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Effect of Surface Treatments of Ceramic Specimens on Shear Bond Strength of Orthodontic Brackets Using Two Different Prime Systems

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Abstract

To compare the shear bond strength (SBS) of metal brackets bonded to ceramic specimens with two surface treatment methods and two different adhesive prime types and to determine adhesive remnant index (ARI). **Materials and Methods:** This in-vitro study included 60 ceramic specimens which divided randomly into two groups of 30 specimens for each according to prime used (Assure® Plus and Transbond™ XT). The labial aspect of each specimen was treated by one of the following three surface preparation: no surface treatment (control group), air abrasion with 50µM aluminum oxide particles (AL_2O_3) and acid etching with 9.6% hydrofluoric acid (HF). Metal orthodontic brackets were bonded to ceramic specimens using Assure® Plus or Transbond™ XT adhesives. The surface roughness was measured by profilometer and then SBS was measured using a universal testing machine at a crosshead speed of o.5 mm/minute. The labial surfaces of specimens were inspected under a stereomicroscope, and the Adhesive remnant index (ARI) scores were determined. Raw data were analyzed using SPSS program. **Results:** The Al_2O_3 air abrasion with Assure® Plus group had a highest mean of SBS values while HF groups with Transbond™ XT adhesive or Assure® Plus give rise a significantly lower SBS values than that obtained for Al_2O_3 group. A significant difference was noted among the groups in the ARI scores. In AL_2O_3 group bonded with Transbond™ XT had a score 1 and 2 which was designated as mix-type failure, indicating a favorable failure mode. **Conclusion:** This study showed that air abrasion of ceramic specimens had a significant effect on SBS of metal brackets bonded to ceramic and the Transbond™ XT prime is a applicable bonding material for clinical use for ceramic restoration.

Introduction:

In the last years, the adult patients seeking orthodontic clinics has increased in number[1,2] and these patients have a different complex restoration like ceramic partial prosthodontics. These ceramic restorations are widely used in esthetics dentistry due to their multiple advantages such as esthetics, biocompatibility, low thermal expansion, decreased bacterial accumulation, color stability and resistance to fracture and abrasion[3-5]. However, difficulty may occur when orthodontic brackets bonded to ceramic prosthesis because conventional enamel bonding protocols may be ineffective for ceramic surface[6] and the adhesion process is hinder due to the presence of glazed layer[5,7-9]. The bonding failure rates of orthodontic brackets are 9.8% on ceramic restoration [5]. As a response to these problem, multiple surface treatments for ceramic restorations have emerged which are chemical, mechanical or combination of both that change the properties of ceramic and produce porous surface and enhance the bonding strength[8,10].

The most commonly used chemical methods include surface etching by hydrofluoric acid, phosphoric acid, maleic acid and saline[3,5,7-12]. Mechanical methods such as air abrasion by AL_2O_3 particles, Laser or diamond bur[13]. Several previous studies revealed that the HF acid etching with different concentrations is typically utilized to improve bracket bonding to traditional ceramics[14]. Sandblasting is a technique used aluminum oxide particles, generally 50 µm, which projected to produce abrasion by high air pressure on the surface of ceramic or another fixed prosthesis[15]. The adhesive materials that is used for bonding orthodontic brackets to enamel or ceramic surfaces should be withstand the forces of orthodontic treatment and masticatory forces and furthermore maintain the integrity of the enamel or ceramic surfaces when debonding of the brackets at the finish of the orthodontic treatment [16]. The Transbond™ XT adhesive material is considered the gold standards adhesive material that is used to compare and test the other orthodontic adhesive systems[9]. A recent advance in bonding material is the Assure®Plus [1,6,9] that it provide high bond strength to zirconia, ceramic, stainless steel, amalgam restoration, gold or irregular metal surface[9]. The objective of this study was to compare the shear bond strength (SBS) of metal brackets bonded to a zirconia faced ceramic using the two surface conditioning methods and two different prime systems.

Materials and Methods:

The ethical approval with Ref. No. (UoM.Dent.23/33) for this research was obtained from the research ethics committee of collage of dentistry/Mosul University.

