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Micro-tensile Versus Micro-shear Bond Testing of Dental Adhesive Systems to Dentin

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Abstract
Keywords: *micro tensile bond strength , micro Shear bond strength, adhesive system , Occlusal dentin, Thermocycling.*
Purpose: *to Compare between micro tensile and micro shear bond strength of three adhesive systems (Futurabond U adhesive, Single Bond Universal adhesive, Swiss TEC SL Bond) to dentin.*
Materials&Methods: *sixty freshly extracted sound human third molars were selected from patients aged 20- 25 years. The occlusal enamel is removed to expose the occlusaldentin, The prepared specimens were randomly divided into three groups according to the type of adhesive used (n=20 each): each adhesive were applied on dentin surface then composite resin were applied . All specimens were stored in distilled water for 24 hours. specimens of each group were subjected to thermocycling. Each group was subdivided into 2 subgroups according to bond strength test used (micro tensile or micro shear) .The bond strength of specimens were measured using an instron machine at a cross head spead of 0.5 mm/min. The debonded surfaces were examined under a stereomicroscope at magnification 40X to determine the mode of failure. All data was collected, tabulated and statistically analysed.*
Results: *Group III recorded a statistical significant most high micro tensile and micro shear bond strength values (38.01±8Mpa),(12.07±2.08Mpa)respectively. followed by group II recording micro tensile and micro shear bond strength (26.7±7.52 Mpa), (11.59±1.82 Mpa) respectivly. while the lowest values were found at group I with mean values of micro tensile and micro shear bond strength (25.31±5.2 Mpa), (12.07±2.08 Mpa) respectivly. ANOVA test was used to compare the three tested groups in each subgroup at a level of significance 0.05.*
Conclusion: *Under the present situation of this research, it was concluded that there is a good bond between all the tested adhesives and sound dentin.*

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I. Introduction

The goal in adhesive dentistry is to achieve an adequately strong bonding of the restorative resin to the tooth structure so that there is optimum retention, decreased microleakage and hence, superior color stability and clinical longevity of the restoration.[1]

The functions of dental adhesives is to promote conservation of tooth structure and enable minimally invasive dentistry, reinforcement of weakened dentin or enamel, reduce marginal staining, reduce microleakag and may also reduce postoperative sensitivity when used appropriately. [2,3]

The main challenge for a dental adhesive is the ability to bond effectively to substrates of different nature. Adhesion to dentin poses a difficult challenge. This is partly due to the biological characteristics of dentin, namely its highly organic content, its tubular structure, and the presence of the dentin smear layer that forms immediately after cavity preparation. [4,5]

A new type of single step self-etch adhesives has been introduced. This type of self-etch adhesive is categorized as ‘universal’ or ‘multi-mode’ as they can be used either with the total-etch mode or the self-etch mode or as ‘selective’ enamel etching mode; self-etch on dentin and etch and rinse on enamel.[6]

Compared with conventional tensile and shear tests both microtensile and microshear tests allow standard tooth regions to be selected, thus preserving the uniformity of the testing area.[7]

The micro tests are characterized by the use of specimens with small bonding areas (less than $2mm^2$).[8]The micro shear test is normally considered easy to execute,[9] Where a knife-edge-chisel or an orthodontic wire loop are closely positioned at the adhesive interface, generating a shear (parallel) stress at the edge of the bonding area.[10]

Maintaining the strong bond of these materials to dentin is mandatory for their success and durability,so, the present study is designed to evaluate micro tensile and micro shear bond strength tests of different adhesive systems to dentin.

The current research hypothesis is to prove that recent contemporary adhesive systems add a new bond strength relationship to dentin structure

II. Materials

Three adhesive systems and their composition were used in this invitro study as shown in table (1).

III. Methods

A total of sixty sound , freshly extracted, sound,non-carious human third molars from patients aged (20-25) years old were collected from the Department of oral and maxillofacial surgery of faculty of Dentistry Tanta University .

The patients signed a written consent. The teeth were cleaned of debris and calculus using periodontal scalers and polished with pumice. They stored in an incubator of 37 °C using distilled water which is changed daily for a period of month maximum 11

Specimens preparation and grouping: Each tooth was embedded in plastic molds, with self-cure acrylic resin till the cervical line . The occlusal enamel was removed from each tooth by using low speed diamond disk[1] with coolant water to expose the coronal dentin.

