

MICROSHEAR BOND STRENGTH OF INDIRECT COMPOSITE RESIN AFTER DIFFERENT SURFACE TREATMENTS (IN-VITRO STUDY)

Dina G. Abd El-Sadek^{1*} MSc, Wegdan M. Abdel-Fattah² PhD, Rania R. Afifi³ PhD

ABSTRACT

INTRODUCTION: An essential part in the successful clinical specifications of any indirect restoration is bond strength. Several surface treatments might be used to improve bond strength between resin cement and indirect composite restorations.

OBJECTIVES: The purpose of the study was to evaluate microshear bond strength of two different types of indirect composite resin after different surface treatments before cementation.

MATERIAL AND METHODS: sixty cylindrical specimens were prepared from two types of indirect composite and divided into 2 groups (n=30). Group A: SR NEXCO (Ivoclar Vivadent AGS chaan, Liechtenstein) and Group B: Gradia Plus Indirect composite (GC, Tokyo, Japan). A polyethylene tube was used to prepare specimens with dimensions (2 mm length and 1 mm width). Composite specimens were precured for 20 seconds by Light Emitting Diode curing unit and the secondary curing was done inside a Photopol pro vacuum light cure box for Gradia Plus for 3 minutes and 11 minutes for SR NEXCO. After secondary curing each main group was subdivided into three subgroups according to their surface treatment: subgroup 1A (sandblasting by aluminum oxide particles followed by silane), subgroup 2A (sandblasting by aluminum oxide particles + Hydrofluoric acid etching+silane) and subgroup 3A (sandblasting by aluminum oxide particles + MONOBOND Etch and prime) same surface treatments were used at subgroups 1B,2B&3B respectively. The treated composite specimens were cemented to a flat dentin surface of 10 molar teeth six samples on each tooth (three samples from each group) using G-CEM LINKACE™ (GC, Tokyo, Japan). All the specimens were thermocycled for 500 cycles from 5 to 55C, and then microshear bond strength test was done in a Universal Testing Machine 0.5 mm/min (Instron model3345, England). The mode of failure determined under stereomicroscope.

RESULTS: Statistical significant difference was noticed between subgroup 1A which was significantly higher than subgroup 2A (p=0.043) and subgroup 3A (p=0.013). Moreover, subgroup 1B recorded significantly higher microshear bond strength values than subgroup 2B (p1=0.002) and subgroup 3B (p2=0.044). Mixed failures were more prominent in subgroup 1A, 1B and 3B while adhesive failures were more prominent in other subgroups except for 2B half of the failures were adhesive and the other half mixed.

CONCLUSION: Sandblasted subgroups with aluminum oxide particles followed by silane recorded the greatest microshear bond strength values regardless of the type of composite.

KEYWORDS: Indirect composite, surface treatment, microshear bond strength.

1 BDS, Faculty of Dentistry, Alexandria University, Alexandria, Egypt.

2 Professor of Operative Dentistry, Conservative Dentistry Department, Faculty of Dentistry, Alexandria University, Alexandria, Egypt.

3 Lecturer of Conservative Dentistry Department, Faculty of Dentistry, Alexandria University, Alexandria, Egypt.

*Corresponding author:

E-mail: dinagamalabdelsadek@gmail.com

INTRODUCTION

The increase in demand for esthetic restorations that resemble the natural tooth structure put composite resin a perfect option that is widely used between dentists for posterior tooth restorations (1).

However, composite restorations have manifested their limited resistance to wear, disintegration, fracture, and secondary caries over time due to polymerization shrinkage (2).

Indirect composite restorations have solved this problem by secondary curing which enhanced the mechanical properties of the composite. Secondary curing is done in a laboratory oven under specific conditions like light, heat, vacuum, and

pressure. This will lead to increase degree of conversion (3) and decrease the number of unreacted C=C which will limit the indirect composite bonding. Therefore chemical bonding and micro-mechanical retention and are mandatory to create a successful bond between the luting composite resin and the indirect restorations especially in non retentive restorations, like inlays and onlays (4, 5).

