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EVALUATION OF THE EFFECT OF DIFFERENT SURFACE TREATMENTS ON MICRO TENSILE BOND STRENGTH AND COLOR STABILITY OF HYBRID CERAMIC REPAIRED WITH COMPOSITE

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ABSTRACT

Aim: The current study sought to assess the impact of various surface treatments on microtensile bond strength (TBS) and colour stability in hybrid ceramic mended with composite.

Materials and Methods: Fifty specimens were prepared from available nano ceramic hybrid CAD /CAM blocks then divided into : Group I, micro bars shaped specimens for micro tensile bond strength testing and group II, slices shaped specimens for color stability testing .Then specimens from each group were divided to five sub-groups according to the surface treatment used diamond abrasion(**D**), diamond abrasion and silane coupling agent(**DS**), sandblasting(**B**), sandblasting and silane (**BS**), and silane coupling agent (**S**).Dual cure resin cement was applied to the specimens , then composite was built up and photoactivated to form ceramic-composite complex .The microbars was subjected to microtensile bond strength test using universal testing machine .Reflective spectrophtometer was used to determine the color of the ceramic-composite slices specimens in CIELAB system.

Results: The highest color change was seen in the silane subgroup (**S**), while the diamond abrasion subgroup (**D**) showed the lowest color change followed by diamond abrasion and silane subgroup (**DS**). sandblasting and silane (**BS**) showed the highest μTBS values followed by diamond and silane (**DS**). The lowest μTBS was seen in the silane (**S**) subgroup.

Conclusion: Different surface treatments increased microtensile bond strength and had an effect on color stability of hybrid ceramic repaired with composite.

KEYWORDS: Hybrid Ceramic, Composite ,Micro Tensile Bond Strength ,Color Stability ,

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INTRODUCTION

Recent advancements in dentistry have seen a surge in the use of all-ceramic restorations, favored for their biocompatibility and aesthetic appeal. Despite their advantages, these restorations are prone to fractures and chips due to their inherent brittleness and structural limitations [1]. To address this, resin-matrix ceramic materials have been introduced, merging the strengths of both composite resin and ceramic materials. This innovation offers a blend of ceramic's durability and color stability with the machinability, low abrasiveness, and easy intra-oral repair qualities of composite resin [2].

Replacing failed dental restorations is both costly and time-consuming. To mitigate this, a shift towards more conservative, minimally invasive repair methods instead of full restoration replacement is gaining traction, especially in Western dental schools [3].

This approach emphasizes the repair of fractures, which can arise from various factors like trauma or design flaws, over complete removal [4] . The removal process can further damage healthy tooth tissue and weaken the structure. In contrast, bonding composites directly to exposed ceramic for repairs is an economical, aesthetically pleasing, and simpler solution [5].

Intra-oral repair, an alternative to extra-oral repair or full restoration replacement, is favored for its cost-effectiveness, resource efficiency, reduced treatment time, and preservation of tooth structure[6]. The success of this method hinges on the bond strength between the ceramic surface and the composite resin [7]. Resin matrix ceramics are particularly effective for repairs due to their high polymer content, which facilitates a strong chemical bond with resin materials [8].

Mechanical and chemical methods for conditioning ceramic surfaces have been presented in order to achieve optimal binding strength between ceramic and composite. Mechanical roughening of the surface using a coarse diamond bur, AL2O3 sandblasting, HF etching, and silane usage all result in satisfactory bond strength. [9].

The market offers various ceramic repair kits, each proposing different techniques like diamond surface roughening, hydrofluoric acid etching, and silanization [10] .However hybrid ceramics lacking silica and glass phases do not respond well to hydrofluoric acid etching.