After enamel removal, the resulting dentin surfaces were flattened and finished using 600 grit Silicon Carbid papers[2] to create a standardized smear layer.

The prepared specimens were randomly divided into three groups of twenty teeth each **(n=20)** according to the type of adhesive system to be investigated.

Groups:

- **Group 1**: Futurabond U adhesive was applied as follow: Single dose is activated by pressing the posterior chamber; liquid was pushed and mixed with the liquid in anterior chamber. Single Tim applicator used and the adhesive was applied and rubbed for 20 seconds, Gentle air drying for 5seconds then Light curing for10 seconds as shown in(figure 1).
- **Group 2:** Single Bond Universal adhesive was rubbed gently for 20seconds, Gentle air drying for 5seconds then Light curing for 10 seconds as in(figure 2).
- **Group 3:** Swiss TEC SL Bond was applied as follow: Swiss TEC SL Etchant Gel was applied directly from the syringe for 15 seconds then Rinsing for 20 seconds. Excess water was removed from the surface with a cotton pellet or with a short spray from the air gun. The surface was not left to dry completely, as the exposed collagen layer might collapse and reduce adhesion.

Swiss TEC SL Bond was applied directly from the bottle onto a disposable brush and rubbing onto dentin for 20 seconds. The rubbing movement on the surface supports this process. Lightly air drying then Curing for 30 seconds.After application of the adhesive system to each specimen, Composite resin was applied to the conditioned dentin surfaces and then cured for 40 s using a Bluephase C5 LED visible light curing unit[3] at a light intensity of 500 mW/cm^2 at zero distance. All bonded specimens were stored in distilled water at 37 $^\circ$C for 24h before testing as in(figure 3)

for micro-tensile specimens: The composite applied to the conditioned dentin surface using universal tofflemire band retainer which encircle the prepared tooth and the composite resin applied onto the surface area (Figure IV-3), then cured for 40 sec with Blue phase C5 LED visible light curing unit4 at a light intensity of 500 mW/cm^2 at zero distance.

for micro-shear specimens: A polyethylene tube of a 2 mm^2 internal diameter and 2 mm height were firmly attached to the exposed dentin and the composite resin packed into the tube using small diameter plastic instrument then it was cured for 40 sec with Blue phase C5 LED visible light curing unit5 at a light intensity of 500 mW/cm^2 at zero distance.

Sub grouping: The final specimen of each group were subdivided randomly into two equal subgroups A and B (10 specimens each) according to the bond strength test used if micro tensile or micro shear bond strength ,all specimens were subjected to thermal stresses using athermocycling apparatus for5000 cycles (5°C to 55°C) with 30 sec. dwell time and 20 seconds transfer time 12.

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[1] Isomet, Bueehler, Lake Bluff, IL , US
[2] waterproof Silicon Carbide paper; Atlas,UK
[3] Bluephase N; Ivoclar Vivadent
[4] Bluephase N; Ivoclar Vivadent
[5] Bluephase N; Ivoclar Vivadent

Bond strength testing:

Microtensile bond strength testing (μTBS):

The specimens were sectioned perpendicular to the adhesive/tooth interface using IsoMet saw at a direction then the specimens were rotated for 90 degree and were sectioned again to obtain rectangular beams 1 x 1 mm with length of 6mm (3 mm for composite and 3 mm for tooth structure) for μTBS testing.Then the specimens were mounted in an Instron universal testing machine. Each specimen was attached with its ends to a specially designed, modified version of Ciucchi's jig using tetric-flow flowable composite (3M adhesive) .

The attachment jig consisted of two aluminum articulating parts, one is fixed and the other is moving. Two cylindrical copper rods were attached to the fixed part . The use of two rods instead of just one was done to prevent the rotation of the assembly. The moving part enclosed two matching grooves to guide the movement of the rods. The lengths and diameters of the grooves were slightly greater than those of the copper rods to allow frictionless movement of the rods. The force was applied to the moving part through an aluminum rod fitted to its end.

The final assembly was then mounted on a universal testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA). The data was recorded using computer software (Instron® Bluehill Lite Software). A tensile load with compression mode of force was applied via materials testing machine at a crosshead speed of 1 mm/min using a load cell of 500 N. The μTBS was expressed in MPa. The applied tensile force resulted in debonding along the substrate-adhesive interface (Figure 4).