Several surface treatments have been used for micromechanical retention for instance sandblasting with aluminum oxide particles, hydrofluoric acid etching, roughening with diamond points or carbide burs and laser (6).

Additionally, Silane coupling agent is important for chemical bonding. It is a bifunctional molecule used to create a chemical bond between inorganic fillers of the laboratory processed composite and the methacrylate monomers of the resin cement matrix, besides it enhances the wettability of the fitting surface of the restoration and allows the resin cement to flow inside the irregularities and concavities of the treated surface of the restoration (7).

Bond strength between restoration and resin cement is a crucial factor for successful indirect restorations. The micro-shear bond strength (μ -SBS) test has been introduced as an altered method for calculating the bond strength of dentin-adhesive assembly using smaller specimen. Besides, after the bonding procedure it does not require an extra trimming process for the specimens which maintain the integrity of the specimens and prevent pre-testing failures (8).

The objective of this study is to investigate microshear bond strength of two types of laboratory processed composite resin materials using three different surface treatments before cementation. The null hypothesis is that there would be no difference in the microshear bond strength of the two types of indirect composite after different surface treatments.

MATERIAL AND METHODS

Material used in this study presented in (Table 1).

Sample size estimation

Sample size was predicted to be 60 composite samples in microshear bond strength. The predicted sample size is made at expectation of 95% assurance level and 80% power of study. (30 samples in each of main two groups, and 10 sample in each of three subgroups) (9, 10)

Composite samples and their preparation for micro shear test:

The teeth selected for this study were mandibular human molars (n=10) with similar normal morphology (free from any tooth anomalies; dens evaginatus and dens invaginatus) and free from cracking, abrasion, previous restorations, caries, attrition and erosion. The teeth were gathered from the out-patient clinics at the faculty of dentistry, oral surgery department, Alexandria University, Egypt. The study was conducted after the permission of the ethical committee at the faculty of dentistry; Alexandria University.

The teeth were cleaned with a hand scalar (Zeffiro, Lascod, Florence, Italy), to remove calculus and soft tissues. They were reserved in 0.1% thymol solution at room temperature until used, and then the roots of the teeth were embedded in a copper ring filled with auto polymerizing resin (14 mm in diameter) (11). The occlusal third of each molar was ground flat using a low-speed motor with a diamond disc with a cooling system to remove enamel and prepare a flat superficial dentin surface (12).

Two types of indirect composite were used (Group A: SR NEXCO, group B: Gradia plus Indirect composite). The composite was built up using a polyethylene tube (BioFlonIV cannula, India) with dimensions of 2 mm length and 1 mm width and cured using a light-emitting diode curing unit (LED) (Woodpecker, LED.D, China) for 20 seconds.

Secondary curing was done in PHOTOPOL PRO VACUUM light cure box for 11 minutes to final cure the SR NEXCO composite and 3 minutes for GRADIA plus indirect composite (13). The two main groups were subdivided according to their surface treatment n=30 into 3 subgroups.

Group A: SR NEXCO indirect composite samples (n=30)

Subgroup 1A. composite air-abraded using 50 μ m aluminum trioxide particles Al₂O₃ particles (Renfert GmbH, Hilzingen, Germany) for 10 s followed by silane coupling agent (405-AUltradent).

Subgroup 2A: composite air-abraded using 50 μ m aluminum trioxide particles Al₂O₃ particles for 10 s, then etched with 9 % hydrofluoric acid for 60 s (405-AUltradent), washed and dried then silane coupling agent was applied.

Subgroup 3A: composite air-abraded using 50 μ m aluminum trioxide particles Al₂O₃ particles for 10 s, then a thin coat of the Monobond etch and prime (IvoclarvivadentAGSchaan, Liechtenstein) was applied using a micro-brush; for 60 s then washed and dried.

