Evaluation of Resin-Resin Interface in Direct Composite Restoration Repair

S Stoleriu*, S Andrian, G Pancu, I Nica and G Iovan
Odontology, Periodontology and Fixed Prosthodontics Department, “Grigore T. Popa” University of Medicine and Pharmacy, Faculty of Dental Medicine, Iași, Romania

E-mail: stoleriu_simona@yahoo.com

Abstract. The aim of this study was to evaluate the resin-resin interface when a universal bonding agent was used in two different strategies in direct restoration repair. Two composite resins (a micro-filled hybrid and a nano-filled hybrid) as old restorations that have to be repair, a universal bonding agent and a micro-filled hybrid composite resin (different then that aged) as new material for repair were chosen for the study. Non-aged samples were used as control and aged samples were used as study groups. The universal bonding agent was applied in etch-and-rinse and in self-etch strategies. The interface between old and new composite resins was evaluated by SEM and the microleakage was assessed by scoring the dye penetration. Very good adaptation of the two different composite resins placed in direct contact in non-aged samples was recorded. No gaps or defects were visible and strong resin-resin contact was observed. After aging, enlargement of resin-resin junction were observed in most of the samples and a increased dye penetration was recorded irrespective of the strategy (etch-and-rinse or self-etch) used for bonding agent application.

1. Introduction
Since their use in dentistry in early 60s, composite resins have been the most used material for final direct restorations due to their continuous improvement of esthetic quality, mechanical properties and ability to bond to dental tissues mediated by adhesive systems [1]. Unfortunately, in oral environment, composite resins are prone to deterioration and degradation [2,3]. For many years the treatment of failed restorations consisted in complete removal of fillings. This approach leaded to excessive removal of sound enamel and dentine due to the practitioner’s need to „do something”, while trying to discriminate between the perfect matched composite resin and the dental tissues and providing freshly new cut enamel for bonding the new material [4,5]. The repetitive cycle of replacing old restorative materials would lead to larger cavities [6-8], increased risk to harm the pulp during the removal of the filling and increased cost of the procedure.

The minimally invasive concept was introduced in dentistry in early 70s with the major goal to preserve as much as possible the tooth structures. This approach focuses on early diagnosis of caries disease, remineralization of incipient lesions and reduction of cariogenic bacteria, minimum preparation in cavitated caries lesions, and repair rather than replacement of defective restorations [9-13]. Composite repair is considered a conservative procedure, which is increasingly accepted and practiced [14,15]. It is estimated nowadays that 50 to 71 percent of the dentist activity is represented by the repair of existing restorations [10]. Repair is indicated in fillings fracture [16], material discoloration or worn areas [17], poor anatomic form, secondary caries, tooth fracture and tooth
pain/sensitivity. This procedure consists in adding a fresh new layer of material in contact with an old restorative material from which a part had been removed by fracture, by wear or by the practitioner. The success of the repair relies on interfacial coupling and long-term retention between the surfaces involved in repair.

Factors like composite surface contamination [18], polishing [5] and ageing [19] are responsible for impairing of direct bonding. Chemical compatibility (related to organic matrix and polymerisation method) between the two composite resins could also affect bonding, since the brand of pre-existing composite resins is often unknown [20,21]. The age of the old restoration and the elapsed time since the repair are also important factors that can affect the bonding [22]. In oral environment, the bond between old and new composite resins is prone to degradation promoted by the direct contact with moisture and by salivary enzymatic activity [22].

Different methods for surface treatment and various intermediary agents were tested in previous studies in order to obtain an ideal adhesion between the two different materials used in the repair procedure [17,23,24]. It was demonstrated that an intermediate adhesive agent increases the repair bond significantly due to surface wetting and chemical bond with the new composite [25-29]. All dental adhesive systems have three different components: etching agent, primer and bonding agent. Clinically, acid-etching might be applied in a separate step (etch-and-rinse technique) or it might be provided by acidic functional monomers (self-etch technique). The most recent novelty in dental adhesion was the introduction of universal or multi-mode bonding agents. Often these materials consist in a mixture of all three components in the same bottle. According to the manufacturer, they can be applied either in etch-and-rinse as in self-etch procedure.

