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Comparison of the Micro-Tensile Bond Strength of Composite Resin Restoration in Micro- and Nano-hybrid Composite Resins using Different Interfacial Materials

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ABSTRACT

Micro-tensile bond strength of new to old composite in micro- and nano-hybrid composites was studied using two interfacial materials of composite flow and silane-bonding compound in samples prepared using aluminum generator in an in vitro manner. Sixty samples prepared after one month of aging process, were divided into three groups: A (nanohybrid to nanohybrid), B (microhybrid to microhybrid) and C (nanohybrid to microhybrid) (20 samples for each group); each group was again divided into 4 subgroups: in the subgroup 1, flow composite and in subgroup 2, silane-bonding compound, were used for bonding new to old composite after sandblasting process. The two subgroups were also labeled as positive controls (integrated nanohybrid and microhybrid composite for groups A and B, and half nanohybrid for group C) and negative controls (bonding old to new composite without interfacial mediums). The prepared specimens were divided into 1 mm pieces and the micro-tensile bond strength of each piece was measured and recorded using the Universal Testing Machine. Data analysis was done using SPSS 17 software and P-value less than 0.05 was considered as significant. The highest and lowest micro tensile bond strengths in all the studied groups belonged to the positive and negative control subgroups, respectively. Also, in all the groups, the subgroup in which silane-bonding compound was used as interfacial material had significantly stronger bond strength than the composite flow. In a general comparison between the groups, group C showed better results than group B and group B showed better results than group A (P <0.001). The results of this study show that the repair of old composite with the help of silane-bonding is the best way to increase bond strength of the composite, and also, the nanohybrid composite showed a weaker repair-ability as compared to the micro hybrid composite.

Key words: Composite Resin, Dentin-Bonding Agents, Micro-tensile Bond Strength, Silane

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INTRODUCTION

With regards to the increasing tendency for isochromatic resin restorations without mercury in accordance with the Minimally Invasive principles, and major advances in the physical and chemical properties of these materials, there are still some concerns such as color change, color discontinuity, edges, abrasion, and fracture in the composite itself [1-4]. Clinicians should therefore consider replacing or restoring the composite for aesthetic and functional reasons. In cases where there is no color change on the margin of restoration and tooth or there is no evidence of secondary caries under restoration, full replacement of composite restorations may result

in unintended spreading of the cavity and extra carving from healthy teeth and even health risk for pulp; also, this process is time-consuming and not economical [5-7].

One of the main problems with isochromatic tooth-resin composites is the formation of strong bond between new and old composites. Important factors that can affect the bond of new to old composites are: 1. interfacial material used between two composite surfaces; 2- different preparation methods of the surface of the composite for more adhesion; 3. composite type used [8]. In a study on the use of composite flow as interfacial material without any other adhesive in bonding old to new composites, Papacchini et al. [9] concluded that bond due to the use of non-adhesive composite flow was higher than that of other methods. In another study, these researchers investigated the micro tensile strength in micro-filled composite restorations and studied the different preparation techniques of the composite surfaces and the effect of oxygen inhibition layer. The results showed that the samples in which the old composite surface were sandblasted by 50 μ aluminum oxide at a distance of 5 mm for 10 s, had the highest bond strength, and the presence or absence of oxygen inhibition layer had no effects on the strength of bonding of the new to old composite [10].

A study conducted by Dall'ora et al., [11] on the effect of Oxygen Inhibition Layer on composite restoration bond strength showed that the presence or absence of oxygen inhibition layer had no visible effect on the strength of the new to old composite bond. However, bond strength decreased with time due to decrease in the number of free radicals.

Another study showed that the highest micro-tensile bond strength was observed in the group in which the old composite surface was prepared by sandblasting with 50 μ aluminum oxide, but there was no difference in the micro-tensile bond of the group prepared by hydrogen peroxide and without surface preparation [12]. Other studies have shown that both preparation of the old composite and the applied materials in bonding are effective in bond strength, and silica coating, together with silane provides a stronger bond in all the groups [13].

Study on the effect of interfacial material and warming up of new composite before restoration on bond strength of composites showed that both are effective in bond strength between two composites, and the highest bond strength was reported in a group in which composite flows were used as interfacial material and the new composite was stored at 37°C [14].