Microshear bond strength testing(μSBS):

Each disc with its own bonded micro-cylinders was secured horizontally with tightening screws to the lower fixed compartment of a materials testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA) (Figure IV-9), with a load cell of 5 kN and data were recorded using computer software (Instron® Bluehill Lite Software). Shearing test was done by compressive mode of load applied at tooth-resin interface using a mono-bevelled chisel shaped metallic rod attached to the upper movable compartment of testing machine traveling at cross-head speed of 1 mm/min. The load required to debonding was recorded in Newton (Figure 5).

The μSBS values (MPa) were calculated from the peak load at failure divided by the bonded surface areas follow: μSBS (MPa)$=N/A \quad A=\pi \times r^2$

(A = the bonding area) (r= the radius of composite cylinder)

Mode of failure analysis: The fractured surfaces of the debonded specimens were inspected under a stereomicroscope at 40x magnification to determine the mode of failure for each specimen15**.**

Adhesive failure; at dentin-restoration interface where no observable restorative material remained on the dentin surface.

Cohesive failure; either in dentin or restoration wherea visible thin coating or bulk of a restorativematerial remained on the dentin surface. Or **Mixed failure**; if a part of restorative materialwas left on dentin surface and the rest of the surface had a partial adhesive failure**.** Mode of failure data was also collected, calculated, tabulated and the percentage of each type of failure wasobtained to bestatistically analyzed.

IV. Result

Regarding the micro tensile bond strength values of specimens data of subgroup A of all tested adhesives showen in(table2) (fig.6) The highest mean value was recorded for group III *(Swiss TEC SL Bond)*, recording(*38.01±8*Mpa) followed by group II (*Tetric n universal bond)*, recording (*26.7±7.52* Mpa), , while the lowest mean bond strength value (*25.31±5.2* Mpa), was found at group I (*(futurabond u)* and there are statistical significant difference was reported with P-value 0.001 between different groups.

Regarding the micro shear bond strength values of specimens data of subgroup B of all tested adhesives showen in(table3) (fig.7) The highest mean value was recorded for group III *(Swiss TEC SL Bond)*, recording (*12.07±2.08*Mpa) followed by group II (*Tetric n universal bond)*, recording (*11.59±1.82* Mpa), while the lowest mean bond strength value (*12.07±2.08* Mpa, was found at group I (*(futurabond u)* and there are statistical significant difference was reported with P-value 0.001 between different groups

Modeoffailure:

The tested specimens of all materials showed different modes of failure of fractured specimens. micro tensile sub group (subgroup A),

Chi square test showed significant difference in failure mode distribution between groups (p=<0.0001< 0.5). table 4 and figure 8

group I showed that 30.77 % of tested samples revealed adhesivemode of failure (fig. 8), and 69.23 % a mixed mode of failure. Concerning group II ,44.44 % adhesive failure, 11.11 % a cohesive mode of failure and 44.44

% mixed mode of failure. However, group III revealed 15.39% adhesive mode of failure while 15.38% cohesive mode of failure and 69.23% mixed mode of failure.
micro shear subgroup (subgroup B). Data was collected in (table 5) (fig.9).Regarding group (I) 67% revealed adhesive mode of failure while 33% were mixed mode of failure. However for group (II) 33% adhesive mode of failure have been recorded and 67% mixed mode of failure. Concerning specimens of group (III)recorded 25% adhesive mode of failure and 75% mixed mode of failure. Chi-square test was used to compare the three modes of failure of both subgroups. There was a statistical significant difference with P-value 0.0001 as in figure 10.

V. Discussion

The current in-vitro study evaluate the micro tensile and micro shear bond strength of different adhesives (*Swiss TEC SL Bond, Tetric n universal bond, futurabond u)* to dentin.

