo de prótese e, assim estimar o comportamento clínico da estrutura protética.

Boa parte das propriedades mecânicas de um material são estimadas através de protocolos de carregamento estáticos, tais como módulo de elasticidade, resistência flexural, e resistência à fratura. (25,26) Entretanto, os eventos de sobrecarga que induzem a fratura em ambiente oral são raros. (27) Assim, com o intuito de simular condições e carregamentos mais fiéis às encontradas na cavidade oral e na mastigação, propõe-se a utilização de carregamentos dinâmicos, para a indução de fadiga. (25) Alguns estudos sugerem que estas condições são mais preditivas do comportamento clínico dos materiais. (24,25)

A fibra de vidro usinável usada neste estudo é um material experimental desenvolvido pela *Angelus Indústria de Produtos Odontológicos S/A* para infra-estrutura de próteses parciais ou totais fixas dento- ou implanto-suportadas. Espera-se que uma fibra de vidro usinável aplicada como infra-esturtura de prótese apresente adesão aos materiais de cobertura, com as vantagens adicionais inerentes ao fluxo digital tais como eliminação do passo de moldagem, adaptação superior, melhor resistência pela distribuição das fibras, propriedades isotrópicas e espessura homogênea.

Nesse sentido, esta tese tem dois objetivos. No primeiro artigo avaliou-se o efeito de diferentes tratamentos de superfície na resistência de união ao microcisalhamento de uma fibra de vidro experimental ao polimetil-metacrilato (PMMA). A hipótese nula testada foi que o tratamento de superfície empregado na fibra não levaria a uma diferença estatisticamente significante nos valores de resistência de união entre fibra de vidro e PMMA.

No segundo artigo, o objetivo foi avaliar a resistência a fratura após ciclos de fadiga de modelos de próteses parciais fixas fabricadas com infraestrutura de fibra de vidro usinada por CAD-CAM e compará-la com uma liga metálica pré-sinterizada de Co-Cr usinada em duas regiões de carregamento, pôntico intermediário e cantilever. A hipótese testada foi que o material da infraestrutura e o local de carregamento não levaria a uma diferença estatisticamente significante nos valores de resistência à fratura.

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ARTIGO 1

Title page

Bond strength of veneering material to an experimental CAD-CAM glass fiber for fabrication of fixed-bridges infrastructures

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Abstract

The aim of the present study is to evaluate the effect of different surface treatments on microshear bond strength of an experimental CAD-CAM glass fiber to polymethyl-methacrylate (PMMA). Eighteen discs (25mm in diameter) were fabricated from the experimental block. The discs were cleaned in ultrasonic bath in distilled water for 180s and divided in six groups according to the surface treatment. C: no surface treatment, S: silane for 60s, A: air-born abrasion (Al_2O_3, 10s, 50µm, 2bar), M: immersion into MMA monomer for 10s, PS: phosphoric acid (37%, 30s) and silane (60s), APS: air-born abrasion, phosphoric acid application and silane. A silicon mold was used to build eight acrylic resin cylinders (1 mm in diameter) over each disc immediately after surface treatment. The specimens were stored in distilled water for 24h under 37ºC and then submitted to a microshear bond strength test until failure (0.5mm/min). Values for bond strength (MPa) were obtained and failure modes were determined using light microscopy under 50x magnification. Shapiro-Wilk test revealed normal distribution (p>0.05) and Levene's test revealed heterogeneity of variances (p<0.05). Data was analyzed using 1-way ANOVA and Games-Howell test (α=5%). The microshear bond strength results were significantly different among groups (P<.001). Bond strength values of the APS and PS groups were significantly higher (P<.001), but no significant differences were observed among each other (P=.985). Results of the S group presented significantly higher bond strength compared to C group (P<.001). Bond strength values for A and C presented no significant differences (P=1.0). Results for M group were significantly lower than that of all groups (P<.008). The most frequent failures observed on APS were cohesive fails at the veneering material (83.34%) followed by PS (68.19%) while the M group presented 100% of adhesive fails. Cohesive fails at the fiber or mixed fails were not observed. The use of silane associated to phosphoric acid as surface treatment increased microshear bond strength of the glass fiber to PMMA. The use of phosphoric acid and silane should be encouraged as surface treatment before veneering the experimental fiber with PMMA. The high prevalence of cohesive failures at the resin associated to high values of bond strength represents an evidence of good structural resistance of the fiber and sufficient bond strength between the fiber and the veneering material.

Keywords: Microshear bond strength, Silane, Dental prosthesis, Glass Fiber.

1. Introduction

Implant-fixed complete dentures (IFCD) are typically fabricated using a metallic infrastructure, stock teeth, and an acrylic resin base. (1) While cast metal or milled alloys offer excellent mechanical properties for prosthetic frameworks, they exhibit drawbacks such as poor adhesion to the veneering material and limited esthetic appeal. (2) As an alternative, metal-free materials have been proposed and have shown satisfactory longevity. (3) Zirconia infrastructure provides a comparable reinforcing effect with enhanced esthetic properties, though at a higher cost compared to other treatment options. (4)

Fiber-reinforced composites (FRC) serve as a metal-free option for fabricating infrastructure of teeth- or implant-supported partial and complete fixed dentures. (5) FRC offers advantages in terms of esthetics, cost, and effective adhesion to PMMA veneering material. (6) The use of beam-shaped analogic manual manufacturing glass fiber seems to be promising. (7) Additionally, a case report on an IFCD described the use of a FRC prosthetic framework as a favorable long-term interim restoration. (8) Furthermore, an in vitro study reported intermediate fracture resistance values when compared to metal frameworks, with more favorable failure modes. (9)

The adhesion between the infrastructure and the veneering material plays a crucial role in the clinical success and longevity of a prosthetic framework. (10)
Biomechanical behavior and more favorable failure patterns is observed on implant-fixed prostheses when the infrastructure is bonded to the veneering material. (7,11) Catastrophic failures, characterized by the fracture of the prosthesis into two pieces, hinder the repair procedure. (12) Such failures are more commonly observed with metallic frameworks due to their poor adhesion capabilities. (13)

Although FRC exhibit the potential for favorable bonding to resinous materials, a high prevalence of veneer fractures attributed to inadequate bond strength between the infrastructure and veneering material. (11) Therefore, the surface treatment of the fiber plays a crucial role in determining the prognosis of the prosthesis by enhancing micromechanical interlocking. (14,15)

Various surface treatments have been used to enhance the bond strength to glass fibers. (16) One of these methods is sandblasting achieved with aluminum oxide particles, which promotes micromechanical retention on the surface of different dental materials, including metals, ceramics, polymers, fibers and even dental substrates to improve bond strength. (17) Additionally, considering the fiber network, design, and composition, certain low molecular weight monomers are able to dissolve the linear polymeric chain improving bonding between FRC and resinous veneering material. (18)

Silane treatment is recommended to promote reliable chemical bonding of resin-based materials to glass fibers. It increases the wettability of the glass fiber surface, allowing resin penetration, and establishes a covalent bond by exposing a functional methacrylate group that can co-polymerize with the resin matrix. (19) Another surface treatment, etching using 37% phosphoric acid, selectively removes the superficial epoxy resin layer while maintaining the fiber integrity. (20)

Hydrofluoric acid has been proposed to treat the surface of glass fibers. However, it shows low bond strength values (21) due to the creation of voids in the polymeric matrix, which allows water sorption and subsequent hydrolysis of the silane. (22) It is important to note that information regarding FRC bond strength and surface treatments is limited to push-out and pull-out methods using intra-radicular posts. (23,24)

Few studies have evaluated surface treatments and bond strength of glass fibers used in prosthesis infrastructures. (25,26) It is important to evaluate the effect of surface treatments on the microshear bond strength of this experimental fiber to understand its behaviour and determine the correct protocol for the application of the material. Therefore, the aim of the present study is to evaluate the effect of various surface treatment protocols on the microshear bond strength of an experimental CAD-CAM mesh glass fiber prior to bonding with its veneering material. The null hypothesis of this study was that the surface treatment would not have significant influence on the microshear bond strength values between the fiber and veneering material.

2. Materials and Methods

The materials used in this study are described in Table 1. A total of 18 discs (Ø25mm and 2mm thickness) were obtained by milling an experimental glass fiber CAD-CAM block. The discs were included into polyvinyl chloride (PVC) tubes using acrylic resin to facilitate its fit to the universal testing machine (Figure 1).

Table 1. Experimental fiber, veneering resin and materials used on surface treatment before bonding.

