Evaluation of Adhesive Capacity of Universal Bonding Agents Used in Direct Composite Resins Repair

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The aims of the study were to characterise the resin-resin interface when a universal bonding agent (UBA) was used in two different strategies in direct composite repair and to evaluate the bonding capacity of UBA by microleakage assessment. In study groups a micro-filled hybrid and a nano-filled hybrid composite resins were aged in order to simulate an old restorations. As a repair material was chosen the same micro-filled hybrid composite resin that was used as an old restoration. UBA was applied in etch-and-rinse and self-etch strategies and was used as an intermediate layer in repair procedure. After the repair the samples were aged again. In control groups were included non-aged, repaired composite resins samples. The resin-resin interface was characterised on SEM images and the microleakage at the interface was evaluated by dye penetration assessment. Universal bonding agent used in direct composite resins repair showed a very good adaptation to non-aged micro-filled hybrid and nano-filled hybrid composite resins. Aging by saliva storage of repaired composite resins leaded to an enlargement of resin-resin junction and a increased microleakage irrespective of the strategy (etch-and-rinse or self -etch) used for bonding agent application. Etch-and-rinse strategy for universal bonging agent application determined a better interface bonding when compared to self –etch strategy.

Keywords: universal bonding agent, direct composite resins, repair, microleakage, SEM

Oral cavity is a complex environment, were restorative materials are exposed to various challenges and prone to degradation. Erosion, abrasion, termal variation, acidic attacks, salivary enzymes and hydrolysis are some of the factors implicated in composite resin failure [1, 2]. Clinical studies claimed that the composite resin failure varied between 5 and 45% over a period of 5–17 years [1, 3]. Generally, two different procedures are used in dental office for failed restoration treatment: repair or replacement. Unfortunately total replacement of the restoration lead most of the time to excessive removal of sound enamel and dentine, bigger cavity preparation, pulp harm and weaker remaining tooth structure [4]. Partial replacement of the restoration or the repair has the advantages of being minimally invasive [5, 6], less time consuming [7] and increasing the longevity of the restoration [8-11]. The bond between the existing composite resin and the new one used for repair represent a major issue. It was generally considered that the bond relies on micromechanical retention, so different techniques were used during time for surface treatment, as air abrasion or abrasive diamond bur use [12] in order to increase the surface irregularities, the surface roughness and total surface area [13]. In addition, phosphoric acid was used to remove the debris and the smear layer [14-20] and hydrofluoric acid was used to increase the surface area by dissolving the silica fillers [12, 21-23]. It was stated and now it is generally accepted that an intermediate layer, represented often by adhesive system, is mandatory to obtain a good adhesion between composite resins layers [24]. The significantly increased repair bond when using an intermediate adhesive agent relies on improving the surface wetting and chemical bond with the new material used in repair procedure [25-27].

Universal or multi-mode bonding agents are the newest type of dental adhesives which consist in a mixture of etching agent, primer and bonding agent in the same bottle. The producers claimed that they can be applied either in etch-and-rinse and in self-etch procedure. One our previous study aimed to evaluate the interface between composite resins used in a repair procedure when a universal bonding agent was used as intermediate layer [28]. The results showed a very good immediate adaptation of composite resin used as repair material to non-aged composite resins. Also, an enlargement of resins junction and an increased microleakage were recorded after aging the composite resins, irrespective of etch-and-rinse or self-etch strategy of application. The limitations of that study represented by the short time aging of composite resins in citric acid and by the absence of aging of repaired composite resins, together with the lack of information regarding the long term behaviour of universal adhesive agents used in composite repair determine us improve the research on this topic.

The aims of present study were to characterise the resin-resin interface when a universal bonding agent was used in two different strategies in direct composite repair and to evaluate the bonding interface by microleakage assessment.

Experimental part

Composite resin specimen preparation and aging

Two different composite resins were used in this study: a micro-filled hybrid (Zmack, Zhermack Sp.A, Germany) (MH) and a nano-filled hybrid (Premise, Kerr Co) (NH). Data regarding the chemical characteristics (matrix type, filler type and filler content) are presented in table 1. From each

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material forty specimens were obtained by placing the composite resin into the moulds having an intern diameter of 5 mm and a height of 6 mm. The materials were applied in two layers using incremental technique. Each layer of 1.5 mm has being polymerized for 40 seconds using a LED unit (LED B, Guilin Woodpwcker Medical Instrument Co., Ltd, China). The moulds were placed in direct contact with a translucent Mylar strip, on a glass slab to flatten the surface and to prevent the formation of the oxygen inhibited layer. Half specimens of each material were used to obtain control samples and the other half was aged by storing in artificial saliva (AFNOR NF S90-701) for four months.

