

# Surface properties of composite resins with and without fluorides for bracket bonding

# Propriedades de superfície de resinas compostas com e sem fluoretos para colagem de suportes

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### **ABSTRACT**

The aim of this study was to evaluate the surface properties of orthodontic resins with and without fluoride. Forty disks, measuring 2 mm thick by 6 mm in diameter, were made of 4 bracket-bonding composite resins (n=10): Transbond Plus Color Change-3M/Unitek (TPCC); Transbond XT- 3M/Unitek (TXT), Orthocem -FGM (OC); Orthocem UV Trace-FGM (OCUV). The discs were photoactivated for 40 seconds with irradiance of 450 mW/cm2 and manually polished in sequence by silicon carbide sandpapers with 1200 and 2000 grain size and finished with diamond paste and felt disc. The surface microhardness



analysis was performed using a Shimadzu Micro Hardness Tester HMV-2,000 (Shimadzu Corporation, Kyoto, Japan) with a load of 50 gF and a 5 second penetration time. Surface roughness readings were taken using a Surf Corder Roughness Meter (SE 1700- Kosaka, Lisboa-Portugal). For data analysis, ANOVA (one-way) was used, followed by Tukey's post-test ( $\alpha$ =0.05). The microhardness results showed a difference ( $p\le0.05$ ) in the means of the orthodontic resins between TPCC and TXT with the other groups. After the surface roughness analysis, the averages showed that TPCC resin showed higher roughness compared to OC and OCUV (p≤0.05), and there was no statistical difference with TXT. It was concluded that statistically the composite resins with fluoride showed significant difference regarding hardness and roughness.

**Keywords:** Orthodontics, Hardness, Composite Resin.

### RESUMO

O objectivo deste estudo era avaliar as propriedades de superfície das resinas ortodônticas com e sem flúor. Quarenta discos, medindo 2 mm de espessura por 6 mm de diâmetro, foram feitos de 4 resinas compostas de colagem de braquetes (n=10): Transbond Plus Color Change-3M/Unitek (TPCC); Transbond XT- 3M/Unitek (TXT), Orthocem -FGM (OC); Orthocem UV Trace-FGM (OCUV). Os discos foram fotoactivados durante 40 segundos com irradiação de 450 mW/cm2 e polidos manualmente em sequência por lixas de carboneto de silício com 1200 e 2000 gramas e acabados com pasta de diamante e disco de feltro. A análise da microdureza da superfície foi realizada utilizando um aparelho Shimadzu Micro Hardness Tester HMV-2,000 (Shimadzu Corporation, Kyoto, Japão) com uma carga de 50 gF e um tempo de penetração de 5 segundos. As leituras da rugosidade superficial foram feitas utilizando um Medidor de Rugosidade de Fronteira de Surf (SE 1700- Kosaka, Lisboa-Portugal). Para análise de dados, foi utilizada ANOVA (one-way), seguida pelo pós-teste de Tukey (α=0,05). Os resultados da microdureza mostraram uma diferença (p≤0.05) nos meios das resinas ortodônticas entre TPCC e TXT com os outros grupos. Após a análise da rugosidade superficial, as médias mostraram que a resina TPCC mostrou maior rugosidade em comparação com o OC e OCUV (p≤0.05), e não houve diferença estatística com o TXT. Concluiu-se que estatisticamente as resinas compostas com flúor mostraram diferenças significativas em relação à dureza e rugosidade.

Palavras-chave: Ortodontia, Dureza, Resina Composta.

# 1 INTRODUCTION

One of the major clinical challenges encountered during orthodontic therapy is the emergence of enamel demineralization, which are called white spot lesions. The emergence of this process occurs due to the use of fixed appliances and accessories, leading to greater retention of bacterial biofilm, as well as greater difficulty in cleaning by the patient [1]. These lesions or decalcifications of the enamel are characterized by the color opaque white, and over time, this white spot can recalcify, but the opaque white color usually remains [2-5].



Moreover, the roughness of a resin composite can also generate greater retention of bacterial plaque, enabling gingival irritation, development of stains and secondary caries. This characteristic, because it is associated with the presence of irregularities on the material surface, can be influenced by the size, type, and amount of fillers present in the composite resin [6].

Given this, several biomaterials have been developed aiming to improve their properties and interaction with the biological host system. Within this context, it is known that fluoride is a fundamental ion for caries disease control, since, due to its high electronegative potential, it can form a protective barrier, binding to calcium ions present in the oral environment. Benson et al.7 have reported that the use of fluoride agents as a preventive measure is effective in reducing caries in orthodontic treatment [7,8].

