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# SURFACE ROUGHNESS AVERAGE AND SCANNING ELECTRON MICROSCOPIC OBSERVATIONS OF RESIN LUTING AGENTS

## *ALTERAÇÃO DE RUGOSIDADE SUPERFICIAL E OBSERVAÇÕES EM MICROSCOPIA ELETRÔNICA DE VARREDURA DE CIMENTOS RESINOSOS*

### **Rafael Francisco Lia MONDELLI, PhD**

Assistant Professor at Operative Dentistry Department, Bauru Dental School - São Paulo State University, São Paulo, Brazil.

### **Anuradha PRAKKI, DDS, MSC**

Graduate Student at Bauru Dental School - São Paulo State University, São Paulo, Brazil.

### **Renato CILLI, DDS, MSC**

Graduate Student at Bauru Dental School - São Paulo State University, São Paulo, Brazil.

### **Maria Fidela de Lima NAVARRO, PhD**

Associate Professor at Operative Dentistry Department, Bauru Dental School - São Paulo State University, São Paulo, Brazil.

### **José MONDELLI, PhD**

Associate Professor at Operative Dentistry Department, Bauru Dental School - São Paulo State University, São Paulo, Brazil.

The objective of this work was to evaluate the surface roughness changes of three current resin cements after tooth brushing simulation, as well as discuss its relation with scanning electron microscopic observations. The materials employed were Enforce Sure Cure (Dentsply), Rely X (3M-ESPE) and Variolink II (Vivadent). They were subjected to brushing abrasion (100,000 strokes for each specimen) and the surface roughness alterations (before and after strokes) were detected. For each roughness test condition, specimens were coated with gold-palladium and observed on a DSM 900 Zeiss scanning electron microscope. Roughness changes values (Ra) were statistically increased after brushing strokes. Based on the microscopic observations and roughness changes analysis, all cements studied became rougher after brushing strokes.

**UNITERMS:** Roughness; Luting cement, wear; Toothbrush abrasion.

## INTRODUCTION

The fitting of indirect restorations in the oral cavity implies directly on the gap formation between the tooth structure and the restorative material. This leads cements to exposure and degradation in the oral environment and includes several phenomena such as sliding, abrasion, chemical degradation and fatigue<sup>20</sup>. In addition, these mechanisms may operate either alone or in combination with others and, considering the intricacy of the oral environment, the breakdown of dental materials mediated by biological activity is very complicated<sup>15</sup>.

Various methods were utilized to predict degradation of restorative materials, such as leaching of filler elements<sup>19</sup>, scanning electron microscopy<sup>8</sup>, polarized light microscopy<sup>23</sup> and weight change<sup>9</sup>. One of the most utilized methods

regards to surface roughness alterations<sup>3,5,21</sup>, expressed in Ra ( $\mu\text{m}$ ) values, or roughness average.

It is well documented that surface roughness changes of restorative materials caused by degradation may be a factor in bacterial colonization and in the maturation of plaque on restorative materials<sup>17</sup>. This may predispose a restoration to the development of secondary caries and periodontal disease<sup>2</sup>. Staining of indirect restorations<sup>10</sup> and their complete loss were also described<sup>16</sup>. The present work aimed at evaluating the surface roughness changes of three current resin cements after tooth brushing simulation and discuss its relationship with scanning electron microscopic findings.

## METHODS AND MATERIALS

The materials employed are listed in Table 1. Ten specimens were performed of each resin cement. Materials were handled according to the respective manufacturer's instructions.

