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EVALUATION THE EFFECT OF COLORING TECHNIQUE ON SURFACE HARDNESS OF MONOLITHIC ZIRCONIA

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ABSTRACT

Statement of problem. The effect of the coloring technique on the surface hardness of monolithic zirconia blocks is unknown.

Purpose. This study aimed to investigate the effect of the coloring technique on the surface hardness of monolithic zirconia blocks.

Materials and Methods. Monolithic zirconia blocks were milled from monolithic zirconia blanks by using a CAD/CAM system, then, using a water-cooled diamond blade, thirty pre-sintered disk-shaped specimens (2 mm 10 mm) were made from monolithic zirconia blocks. Ten disks were cut from precolored blocks (A2) and twenty from pure white zirconia blocks (A2). The samples were divided into three main groups (n=10) based on the coloring procedure used: **Group (WZ)** white zirconia, **Group (ISZ)** internal staining group (pre-colored zirconia block, A2), and **Group (ESZ)** external staining group, where white zirconia discs were dipped in coloring liquid (A2). Following the instructions from the manufacturer, the coloring liquid was applied. The acrylic resin was used to mount each specimen from the three groups in the center of the polypropylene holders. Each group tested for the surface microhardness test (n =5). Data were analyzed using a **one-way ANOVA** test to compare the means of three groups with the Bonferroni post-hoc test. SEM photomicrographs were taken at ×500 and ×5000 magnification and observed under a (Quanta 250; FEG) to evaluate the surface morphology and visual inspection.

Results. The results showed the highest Vickers hardness number in the group (ISZ), where zirconia samples were pre-colored (A2).

Conclusion. Coloring techniques affect the surface hardness of monolithic zirconia blocks—the highest Vickers hardness number in precolored zirconia samples.

KEYWORDS: Ceramics, Dipping, Bruxzir, Precolored

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INTRODUCTION

All ceramic crowns are among the solutions that provide the most ideal aesthetic result out of all the available restorations. Compared to alternative restorations, all ceramic crowns more closely resemble the natural tooth structure in color and translucency. (1)

Clinicians regard zirconia-based ceramics as the ideal option among all ceramic systems because they meet the biomechanical requirements for high mechanical strength, fracture toughness, and chemical and dimensional stability. (2)

Zirconia is a polymorph material that occurs in three phases: monoclinic (M) (from room temperature up to 1170°C), tetragonal (T) (1170°C to 2370°C), and cubic (C) (above 2370°C). (3)

Three main types of zirconia are available for use in clinical dentistry. Although chemically identical, they have slightly different physical properties (e.g., porosity, density, purity, strength), which may or may not be clinically relevant. Blocks can be milled either at the green stage, the pre-sintered stage, or the wholly sintered stage. Frameworks made from green and pre-sintered zirconia are milled in an enlarged form to compensate for the shrinkage during sintering, usually 20%-25% for a partially-sintered framework.(4)

One of the most critical advantages of zirconia ceramic restorations over traditional metal-ceramic restorations is their more aesthetic appearance. Notwithstanding these advantages, the color of zirconia is white, and obtaining a natural tooth color may be challenging even after veneering because the framework's color affects the restoration's final color. These explanations suggest that shaded zirconia could provide a more natural look. (5)

Three techniques are frequently used to color zirconia for dental applications; one involves combining metal oxides with ZrO_2 powder during production to produce precolored blocks. Pre-sintering infiltration of the green-stage frameworks with particular coloring liquids is another technique. The third technique requires firing in a dental ceramic furnace and involves coating the zirconia with liners after post-sintering. (2)

According to one study, when a pink liquid containing Er and Nd ions was used to shade zirconia, its strength and surface hardness significantly decreased. Other research, however, found no considerable alteration in flexural strength or hardness. To the authors' knowledge, there have not been any reports on how using aqueous coloring liquids affects zirconia's mechanical properties. (6)

Among its properties, zirconia exhibits high Vickers hardness around 1300VHN and must comply with criterion F1873 of the American Society for Testing and Materials (ASTM), which suggests values above 1200HV.(3)

This study aimed to investigate the effect of the coloring technique on the surface hardness of monolithic zirconia blocks. The null hypothesis of this study was that the coloring technique would not affect surface hardness on monolithic zirconia.