Clinically, good bond strength is reflected by the interface ability to prevent the microleakage [30]. The leakage is responsible for bond degradation, marginal staining of the restoration, biofilm accumulation and the risk of caries lesions onset. Previous studies that investigated microleakage of the composite repair interface showed differences between the adhesive systems investigated, but no data are available regarding universal bonding agents [31].

The aim of this study was to evaluate the resin-resin interface when a universal bonding agent was used in two different strategies in direct restoration repair.

2. Experimental procedures

2.1. Obtaining the composite resin specimens

Two different composite resins were chosen for this study: a micro-filled hybrid (Filtek Z250, 3M ESPE-MH1) and a nano-filled hybrid (Premise, Kerr NH). Forty quadrangular specimens of each material were obtained by placing unpolymerized composite resin into molds having 5 mm in length, 5 mm in width and 2 mm in high. One layer of 2 mm was placed using a buccal spatula and a condenser, and was polymerized for 40 seconds using a LED light unit (LED B, Guilin Woodpecker Medical Instrument Co., Ltd, China) having the light intensity of 850-1000mW/cm² and the wavelength of 420-480 nm. Before the last increment polymerization, a translucent Mylar strip and a 2mm thick glass slab will be place on top of the surface layer in order to obtain a flat, smooth surface and to prevent the formation of the oxygen inhibited layer. Twenty specimens of each material were used to obtain control samples and twenty specimens were aged by storing in citric acid for one week.

2.2. Composite resin repair and samples preparation

One composite resin (Z-mack, Zermack- MH2) was chosen to be place in contact with the non-aged and aged composite resins as a repair material. For bonding the micro-filled hybrid resin to aged resins, an universal bonding agent (G Premio Bond, GC Corporation- UBA) was used in two different strategies: etch-and-rinse (strategy 1), and self-etch (strategy 2). The layout of the groups is presented in table 1.
Table 1. The layout of the groups.

|       | non-aged MH1 | non-aged NH | aged MH1 | aged NH | UBA strategy 1 | UBA strategy 2 | MH2 |
|-------|--------------|-------------|----------|---------|----------------|----------------|-----|
| Group 1 | X            | X           |          |         |                |                | X   |
| Group 2 | X            |             | X        |         |                |                | X   |
| Group 3 |              | X           |          |         |                |                | X   |
| Group 4 |              | X           |          |         |                |                | X   |
| Group 5 |              | X           | X        |         |                |                | X   |
| Group 6 |              | X           |          |         |                |                | X   |
| Group 7 |              | X           | X        |         |                |                |     |
| Group 8 | X            |             | X        |         |                |                |     |

In strategy 1, 35% phosphoric acid etchant gel (3M-ESPE, St. Paul, MN, USA) was applied for 15 seconds on one of 2mmx5mm surface of the resin specimens using the tip of the syringe, then the acid was removed using the water spray from the dental unit and gently dry using the air spray. For bonding procedure, the application of UBA was made according to the producer instructions. Using disposable applicator brushes, the universal adhesive agents was applied by scrubbing on the resin surface for 20 second, gently air dry for 5 seconds using medium air pressure and then lightcured for 20 seconds.

In order to obtain the samples, the repair composite was applied in contact with non-aged or aged specimens. For that non-aged and aged composite specimens were reassembled in 10 mm length, 5 mm width and 2 mm high molds, with the surface where the universal adhesive agent was applied toward the middle of the mold. MH2 was inserted in one increment of 2mm, using the same procedure as for MH1 and NH. Different shade was chosen for repair material for a better assessment of the repair interface. The samples were then removed from the molds and stored in distilled water for 24 hours.

