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Influence of different surface treatments on the shear strength between acrylic resin and two materials: Polyether Ether Ketone (PEEK) / ZANTEX^R

A influência de diferentes tratamentos de superfície na resistência ao cisalhamento entre resina acrílica e dois materiais: Poli éter éter cetona (PEEK) / ZANTEX^R

La influencia de diferentes tratamientos superficiales en la resistencia al corte entre la resina acrílica y dos materiales: poli éter éter cetona (PEEK) / ZANTEX^R

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Abstract
The present study assesses the shear strength of Polyether Ether Ketone (PEEK) and Zantex to thermopolymerizable acrylic resin (TAR) with different surface treatments. To this end, 100 test specimens were produced and divided into two groups: G1(PEEK + TAR) and G2 (Zantex + TAR). Each group was subdivided into the subgroups A, B, C, D and E (n=10) according to surface pre-treatment: A: TAR + PEEK/Zantex without surface treatment (control); B: TAR + PEEK/Zantex treated with Palabond; C: TAR + PEEK/Zantex blasted with aluminum oxide (Al_2O_3) of 125 µm; D: TAR + PEEK /Zantex treated with Al_2O_3 of 125 µm + Palabond; and E: TAR + PEEK/Zantex prepared with groove-shaped retentions. The structures were tested in a Universal testing machine EMIC DL2000. Two-way variance analysis showed significant difference between materials and surface treatments ($p < 0.001$). Multiple comparisons were done with Tukey's test and failure modes were analyzed with the G test. Statistical analysis showed

that Zantex outperformed PEEK for most treatments and showed similar performance in the subgroups treated with Al_2O_3. Within the PEEK group, the use of grooves and Palabond combined with blasting showed the best results; in the Zantex group, the best results were obtained with Palabond and Palabond combined with blasting. Regarding failure modes, PEEK showed only adhesive failures and Zantex's failures were dependent on treatment. Therefore, the surface treatment method applied to the polymers studied here contribute to the material's adhesion to acrylic resin.
Keywords: Shear strength; Dental prosthesis; Polymer; Health teaching.

Resumo
Este estudo avaliou a resistência ao cisalhamento do Poli éter éter cetona (PEEK) e do Zantex à resina acrílica termopolimerizável de recobrimento (RAT) com o objetivo de avaliar a influência de diferentes tratamentos de superfície nestes materiais. Para isso, utilizou-se 100 corpos de prova divididos em 02 grupos: G1(PEEK + RAT) e G2 (Zantex + RAT). Cada grupo foi dividido nos subgrupos A, B, C, D e E com n=10, distintos pelo tratamento de superfície dado ao PEEK e ao Zantex previamente à prensagem da RAT. O subgrupo A foi composto por (RAT) + PEEK/Zantex sem tratamento de superfície (controle); B: RAT + PEEK/Zantex tratados com Palabond; C: RAT + PEEK/Zantex jateados com óxido de alumínio (Al_2O_3) a 125 µm; D: RAT + PEEK /Zantex Al_2O_3 a 125 µm + Palabond; e E: RAT + PEEK/Zantex preparados com retenções em forma de canaletas. As estruturas foram testadas na máquina de ensaios Universais EMIC DL2000. A análise de variância a dois critérios demonstrou significância entre os materiais e os tratamentos de superfície aplicados ($p < 0,001$). Para as comparações múltiplas empregou-se o teste de Tukey e para os modos de falha o teste G. A análise estatística mostrou que o material Zantex superou o PEEK para a maioria dos tratamentos, assemelhando-se somente para os preparos com Al_2O_3 especificamente. Na avaliação interna dos grupos, o emprego de canaletas e jateamento + Palabond representaram os maiores valores para o material PEEK; e para o Zantex, Palabond e jateamento + Palabond. Quanto aos modos de falha, para o PEEK 100% foram do tipo adesiva, e para o Zantex, houve variação dependente do tratamento. Portanto a aplicação de diferentes métodos de tramentos de superfície sobre os respectivos polímeros abordados neste estudo,contribui para maior adesão desses materiais à resina acrílica de recobrimento.
Palavras-chave: Resistência ao cisalhamento; Prótese dentária; Polímero; Ensino em saúde.