MATERIALS AND METHODS

Thirty disk-shaped specimens (2mm×10mm) were cut from monolithic zirconia blocks (Bruxzir, Prismatik Dental Craft, Inc., USA).

Preparation of samples

Monolithic zirconia blocks were milled from monolithic zirconia blanks (Bruxzir, Prismatik Dental Craft, Inc., USA) using a CAD/CAM system (Roland DWX-51D, USA). Then, using a water-cooled diamond blade (Isomet 4000; Buehler), thirty pre-sintered disk-shaped specimens (2 mm 10 mm) were made from monolithic zirconia blocks (Bruxzir, Prismatik Dental craft, Inc.).Twenty disks were cut from pure white zirconia blocks (BruxZir® HT 2.0, Prismatik Dental Craft, Inc., USA) and ten from pre-colored blocks (A2)((BruxZir® Shaded 16

Plus, Prismatik Dental Craft, Inc., USA). To confirm that the test surfaces were in identical circumstances, 400-grit wet silicon carbide abrasive paper was used to abrade each disc. The samples were divided into three main groups (n=10) according to the employed coloring process: Group white zirconia (WZ), Group internal staining group (precolored zirconia block, A2) (ISZ), and Group external staining group, in which white zirconia disks were immersed into coloring liquid (A2) (ESZ). The coloring liquid (BruxZir Coloring Liquid, Prismatik Dental Craft, Inc., USA) was applied according to the manufacturer's recommendations. All the specimens of the three groups were mounted into the center of the polypropylene holders with acrylic resin. Each group tested for the surface microhardness test (n =5). Monolithic zirconia had a percentage of shrinkage after sintering, which was considered during sample preparation.

External staining procedures

Bruxzir coloring liquids (Prismatik Dental Craft, Inc.) were used for the external staining of the Group (ESZ). The coloring liquid (A2) was applied according to the manufacturer's recommendations. The samples were positioned in the middle of the dry, clean dipping jar, and enough coloring liquid was added to cover them by at least one millimeter. Ensure that the samples are dry and dust-free. Samples were placed in an ultrasonic water bath for thirty seconds up to a maximum of one minute. Samples were removed from the water bath and gently dried with a clean, absorbent tissue. Samples were put in a sintering tray with clean zirconia sintering beads, margin side up. Trays were placed in a microwave for ten minutes at a recommended temperature of 450 ^{o}F. Units were removed and placed under a heat lamp for fifteen minutes to ensure all moisture from the samples had been removed. The samples were submerged in the bubble-free liquid in the dipping jar for fifteen minutes with clean, metal-free tweezers. Samples were removed with clean metal-free tweezers from the coloring liquid, and then samples were left to dry. Samples were placed under a heat lamp for fifteen minutes before sintering. In addition, samples were not allowed to touch one another or the sides of the sintering tray during the sintering cycle.

Surface Microhardness test procedures

A micro-Vickers hardness tester (Tukon 1102 Wilson; Buehler) was used to evaluate the surface hardness of specimens. Surface hardness was measured using the indentation technique to determine the specimens' Vickers hardness number (VHN) on applying a load of (9.81 N). Three indentations were placed in the center of each specimen, and an average hardness value (HN) was calculated from the three measurements.

