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Abstract

Aim of Study :

The aim of the study is directed to evaluate effect of different bonding stategies on clinical behavior, micro tensile bond strength, and depth of cure of bulk fill resin composites Conclusions

Under the results of the present study, the following conclusions could drowns:

- The total-etch adhesive approach provides higher bond strength, and depth of cure, with noticeably better adaptability and lower postoperative hypersensitivity.
- The self-etch adhesive approach proved the lower bond strength, depth of cure, and higher postoperative hypersensitivity with simplified bonding steps when compared with the total-etch approach.
- Post-operative hypersensitivity is affected by the bonding procedures.
- The self- adhesive approach of Bioactive Activa restorations proved the lower bond strength, depth of cure, and lower postoperative hypersensitivity with simplified bonding steps when compared with the bonded Bulkfill restorations.
- Bulk fill composite with bulk packing in 4mm thickens increment together with total-etch adhesive is considered a practical approach in class II cavity restorations regarding bond strength, depth of cure, esthetic, and marginal adaptability

Sample size calculation

According to a previous study by Fahim et al. 2019 (122), the sample size calculation that was performed G power test analysis recorded a total of 66 samples. The total sample size was assigned to 3 main groups (n=22) according to the type of restorative materia

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Introduction

The increasing attractiveness of tooth-colored restoration has promoted research in this paeicular area of restorative dentistry in the last few years. Resin composites are used extensively in tooth restoration because they are popular with both dentists and patients. Amongst other benefits, their color is similar to that of a real tooth, they have good physical properties and can be used in conservative cavity preparation 'l.

- Different methods had been formulated for composite resin insertion including incremental and bulk fill techniques. One obvious advantage for the incremental technique is the limitation of the thickness of resin, which provides adequate light peneration and subsequent polymerization that results in enhanced physical properties and improved marginal adaptation ')Another reason to use the incremental technique is to decrease the amount of shrinkage occurring during polymerization, which is beneficial because the developing stress can cause cuspal deformation with resulting sensitivity or microcracks in resin or tooth structure. The stress can also cause adhesive failure at the tootYresin interface resulting in marginal gap, microleakage, and secondary caries(34)
- Despite these benefits, the incremental technique has disadvantages, that may include; the possibility of incorporating voids or contamination between composite layers, bond failures between increments, difficulty in placement because of limited access in large cacity preparations, and the increased time required to place and polymerize each layer(5 '
- Lately, there is a direction to decrease ie number of increments for direct composite restoration and support the use of a bulk fill technique. Several manufacturershave developed *buk fill" resin composites that can be applied to the cavity in a thicknessof 4 mm with enhanced curing and controlled shrinkage(7.
- Bulk fill resin composites have been proven in several studies to enable restoration in thick layers, up to 4mm, maintaining the mechanical properties and the degree of conversion within the whole increment".Besides, decrease polymerization shrinkage stress and reduced cusp deflection in standardized class II cavities"
- Adhesive dentistry is a rapidly changing and evolving field. The basic principle of adhesion of composite resins to dental substrate is based on exchange processes in which inorganic dental material is replaced *Eur. Chem. Bull.* 2023,12(issue 11),273-301

by synthetic resin 'l' The establishment of effective inter locking occurs when the adhesive penetrates into the intratubular and intertubular dentin'l' During dentin acid- etching, the minerals content of the dentin surface is removed, and the collagen fibrils remain supported by water'l". After decades of evaluation, adhesives may include different formulations and, consequently, their bond values may vary in relation to dental substrate. Currently there is a tendency to simplify bonding procedures which introduced the self-etching adhesive concept"3'

- Bioactive materials are "smart," moisture-friendly. By responding to ambient conditions in the mouth, they play an active role in the oral environment and stimulate formation of a layer of protective, apatite-like crystal deposits at the material-tooth interface that forms a natural bond between the material and living tissue l°.Among this new class of materials is ACTIVA bioactive restorative material(self adhering composite).ACTIVA is a new category of strong, esthetic, smart bioactive composite resins that release and recharge calcium, phosphate and fluoride ions and are more bioactive than glass ionomers ¹.