Materials (Manufacturer)	Description	Application mode
Experimental glass fiber (Angelus, Londrina, PR, Brazil)	Experimental Glass Fiber	Milled by CAD-CAM
Jet (Classico, São Paulo, SP, Brazil)	Self-polymerizing acrylic resin (liquid and powder)	Veneering material used for microshear bond strength specimen construction
Prosil (FGM, Joinvile, SC, Brazil)	Silane	Applied for 60s on the surface and air dried of the fiber
Condac 37(FGM, Joinville, SC, Brazil)	Phosphoric acid 37%	Applied for 30s on the surface of the fiber
Jet (Classico, São Paulo, SP, Brazil)	Self-polymerizing acrylic resin monomer (liquid)	Applied for 10s on the surface of the fiber
MicroJato Standard (Bio Art, São Carlos, SP, Brazil)	Aluminum Oxide Sandblasting	Al_2O_3, 50μm, 10s, 2bar

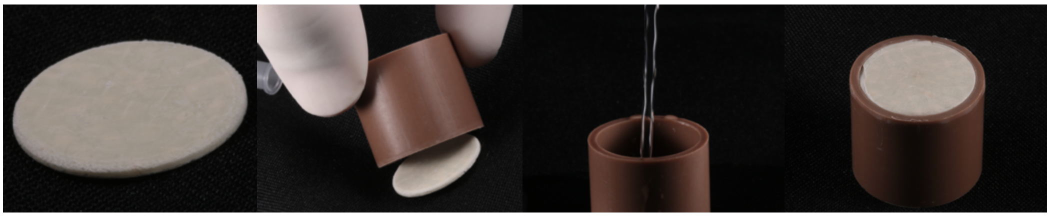

Figure 1. Disc obtained from the glass fiber block embedded on PVC tubes using acrylic resin.

A master cast with 8 equidistant cylinders (Ø1mm) was milled from CAD-CAM PMMA blocks. This master cast was molded with a light body polyvinyl siloxane. This mold was used to construct the specimens for the microshear bond strength tests.

All fiber discs were cleaned on ultrasonic baths in distilled water for 180s and air dried. The discs were divided into 6 groups according to the surface treatments. (Table 2)

Table 2. Description of the experimental groups according to the surface treatment

Group	Description
C	No surface treatment applied
S	Silane application (60s), air-drying
A	Sandblasting (Al_2O_3, 50µm, 10s, 2 bar), and rinsing (60s)
M	Methyl methacrylate self-polymerizing monomer application (10s), air-drying
PS	Phosphoric acid 37% application (30s), silane application (60s), air-drying
APS	Sandblasting (Al_2O_3, 50µm, 10s, 2 bar), phosphoric acid 37% (30s), silane (60s) and air drying

Immediately after the surface treatment, self-polymerizing acrylic resin was manipulated, inserted into a syringe, and poured into the silicon mold to build the cylinders (1mm diameter and 2mm high). Each group had 24 specimens. The acrylic resin was polymerized into a compression capsule (25 bar) to avoid gaps or void into the resin (Figure 2).

Figure 2. A. PVC ring with embedded glass fiber; B. Silicon mold with 1mm diameter orifices; C. Injection of PMMA into the orifices; D. PMMA cylinders for microshear bond strength test.

After polymerization of the acrylic resin, the silicon mold was removed carefully using manual instruments to avoid damage to the specimens. All the specimens were analyzed using light microscopy (Olympus BX60, Olympus Corp, Tokyo, Japan) under 50x magnification to identify possible flaws, such as gaps, voids, bubbles or other defects. Specimens presenting these defects were excluded from the study. Specimens were stored in distilled water at 37ºC for 24h.

Specimens were placed on the universal testing machine (EMIC 2000 Instron, Illinois Tool Works Inc, Norwood, MA) and tested in microshear testing device using a load cell of 50N with a force applied to the adhesive interface until failure. (Figure 3) The specimens were loaded at 0.5mm/min crosshead speed. Values for microshear bond strength were obtained in N and converted to MPa. Pre-testing failures were recorded as 0 MPa.

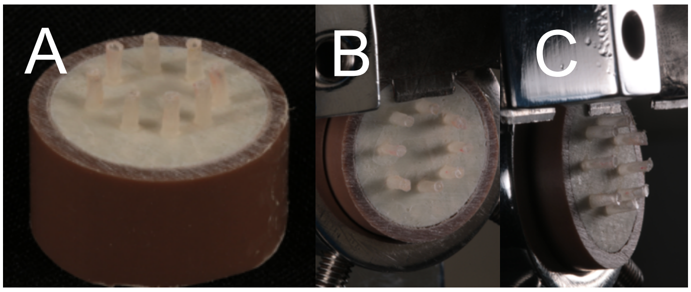

Figure 3. (A) Eight specimens on the fiber surface, and microshear testing device applying load to the adhesive interface of the specimens on the universal testing machine (B) frontal and (C) lateral view.

The mode of failures was determined using light microscopy under 50x magnification and registered as ‘adhesive’, ‘cohesive’ at the resin or the fiber, or ‘mixed failure’. The frequency of the failure modes was recorded.

Micro-shear bond strength values of all specimens were reported as mean and standard deviation (SD). The bond strength data of the groups were normally distributed (Shapiro-Wilk, $P>.05$), and Levene’s test revealed heterogeneity ($P<.001$). The data were compared using a 1-way analysis of variance (ANOVA) and Games-Howell post-hoc non parametric test was used for pairwise comparison ($\alpha=5\%$). A statistical software program (IBM SPSS Statistics, v24.0; IBM Corp) was used for the analysis.

3. Results

The microshear bond strength results were significantly different among groups (P<.001). Bond strength values of the APS and PS groups were significantly higher (P<.001), but no significant differences were observed among each other (P=.985). Results of the S group presented significantly higher bond strength compared to C group (P<.001). Bond strength values for A and C presented no significant differences (P=1.0). Results for M group were significantly lower than that of all groups (P<.008). (Table 3 and Figure 4)

Table 3. Mean values, standard deviation, and significant differences for microshear bond strength (MPa).

Group	Mean±SD
C	16.61±4.9 [c]
S	24.66±8.65 [b]
A	16.93±5.35 [c]
M	11.34±3.47 [d]
PS	36.60±11.59 [a]
APS	38.99±13.04 [a]

Groups with same superscript letters not significantly different (Games-Howell P>.05)

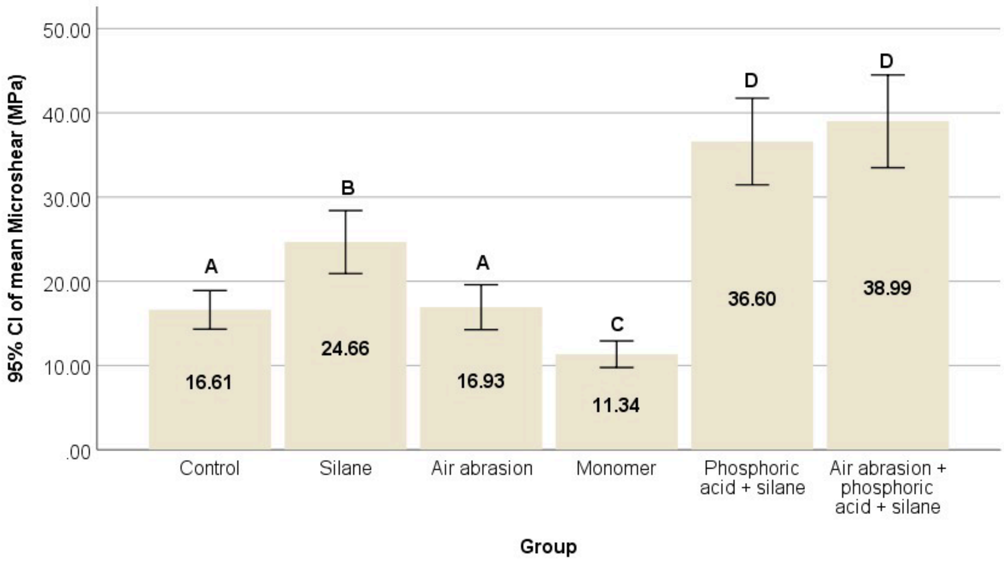

Figure 4. Bar chart showing mean and confidence interval of bond strength values. Crossed confidence intervals reveals no statistical differences among groups. Same capital letters means no significant differences (Games-Howell P>.05).