The effect of a single variable is easily to be evaluated in-vitro while keeping all other variables such as tooth structure, morphology contamination and oral environmental conditions constant. In-vitro tests are also easy, fast and relatively cheap to screen new materials and techniques**.** It was therefore chosen to perform the present study in the laboratory of faculty of dentistry tanta university.**[12]**

Clinically, the main cause of failure of composite fillings is related to the occurrence of marginal leakage, which eventually leads to marginal discolouration, secondary caries, and subsequent loss of retention.[13,14]

In addition selecting micro-shear bond strength test is justified because it is easy to perform, requiring minimal equipment and specimen preparation. It is a valuable factor that indicates the degree of adhesion of material to tooth structure**.** Micro-shear bond strength test show a trend for increased bond strength values with the using of small bonding areas, thus using polyethylene tube currently with 2 mm^2 in diameter and 3 mm in height was performed to obtain a standard and small bonding area.[15,16]

Also the micro-tensile method of adhesion testing permits a more uniform stress distribution along the bonded interface. In addition, this technique enables the measurement of bond strength to very small areas decreasing the probability of air voids incidence in the prepared specimens.[17]

In this study Futurabond U adhesive which is manufactured by voco was used as a self etch bonding agent.It is composed of Water, Ethanol, Silicium dioxide, Acid modified methacrylate (methacrylate ester), HEMA(2-hydroxyethyl methacrylate) and Camphorquinone .

Futurabond U adhesive is a self-etch adhesive system which was selected currently because Rosa WL et al. reported that it is (a self-etch system) one-bottle, no mixing system decreasing post-operative sensitivity.[18]

Poptani et al,[19] stated that it uses methacrylate ester as a resin monomer, adding that, the balance of water-acidic monomers and resin monomers in self etch adhesives is paramount in optimizing bond efficacy to dentin.

Also Tetric N Bond Universal which is manufactured by ivoclar was used as universal adhesive which is characterized by being Universally suitable for direct and indirect bonding procedures and all etching protocols and Minimal risk of postoperative sensitivity due to the integrated desensitizing effect.[20]

Tetric N-Bond Universal contains low levels of acidic monomer, and are therefore “mild-etching” adhesives. Tetric N-Bond Universal has a pH of approximately 2.5 –3.0. The Tetric N-Bond Universal matrix is based on a combination of monomers of hydrophilic (hydroxyethyl methacrylate/HEMA), hydrophobic (decandiol dimethacrylate/ D3MA) and intermediate (bis-GMA) nature. This combination of properties allows Tetric N-Bond Universal to reliably bridge the gap between the hydrophilic tooth substrate and the hydrophobic resin restorative, under a variety of surface conditions.[21]

The last bonding agent was Swiss TEC SL Bond manufactured by Coltene/Whale dent AG which consists of (HEMA, Hydroxpropyl methacrylate, Glycerol dimethacrylate, Polyalkenoate methacrylized, UDMA) to be used in a total etch mode (etch-and-rinse technique). It’s a two-step bonding technique in which 37% phosphoric acid (H3PO4) which removes the smear layer and conditions dentin before bonding agent application.[22] Some studies reported that the bond strength is largely influenced by the resin composite used, thus this variable was excluded by using one type of composite resin. As the use of stiffer composites resin restoration may significantly increase bond strength values,[23]thus **Filtek Z350 XT** Nano composites manufactured by 3M which is universal Restorative is a visible light-activated composite designed for use in anterior and posterior restorations was selected. It enhanced optical properties, excellent handling characteristics and be used mostly in other researches.[24]

The use of the thermo-cycling of 5000 cycles (5-55°C) was selected to subject the bonded interface of composite resin to thermal stresses generated by the different thermal conductivities and coefficient of thermal expansion of the substrates and bonded materials simulating the clinical situation. **Chao Xie et al**, [25] studied the the effects of thermocycling on Microtensile bond strength of one- and two-step self-etching adhesives on sclerotic dentin. The author used 5000 cycles as representative of 6 months clinical follow up.

The three adhesive systems presented µTBS results three -to-four times higher than those obtained in the µSBS test, which corroborates with the findings of other studies.

This can be explained by the difference in loading forces between both bond strength tests during their execution in the universal testing machine. while the micro tensile test stresses the adhesive interface perpendicularly, leading to the separation of the composite resin and the dentin substrate by opposite forces, the microshear test produces severe stress concentrations near the desired test site by the occurrence of parallel forces.[26]

According to **Stape et al**, [27] as higher the bonding area, higher the probability of some defects occur in the specimen (even at the dentin, composite, or adhesive sites),which may reduce the bond strength values. Considering that the specimens from the µSBS test showed a bonding area more than twice of those from the µTBS test, it can be inferred that this fact might have added a complimentary benefit for the better performance of the micro tensile bond strength test compared to the micro shear one.