The samples:

The samples included 60 CAD/CAM zirconia faced porcelain specimens, each specimen consist of two parts Figure(1), The upper part of the specimen is a crown of upper left central incisor with a diameters determined according to Ash and Nelson(2010)[17] and Sangalli *et al.*(2022)[18]**.** The cylindrical base with the diameter of 10mm in height and a radius length of 10mm. The sample size was calculated using sample size calculation formulas by Charan *et al.*(2021)[19] and based on a study done by Mehta *et al*.(2016)[1].

Ceramic specimens fabrication :

A three dimensional program (Exocad galawy) was used to design the samples. Subsequently, the zirconia specimens were milled using the Go2dent digital software (Go2dental program) and a CAD/CAM milling machine (Maxx200, Korea) then the labial surface of zirconia specimens was faced by ceramic. All the steps of laboratory processes for the models design, construction, facing by ceramic and glazed was carried out according to the manufacturer's recommendations by a single dental technician to ensure consistency. All the specimens were cleaned by a polishing paste without fluoride then thoroughly washed and dried by the air for 5 second.

Criteria of Sample Selection:
The labial surface of specimens examined by a stereomicroscope(Japan/Union/ME3138) under 10X magnification power Figure(2) to confirm that selected specimens clear from any impurities, any porosities, cracks or irregularities[20].

Sample grouping:
The specimens were randomly divide in two main groups according to prime type into: Assure® Plus bonding group and Transbond™ XT group. Then each group subdivided into three subgroups according to surface treatment methods as follow control group, hydrofluoric acid treatment group(HF) and aluminum oxide air abrasion group(AL_2O_3).

Surface treatment procedure:
For hydrofluoric acid groups, the labial aspect of the specimens at middle third etched by 9.6% HF acid for 1minute then rinsed for 30 seconds, and air-dried[21].
For Aluminum oxide group(AL_2O_3) groups: The specimens fixed in a special design base to ensure standardization of distance and direction between microetcher (Ortho Technology, Emergo Europa) and specimens surface as shown in the Figure(3).Then the labial aspect at middle third of the specimens were undergone sandblast by 50μm AL_2O_3 particles using the microetcher at distance of 10mm and in direction perpendicular to the labial surface of specimens with pressure of 0.25Mps for 15seconds. Then the samples were washed thoroughly by tap water to remove AL_2O_3 particles and then dried by air spray[22].

Surface roughness measurement:
The surface roughness(Ra) for all groups(control, AL_2O_3 and HF) was measured using a profilometer device(Surfatest SJ-201p, Mitutoyo, Japan). The Ra of each specimen was measured and ten readings of each group were recorded[23].

Bonding the brackets:
For the groups of Transbond™ XT prime, A single coat of adhesive primer (Transbond XT; 3 M Unitek®, Monrovia, CA, USA) was applied and well distributed on the center of the labial aspect of the specimens and left for 30 seconds and then dry with oil free air for 10 seconds to remove excess Then curing started using LED light curing device for 10 seconds according to manufactured instruction.
For the Assure® Plus groups, a thin layer of the Assure® Plus prime was applied and well distributed on the center of the labial aspect of the specimens and left for 2 minutes then thoroughly dry for 3-5 seconds and then curing started using LED light curing device for 10 seconds. All these procedure followed the manufactured instruction.
For all samples, the Transbond™ XT adhesive paste(Transbond XT; 3 M Unitek®, Monrovia, CA, USA) was put over the bracket base of upper central incisor bracket(standard Edgewise 0.022 inch slot metal bracket, Dentaurum**)**. After that, the brackets were fixed at the treated center of the labial aspect of the specimens at a distance of four millimeters from the incisor edge. Bracket positioning gauge was used to ensure correct bracket position, Figure(4). After that, The model insulted on the a customized mold and transfer to the stage of universal testing machine. A load of 200 gm was applied by universal testing machine at a bracket slot for 10 second to confirm uniform thickness of adhesive[24] Figure (5). The excess adhesive was removed by dental explorer. Then the adhesive was photo polymerized using LED light curing device with wave length(420-480nm) and illumination of(1200-1500) mw/cm^2.The curing light was applied for 20 second for the mesial side and 20 seconds for the distal side, The tip of the light cure device was at a distance of 2mm from the mesial and distal edges of the bracket base[25]. The specimens was allowed to bench rest for 30 minutes and then put in a sealed container filled with a distilled water and stored in an incubator at 37^{0}C for 24hours before testing[26].