The failure modes observed were described as adhesive or cohesive at the resin. Mixed or cohesive failures at the fiber were not observed. (Table 4) Representative images of the failures were obtained by light microscopy. (Figure 5)

Table 4. Absolute and relative frequency of failure modes between the groups.

Group	Cohesive failures at the Resin	Adhesive failures
C	4 (20%)	16 (80%)
S	10 (43.47%)	13 (56.53%)
A	5 (25%)	15 (75%)
M	0 (0%)	21 (100%)
PS	15 (68.19%)	7 (31.81%)
APS	20 (83.34%)	4 (16.66%)

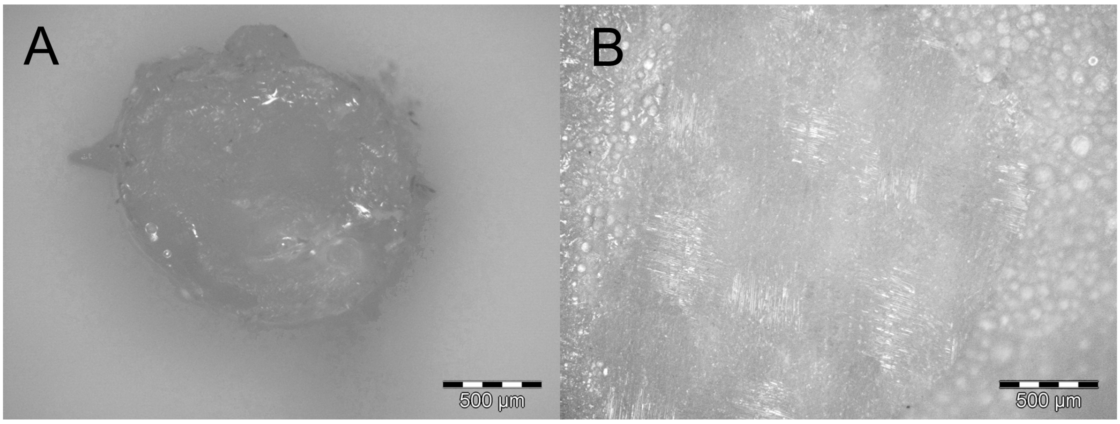

Figure 5. Representative images of (A) cohesive failures at the resin and (B) adhesive failure under 50x magnification.

4. Discussion

The bond strength between the infrastructure and veneering material of prosthetic frameworks is a critical factor in determining their clinical longevity. This experimental study aimed to evaluate the in vitro microshear bond strength of a CAD-CAM glass fiber, specifically indicated for the fabrication of infrastructure in implant-supported fixed partial or complete dentures, following various surface treatment protocols. The findings of this study demonstrates that the use of a silane coupling agent resulted in higher bond strength, while sandblasting of the fiber surface is unnecessary. Furthermore, the immersion of the fiber in a monomer solution was found to have a negative impact on bond strength. As a result, the null hypothesis that the surface treatment would not have significant influence on microshear bond strength values between fiber and veneering material was rejected.

The use of glass fiber as reinforcement in removable denture bases, veneered with PMMA, resulted in increased flexural strength, impact strength, and fatigue resistance, acting as crack stoppers. (27) Furthermore, fibers have been proposed as infrastructures for dental- and implant-supported fixed prostheses. (7–9) The use of a manufactured glass fiber infrastructure technique is easier and a less time-consuming alternative compared to traditional metal-plastic or metal-ceramic prosthesis. (8) Unidirectional and braided glass fibers used in implant-supported fixed complete dentures infrastructures is suitable for long-term interim restorations, compared to titanium infrastructures and unreinforced prosthetic frameworks. (7) However, a finite element analysis study demonstrated that implant-supported complete dentures showed similar stress distribution patterns when using metallic or fiber infrastructures. (28) Despite the promising aspects of manually constructed frameworks using glass fibers, there are inherent disadvantages related to their handling and manufacturing. (9) As a result, the reported results showed high variation, thus they are typically considered semi-permanent restorations. (29–31) Therefore, CAD-CAM glass fiber milled infrastructures could offer an optimal solution to overcome these disadvantages by standardizing their fabrication and precisely calculating the size and geometry according to the final shape of the prosthesis.

Silane is widely recognized and employed by clinicians and researchers as a surface treatment due to its ease of use, low sensitivity to technique, and reliable bonding performance. (18) The bonding mechanism of silane to glass relies on the formation of a strong siloxane linkage through condensation with surface hydroxyl groups on the substrate. Additionally, silane forms a bond with the resin by reacting with the organofunctional groups of silane and the functional groups of the monomers, facilitated by reactive free radicals (16) The bond between the glass fiber and the resin matrix further contributes to the enhancement of mechanical and physical properties. (32) In the present study, all groups subjected to silane surface treatment demonstrated superior performance, particularly when preceded by phosphoric acid treatment. To the best of the authors' knowledge, there is a dearth of published information concerning this experimental fiber and its bond strength. Literature reviews consistently emphasize the imperative role of silane application on commonly employed fiber-reinforced composites (FRCs), as it significantly enhances the bonding between the fibers and the resin-based materials. (16,33)

Bonding to non-silica based materials using silane alone may not provide adequate results, and a specific surface pretreatment, such as acid etching, can help overcome this limitation. (34) While some manufacturers recommend phosphoric acid as a surface treatment for glass fiber posts, phosphoric acid acts primarily as a cleaning agent since both the fibers and resin matrices are inert to this substance. (25) However, in the present study phosphoric acid played a significant role as a surface pretreatment prior to silane application, as demonstrated by the superior bond strength observed in the APS and PS groups. Similar findings were reported in a previous study which emphasized the essential role of phosphoric acid in improving the adhesion of FRC. (35)

Sandblasting with aluminum oxide particles is commonly recommended as a surface treatment before resin bonding for various indirect restorative materials. (36) Typically, it involves the use of 30 to 110 µm particles for 5 to 15 seconds, under a constant air pressure of 2 to 4 bar, perpendicular to the surface at a short distance. (22) The purpose of air abrasion is to increase surface roughness, which enhances bond strength through micro-mechanical interlocking. (20,25) In the present study, the groups which were sandblasted presented no statistical differences on bond strength values.

Fiber reinforced composite resin matrix can be composed of different monomers and polymers, such as PMMA, Bis-GMA, Bis-EMA, TEG-DMA, UDMA and others. (37) Resin MMA monomers are able to partially dissolve semi-interpenetrating polymer networks (Semi-IPN), which can improve bond strength of FRC substrates. (38) This benefit is especially usefull after polymerization is complete and there is absence oxygen inhibition layer, (39) which is observed in the experimental CAD-CAM glass fiber used for the fabrication of the specimens of FP and FC groups. Although fresh monomers are able to penetrate semi-IPN FRC, this surface treatment is not effective on cross-linked polymers FRC. (40) In the present study, the use of MMA monomer prior to veneering material showed the lowest bond strength values, even worse than control group, which had no surface treatment.

Groups which presented the higher values of microshear bond strength, such as S, PS, and APS, also showed mostly cohesive failures, which could be an indication of favorable adhesion. (41) Moreover, the cohesive failures were predominantly observed in the veneering material, which can be an indicative of good structural resistance of the FRC. (42) Contrarily, groups with lower bond strength, such as C, A, and M, presented lower means of bond strength values and higher frequency of adhesive failures.

The limitations of the study can be attributed to the lack of information available in the literature regarding the experimental fiber used in this research, which hinders comprehensive comparisons of microshear bond strength results. Additionally, alternative surface treatments, such as silica-coating, different types of acid etching, or laser treatments, were not evaluated in this study. Furthermore, the bonding behavior of phosphoric acid at different application times, could also be investigated in future research. It should be noted that the results obtained in this study are specific to the particular fiber investigated. Further research is necessary to investigate the adhesion between resin cements and the fiber, particularly when bonding the fiber to implant abutments. Additionally, a comprehensive understanding of the bonding patterns between the fiber and composite resin veneering material is needed. Further laboratory investigations and randomized clinical trials are necessary to better comprehend the biomechanical behavior of this fiber.

5. Conclusion

Based on the findings of this *in vitro* study, surface treatment with phosphoric acid and silane as surface treatment provided higher bond strength to the veneering material. Sandblasting with aluminum oxide did not improve bond strength when used alone or associated to silane. Self-polymerizing acrylic resin monomer immersion is not indicated as surface treatment for this kind of fiber, since it reduces the bond strength.

Cohesive failure modes at the resin, associated to high values of bond strength, observed in the groups which used phosphoric acid and silane as surface treatment, may be a good indicative of sufficient bond strength and good structural resistance of the experimental fiber.