Simulation of repair procedure and preparation of the samples

In order to simulate the repair procedure, the same micro-filled hybrid composite resin used for specimens preparation (Z-mack, Zermack Sp.A.) was placed in direct contact with the non-aged and aged NH and MH composite resin specimens. As an intermediate layer, a universal bonding agent (G Premio Bond, GC Corporation) (UBA) was applied between the composite resins used in dental repair in two different strategies: etch-and-rinse (strategy 1), and self-etch (strategy 2). The protocol of samples distribution in groups and groups setup are listed in table 2

In etch-and-rinse strategy, the surface of the resin specimens were etched using 35% phosphoric acid gel (3M-ESPE, St. Paul, MN, USA) for 30 s, then removed with water and gently dry using the dental unit air spray. The application of UBA followed the producer instructions: scrubbing the sample surface for 20 s, gently air drying for 5 seconds and lightcure for 20 s. In self-etch strategy the same steps were followed, except the etchant application.

The repair composite was applied in two increments of 1.5 mm each. In order to identify the two different materials used in restoration repair, different shades were chosen for composite resins.

The samples considered as control (groups 1, 2, 5, 6) were removed from the moulds. The samples included in the study groups (3, 4, 7, 8) were immersed in artificial saliva for 2 months and then removed from the moulds.

Microleakage evaluation by dye penetration and interface characterisation using SEM evaluation

The extern surfaces of the samples were coated with two layers of water resistant nail varnish, except a 10 mm x5 mm surface. The samples were immersed in 2% methylene blue buffered dye solution (*p*H=7) for 4 hours [29]. Then the samples were transversely sectioned using diamond discs (Komet Dental, Brasseler GmbH&Co, Germany) at low speed, under cooling water. The sample sections were examined using an optical microscope (Carl-Zeiss AXIO Imager A1m) at 50x magnification. The dye penetration was evaluated according to four scores: 0 - no dye penetration, 1 - dye penetration less than a half of the interface, 2 - dye penetration more than a half of the interface, but less then whole interface, 3 - complete dye penetration of the interface. The characterisation of the repaired composite resins interface was performed on the images recorded using a scanning electron microscope (VEGA II LSH, Czech Republic). The morphology of UBA layer and the micro-gaps formation between UBA and composite resins were evaluated at 500X magnification.

Results and discussions

SEM aspects of some samples included in groups 1-8 are showed in figure 1. In groups 1, 2, 5 and 6 a very tight adaptation of the two composite resins placed in contact was observed. There were no gaps or defects at resin-resin interface. In groups 3, 4, 7 and 8 a slightly enlargement of the resin-resin junction was recorded and in some samples little gaps and defects were visible (fig.1-groups 3, 7).

Optical images of the resin-resin junction and examples of dye penetration scored as 0 and 1 in groups1-8 are presented in figure 2. The scores for dye penetration recorded for the samples in all the groups and the mean score values are presented in table 3. In groups 1, 2, 5 and

Product	Filler type	Filler content in weight	Filler content in volume	Matrix type
Z-mack	Barium glass, silica	77%	57%	Bis-GMA, Bis-EMA TEGDMA
Premise	Barium alumino borosilicate glass, silica, PPF, barium glass	84%	69%	Bis-GMA Bis-EMA TEGDMA

Table 1
CHEMICAL COMPOSITION AND CHARACTERISTICS OF COMPOSITE RESINS

Group	Initial composite resin				UBA		Composite resin for repair
	non-aged MH	aged MH	non-aged NH	aged NH	strategy 1	strategy 2	MH
1	X					X-	X
2	X				X-		X
3		X			-	X-	X
4		X			X		X
5			X			X	X
6			X		X		X
7				X		X	X
8				X	X		X