This was against what **Andermatt L, Özcan M.**[28] reported as they repoted that in the microshear bond test, loading forces are applied as close as possible to the interface, [29] Resulting in severe stress concentrations near the desired test site, but In case of the microtensile bond test, the loading force passes through the tooth substrate and resin composite before reaching the adhesive interface, with subsequent stress concentration in these materials. These stress concentrations may lead to frequent cohesive failures within the tooth substrate.

This finding is consistent with previous studies, which have indicated that the Swiss TEC SL bond (total etch adhesive) represented high bonding performance in both laboratory and clinical studies followed by universal bond adhesive. The lowest bonding results belonged to futura u bond.[30]

This result was explained and agreed with **Isabella A. Gomes et al,** [31] who repoted that the bond strength values obtained for the two-step total etch adhesive system were greater when compared to those observed with the self-etch systems . They compared the bond strength of different adhesive systems to dentin substrate after 24h storage in distilled water (DW), or 180-day aging protocols in DW, artificial saliva (AS), citric acid (CA), or thermal cycling (TC).

The high values obtained from group III also was explained and agreed with Nassar et al. who found removal of the smear layer and opening of the dentinal tubules by means of complete acid etching allow free diffusion of the adhesive agents and may contribute to the bond effectiveness of etch-and-rinse adhesive systems.[32]

On the other hand**, Daneshkazemi et al,** [33] demonstrated that Scotch bond Universal using self-etch strategy has the highest bond strength values and single bond II using total etch strategy had the lowest bond strength values.

The Tetric N Bond Universal basically one-step self-etch adhesives that can be used under diferent adhesion strategies, but the higher values obtained over that that of Futurabond U adhesive because it contain 10-MDP (10-Methacryloyloxydecyl dihydrogen phosphate) as a functional monomer.[34]

Muñoz et al, [35] agreed with this hypothesis as compared the longevity results of Single bond universal (contains both MDP-VBCP) with Adper single bond 2 (contains only VBCP), two materials with similar compositions, the only difference being the presence of MDP in the former, it seemed that the association MDP-VBCP enhanced the bonding ability, since Single bond universal in both etching modes showed stable bonds even after 6 months of water storage

On the other hand This comes in disagreement with **Wagner et al**. [36] who stated that there was not a statistically significant difference in mean micro bond strength when Single bond universal in self-etch mode was compared to All bond universal; which only contains MDP. They reported that The VBCP may compete with the MDP monomer for Ca-bonding sites located in hydroxyapatite and due to its high molecular weight, could even prevent VBCP approximation during polymerization.

In the current results, Different modes of failure were recorded after micro-tensile and micro-shear bond strength test.

For Group I , there was increased percentage of mixed mode of failure for both micro-tensile and micro-shear bond strength tests. This become in agreement with Campos MD et al., who studied Influence of acid etching and universal adhesives on the bond strength to dentin.[37]

There was no cohesive mode of failure in the micro shear tested specimens this could result from high stress concentrated at the dentin-composite interface in contrast to micro tensile bond test in which the loading force passes through the tooth substrate and resin composite before reaching the adhesive interface, with subsequent stress concentration in these materials which lead to frequent cohesive mode of failure. [38,39]

These results are in agreement with previous studies which suggested that the mode of failure is an indicator to the strength of bond. Adhesive failure usually indicated low bond strength while cohesive failure resembles high bond strength values.[40]

VI. Conclusions

Under the limitations of this study, the results suggest that:

1- Both the micro-shear and micro- tensile bond strength test are highly reliable and accepted for bond strength testing.

2- The total etch technique is the gold standard method for bonding.

3- According to the mode of failure, the highest percentage was for mixed failure showing that all the tested adhesives provide good adhesion to the tooth structure, but the best results goes to Swiss TEC SL Bond (etch and rinse technique).

