Original Research
Dental Materials

Surface roughness and wear of resin cements after toothbrush abrasion

Sérgio Kiyoshi ISHIKIRIAMA
Juan Fernando ORDOÑÉZ-AGUILERA
Rafael Massunari MAENOSONO
Fernanda Lessa Amaral VOLÚ
Rafael Francisco Lia MONDELLI

Universidade de São Paulo, Bauru School of Dentistry, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru, SP, Brazil.

Declaration of Interests: The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript.

Corresponding Author:
Sergio Kiyoshi Ishikiriama
E-mail: serginho@usp.br

DOI: 10.1590/1807-3107BOR-2015.vol29.0011

Submitted: May 06, 2014
Accepted for publication: Aug 29, 2014
Last revision: Oct 20, 2014

Abstract: Increased surface roughness and wear of resin cements may cause failure of indirect restorations. The aim of this study was to evaluate quantitatively the surface roughness change and the vertical wear of four resin cements subjected to mechanical toothbrushing abrasion. Ten rectangular specimens (15 × 5 × 4 mm) were fabricated according to manufacturer instructions for each group (n = 10): Nexus 3, Kerr (NX3); RelyX ARC, 3M ESPE (ARC); RelyX U100, 3M ESPE (U100); and Variolink II, Ivoclar/Vivadent (VL2). Initial roughness (Ra, μm) was obtained through 5 readings with a roughness meter. Specimens were then subjected to toothbrushing abrasion (100,000 cycles), and further evaluation was conducted for final roughness. Vertical wear (μm) was quantified by 3 readings of the real profile between control and brushed surfaces. Data were subjected to analysis of variance, followed by Tukey's test ($p < 0.05$). The Pearson correlation test was performed between the surface roughness change and wear ($p < 0.05$). The mean values of initial/final roughness (Ra, μm)/wear (μm) were as follows: NX3 (0.078/0.127/23.175); ARC (0.086/0.246/20.263); U100 (0.296/0.589/16.952); and VL2 (0.313/0.512/22.876). Toothbrushing abrasion increased surface roughness and wear of all resin cements tested, although no correlation was found between those variables. Vertical wear was similar among groups; however, it was considered high and may lead to gap formation in indirect restorations.

Keywords: Dental Cements; Resin Cements; Dental Restoration Wear.

Introduction

An ideal luting procedure consists of a stable and long-lasting seal between an indirect restoration and the tooth, avoiding infiltration and preventing plaque accumulation.[1] Due to high demand by patients for cosmetic procedures using all-ceramic systems, cements also need to provide adhesion and aesthetics. In this context, resin cements have some advantages, since these materials provide not only aesthetics and adhesion but also lower solubility than traditional dental cements based on glass ionomers and zinc phosphate.[2,3]

Despite these advantages, in a luting procedure, a cement line always remains,[4,5] and the exposure of this cement in the oral cavity makes it susceptible to degradation, which may occur as a result of intraoral mechanisms such as water sorption, hydrolysis, dynamic fatigue,[6] or even toothbrushing abrasion.[7] As a consequence, increased surface roughness and wear can promote the retention of dental plaque, leading to a greater risk of developing secondary caries, periodontal diseases, discoloration, and even loss of the restoration.[8]

In 2- and 4-year clinical trials,[9,10] resin cements have already shown an increased loss of marginal integrity on indirect restorations, which could lead to premature failure of these restorations. Some authors[11,12,13] have already assessed different characteristics of resin cements for a better understanding of this issue; however, still more evidence is necessary, particularly regarding surface roughness and wear of resin cements. Therefore, the aim of this study was to evaluate *in vitro* the surface roughness change and the vertical wear of different resin cements after mechanical toothbrushing abrasion.