Group B: Gradia plus indirect composite resin samples (n=30)

Subgroup 1B. composite air-abraded using 50 μ m aluminum trioxide particles Al₂O₃ particles (Renfert GmbH, Hilzingen, Germany) for 10 s followed by silane coupling agent (405-AUltradent).

Subgroup 2B: composite air-abraded using 50 μ m aluminum trioxide particles Al₂O₃ particles for 10 s, then etched with 9 % hydrofluoric acid for 60 s (405-AUltradent), washed and dried then silane coupling agent was applied(14).

Subgroup 3B: composite air-abraded using 50 μ m aluminum trioxide particles Al₂O₃ particles for 10 s, then a thin coat of the Monobond etch and prime (IvoclarvivadentAGSchaan, Liechtenstein) was applied using a micro-brush; for 60 s then washed and dried.

Cementation of composite samples

Etching of the dentin surface of the teeth was done with N-etch (37% phosphoric acid etching, ivoclar, vivadent) for 15 seconds then washed and dried then a thin layer of Te-Econom (universal dental adhesive bond, Ivoclar Vivadent) was applied and cured for 20 seconds, after that six samples (three samples from each group) were cemented to the dentin surface of each tooth using self-etch dual-cure adhesive resin Cement (GCEM-linkAce™) according to manufacturer's instructions. Specimens were photo-polymerized for 20 s (14). After that,

thermocycling was done for all the specimens (500 cycles from 5 to 55C).

Micro shear bond strength test (Figure 1)

Each Bonded specimen was attached with screws to the fixed lower compartment of the universal testing machine (Instron model3345, England) with a load cell 500 Newton. An orthodontic wire 0.14 mm in diameter was prepared as a loop and was wrapped around the cylindrical. The wire was straightened with the loading axis of the upper movable part of the testing machine. The specimens were subjected to microshear load at a constant crosshead of 0.5 mm/min until debonding failure occurs. The micro shear bond strength values were evaluated from the maximum load at failure divided by the bonded surface area. The composite-dentin fractured specimens were evaluated using stereomicroscope and classified as adhesive (failure at the dentin/composite interface), cohesive (failure within the resin composite) or mixed (partial adhesive/partial cohesive fracture) (15).

Statistical analysis of the data

Data were inserted to the computer and investigated with IBM SPSS software package version 20.0. (Armonk, NY: IBM Corp). The Kolmogorov-Smirnov test was used to confirm the normality of distribution. Quantitative data were described using range (minimum and maximum), mean, standard deviation, median and interquartile range (IQR). Significance of the obtained results was judged at the 5% level. F-test (ANOVA) was used for normally distributed quantitative variables, to compare between more than two groups, and Post Hoc test (Tukey) for pairwise comparisons.

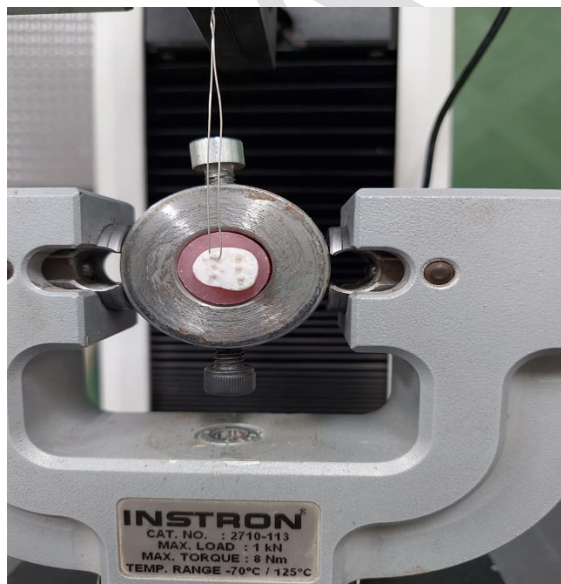


Figure (1): Microshear specimens in universal testing machine.

Table (1): Materials used in this study with their composition.