Micro tensile bond strength is one of the most important properties used to compare between the restorative materials that subjected to high masticatory forces to evaluate the long term durability of resin hard tissue bonds(l'

The intensity of light at a given depth and for a given irradiance period is a critical factor in determining the extent of reaction of monomer into polymer, and significantly associated with values of mechanical properties, biocompatibility, color stability and would therefore be expected to be associated with clinical success of the restoration l'.

New clinical criteria were approved by the FDI World Dental Federation, these criteria were categorized into three groups: esthetic parameters (four criteria), functional parameters (six criteria) and biological parameters (six criteria). Each criterion can be expressed with five scores, three for acceptable and two for non-acceptable (one for reparable and one for replacement). The criteria have been used since 2007(18919) From the previous review; it was assumed that it would be importance to evaluate Micro tensile bond strength, depth of cure and FDI criteria of three types of bulk fill resin composite with different bonding strategies at different storage times.

Results

I. In vitro Results:

1. Microtensile bond strength results:

1.1. Microtensile bond strength concerning the tested material:

The microtensile bond strength results concerning the type of restorative material are recorded in mega Pascal

(MPa) and represented as a mean and standard deviation (SD) in (Table 3) and shown in (Figure 21).

The microtensile bond strength results reveal that the difference in the bond strength between the tested

restorative materials was statistically significant at 24 hours, 3 months, and 6 months of follow-up periods

with a level of significance of (P<0.05) as indicated by the One-way ANOVA test.

Among the groups, Tukey's HSD test showed that the microtensile bond strength results exhibited a statistically significant difference between Tetric N Ceram/ Total-etch and the other two tested groups as well as between X-tra fil/ Self-etch and Activa Bioactive at 24 hours, 3 months, and 6 months of follow-up periods with a level of significance of (P<0.000).

The higher mean \pm SD values were recorded with the Tetric N Ceram/ Total-etch group (26.6 \pm 0.55 MPa), (18.9 \pm 0.79 MPa), and (14.8 \pm 1.39 MPa) followed by X-tra fil/ Self-etch group (21.5 \pm 0.85 MPa), (14.3 \pm 0.49 MPa), and (10.9 \pm 0.59 MPa) at 24 hours, 3 months, and 6 months of follow-up periods respectively. While the lower significant mean \pm SD values were recorded with the Activa Bioactive (14.9 \pm 0.41 MPa), (10.2 \pm 0.38

MPa), and (6.9±0.56 MPa) at 24 hours, 3 months, and 6 months of follow-up periods respectively.

Materials and methods

Materials and Methods

I. Materials

The materials used in this study were 3 different bulk-fill composites restorative materials of different compositions and 2 different bonding agent materials of different bonding strategies as follows:

A. Bulk-fill composite materials

1) Nano-optimized bulk-fill posterior resin composite (Tetric N-Ceram) in Etch & rinse mode.

- 2) Bulk-fill microhybrid posterior composite (X-tra fil, VOCO) in SE mode.
- 3) Bioactive bulk-fill resin composite (Activa BioActive Restorative) in self-adhesive.

B. Bonding agents

- 1) Total-etch adhesive (Tetric N-bond)
- 2) Self-etch adhesive (Universal adhesive Futurabond U).
- All materials that were used in this study and their category, composition, and manufacturer were listed in Table (1) and shown in Figures (1-5)

Category	Material name	Composition	Manufacturer
1. Posterior Bulk-fill compo site	Material name Tetric N-Ceram bulk-fill composite (Figure 1)	 Composition Monomer matrix: UDMA (19-21% weight) Inorganic fillers: 75-77% weight or 53-55% volume Glass filler: 0.4 – 0.7 micron YbF3: 80 – 120 nm Mixed oxide: 170 – 	Manufacturer Ivoclar Vivadent dental product, Liechtenstein
2. Bulk-fill nanohybrid posterior composite	X-tra microhybrid bulk- fill composite (Figure 2)	 230 nm Nano-hybrid. Barium-boron-alumino-silicate glass (2–3 µm) 84 % inorganic fillers. Organically modified silicic acid 10-25%. Resin matrix: Bis-GMA, UDMA, TEGDMA 	VOCO; Cuxhaven, Germany
3. Self- adhesive Bioactive	Activa BioActive Restorative Bulk-fill composite	Resin matrix: Blend of diurethane and	Pulpdent Corporation,

