

## Shear bond strength of resinous cements to Zirconia Katana™ UTML

Resistência de união ao cisalhamento de cimentos resinosos a Zircônia Katana™ UTML

Resistencia de la unión al cizallamiento de los cementos de resina a Zirconia Katana™ UTML

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### **Abstract**

The aim of this study is to assess the bond strength of three resinous cements to Zirconia Katana™ UTML (Kuraray Noritake Dental Inc, Miyoshi, Aichi, Japan). Thirty-six blocks of Zirconia were milled and included in polyvinyl chloride (PVC) pipes with self-polymerizable acrylic resin. Each sample was polished in a polishing machine, blasted with aluminum particles, cleaned with ultrasound, and dried with compressed air. The cementation was done according to the protocol recommended by the manufacturer. The samples were divided in 3 groups (n=12) according to the resinous cement used: Group R - RelyX™ U200 Automix (3M ESPE, Sumaré, São Paulo, Brazil); Group M - Multilink® N (Ivoclar Vivadent, Schaan, Liechtenstein); and Group P - Panavia™ V5 Paste (Kuraray Noritake Dental Inc, Cotia, São Paulo, Brazil). The shear stress test was performed in EMIC DL2000 (EMIC, São Paulo, SP, Brazil), and failure type was analyzed. Average bond strengths were 5.4 kgf, 5.8 kgf, and 6.68 kgf for groups R1, M1 and P1, respectively. Variance analysis showed no significant difference between groups. None of the groups showed cohesive failure; there was no significant difference in the frequency of mixed and adhesive failure between groups. All three cements showed similar performance in the tests.

**Keywords:** Cementing; Cementing agents; Shear strength.

### **Resumo**

O objetivo deste estudo foi analisar a resistência de união entre três cimentos resinosos à Katana™ Zircônia UTML (Kuraray Noritake Dental Inc, Miyoshi, Aichi, Japão). Para isso, 36 blocos de zircônia foram fresados e incluídos em tubos de policloreto de vinila (PVC) com resina acrílica autopolimerizável. Cada amostra foi polida na politriz, jateada com partículas de alumina e limpas em ultrassom. Os blocos foram secos com jato de ar antes de receberem seus

respectivos cimentos, respeitando o protocolo de cimentação recomendado pelos fabricantes. As amostras foram divididas em três grupos (n=12) de acordo com o cimento resinoso utilizado: Grupo R - RelyX™ U200 Automix (3M ESPE, Sumaré, São Paulo, Brasil); Grupo M - Multilink® N (Ivoclar Vivadent, Schaan, Liechtenstein); e Grupo P - Panavia™ V5 Paste (Kuraray Noritake Dental Inc, Cotia, São Paulo, Brasil). O ensaio mecânico de cisalhamento foi realizado na EMIC DL2000 (EMIC, São Paulo, SP, Brasil) e também foi analisado o modo de falha. Os resultados mostraram média do valor de resistência de união de 5,4kgf para o grupo R1, 5,8kgf para o grupo M1 e 6,68 kgf para o grupo P1. A análise de variância demonstrou não haver diferença estatisticamente significativa entre os grupos. Não houve falha coesiva em nenhum grupo; e não houve diferença nos modos de falha adesiva e mista entre os grupos. Concluiu-se que os três cimentos apresentaram o mesmo desempenho frente ao teste realizado.