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Table 1: materials used in this study:

Material	Chemical Composition	Manufacture	Batch no.
Futurabond U adhesive Fig(IV -2)	Water, Ethanol, Silicium dioxide, Acid modified methacrylate (methacrylate ester),HEMA(2-hydroxyethyl methacrylate), Camphorquinone	Voco GmbH, Cuxhaven, Germany	**184095**
Tetric NBond Universal Fig (IV -3)	Bis-acrylamide derivatives, water, 10 bis-methacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxy alkyl methacrylamide,Vitrebond Copolymer,highly dispersed silicon dioxide, catalysts and stabilizers	Ivoclar Vivadent, Schaan, Liechtenstein	**690104**
Swiss TEC SL Bond Fig (IV -4)	HEMA, Hydroxpropyl methacrylate, Glycerol dimethacrylate, Polyalkenoate methacrylized, UDMA	Coltene/Whale dent AG	**J77223**
Filtek Z350 XT Nanocomposites	Resin [bis-GMA, UDMA, TEGDMA, bis-EMA. Filler (silica filler,	3M ESPE/USA	**NC68412**

Figure 1: Futurabond U


Figure 2: Tetric n bond

Figure 3: Swiss TEC SL Bond

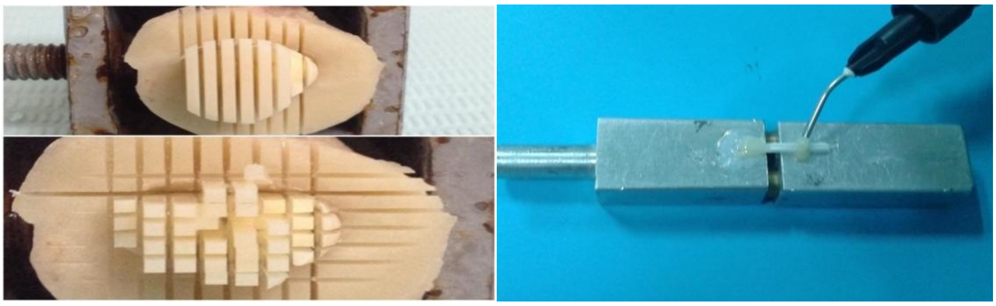
Figure 4: specimen for micro tensile bond strength

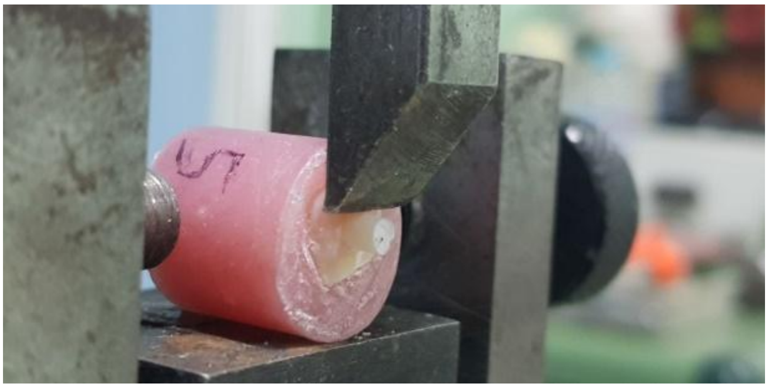

Figure 5: specimen for micro shear bond strength

Table 2: showing µ-tensile bond strength results for three tested adhesive systems.

Variables		Statistics		
		Mean±SD	95% confidence intervals	
			Lower	Upper
Adhesive groups	Gr_I	25.31^{B}±5.2	22.43	28.19
	Gr_II	26.7^{B}±7.52	22.54	30.87
	Gr_III	38.01^{A}±8	33.58	42.44
ANOVA test	P value	<0.0001*		