2.3. Microleakage assessment by dye penetration and SEM evaluation of the samples interface

The samples from each group were used for evaluation of adhesive efficiency by microleakage assessment and for SEM evaluation of the interface. All the extern surfaces of the samples will be coated with two layers of water resistant nail varnish, except the 10mm x5mm surface that was finished. The samples were immersed in 2% methylene blue buffered dye solution (pH=7) for 4 hours (Cavalcanti, 2004).

After that the samples were transversely sectioned using diamond discs (Komet Dental, Brasseler GmbH&Co, Germany), under watercooling. The sections were examined using a optical microscope (Carl-Zeiss AXIO Imager A1m) coupled with a high resolution digital camera at 50x magnification. The dye penetration was assessed according to the following scores: 0- no dye penetration, 1- dye penetration on less then a half of the interface, 2- dye penetration on more then a half of the interface, but less then whole interface, 3- complete dye penetration of the interface.

A scanning electron microscope (VEGA II LSH, Czech Republic) was used to analyze the aspect of the repair interface. The morphology of UB layer in groups 1-8, and the microgaps formation between UB and MH1, NH, and MH2 were performed at x100, x300, and x1000 magnification.

3. Results and discussions

Some SEM aspects of resin-resin interface in groups 1-8 are presented in figure 1. Very good adaptation of the two different composite resins placed in direct contact in all control groups was recorded. No gaps or defects were visible and strong resin-resin contact was observed. In groups 4-8, enlargement of resin-resin junction were observed in most of the samples.
### Table 2. Dye penetration scores in control and study groups.

| No of samples | In groups | score 0 | score 1 | score 2 | score 3 | score 4 | Mean score |
|---------------|-----------|---------|---------|---------|---------|---------|------------|
| Group 1       | 10        | 10      | -       | -       | -       | -       | 0          |
| Group 2       | 10        | 10      | -       | -       | -       | -       | 0          |
| Group 3       | 10        | 10      | -       | -       | -       | -       | 0          |
| Group 4       | 10        | 10      | -       | -       | -       | -       | 0          |
| Group 5       | 10        | 1       | 6       | 1       | 2       | -       | 1.4        |
| Group 6       | 10        | 1       | 7       | 1       | 1       | -       | 1.2        |
| Group 7       | 10        | 1       | 6       | 2       | -       | 1       | 1.4        |
| Group 8       | 10        | 1       | 7       | 1       | 1       | -       | 1.2        |

In figure 1 some samples scored with 1 according to dye penetration are exemplified (groups 5 and 8). The dye penetration scores in all eight groups are presented in table 2. In groups 1-4 there was no sample scored more than 0 for dye penetration. In groups 5-8, increased values of scores were registered when compared to control.
It was demonstrated that microleakage evaluation is a suitable method to assess the performance of adhesive systems especially when few clinical data are available [31-35]. In order to obtain the composite resins aging, different storage media and techniques were used in previous studies: immersion in artificial saliva, saline solution, deionized water, water [36], citric acid [37] or thermocycling. The idea of using citric acid for aging was to mimic the action of acidic beverages or food. Studies that analysed the effect of different storage media showed similar effects on filler exposure when using citric acid, water storage [5] and termocycling. Despite termocycling and water storage, citric acid leads to resin matrix degradation [28] and a lesser surface roughness. The increased surface roughness after immersion in water might be explained by swelling instead of matrix degradation [28]. Resin-to-resin bonding after aging was evaluated in previous studies [38].

In composite resin repair a big challenge is represented by the adhesion between a new composite resin and a pre-existing one, which is unknown most of the time. Generally, when placing two composite layers in contact, the adhesion is obtained through the oxygen-inhibited un-polymerized resin layer [39]. Aged composite resins have lesser or no un-reacted monomers, so poor adhesion is expected at the interface with the newer resin. In our study increased values of dye penetration were recorded at resin-resin interface after both composite resins aging as a result of the less efficient adhesion.