In recent years, there have been many advances in composite resins, including nanoscale composites. These composites are claimed to have higher mechanical and visual properties, better aesthetics, high abrasion resistance and lower polymerization contraction [15].

With regards to these composites, little research has been performed and in most studies, micro-hybrid composites have been used for the bond of new to old composites. Even though nano hybrid composites have been recently introduced and many studies have suggested the high physical properties of these materials due to the high percentage of fillers, there is a lack of information on their bond strength to the old composite. The purpose of this study was to compare the micro-tensile bond strength of the new to old composite in the micro- and nano-composites. At the end of this research, it will be determined whether nanohybrid composites have acceptable physical properties and whether they have similar properties in the field of bonding new to old composites.

MATERIALS AND METHODS

This is a laboratory research and the number of samples was estimated based on similar research and the research design was 60 composite blocks. The sampling method was simple random sampling and sample preparation was done according to the objective.

Micro- and nano-hybrid composite blocks were prepared by special 8.8 mm aluminum cylinders without any crack and bubble, and divided into three general groups (A, B and C). In this study, the Gradia direct anterior micro hybrid composite manufactured by GC America with color codes A3 and B3, filtek supreme XT nano-hybrid composite, manufactured by 3M ESPE in USA with color codes A3 and B3, and a flowable filtek supreme XT flow composite, a combination of silane-bonding, PBA silane, manufactured by 3M ESPE and Clearfil SE bond, manufactured by the Kuraray Japan company, were used.

Preparation of resin blocks

Forty five composite samples (15 samples of A3 nano-hybrid composites and 30 A3 micro-hybrid composites) were prepared by special aluminum generator with a 4.8 mm cylinder, and 15 integrated composite samples (5 nano-hybrid composite samples, 5 micro-hybrid composite samples and 5 half micro/half nano-hybrid composite samples) were prepared with the 8.8 mm cylinder. The composites were placed in 2 mm increments and condensed with a plastic instrument without any contamination in all the samples. They were then cured by the 7 Light Cure Astralis with a power of 750 mW/cm2 for 40 s (12, 16 and 17). After placing the increment, the composite surface was covered by a Mylar Strip to obtain a smooth surface after the curing (9,10,12,16 and 17). Then, all the samples were stored in normal saline (37°C) for one month while the normal saline was changed daily.

All the samples were sandblasted after aging by 50 µ aluminum oxide for 10 s at a distance of 5 mm under pressure of 60-100 psi. Then, phosphoric acid 37% was applied to the surface for 30 s and then washed with water for 30 s and dried at atmospheric pressure from a distance of 5 mm for 10 s [9,10,12,16,17).

A total of 15 samples of composite nanohybrid samples prepared (group A) were randomly divided into three 5-item subgroups as follows:

- Group A1: A composite flow was used as a thin layer on the surface of the old composite and the sample was placed in a special aluminum generator at the position of 8.8 mm and the new nanohybrid composite B3 was added as 2 mm increments and each layer was cured separately.
- Group A2: Silane-bonding compound was used according to the protocol on the composite surface, then the sample was placed in the special aluminum generator at 8.8 mm, and then the new nanohybrid composite B3 was added as 2 mm increments and each layer was cured separately.
- The silane was first used and after waiting for 1 min, the applied bonding was cured for 10 s (as instructed by the manufacturer).
- Negative control group: The new nanohybrid composite B3 was placed in 2 mm increments and cured without applying any mediator materials inside the generator at position 8.8 mm.
- Positive control group: This is composed of the same five nanohybrid composite samples with the size of 8.8 mm.

- A total of 30 microhybrid composite samples were randomly divided into two 15–member groups (Groups B and C).