Resumen
Este estudio evaluó la resistencia al corte de la poliéter cetona (PEEK) y Zantex a la resina de recubrimiento termopolimerizable acrílica (RAT) para evaluar la influencia de diferentes tratamientos superficiales en estos materiales. Para ello se utilizaron 100 especímenes, divididos en 02 grupos: G1 (PEEK+RAT) y G2 (Zantex+RAT). Cada grupo se dividió en subgrupos A, B, C, D y E con n=10, que se distinguen por el tratamiento superficial dado a PEEK y Zantex antes de prensar el RAT. El subgrupo A consistió en (RAT) + PEEK/Zantex sin tratamiento superficial (control); B: RAT + PEEK/Zantex tratado con Palabond; C: RAT + PEEK/Zantex arenado con óxido de aluminio (Al2O3) a 125 µm; D: RAT + PEEK/Zantex Al2O3 a 125 µm + Palabond; y E: RAT + PEEK/Zantex preparado con retenciones en forma de canal. Las estructuras se ensayaron en la máquina de ensayo universal EMIC DL2000. El análisis de varianza de dos vías mostró significación entre los materiales y los tratamientos superficiales aplicados ($p < 0,001$). Para las comparaciones múltiples se utilizó la prueba de Tukey y para los modos de falla la prueba G. El análisis estadístico mostró que el material Zantex superó al PEEK para la mayoría de los tratamientos, siendo similar solo para las preparaciones con Al2O3 específicamente. En la evaluación interna de los grupos, el uso de canales y arenado + Palabond representó los valores más altos para el material PEEK; y para Zantex, Palabond y arenado + Palabond. En cuanto a los modos de falla, para 100% PEEK fueron del tipo adhesivo, y para Zantex hubo una variación dependiente del tratamiento. Por lo tanto, la aplicación de diferentes métodos de tratamientos superficiales sobre los respectivos polímeros abordados en este estudio, contribuye a una mayor adherencia de estos materiales al recubrimiento de resina acrílica.
Palabras clave: Resistencia al corte; Prótesis dental; Polímero; Enseñanza en salud.

1. Introduction

Evolution of materials with potential for the use in protocol-type prosthesis infrastructures has been the focus of current studies. Although employed in prosthesis over implants structures, such as bars and prosthetic components, for many years, the use of metal alloys presents some constraints. Aesthetical issues and adverse reactions are the main constraints in the use of metal in oral rehabilitations (Campbell et al., 2017). In addition to that, the high rigidity and elasticity module largely different from the bone hinder an adequate load transmission to the support structures (Manolea et al., 2017). Hence, lighter materials such as polymers have become the target of investigations.

Among new materials currently commercialized is the Polyether ether ketone (PEEK), widely used in orthopedic devices and prostheses (Manolea et al., 2017). This polymer was first used in Medicine with the Brantigan cage – an orthopedic device used for spine rehabilitations. Its success motivated the use of PEEK in other healthcare areas. Due to its favorable physical properties, such as good mechanical strength and elasticity module similar to the bone's, PEEK became the focus of Dentistry as well (Manolea et al., 2017). However, despite good results, researchers point to the need of further studies (Najeeb et al., 2016; Carvalho et al., 2017; Jaros et al., 2018) to better understand, for instance, its adherence to different structures (Rocha et al., 2016; Botel et al., 2018).

Another material that has recently caught the attention of scientific research is Zantex (Biofunctional Materials-USA). Composed by a polymer matrix (epoxy substrate) reinforced with a dense tridimensional network of glass fibers, it presents small elasticity module, flexibility similar to the bone's and biocompatibility. The epoxy resin matrix presents favorable features such as high tensile strength, dimensional stability, small elasticity module, that are enhanced with the insertion of glass fibers (Bonon et al.,2016). When used as infrastructure in fixed partial prosthesis, this composite showed reliability in terms of fracture strength in comparison with metal and Zirconia (Bergamo et al.,2019).

This study assesses the applicability of PEEK and Zantex as alternatives to metal alloys in infrastructures of implant-supported prostheses through shear mechanical test with a thermopolymerizable acrylic resin under different surface treatment conditions.

2. Methods

This study was approved by the Research Ethics Committee (#2020 0480) of the Dentistry School of São Leopoldo Mandic University.