Shear bond strength measurement:
The Universal testing machine(GESTER, Fujian, China)was used to measure the SBS at postgraduate laboratory, College of Dentistry, University of Mosul, with across speed of 0.5mm/minute. The

specimen was secure seat A prefabricated holder for the specimens has been constructed to ensure proper and secure seating of the specimen so that the bracket base was parallel to the direction of the shear force, Figure (6).The chisel shape blade was directed toward the tooth – bracket interface in an occluso-gingival direction. The necessary load to debond or initiate bracket failure was recorded in Newton unit and converted to MPa unit by dividing the failure load or force in Newton unit to the surface area of the bonded bracket base(mm^2) which was measured by digital caliper(23).

Adhesive Remnant Index (ARI) Measurement:

After debonding of the brackets, the labial surface of the crown of the models were examined under Stereomicroscope at X10 magnification power(Optica, Italy), to assess the amount of the adhesive material left on the models surfaces. The criteria that were used for measuring ARI scores were(9):

- Score 0= No adhesive remnant on the labial surface of the model.
- Score 1= Less than half of the adhesive remained on the labial surface of model.
- Score 2= More than half of the adhesive remained on the labial surface of model.
- Score 3= All of the adhesive remained on the labial surface of model, with a distinct impression of the bracket's mesh as in the Figure (7).

Statistical analysis:

The raw data was analyzed by SPSS program V.23 software(New York, USA). The Shapiro-Wilk test showed that the SBS raw data were normally distributed, and Levene's test confirmed homoscedasticity. One-way ANOVA was used to compare the mean SBS values of the groups at significance level at $P \leq 0.05$ followed by Duncan's Multiple Range test. The independent T test was performed to compare between two adhesive system. The nonparametric data of surface roughness and ARI scores compared by Kruskal-Wallis test.

Results:

The AL_2O_3 group had a highest amount of surface roughness and Kruskal-Wallis test revealed a significant difference among group in amount of surface roughness as in Table(1).

Table(2) showed the descriptive statistic of SBS values of each groups. The Al_2O_3 with Assure® Plus group had a highest mean value. Also, Table (2) showed a significant difference between the means of SBS values of the groups at $P \leq 0.05$. Using Duncan's Multiple Range test for SBS (Table 2), significant discrepancies were detected and the highest SBS was found in Al_2O_3 groups. The SBS of HF groups for Transbond™ XT adhesive and Assure® Plus was lower than that one's obtained by Al_2O_3. Conversely, the control groups had the lowest mean SBS values.

The Independent t-test (Table 3) revealed that there was a significant difference at $P \leq 0.05$ between Assure® Plus and Transbond™ XT adhesive type in Al_2O_3 groups and there was no significant difference between them among HF or control groups.

Table (4) illustrated the ARI scores distribution. The majority of the samples in control had score 0(all the adhesive remained on the bracket base) while for HF had score 1. Furthermore, most of the samples of Al_2O_3 group with Assure® Plus had score 2 and 3 (most of the resin material remained on the ceramic surface) while Al_2O_3 group with Transbond™ XT had score 1 and 2. Table (5) showed the descriptive statistics and Kruskal-Wallis test which revealed a significant differences in the ARI scores at $P \leq 0.05$.