Bulk-fill composite	(Figure 3)	other methacrylates with modified polyacrylic acid. Filler: Silica,	Watertown, MA, USA
		amorphous, and Sodium fluoride.	
4. Total- etch adhesive	Tetric N-bond (Figure 4)	HEMA, UDMA, Bis- GMA, phosphoric acid acrylate, catalysts, and stabilizers. Ethanol. Silica nanofillers: <1% weight.	Ivoclar Vivadent dental product, Liechtenstein
5. Self- etch adhesive	Universal adhesive Futurabond U (FbU) (Figure 5)	Liquid 1: Acidic adhesive monomer, HEMA, BIS GMA, HEDMA, UDMA, Catalyst. Liquid 2: Ethanol, Initiator, catalyst.	VOCO; Cuxhaven, Germany







Figure (2): X-tra fil bulk-fill microhybrid posterior composite.



Figure (3): Activa BioActive Restorative bulk-fill composite.



Figure (4): Tetric N-bond (Total-etch adhesive).



Figure (5): Futurabond U (Self-etch adhesive)

Table (2): FDI Criteria

EDI	au!4au!a	Clinically very	Clinically good	Clinically	Clinically	Clinically
FDI	criteria	good		sufficient	unsatisfactory	poor
Esthetic	Color	The translucency	The translucency and	The translucency	The	The shade
parameter	stability and	and shade of the	shade of the filling	and shade of the	translucency	difference
	translucency	filling confound	present a very slight	filling do not	and shade of	is severe
		perfectly with the	difference with the	confound	the filling do	
		surrounding tooth	surrounding tooth	perfectly with the	not confound	
		structure	structure	surrounding tooth	perfectly with	
				structure but the	the	
				difference remains	surrounding	
				acceptable	tooth structure.	
					The difference	
				~	is important	FF1 (111)
Functional	Marginal	Harmonious	Marginal gap (50 μ m)	Gap	3.4 Gap	The filling
parameter	adaptation	outline; no gaps;	3.2 Small marginal	$<150 \ \mu m, \text{ not}$	$>250 \ \mu m \text{ or}$	18 loose but
		no discoloration	fracture, removable by	removable $3.3.2$.	dentin/base	in situ
			polisning	Several small	exposed	
				fractures	5.4 Chip	
				mactures	demoging	
					marging	
					3.4 Notable	
					enamel or	
					dentin wall	
					fracture	
Biological	Postoperative	No	Low hypersensitivity	Premature/	Premature/	Very
parameter	sensitivity	hypersensitivity	for a limited period of	slightly more	very intense	intense,
			time	intense	4.4. Extremely	acute
				4.3. Delayed/	delayed/weak	pulpitis or
				weak sensitivity;	with	non-vital;
				no subjective	subjective	endodontic
				complaints;	complaints	treatment
				no treatment	4.4. Negative	necessary;
				needed	sensitivity;	restoration
					intervention	has to be
					necessary but	replaced
					not	
					replacement	

• In vitro intervention:

A. Micro tensile bond strength test:

I. Specimens preparation:

1.Molds fabrications:

a) Mold fabrications for preparation of standardized acrylic block:

A specially fabricated cylindrical plastic mold of internal diameter

15mm and 20mm in height was fabricated. A separating medium was used to coat the internal surface of the mold. The mold was filled with self-curing acrylic resin, the base of the mold rested on a glass slab in order to obtain a flat smooth surface base. Each tooth was embedded vertically in the mold while the acrylic resin still in the dough stage to the level of the cemento-enamel junction of the tooth leaving the occlusal surface projecting above the surface of the mold⁽⁵²⁾ (Fig.5). Figure (5): A specially fabricated mold with the



standardized acrylic blocks

b) Mold fabrications for preparation of specimens:

A cylindrical mold was fabricated to restore the specimens with bulk fill composite resin. This mold consists of an external plastic ring (A) and internal splitted Teflon disc (B) (Fig.6).

The external plastic ring has internal diameter of 25 mm, and 4 mm height. It held the halves of the splitted Teflon dis (B) snugly fit together. The internal splitted Teflon disc has an external diameter of 25mm and central hole of 4mm x 4mm dimension (Fig.7). Midway marked was done at 2 mm height in the internal surface of the central hole to allow two equal increments of composite resin. The splitted disc was used to restore specimens with 4mm of composite resin ⁽⁵³⁾.