While repairing fractured ceramic restorations is a practical and preferred option, it presents challenges. Post-repair, there can be noticeable color changes over time [11]**,** often due to the differing compositions of ceramic and composite materials. By the passing time, discoloration of dental materials in the oral environment is produced by the resin matrix's composition, which comprises BIS-GMA, BIS-EMA, and UDMA, all of which have higher water sorption and are thus more sensitive to staining agents and discoloration. [11]. Additionally, artificial aging factors like thermal cycling and exposure to colored beverages can significantly alter the optical properties of resin-based ceramics, impacting the aesthetic outcome **[2].**

The aim of this study was to assess the impact of various surface treatments (D, DS, B, BS, and S) on the colour stability and microtensile bond strength of hybrid ceramic (Grandio blocs: nano ceramic hybrid CAD/CAM blocks) as measured by a spectrophotometer using a universal testing machine. restored utilising composite.

MATERIALS AND METHODS

1- Specimens Grouping

Fifty specimens were fabricated from commercially available hybrid ceramic (**Grandio blocs).** Specimens were divided according to their shape to two groups: **group 1** micro bars shaped(twenty five microbar) and **group 2** slices shaped (twenty five slice).Each group was divided according to surface treatments done to five subgroups: **D:** diamond bur abrasion(n=5), **DS**: diamond and silane coupling agent,(n=5), **B**: sandblasting by silica powder (n=5), **BS:** sandblasting and silane coupling agent (n=5), and **S**: silane coupling agent (n=5) Microbars shaped specimens were subjected to micro-tensile bond strength test by microtensile tester then were viewed using stereomicroscope (Nikon SMZ745T ,Japan) to view the fracture pattern and detect the mode of failure..The slices shaped specimens were analyzed using clinical spectrophotometer (X-Rite, model RM200QC ,Neu-Isenburg, Germany) to detect color changes.

2- Specimens Preparation

Microbars fabrication (for microtensile bond strength testing)

****Ceramic Blocks Cutting***

Every ceramic grandio block (grandio blocs) was cut horizontally using low speed percision cutting machine.(isomet 4000, BUEHLER, Germany) with diamond saw to mini-blocks (4 mm each).Mini blocks were polished using 600 grit sandpaper .

****Composite Specimens Construction***

Mold was formed using elastomeric silicon impression material (Zhermack, Badia Polesine, Rovigo, Italy) in round base(Ready made, Egypt) by taking impression of the last part of ceramic block with size of 4 mm and was left to set forming silicon index. Excess impression material was removed with scalpel blade(Miltex, stainless steel, Pakistan).Composite was built up incrementally in the rubber and photoactivated for 40 seconds by light cure device (Bluephase Ivoclar Vivadent, Schaan, Lichtenstwin).

Following the first polymerization, the resin micro block was taken from the mold, and the side in touch with the silicone, as well as the other sides, were polymerized for 40 seconds each to give mini blocks equal to that of ceramic that was ground on sand papers to obtain flat and smooth surface.

Slices Fabrication: (For Color Measurments)

Ceramic blocks was cut by diamond saw (isomet 4000, BUEHLER, Germany) to slices 1mm in size. Digital caliper (TMT32150,China) was used to make sure specimen have equal size $1mm^2$. Impression was taken to a ceramic slice by silicon impression material (Zhermack, Badia Polesine, Rovigo, Italy2) to form a silicon mold, and was left to set. Composite was built up in the mold and photo activated. (Bluephase Ivoclar Vivadent, Schaan, Lichtenstwin) for 40 second then removed from the mold and polymerized in all sides of the composite slice.

3 - Surface Treatment Methods

The following surface treatment methods were done in the surfaces of ceramic that were subjected to cementation as a repair ceramic part :

Subgroup D: bonding surfaces of hybrid ceramic slices (grandio blocs) were abraded by diamond bur (fissure diamond bur, china) and high speed hand piece (Sirona dental system, Germany) with constant water spray, sweeping motion perpendicular to surface with light pressure , then specimens were rinsed with distalled water and air dried. **Subgroup DS:** diamond abrasion was done as mentioned before, then silane application, :bonding surfaces of slices were brushed with silane coupling agent (VOCO, Cuxhaven, Germany) for 90 seconds. The specimens were cleaned in distilled water for 20 seconds, then dried with oil- water free compressed air. **Subgroup B :** Sandblasting of the bonding surfaces with silica (3M ESPE, Seefeld, Germany). powder at a pressure of two bars, where the distance between the nozzle and the surface was 10 mm and perpendicular to the treated surface for twenty seconds. The specimens were cleaned in distilled water, and sprayed with alchol to clean the surface then dried with oil- water free compressed air. **Subgroup BS:** sandblasting as mentioned, then silane application as mentioned before And **Subgroup S**: silane application as mentioned before. After all surface treatments the ceramic slices were ultrasonically cleaned in distalled water (for five minutes) and air dried for 60 seconds.