The first group (group B) was randomly divided into three 5-member subgroups:

- Group B1: The composite flow was used as a thin layer on the surface of the old composite, and the sample was placed in a special aluminum generator at 8.8 mm position and the new microhybrid composite B3 was added in increments of 2 mm, and each layer was cured.
- Group B2: Silane-bonding compound was applied according to the protocol on the old composite surface, then the specimen was placed in a special aluminum generator at 8 mm, and the new microhybrid composite B3 was added in increments of 2 mm, and each layer was cured.
- Negative control group: New microhybrid composite B3 was placed inside a special aluminum generator at 8.8 mm on the old composite in increments of 2 mm without application of any mediator material and was then cured.
- Positive control group: This is composed of the same five microhybrid composite samples with the size of 8.8 mm.

The second group (Group C) is randomly divided into three 5-member subgroups:

- Group C1: The composite flow was used as a thin layer on the surface of the old composite, and the sample was placed in a special aluminum generator at 8.8 mm position and the new nanohybrid composite B3 was added in increments of 2 mm, and each layer was cured.
- Group C2: Silane-bonding compound was applied according to the protocol on the old composite surface, then the specimen was placed in a special aluminum generator at 8 mm, and the new nanohybrid composite B3 was added in increments of 2 mm, and each layer was cured separately.
- Negative control group: New nanohybrid composite B3 was placed inside a special aluminum generator at 8.8 mm on the old composite in increments of 2 mm without application of any mediator material and was then cured.
- Positive control group: This is composed of the same five half micro-/half nano-hybrid composite samples with the size of 8.8 mm.

Table 1: Micro-tensile bond strength (MPa) of the samples studied

	A_{PC}	A_{NC}	A_1	A_2	B_{PC}	B_{NC}	B_1	B_2	C_{PC}	C_{NC}	C_1	C_2
Sample1	179.65	49.9	56.84	75.06	161.38	44.82	80.54	112.65	149.85	*	107.86	**116.29**
Sample2	185.35	38.4	61.78	78.12	166.48	61.24	79.23	103.91	143.77	*	107.71	**114.63**
Sample3	182.22	40.4	62.74	75.66	162.74	48.28	80.21	106.01	144.6	*	103.39	**113.43**
Sample4	180.75	42.8	58.38	76.77	163.33	54.65	79.68	110.4	146.25	*	104.46	**115.75**
Sample5	183.6	45.48	60.64	77.2	165.74	58.8	79.3	105.65	145.65	*	105.67	**115.44**
Average	182.314	43.396	60.076	76.562	163.934	53.558	79.792	107.724	146.024	*	105.818	115.108

A_{PC} group: positive control A, **A_{NC}** group: negative control A, group **A_1**: old nanohybrid sandblasted composite + composite flow + new nanohybrid composite, group **A_2**: old nanohybrid sandblasted composite + silane-bonding compound + new nanohybrid composite, **B_{PC}** group: Positive control B, **B_{NC}** group: Negative control B, Group **B_1**: old microhybrid sandblasted composite + composite flow + new microhybrid composite, group **B_2**: old microhybrid sandblasted composite + silane-bonding compound + new micro hybrid composite, **C_{PC}** Group: Positive control C, **C_{NC}** Group: Negative control C(*samples were debonded when cutting) , Group **C_1**: old microhybrid sandblasted composite + composite flow + new nanohybrid composite, Group **C_2**: old microhybrid sandblasted composite + silane-bonding compound + new nanohybrid composite

Then, all samples (N = 60) were stored for 24 h in normal saline at 37°C and then examined by micro tensile bond strength test (Figure 1). Each sample was divided into 1 mm section by a diamond cutting machine containing a coolant flow, such that half of each section consists of old composite and half new composite. Then, each section was individually made to have the form of an hourglass with a cross section of 0.37-0.67 mm^2 by turbine and placed in a universal testing machine under a pure tensile force (at a speed of 0.5 mm/min) and force at failure was recorded for each sample [9,10,12,16,17].

Then, according to the formula F/A, with F, the force at failure and A cross-section in 2 mm^2, the micro tensile strength of each recorded section was calculated, and by taking the average of the sections, the micro tensile strength of each sample was obtained. Data were analyzed using SPSS17 software and ANOVA test.

RESULTS

The results obtained from this study are as follows: The micro tensile bond strength of the samples expressed in MPa is shown in Table 1.