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ARTIGO 2

Title page

Fracture resistance of an implant-supported fixed partial prosthesis fabricated with an experimental CAD-CAM glass fiber infrastructure

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Abstract

The aim of the present study is to evaluate the fracture resistance of implant-supported fixed partial prosthesis models with experimental CAD-CAM milled glass fiber infrastructure compared to milled pre-sintered Co-Cr infrastructure loaded in two different areas, intermediate pontic and cantilever. Thirty-two epoxy resin models were constructed from a master cast with two abutment analogues 20mm apart. The models were scanned with a bench scanner and the infrastructures (n=16) were milled either from cobalt-chromium alloy (Ceramill Sintron, Amann Girrbach) or experimental glass fiber and veneered by thermopolymerized acrylic resin with a 20mm cantilever. The specimens were randomly assigned into four groups based on its loading site and infrastructure material: FP – Glass fiber infrastructure loaded on the intermediate pontic; FC – Glass fiber infrastructure loaded on cantilever; MP – Cast metal infrastructure loaded on the intermediate pontic; and MC – Cast metal infrastructure loaded on cantilever. The specimens were submitted to fatigue loading (50N, 100 000, 1Hz, Biocycle, Biopdi) and fracture loading (Emic, instron). Values for facture resistance were obtained and the fracture patterns were observed using optical microscopy. Shapiro-Wilk test revealed normal distribution ($p>0.05$) and Levene's test revealed homogeneity of variances ($p>0.05$). Data was analyzed using 2-way ANOVA, for infrastructure material and loading site, and Tukey test ($\alpha=5\%$). The fracture resistance results were significantly different among groups ($P<.001$). Fracture resistance of MP were significantly higher among groups ($P<.001$). Results for MC and FC were significantly lower among groups ($P<.001$), but no statistical differences were observed between them ($P=.633$). The failure modes observed were more critical on the metal groups where the fractured pieces debonded from the infrastructure. The experimental fiber infrastructure seems to be a promising material, especially on cantilever pontics. The failure patterns observed on the fiber may facilitate the repair *in situ*.

Keywords: Fatigue, Fatigue Fracture, Fracture resistance, Dental prosthesis, Implant and Prostheses

1.Introduction

The restoration of edentulous spaces using implant-supported fixed partial prostheses has become a widely accepted treatment modality in modern restorative dentistry. (1,2) Implant-supported fixed partial prostheses offer numerous advantages, including improved oral function, aesthetics, and patient satisfaction. (3) Traditional frameworks for such prostheses have primarily relied on metal-based materials, which provide excellent mechanical properties but may present limitations in terms of esthetics and potential complications such as corrosion and allergic reactions. (4–6) Consequently, there has been growing interest in the development of alternative materials that can combine the desirable mechanical properties of metal frameworks with improved esthetics and biocompatibility. (7,8) One material showing promise in this regard is glass fiber-reinforced composite (FRC) infrastructure. (9)

Fibers have found extensive applications in dentistry, assuming various forms such as posts, ribbons, bands, mesh, beams, and more recently, CAD-CAM blocks or discs. (10) These fibers exhibit diverse properties and reinforcement behavior dictated by their direction, geometry, and distribution. (11,12) Different applications have been identified, including the use of fibers as infrastructures for dental prostheses to enhance mechanical properties. (13) Among dental fibers, FRC stand out due to their exceptional esthetic properties, dentin-like elastic modulus, and favorable mechanical characteristics. (14) Moreover, FRC has been suggested as a viable alternative to traditional metal casts. (8) Comparisons between metal and fiber-based structures are a common subject in dentistry, since metallic frameworks present poor adhesion, compromised esthetics, and high elastic modulus. (15)

Although FRC has gained attention in restorative dentistry, there is limited information available regarding the effect of fatigue and fracture loading on implant-supported fixed partial prosthesis models incorporating CAD-CAM glass fiber infrastructure. (13) Understanding the behavior of these prosthetic systems under cyclic loading conditions is crucial for ensuring their long-term durability and clinical success. (16) Moreover, as implant-supported restorations are subjected to occlusal forces during function, it is essential to investigate the performance of FRC infrastructure under conditions that mimic the dynamic oral environment. (17)

Therefore, there is a need for comprehensive studies examining the fatigue and fracture properties of implant-supported fixed partial prosthesis models with glass fiber infrastructure to fill this research gap.

The utilization of a manually fabricated glass fiber infrastructure technique is described as a more convenient and time-efficient alternative to conventional metal-plastic or metal-ceramic prosthesis. (18) A study which investigated the fracture resistance of unidirectional and braided glass fibers used in implant-supported fixed complete dentures, revealed their suitability for long-term interim restorations compared to titanium infrastructures and unreinforced prosthetic frameworks. (7) However, a finite element analysis study demonstrated that the stress distribution patterns in fixed implant-supported complete dentures were similar regardless of whether metallic or fiber infrastructures were employed. (19) Although manually constructed frameworks using glass fibers show promising results, they are associated with inherent challenges in handling and manufacturing. (13) Consequently, the reported outcomes are heterogeneous, leading to their classification as semi-permanent restorations. (20–22) To address these limitations, CAD-CAM glass fiber milled infrastructures represent an advantageous approach, allowing for standardized fabrication and precise calculation of size and geometry in accordance with the final prosthesis shape.

Therefore, the objective of this study is to evaluate the effect of fatigue and fracture loading on implant-supported fixed partial prosthesis models with experimental CAD-CAM milled glass fiber infrastructure compared to soft metal milling cobalt-chromium alloy infrastructure loaded in two different areas, intermediate pontic and cantilever. The null-hypothesis was that the loading protocol or infrastructure material would not have a significant influence on the fracture strength of the prosthesis.

2. Materials and Methods

A master cast was constructed using two mini-abutments transfers on a flat glass surface and splinted using acrylic resin, with a standardized 20mm distance. The transfers were screwed to abutment analogues and positioned on a acrylic resin base. (Figure 1)

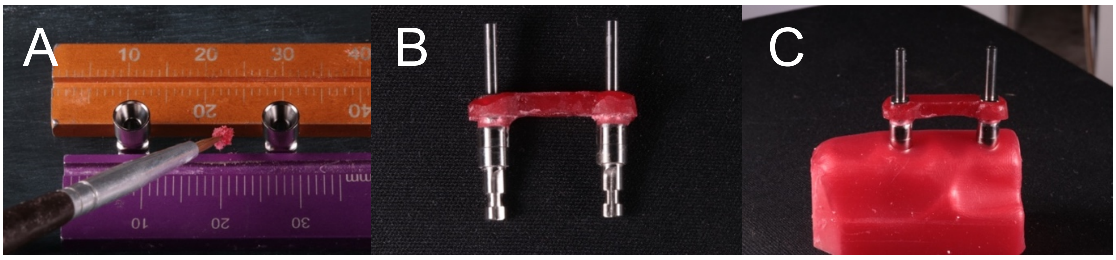

Figure 1. A. Splinting abutment transfers standardizing 20mm distance; B. abutment analogues screwed to the transfers; C. Abutment analogues placed on the master cast.

Individual acrylic resin open-trays were constructed and polyvinylsiloxane molds were obtained from the master cast. Thirty-two epoxy resin models were constructed using two abutment analogues each. (Figure 2)

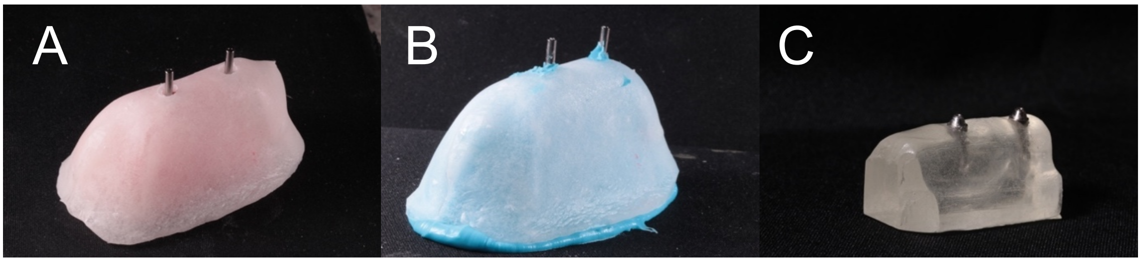

Figure 2. A. Custom tray built for impression of the master cast; B. Polyvinil Siloxane impression; C. Epoxy resin model.