Figure (6): Teflon split mold

- A. External plastic ring
- B. Internal splitted mold



Figure (7): External ring with an internal splitted mold for produce specimens

Preparation of the occlusal dentin surface:

The teeth were removed from the mold (Fig.8). Grinding machine with a grit carborundum disc was used to wet grind two mm from the occlusal surface projecting above the acrylic surface by using caliber. The dentin surface was further abraded using a #600 grit wet silicon carbide abrasive paper for 60 seconds under running water to produce a polished surface and uniform smear layer ⁽³⁾. Then the specimens were washed in running tap water and blotted using wet gauze



Figure (8): The tooth after removed from the mold



Figure (9): The prepared tooth specimen embedded in the acrylic block

d) Restoration protocols: ^(9, 18)

 After occlusal surface preparation the teeth in each main group were restored according to the assigned restoration protocol as follows:

Group I: Total-etch adhesive system was used for dentine surface treatment by etch was applied for 20 s on the flat dentin surfaces, then rinsed thoroughly with an oil-free stream of water for 10 s. The excess water was removed with gently drying leaving the dentin surfaces moist. The adhesive was applied, gently dried and light polymerized using a LED light-curing unit with power output of 1000mW /cm^{2**} (figure 9) for 20 seconds according to the manufacture's instructions.

Group II: Activa BioActive Restorative Bulk-fill resin composite was applied

by the following selective etching for enamel 37% phosphoric acid etch was applied for 15 seconds then rinsed and dried.

- No adhesive was applied.
- BioActive Restorative Bulk-fill resin composite was placed in a single increment (4 mm bulk-filling) and packed to the cavity with composite applicator.
- Polymerization was performed with an LED unit for 40 seconds

Group III: Self-etch adhesive protocol was used for dentine surface treatment by application of futurabond self etch adhesive followed by application of x_tra microhyprid Bulk-fill resin composite.



Figure (11): The acrylic block with the bonded composite resin



Figure (10): LED light curing unit

e) Storage of specimens:

The specimens were stored in distilled water at 37°C in an incubator with

100% humidity at different storage time (24 hours , one month, and six months) until microtensile bond strength testing was performed.

f) Beam preparation for microtensile bond strength testing: ⁽¹⁸⁾

- Each tooth in each main group was positioned on the cutting machine and sliced into a succession of 1X1 mm-thick slabs while being thoroughly cooled by water to obtain 3 specimens from each tooth. (Figure 12 and 13)
- The slab thickness was confirmed by a digital caliper ^(v).
- The tooth was rotated 90 degrees and lengthwise sections of restoration and dentine were cut using a diamond disc.
- The prepared slab samples of each assigned group were stored in artificial saliva for 24 hours, 3 months, and 6 months before testing.
- Then microtensile bond strength testing was performed on each slab sample after different storage times.



Figure(12): IsoMet 4000 Microsaw Germany Cutting Machine



Figure(13): A beam consisting of a composite, bonding agent and dentin parts.

2.Microtensile bond strength measurement:

- After the end of each storage period, each composite sample in each main group was subjected to microtensile bond strength testing.
- Each slab sample had its ends connected using cyanoacrylate adhesive to a specifically created, modified Ciucchi's jig.
- The final assembly was then mounted on a universal testing machine (vi).
- Through an aluminum rod attached to the end of the jig, the force was applied to the moving component.
- A tensile load was applied via the materials testing machine at a crosshead speed of 0.5 mm/min.
- The applied tensile force resulted in debonding along the substrateadhesive interface.
- The load required for debonding of each stick was recorded in megapascal (MPa).
- The data was recorded using computer software ^(vii).
- The microtensile bond strength ($\mu\delta$) was measured as Newton divided by the area and calculated using the following equation:

$$\mu\delta = L/A,$$

Where; L: is the load (N) at the failure of the sample and A: is the interfacial area of the sample (mm^2) as measured with the digital caliper.