4- Ceramic-Composite Blocks Fabrication

The dual-curing adhesive luting system that was supplied in a form of auto-mix syringe with disposable applicators, it was applied evenly on the ceramic treated surface with a disposable applicator. The composite resin mini-blocks were cemented above the ceramic mini-blocks ,the excess cement was removed by micro brush. Light curing was done according to manufacture's instructions , the cement was polymerized for 40 seconds on each to from ceramic-composite block. Specimens were stored in coded pouches. The same procedures were made for ceramic and resin slices to form ceramic-composite slices.

5 - Thermocycling of the Specimens

Prior to conducting the microtensile bond strength test, all specimens were preserved in distilled water at 37o C for one day in an incubator (BST50 20, VEB MLW, Leipzig, Germany). For the purpose of simulating clinical service in the lab, specimens from each subgroup were run through 5,000 cycles in a thermocycling machine (robota automated thermal cycle; BILGE, Turkey) with water temperatures ranging from 5°C (the low point) to 55°C (the high point). Each cycle lasted 25 seconds, and there was a 10-second lag time between cycles. One common and accurate way to mimic the conditions of intraoral thermal change that a restoration would have experienced is thermal ageing. Bayne (2011) states that one year of clinical survival is equivalent to 5,000 cycles.

6 -Preparation for Microtensile Bond Strength Test

The ceramic composite blocks were cut into bar-shaped specimens with a cross area of 1mm2 using a persion saw (isomet 4000, BUEHLER, Germany)[25] along the X- and Y-axes (1x1x6 mm). leaving out the usage of slices on the periphery in order to prevent using slices with an inadequate or excessive quantity of cement at the interface.

7 -Microtensile Bond Strength Test

To ensure that all the microbar specimens were of uniform size, their bonding areas were measured using digital callipers (Total TMT32150, China) prior to testing. Using cyanoacrylate adhesive, the specimens were fastened to the flat metal plate of the microtensile tester (5ST, Tunius Olsen, England) (universal testing equipment) (Super Glue ,Taizhou Henco-glue). All forces acting on the ceramic-composite bonding contact were maintained at a free and perpendicular position. A velocity of 1 mm/min across the head was applied to the complex until it broke.

It was possible to determine the bond strength in MPa by dividing the force needed to break the specimen (in N) by the area of the bond (in mm2). Specimens that did not pass the tests were marked as having no μTBS (Microtensile bond strength) values. Statistics were used to examine the variations in μTBS between the categories.

8 -Mode of Failure:

After fracture specimens were viewed using stereomicroscope (Nikon SMZ745T ,Japan) with FHD camera DX-230 connected to computer screen to view the pattern of fracture..Two types of failure was seen: 1.Adhesive failure (hybrid ceramic surface was visible no layer af resin cement remain on the surface). 2.Mixed failure in hybrid ceramic and luting resin cement (fracture line include both ceramic, composite and luting cement).

9 -Color Measurments:

The specimens of ceramic-composite slices were measured using a reflective spectrophotometer in the CIELAB (Commission International de l'Eclairage) system. The instrument utilized for this purpose was a German-made X-Rite model RM200QC. In order to ensure precise alignment of the specimens with the apparatus, the average aperture size was adjusted to 4 mm. The measurements were taken using the CIB standard illuminant D56, and a white backdrop was used. D65 is also known as a daytime illuminant because it generally represents the normal noon light in Western Europe and Northern Europe, which consists of both direct sunlight and light dispersed by a clear sky.