They were inserted in a silicon matrix with internal dimensions of 5mmX3mm and covered with a polyester strip (TDV Dental Ltda, Santa Catarina, Brazil) and a glass slab (Corning Mexicana S.A, Mexico) that was pressed with an axial load of 500g for 30 seconds, to flow out excess of material. Following, the polymerization was carried out for the manufacturer's recommended exposure times (60s through the strip) with a visible light unit with halogen lamp (Optilight 600, Gnatus, Ribeirao-Preto, Brazil). The opposite surfaces of the specimens were also polymerized (60 seconds). After, they were finished using in order medium, fine and superfine aluminum oxide finishing system (Sof-lex, 3M-ESPE, Sumare, Brazil). Then, specimens were ultrasonically cleaned for 10 minutes and individually stored at 37°C in deionized water.

The surface roughness analysis of the specimens was conducted using a Hommel Tester Basic T1000 machine (Hommelwerke GmbH ref. # 240851 – Schweningem - Germany) and expressed in Ra values (Roughness average –  $\mu\text{m}$ ). Each specimen was gently dried with absorbent paper and the tracings carried out for five times, in five different locations, for each surface. The baseline roughness was obtained by the arithmetic mean of these readings.

Then the specimens were subjected to tooth brushing abrasion test. This was performed with an automatic tooth brushing equipment (M.N. São Carlos – Brazil). Soft nylon bristles (Oral B 35) were applied with a load of 300g, horizontal length of 20mm and 4.5 strokes per second speed. The slurry was prepared by mixing 2:1 of deionized water and Colgate MFP (Colgate Palmolive – Osasco – São Paulo – Brazil) dentifrice by weight. One hundred thousand brushing strokes were done for each group (total of 21 hours of brushing test). The specimens were then rinsed under running tap water for one minute (each sample)

**TABLE 1-** Materials tested

Material	Manufacturer	Batch #	Filler Size (mm)	Filler content (% weight)
Enforce	Dentsply	176	1	60
Rely X	3M-ESPE	27071	1.5	67.5
Variolink II	Vivadent	C55626	0.7	73.4

and ultrasonically cleaned for 10 minutes. Thereafter, the same protocol utilized for baseline roughness determination was repeated. Roughness variation was observed as the difference between baseline and final readings<sup>18</sup>.

For each roughness test condition, before and after brushing strokes, 6 specimens were coated with gold-palladium with a MED 010 Balzers sputter coater for 3min and observed under a DSM 900 Zeiss scanning electron microscope.

## RESULTS

Significant roughness alterations after toothbrushing abrasion were analyzed using paired t-tests for each material ( $\alpha=0.05$ ) and the statistical difference between the roughness changes of the materials was obtained with one-way variance analysis at a significance level of 0.05.

All materials presented an increase in Ra values after brushing strokes, they were statistically significant (Table 2). The variance analysis showed no statistically differences in the roughness changes between the materials tested ( $F=2.40$ ,  $P=0.2$ ). The roughness tracings and SEM of cements are shown in Figures 1, 2 and 3. Generalized wear of the luting agents was minimal.

## DISCUSSION

According to the results of this study, all resin cements investigated presented an increase in roughness average (Ra) values after the toothbrushing test, they were statistically significant. Also, no statistically significant differences were found for the roughness average changes between the materials tested.

These findings may be explained by the roughness parameter utilized in this study. The most utilized parameter is Ra, roughness average, the arithmetic mean of the deviations of the roughness profile about the centerline<sup>3,21</sup>. It is also known as CLA or Center Line Average. Thus the centerline corresponds to that section through the profile, which cuts off equal areas above and below it<sup>4</sup>. However, the same value of Ra can be obtained by a wide range of different profiles and textures. Conversely, this is one of the most employed parameters<sup>3,14,18</sup> and was adopted in the present study to provide comparison to others. Five readings were performed for each specimen in order to increase the surface measurement accuracy of the present study.