Figure (14): Attachment jig



Figure (15): spicemen Attachment to jig



Figure (16): Jig mounted on the universal testing machine



Figure (18): Schematic diagram showing the beam before (left) and after(right) being stressed to failure

B. Depth of Cure by Vickers Microhardness: ⁽¹¹⁹⁾

1. Sample preparation:

- The composite samples for each main group were created using a 4 mm long by 4 mm internal diameter opaque Teflon mould. (Figure 19).
- The composite restoration of each main group was then filled in bulk (one increment) with each material once the mould had been set up on a glass slide coated with a Mylar strip.
- A glass slide was placed on top of the composite-filled upper surface of the mould before it was coated with a Mylar strip.
- The sample was then polymerized for 20 seconds while the light-curing unit's tip remained in close proximity to the 1.2-mm-thick glass slide to maintain a constant distance from the sample.
- Each sample was taken from the mould after polymerization.(Figure 20)
- The samples were kept in artificial saliva in refrigerator for 24 hours, 3 months, and 6 months before microhardness testing.





Figure (19) : Teflon mould.



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Figure (20): Bulk-fill Composite samples for depth of curing test.

2. Vickers Microhardness measurement: (119)

- After the end of each storage period, each composite sample in each main group was subjected to microhardness testing.
- Vickers microhardness tester device ^(viii) was used to measure the top and bottom surface hardness of each 4-mm high sample during testing.
- A force of 5 N was applied to the Vickers pyramid measuring indenter for five seconds as it was pressed against the composite sample. (Figure 21)
- To reduce measurement errors within a sample, the surface Vickers hardness (HV) of each sample was tested three times.
- The depth where HV equals the surface value multiplied by an arbitrary ratio, typically 0.8 (HV-80%), is where the depth of curing is typically defined as the thickness of the composite that is properly polymerized.
- Therefore, the HV of the lower surface of each sample was compared to the value of the upper surface, and it was noted when it fell below HV-80%



Figure (21): Depth of curing test by Vickers microhardness test.

Results

I. In vitro Results:

1. Microtensile bond strength results:

1.1. Microtensile bond strength concerning the tested material:

The microtensile bond strength results concerning the type of restorative material are recorded in mega Pascal (MPa) and represented as a mean and standard deviation (SD) in (**Table 3**) and shown in (**Figure 28**).

The microtensile bond strength results reveal that the difference in the bond strength between the tested restorative materials was statistically significant at 24 hours, 3 months, and 6 months of follow-up periods with a level of significance of (P<0.05) as indicated by the One-way ANOVA test.

Among the groups, Tukey's HSD test showed that the microtensile bond strength results exhibited a statistically significant difference between Tetric N Ceram/ Total-etch and the other two tested groups as well as between X-tra fil/ Self-etch and Activa Bioactive at 24 hours, 3 months, and 6 months of follow-up periods with a level of significance of (P<0.000).

The higher mean \pm SD values were recorded with the Tetric N Ceram/ Total-etch group (26.6 \pm 0.55 MPa), (18.9 \pm 0.79 MPa), and (14.8 \pm 1.39 MPa) followed by X-tra fil/ Self-etch group (21.5 \pm 0.85 MPa), (14.3 \pm 0.49 MPa), and (10.9 \pm 0.59 MPa) at 24 hours, 3 months, and 6 months of follow-up periods respectively. While the lower significant mean \pm SD values were recorded with the Activa Bioactive (14.9 \pm 0.41 MPa), (10.2 \pm 0.38 MPa), and (6.9 \pm 0.56 MPa) at 24 hours, 3 months, and 6 months of follow-up periods respectively.

Variable	Tetric N Ceram/ Total-etch	X-tra fil/ Self-etch	Activa Bioactive	F-ratio	p-value		
24 hours	26.6±0.55 ^A	21.5±0.85 ^B	14.9±0.41 ^C	689.63	0.000*		
Sig.							
between	P1<0.0	P1<0.000*, P2<0.000**, P3<0.000*					
groups							
3 months	18.9±0.79 ^A	14.3±0.49 ^B	10.2±0.38 ^C	439.68	0.000*		

Table (3): Comparison of microtensile bond strength results concerning material:

Sig. between groups	P1<0.0	000*, P2<0.0	00**, P3<0.0	00*	
6 months	14.8±1.39 ^A	10.9±0.59 ^B	6.9±0.56 ^C	140.83	0.000*
Sig. between groups	P1<0.0	000*, P2<0.0	00**, P3<0.0	00*	

*; Significant at p<0.05. ns; non-significant at p>0.05

; Different uppercase letters mean statistically significant.