Using a CAD-CAM system in a central milling unit (Bit milling machine, Isolaplast Isolantes e Plásticos. Ltda, São Paulo, SP, Brazil), 100 blocks measuring 10x10x10mm were produced - 50 PEEK blocks (Isolaplast Isolantes e Plásticos. Ltda, São Paulo, SP, Brazil) and 50 Zantex blocks (Biofunctional Materials, imported from the USA by Intra lock Tissue Tech, São Paulo, SP, Brazil).

All PEEK and Zantex blocks were rinsed in running water, inserted in 15 mm ½" PVC pipes (Amanco Brasil SA, São Paulo, SP, Brazil), standardized and cleaned. The blocks were included in polyurethane resin CST-47 (Redelease Produtos para Indústrias. Ltda, São Paulo, SP, Brazil), leaving the upper PEEK or Zantex surface exposed (Figure 1) by using an adhesive film to protect the surfaces during inclusion.

Figure 1 – Test specimens.

Source: own authorship.

Following milling, all samples were polished to homogenize and standardize the surfaces and remove any residues adhered to the surfaces after inclusion. This procedure was conducted using a polishing machine (Arotec S/A. Indústria e Comércio, Cotia, SP, Brazil) with sandpaper (silicon carbide) with 600, 800 and 1200 grit for 30 seconds at 300 rpm.

The blocks were divided into 2 groups: Group 1 – PEEK, Group 2 – Zantex. Each group was subdivided into 5 subgroups (n=10) (Pulici et al., 2017) differing by surface treatment:

Group1/Group2: PEEK/Zantex + Thermopolymerized acrylic resin with classical coating (Clássico Artigos Odontológicos. Ltda, São Paulo, SP, Brazil).

-G1A/ G2A = PEEK/ Zantex without surface treatment

-G1B/ G2B = PEEK/ Zantex treated with Palabond

-G1C/ G2C = PEEK/ Zantex blasted with 125 µm aluminum oxide

-G1D/ G2D = PEEK/ Zantex blasted with 125 µm aluminum oxide + Palabond (Kulzer South América. Ltda, São Paulo, SP, Brazil)

-G1E/ G2E = PEEK/ Zantex prepared with surface grooves

The thickness of the acrylic resin sample coating was determined by juxtaposed wax strips cut with punch scalpel to have 7 mm of thickness and the shape of a cube. The samples were included in a muffle and the wax strips were laid on the entire PEEK/Zantex exposed surface. The Zetalabor silicone (Zhermack SpA, Badia Polesine, Roma, Italy) was placed and the muffle was taken to cooking using the lost-wax casting.

Following dewaxing, PEEK and Zantex's surfaces were cleaned with isopropyl alcohol and submitted to treatment according to the proposed method:

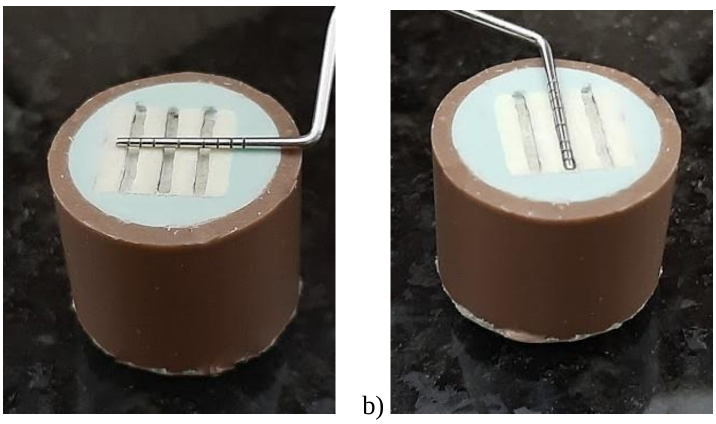
-Blasting with aluminum oxide at 125µm, 40psi/10s (Defama. Bettinelli e Betineli. Ltda, Porto Alegre, RS, Brazil) of subgroups C and D of groups G1 and G2. Application was done at a distance of 10mm, measured from the tip of the blaster (Bio-Art. Equipamentos Odontológicos. Ltda, São Carlos, SP, Brazil) to the surface of the test specimen. The test specimen was positioned perpendicularly to the direction of the blast (Pulici et al., 2017).

- Palabond application (according to the manufacturer's recommendations) on the structures of subgroups B of G1 and G2.

- Palabond application (according to the manufacturer's recommendations) on the structures of subgroups D of G1 and G2 following aluminum oxide blasting.