Two materials were used to fabricate the prosthetic infrastructures (Table 1). The epoxy resin models were scanned using a bench scanner (Ceramill Map 400, Amann Girbach) and sixteen frameworks (N=32) were designed (Ceramill Mind, Amann Girbach) and milled using the experimental fiber and a pre-sintered Co-Cr metallic alloy (Ceramill Motion 2, Amann Girbach).

Table 1. Infrastructure materials.

Material (Manufacturer)	Description
Experimental Glass Fiber (Angelus, Londrina, PR, Brasil)	Experimental CAD-CAM Glass Fiber
Ceramill Sintron (Amann Girrbach, Koblach, Voralrberga, Austria)	Soft metal milling cobalt-chromium alloy

The frameworks were divided in four groups (n=8) according to its material, which could be fiber or metallic, and loading protocol, which could be on the intermediate pontic or on cantilever region. (Table 2)

Table 2. Description of the experimental groups.

Group	Infraestructure	Loading protocol
FP	Experimental Glass Fiber	Intermediate pontic
FC	Experimental Glass Fiber	Cantilever pontic
MP	Soft metal milling cobalt-chromium alloy (Sintron)	Intermediate pontic
MC	Soft metal milling cobalt-chromium alloy (Sintron)	Cantilever pontic

The milled infrastructures had standardized dimensions (5x5x40mm). The metallic infrastructures were built directly on the abutment platform. The fiber infrastructure was luted to a screw retained titanium base using a self-adhesive resin cement (Relyx u200, 3M ESPE, St Paul, MN, USA) and placed on the abutment platform (Figure 3).

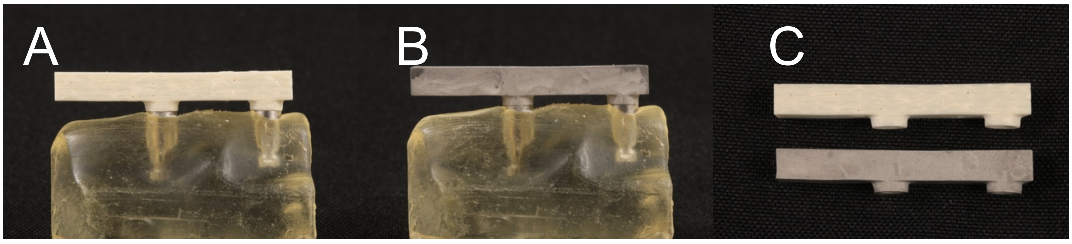

Figure 3. A. Fiber infrastructure screwed to the epoxy resin model; B. Metallic infrastructure screwed to the epoxy resin model; C. Identical design and shape of the glass fiber and metallic infrastructure.

The passive fit and correct adaptation of the infrastructure was confirmed by the one-screw test. (23) Both, metal and fiber, infrastructures were veneered using thermopolymerized acrylic resin (Clássico, Clássico Artigos Odontológicos, São Paulo, SP, Brasil). The fiber surface treatment before veneering was performed according to the previous study. The metal surface treatment consisted of aluminum oxide sandblasting (10s, 50um, 2bar) and application of a metallic primer based on 10-MDP functional monomer. The final dimensions of the specimens were 8x8x43mm, since the acrylic resin was 1.5mm thick. (Figure 4) Transparent acrylic resin was selected for better observation of the fracture behavior of the fiber infrastructure. The specimens were screwed to the epoxy resin models using a torquemeter (10N) as recommended by the manufacturer.

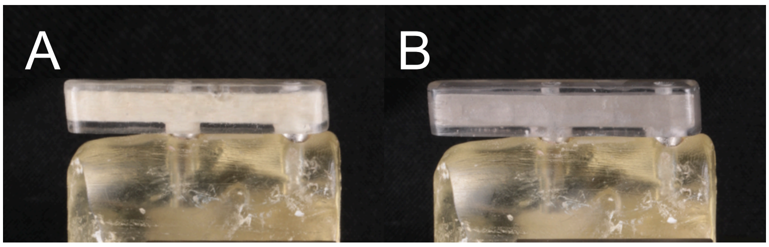

Figure 4. A. Fiber infrastructure with PMMA veneering material; B. Metallic infrastructure with PMMA veneering material.

The 16 specimens from each group were submitted to 100,000 fatigue cycles (50 N, 1 Hz) using a mechanical cycling machine (Biocycle, Biopdi). According to the loading protocol, half of the specimens had the loading sphere positioned between implants, on intermediate pontics (P), and the other half will on the most distal portion of the cantilever region (C), as illustrated in Figure 5. The specimens were continually evaluated to analyze possible failures. In case of mechanical failure, the specimen would be removed from the fatigue machine and excluded from the study. The specimens that survived all fatigue cycles were subjected to a fracture test using a universal testing machine (EMIC DL2000, Instron Corp.) with a load cell of 2000N at a speed of 1mm/min. The fracture resistance values will be obtained in Kgf and converted to N (Figure 6).

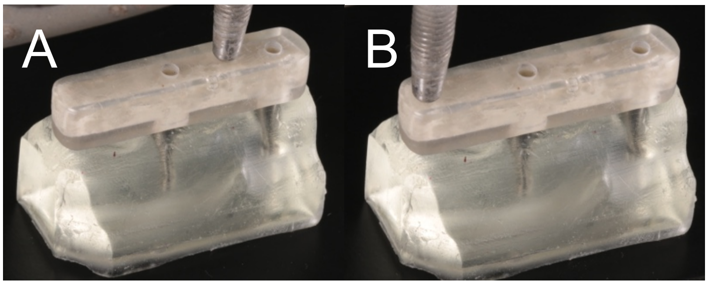

Figure 5. A. FP loading protocol, on intermediate pontic; B. FC loading protocol, on cantilever region, both placed on the cycling machine.

The failure patterns were observed and categorized as the repetition of the events. Fracture resistance values of all specimens were reported as mean and standard deviation (SD). The fracture resistance data of the groups were normally distributed (Shapiro-wilk, $P>.05$), and Levene's test revealed homogeneity of variances ($P>0.05$). The data were compared using 2-way analysis of variances (ANOVA) for loading protocol and infrastructure material and Tukey post-hoc test was used for pairwise comparison ($\alpha=5\%$). A statistical software program (IBM SPSS Statistics, v24.0; IBM Corp) was used for the analysis.

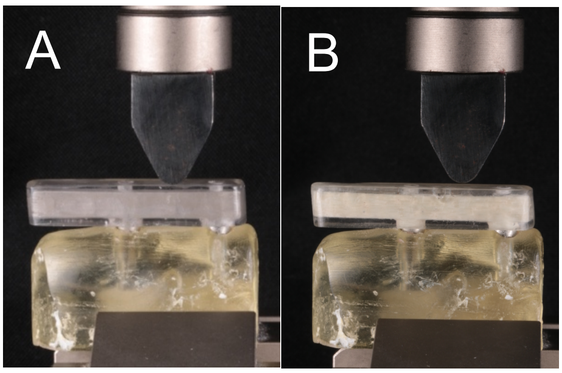

Figure 6. A. MP; and B. FP loading protocol on the fracture resistance testing machine (EMIC DL2000, Instron Corp.).

3. Results

The fracture resistance results were significantly different among groups for infrastructure material and loading protocol (P<.001). Fracture resistance values of MP were significantly higher among groups (P<.001). The FP group presented fracture resistance value significantly lower than MP, but higher than MC and FC (P<.001). Groups which presented loading protocol on cantilever region, such as MC and FC, showed fracture resistance values significantly lower among groups (P<.001), but no significant differences were observed among each other (P=.633). (Table 3 and Figure 7)

Table 3. Mean values, standard deviation, and significant differences (N).

