# FILM THICKNESS OF RESIN CEMENTS USED WITH ADHESIVE SYSTEMS

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***ABSTRACT***

*The final film thickness of a resin adhesive and a resin cement could be affected by previous polymerization of the adhesive systems on dentin surfaces. The aim of this work was to evaluate changes in the film thickness of dual resin based cements with their adhesives as a function of polymerization of the latter on dentin surfaces.*

*The materials used were: RelyX ARC (R) + Single Bond (SB) and Variolink base (VB) and high (HV) or low (LV) viscosity catalyst + Syntac Classic (S) or Excite DSC (E); 56 human dentin discs and 56 composite resin discs (Z250). Dentin disc surfaces were treated with 35% phosphoric acid (except for S) and the adhesive system was either polymerized or not polymerized. A 0.05 ml increment of cement mixture was placed on the dentin disc and covered with the resin disc. A 25 N load was applied for ten minutes and then, the combined thickness was measured with a digital micrometer. Sample size (n) was 4 for each cement or condition. A two-way analysis of variance was performed with a level of significance of $p<0.05$.*

*The mean film thickness (and standard deviations) in µm, with and without previous polymerization of the adhesive layer, were: R+SB: 16.50 (2.64) and 17.00 (1.41); VB+S: 21.75 (5.37) and 62.25 (0.95); VB LV+S: 24.50 (3.87) and 72.75 (1.89); VB HV+S: 28.75 (8.46) and 93.00 (53.63); VB+E: 31.75 (8.38) and 42.75 (4.34); VB LV+E: 47.75(2.50) and 45.75 (3.20); VB HV+E: 49.25 (25.50) and 45.75 (2.75). Significant differences ($p<0.01$) were found for the cements and polymerization condition as well as for the interaction between them.*

*Instructions regarding polymerization of the adhesive layer must be followed when adhesive systems are used in combination with dual polymerized resin based cements. Otherwise, final film thickness of the adhesive and the resin cement could be affected.*

**Key words:** *Film thickness, consistency, resin cements, adhesive systems.*

## ESPESOR DE PELÍCULA DE CEMENTOS RESINOSOS UTILIZADOS CON SISTEMAS ADHESIVOS

***RESUMEN***

*El objetivo de este trabajo fue evaluar el espesor de película de dos cementos resinosos duales utilizados con los correspondientes sistemas adhesivos, en función de la polimerización de este último sobre la dentina.*

*Los materiales utilizados fueron: RelyX ARC (R) + Single Bond (SB) y Variolink base (VB) y catalizador de alta (HV) o baja (LV) viscosidad + Excite DSC (E). Se seleccionaron 56 discos de dentina humana y 56 discos de resina compuesta (Z250). Las superficies de los discos de dentina fueron tratados con ácido fosfórico al 35% (excepto S) y el adhesivo fue polimerizado o no. Se ubicó un volumen de 0.05 ml de material sobre el disco de dentina y se ubico un disco de composite sobre él. Se aplicó una carga de 25 N durante diez minutos y posteriormente se midió el espesor de película total con un micrómetro digital. El tamaño de la muestra fue de 4 probetas para cada situación experimental. Los datos obtenidos fueron analizados estadísticamente mediante análisis de variancia de 2 vías con un nivel de significancia de $p<0.05$.*

*Las medias aritméticas (desviación estándar) en µm, con y sin previa polimerización del sistema adhesivo, fueron: R+SB: 16.50 (2.64) y 17.00 (1.41); VB+S: 21.75 (5.37) y 62.25 (0.95); VB LV+S: 24.50 (3.87) y 72.75 (1.89); VB HV+S: 28.75 (8.46) y 93.00 (53.63); VB+E: 31.75 (8.38) y 42.75 (4.34); VB LV+E: 47.75(2.50) y 45.75 (3.20); VB HV+E: 49.25 (25.50) y 45.75 (2.75). Se encontraron diferencias estadísticamente significativas ($p<0.01$) para los cementos, la polimerización o no del adhesivo y la interacción entre ellos.*

*La polimerización o no del adhesivo debe ser considerada cuando éste se utiliza en combinación con cementos a base de resinas, de modo de no alterar el espesor de película total.*