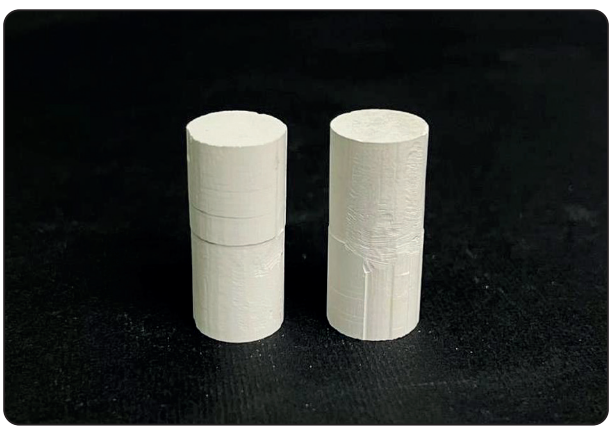

Fig. (1) Monolithic Zirconia blocks milled from zirconia blank

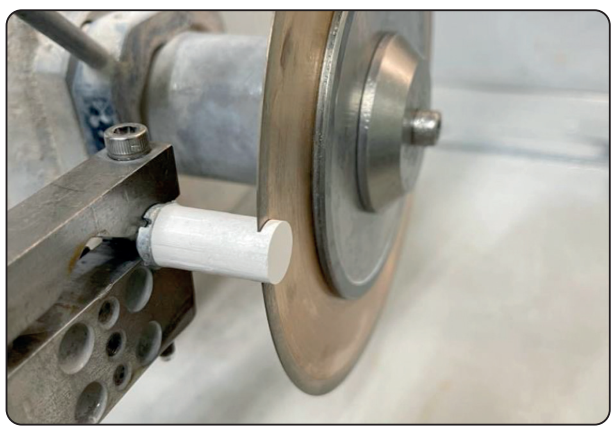

Fig.(2) Disk-shaped specimens fabricated from monolithic zirconia blocks

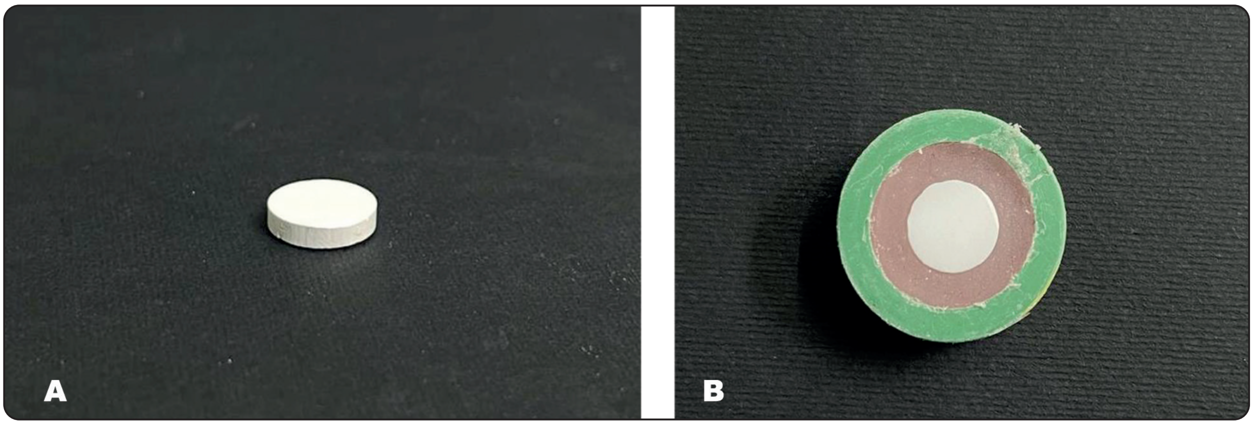

Fig.(3) (A); Disk-shaped specimens [2 mm×10 mm] fabricated from monolithic zirconia blocks – (B); Sample placed using acrylic resin in the center of the polypropylene holder.

Fig.(4) Samples submerged in bubble-free liquid in dipping jar.