Group	Mean±SD
FP	4206.31 ± 469.11^{b}
FC	1130.85 ± 245.89^{c}
MP	5850.28 ± 607.25^{a}
MC	1388.18 ± 297.27^{c}

Groups with same superscript letters not significantly different (Tukey P>.05)

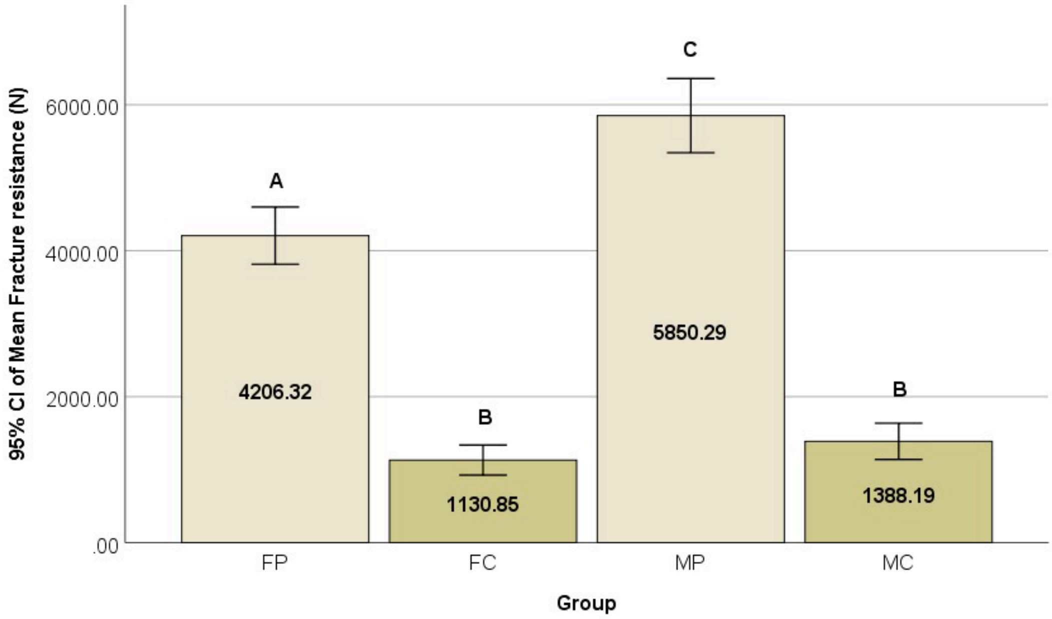

Figure 7. Bar chart showing mean and confidence interval of fracture resistance values. Crossed confidence intervals reveals no statistical differences among groups. Same capital letters means no significant differences (Tukey P>.05).

Failure observed occurred only on the veneering material and the patterns could be addressed to the bonding ability of the infrastructure material. All the specimens which used experimental fiber infrastructure (FP and FC) presented fractures in the veneering material but the pieces were still bonded to the infrastructure. (Figure 8) Contrarily, on the specimens which used metallic infrastructures, the veneering material debonded from the infrastructure. (Figure 8)

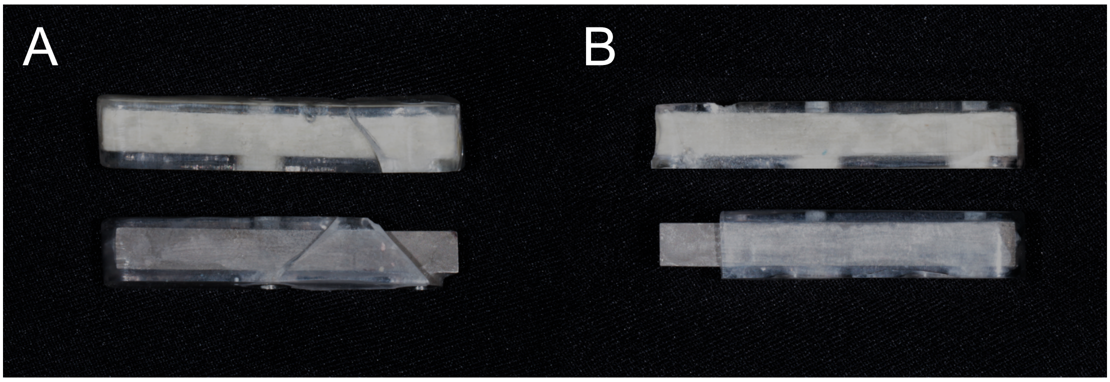

Figure 8. Representative images of the failure patterns in (A) intermediate pontic and (B) cantilever loading protocols.

4. Discussion

The fracture resistance isolated represents a high static load that usually exceeds the functional cyclic loading present in the oral environment. Thus it is important to associate this test with fatigue loading, to simulate functional aging of the dental materials and collaborate on the prediction of clinical behavior. This experimental study aimed to evaluate the fracture resistance after fatigue cycling of pre-clinical bar shaped specimens of implant-fixed partial dentures with a CAD-CAM milled glass fiber infrastructure compared to a CAD-CAM soft metal milling cobalt-chromium alloy infrastructure with two different loading protocols, on the intermediate pontic or on cantilever. The findings of this study demonstrates that based on the fracture resistance values of the specimens, the experimental fiber infrastructures were comparable to the metallic ones with cantilever load, which is a very promising result. Nevertheless, the intermediate pontic at the metallic infrastructure still presented a significantly higher fracture resistance. As a result, the null hypothesis that the loading protocol or infrastructure material would not have a significant influence on the fracture strength of the prosthesis was rejected.

Metallic infrastructure is frequently reported for dental- or implant-supported fixed partial or complete denture frameworks. (7,24–26) Different alloys have been proposed, such as titanium or Co-Cr. (6,7) These alloys present good fracture resistance and hardness, radiopacity, and relatively good biocompatibility, corrosion resistance, and low cost. (27) In the present study, metallic infrastructure performed better than glass fiber material when intermediate pontic was tested. Nonetheless, metal still present disadvantages, such as poor esthetic and low adhesion to polymeric veneering materials. Also, the material can be allergic, present disgusting taste in some situations, and some level of oxidation. (15) Those drawbacks may lead to relative contraindications, since nearly all those conditions occurs in specific situations, except for esthetic and adhesion.

Alternatively, glass fiber have been proposed as fixed prosthesis infrastructure with great esthetic and adhesion to polymers. (9) The fabrication methods vary, and can be manually or digitally constructed using CAD-CAM technology. (8,14) The mechanical reinforcement effect of FRC on infrastructures of fixed dental prosthesis to static loading have been reported. (11,12) Usually, the fracture resistance of fiber infrastructure is worse that of metallic infrastructure, and better that of unreinforced framework designs. (7,13) Still, other study revealed important results that supports the use of FRC infrastructures in implant-supported prosthesis by enhancing the fracture resistance in more than 500%, when compared to unreinforced photo-polymerized composites or thermo-polymerized resin. (28) In the present study, glass fiber presented a worse fracture resistance when compared to metallic infrastructure on the intermediate pontic loading protocol. Contrarily, on the cantilever loading protocol, the FRC presented no statistical difference from the metallic infrastructure. The dynamic observation of the fracture resistance test revealed an interesting behavior of the fiber in the cantilever region that could answer this difference, which was the ability to bend before fracture. This characteristic may be attributed to the low elastic modulus and good resilience of the fiber. (10,29,30)

Fracture is a common complication in implant-supported fixed partial or complete dentures, and the clinical approach for repairing or replacing the prosthesis depends on the location and extent of the failure. (31) The failure modes observed in the current study exhibited a uniform distribution. In general, the infrastructures remained intact, while failures were observed in the veneering material. However, the crucial role of the infrastructure material was evident as the fracture resistance values exhibited statistically significant differences between the FP and MP groups. A comparison of fracture resistance between fiber and titanium infrastructures in implant-supported fixed complete dentures with cantilever designs revealed superior performance in the metallic group, with similar failure modes. (7) In contrast, in the present study, the observed failure modes were more favorable for the groups with FRC infrastructures, where the veneering material fractured but remained bonded to the infrastructure. The Co-Cr infrastructure exhibited debonding of the fractured veneering material. This may be attributed to the superior adhesive properties of the fiber against poor bonding of the Co-Cr infrastructure. The repair of the prosthetic framework varies depending on the specific failure mode, (29) and the findings from this study suggest that FRC infrastructures may facilitate in situ repair. Also, that failure pattern may be capable of maintaining function of the prosthesis until reparation process is concluded.

The limitations of the study can be attributed to the pre-clinical bar shaped geometry of the infrastructure and the veneering material of the specimen.

Prosthesis models presents a more complex shape, which can contain connectors between pontics that can be fulcrum to possible stress concentration and fracture. However, this kind of in vitro simulation is important to understand the real behavior of dental materials isolated from possible biases present in different designs of in vitro studies or in real clinical situations. Other limitations includes the need to understand possible biomechanical behaviors after the repair of the fractured specimens. In general the failures occurred in the veneering material rather than the infrastructure, in that way the fracture resistance of the infrastructure material was not calculated. Future studies should address those limitations and clinical trials should be encouraged.

5. Conclusion

Based on this in vitro study results, it can be concluded that this fiber presents promising results especially in cantilever region, where the fracture resistance was comparable to metal. Still, on intermediate pontics, metallic infrastructure presented significantly higher fracture resistance.