Some works examined the surface roughness of composites. When different brands were studied, Willems et al.<sup>22</sup> (1992) found

**TABLE 2-** Paired "T" test\*

Material	"T" value	p
Enforce	-6.6719	0.0001
Rely X	-8.5517	0.00001
Variolink II	-6.2459	0.0001

\* Significance level of 0.05

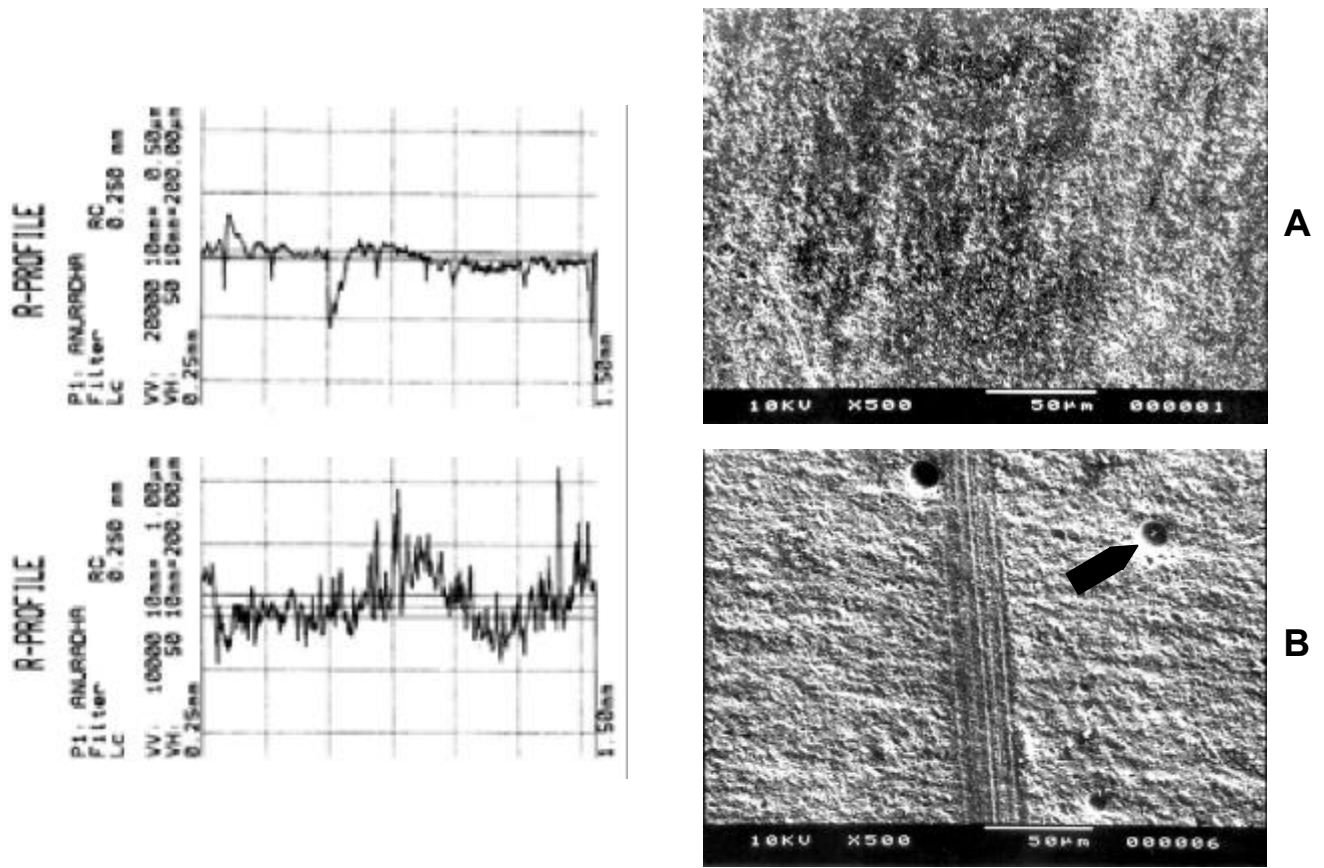


FIGURE 1- Roughness and microscopic evaluation of Enforce (Dentsply) resin cement before (a) and after (b) brushing simulation