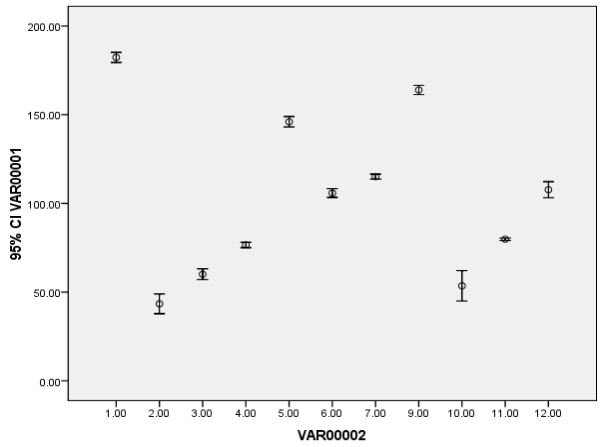
As shown in Tables 1 and 2, in all the three groups, the micro-tensile bond strength of the positive control group was significantly higher than in the other groups. In group A, the micro-tensile bond strength of the A2 group was significantly higher than that of the A1 and the negative control group, and the micro-tensile bond strength of the A1 group was significantly higher than that of the negative control group. In groups B and C, the micro-tensile bond strength of the second subgroup is higher than that of the first subgroup and the negative control group, and the micro-tensile strength of the first subgroup is higher than that of the negative control group.

Based on the statistical analysis, there is a significant difference between the mean of group A, and other groups and the mean of subgroups of A ($p < 0.001$). Also, the results show that there is no significant difference between the mean of subgroups of group C (C1 and C2) ($P = 0.34$) but there is a significant difference between group C and other groups and between subgroups of group B and other groups ($P < 0.001$).

Table 2: Mean, standard deviation, maximum and minimum of the studied groups

Micro tensile bond strength (MPa) Groups	Mean ± SD	Minimum	Maximum
Positive control group A	182.3 ± 2.268	180	185
Negative control group A	43.4 ± 4.498	38	50
Group A_1	60.08 ± 2.432	57	63
Group A_2	76.56 ± 1.29	75	78
Positive control group B	163.93 ± 2.125	161	166
Negative control group B	53.56 ± 6.92	44	61
Group B_1	79.79 ± 0.571	79	80
Group B_2	107.72 ± 3.647	103	112
Positive control group C	146.02 ± 2.17	143	149
Negative control group C	*	*	*
Group C_1	105.82 ± 1.969	103	107
Group C_2	**115.11 ± 1.114**	**113**	**116**

SD: Standard Deviation; *samples were debonded when cutting



[1]Positive control group A, [2]Negative control group A, [3]Group A1, [4]Group A2, [5]Positive control group C, [6]Group C1, [7]Group C2, [8]Negative control group C (samples were debonded when cutting), [9]Positive control group B, [10]Negative control group B, [11]Group B1, [12]Group B2

Diagram 1: Dispersion diagram of the micro tensile bond strength in the studied groups

Figure 1: Universal Testing Machine

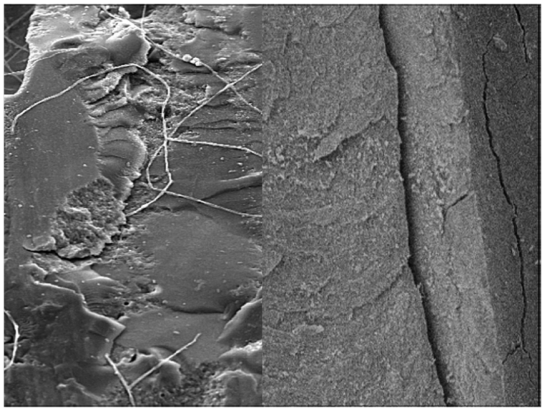


Figure 2: SEM image of bonding nanohybrid and nanohybrid composites using a composite flow