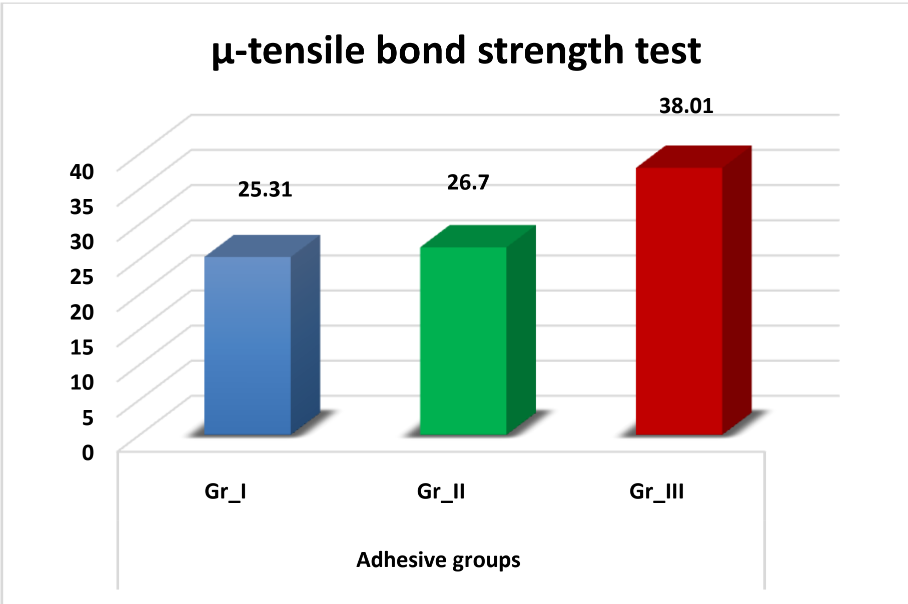

Figure 6: Column chart representing the mean values of µ-tensile bond strength between all adhesive groups

Table 3: showing µ-shear bond strength results for three tested adhesive systems.

Variables		Statistics		
		Mean±SD	95% confidence intervals	
			Lower	Upper
Adhesive groups	Gr_I	7.09^{B}±1.87	6.06	8.13
	Gr_II	11.59^{A}±1.82	10.58	12.59
	Gr_III	12.07^{A}±2.08	10.91	13.22
ANOVA test	P value	<0.0001*		

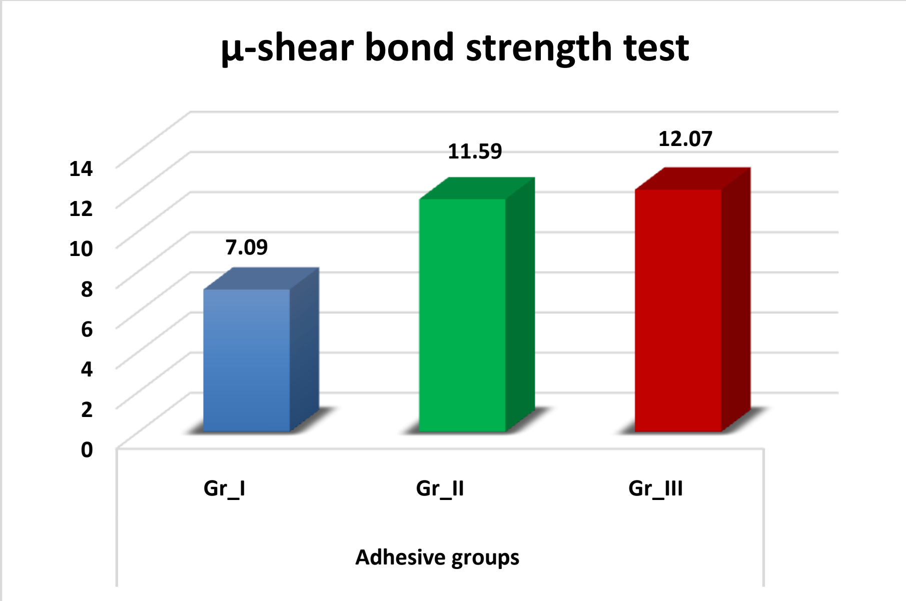

Figure 7: Column chart representing the mean values of µ-shear bond strength between all adhesive groups

Table 4: Frequent distribution of failure mode pattern (%) as function of adhesive groups for micro tensile bond strength test subgroup(subgroup A)

Variables		Failure mode pattern			Statistics
		Adhesive	**Cohesive**	**Mixed**	**P value**
Adhesive groups	**Gr_I**	30.77%	0%	69.23%	<0.0001 *
	Gr_II	44.44%	11.11%	44.44%	
	Gr_III	15.39%	15.38%	69.23%	