Material	Composition	Manufacture
Gradia Plus (indirect composite)	1–5%: bisphenol A-glycidyl methacrylate, 5–10%: triethylene glycol dimethacrylate, 1–5%: urethane dimethacrylate, ceramic filler (barium glass, silica)	GC, Tokyo, Japan
SR Nexco (indirect composite)	Dimethacrylates (17-19 wt%), copolymer and silicon dioxide (82-83%) additional contents are stabilizers, catalysts and pigments (<1 wt.%)	Ivoclar Vivadent, Shaan, Liechtenstein
GCem linkAce™	Fluoroaminosilicate glass, initiator, pigment, 4-MET, phosphoric acid ester, monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer	GC, Tokyo, Japan
Monobond plus etch&prime	Tetrabutyl ammonium dihydrogentrifluoride, methacrylated phosphoric acid ester, trimethoxysilylpropyl methacrylate, alcohol, water	Ivoclar Vivadent, Shaan, Liechtenstein

RESULTS

Microshear bond strength results

The highest mean of microshear bond strength values were recorded in subgroup 1B (sandblasted group followed by saline) of GRADIA PLUS indirect composite (28.75±2.46) while the lowest mean of microshear bond strength values were recorded in subgroup 3A (Monobond etch and prime) of SR NEXCO (18.99±3.32). (Figure 2& 3) In group A, post Hoc test revealed significance difference between subgroups 1A and 2A (p=0.043) and subgroups 1A and 3A (p=0.013) (Table 2)

The highest mean value of microshear bond strength was in subgroup 1A with minimum to maximum values (17.78-26.38) respectively; with a mean±Sd (23.41±3.23) followed by subgroup 2A with minimum to maximum values (15.75-24.31)

respectively; with a mean \pm sd of (19.74+3.10) then followed by subgroup 3A with minimum to maximum values (13.81 – 26.43) respectively; with a mean \pm sd (18.99+3.32) (Figure 2).

In group B, post Hoc test revealed significance difference between subgroups 1B and 2B ($p_1=0.002$) and subgroups 1B and 3B ($p_2=0.044$) (Table 3)

The highest mean value of microshear bond strength was found in subgroup (1B) with minimum to maximum values (25.01-32.33) respectively; and a mean \pm Standard deviation (28.75 \pm 2.46), followed by subgroup (3B) with minimum to maximum values (21.86 - 28.84) respectively and a mean \pm Sd (25.89+2.55) then followed by subgroup (2B) with minimum to maximum values (21.31-30.69) respectively; with a mean \pm sd (24.49+2.56).(Figure 3)

Failure mode analysis

When the failure mode was analyzed, in general, mixed failures were the more prominent type of failures in subgroups 1A, 1B and 3B as a percentage (70%,70% and 60%) respectively, while adhesive failures were more prominent in the remaining subgroups except for subgroup 2B half of the specimens were mixed and the other half adhesive failure. Cohesive failure found in one specimen in subgroup 1A. (Table 4)

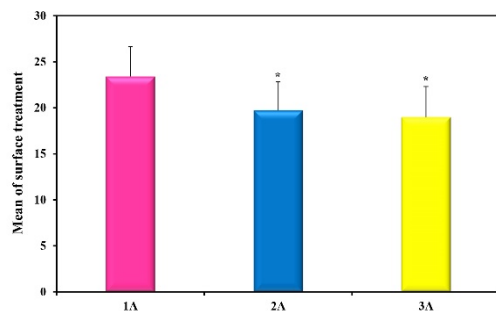


Figure (2): Comparison between the three sub groups according to surface treatments in group A.

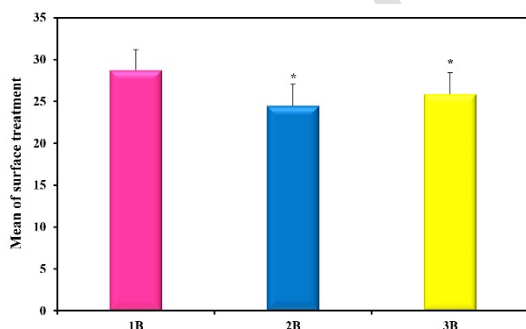


Figure (3): Comparison between the three sub groups according to surface treatments in group B.