Previous investigations showed that microleakage at the resin-resin interface is not correlated to the type of pre-existing composite resin [31]. Almost similar dye penetration was recorded in present study when nano-filled hybrid and micro-filled hybrid were used as pre-existing materials.

In order to obtain a good composite repair procedure, micromechanical adhesion is desirable. For that, increasing the surface area of the pre-existing resin or application of an intermediate layer (like bonding agent) was recommended [5, 40, 41]. For self-etch bonding systems, the acidic primer seems to provide a clean surface [42] and for etch and rinse bonding systems phosphoric acid is responsible for this action. When phosphoric acid was used in etch and rinse bonding systems, it was demonstrated that no significantly effect on improving the bonding in composite repair procedure [29, 43]. Previous studies demonstrated that both etch and rinse and two-step self-etching systems lead to similar results in composite repair. Universal adhesives or multi-mode adhesives contain all the bonding components in one bottle and they can be applied either in etch-and-rinse or self-etch approaches. In our study the universal bonding agent applied in both strategies determined comparable results when used as intermediate layer in composite repair.

Still conflicting results were reported when evaluating the multiple variables involved in bonding at the resin-to-resin interface. The literature reveals a lack of common opinion regarding the behaviour of different bonding agents used in direct composite resins repair and very little information regarding the efficiency of universal bonding agents. The few studies that have been published aimed only to evaluate the bond of universal adhesive agents to dental hard tissues. In our study the use of universal adhesive system in both strategies for composite repair conducted to similar result regarding the

Figure 1. Aspects of dye penetration and SEM aspect of the interface in control and study groups.
adhesion efficiency. The results of our study are preliminary, future studies being necessary to investigate more the bonding capacity of universal adhesive agents used in composite resin repair.

4. Conclusions

Very good adaptation of micro-filled hybrid composite resin used to repair the non-aged micro-filled hybrid and non-aged nano-filled hybrid composite resin was recorded. The direct contact between both non-aged composite resin and the new composite resin when using universal bonding agent as intermediate layer did not lead to gaps formation or visible defects. After aging, enlargement of resin-resin junction and a increased microleakage was recorded, irrespective of the strategy (etch-and-rinse or self-etch) used for bonding agent application.