The failure modes were more favorable on the fiber infrastructure specimens, in which the veneering material kept bonded to the infrastructure regardless the loading protocol.

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CONCLUSÕES GERAIS

Baseado nos resultados do primeiro artigo, pode-se concluir que o protocolo adequado de tratamento de superfície para esta fibra de vidro experimental deve incluir o condicionamento prévio com ácido fosfórico por 30 segundos, seguido da aplicação de silano por 60 segundos.

Deve-se orientar os laboratórios de prótese que irão manusear esta fibra a evitar a imersão da fibra em monômero de resina acrílica pois, apesar deste tratamento apresentar aumento na resistência de união de resina em alguns tipos de fibra de vidro, para a fibra experimental usada neste estudo houve uma piora na resistência de união até mesmo quando comparado ao grupo controle, sem nenhum tratamento prévio.

Para os grupos com valores mais altos de resistência adesiva ao microcisalhamento, os padrões de falha foram principalmente compostos de falhas coesivas na resina, o que pode indicar boa resistência estrutural da fibra de vidro experimental e valores suficientes de resistência de união.

Sobre o comportamento mecânico de resistência à fratura, esta fibra experimental mostrou-se um material muito promissor por conseguir igualar-se ao metal em região de cantilever, apesar de ter apresentado um comportamento inferior em região de pônticos intermediários.

Ainda, ao analisar os padrões de falhas do teste de resistência à fratura, todas os eventos observados nos grupos com infraestrutura de fibra de vidro foram mais favoráveis do que as observadas nos grupos com infraestrutura de metal.

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ANEXOS

ANÁLISE ESTATÍSTICA ARTIGO 1

Case Processing Summary

Group		Valid N	Valid Percent	Missing N	Missing Percent	Total N	Total Percent
Microshear (MPa)	Control	20	100,0%	0	0,0%	20	100,0%
	Silane	23	100,0%	0	0,0%	23	100,0%
	Air abrasion	18	100,0%	0	0,0%	18	100,0%
	Monomer	21	100,0%	0	0,0%	21	100,0%
	Phosphoric acid + silane	22	100,0%	0	0,0%	22	100,0%
	Air abrasion + phosporic acid + silane	24	100,0%	0	0,0%	24	100,0%

Descriptives

Group				Statistic	Std. Error
Microshear (MPa)	Control	Mean		16,608	1,0966
		95% Confidence Interval for Mean	Lower Bound	14,313	
			Upper Bound	18,903	
		5% Trimmed Mean		16,619	
		Median		17,280	
		Variance		24,051	
		Std. Deviation		4,9042	
		Minimum		7,7	
		Maximum		25,3	
		Range		17,7	
		Interquartile Range		8,2	
		Skewness		-0,394	0,512
		Kurtosis		-0,569	0,992
	Silane	Mean		24,659	1,8041
		95% Confidence Interval for Mean	Lower Bound	20,918	

		Upper Bound	28,401	
	5% Trimmed Mean		24,498	
	Median		25,730	
	Variance		74,856	
	Std. Deviation		8,6519	
	Minimum		10,1	
	Maximum		42,2	
	Range		32,1	
	Interquartile Range		13,2	
	Skewness		0,110	0,481
	Kurtosis		-0,513	0,935
Air abrasion	Mean		16,926	1,2621
	95% Confidence Interval for Mean	Lower Bound	14,263	
		Upper Bound	19,588	
	5% Trimmed Mean		16,801	
	Median		16,510	
	Variance		28,673	
	Std. Deviation		5,3548	
	Minimum		9,6	
	Maximum		26,5	
	Range		16,9	
	Interquartile Range		8,1	
	Skewness		0,483	0,536
	Kurtosis		-0,693	1,038
Monomer	Mean		11,337	0,7578
	95% Confidence Interval for Mean	Lower Bound	9,756	
		Upper Bound	12,918	
	5% Trimmed Mean		11,429	
	Median		11,520	
	Variance		12,060	
	Std. Deviation		3,4727	
	Minimum		3,8	
	Maximum		17,2	
	Range		13,3	
	Interquartile Range		5,4	
	Skewness		-0,412	0,501
	Kurtosis		-0,383	0,972
	Mean		36,597	2,4729

		95% Confidence Interval for Mean	Lower Bound	31,454	
			Upper Bound	41,740	
		5% Trimmed Mean		36,734	
		Median		35,775	
		Variance		134,540	
	Phosphoric acid + silane	Std. Deviation		11,5991	
		Minimum		12,7	
		Maximum		58,5	
		Range		45,8	
		Interquartile Range		13,5	
		Skewness		-0,367	0,491
		Kurtosis		0,009	0,953
	Air abrasion + phosporic acid + silane	Mean		38,992	2,6621
		95% Confidence Interval for Mean	Lower Bound	33,485	
			Upper Bound	44,499	
		5% Trimmed Mean		38,699	
		Median		40,260	
		Variance		170,077	
		Std. Deviation		13,0414	
		Minimum		14,9	
		Maximum		68,5	
		Range		53,6	
		Interquartile Range		13,3	
		Skewness		0,399	0,472
		Kurtosis		0,805	0,918

Tests of Normality

		Kolmogorov-Smirnov[a]			Shapiro-Wilk		
Group		Statistic	df	Sig.	Statistic	df	Sig.
Microshear (MPa)	Control	0,192	20	0,052	0,945	20	0,293
	Silane	0,109	23	,200*	0,974	23	0,782
	Air abrasion	0,131	18	,200*	0,937	18	0,261
	Monomer	0,099	21	,200*	0,977	21	0,875
	Phosphoric acid + silane	0,169	22	0,104	0,960	22	0,486

	Air abrasion + phosporic acid + silane	0,165	24	0,090	0,948	24	0,244

*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Univariate Analysis of Variance

Between-Subjects Factors

		Value Label	N
Group	0	Control	20
	1	Silane	23
	2	Air abrasion	18
	3	Monomer	21
	4	Phosphoric acid + silane	22
	5	Air abrasion + phosphoric acid + silane	24

Descriptive Statistics

Dependent Variable:

Group	Mean	Std. Deviation	N
Control	16,608	4,9042	20
Silane	24,659	8,6519	23
Air abrasion	16,926	5,3548	18
Monomer	11,337	3,4727	21
Phosphoric acid + silane	36,597	11,5991	22
Air abrasion + phosphoric acid + silane	38,992	13,0414	24
Total	24,867	13,6692	128

Levene's Test of Equality of Error Variances[a,b]

		Levene Statistic	df1	df2	Sig.
Microshear (MPa)	Based on Mean	5,074	5	122	0,000
	Based on Median	4,702	5	122	0,001

	Based on Median and with adjusted df	4,702	5	70,370	0,001
	Based on trimmed mean	5,187	5	122	0,000

Tests the null hypothesis that the error variance of the dependent variable is equal across groups.
a. Dependent variable: Microshear (MPa)
b. Design: Intercept + Grupo

Tests of Between-Subjects Effects

Dependent Variable:

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Noncent. Parameter	Observed Power[b]
Corrected Model	14159,992[a]	5	2831,998	36,104	0,000	180,522	1,000
Intercept	74212,015	1	74212,015	946,111	0,000	946,111	1,000
Grupo	14159,992	5	2831,998	36,104	0,000	180,522	1,000
Error	9569,556	122	78,439				
Total	102881,806	128					
Corrected Total	23729,548	127					

a. R Squared = ,597 (Adjusted R Squared = ,580)
b. Computed using alpha = ,05

Post Hoc Tests

Group

Multiple Comparisons

Dependent Variable:

(I) Group			Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
~~Tukey HSD~~	~~Control~~	~~Silane~~	~~-8,051*~~	~~2,7078~~	~~0,041~~	~~-15,892~~	~~-0,211~~
		~~Air abrasion~~	~~-0,318~~	~~2,8774~~	~~1,000~~	~~-8,649~~	~~8,014~~
		~~Monomer~~	~~5,271~~	~~2,7672~~	~~0,404~~	~~-2,742~~	~~13,283~~
		~~Phosphoric acid + silane~~	~~-19,989*~~	~~2,7363~~	~~0,000~~	~~-27,912~~	~~-12,066~~
		~~Air abrasion + phosphoric acid + silane~~	~~-22,384*~~	~~2,6815~~	~~0,000~~	~~-30,148~~	~~-14,620~~
	~~Silane~~	~~Control~~	~~8,051*~~	~~2,7078~~	~~0,041~~	~~0,211~~	~~15,892~~