Thus, fluoridated materials are an advantage during orthodontic treatment because they do not depend on the patient to be used and, when bonding, they allow the ion to be close to where it will be needed (around the bracket) in cases of poor hygiene and sugar ingestion [9]. Therefore, fluoride-releasing orthodontic adhesive materials have been developed and, with regard to their release efficacy and amount of fluoride on reducing demineralizations, it has been observed that these fluoride-releasing resin composites are useful in reducing white spot lesions and, consequently, are able to avoid topical fluoride applications [10-12].

Due to the development of several materials for orthodontics with fluoride additive characteristics, and the studies in the literature on the various orthodontic composites being correlated to shear bond strength tests, [10,13,14] the study of surface mechanical properties tests, such as microhardness and roughness, have become important, thus allowing the definition of these materials, being more frequently performed to evaluate restorative composite resins [15].

Therefore, the aim of this study was to evaluate the surface mechanical properties of orthodontic resins with and without fluoride. The null hypothesis of the study was that the different groups of resins used showed no difference in surface roughness and microhardness.

# 2 MATERIALS AND METHODS

The present research was approved by the Research Ethics Committee - Protocol 259/2017 (APPENDIX A).



### 2.1 EXPERIMENTAL DESIGN

In this study, 4 light-cured composite resins from 2 commercial brands were evaluated: Without fluoride: Transbond XT - TXT (3M/Unitek®), Orthocem - OC (FGM). With fluoride: Transbond Plus Color Change - TPCC (3M/Unitek®), Orthocem UV Trace - OCUV (FGM®).

# 2.2 SAMPLE PREPARATION

Forty resin disks (n = 10) were made using an elastomer mold measuring 6 mm in diameter by 2 mm thick. The composite was inserted into the mold with the aid of a resin insertion spatula, performed in increments. After composite application, polyester matrices were positioned for resin planning and extravasation of the excess, exerting pressure with a glass plate. Each disc was then light-cured for 40 seconds using a Radii Cal® (SDI Dental Product, Bayswater, Victoria, Australia) light curing unit with an irradiance of 450 mW/cm<sup>2</sup>, measured regularly with a radiometer (Demetron, Danburry, CT, USA).

Subsequently, manual polishing was performed with silicon carbide sandpaper with 1200 and 2000 grain size, and felt disc with Diamond R paste (FGM®) based on aluminum oxide, with grain size of 6 to 8 microns. All discs were stored dry, separately divided by groups.

PRODUCT	MANUFACTURER	GROUP	FLUORIDE RELEASE	COMPOSITION
TPCC	3M/Unitek (Monrovia - Califórnia - EUA)	n=10	YES	Organic Matrix: Bis-GMA and TEGDMA Inorganic part: Quartz silanized in 40% of the volume, glass with silane hydrolyzed in 40% of the oligomer volume of citric acid dimethacrylate.
TXT	3M/Unitek (Monrovia - California - USA)	n=10	NO	Organic matrix: Bis-GMA and TEGDMA Inorganic part: silanized silica with 70 to 80% by volume ndimethyl benzocaine, hexafluorophosphate, camphoroquinone



OCUV	FGM (Joinville – SC –	n=10	NO	Methacrylic monomers such as BisGMA, TEGDMA, and phosphated methacrylic monomers,
	Brazil)			stabilizer, sodium fluoride, camphorquinone, and co-initiator. Inactive ingredients: Inorganic fillers of nanometer silanized silicon dioxide and luminescent pigment.
OC	FGM (Joinville – SC – Brazil)	n=10	YES	Methacrylic monomers such as BisGMA, TEGDMA and phosphated methacrylic monomers, stabilizer, camphorquinone, co-initiator and silicon dioxide nanometer filler.

Source: Research data (2017).

### 2.3 SURFACE MICROHARDNESS ANALYSIS OF ORTHODONTIC RESINS

The Knoop microhardness analyses were performed 5 days after the specimens were made using a Shimadzu Micro Hardness Tester HMV-2,000 (Shimadzu Corporation, Kyoto, Japan) with a load of 50g/Force and an indentation time of 5 seconds, with 3 indentations on the upper surface of each disc.

# 2.4 SURFACE ROUGHNESS ANALYSIS OF ORTHODONTIC RESINS

The surface roughness readings were taken 5 days after the specimens were made and stored dry, using the Surf Corder device (SE 1700- Kosaka, Lisbon, Portugal). Each reading was measured with the profilometer needle in three different referential positions, obtained by rotating the specimen. The roughness considered was the arithmetic mean between the three peaks and valleys (Ra) traversed by the profilometer in a measuring stretch calibrated for 4.8mm. Three readings were taken in different positions for each specimen and an average was obtained for each resin disk.