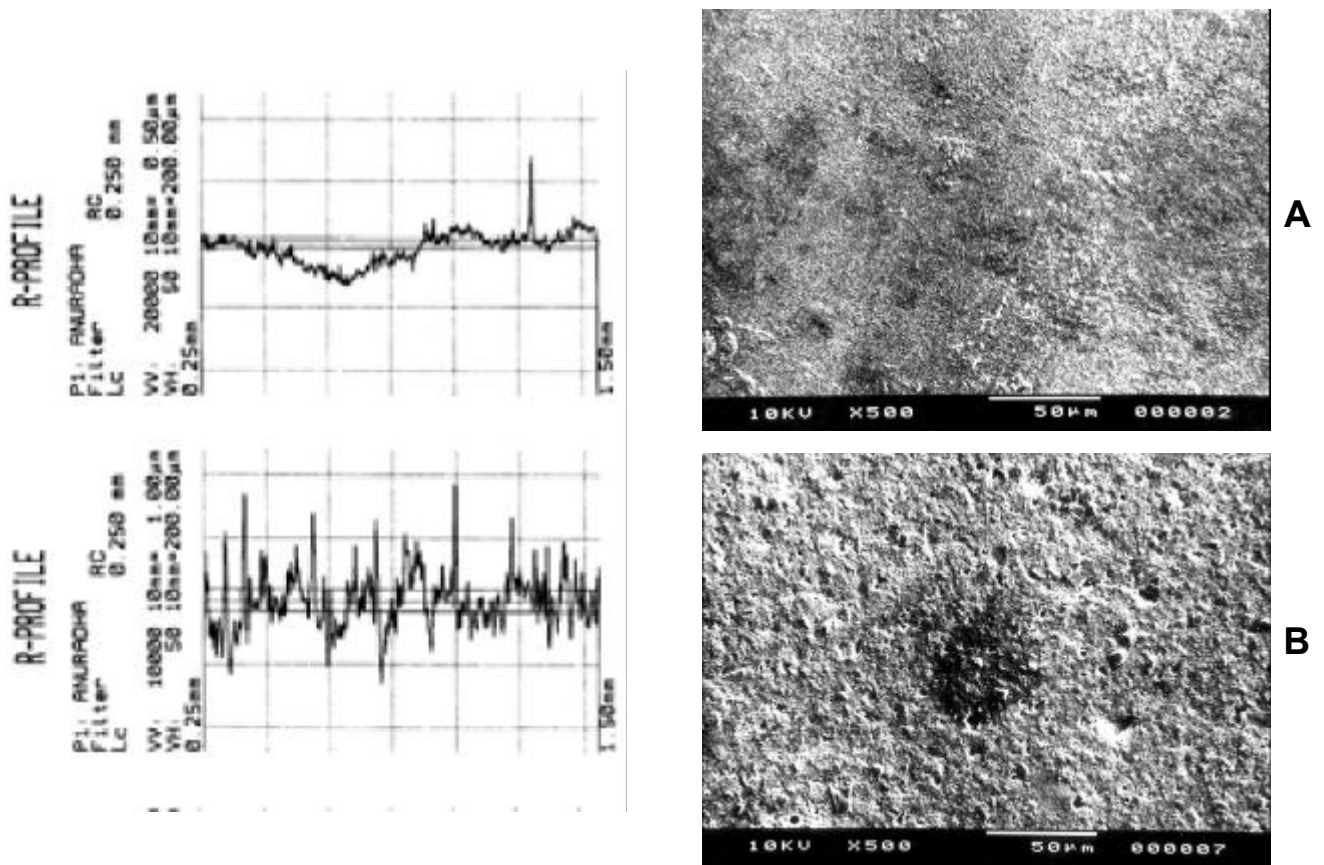


FIGURE 2- Roughness and microscopic evaluation of Rely X (3M) resin cement before (a) and after (b) brushing simulation