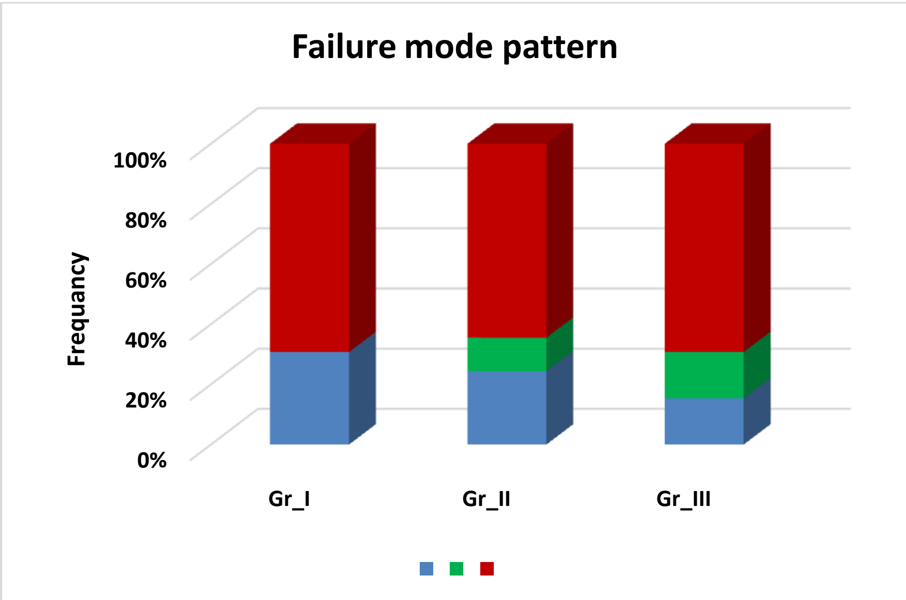

Figure 8: Stacked column chart showing frequent distribution of failure modes analysis (%) as function of adhesive groups (subgroup A)

Table 5: Frequent distribution of failure mode pattern (%) as function of adhesive groups for micro shear bond strength test subgroup(subgroup B)

Variables		Failure mode pattern			Statistics
		Adhesive	**Cohesive**	**Mixed**	**P value**
Adhesive groups	**Gr_I**	67%	0%	33%	<0.0001 *
	Gr_II	33%	0%	67%	
	Gr_III	25%	0%	75%	

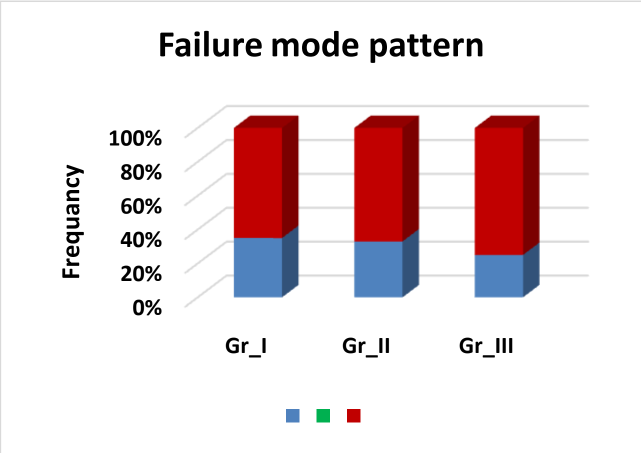

Figure 9: Stacked column chart showing frequent distribution of failure modes analysis (%) of micro-shear test as function of adhesive groups (subgroup B)

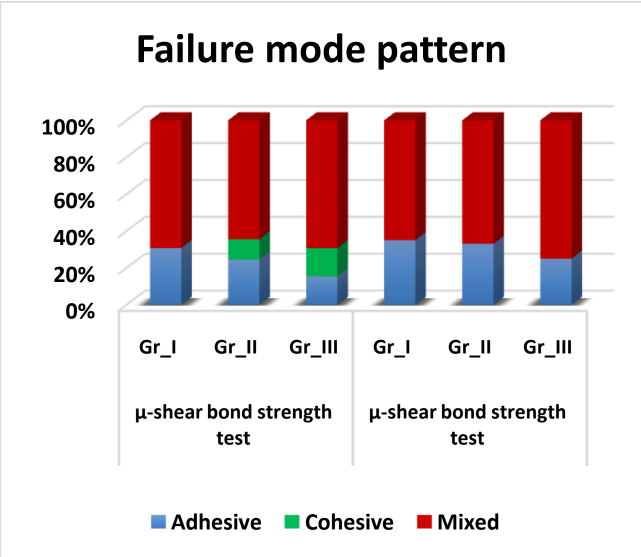

Figure 10: stacked Column chart showing frequent distribution of failure modes analysis (%) of both µ-tensile and µ-shear bond strength as function of adhesive groups

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