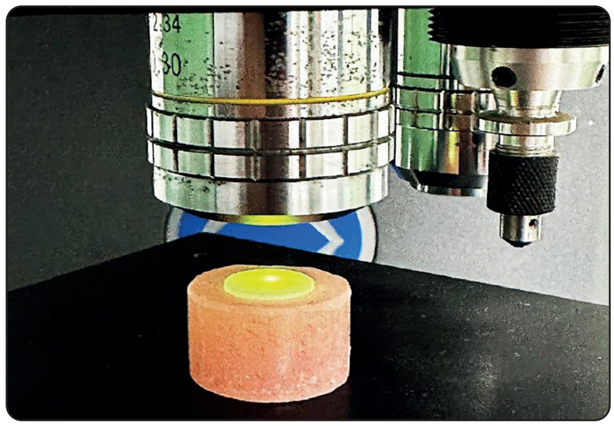

Fig. (5) Surface hardness measured using indentation technique to determine Vickers hardness number (VHN) of specimens on application of a load of 9.81 N

RESULTS

1. Effect of coloring techniques on the surface microhardness of monolithic zirconia blocks

The results showed that the highest Vickers hardness number in the group (ISZ) in which zirconia samples were pre-colored (A2) were mean (±SD=1302.95 ±31.60 HN). and the collective average [Median (range)] of the three readings were {=1309.1 (1253.33:1341.1) HN}. The lowest values in the group (ESZ) in which samples were immersed in coloring liquid were mean (±SD=1151.41 ±81.09 HN). and the collective average [Median (range)] of the three readings were{=1309.1 (1253.33:1341.1) HN} (Figure 7).

The highest surface hardness was achieved in group (ISZ) and then group (WZ). The lowest bond strength was achieved in the group (ESZ), and the three groups' differences were statistically significant ($P< 0.05$) (Table 1, Fig. 6).

The result also showed that there was statistical significance between group (WZ) and group (ISZ), and there was statistical significance between group (WZ) and group (ESZ). However, there was no difference between Group (ISZ) and Group (ESZ).

TABLE (1) Vickers hardness number (VHN) of specimens

Roughness results	**Group (WZ) N=5**	**Group (ISZ) N=5**	**Group (ESZ) N=5**	***P* value**	**P1**	**P2**	**P3**
Reading 1	1323.58±69.24	1332.4±67.65	1144.82±108.90				
Mean ± SD	1323.3	1317.6	1086.3	0.006	1.00	0.02	0.01
Median (range)	(1247.2:1430.9)	(1274.8:1448.1)	(10.45.5:1265.5)				
Reading 2	1286.76±27.92	1349.82±32.04	1146.88±58.57				
Mean ± SD	1281	1333.9	1132.4	<0.0001	0.10	0.001	<0.0001
Median (range)	(1261.5:1333.9)	(1326.8:1333.9)	(1081.1:1213.9)				
Reading 3	1298.5±27.37	1348±36.91	1162.52±86.05				
Mean ± SD	1306.8	1353.8	1166.8	0.001	0.57	0.007	0.001
Median (range)	(1251.3:1321.5)	(1305.8:1392.6)	(1041.2:1243.7)				
Mean hardness	1302.95±31.60	1343.41±38.40	1151.41±81.09				
Mean ± SD	1309.1	1343.97	1111.4	0.0003	0.80	0.003	<0.0001
Median (range)	(1253.33:1341.1)	(1302.93:1399.4)	(1064.07:1241.03)				