- P1; between Tetric N Ceram and X-tra fil.
- P2; between Tetric N Ceram and Activa Bioactive.
- P3; between X-tra fil and Activa Bioactive.



Figure (28): Comparison of microtensile bond strength results concerning material

1.2. Microtensile bond strength concerning the follow-up time:

The microtensile bond strength results concerning the follow-up time are recorded in mega Pascal (MPa) and represented as a mean and standard deviation (SD) in (**Table 4**) and shown in (**Figure 29**).

The microtensile bond strength results reveal that the difference in the bond strength between the different follow-up periods (24 hours, 3 months, and 6 months) was statistically significant for the different tested materials with a level of significance of (P<0.05) as indicated by the One-way ANOVA test.

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Among the groups, Tukey's HSD test showed that the microtensile bond strength results exhibited a statistically significant difference between 24 hours and the other two tested periods (3 and 6 months) as well as between 3 months and 6 months of follow-up periods with a level of significance of (P<0.05).

The higher mean \pm SD values were recorded with the Tetric N Ceram/ Total-etch group at 24 hours (26.6 \pm 0.55 MPa) followed by 3 months (18.9 \pm 0.79 MPa), and 6 months (14.8 \pm 1.39 MPa). Also, the X-tra fil/ Self-etch group recorded the higher mean \pm SD values at 24 hours (21.5 \pm 0.85 MPa), followed by 3 months (14.3 \pm 0.49 MPa), and 6 months (10.9 \pm 0.59 MPa). Furthermore, the higher significant mean \pm SD values were recorded for the Activa Bioactive group at 24 hours (14.9 \pm 0.41 MPa), followed by 3 months (10.2 \pm 0.38 MPa), and 6 months (6.9 \pm 0.56 MPa) respectively.

 Table (4): Comparison of microtensile bond strength results of each tested material at different follow-up periods:

Variable	24 hours	3 months	6 months	F-ratio	p-value		
Tetric N Ceram/ Total-etch	26.6±0.55 ^A	18.9±0.79 ^B	14.8±1.39 ^C	301.63	<0.000*		
Sig. between groups	P1<0.0	P1<0.000*, P2<0.000**, P3<0.000*					
X-tra fil/ Self-etch	21.5±0.85 ^A	14.3±0.49 ^B	10.9±0.59 [°]	526.32	0.000*		
Sig. between groups	P1<0.0						
Activa Bioactive	14.9±0.41 ^A	10.2±0.38 ^B	6.9±0.56 ^C	612.10	<0.000*		
Sig. between groups	P1<0.0	P1<0.000*, P2<0.000**, P3<0.000*					

*; Significant at p<0.05. ns; non-significant at p>0.05

; Different uppercase letters mean statistically significant.

- P1; between 24 h Ceram and 3 months.
- P2; between 24 h Ceram and 6 months.
- P3; between 3 months and 6 months.



Comparison of microtensile bond strength results of each tested material at different follow-up periods

1.3. Microtensile bond strength concerning material and time:

The microtensile bond strength results concerning the type of restorative material and the different follow-up periods are represented in (**Table 5**) and shown in (**Figure 30**).

With regard to material, the overall statistical results of the microtensile bond strength between the tested restorative materials were statistically significant at 24 hours, 3 months, and 6 months of follow-up periods with a level of significance of (P=0.000) as indicated by the Two-way ANOVA test. Also, the overall statistical results showed that the difference between the averages of some groups is big enough to be statistically significant (P<0.05) as indicated by the Two-way ANOVA test.

With regard to time, the overall statistical results of the microtensile bond strength of each restorative material were statistically significant ($P=0.000^*$) as indicated by the One-way ANOVA test. Also, the overall statistical results showed that the difference between the averages of some groups is big enough to be statistically significant (P<0.05) as indicated by the Two-way ANOVA test.

However, the interaction between the different tested materials and the different follow-up periods showed that the difference between the averages of all groups is not big enough to be statistically significant as indicated by the Two-way ANOVA test (P<0.000).