**Palavras-chave:** Cimentação; Agentes cimentantes; Resistência ao cisalhamento.

### Resumen

El objetivo de este estudio fue analizar la fuerza de unión entre tres cementos de resina a Katana™ Zirconia UTML (Kuraray Noritake Dental Inc, Miyoshi, Aichi, Japón). Para ello se fresaron 36 bloques de zirconia y se incluyeron en tubos de policloruro de vinilo (PVC) con resina acrílica autopolimerizable. Cada muestra fue pulida en la pulidora, arenada con partículas de alúmina y limpiada ultrasónicamente. Los bloques fueron secados al aire antes de recibir sus respectivos cementos, respetando el protocolo de cementación recomendado por los fabricantes. Las muestras se dividieron en tres grupos (n=12) según el cemento resinoso utilizado: Grupo R - RelyX™ U200 Automix (3M ESPE, Sumaré, São Paulo, Brasil); Grupo M - Multilink® N (Ivoclar Vivadent, Schaan, Liechtenstein); y Grupo P - Pasta Panavia™ V5 (Kuraray Noritake Dental Inc, Cotia, São Paulo, Brasil). El ensayo de corte mecánico se realizó en el EMIC DL2000 (EMIC, São Paulo, SP, Brasil) y también se analizó el modo de falla. Los resultados mostraron un valor promedio de fuerza de unión de 5,4 kgf para el grupo R1, 5,8 kgf para el grupo M1 y 6,68 kgf para el grupo P1. El análisis de varianza no mostró diferencias estadísticamente significativas entre los grupos. No hubo falla cohesiva en ningún grupo; y no hubo diferencia en los modos de falla adhesivos y mixtos entre los grupos. Se concluyó que los tres cementos presentaron el mismo comportamiento frente al ensayo realizado.

**Palavras clave:** Cementación; Agentes cementantes; Resistencia a la cizalladura.

## 1. Introduction

In recent years, dentistry has witnessed a growing demand for aesthetic materials. One of the reasons for such growth might be the implementation of CAD/CAM technologies and automated systems for the manufacturing of prosthetic parts. In addition to that, patients have become more demanding in their aesthetic needs. Hence, metal-free ceramic crowns have become an interesting alternative for metallo ceramics due to their excellent optical properties (Carvalho et al., 2016; Lee et al., 2017; Colombo et al., 2020).

Zirconia is widely used as a prosthetic material; yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) is considered the ceramic material with the best mechanical properties (Yue et al., 2018; Araújo-Júnior et al., 2020; Kang et al., 2020).

Considering the improvement of ceramic materials, Kuraray Noritake Dental Inc. released Katana™ Ultra Translucent Multilayered Zirconia (UTML) and Super Translucent Multilayered Zirconia (STML) in 2015. One of their greatest advantages is reaching the highest aesthetic level in fewer steps. This is possible because the milling disks are available in several layers of color, using the multilayered technology, creating a smooth gradual color transition (Kolakarnprasert et al., 2019).

In clinical practice, cementing is key to a better result with such materials. Literature shows that cementing of Y-TZP is still a challenge since the material does not contain silica, thus presenting low adhesive potential. Hence, conventional cementing procedures used with porcelain are not recommended because the bonding of Y-TZP to resinous cement is difficult, requiring a controversial protocol, often subject to debate (Negreiros et al., 2017; Lee et al., 2017; Magalhães et al., 2020).

There is no consensus about the ideal cementing protocol concerning Y-TZP (non-vitreous ceramic) in the literature; this study aims to investigate the adhesion of various resinous cements to yttria-stabilized tetragonal zirconia polycrystal of the brand Katana™ Zirconia UTML (Kuraray Noritake Dental Inc, Miyoshi, Aichi, Japan) and to determine which one presents the highest bond strength in the shear test.

## 2. Methods

The present research is an experimental study with a quantitative research methodology. Thirty-six blocks of Katana™ Zirconia UTML (Kuraray Noritake Dental Inc, Miyoshi, Aichi, Japan) were milled using CAD/CAM (Roland DWX-50, Irvine, CA, USA) to have a rectangular shape measuring de 5x10x10mm (Pulici et al., 2017).

Sample calculation was run on G\*Power 3.1.9.4 with one-way variance analysis. N was defined as 12 (n=12). The Zirconia blocks were milled, included in self-polymerizable acrylic resin Jet (Artigos Odontológicos Clássico Ltda., São Paulo, Brazil) in ½ inches x 15 mm polyvynyl chloride (PVC) pipes (Tigre, Castro, Paraná, Brazil).