Discussion:

Debonding of orthodontic brackets is a major cause for lengthening orthodontic treatment(27) In general, the brackets are bonded to enamel surface but the challenge is when orthodontists are required to bond brackets to restorative surface rather than enamel(28). Ceramic crowns are the most commonly used when esthetic is required to restore damage or lost teeth(29)so bonding of bracket to glazed ceramic surface required different surface treatment procedures than natural enamel surface to increase the bonding strength of the bracket to ceramic restoration[30-31]. In the present study, the SBS of metal bracket bonded to ceramic specimens were tested using two surface conditioning methods and two prime protocols also in order to verify which procedure is the most suitable for clinical use. The air abrasion byAL_2O_3 particles provide a roughest surface than HF etching because in this technique the AL_2O_3particles are projected to produce abrasion by high air pressure on the surface of ceramic or another fixed prosthesis and similar result conducted by Kwak *et al*.(2016)[23].

According to surface treatment methods, the AL_2O_3 group had the highest value of SBS among group while the control and HF groups had a SBS which is so low and not reach to a acceptable clinical range of bonding strength that described by Reynolds and colleagues who proposed. that the reasonable clinical bond strength values is 5.9 to 7.8 MPa[32,33].

Several studies were controversy to our results such as stella *et al*.(2015) concluded that etching of porcelain surface by 10% hydrofluoric acid result in higher SBS of (16.42MPa)[30]. Moreover, results obtained by Yassaei *et al*.(2013)[34] and Kurt *et al*.(2019)[35] showed that etching of felspathic ceramic by HF 9.6% for 2 minutes result in SBS of 7.4MPa and 8.84MPa respectively. The literature review done by Alzainal *et al.* (2020)[11] mentioned that the HF 9.6% consider as golden methods of surface treatment of ceramic prosthesis. In another study by Mageet *et al*.(2023), they note a higher SBS when porcelain surface etching by HF for 1minutes[31]. However, the intraoral HE etching can be irritant and toxic to soft tissue so another method should be used as alternative such as laser, sandblasting, bur, maleic acid or orthophosphoric acid[11]. The contrary in the our results with these studies mentioned above should be related to use of different types of ceramic materials which differ in particle size and the form of their crystalline structure which may be responsible for different values of bond strength[36].

The SBS recorded using AL_2O_3 air abrasion has a highest value(16.9 M,Pa) and this finding similar to a study by Naseh *et al.* (2018) showed that when the feldspathic porcelain discs sandblasted by aluminum oxide particle and followed by HF etching for 2 minutes, the values of SBS were above the minimum acceptable values[9]. Moreover, Mageet *et al*.(2023) concluded that the strongest SBS (18.63MPa) was obtained when used sandblasting alone without etching[31].

In the present study, the SBS values obtained using Assure® Plus prime with AL_2O_3 abrasion were higher than those obtained when using Transbond™ XT prime and this due to higher flowability of Assure® Plus prime compared with Transbond™ XT prime a similar results recorded by Pulido *et al.* (2023) who found that the SBS recorded when metal orthodontic brackets bonded to ceramic specimens using Assure® Plus were satisfactory for feldspathic porcelain[37]. Also. Jamal *et al*.(2021) reported that a high shear bonding strength(17.29MPa)was recorded when Assure® Plus bonding ceramic brackets to feldspathic ceramic[38]. Amirabadi *et al*.(2015) found that the application of Assure® Plus on feldspathic porcelain yielded a higher SBS than those treated by Transbond™ XT[39] . The finding of Toodehzaeim *et al*.(2017) reported that the application of Assure® Plus on ceramic, amalgam or enamel surface provided a bonding strength values within acceptable clinical mean and considered Assure® Plus as a multipurpose prime[6].

A controversy result by El-Ramly *et al*.(2022) which showed that the air abrasion with Reliance Assure Plus yielded a lower SBS value(1.84MPa)of orthodontic brackets bonded to porcelain surfaces[28]. Also Mehta *et al.* (2016)reported no significant differences in SBS between Transbond™ XT and Assure® Plus bonding agent[1]. A universal bonding(Transbond™ XT prime)revealed a high SBS(14.369MPa) with AL_2O_3 surface treatment while a low SBS(4.30 and 2.39 MPa)values with HF etching and control groups respectively. A research by Tahmasbi *et al*.(2020) tested the SBS of metal brackets to ceramic bonded by universal prime and the results found that the SBS values were 4.4MPa and the author reported that the universal prime did not provide acceptable SBS[8]. Furthermore, Isolan *et al.*(2014) evaluated the bonding strength of brackets bonded to porcelain surface using universal bonding agent after etched by 10% HF and showed a high bonding strength of 29MPa and this finding was contrary to our results[40].