		Air abrasion	7,734	2,7871	0,069	-0,337	15,804
		Monomer	13,322*	2,6731	0,000	5,582	21,062
		Phosphoric acid + silane	-11,938*	2,6412	0,000	-19,585	-4,290
		Air abrasion + phosphoric acid + silane	-14,333*	2,5843	0,000	-21,816	-6,850
	Air abrasion	Control	0,318	2,8774	1,000	-8,014	8,649
		Silane	-7,734	2,7871	0,069	-15,804	0,337
		Monomer	5,588	2,8448	0,369	-2,649	13,826
		Phosphoric acid + silane	-19,671*	2,8148	0,000	-27,822	-11,521
		Air abrasion + phosphoric acid + silane	-22,067*	2,7615	0,000	-30,063	-14,070
	Monomer	Control	-5,271	2,7672	0,404	-13,283	2,742
		Silane	-13,322*	2,6731	0,000	-21,062	-5,582
		Air abrasion	-5,588	2,8448	0,369	-13,826	2,649
		Phosphoric acid + silane	-25,260*	2,7020	0,000	-33,083	-17,436
		Air abrasion + phosphoric acid + silane	-27,655*	2,6464	0,000	-35,318	-19,992
	Phosphoric acid + silane	Control	19,989*	2,7363	0,000	12,066	27,912
		Silane	11,938*	2,6412	0,000	4,290	19,585
		Air abrasion	19,671*	2,8148	0,000	11,521	27,822
		Monomer	25,260*	2,7020	0,000	17,436	33,083
		Air abrasion + phosphoric acid + silane	-2,395	2,6141	0,942	-9,965	5,174
	Air abrasion + phosphoric acid + silane	Control	22,384*	2,6815	0,000	14,620	30,148
		Silane	14,333*	2,5843	0,000	6,850	21,816
		Air abrasion	22,067*	2,7615	0,000	14,070	30,063
		Monomer	27,655*	2,6464	0,000	19,992	35,318
		Phosphoric acid + silane	2,395	2,6141	0,942	-5,174	9,965
Games-Howell	Control	Silane	-8,051*	2,1112	0,006	-14,406	-1,696
		Air abrasion	-0,318	1,6720	1,000	-5,358	4,723
		Monomer	5,271*	1,3330	0,005	1,248	9,293
		Phosphoric acid + silane	-19,989*	2,7052	0,000	-28,239	-11,739
		Air abrasion + phosphoric acid + silane	-22,384*	2,8791	0,000	-31,133	-13,635
	Silane	Control	8,051*	2,1112	0,006	1,696	14,406
		Air abrasion	7,734*	2,2017	0,014	1,122	14,345
		Monomer	13,322*	1,9567	0,000	7,363	19,281
		Phosphoric acid + silane	-11,938*	3,0611	0,005	-21,111	-2,765
		Air abrasion + phosphoric acid + silane	-14,333*	3,2158	0,001	-23,954	-4,712

	Air abrasion	Control	0,318	1,6720	1,000	-4,723	5,358
		Silane	-7,734*	2,2017	0,014	-14,345	-1,122
		Monomer	5,588*	1,4722	0,008	1,093	10,083
		Phosphoric acid + silane	-19,671*	2,7764	0,000	-28,102	-11,241
		Air abrasion + phosphoric acid + silane	-22,067*	2,9461	0,000	-30,986	-13,147
	Monomer	Control	-5,271*	1,3330	0,005	-9,293	-1,248
		Silane	-13,322*	1,9567	0,000	-19,281	-7,363
		Air abrasion	-5,588*	1,4722	0,008	-10,083	-1,093
		Phosphoric acid + silane	-25,260*	2,5864	0,000	-33,233	-17,286
		Air abrasion + phosphoric acid + silane	-27,655*	2,7678	0,000	-36,143	-19,167
	Phosphoric acid + silane	Control	19,989*	2,7052	0,000	11,739	28,239
		Silane	11,938*	3,0611	0,005	2,765	21,111
		Air abrasion	19,671*	2,7764	0,000	11,241	28,102
		Monomer	25,260*	2,5864	0,000	17,286	33,233
		Air abrasion + phosphoric acid + silane	-2,395	3,6335	0,985	-13,219	8,429
	Air abrasion + phosphoric acid + silane	Control	22,384*	2,8791	0,000	13,635	31,133
		Silane	14,333*	3,2158	0,001	4,712	23,954
		Air abrasion	22,067*	2,9461	0,000	13,147	30,986
		Monomer	27,655*	2,7678	0,000	19,167	36,143
		Phosphoric acid + silane	2,395	3,6335	0,985	-8,429	13,219

Based on observed means.
The error term is Mean Square(Error) = 78,439.
*. The mean difference is significant at the ,05 level.

ANÁLISE ESTATÍSTICA ARTIGO 2

Tests of Normality

Group		Kolmogorov-Smirnov[a] Statistic	df	Sig.	Shapiro-Wilk Statistic	df	Sig.
Fracture resistance (N)	FP	0,174	8	,200*	0,925	8	0,469
	FC	0,216	8	,200*	0,916	8	0,399
	MP	0,208	8	,200*	0,908	8	0,342
	MC	0,229	8	,200*	0,902	8	0,303

*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Levene's Test of Equality of Error Variances[a,b]

		Levene Statistic	df1	df2	Sig.
Fracture resistance (N)	Based on Mean	1,471	3	28	0,244
	Based on Median	1,085	3	28	0,372
	Based on Median and with adjusted df	1,085	3	16,719	0,383
	Based on trimmed mean	1,477	3	28	0,242

Tests the null hypothesis that the error variance of the dependent variable is equal across groups.

a. Dependent variable: Fracture resistance (N)

b. Design: Intercept + Framework + Design + Framework * Design

Tests of Between-Subjects Effects

Dependent Variable:

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Noncent. Parameter	Observed Power[b]
Corrected Model	124705287,433[a]	3	41568429,144	225,408	0,000	676,223	1,000
Intercept	316293568,576	1	316293568,576	1715,123	0,000	1715,123	1,000
Framework	7229940,419	1	7229940,419	39,205	0,000	39,205	1,000
Design	113629847,634	1	113629847,634	616,165	0,000	616,165	1,000
Framework * Design	3845499,380	1	3845499,380	20,852	0,000	20,852	0,993
Error	5163606,430	28	184414,515				
Total	446162462,439	32					
Corrected Total	129868893,863	31					

a. R Squared = ,960 (Adjusted R Squared = ,956)

b. Computed using alpha = ,05

Descriptive Statistics

Dependent Variable:

Group	Mean	Std. Deviation	N

FP	4206,3175	469,10847	8
FC	1130,8500	245,89055	8
MP	5850,2875	607,25561	8
MC	1388,1875	297,27725	8
Total	3143,9106	2046,78264	32

Between-Subjects Factors

		Value Label	N
Group	1	FP	8
	2	FC	8
	3	MP	8
	4	MC	8

Multiple Comparisons

Dependent Variable:

Tukey HSD

(I) Group		Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
FP	FC	3075,4675*	214,71756	0,000	2489,2217	3661,7133
	MP	-1643,9700*	214,71756	0,000	-2230,2158	-1057,7242
	MC	2818,1300*	214,71756	0,000	2231,8842	3404,3758
FC	FP	-3075,4675*	214,71756	0,000	-3661,7133	-2489,2217
	MP	-4719,4375*	214,71756	0,000	-5305,6833	-4133,1917
	MC	-257,3375	214,71756	0,633	-843,5833	328,9083
MP	FP	1643,9700*	214,71756	0,000	1057,7242	2230,2158
	FC	4719,4375*	214,71756	0,000	4133,1917	5305,6833
	MC	4462,1000*	214,71756	0,000	3875,8542	5048,3458
MC	FP	-2818,1300*	214,71756	0,000	-3404,3758	-2231,8842
	FC	257,3375	214,71756	0,633	-328,9083	843,5833
	MP	-4462,1000*	214,71756	0,000	-5048,3458	-3875,8542

Based on observed means.
The error term is Mean Square(Error) = 184414,515.

*. The mean difference is significant at the 0,05 level.

Homogeneous Subsets

Fracture resistance (N)

Tukey HSD[a,b]

		Subset		
Group	N	1	2	3
FC	8	1130,8500		
MC	8	1388,1875		
FP	8		4206,3175	
MP	8			5850,2875
Sig.		0,633	1,000	1,000

Means for groups in homogeneous subsets are displayed.
Based on observed means.
The error term is Mean Square(Error) = 184414,515.
a. Uses Harmonic Mean Sample Size = 8,000.

b. Alpha = 0,05.