Methodology

Experimental Design

This study was performed with 10 experimental specimens per group (n = 10). The following 2 quantitative response variables were obtained: roughness (Ra, µm) and vertical wear (µm). For roughness, two variation factors were present as follows: Resin Cement at four levels, Nexus 3 (NX3) (Kerr, Washington, USA); RelyX ARC (ARC) (3M ESPE, St. Paul, USA); RelyX U100 (U100) (3M ESPE); and Variolink II (VL2) (Ivoclar/Vivadent, Schaan, Liechtenstein) (Table 1) and Time at two levels (initial and final). For vertical wear, the only variation factor present was Resin Cement at four levels (NX3, ARC, U100, VL2).

Specimen preparation

Ten specimens per group were prepared with a metallic matrix (4 × 5 × 15 mm).[14] Light cure was performed at three different points, each for 40 seconds, to ensure light exposure throughout the sample. The specimen surfaces were polished using a metallographic polishing machine (Arotec, Cotia, Brazil) with silicon carbide papers at 600, 800, and 1200 grit (Buehler, Lake Bluff, USA) and regular cooling water. All specimens were stored hermetically in sealed bottles containing deionized water at 37 ± 1 °C and 100% absolute humidity for 7 days.

Toothbrushing abrasion

Before the abrasive challenge, half of the surface area was protected with isolation tape to serve as the control side. Toothbrushing abrasion was performed in a toothbrushing machine (Elquip, Sao Carlos, Brazil) particularly developed for testing purposes[15,16] with soft-bristle dental brushes (Johnson & Johnson Industrial Ltda., Sao José dos Campos, Brazil) and abrasive toothpaste (Colgate Palmolive Ltda., Sao Bernardo do Campo, Brazil). Toothbrushes were positioned to have contact with only half of the specimen surface, and the other half remained protected with isolation tape. In total, 100,000 cycles were performed, at 4.5 cycles per second, with load of 300 g over the toothbrush heads.[14,15] Every 2 minutes, a 0.4-mL quantity of toothpaste and distilled water (ratio, 1:2) was injected, and the toothbrushes were changed at the end of 50,000 cycles. After abrasion testing, specimens were rinsed under running water and cleaned in a sonic device for 5 minutes and then stored in deionized water (37 °C).

Roughness and vertical wear measurements

Surface roughness was analyzed with a roughness meter (Jenoptik AG, Jena, Germany) and Turbo

Table 1. Commercial brands, polymerization type, composition, filler size, and ratio of resin cements evaluated.

Commercial brands	Composition	Filler loading (vol/wt%)	Filler size
Nexus 3 (Kerr)	Uncured methacrylate ester monomers, inert mineral fillers, activators and stabilizers, and radiopaque agent	47 / 67.5	0.6 µm
RelyX ARC (3M ESPE)	BisGMA, TEGDMA, polymer, zirconia/silica filler	* / 67.5	1.5 µm
RelyX U100 (3M ESPE)	Glass powder, methacrylated phosphoric acid esters, TEGDMA, silane-treated silica, sodium persulfate	54 / 72	< 9.5 µm
Variolink II (Ivoclar/Vivadent)	Bis-GMA, UDMA, TEGDMA, inorganic fillers	46.7 / 73	0.7 µm

*Information not provided by manufacturer.

Datawin software (Hommel-Etamic, Schwenningen, Germany). Five random readings were obtained from each specimen surface, and mean roughness values (Ra) were obtained.[17] Initial roughness (baseline) was performed before the toothbrushing test, while final roughness was obtained after the test. The parameters for roughness measurements are listed in Table 2.

For vertical wear evaluation, the same equipment was used in the profilometer function, and the parameters are also listed in Table 2. Wear was calculated by the mean of three readings of the real profile. The active tip of the profilometer traced the specimen's surface from the control side to the test side (Figure 1).

Statistical analysis

Roughness data were subjected to repeated-measures analysis of variance (ANOVA), followed by Tukey's test ($p \leq 0.05$). Vertical wear data were initially subjected to the Shapiro–Wilks normality test, and because data presented a normal distribution, the parametric one-way ANOVA was performed. The Pearson correlation test was also performed for correlation between wear and roughness ($p \leq 0.05$). All statistical analyses were performed with the software Statistica 10.0 (StatSoft, Tulsa, USA).