Table (2): Comparison between the three sub groups according to surface treatments in group A.

Surface treatment	1A (n = 10)	2A (n = 10)	3A (n = 10)	F	p
Min. – Max.	17.78 – 26.38	15.75 – 24.31	13.81 – 26.43	5.414*	0.011*
Mean \pm SD.	23.41 \pm 3.23	19.74 \pm 3.10	18.99 \pm 3.32		
Median (IQR)	24.45 (20.24 – 26.08)	20.58 (16.89 – 21.98)	18.40 (17.35 – 20.45)		
Sig. bet. grps.	p ₁ =0.043*, p ₂ =0.013*, p ₃ =0.862				

IQR: Inter quartile range
 SD: Standard deviation
 F: F for ANOVA test, Pairwise comparison bet. each 2 groups was done using Post Hoc Test (Tukey)
 p: p value for comparing between the studied groups
 p₁: p value for comparing between 1A and 2A
 p₂: p value for comparing between 1A and 3A
 p₃: p value for comparing between 2A and 3A
 *: Statistically significant at $p \leq 0.05$

Table (3): Comparison between the three sub groups according to surface treatments in group B.

Surface treatment	1B (n = 10)	2B (n = 10)	3B (n = 10)	F	p
Min. – Max.	25.10 – 32.33	21.31 – 30.69	21.86 – 28.84	7.441*	0.003*
Mean \pm SD.	28.75 \pm 2.46	24.49 \pm 2.56	25.89 \pm 2.55		
Median (IQR)	28.97 (26.5 – 30.40)	24.54 (22.7 – 25.03)	27.09 (22.8 – 27.82)		
Sig. bet. grps.	p ₁ =0.002*, p ₂ =0.044*, p ₃ =0.437				

IQR: Inter quartile range
 SD: Standard deviation
 F: F for ANOVA test, Pairwise comparison bet. each 2 groups was done using Post Hoc Test (Tukey)
 p: p value for comparing between the studied groups
 p₁: p value for comparing between 1B and 2B
 p₂: p value for comparing between 1B and 3B
 p₃: p value for comparing between 2B and 3B
 *: Statistically significant at $p \leq 0.05$

Table (4): Mode of failure of the six subgroups.

Mo de of fail ure	1A (n = 10)		2A (n = 10)		3A (n = 10)		1B (n = 10)		2B (n = 10)		3B (n = 10)	
	N	%	N	%	N	%	N	%	N	%	N	%

Adhesive	2	20	6	60	7	70	3	30	5	50	4	40
Cohesive	1	10	0	0	0	0	0	0	0	0	0	0
Mixed	7	70	4	40	3	30	7	70	5	50	6	60

DISCUSSION

Indirect or laboratory-processed resin generally needs extra polymerization to increase the polymeric conversion, and consequently increases the mechanical properties. The degree of conversion after initial curing is about 42-45% and reaching 68 % after secondary polymerization (16). This increase in the degree of conversion reduces the number of unreacted methacrylate groups available for creating a bond between the restoration and the resin cement, (17) leading to a decline in the bond strength about 25-80%. Thus, surface treatment of the indirect restoration is mandatory to improve bonding (16).

This study tested the influence of three surface treatments on microshear bond strength of two types of laboratory-processed composite resin. The results showed a significant difference between all subgroups for the two types of composites, thus the null hypothesis was rejected.