**Palabras clave:** *Espesor de película, consistencia, cementos resinosos, adhesivos.*

## INTRODUCTION

A wide variety of dental cements are used to lute restorations such as veneers, crowns or fixed partial dentures to dental structures [1]. Conventional cements – zinc phosphate, zinc polycarboxylate and glass ionomers – are based on an acid-base reaction with formation of an ionic salt. On the other hand, there are products that are partially or completely based on polymerization reactions: hybrid ionomer cements and composite resins, respectively [2]. The

**TABLE 1. Products used in this study**

| Type of Material | Trade Name | Code | Batch Number | Manufacturer |
|---|---|---|---|---|
| Dual resin cement | RelyX ARC | R | 20011024 | 3M ESPE |
| Single-bottle adhesive system | Single Bond | SB | OEY | 3M ESPE |
| Light polymerized resin cement | Variolink II base | VB | E13253 | Ivoclar Vivadent |
| Dual resin cement | Variolink II base + low viscosity | VB LV | D17352 | Ivoclar Vivadent |
| Dual resin cement | Variolink II base + high viscosity | VB HV | D20550 | Ivoclar Vivadent |
| Multi-bottle adhesive system | Syntac | S | B47493 | Ivoclar Vivadent |
| Single-bottle adhesive system | Excite DSC | E | D60127 | Ivoclar Vivadent |
| Hybrid composite | Z250 | --------- | OGB A3 Shade | 3M ESPE |

mechanisms that allow for retention of the restoration include mechanical and molecular adhesion [2]. Clinical success is related to luting procedures as well as the intrinsic characteristics of the luting material [3]. Some type of bonding between the two structures, sealing the gap between both substrates to prevent leakage and a correct seating of the restoration, are necessary for an adequate final result [3, 4]. Consistency and film thickness are characteristics of a luting product that influence that final result [5-10].

When resin cements are used, adhesive procedures are recommended to generate an intimate bond between dental and restoration structures. These procedures are mainly used to generate micromechanical bonding. When dentin structures are involved, different types of adhesive systems can be used to generate the so-called hybrid layer and promote adhesion [11, 12]. All of these adhesive procedures can generate a layer with a finite thickness that could impair adequate seating of the restoration and influence the overall film thickness between structures [12].

The manufacturer's instructions regarding the activation or lack of activation of the adhesive layer before the definitive luting procedure intend to produce minimal modifications in that final film thickness. The objective was to evaluate the film thickness of two dual resin cements with and without previous photopolimerization of their respective adhesive systems.

## MATERIAL AND METHODS

The methodology that was established by the ANSI/ADA Specification Nº 96 for zinc phosphate cement and the ISO Specifications 9917:2003 and 4049:2000 was adapted and used [13-16]. These specifications admit a maximum film thickness of 50 µm for resin cements. Tests were conducted under standardized temperature (21ºC ± 2ºC) and humidity (60% ± 10%) conditions. The materials selected for the experiments are shown in Table 1. RelyX ARC (R) (3M ESPE) and Variolink II (V) (Ivoclar, Vivadent) were chosen on the basis of their different polymerization mechanisms and consistency. Using a consistency test based on the flow of the single material or the mix between glass plates under a 25 N load, the cements were ranked in decreasing viscosity order as follows: VB HV - VB LV- VB - R.

The base paste (VB) of Variolink II was used both alone and mixed with the low (VB LV) and the high (VB HV) viscosity pastes. The adhesive systems that are supplied by the same manufacturers, Single Bond (SB, 3M ESPE) and Syntac Classic (S) or Excite DSC (E, Ivoclar, Vivadent) were included in the experiments and were chosen on the basis of their different mechanisms of adhesion. Manipulation of the materials was carried out as indicated by the manufacturer's instructions.