The formula was used to determine the specimens' colour changes, denoted as ΔE. $\Delta E^* = [(L^*1-L^*0)2+(a^*1-a^*0)2+(b^*1-b^*0)2]\ 1/2$. Assuming that L* is the brightness scale from 0 to 100, a* is the colour shift from red to green for the axis, and b* is the colour variation from yellow to blue for the axis.

Averaging the values was done. High values of L* indicate a brighter sample, higher values of a* indicate a redder sample, and lower values of a* indicate a greener sample. Similarly, high values of b* indicate a yellower sample, and lower values of b* indicate a bluer sample. Pre-, post-, and thermocycling colour measurements were taken according to the various surface treatments.

RESULTS

Color Changes After Surface Treatments and Repair with Composite :

Comparing the color change (ΔE) between five groups, results showed that there was statistically significant differences in ΔE between all groups. The highest color change was seen in the silane subgroup **(S)** (15.2±1.7), while the diamond abrasion subgroup **(D)** showed the lowest color change (4.5±0.6) followed by diamond abrasion and silane subgroup **(DS)** (6.8±1.3)

No significant difference was seen between sandblasting **(B)**, sandblasting and silane **(BS)**, as shown in **table(2)** and **figure(1).**

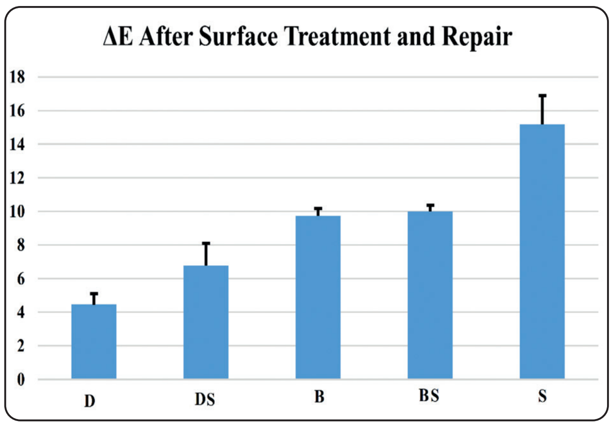

Fig. (1) Histogram showing color change mean values for the five subgroups after surface treatments, repair.

TABLE (1) ΔE After Surface Treatments and Repair in the Five Subgroups

After surface treatments and repair		Diamond (D) N=5	Diamond and Silane (DS) N=5	Sandblasting (B) N=5	Sandblasting and Silane (BS) N=5	Silane (S) N=5	P value
ΔE	Range	(3.4-4.9)	(5.6-8.3)	(9.4-10.4)	(9.4-10.3)	(12.5-16.7)	<0.001*
	Mean ± SD	4.5±0.6	6.8±1.3	9.7±0.4	10±0.4	15.2±1.7	
P value between each two subgroups							
D			0.002*	<0.001*	<0.001*	<0.001*	
DS				<0.001*	<0.001*	<0.001*	
B					0.701	<0.001*	
BS						<0.001*	

Color Changes After Thermocycling:

Examining the five groups after thermocycling, results were shown that there were significant differences between the subgroups in Δ E after thermocycling . The highest Δ E values were seen in silane subgroup **(S)** (14.8±1.6), while the lowestΔ E values were seen in the diamond abrasion **(D)** (5.2±0.6) followed by diamond abrasion and silane subgroup **(DS)** (7.8±0.8) .

No significant difference was seen between diamond **(D)**,diamond and silane**(DS)** or between the two sandblasting subgroups **(B, BS).** ,as shown in **table(3)** and **figure(2)**.

3-Microtensile Bond Strength Measurments in Mpa Between the Five subgroups:

Comparing the micro tensile bond strength (μTBS) between the five subgroups, the sandblasting and silane subgroup **(BS)** showed the highest μTBS values (24.2±1.5) followed by diamond and silane subgroup **(DS)** (21.4±1.4). The lowest μTBS was seen in the silane subgroup **(S)** (15.5±1.2) as shown in **table(4)** and **figure (3)**.