Table (8): The overall comparison of the microtensile bond strength results of each tested

 material at different follow-up periods:

Source	DF	Sum of Square (SS)	Mean Square (MS)	F Statistic (df ₁ ,df ₂)	P-value
Factor A -	2	1058.0275	529.0137	984.2116	0*
rows (A)				(2,63)	
Factor B -	2	1266.79	633.395	1178.4093	0*
columns (B)				(2,63)	
Interaction	4	35.815	8.9538	16.6581	<0.0001*
AB				(4,63)	
Error	63	33.8625	0.5375		
Total	71	2394.495	33.7253		

*; Significant at P<0.05. ns; non-significant at P>0.05



Figure (30): The overall comparison of the microtensile bond strength results of each tested material at different follow-up periods

2. Depth of cure (Hardness):

2.1. Depth of cure concerning the tested material:

The DOC results concerning the type of restorative material is measured by top-bottom

Vickers microhardness and recorded in Gega Pascal (GPa) and represented as a mean and standard deviation (SD) in (**Table 6**) and shown in (**Figure 31**).

The depth of cure results reveals that the difference in the DOC between the tested restorative materials was statistically significant at 24 hours, 3 months, and 6 months of follow-up periods with a level of significance of (P<0.05) as indicated by the One-way ANOVA test.

Variable	Tetric N Ceram/ Total-etch	X-tra fil/ Self-etch	Activa Bioactive	F-ratio	p-value	
24 hours	88.2 ± 1.11^{A}	74.2±0.56 ^E	$69.2 \pm 0.90^{\circ}$	1108.48	0.000*	
Sig. between groups	P1<0.0)00*, P2<0.0	00**, P3<0.0	00*		
3 months	72.6±0.73 ^A	65.1±0.77 ^B	62.7±0.67 ^C	454.95	0.000*	
Sig. between groups	P1<0.0	P1<0.000*, P2<0.000**, P3<0.000*				
6 months	67.1±0.44 ^A	59.8±0.69 ^B	55.6±0.68 ^C	796.04	0.000*	
Sig. between groups	P1<0.0	000*, P2<0.0	00**, P3<0.0	00*		

Among the groups, Tukey's HSD test showed that the DOC results exhibited a statistically significant difference between Tetric N Ceram/ Total-etch and the other two tested groups as well as between X-tra fil/ Self-etch and Activa Bioactive at 24 hours, 3 months, and 6 months of follow-up periods with a level of significance of (P<0.000).

The higher mean \pm SD top-bottom microhardness values were recorded with the Tetric N Ceram/ Total-etch group (88.2 \pm 1.11 GPa), (72.6 \pm 0.73 GPa), and (67.1 \pm 0.44 GPa) followed by X-tra fil/ Self-etch group (74.2 \pm 0.56 GPa), (65.1 \pm 0.77 GPa), and (59.8 \pm 0.69 GPa) at 24 hours, 3 months, and 6 months of follow-up periods respectively. While the lower significant mean \pm SD top-bottom microhardness values were recorded with the Activa Bioactive (69.2 \pm 0.90 GPa), (62.7 \pm 0.67 GPa), and (55.6 \pm 0.68 GPa) at 24 hours, 3 months, and 6 months of follow-up periods respectively.

Table (6): Comparison of the DOC (top-bottom microhardness) results in concerningEur. Chem. Bull. 2023, 12(issue 11), 273-301295

material:

*; Significant at p<0.05. ns; non-significant at p>0.05

; Different uppercase letters mean statistically significant.

- P1; between Tetric N Ceram and X-tra fil.
- P2; between Tetric N Ceram and Activa Bioactive.
- P3; between X-tra fil and Activa Bioactive.



Figure (31): Comparison of DOC results concerning material

2.2. Depth of cure concerning the follow-up time:

The DOC results concerning the follow-up time is measured by top-bottom Vickers microhardness recorded in Giga Pascal (GPa) and represented as a mean and standard deviation (SD) in (**Table 7**) and shown in (**Figure 32**).

The DOC results reveal that the difference in the depth of cure between the different follow-up periods (24 hours, 3 months, and 6 months) was statistically significant for the different tested materials with a level of significance of (p<0.05) as indicated by the One-way ANOVA test.