Results

Roughness analysis

Means and standard deviations of roughness (Ra, µm) before and after toothbrushing abrasion for each group are described in Table 3. Both variation factors (Resin Cements and Time) were significant, with interactions between factors. NX3 and ARC presented lower surface roughness values, while U100 and VL2 presented higher values. Regarding differences between initial and final roughness, ARC, U100, and VL2 presented higher surface roughness values after toothbrushing abrasion.

Vertical wear analysis

Table 4 presents means and standard deviations of vertical wear (µm) after toothbrushing abrasion. No statistically significant differences were observed ($p = 0.052$).

Correlation between surface roughness and vertical wear

No correlation was found by the Pearson correlation test between vertical wear/initial roughness ($p = 0.789$) and vertical wear/final roughness ($p = 0.897$).

Discussion

Surface roughness is a characteristic of resin cement that plays an important role in the adherence and maturation of the bacterial biofilm. According to some authors, surface roughness values higher than 0.2 µm may significantly increase plaque adhesion,[18] supporting the development of secondary caries and periodontal diseases. In the present study, two of four resin cements showed initial surface roughness values higher than 0.2 µm, and after toothbrushing abrasion, only NX3 presented a satisfactory surface roughness. These results are concerning and emphasize the need for more investigations in this area.

U100 and VL2 presented significantly higher surface roughness values than NX3 and ARC. One possible explanation is the percentage of filler loading (Table 1). Both U100 and VL2 have a higher percentage of filler loading, which may lead to {1.1 [EN] Please check the change} increased surface roughness when associated with the less viscous resin matrix needed for cements. Some authors have investigated the effect of toothbrushing abrasion on flowable resin composites, which are also less viscous than restorative composites.

Table 2. Parameters used for initial and final roughness measurements.

Parameter	Roughness	Wear
Tolerance (extreme values to be considered at the readings)	Tmin = 0.01 µm Tmax = 8.00 µm	Tmin = 8.00 µm Tmax = 40 µm
Trial limit (real extension traveled by the active tip)	Lt = 5.00 mm	Lt = 10.00 mm
Measuring limit (extension considered at the readings)	Lm = 4.5 mm	Lm = 9 mm
Cut-off (filtering, minimizing the interference of surface ripple)	Lc = 0.25 mm	Lc = 0.00 mm

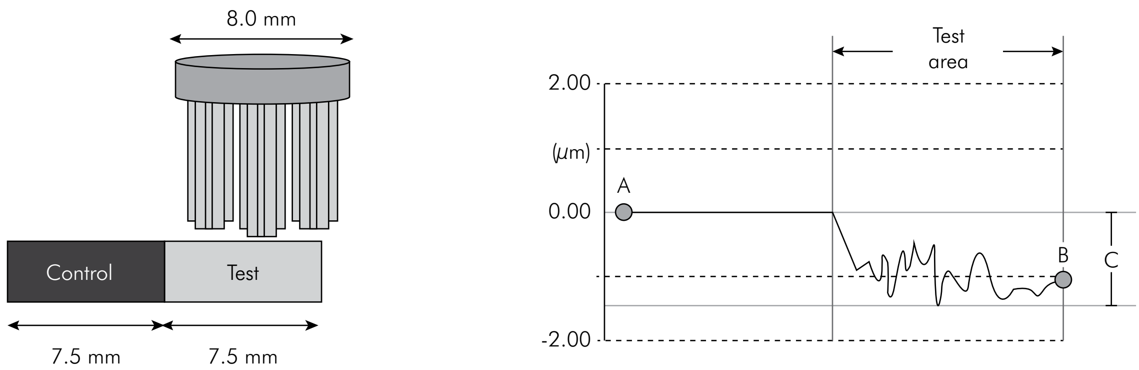

Figure 1. Illustration of toothbrush head positioning over the test side of the specimen surface. Profilometer tip tracing from the control side to the test side (A-B). Maximum wear value (C).