- Grooves designed on the surface in contact with the acrylic block using a diamond tip FG 3097 (KG Sorensen, Cotia, SP, Brazil) with low rotation and active tip of 1.0mm of diameter in subgroups E of G1 and G2, perpendicularly to the direction of the force vector exerted by the test rod. Each test specimen had three evenly spaced parallel grooves measuring 1.0 mm of diameter (Figure 2).

Figure 2 – Grooves.



a) b)

Source: Own authorship.

The thermopolymerizable resin was applied according to the manufacturer's recommendations and pressed and polymerized by the Australian Cycle.

The test specimens were obtained by pressing the thermopolymerizable acrylic resin over the PEEK and Zantex structures in a muffle. Finally, they were immersed in aqueous solution at 37°C for 24h to simulate the oral conditions (Rocha et al.,2016).

All test specimens were subjected to the shear stress test in a universal testing machine EMIC DL 2000 (EMIC, São Paulo, SP, Brazil). The tests were performed with a 50 kgf load at 0.5mm/min, using a chizel rod provided with the machine (Pulici et al., 2017). The load was applied perpendicularly to the PEEK or Zantex surface until the displacement of the fragments (Figure 3).

Figure 3 – Application of the force.

Source: Own authorship.

After the test, the failure mode was analyzed using a stereoscopic magnifying glass (Carl Zeiss, Jena, Germany - 30x magnification). Failure modes were classified as: cohesive of the acrylic resin, cohesive of Zantex/PEEK, adhesive or mixed (Pulici et al., 2017). One sample with cohesive and mixed failures was randomly chosen within group G2E to be analyzed through stereoscopy using an Olympus model SZ61 (Olympus Optical do Brasil, São Paulo, SP, Brazil).

Bond strength data failed both normality and homocedasticity tests. To ensure adhesion to the normal distribution, the square root function transformation was applied to the data. Then, the effect of material and surface treatment was compared using two-way variance analysis. For multiple comparisons, Tukey’s test was applied. Failure modes were analyzed using the G test. All statistical calculations were run on SPSS 23 (SPSS INC., Chicago, IL, USA) and BioEstat 5.0 (Fundação Mamirauá, Belém, PA, Brazil), with 5% of significance.

3. Results

The two-way variance analysis showed significant interaction between material and surface treatment ($p < 0.001$).

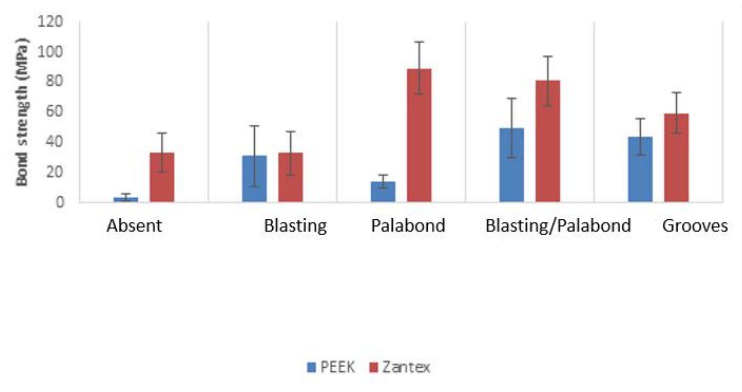
Tukey’s test showed that Zantex with grooves or Palabond®, with or without blasting, presented greater bond strength to the thermopolymerizable acrylic resin in comparison with PEEK. The same pattern was observed for the groups without surface treatment. Blasting was the only treatment resulting in a lack of significant difference between PEEK and Zantex (Table 1 and Graph 1).

Table 1 – Averages and standard deviations of bond strength (MPa) between thermopolymerizable acrylic resin and PEEK or Zantex submitted to different surface treatments.

Type of retention	PEEK	Zantex
Absent	3.58 (2.35) Bd	33.04 (12.81) Ac
Blasting	30.96 (19.92) Ab	32.79 (14.38) Ac
Palabond	14.30 (4.18) Bc	89.40 (17.32) Aa
Blasting/Palabond	49.63 (19.41) Ba	80.86 (15.96) Aa
Grooves	43.78 (12.44) Ba	59.41 (13.33) Ab

Caption: Averages followed by different capital letters indicate significant difference between materials, considering each treatment separately (comparison within the line). Averages followed by different lower-case letters indicate significant difference between treatments, considering each material separately (comparisons within the column). Source: own authorship.