The ARI was used to describe the position and mode of adhesive failure. Several studies have advocated that it is preferable for occurrence of adhesion failure at the tooth adhesive interface so that the resin remnants on the surface can be cleaned safely with rotary instruments [32, 41-43]. When debonding orthodontic bracket from tooth surface, it is important to avoid tooth crack and with minimal adhesive remain on the teeth surface. Likewise, for all restorations, the aim is the debond area has minimum cohesive damage to ceramic or zirconia and at the same time has minimal residual adhesive left[1]. The adhesive failure in control and HF group bonded by Transbond™ XT or Assure® Plus had a score 0 and 1 which was designated to adhesive-zirconia interface failure while the models in AL_2O_3 group bonded by Transbond™ XT had a score 1 and 2 which is a mixed failure, showing a favorable failure mode. Controversy, most of the models in AL_2O_3 group bonded with Assure® Plus had a score 2 and 3 which was appointed to adhesive-bracket interface failure and this findings similar to Tahmasbi *et al.* (2020)[8] and Abou shady *et al.*(2021)[44] concluded that most of failure at bracket adhesive interface. Also, A cohesive porcelain failure of the specimens was reported by Isolan *et al.*(2014)[40]. Controversy to present study, Karan *et al.*(2007)[45] and El-Ramly *et al.*(2022)[28] showed that the samples which treated with air abrasion by AL_2O_3 and bonded by Assure® Plus prime failed at the adhesive porcelain interface. So our study suggested that the Transbond™ XT with AL_2O_3 surface conditioning method is a suitable adhesive for use with ceramic restorations.

Limitation of the study:

This in vitro studies applied to evaluate the effect of two types of adhesive material and two surface treatment methods on SBS but the effect of other factors that intervene in oral environment were not considered in our investigation. These contributing variables that affect the SBS values in the oral environment like pH level of saliva, complex microflora, temperature, stress generated by the orthodontic arch wire and masticatory force.

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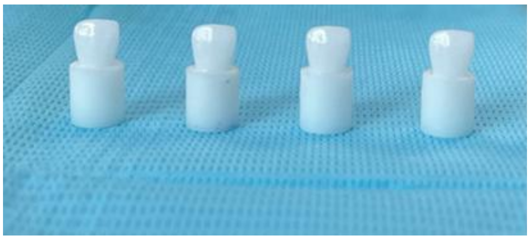
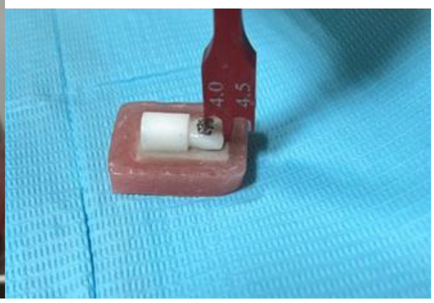
Conflicts of interest: There are no conflicts of interest .



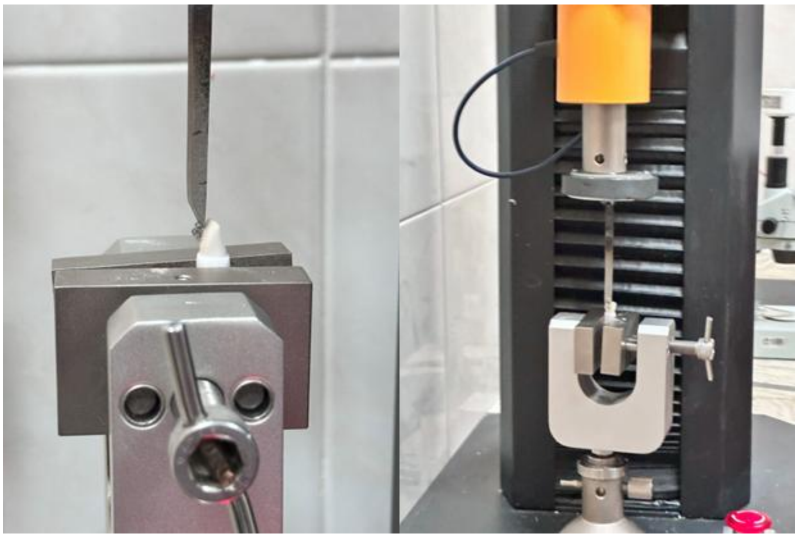
Figure(1) :Zirconia faced by ceramic specimens.