# 2.5 STATISTICAL ANALYSIS

The Knoop microhardness and roughness data were evaluated by Analysis of variance (one-way) and Tukey's test, using a 5% significance level. The statistical tests were performed by the software Statistica for Windows, version 6.0 (StatSoft, Tulsa - OK, USA).

# 3 RESULTS

Table 1 shows the results of Surface Microhardness, where one can observe differences between the averages of the orthodontic resins between TPCC and TXT with



the other groups ( $p \le 0.05$ ). After the surface roughness analyses, averages showed that TPCC resin presented higher roughness compared to OC and OCUV (p≤0.05), and there was no statistical difference with TXT (p=0.05).

Table 1 - Averages (Standard Deviation) of surface roughness and Microhardness of light-cured composite resin discs of two different commercial brands, per group: Averages not sharing a letter are significantly different in column. Tukey's test (p-value  $\leq 0.05$ ).

Mean (Standard Deviation)			
Roughness	Knoop Microhardness		
0.1711 (0.04) <sup>ab</sup>	118.81 (27.01) <sup>a</sup>		
0.2137 (0.05) <sup>a</sup>	103.12 (17.68) <sup>a</sup>		
0.1450 (0.05) <sup>b</sup>	78.22 (15.34) <sup>b</sup>		
0.1382 (0.04) <sup>b</sup>	77.75 (8.58) <sup>b</sup>		
	Roughness  0.1711 (0.04) <sup>ab</sup> 0.2137 (0.05) <sup>a</sup> 0.1450 (0.05) <sup>b</sup>		

The significance level  $\alpha$ =0.05 was considered. Equality of variances was assumed for the analysis. Given the p-value of the F-statistic, computed in the ANOVA table (p=0.005), and given the significance level of 5%, the information brought by the explanatory variables is significantly better than what a basic mean would bring.

### 4 DISCUSSIONS

The present study aimed to evaluate the roughness and hardness properties (in vitro studies) of these materials, which showed a statistically significant difference, rejecting the null hypothesis. There is a significant deficiency in the literature regarding the evaluation of fluoride releasing materials, as reported by previous studies [10,15,16] since orthodontic composites are mostly correlated to shear bond strength tests [10,13,14,17]. One aspect that was not evaluated in the present study, and that should be considered, is that fluoride is believed to be related to the mechanical properties of the resins [15, 17,18] since the evaluation by Bowen [19] in 1963 of Bis-GMA based resins with and without filler particles, it has been shown that the incorporation of filler particles provides beneficial changes in some properties, among them, the resistance to penetration and that the content of inorganic particles directly affected the hardness values, where the hardness of a material depends directly on the bonding forces between atoms, ions, or molecules [15,20]. This justifies the results obtained in the present research with higher hardness presented in the materials with fluoride (TPCC and OCUV), and cooperates to further studies on these mechanical properties.



Surface characteristics of the materials evaluated prove that further exploration is needed for better results and clinical indications in orthodontic practice. The little exploration of these properties on the evaluation of the roughness of these materials had no correlation with the properties added on the resins of the same brand (TXT and TPCC, OC and OCUV), since the surface irregularities present in these materials are influenced by their size, type and amount of fillers present and added to the composite [6,15], which in this study showed a correlation of roughness with the fluoride added to orthodontic resins (TPCC and OCUV), This is important and also relevant for the study of orthodontic resins, with and without particles, since further studies to determine the surface properties and the influence of added particles on their properties will be effective in promoting improvements in their mechanical characteristics, especially in their clinical application.

Literature reports that the incorporation of inorganic fillers in a polymer provides improvement in mechanical properties such as hardness [21]. The study of microhardness testing becomes more necessary because it allows the precise definition of these materials and their use. Moreover, as studies have shown that hardness may be related to the degree of conversion of resin monomers, and that the Transbond XT resin showed statistically superior shear bond strength and hardness values compared to other resins [15], we can say that the properties of fillers added to a resin of the same brand may directly affect the mechanical and superficial properties of the adhesive material. Thus, the choice of orthodontic material should be based on the knowledge of its properties and study of its efficiency, through clinical and laboratory experiments, and is of great importance to provide the orthodontist with better results during and after the patient's orthodontic treatment.

The applicability of orthodontists is limited by the wide range of adhesive composite materials used for bonding, however, with few evaluations of their specificities and it becomes necessary to know their physical properties that do not involve any structural modification at the molecular level of the materials, such as mechanical properties [22,23]. Thus, it is understood the need for evaluation to better understand the mechanical properties of orthodontic materials currently used, as well as those that present the addition of fluoride, and the evaluation of its interference on these properties.



# **5 CONCLUSIONS**

It was concluded that the hardness and roughness properties of orthodontic composite resins showed differences between the composite resins with fluoride, and that fluoride cannot be correlated as an interference factor in these properties.



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