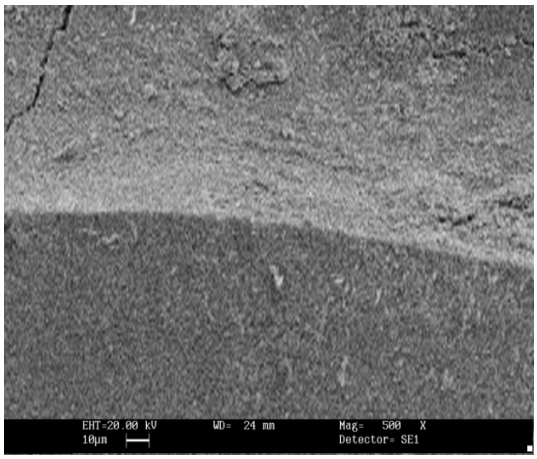


Figure 3: SEM image of bonding nanohybrid and nanohybrid composites using silane-bonding

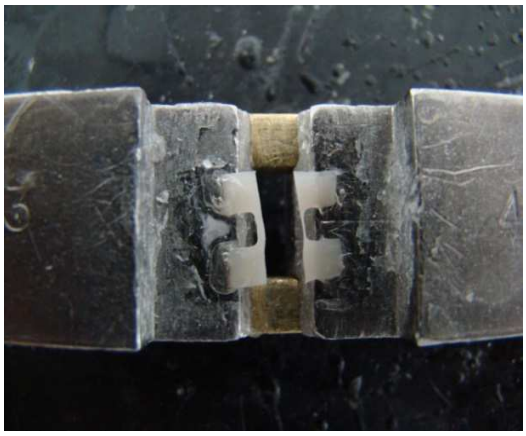


Figure 4: The image of bonding nanohybrid and nanohybrid composites separated from the bonded area

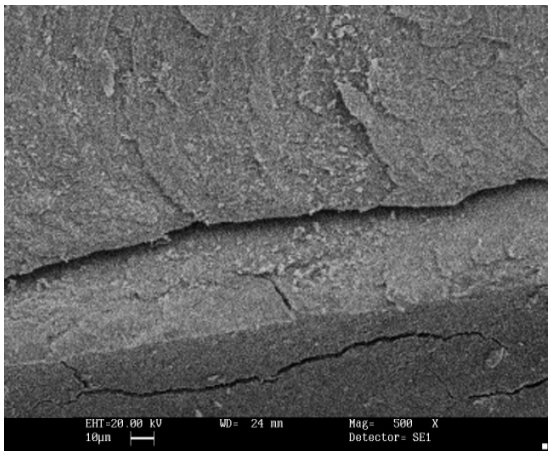


Figure 5: SEM image of bonding nanohybrid and microhybrid composites using a composite flow

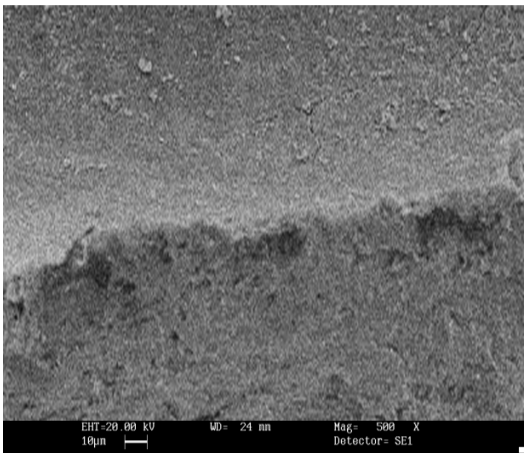


Figure 6: SEM image of bonding nanohybrid and microhybrid composites using silane-bonding

Figure 7: Bonding of nanohybrid and microhybrid composites together with the cohesive fracture

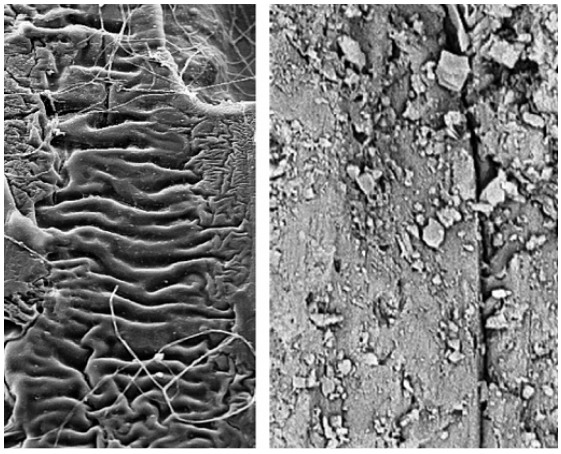


Figure 8: SEM image of bonding microhybrid and microhybrid composites using a composite flow as the interfacial material