Table 3. Means and standard deviations of initial and final roughness (Ra, μm) after toothbrushing abrasion.

Groups	Initial Roughness	Final Roughness
NX3	0.078 ± 0.044 aA	0.127 ± 0.043 aA
ARC	0.086 ± 0.057 aA	0.246 ± 0.042 bA
U100	0.296 ± 0.125 aB	0.589 ± 0.168 bB
VL2	0.313 ± 0.086 aB	0.511 ± 0.110 bB

Lowercase letters - analysis between columns (initial × final).
Uppercase letters - analysis between rows (group comparison).

Table 4. Means and standard deviations of wear for all tested cements (μm).

Groups	Wear
U100	16.95 ± 4.36A
ARC	20.26 ± 6.00A
VL2	22.88 ± 5.22A
NX3	23.18 ± 5.97A

Uppercase letters - analysis between rows (group comparison).

Similar results are observed, with increased surface roughness after the toothbrushing test.[15]

The mechanism by which resin cements become rougher after toothbrushing abrasion is still not well established. The authors hypothesize that mechanical abrasion may degrade the organic matrix and expose filler particles, leading to increased surface roughness, particularly in resin cements with higher filler loading. Analysis such as by atomic force microscopy may be useful for a better understanding of this issue.

In terms of wear, no statistically significant difference was observed ($p = 0.052$). The great standard deviation determined by this methodology did not provide enough test power, which was considered lower (0.43) than that desired (0.80). Further investigations with this methodology must consider a higher number of specimens to avoid the lack of differences between resin cements. Some authors have observed lower numerical values of vertical wear after toothbrushing abrasion of resin cements positioned between layers of lithium disilicate.[4] Despite the fact that this situation is more similar to the clinical usage of resin cements, the ceramic layers could protect resin cements from further wear beyond the positioning of the toothbrushes, which may have led to the lower wear values. Both results, however, are indicative that resin cements lack significant wear resistance and may be susceptible to the development of gaps in indirect restorations over time. These findings are in agreement with those of a recent controlled clinical trial that reported 90% of small marginal deficiencies, probably caused by increased wear of RelyX Unicem over a 4-year period.[9]

The authors believe that the mechanisms by which an abrasive challenge leads to the surface roughness change and wear are considerably different, which may have resulted in the lack of correlation between these response variables. The former response variable may be more related to stability between fillers and organic matrix: for example, some authors have observed that silorane resin composites are very stable in aqueous solution, and they show smooth surface roughness even after 1-year water storage.[19] Conversely, vertical wear may be more associated with individual characteristics of fillers and organic matrix. Restorative resin composites, which have formulations very similar to those of resin cements, present satisfactory wear resistance and can be used safely in posterior teeth restorations. They differ from resin cements mainly in filler loading, shape and size, and a higher viscosity of the organic matrix, which may lead to increased wear resistance.

Conclusion

It is possible to conclude that resin cements present increased surface roughness after toothbrushing abrasion. Cements with higher percentages of filler loading presented higher surface roughness values. All cements showed similar but high rates of vertical wear after toothbrushing.

Acknowledgements

The authors acknowledge FAPESP (*Fundação de Amparo à Pesquisa do Estado de São Paulo*) for financial support (process no. 2010/14111-0).