Graph 1 – Bar chart of the bond strength between thermopolymerizable acrylic resin and PEEK and Zantex submitted to different surface treatments.



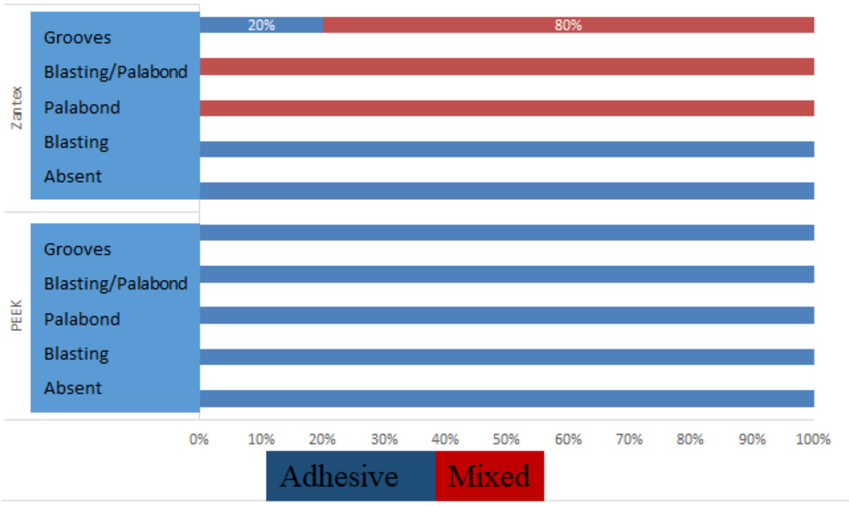
Source: Own authorship.

Considering only PEEK, the greatest bond strength was obtained with grooves and blasting with Palabond® (no significant difference between these two treatment). The subgroup submitted to blasting showed greater bond strength than the subgroup treated with Palabond®, followed by the subgroup with no surface treatment (Table 1 and Graph 1).

For Zantex, Palabond®, with or without blasting, showed greater bond strength than the presence of grooves. The bond strength did not differ between subgroups with blasting only and no surface treatment (Table 1 and Graph 1).

While for PEEK 100% of failures were adhesive, regardless of surface treatment, for Zantex, failure mode was dependent on the surface treatment ($p < 0.001$). In the subgroup with grooves, only 20% of failures were adhesive, with the prevalence of mixed failures (80%). In the subgroups treated with Palabond®, with and without blasting, all failures were mixed (Graph 2).

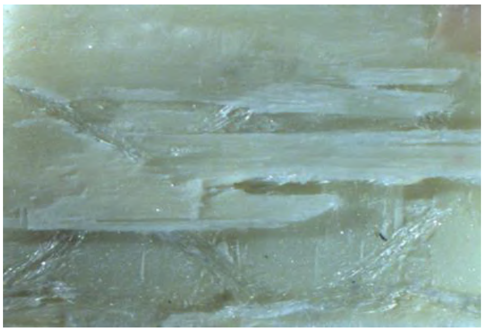
Graph 2 – Stacked bar chart with failure mode observed between thermopolymerizable acrylic resin and PEEK or Zantex, under different surface treatments.



Source: own authorship.

Only Zantex surface was analyzed through stereoscopy since none of the groups showed cohesive failures and mixed failures represented only this material. In the images, it is possible to observe the detachment of the glass fibers from the epoxy resin matrix (Figure 4).

Figure 4 – Stereoscopy of Zantex surface (group G2E).



Source: own authorship.

4. Discussion

The effect of surface treatments on different polymer-based materials for rehabilitation is the target of great interest in Dentistry. Particularly, in the case of PEEK, studies target osteogenic activity (Liao et al.,2020, Torstrick et al.,2020), and the improvement of PEEK adhesion to human dentin, compound resin and acrylic coating resin (Dede et al.,2021, Escobar et al.,2021). Regarding Zantex, the manufacturer's reports mention versatility in terms of adhesion to polymethacrylate, Zirconia, lithium disilicate and photopolymerizable resin and is presented as an efficient material for the attachment of different aesthetic structures such as ceramics and compound resins. Hence, in face of a lack of studies on Zantex, it is important to assess its shear strength to different adhesion artifacts.