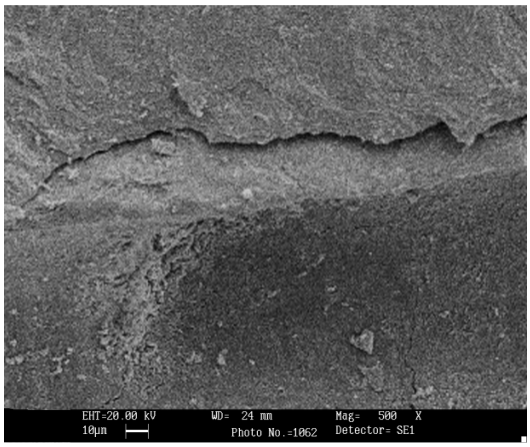


Figure 9: SEM image of bonding microhybrid and microhybrid composites using silane-bonding as the interfacial material

Figure 10: Bonding microhybrid and microhybrid composites together with the cohesive fracture

DISCUSSION

Composite restorations are commonly used in dentistry to repair or restore damaged teeth due to caries or trauma based on the new dental practice, the Minimally Invasive. Prescription cases include: the need to correct marginal defects, change in surface color, lump loss, wear or abrasion, and mass fracture of the anterior and posterior restorations [18].

In this study, the micro-tensile bond strength of restoration in micro and nano-hybrid composites was investigated using different intermediate materials (composite flow and silane-bonding composites in prepared samples). The results indicated that repairing the old composite by silane-bonding is the best way to increase the bond strength of the composite, and that the nano-hybrid composite exhibits weak repair-ability as compared to the micro-hybrid one.

According to the studies, important factors that can affect the bonding of new to old composite are: 1. Different methods of preparation of the old composite surface to create more adhesion; 2- Composite type used; 3. Interfacial medium used between two levels of composite. All these cases are fully described below [8]. Various methods such as sandblasting, milling and chemical conditioning are used for surface preparation. Sandblasting is considered as the golden standard because of the formation of homogeneous porosities [10]. Surface preparation of all samples was performed in this study using the Sandblast method. With the advent of nanohybrid composites, new opportunities to improve the physical properties of without-mercury isochromatic tooth materials have emerged. However, most claims, including the possibility of repair, are at theoretical level and there is lack of information in this regard. In this study, the possibility of repair of these composites was investigated. There are disagreements in interfacial materials between the two levels of composite, and even some clinicians are against the use of any interfacial substance. One of the characteristics of this research is that a subgroup of negative control was designed in each group, in which the new and old composite materials were bonded to each other without the use of any interfacial material after sandblasting; and the results were assessed.

Recently, composite flow and silane-bonding compounds were proposed as interfacial substances. Papacchini et al. used these two interfacial materials in two separate studies for the repair of micro-tensile composites, and in each research, very acceptable results were obtained. Therefore, in the two studies, the strongest bond was obtained using these two interfacial materials; however, these two intermediate materials were not compared in these studies, but there was comparison in the current study [9,14,16].

In this study, for three general groups: A (a new composite of nanohybrid bonded to an old nanohybrid composite), B (a new micro-hybrid composite bonded to an old micro-hybrid composite), and C (new nanohybrid composite bonded to an old micro-hybrid composite), the micro tensile bond strength of two interfacial materials of composite flow and silane-bonding compound was compared in the two subgroups for each group. The compound was compared in the two subgroups of each group. For each group, positive (integrated composite samples) and negative controls (bonding new composite to the old without application of any interfacial materials) were considered. Eventually, the interface surfaces were examined by electron microscopy (SEM) with magnifications of 500 and 2000 x.

As expected, the micro-tensile strength of the positive control subgroup (cohesive composite samples) was higher in each group, than in other subgroups. The micro-tensile strength of the negative control subgroup (bonding the new to the old composites without the use of any interfacial materials) was lower than in the other subgroups, which indicates that bonding the new to the old composite without the use of any interface and only based on the preparation of the surface of the old composite will result in a weak bond, and even in the negative control subgroup C, all samples were removed from the bonding region during cutting.