References

[1]. Roulet J F, Roulet J F, Wilson N H F, and Fuzzi M 2001 Advances in Operative Dentistry. vol 1, Quintessence Publishing Co Inc
[2]. Soderholm K-J, Zigan M, Ragan M, Fischlschweiger W, and Bergman M. 1984 J. Dent. Res. 63 1248–1254
[3]. Finer Y, and Santerre J P 2004 J. Dent. Res. 83: 22-26
[4]. Söderholm K-J M, and Roberts MJ 1991 Scand. J. Dent. Res. 99 2 173- 80
[5]. Shahdad S A, and Kennedy J G 1998 J. Dent. 26 8 685-94
[6]. Krejci I, Lieber C M, and Lutz F 1995 Dent. Mater. 11 34-40
[7]. Gordan V V 2000 J. Esthet. Dent. 12 209-215
[8]. Gordan V V, Mondragon E, and Shen C 2002 Quintessence Int. 33 273-278
[9]. Murdoch-Kinch C A, and McLean M E 2003 J. Am. Dent. Ass. 134 87-95
[10]. Tyas M J, Anusavice K J, Frencken J E, and Mount G J 2000 Int. Dent. J. 50 1 1-12
[11]. Mjör I A, and Gordan V V 2002 Oper. Dent. 27 528-534
[12]. Staehle H J. Minimally invasive restorative treatment. J Adhes Dent 1999; 1: 267-284
[13]. Chalmers J M 2006 J. Can. Dent. Ass. 72 435-440
[14]. Gordan V V, Mjör I A, Blum I R, and Wilson N 2003 J. Am. Dent. Ass. 134 317-323
[15]. Blum I R, Schriever A, Heidemann D, Mjör I A, and Wilson N H F 2003 Eur. J. Dent. Educ. 7 41-48
[16]. Chalkley Y, and Chan D C N 1986 J. Prostheth. Dent. 56 4 441-444
[17]. Brosh, T, Piló R, Bichacho N, and Blutstein R 1997 J. Prostheth. Dent. 77 2 122-126
[18]. Eriiksonn S O, Pereira P N R, Swift E J Jr, Heymann H O, Sigurdsson A 2004 Dent. Mater. 20 37-44
[19]. Mitsaki-Matsou H, Karanika-Kouma A, Papadoyiannis Y, and Theodoridou-Pahine S 1991 Quintessence Int. 22 475-481
[20]. Kao E C, Pryor H G, and Johnston W M 1988 J. Prostheth. Dent. 60 328-333
[21]. Gregory W A, Pounder B, and Bakus E 1990 J. Prostheth. Dent. 64 664-668
[22]. Finer Y, and Santerre J P 2004 J. Dent. Res. 2004 83 22-26
[23]. Lucena-Martin C, González-López S, and Navajas-Rodrigues de Mondelo J M 2001 J. Prostheth. Dent. 86 5 481-8
[24]. Öztas N, Alaçan A, and Bardakcy Y 2003 Oper. Dent. 28 2 149-54
[25]. Padipatvuthikul P, and Mair I 2007 Dent. Mater. J. 23 4 519-525
[26]. Yesilyurt C, Kusgoz A, Bayram M, and Ulker M 2009 J. Esthet. Rest. Dent. 21 4 251-260
[27]. Junior S, Ferracane J, and Bona A 2009 Dent. Mater. J. 2009 25 4 442-451
[28]. Rinastiti M, Özcan M, Siswomihardjo W, and Busscher H 2010 J. Dent. 38 1 29-38
[29]. Papacchini F, DallOca S, Chieffi N, Goracci C, Sadek F T, Suh B I, Tay F R, and Ferrari M 2007 J. Adhes. Dent. 2007 9 25–31
[30]. Hadavi F, Hey J H, Ambrose E R, and Elbhadrawy H E 1993 Oper. Dent. 18 1 2-7
[31]. Celik C, Cehreli SB, and Arhun N. 2015 Eur. J. Dent. 2015; 9 1 92–99
[32]. Aschenbrenner C M, Lang R, Handel G, and Behr M. 2012 Clin. Oral Investig. 16 191–200
[33]. Bagis Y H, Baltacioglu I H, and Kahyaogullari S 2009 Oper. Dent. 34 578–85
[34]. Iovan G, Stoleriu S, Pancu G, Topoliceanu C, Ciobanu M C, and Andrian S 2014 Romanian Journal of Oral Rehabilitation 6 2 18-23
[35]. Iovan G, Stoleriu S, Andrian S, Dia V, Căruntu I D 2004 Rev Med Chir Soc Med Nat Iaşi 108 4 894-898
[36]. Yap A U J, Sau C W, Lye K W 1999 Oper. Dent. 24 371–376
[37]. Gregory W A, Pounder B, and Bakus E 1990 J. Prosthet. Dent. 64 664-668
[38]. Rasmussen S 1996 Int J Adhesion and Adhesives 16 147–154
[39]. Ozcan M, Kojima A N, Pekkan G, Mesquita A M, and Bottino M A 2014, Int. J. Adh. Adhes. 49 97–102
[40]. Costa T R, Ferreira S Q, Klein-Júnior C A, Loguercio A D, and Reis A 2010 Oper. Dent. 35 231–7
[41]. Cehreli S B, Arhun N, and Celik C. 2010 Oper. Dent 35 337–44
[42]. Cavalcanti A N, De Lima A F, Peris A R, Mitsui F H, and Marchi G M 2007 J. Esthet. Restor. Dent. 19 90–8
[43]. Hannig C, Laubach S, Hahn P, and Attin T. 2006 J. Adhes. Dent. 8 35–40