The surface was treated following the resin milling. The samples were polished to rectification and to remove any material adhered to the surface during the inclusion process. This procedure was done with a polishing machine (Arotec, Cotia, São Paulo, Brazil) with 320, 400, 600, 800 and 1200 grit sandpaper for 30 seconds at 300 rpm (Santos et al., 2018). The samples were blasted with 50µm alumina particles for 20 seconds at a distance of 10 mm at 0.2 MPa (below 2 bar), cleaned with ultrasound for 2 minutes and dried with compressed air, according to Katana™ Zirconia UTML manufacturer recommendations.

A bipartite metallic matrix was used to standardize the area where the resinous cements are to be applied (Figure 1). On one end, the matrix has an orifice to fit the PVC pipe and and, on the opposite end, an orifice measuring 5 mm in diameter and 3 mm in thickness where the cements will be placed (Santos et al., 2018). In figure 1, the split matrix can be seen uncoupled and then coupled onto a PVC tube, before the application of cement.

**Figure 1** – Bipartite metallic matrix to accomodate the milled Zirconia included in the PVC pipe and used for cementation.



Source: Authors.

The following brands of cement were used according to the recommendations of their respective manufacturers for Zirconia: RelyX™ U200 Automix (3M ESPE, Sumaré, São Paulo, Brazil), Multilink® N (Ivoclar Vivadent, Schaan, Liechtenstein) and Panavia™ V5 Paste (Kuraray Noritake Dental Inc, Cotia, São Paulo, Brazil). The experimental groups were defined as:

Group R: Katana™ Zirconia UTML + RelyX™ U200 Automix (n=12)

Group M: Katana™ Zirconia UTML + Multilink® N (n=12)

Group P: Katana™ Zirconia UTML + Panavia™ V5 Paste (n=12)

The test specimens were finished after the cement insertion and polymerization, and removal of the bipartite metallic matrix (Figure 2). Figure 2 shows the decoupled matrix after insertion and light-curing of the cement.

**Figure 2** – Removal of the bipartite metallic matrix after cementation.



Source: Authors.

The test specimens were submitted to a mechanical shear test on EMIC DL2000 (EMIC, São Paulo, SP, Brazil) (Figure 3). Shear strength was determined using the machine's test screw, which directed the force to the interface cement/ceramic, at 0.5 mm/min with load cell of 50 kgf until fracture (Pulici et al., 2017).

Figure 3 shows the moment of performing the mechanical shear test.

**Figure 3** – Shear test.



Source: Authors.

By the end of the mechanical test, all samples were analyzed under a stereoscopic magnifying glass using 40x magnification. Failure type was categorized as cohesive of Zirconia, cohesive of cement, adhesive, or mixed (Pulici et al., 2017).

### Statistical analysis

Assuming normal distribution and homoscedasticity, the bond strengths of resinous cements to Zirconia Katana™ UTML were compared with one-way variance analysis. The fracture types were analyzed with Fisher's exact test with Freeman-Halton extension. Statistical calculations were run on SPSS 23 (SPSS Inc., Chicago, IL, USA) and on vassarstats.net, with 5% of significance.

### 3. Results

One-way variance analysis showed no significant difference between the cement brands RelyX™ U200, Multilink® N and Panavia™ V5 Paste regarding bond strength to Zirconia Katana™ UTML ( $p = 0.523$ ; Table 1 and Graph 1).