TABLE (3) Δ E After Thermocycling Between the Five subgroups

Δ E after thermocycling		Diamond (D) N=5	Diamond and Silane (DS) N=5	Sandblasting (B) N=5	Sandblasting and Silane (BS) N=5	Silane (S) N=5	P value
ΔE	*Range*	(4.5-6.1)	(5.1-7.6)	(6.5-8.4)	(7.4-10.2)	(13-17.1)	*<0.001**
	Mean ± SD	5.2±0.6	6.5±1	7.8±0.8	9±1.1	14.8±1.6	
P value between each two subgroups							
D			*0.078*	*0.001**	*<0.001**	*<0.001**	
DS				*0.067*	*0.002**	*<0.001**	
B					*0.101*	*<0.001**	
BS						*<0.001**	

One way ANOVA test for quantitative data between the 5 subgroups followed by LSD analysis between each subgroup*: Significant level at P value < 0.05

TABLE (4) Comparison of μTBS Between the Five subgroups

		Diamond (D) N=5	Diamond and Silane (DS) N=5	Sandblasting (B) N=5	Sandblasting and Silane (BS) N=5	Silane (S) N=5	P value
MTBS	*Range*	(18.3-21)	(19.5-23.1)	(19.4-22)	(22.4-26)	(14.3-17.3)	*<0.001**
	Mean ± SD	19.6±1.1	21.4±1.4	20.8±1	24.2±1.5	15.5±1.2	
P value between each two subgroups							
D			*0.036**	*0.154*	*<0.001**	*<0.001**	
DS				*0.452*	*0.002**	*<0.001**	
B					*<0.001**	*<0.001**	
BS						*<0.001**	

One way ANOVA test for quantitative data between the 5 subgroups followed by LSD analysis between each subgroup*: Significant level at P value < 0.05

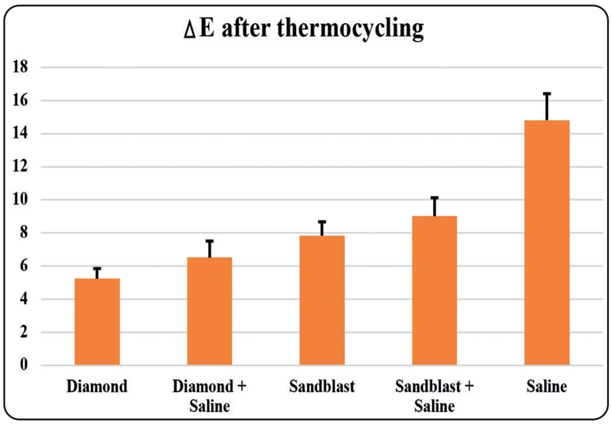

Fig. (2) Histogram showing color change mean values for the five groups after thermocycling

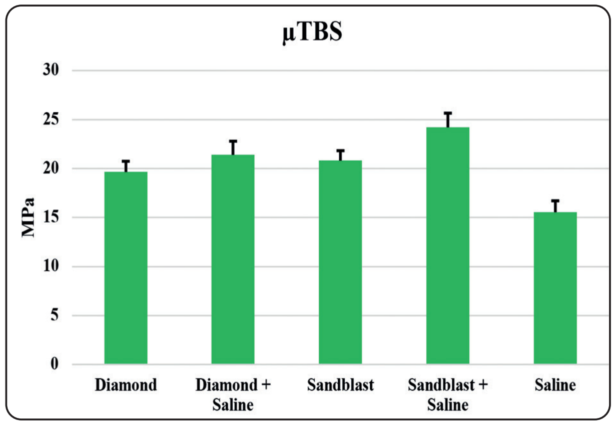

Fig. (3) Histogram showing microtensile bond strength mean values for the five subgroups

DISCUSSION

For many years, resin matrix ceramics have been a staple in creating permanent dental restorations. Their popularity stems from their resemblance to natural tooth material, primarily due to a dentine-like modulus of elasticity. These materials also boast a high filler content and minimal shrinkage, making them user-friendly in dental procedures. Grandio blocs are highly filled nano-ceramic hybrid CAD/CAM blocks, designed for a range of dental applications including crowns, inlays, onlays, veneers, and implant-supported crowns. Their composition includes an exceptionally high filler content of 86%, contributing to their optimal tooth-like characteristics. Its exceptional tooth-like qualities, industry-leading compressive strength, ultra-low water absorption, and natural aesthetics—along with improved color stability and superb polishability are all the result of its extraordinarily high filler degree of 86%.