Table 2
CONTROL AND STUDY GROUPS SETUP

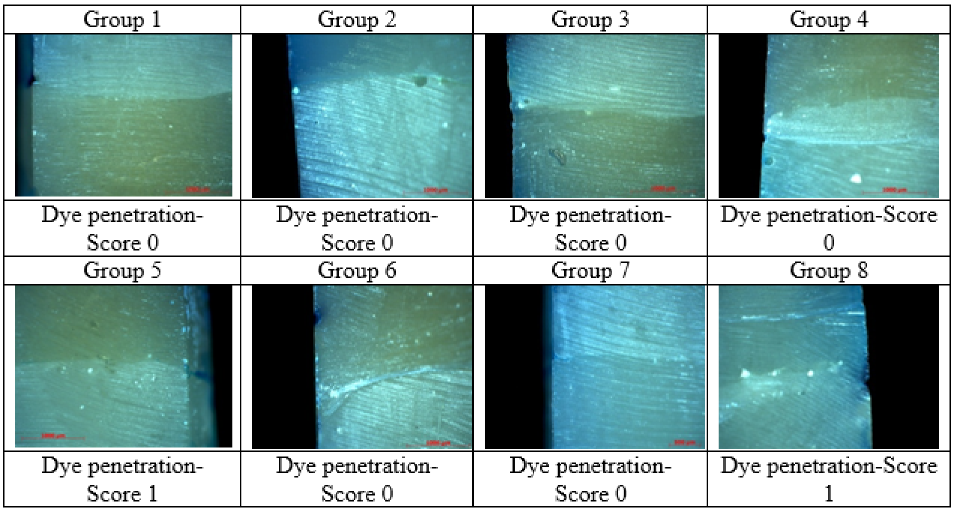

Fig. 1. Cross sections of the adhesive joint created using the universal bonding system

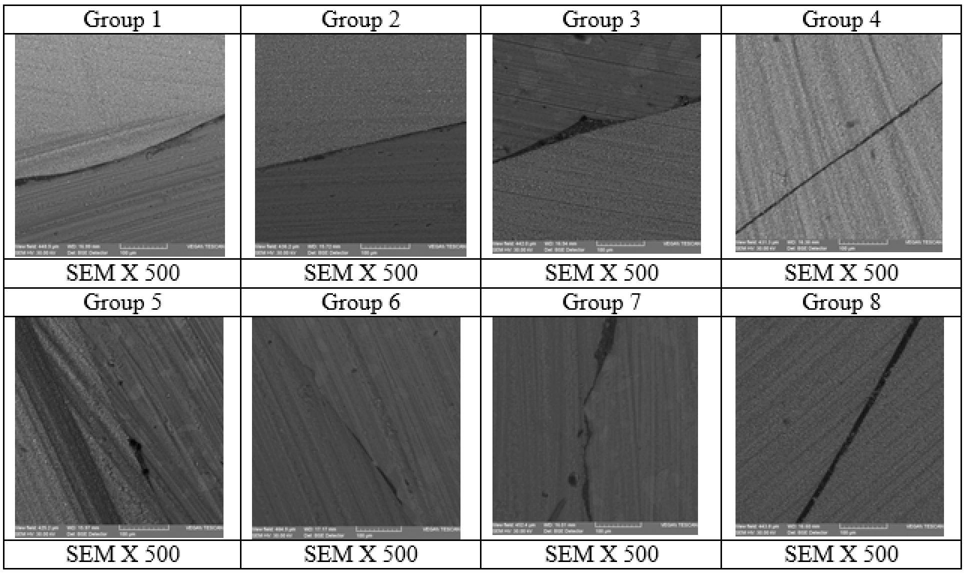

Fig. 2. Aspects of dye penetration at the resin-resin interface in control and study groups

Table 3
DYE PENETRATION SCORES IN CONTROL AND STUDY GROUPS

Group	No of samples					Mean score value
	score 0	score 1	score 2	score 3	score 4	
1	10	-	-	-	-	0
2	10	-	-	-	-	0
3	2	4	3-	1	-	1.3
4	1	5	3	1	-	1.4
5	10	-	-	-	-	0
6	10	-	-	-	-	0
7	1	4	4	1	-	1.5
8	1	3	4	2	-	1.7

6 all the samples were scored as 0 for dye penetration. Higher scores were registered in groups 3, 4, 7 and 8 when compare to 1, 2, 5 and 6. The lower mean value of dye penetration was recorded in group 3 and the highest in group 8.