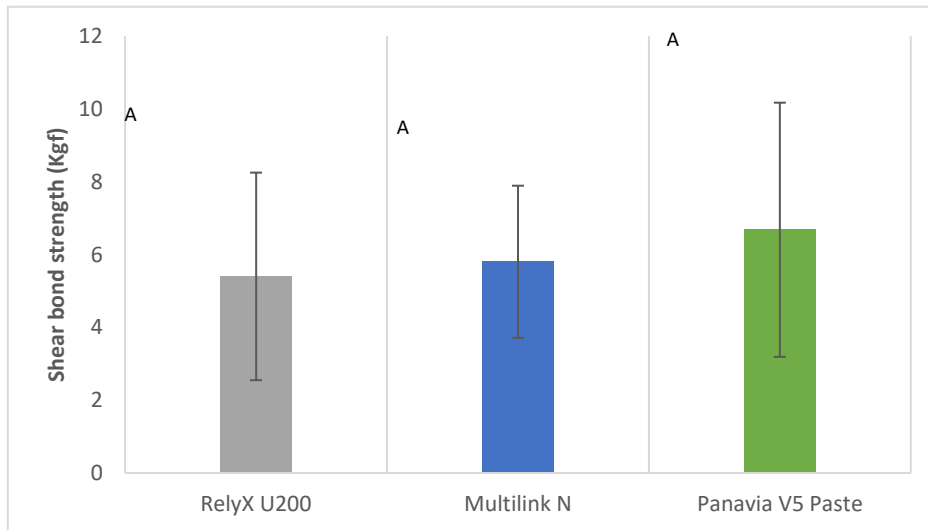
**Table 1** – Averages and standard deviations of bond strength, in Kgf, between Zirconia Katana™ UTML and the resinous cements.

Resinous cement	Average	Standard deviation	Minimum and maximum values
RelyX™ U200	5.40 A	2.85	1.35 and 10.18
Multilink® N	5.80 A	2.09	3.52 and 9.75
Panavia™ V5 Paste	6.68 A	3.49	0.76 and 13.77

Source: Authors.

Caption: Averages followed by the same letters indicate lack of statistical difference between resinous cements.

**Graph 1** – Bar chart of average bond strength between Zirconia Katana™ UTML and resinous cements.

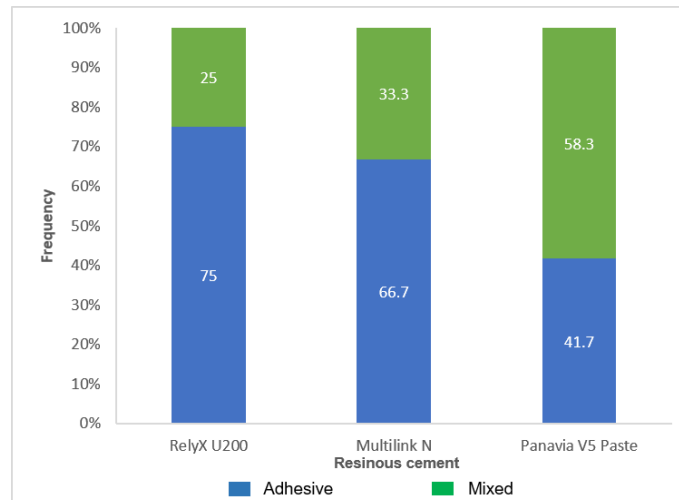


Source: Authors.

Caption: Bars with the same letters indicate lack of statistical difference between resinous cements.

Graph 2 presents the distribution of failure types among the samples; none of the groups presented exclusively cohesive failures. Adhesive failures were more abundant with RelyX™ U200 and Multilink® N, while mixed failures were more prevalent with Panavia™ V5. However, regarding failure type, all three groups were statistically similar, according to the Friedman's test with Freeman-Halton extension ( $p = 0.321$ ).

**Graph 2** – Bar chart of relative frequencies of failure types between Zirconia Katana™ UTML and resinous cements.



Source: Authors.

#### 4. Discussion

Katana™ Zirconia UTML (Kuraray Noritake Dental Inc, Miyoshi, Aichi, Japan) is characterized as a third-generation Zirconia, completely stabilized, arranged in multilayered disks for milling. Shows great optical and mechanical properties, which makes it a versatile material, capable of meeting the aesthetical needs of prosthetics (Colombo et al., 2020; Kang et al., 2020).