It mimics human dentition properties such as modulus of elasticity that produced better biochemical behavior [12] it has superior strength and marginal integrity excellent polishability. The materials' simplicity of usage provides further benefits, simplifies polishing and allows for intraoral repairs.it has high flexural strength, compressive strength and flexural modulus[13]. Grandio blocks had the highest elasticity modulus and the lowest water sorption. It exhibited the lowest roughness characteristics and had higher flexural and ultimate tensile strength both at baseline and after aging[14].

The present study aimed to evaluate the impact of various surface treatments on the microtensile bond strength and colour stability of composite-repaired hybrid ceramic.

Introduced in 1994, the Microtensile Bond Strength (µTBS) test has become a cornerstone in the realm of bond strength testing. Over the years, it has gained widespread adoption across numerous laboratories, establishing itself as a standard and highly versatile method for evaluating bond strength. Recognized for its sensitivity, the µTBS test is considered among the most reliable techniques for assessing the bonding performance of materials in vitro [16].

The study's findings reveal that surface treatments significantly improve the microtensile bond strength (µTBS) of materials. Notably, the µTBS values recorded from all groups fell within the clinically acceptable range. This aligns with the standards set by **Atalay C et al (2018)** [17] who posited that a bond strength in the range of 15 to 25 MPa is indicative of favorable durability in repair works.

Furthermore, the study observed that the results obtained from methods like bur abrasion and sandblasting, with or without the addition of silane, surpassed the values achieved by the silane-only subgroup. This observation is consistent with the findings of **Frankenberger, R. et al (2015)** [18]. They highlighted that micromechanical roughening significantly enhances bond strength values in CAD/CAM hybrid materials, sometimes even outperforming chemical conditioning. This indicates the effectiveness of mechanical surface treatment methods in improving the bonding performance of these materials.

The process of surface roughening plays a crucial role in enhancing the bonding strength between composites. This technique involves the removal of the superficial layer of the material, which is typically more exposed to degrading agents. As the surface is roughened, there's an increase in surface energy. This heightened energy improves the wetting of the surface by the bonding agent. Consequently, a stronger bond is formed at the interface between the two composites [19]. Moreover, the application of surface treatments to the restoration surface is recommended to further enhance this bond. These treatments aim to increase the micromechanical interlocking with the luting cement, thereby improving the interfacial bond strength. Additionally, the use of a silane agent is particularly beneficial as it enhances chemical adhesion.

From our results sandblasting and silane subgroup showed the highest µTBS values followed by diamond and silane subgroup. This results come in agree with **Zhang HB et al (2020)** [20] who found that surface treatments such as sandblasting, sandblasting and silane, hydrofluoric acid and silane, and others may enhance the bond strength of resin nanoceramics. Among the groups, the bond strength was strongest in the sandblasted and silane group. Sandblasting followed by silanization produced the maximum µTBS for resin ceramic, according to **Pinto RDS et al(2022)** [21]. study conducted by **Huang B et al (2013)** [22] found that combining bur abrasion with silanization, as opposed to just using bur abrasion, improved the binding strength between composites and ceramics based on lithium disilicate. As a result, diamond abrasion and silane applied together are preferable. In a study conducted by **Swarnakar A et al(2023)** [23], it was shown that sandblasting ceramic samples resulted in a much stronger bond than those treated with laser or silane-coupling agents alone.

In contrast with **Colares RCR et al (2013)** [24] recommended avoiding sandblasting as a surface preparation owing to its negative impact on bond strength, sandblasting should be avoided on ceramics because to the potential for volume loss and alterations in morphology.