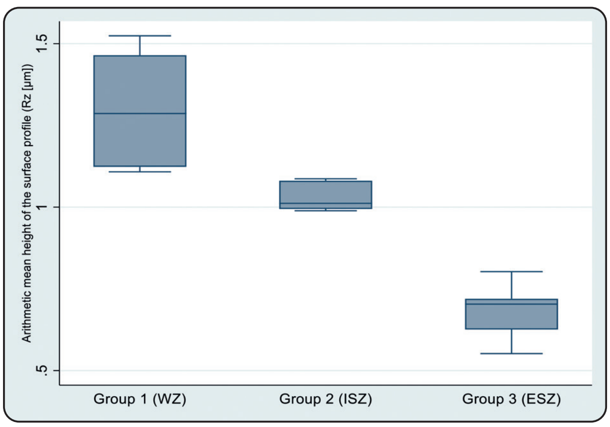

Fig.(6) Hardness number results among studied groups

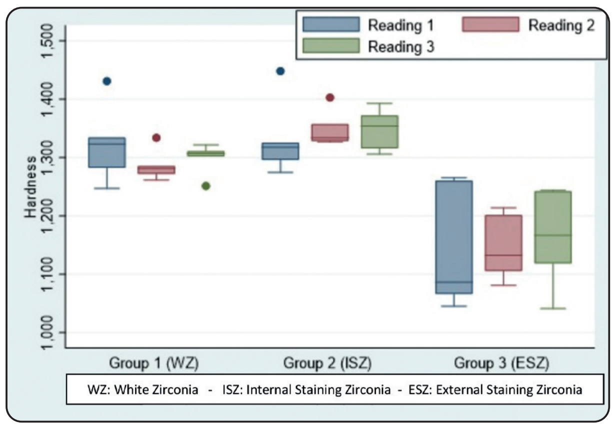

Fig.(7) Mean hardness among studied groups (HN)

A one-way ANOVA test was used with the Bonferroni post-hoc test. *P* value compared the three groups. P1 compared [Group (WZ)] & [Group (ISZ)], P2 compared [Group (WZ)] & [Group (ESZ)], and P3 compared [Group (ISZ)] & [Group (ESZ)].

2. Effect of coloring techniques on the surface morphology of monolithic zirconia blocks.

SEM evaluation revealed that the surface morphology was modified after coloring zirconia samples. Colored with any coloring technique showed multiple deep multidirectional scratches with irregular texture. (Figure 8).