References

1. Pegoraro TA, da Silva NR, Carvalho RM. Cements for use in esthetic dentistry. Dent Clin North Am. 2007 Apr;51(2):453-71.
2. Vrochari AD, Eliades G, Hellwig E, Wrbas KT. Water sorption and solubility of four self-etching, self-adhesive resin luting agents. J Adhes Dent. 2010 Feb;12(1):39-43.
3. Cruz CAS, Adabo GL, Rettondini WC, Sa DN, Silva Filho FP. Penetration of calcium hydroxide based cements by dental enamel conditioning acids. Rev Odontol UNESP. 1990 Jan;19(1):173-82. [Portuguese].
4. Belli R, Pelka M, Petschelt A, Lohbauer U. In vitro wear gap formation of self-adhesive resin cements: a CLSM evaluation. J Dent. 2009 Dec;37(12):984-93.
5. Prakki A, Cilli R, Da Costa AU, Goncalves SE, Mondelli RF, Pereira JC. Effect of resin luting film thickness on fracture resistance of a ceramic cemented to dentin. J Prosthodont. 2007 May-Jun;16(3):172-8.
6. Cilli R, Mattos MC, Honorio HM, Rios D, Araujo PA, Prakki A. The role of surface sealants in the roughness of composites after a simulated toothbrushing test. J Dent. 2009 Dec;37(12):970-7.
7. Goldstein GR, Lerner T. The effect of toothbrushing on a hybrid composite resin. J Prosthet Dent. 1991 Oct;66(4):498-500.
8. Bollen CML, Lambrechts P, Quirynen M. Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. Dent Mater. 1997 Jul;13(4):258-69.
9. Peumans M, Voet M, De Munck J, Van Landuyt K, Van Ende A, Van Meerbeek B. Four-year clinical evaluation of a self-adhesive luting agent for ceramic inlays. Clin Oral Investig. 2013 Apr;17(3):739-50.
10. Taschner M, Krämer N, Lohbauer U, Pelka M, Breschi L, Petschelt A, et al. Leucite-reinforced glass ceramic inlays luted with self-adhesive resin cement: a 2-year in vivo study. Dent Mater. 2012 May;28(5):535-40.
11. Calgaro PA, Furuse AY, Correr GM, Ornaghi BP, Gonzaga CC. Influence of the interposition of ceramic spacers on the degree of conversion and the hardness of resin cements. Braz Oral Res. 2013 Sep-Oct;27(5):403-9.
12. Ishikiriama SK, Maenosono RM, Oda DF, Ordoñez-Aguilera JF, Wang L, Mondelli RF. Influence of volume and activation mode on polymerization shrinkage forces of resin cements. Braz Dent J. 2013 July-Aug;24(4):326-9.
13. Jongsma LA, Kleverlaan CJ, Pallav P, Feilzer AJ. Influence of polymerization mode and C-factor on cohesive strength of dual-cured resin cements. Dent Mater. 2012 Jul;28(7):722-8.
14. Prakki A, Cilli R, Araujo PA, Navarro MF, Mondelli J, Mondelli RF. Effect of toothbrushing abrasion on weight and surface roughness of pH-cycled resin cements and indirect restorative materials. Quintessence Int. 2007 Oct;38(9):e544-54.
15. Garcia FC, Wang L, D'Alpino PH, Souza JB, Araujo PA, Mondelli RF. Evaluation of the roughness and mass loss of the flowable composites after simulated toothbrushing abrasion. Braz Oral Res. 2004 Apr-Jun;18(2):156-61.
16. Mondelli RF, Prakki A, Cilli R, Navarro MF, Mondelli J. Surface roughness average and scanning electron microscopic observations of resin luting agents. J Appl Oral Sci. 2003 Dec;11(4):327-31.
17. Prakki A, Cilli R, Mondelli RF, Kalachandra S. In vitro wear, surface roughness and hardness of propanal-containing and diacetyl-containing novel composites and copolymers based on bis-GMA analogs. Dent Mater. 2008 Mar;24(3):410-7.
18. Quirynen M, Bollen CM. The influence of surface roughness and surface-free energy on supra- and subgingival plaque formation in man. A review of the literature. J Clin Periodontol. 1995 Jan;22(1):1-14.
19. Giannini M, Di Francescantonio M, Pacheco RR, Cidreira Boaro LC, Braga RR. Characterization of water sorption, solubility, and roughness of silorane- and methacrylate-based composite resins. Oper Dent. 2014 May-Jun;39(3):264-72.