Among the groups, Tukey's HSD test showed that the depth of cure results exhibited a statistically significant difference between 24 hours and the other two tested periods (3 and 6 months) as well as between 3 months and 6 months of follow-up periods with a level of significance of (p<0.05). *Eur. Chem. Bull.* 2023,12(issue 11),273-301 296 The higher mean \pm SD top-bottom microhardness values were recorded with the Tetric N Ceram/ Total-etch group at 24 hours (88.2 \pm 1.11 GPa), followed by 3 months (72.6 \pm 0.73 GPa), and 6 months (67.1 \pm 0.44 GPa). Also, the X-tra fil/ Self-etch group recorded the higher mean \pm SD top-bottom microhardness values at 24 hours (74.2 \pm 0.56 GPa) followed by 3 months, (65.1 \pm 0.77 GPa), and 6 months (59.8 \pm 0.69 GPa) respectively. Furthermore, the higher significant mean \pm SD top-bottom microhardness values were recorded with the Activa Bioactive at 24 hours (69.2 \pm 0.90 GPa), (62.7 \pm 0.67 GPa) followed by 3 months, and 6 months (55.6 \pm 0.68 GPa) respectively.

Variable	24 h	3 months	6 months	F-ratio	P-value
Tetric N Ceram/	88.2±1.11 ^A	72.6±0.73 ⁴	A 67.1±0.44 ^A	1640.38	0.000*
Total-etch					
Sig. between groups	P1<0.				
X-tra fil/ Self-etch	$74.2{\pm}0.56^{\rm B}$	65.1 ± 0.77^{B}	$59.8{\pm}0.69^{B}$	1032.02	0.000*
Sig. between groups	P1<0.				
Activa Bioactive	69.2±0.90 ^C	62.7±0.67 ^C	55.6±0.68 ^C	715.47	0.000*
Sig. between groups	P1<0.	000*, P2<0.0	000**, P3<0.0	000*	

Table (7): Comparison of the DOC (top-bottom	microhardness)) results of each tested
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material at different follow-up periods:

*; Significant at p<0.05. ns; non-significant at p>0.05

; Different uppercase letters mean statistically significant.

- P1; between 24 h Ceram and 3 months.
- P2; between 24 h Ceram and 6 months.
- P3; between 3 months and 6 months.



Figure (32): Comparison of the DOC results of each tested material at different follow-up periods

2.3. Depth of cure concerning material and time:

The DOC results concerning the type of restorative material and the different followup periods are represented in (**Table 8**) and shown in (**Figure 33**).

With regard to material, the overall statistical results of the DOC between the tested restorative materials were statistically significant at 24 hours, 3 months, and 6 months of follow-up periods with a level of significance of (p=0.000) as indicated by the Two-way ANOVA test. Also, the overall statistical results showed that the difference between the averages of some groups is big enough to be statistically significant as indicated by the Two-way ANOVA test.

With regard to time, the overall statistical results of the DOC of each restorative material were statistically significant (p=0.000) as indicated by the One-way ANOVA test. Also, the overall statistical results showed that the difference between the averages of some groups is big enough to be statistically significant (p<0.05) as indicated by the Two-way ANOVA test.

However, the interaction between the different tested materials and the different follow-up periods showed that the difference between the averages of all groups is not big enough to be statistically significant as indicated by the Two-way ANOVA test (P=0.000).

Table (8): The overall comparison of DOC (top-bottom microhardness) results of each tested material at different follow-up periods:

Source	DF	Sum of	Mean	F Statistic	p-
		Square	Square	$(\mathbf{df_1}, \mathbf{df_2})$	value
		(SS)	(MS)		
Factor A -	2	2601.944	1300.972	2292.5863	0*
rows				(2,72)	
(material)					
Factor B -	2	3709.1528	1854.5764	3268.1538	0*
columns				(2,72)	
(time)					
Interaction	4	239.8398	59.9599	105.662	0*
AB				(4,72)	
Error	72	40.8578	0.5675		
Total	80	6591.7943	82.3974		

*; Significant at p<0.05. ns; non-significant at p>0.05



Figure (33): The overall comparison of DOC results of each tested material at different follow-up periods

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