that Heliosit (Vivadent), Certain (Johnson & Johnson) and Heliomolar (Vivadent) were the smoothest ( $0.07\mu\text{m}$ ,  $0.08\mu\text{m}$  and  $0.09\mu\text{m}$ ) Ra values, respectively, whereas Litefil A (Shofu Inc.) was initially the roughest ( $R_a = 1.56\mu\text{m}$ ). All studies showed Ra values ranging from  $0.03\mu\text{m}$  up to  $0.2\mu\text{m}^2$  after polishing. In the present work the baseline Ra means were Enforce:  $0.07\mu\text{m}$ ; Rely X:  $0.11\mu\text{m}$  and Variolink II:  $0.05\mu\text{m}$ .

According to Nowicki<sup>14</sup> (1985), roughness height is merely one estimator of surface quality and as stated by Jung<sup>11</sup> (1997) the horizontal aspect of roughness remains largely unconsidered when Ra parameter is used. The same author proposed the measurement of the LR, profile length ratio, which takes into account the vertical and horizontal dimensions of roughness at the same time. As mentioned, although several methods were used to predict degradation of restorative materials, changes in the surface profile have been determined as being useful for measuring the effects of abrasion resistance of restorative materials<sup>6</sup>. Due to the complexity of the surface of restorative materials, a multi parameter representation must be advocated as a method capable of providing full description.

Similarly to the profile tracings, the microscopic findings also indicated differences between the cement surfaces before and after they were subjected to brushing simulation. These characteristics are correlated to the material composition, percentage conversion of monomer to polymer during curing, medium filler size, problems with coupling agent effectiveness and hydrolysis that have also been proposed as possible weak links<sup>7, 13</sup>. As described by Kawai, Iwami, Ebisu<sup>12</sup> (1998),

composites abrasion occurs in two steps: initially there is a selective wear in organic matrix, which leads to inorganic particles exposure and protrusion. Afterwards, the mechanical stress dislodges these particles from the matrix.

While comparing Enforce (Dentsply) and Rely X (3M-ESPE) profiles with microscopic photographs before and after the abrasion test (Figs. 1 and 2), loss of organic matrix and inorganic particle protrusion were observed. These characteristics were also observed in fig. 3, in a less expressive way, what may be explained to Variolink II medium filler size,  $0.7\mu\text{m}$ , which is smaller than Enforce and Rely X,  $1.0\mu\text{m}$  and  $1.5\mu\text{m}$  respectively (Figures 1, 2 and 3). For specimen's preparation, the base and catalyst luting composite were mixed. This type of manipulation can guide to bubble incorporation<sup>1</sup> as it is shown in figs. 1 and 3 (arrows). In these cases there must be a concern, since the diamond needle of some equipments can penetrate in these bubbles and neglect the internal surface contact, omitting the correct surface average roughness description. In the present study these readings were disregarded.

## CONCLUSION

All resin cements studied became rougher after being submitted to brushing strokes, they were statistically significant. No statistical differences were found in the roughness changes between different materials. SEM observations revealed that the generalized wear of the luting agents was minimal.