The degradation of composite resins in oral cavity might be due to fatigue, enzymatic activity, chemical agents [30], termal variation, wear, hydrolytic action [31]. Restoration failure is often a result of the degradation process which leads to microleakage, discoloration, marginal ditching, delamination or fracture. In case of failed restoration there are two different clinical approaches: repair or replacement. In repair procedure a freshly new material is placed in direct contact with an old material, aged and mostly unknown. In our study using the same material for repair as it was applied before leaded to better results regarding the microleakage when compared to the use of dissimilar composite resins in repair procedure.

Generally, the adhesion between two composite layers is obtained by oxygen-inhibited layer of unpolymerized resin due to covalent bonds formation [32,33]. After polymerization, 40–50% of the unreactive methacrylate groups is present and lead to the adhesion of a new composite layer. In time the percent of these groups decrease and the bonding potential become lower [34]. This can be the explanation the lower microleakage registered in control groups when compare to study groups.

Different methods of surface treatment were recommended during time in order to improve the resin-resin adhesion. For both similar and dissimilar combination of composite resins, using universal adhesive system in etch-and-rinse strategy showed lower microleakage when compared to self-etch strategy. This finding is in contrast with the results of previous studies which demonstrated that the use of phosphoric acid in etch-and-rinse bonding systems, did not leaded to significantly improved effect on repair bonding [13, 35].

In the present study saliva storage was chosen as aging regimen. Water storage simulates aging by water uptake and the level of water-sorbtion of composite resins is correlated to matrix type [36]. After a specific point it is expected the saturation to appear. In our study both types of composite resins that were aged presented increased microleakage at the interface with the resin used for repair when compared to non-aged specimens.

Conclusions

Universal bonding agent used in direct composite resins repair showed a very good adaptation to non-aged micro-filled hybrid and nano-filled hybrid composite resins. Aging by saliva storage of repaired composite resins leaded to an enlargement of resin-resin junction and a increased microleakage irrespective of the strategy (etch-and-rinse or self -etch) used for bonding agent application. Etch-and-rinse strategy for universal bonging agent application determined a better interface bonding when compared to self –etch strategy.

References

1.DA ROSA RODOLPHO, P.A., CENCI, M.S., DONASSOLLO, T.A., LOGUERCIO, A.D., DEMARCO, F.F., J. Dent. **34**, 2001, p. 427.

2.ROULET, J.F. Degradation of dental polymers. Basel: Karger, 1987, p. 40.

3.HICKEL, R., MANHART, J., J. Adhes. Dent., **3**, 2001, p. 45.

4.MJØR, I.A., Oper. Dent., **6,** 1981, p. 49.

5.GORDAN, V.V., SHEN, C., RILEY, J. 3rd, MJOR, I.A., J. Esthet. Restor. Dent., **18**, 2006, p. 144.

6.MONCADA, G., MARTIN, J., FERNANDEZ, E., HEMPEL, M.C., MJOR, I.A., et al., J. Am. Dent. Assoc., **140,** 2009, p. 425.

7.KREJCI, I., LIEBER, C.M., LUTZ, F., Dent. Mater., **11,**,1995, p. 34.

8.FERNANDEZ, E., MARTIN, J., VILDOSOLA, P., OLIVEIRA JR. O.B., GORDAN, V., et al., J. Dent., **43,** 2015, p. 279.

9.TANCULESCU, O., DOLOCA, A., VIERIU, R.M., MOCANU, F., IFTENI, G., VITALARIU, A., SOLOMON, S., IOANID, N., IOVAN, G., Rev. Chim. (Bucharest), **68,** no. 1, 2017, p. 94

10.GAVRILA, L., BALAN, A., MURARIU, A., SANDU, A.V., SAVIN, C., Rev. Chim. (Bucharest), **67,** no. 11, 2016, p. 2228

11. MURARIU, A., SAVIN, C., FEIER, R., BALAN, A., Rev. Chim. (Bucharest), **67,** no. 9, 2016, p. 1876

12.CELIK, E.U., ERGUCU, Z., TURKUN, L.S., ERCAN, U.K., J. Adhes. Dent., **13**, 2011, p. 359.

13.HANNIG, C., LAUBACH, S., HAHN, P., ATTIN, T., J. Adhes. Dent., **8**, 2006, p. 35.