The results of the present study revealed that the two types of indirect composite treated by sandblasting and silane showed significantly higher microshear bond strength values than sandblasting followed by Hydrofluoric acid etching and silane. **Our results are consistent with Poskus et al (18) and Soares et al (19) who found that using sandblasting followed by hydrofluoric acid and**

silane weakened the bond strength of indirect composite compared with sandblasting followed by silane. This could be attributed to the effect of sandblasting with aluminum oxide particle abrasion (50 um) in the exposure of the filler particles and facilitates bonding in the silanization step. However acid conditioning might cause resin melting of the matrix and complete disintegration of the glass fillers on the composite which was important for bonding and silanization step (19).

On the other hand the results of this study contradicting with Hori S (20) et al who used 1% hydrofluoric acid etching for five minutes and found that hydrofluoric acid etching with silane application recorded higher bond strength values than sandblasting with silane application only. This disparity in the results might be due to the difference in the composite type and the concentration of hydrofluoric acid used. In the above mentioned study, the authors used Estenia C&B indirect composite which has alumina particles that were not degraded by hydrofluoric acid etching when observed under scanning electron microscopy while Gradia plus composite resin used in the present study contains barium glass filler and silica ,and SR NEXCO composite resin contains silicon dioxide as inorganic filler particles. These fillers might be less resistant to the disintegration of hydrofluoric acid etching. Moreover, the concentration of the hydrofluoric acid etching used in the current study was higher (9% for 60 seconds). This concentration might have a negative effect on the indirect composite because it had a dissolving action on the filler particles used (20, 21)

In the present study, the results exhibited that the two types of indirect composite treated by sandblasting followed by silane application showed significant higher bond strength values than sandblasting followed by monobond etch and prime. These findings agreed with those reported by El Damnhoury et al (23) who concluded that sandblasting followed by silane application recorded higher bond strength values than monobond etch and prime when applied in indirect composite resins. It might be attributed to the composition of Monobond etch and prime that contains ammonium polyfluoride acid and silane in one bottle, polyfluoride acid is responsible for dissolving the glass particles, while silane remains on the surface of the composite after rinsing with water to create chemical bond with the resin cement. However the acidity of the polyfluoride might cause excessive softening of the glass filler particles and the absence of glass filler particles make the silanization step useless (22, 23).

According to the results obtained from this study the mean microshear bond strength values of sandblasting with silane application surface treatment of Gradia plus composite was higher than SR Nexco. The difference in particle size and

microstructure of the composite might affect the bond strength values for the two types of composite. SR Nexco has highly scattered silicon dioxide act as microfiller particles which are different in size and non-homogeneous due to the copolymer and prepolymer composition (24). This non-homogenous structure might increase the propability of cracks on the composite surface during the sandblasting process. However Gradia Plus indirect composite is a nanohybrid lab composite with barium glass and silica nano filler with ultrafine filler with homogenous distribution mixed with the resin matrix (25, 26), this might cause a decline in the probability of crack propagation in the composite surface. It was reported by Yoshihara et al (27) that sandblasting increased the surface roughness with filler exposure; however when observed under scanning electron microscopy it also might cause subsurface and surface cracks in between the resin matrix and in the interface between the filler particles and resin matrix which might decrease the bond strength values (27).

The mode of failure according to the results obtained from this study, mixed failures were more prominent in sandblasted subgroups followed by silane in the two types of composite. Meanwhile, the adhesive failure was in the groups related to hydrofluoric acid and monobond etch and prime, these findings agreed with a previous study done by Pokus et al (18) who compared sandblasted followed by silane indirect composite with sandblasted followed by hydrofluoric acid etching and silane. The auther stated that this might be occur due to the higher values of the bond strength which may have a pivotal role in these finding (18).

CONCLUSION

Within the limitations of this in-vitro study, it can be concluded that:

The treatment of the fitting surface of the indirect composite restorations is mandatory to enhance the bond strength of the restoration.

Aluminum oxide particles with 50 um as sandblasting followed by silane remains the golden standered in indirect composite surface treatments. Gradia plus indirect composite treated with sandblasting followed by silane recorded superior results in mean microshear bond strength compared with sr nexco

Hydrofluric acid etching and monbond etch and prime weaken the bond strength of the tested indirect resin composite.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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