The use of silane following surface treatments greatly enhanced the μTBS. These results are consistent with the studiy of **Elsaka SE(2014)** [25] found similar results when they studied the impact of surface treatments and silane on the binding strength of nano ceramic and hybrid ceramic resin. After surface treatments, silanization greatly improved the binding strength to resin cement for hybrid ceramics as concurred by **Demirtag Z and Culhaoglu AK(2019)** [26].

According to **Spitznagel FA et al(2014)** [27] the application of silane treatment was found to significantly increase the bond strength. In contrast, **D'Arcangelo and Vanini (2007)** [28] concluded that silane treatment did not significantly affect the bond strength of resin materials.

Based on what was found in this study, the μTBS test yielded mixed results for the remaining failures, with adhesive failures being the most common. The findings were in line with those of **Sano H et al(1994)** [29] who noted that the μTBS test was helpful in revealing adhesive failures at the bonded interface due to the ability to examine tiny surface areas. This aligns with the findings **Della Bona A et al(2016)** [30] who discovered that the μTBS test and microbar shaped specimens are related with a higher number of adhesive fractures. Reducing μTBS is achieved by applying thermal-cycling on bar-shaped specimens rather than blocks. The impact of temperature fluctuations on materials and bond strengths may be better measured with this method. Additionally, Cekic-**Nagas et al (2016)** [31] concurred that adhesive failures accounted for 54% of all observed failure modes in both materials.

When considering the aesthetic result and practical application, color stability of a restoration is a crucial criterion. All groups displayed noticeable color changes after cementation, which might be attributed to the specimens' rapid artificial ageing. In terms of dressings for the outside. In this study subgroup which was treated by diamond abrasion was more color stable than the other subgroups, followed by diamond abrasion and silane, while surface treatment with silane only had the lowest color stability.

Kilinc H and Turgut S (2018) [32] reported thar hybrid ceramics and resin nano ceramics are more liable to color changes and the discoloration is also dependent on material composition .They found that the optical properties of CAD/CAM material were affected by the type of the material, the color stability of polymer- based resin ceramics is less color stable than other ceramics.

The colour stability of various ceramics after 300 hours of artificial ageing was compared by **Karaokutan I et al(2016)** [33] According to their findings, resin nanoceramics had a much higher colour change value compared to the others. This is in line with the findings of **Saba DA et al (2017)** [34] who examined the consistency of hybrid ceramic colours following 28 days of immersion. According to their findings, the resin matrix in hybrid ceramics caused far larger colour change values in distilled water compared to feldspathic ceramics. Additionally, in line with the findings of **Al Amri et al (2021)** [2], the colour stability of restorative materials that are 1 mm thick during thermal ageing and immersion in a staining solution was assessed. Compared to glass-ceramic or ceramics reinforced with lithium disilicate or ceramics infiltrated with polymers, the resin nano ceramic specimens exhibited a much greater ΔE.

Thermocycling was used to simulate the effects of long-term oral conditions on the surface of the hybrid ceramic material. The hue and translucency of the specimens following various finishing techniques were determined to be clinically undesirable ($\Delta E=5.03$) due to the ageing process, according to **Kurt M and Bal BT (2019)** [35].

CONCLUSIONS

Within the limitations of this study, it was found that;

1. Surface treatment with sandblasting and silane application increase the microtensile bond strength significally.
2. Surface treatment with diamond bur has good color stability.
3. Silane application without mechanical surface treatment decrease the microtensile bond strength and color stability .
4. Thermocycling affect negatively the color stability

Clinical recommendations:

Based on the current study findings, the following recommendations can be drawn:

1. The nano-ceramic hybrid material, known as Grandio blocs, demonstrates the ability to be effectively repaired with composite resin. This repair process requires appropriate surface treatments, which include mechanical methods such as diamond abrasion or sandblasting, followed by the application of silane.

2. While the present study has provided valuable insights, it primarily focused on color parameters. Therefore, it is recommended that future research should expand to include assessments of fracture resistance and surface roughness. Additionally, investigating translucency is suggested to gain a more comprehensive understanding of the optical properties of Grandio blocs.

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