14.MURARIU, A., ZALTARIOV, M., VASILIU, L., BALAN, A., SAVIN, C., GAVRILA, L.M., FORNA, N.C., Rev. Chim. (Bucharest), **68,** no. 4, 2017, p. 781

15.BALAN, A., STOLERIU, S., ANDRIAN, S., SANDU, A.V., IOVAN, G., SERBAN-PINTILICIUC, V., Rev. Chim. (Bucharest), **66,** no. 2, 2015, p. 182

16.BALAN, A., ANDRIAN, S., SAVIN, C., SANDU, A.V., PETCU, A., STOLERIU, S., Rev. Chim. (Bucharest), **66,** no. 4, 2015, p. 562

17.BALAN, A., STOLERIU, S., ANDRIAN, S., SANDU, A.V., SAVIN, C., Rev. Chim. (Bucharest), **66,** no. 1, 2015, p. 70

18.TANTBIROJN, D., FERNANDO, C., VERSLUIS, A., Oper. Dent.,**40**, no. 4, 2015, p. 364.

19.SPYROU, M., KOLINIOTOU-KOUMPIA, E., KOUROS, P., KOULAOUZIDOU, E., DIONYSOPOULOS, P., Eur. J. Dent., **8**, 2014, p. 353.

20.ELIASSON, S.T., TIBBALLS, J., DAHL, J.E., Oper Dent., **39**, no.5, 2014, E 206.

21.DE MELO M.A., MOYSES, M.R., DOS SANTOS, S.G., ALCANTARA, C.E., RIBEIRO, J.C., Braz. Oral Res., **25,** 2011, p. 485.

22.LOOMANS, B.A., CARDOSO, M.V., ROETERS, F.J., OPDAM, N.J., DE MUNCK, J., et al., Dent. Mater., **27**, 2011, p. 701.

23.DALL'OCA, S., PAPACCHINI, F., RADOVIC, I., POLIMENI, A., FERRARI, M., J. Oral Sci., **50**, 2008, p. 403.

24.TEZVERGIL, A., LASSILA, L.V., VALLITTU, P.K., J. Dent., **31**, 2003, p. 521.

25.PADIPATVUTHIKUL, P., MAIR, L., Dent. Mater. J., **23, no.** 4, 2007, p. 519.

26.JUNIOR, S., FERRACANE, J., BONA, A., Dent. Mater. J., **25,** no. 4, 2009, p. 442.

27.RINASTITI, M., OZCAN, M., SISWOMIHARDJO, W., BUSSCHER, H., J. Dent., **38, no.** 1, 2010, p. 29.

28.STOLERIU, S., ANDRIAN, S., PANCU, G., NICA, I., IOVAN, G., Mat. Sci. and Eng., **209**, 2017, 012078 doi:10.1088/1757-899X/209/1/012078

29.CAVALCANTI, A.N., DE LIMA, A.F., PERIS, A.R., MITSUI, F.H., MARCHI, G.M., J. Esthet. Restor. Dent., **2**, 2007, p. 90.

30.MUNTEANU, B., ANDRIAN, S., IOVAN, G., GHIORGHE, A.C., NICA, I., STOLERIU, S., Mat. Plast., **51**, no.3, 2014, p. 279.

31.NICA, I., IOVAN, G., GHIORGHE, A.C., STOLERIU, S., PANCU, G., ANDRIAN, S., Mat. Plast., **51**, no.3, 2014, p. 282.

32.TEZVERGIL, A., LASSILA, L.V., VALLITTU, P.K., J. Adhes. Dent.,**11**, 2009, p. 41.

33.VANKERCKHOVEN, H., LAMBRECHTS, P., VAN BEYLEN, M., DAVIDSON, C.L., VANHERLE G., J. Dent. Res., **61**, 1982, p. 791.

34.OZTAS, N., ALACAM, A., BARDAKCY, Y., Oper. Dent., **28**, 2003, p. 149.

35.PAPACCHINI, F., DALLOCA, S., CHIEFFI, N., GORACCI, C., SADEK, F.T., et al. J. Adhes. Dent., **9**, 2007, p. 25.

ÖZCAN, M., KOC-DUNDAR, B., J. Adhes. Sci. Technol., **28**, no. 21, 2014,

Manuscript received: 8.02.2017