Several authors describe the blasting of aluminum oxide to the surface of Zirconia to improve the bond strength to resinous cement (Sharafeddin et al., 2018; Murakami et al., 2017; Sathish et al., 2019; Colombo et al., 2020; Kang et al., 2020; Inokoshi et al., 2020). Colombo et al., 2020 show that Katana UTML samples blasted with abrasive particles perform better in shear stress tests, in agreement with other findings (Sharafeddin et al., 2018; Le et al., 2019); this laid the basis for the methodology used in the present study. Blasting is recommended with caution, though, since some studies (Murakami et al., 2017; Kang et al., 2020) show a potential for the formation of cracks on the surface of Zirconia if done incorrectly, which could lead to poor adhesion and early and catastrophic failure. For this reason, this study followed the manufacturers' blasting protocols. On the other hand, Kang et al., 2020 report better adherence with previous abrasive and acid treatment. Negreiros et al., 2017 show that the use of a cleaning agent combined with a binding agent after blasting with aluminum oxide favors adhesion. Our shear stress test results show no difference between the brands of cements tested in terms of bond strength, regardless of their chemical composition. However, Lee et al., 2015 found differences in bond strength to Zirconia (Fulluster, HASS Co., Gangneung, Korea) of three resinous cements (Calibra®- Dentsply, Konstanz, Germany, MultilinkR N - Ivoclar-vivadent, Schaan, Liechtenstein, RelyX™ Unicem Clicker™ - 3M ESPE, Seefeld, Germany) in the shear stress test. The authors found a correlation between the content of phosphate monomers and the cement performance.

Akay et al., 2016 also found statistical differences between brands of cement (Variolink II / Panavia SA) when tested on the Zirconia surface (ICE Zirkon) with different surface treatments. The study showed that 10-MDP-containing resinous cement combined with the immersion of Zirconia in acid solution for 10 minutes increased the bond strength in the shear stress test. The authors also noticed that 10-MDP increases bond strength to blasted surfaces. Lüthy et al. 2006 also found better results for 10-MDP-containing cements in comparison with cements containing other types of monomers. These results are in disagreement with the results found in the present study.

Magalhães et al., 2020 suggested the use of titanium dioxide nanotubes (TiO<sub>2</sub>-nts) in various concentrations added to Y-TZP and resinous cement to improve the bond strength results in shear stress tests. Their study showed no improvement in resinous cements without MDP in their composition (RelyX U200 (3M ESPE, Saint Paul, USA). However, TiO<sub>2</sub>-nts added to Zirconia in the concentration of 5%, improved adherence to a 10-MDP-containing cement.

Similarly to the findings of Gomes et al., 2015, Lee et al., 2015 and Sharafeddin et al., 2018, cohesive failure was not observed in any of the groups in the present study. On the other hand, Negreiros et al., 2017 observed cohesive failures in all groups tested; these failure types were predominant in groups where the samples were applied with Monobond Plus, which may be related to an effective bond strength in short-term tests.

The present study found mixed failures, but adhesive failures were more frequent. Mixed failures were predominant in the group of 10-MDP-containing cement; adhesive failures were more frequent in the group of RelyX U200 cement, a brand of cement that does not contain 10-MDP, similarly to the results obtained in Gomes et al., 2015 and Magalhães et al., 2020. In contrast, in Le et al., 2019, cohesive and adhesive failures were described, the latter being more frequent in all test groups. Lüthy et al., 2006 also found cohesive and adhesive failures, in opposition to the findings of the present study.

## 5. Conclusion

The three cement brands tested performed similarly in the test;

None of the groups presented cohesive failure; there was no difference in failure type frequency between groups.

The bond strength between Katana™ Zirconia UTML (Kuraray Noritake Dental Inc, Miyoshi, Aichi, Japan) and three resin cements was analyzed in this study, following protocols recommended by the manufacturers of the brands used and also according to literature recommendations. However, it is suggested that further studies be conducted to obtain greater knowledge on the subject, such as thermocycling of specimens, other mechanical tests such as microshear, among other analyzes in the techniques, so that clinical approaches are more predictable and lasting, bringing greater long-term success.

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