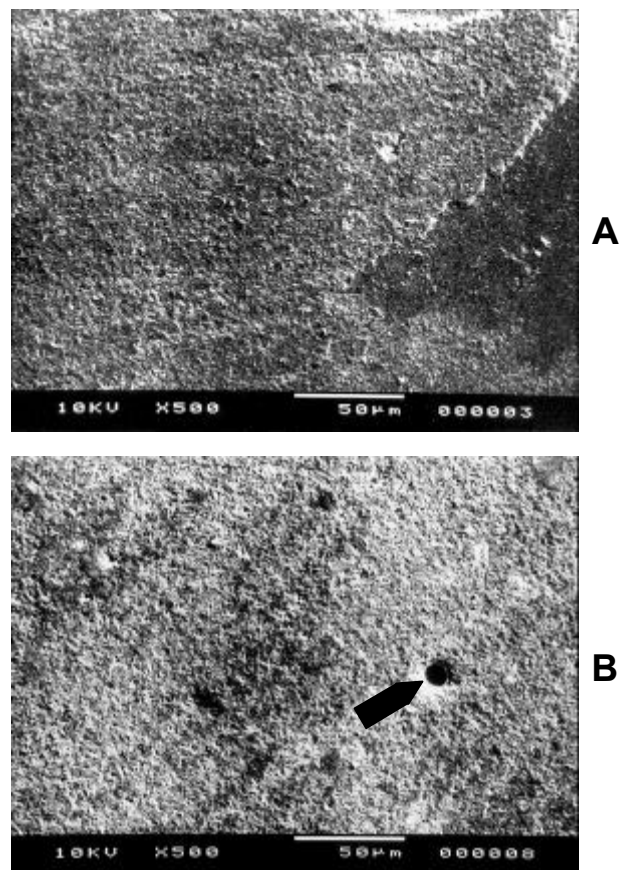
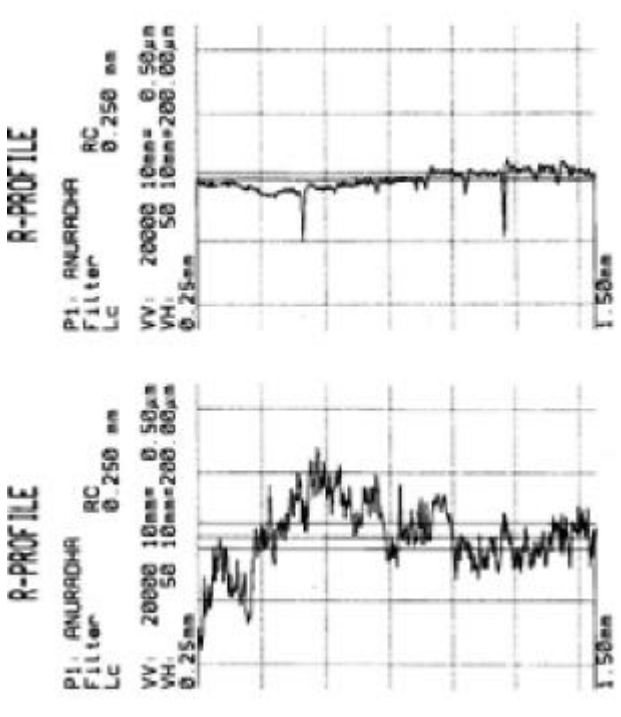


FIGURE 3- Roughness and microscopic evaluation of Variolink II (Vivadent) resin cement before (a) and after (b) brushing simulation

## RESUMO

O objetivo desse estudo foi avaliar a alteração de rugosidade superficial de três cimentos resinosos após submetê-los a ciclos de escovação simulada e analisar qualitativamente a sua superfície através de observações microscópicas. Os materiais empregados neste estudo foram Enforce Sure Cure (Dentsply), Rely X (3M-ESPE) e Variolink II (Vivadent). Estes foram submetidos a ciclos de escovação simulada (100.000 ciclos para cada espécime) e a alteração de sua rugosidade superficial (antes e após escovação) foi avaliada. Para cada material e condição de rugosidade, espécimes foram selecionados, metalizados e observados em microscopia eletrônica de varredura (DSM 900 Zeiss). Baseado nas observações microscópicas e nos valores de alteração de rugosidade aritmética (Ra) após ciclos de escovação simulada.

**UNITERMOS:** Rugosidade superficial; Cimento resinoso, desgaste; Escovação simulada.

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Correspondence for authors:

Rua São Gonçalo 6-73/81 Vila Universitária, Bauru, São Paulo ZIP: 17043-170, e-mail: praksi@yahoo.com, phone: (14) 234 2629.