Discs were obtained from a polymerized composite resin cylinder (Z250, 3M ESPE) and healthy

| TABLE 2. Arithmetic means (A.M.), standard deviations (S.D.) and marginal estimate means with 95% confidence intervals (in µm) for film thickness of resin cements and adhesive systems with (Condition A) and without (Condition B) photopolymerization | | | | | |
|---|---|---|---|---|---|
| GROUPS | ACTIVATION | A.M. | S.D. | LOWER LIMIT | UPPER LIMIT |
| R + SB | No | 17.00 | 1.41 | 0.44 | 33.56 |
| | Yes | 16.50 | 2.64 | 0.06 | 33.06 |
| VB + S | No | 21.75 | 5.37 | 5.19 | 38.31 |
| | Yes | 62.25 | 0.95 | 45.69 | 78.81 |
| VB LV + S | No | 24.50 | 3.87 | 7.94 | 41.06 |
| | Yes | 72.75 | 1.89 | 56.19 | 89.31 |
| VB HV + S | No | 28.75 | 8.46 | 12.19 | 45.31 |
| | Yes | 93.00 | 53.63 | 76.44 | 109.56 |
| VB + E | No | 42.75 | 4.34 | 26.19 | 59.31 |
| | Yes | 31.75 | 8.38 | 15.19 | 48.31 |
| VB LV + E | No | 45.75 | 3.20 | 29.19 | 62.31 |
| | Yes | 47.75 | 2.50 | 31.19 | 64.31 |
| VB HV + E | No | 45.75 | 2.75 | 29.19 | 62.31 |
| | Yes | 49.25 | 25.50 | 32.69 | 65.81 |

R: RelyX ARC, SB: Single Bond, VB: Variolink II Base Paste, S: Syntac, VB LV: Variolink II Base Paste + Low Viscosity Paste, VB HV: Variolink II Base Paste + High Viscosity Paste, E: Excite DSC.

extracted human third molars using a diamond saw (Buehler) and a hard tissue machine cutting device (MicroDisc NH-GP, DHUC Ing. Argentina) operated at. 150 rpm. The resin and dentin discs so obtained were approximately 1 cm in diameter and between 1 and 1.5 mm thickness. All discs were polished with up to 1200 grit sandpapers and the composite discs were abraded with 25 µm diameter aluminium oxide airborne-particles (Lares, Microprep, USA), at a pressure of 80 psi and cleaned with phosphoric acid, rinsed with water and air dried.
The combined thickness of dentin and composite discs pairs was recorded to the 0.1 µm with a digital micrometer (Digimatic, Mitutoyo Corporation) and recorded as Reading A. The adhesive systems were then applied on the occlusal surface of the dentin discs and they were either light activated using an appropriate device (XL 3000, 3M ESPE) (Condition A) or not activated (Condition B). A measured amount (0.05 ml) of each cement mixture was then placed on the adhesive covered dentin surface and the corresponding resin disc was placed on it. A 25 N load was applied with a hydraulic machine (CIFIC, Rosario, Argentina). Ten minutes later, the overall thickness of the discs and the luting cement was recorded (Reading B). The difference between readings A and B was considered as the final combined film thickness for the specimen that was being tested. The sample size was four (n=4) for each cement-condition combination.
Selected specimens were sectioned and covered with an Au/Pd layer (Thermo V6 Scientific, Polaron SC7620) for Scanning Electron Microscopy (XL30, Phillips, Eindhoven, The Netherlands). An image processing and analysis program (Image Tool, Version 3.0, The University of Texas Health Science Center) was used to observe the uniformity of the cement film and to compare the numerical values obtained by the visualization of the film thickness with a digital micrometer. It could be observed that the film thickness of all the cements tested was similar in both cases (Fig. 1).
A two-way analysis of variance was performed with a level of significance of $p<0.05$ to compare quantitative film thickness results.

## RESULTS

Table 2 shows arithmetic means and standard deviations (in µm) for the film thickness of resin cements and adhesive systems with and without previous photopolymerization. Table 3 shows the analysis of

| TABLE 3. Two-way analysis of variance | | | | | |
|---|---|---|---|---|---|
| **SOURCE** | **SC** | **DL** | **CM** | **F** | **P** |
| GROUPS | 8866.9 | 6 | 1477.8 | 5.48 | < 0.01 |
| ACTIVATION | 7223.1 | 1 | 7223.1 | 26.82 | < 0.01 |
| GROUPS + ACTIVATION | 9244.6 | 6 | 1540.7 | 5.72 | < 0.01 |
| Error | 1307.5 | 42 | 269.2 | | |
| **Total** | **36642.2** | **55** | | | |

variance of the results. A significant effect (p<0.01) was found for both factors (cement and adhesive and activation or lack of activation of the adhesive layer) as well as for the interaction between them. Table IV shows means film thickness values (in μm) as well their 95% confidence intervals. Significant differences (p<0.01) in film thickness were found between conditions in which the Syntac adhesive system was prepolymerized or left unpolimeryzed. The opposite was found for Single Bond and Excite DSC when these materials were prepolymerized or left unpolymerized.