Figure(2) : Evaluation of surface of specimens by stereomicroscope.

Figure(3): Air abrasion of specimens.



Figure(4): Bracket positioning.　　Figure(5): A stable pressure applied to bracket.



Figure(6) :Shear bonding strength measurement by universal testing machine.

Figure(7) : ARI score 3.

Table (1): Descriptive Statistics, Kruskal-Wallis Test and Mann- Whitney U test of the amount of surface roughness (μm) for examined groups.

Ceramic	Surface conditioning method	N	Minimum	Maximum	Mean	Std. Deviation	chi2	sig	z-value		
									Al_2O_3 vs control	HF vs control	HF vs Al_2O_3
	HF	10	.40	.60	.4950	.06852	23.698	0.00	- 3.845- **	- 3.856- **	-2.971- *
	Al_2O_3	10	.50	.80	.6400	.09661					
	Control	10	.10	.13	.1110	.01197					

*Significant at P≤0.05 ** Significant at P≤0.001.

Table (2): Descriptive Statistics, One way analysis (ANOVA) and Duncun Multiple Range Test of SBS (MPa) for examined groups.

	Adhesive types	Surface conditioning methods	N	Minimum	Maximum	Mean	Std. Deviation	F	Sig.	Duncun Multiple Range Test
Ceramic specimens	Assure® Plus	Control	10	1.90	2.80	2.4460	.32080	2.325E3	.000	2.4460 c
		HF	10	3.40	4.60	4.1316	.34192			4.1316 b
		Al_2O_3	10	15.74	17.71	16.9406	.76870			16.9406 a
	Transbond™ XT	Control	10	1.85	2.39	2.1454	.18384	4.377E3	.000	2.1454 c
		HF	10	3.39	4.30	3.9490	.28946			3.9490 b
		Al_2O_3	10	13.89	14.93	14.3699	.42528			14.369 a

Table (3): Independent t-test for SBS for examined groups according to adhesive types.

			N	Mean	t-value	Sig	Std. Deviation	Std. Error Mean
Ceramic	Control	Assure® Plus	10	2.4460	2.571	.19	.32080	.10145
		Transbond™ XT	10	2.1454	2.571		.18384	.05814
	HF	Assure® Plus	10	4.1316	1.289	.214	.34192	.10813
		Transbond™ XT	10	3.9490	1.289		.28946	.09154
	Al_2O_3	Assure® Plus	10	16.9406	9.254	.000*	.76870	.24308
		Transbond™ XT	10	14.3699	9.254		.42528	.13449

Table(4):ARI scores distribution.

	Group	**0**	**1**	**2**	**3**
Assure® Plus	HF	1	8	1	0
	Control	6	4	0	0
	Al_2O_3	0	1	6	3
Transbond™ XT	HF	3	7	0	0
	Control	10	0	0	0
	Al_2O_3	0	7	3	0

Table (5): Descriptive Statistics and Kruskal Wallis Test of ARI scores.

	Adhesive types	Surface conditioning methods	N	Minimum	Maximum	Mean	Std. Deviation	Kruskal Wallis Test	
								Chi-Square	Asymp. Sig.
Ceramic	Assure® Plus	Control	10	0.00	1.00	.4000	.51640	20.152	.000
		HF	10	0.00	2.00	1.0000	.47140		
		Al_2O_3	10	1.00	3.00	2.2000	.63246		
	Transbond™ XT	Control	10	0.00	.00	.0000	.00000	20.558	.000
		HF	10	0.00	1.00	.7000	.48305		
		Al_2O_3	10	1.00	2.00	1.3000	.48305		

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