Among all the subgroups, the subgroup that used the silane-bonding compound as an interfacial substance had the strongest micro-tensile bond after the positive control subgroup of each group. This can be due to the fact that the silane-bonding combination not only penetrates the porosities resulting from surface preparation by sandblasting, but also, it helps the new composite to penetrate these porosities by creating a chemical bond with silicate groups of the composite fillers which contributes to the bond's strength. However, the composite flow is the only one responsible for filling the porosities and helps the new composite to penetrate into porosities. With a general review of the three main groups (A, B and C), it can be clearly seen that the group A, a new nanohybrid composite bonded to an old nanohybrid composite, has the weakest micro-tensile bond strength in both of its subgroups among all subgroups. This contradicts the initial hypothesis of this research. That is, although it seems that with increase in the filler percentage, the physical properties of nanohybrid composites are improved, the results of this research are not in favor of this theory. It seems that the hardness of the nanohybrid composite due to the presence of zirconium particles prevents proper surface preparation of these composites, which is also visible in the SEM images provided by the interface (Figs. 2-4); therefore most of the samples

of this group were debonded as adhesive by the introduction of pure micro-tensile strength. It seems that the surface of the nano-hybrid composites could be made porous by increasing the pressure of sandblasting, but it should be noted that increased sandblasting pressure may damage the adjacent soft tissues or even the pulp. On the other hand, the ability of aluminum oxide to make the surface of nanohybrid composites porous with the presence of zirconium particles is questionable (Figs. 2-4).

On the other hand, due to the high percentage of the filler in these composites, cluster accumulation of inactive fillers was observed near the composite surface. After sandblasting, this inactive accumulation created a not-so-strong bond. The results obtained from Group C are somewhat confirmatory of this issue, because in this group, the new nanohybrid composite was bonded to the old micro hybrid composite and in both subgroups (C1, C2), the micro tensile bond strength was acceptable and even higher than that of the other subgroups. Therefore, it could be seen that the nanohybrid composite does not provide an acceptable bond surface after preparation. The SEM images from this group also indicate that the surface of the micro hybrid composite is well prepared and the new nanohybrid composite has penetrated into these porosities through the interfacial material. The images obtained during the fracture of the samples also indicate fracture of the sample as cohesive from the old micro hybrid composite in most samples (Figures 5 to 7). In group B, in the bonding of the new to old micro hybrid composite, an acceptable strength of the micro tensile bond close to that of the Group C, was obtained, which is consistent with the results obtained by Papacchini et al. (10-12). SEM images from the samples of this group also confirm that the preparation of the surface of old micro hybrid composite is properly done and the new micro hybrid composite has also penetrated well into the porosities with the aid of the interfacial material. The images taken from the sample fractures in the group shows that most of the samples were fractured in cohesive form from the old micro hybrid composite (Figures 8-10).

With regards to the limitations of this study, it can be argued that production of the generator with millimeter dimensions is a precise and difficult task and requires a lot of time, specialists and the needed equipments like CNC, etc. Cutting the samples with a Diamond Cutting Machine to 1 mm sections is difficult and requires high precision and skill. Finally, it is suggested that future studies should be conducted in vivo and in real oral conditions, since this study was done in vitro.

CONCLUSION

According to the findings of this study, it can be said that:

- In all the studied groups, the highest and lowest micro-tensile bond strengths belonged to the positive (integrated nanohybrid and micro-hybrid composites for groups A and B, respectively, and half micro-/half nanohybrid for group C) and negative control subgroups (bonding of old to new composite with no interfacial substance).
- In all the groups, the subgroup that used the silane-bonding compound as an interfacial material had significantly stronger bond strength than the composite flow.
- In a general comparison, Group C (old micro hybrid to new nano hybrid) showed better results than Group B (old micro hybrid to new micro-hybrid) and Group B showed better results than Group A (old nanohybrid to new nanohybrid).

Conflict Of Interest

The authors hereby report no conflicts of interest with regards to this work.

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