## DISCUSSION

Resin cements are frequently used in dental practice to lute restorations to dental and other structures [1, 3]. Previous treatment of dental tissues and internal surfaces of rigid restoration with dentin bonding agents is necessary to enhance chemical and/or micromechanical adhesion between them [11, 12].

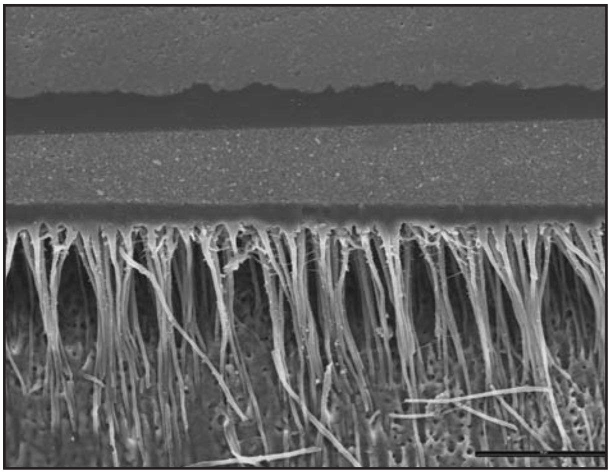

*Fig. 1: Image shows cement Variolink II base paste mixed with high viscosity paste and Excite DSC photopolymerized per manufacturer recommendation. From top to bottom: composite disc, adhesive layer, resin cement, dentin disc, and adhesive system. Total film thickness (cement and adhesive) in central portion is over 50 μm, value within range of values recorded in experimental tests. Original magnification ×530. Scale: 50 μm.*

The film thickness of the adhesive and the resin cement depend on multiple factors [1, 2, 4, 5, 7, 8-10]. Consistency is considered to be one of the principal features that affect the film thickness of resin cements. This is a time-dependent property affected by the temperature of the mix, the filler content of the cement, the composition of the resin matrix and the degree of polymerization [1, 8, 10]. Working and setting time, time elapsed after mixing, magnitude, intensity and way of application of the load and thickness of the adhesive systems are considered the main factors that affect the film thickness of resin cements [1, 3, 4, 8, 9, 10].

The low or very low consistency of most of the available commercial adhesive systems prevents them from reaching an adequate seating of the restoration. Current adhesive systems, total-etch and self-etching systems were used in this study. According to the manufacturer's indications, Single Bond and Excite DSC (filled type bonding system / total-etching adhesives) have to be applied on previously conditioned dental surfaces and should be photopolymerized. On the other hand, the Syntac system works optionally with partial (self-etching adhesive system) or complete removal of the smear layer (acid conditioning followed by primer) and should be applied without photoactivation. The purpose of these different indications is to avoid alterations of the final film thickness and ensure the seating and consequently, the fit of the restoration.

The results obtained in this study showed that previous polymerization of the adhesive layer did not affect the film thickness of Single Bond and Excite DSC. This could be due to the fact that these are low viscosity adhesives and for Excite DSC the filler does not seem to produce enough increase in viscosity to affect the final film thickness. In contrast, when a multi-bottle system like Syntac Classic was light activated before using the cement, a larger and unfavorable film thickness was generated, i.e. greater than the 50 µm ISO specification permits. Syntac Classic could affect final film thickness as shown by the lack of overlap of the confidence intervals, confirming that the manufacturer's indications must be followed to obtain an adequate final film thickness.

The higher dispersion in the results with Variolink II base with the high viscosity paste and Syntac Classic would be due to the fact that the high viscosity paste is more difficult to manipulate than the other pastes provided by the manufacturer and because the adhesive system consists of three chemicals with different consistencies that should be placed on the dentin substrate sequentially.

It can be concluded that instructions regarding polymerization of the adhesive layer must be followed when adhesive systems are used in combination with dual polymerized resin based cements. Otherwise, film thickness of the adhesive and the resin cement could be affected.

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