Studies point to the need of surface pre-treatment and to the use of bonding agents to improve PEEK's adhesive features, given the lack of satisfactory adhesive results otherwise (Sproesser et al., 2014; Ramos et al., 2018). This has been corroborated by our results that show PEEK's low shear strength in the absence of treatment. On the other hand, Zantex, a novelty in the market, showed excellent results even in the absence of surface treatment, showing its potential for application in prosthetics. This increase in shear strength might be due to the epoxy resin present in the Zantex's matrix. The epoxy resin matrix was mentioned as a contributing factor for the interlaminar adhesion between the glass fiber-based polymer and the epoxy resin (Gonçalves et al., 2009). Zantex's roughness, larger than PEEK's, has also been pointed as a contributing factor (Ramos et al., 2018; Kürkçüoğlu et al, 2021).

Here, 125 µm aluminum oxide blasting was chosen in an attempt to reach higher adhesion values than those found in studies that used 110 µm particles (Stawarczyk et al., 2014). In the present study, blasting with aluminum oxide alone did not affect Zantex's adhesion; however, when associated with the bonding agent Palabond, adhesion increased significantly. Contrarily to that, PEEK's adhesion to the acrylic coating resin increased significantly in the groups with and without blasting.

The use of bonding agents is reported as a contributing factor for the adhesion (Dede et al.,2021; Escobar et al.,2021). Their indications depend on the type of material and adhesion required, varying according to substrate and coating structure. The variety of cements and adhesives available in the market demand expertise, while the correct choice is key for the rehabilitation success (Stawarczyk et al., 2018). One study reports an increase in the adhesion between PEEK bases and coating structures with the use of Signum PEEK Bond as bonding agent (Stawarczyk et al., 2014). Another study reports high bond strength values of a glass fiber-reinforced composite, similar to Zantex's structure, to coating resin with the use of a self-etching resinous bond agent (Bergamo et al., 2019).

The lack of specific adhesives for the bonding of PEEK, and consequently Zantex, to thermopolymerizable acrylic resin is a challenge to prosthetics. One study reported unsuccessful bonding of PEEK structures when using a universal primer (Monobond) without previous surface treatment (Ramos et al., 2018). This agrees with the findings of the present study that found unsatisfactory adhesion results of PEEK treated with the silane Palabond alone, whereas previous treatment with aluminum oxide blasting resulted in the largest bond strength values in the PEEK group. This reinforces the idea that PEEK is effective with a variety of coating materials after some surface modification. On the other hand, Palabond produced significant adhesion results, with and without previous treatment, when applied with Zantex.

The creation of grooves produced significant adhesion results for both materials. However, the difference between materials was not larger than that found with Palabond alone and in combination with blasting. Based on the fact that additional mechanical retentions are key to the adhesion between metal and coating resin, this study aimed to assess its effect on the two materials. One aspect worth considering during the production of grooves in polymer structures is the possibility of a weakening of the prosthesis infrastructure due to the disruption of fibers; therefore, this process should be carried out with caution and observing the disposition of the fibers.

Regarding failure modes, 100% of failures were adhesive, in agreement with Rosentritt et al. (2015). Adhesive and mixed failures were observed in PEEK bonded with human dentin (Rocha et al., 2016). In the Zantex groups, only adhesive and mixed failures were observed. In the mixed failures, a detachment of the glass fibers was observed, with the disruption of its tridimensional structure (Figure 6). In the clinical environment, this detachment in protocol-type prosthesis can lead to the replacement of the entire prosthesis infrastructure. Cohesive failures were observed with polymers similar to Zantex bonded to compound resin (Bergamo et al., 2019).

The present study suggests the need for further studies, such as, for instance, the comparison of different methods of application of thermopolymerizable acrylic resins, the use of other chemical bonding agents combined with 125 µm aluminum oxide blasting and other mechanical retention types, particularly added to Palabond.

Conclusion

PEEK showed better adhesion when pre-treated with blasting combined with Palabond® and with the production of grooves. All treatments were more effective than the groups without treatment.

Zantex showed better adhesion with Palabond®, combined or not with blasting. It also showed satisfactory adhesion results in the absence of surface treatment.

Zantex is shown as a promising material for the application in prostheses over implants since it showed better results in comparison with PEEK in all conditions, except when treated exclusively with aluminum oxide blasting, condition in which both materials showed the same behavior.

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