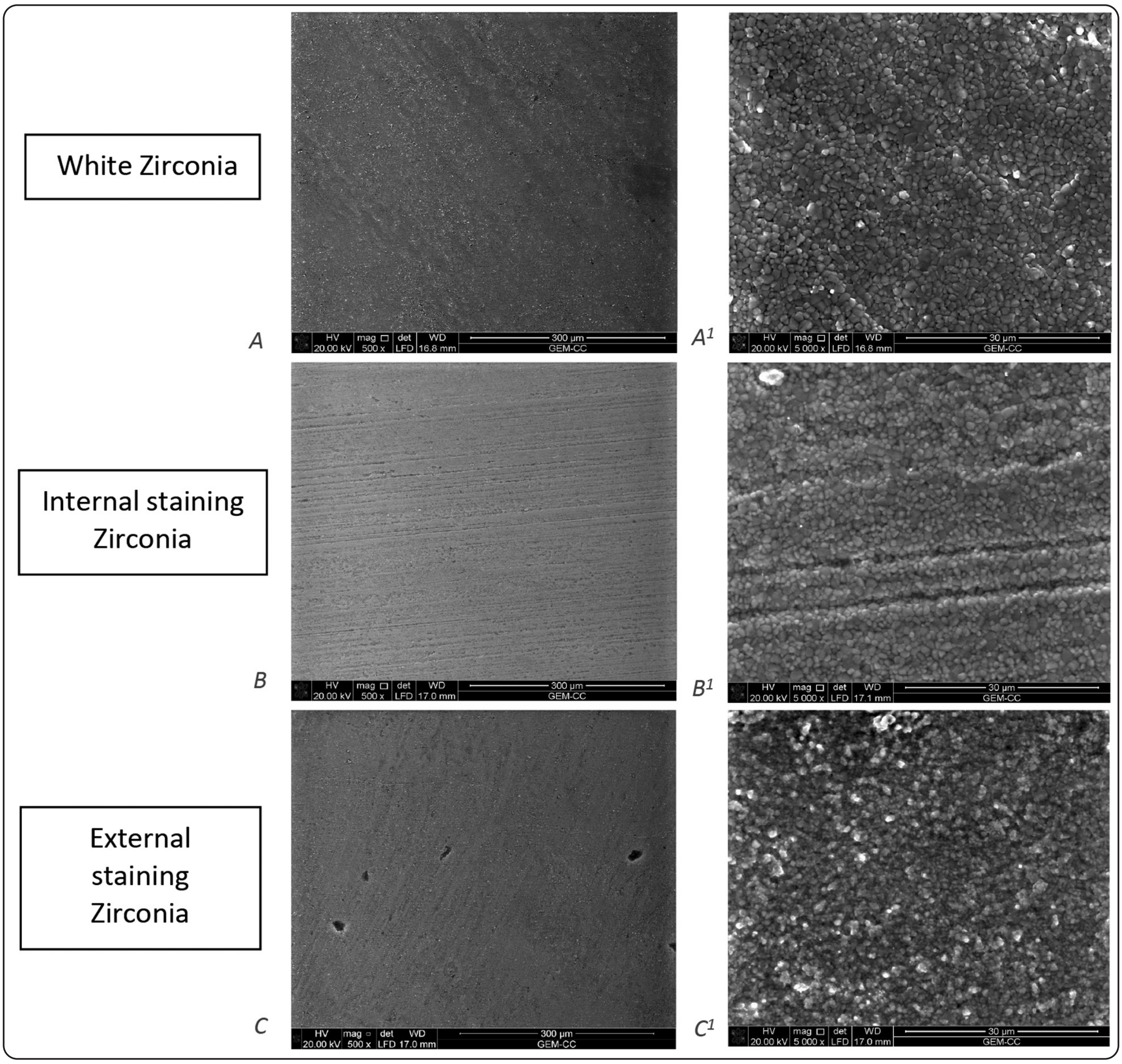

Fig.(8) SEM images of zirconia samples (A; group (WZ) at (× 500) magnification - A1; group (WZ) at (× 5000) magnification – B; group (ISZ) at (× 500) magnification- B1; group (ISZ) at (× 5000) magnification – C; group (ESZ) at (× 500) magnification – C1; group (ESZ) at (× 5000) magnification)

DISCUSSION

All-ceramic restorations have become very popular in dentistry as aesthetics and biocompatibility are considered more. Because of its exceptional strength and dependability, three yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) have been employed to enable longer-span ceramic restorations, even in posterior areas that bear much stress. [7]

A promising new ceramic (zirconia) in 1990 was introduced to resolve the issue of mechanical failure while preserving the aesthetic advantages of all ceramic crowns. The literature indicates that these materials have remarkable five-year survival rates (91.2 percent for individual crowns and 97.6 percent for individual crowns placed on implants). [8]

The current study used monolithic zirconia blocks: White zirconia (BruxZir HT 2.0) and precolored zirconia (BruxZir Shaded 16 PLUS).

Zirconia is white, and because the color of the framework affects the final color of the restoration, it may be challenging to produce a natural tooth color even after veneering. Shaded zirconia may help create a more natural appearance for these reasons. [5]

Two main approaches for coloring monolithic zirconia have been proposed to achieve esthetics comparable to those of veneering porcelain. To create different shades of cores, anatomic contour zirconia restorations are submerged in coloring liquids that contain rare earth element chloride solutions, or metallic pigments are added to the initial zirconia powder before or after pressing the milling blocks. [9]

In this study, the coloring liquid (BruxZir Coloring Liquid) was selected for the shading of BruxZir restorations. Before the last sintering, the BruxZir Coloring Liquid was applied. The restoration was sintered at a high temperature following the application of color. Maximum strength and translucency were displayed by the sintered material, resembling the natural teeth. [10]

According to Hjerppe et al [11], liquid color shades impacted the surface microhardness and biaxial flexural strength of zirconia. Shah et al [12] also found that when the concentration of the liquid color shade increased, the flexural strength of zirconia reduced. The process of coloring, however, had little effect on the material's resistance to degradation at low temperatures.

In this study, twenty samples were cut from pure white zirconia blocks and ten from precolored blocks (A2). To make sure that the test surfaces were in the same condition, 400-grit wet silicon carbide abrasive paper was used to abrade each disc, Which was also done by Giti R et al. (2020). [2]

In this study, the coloring liquid (A2) was applied according to the manufacturer's recommendations. Enough coloring liquid was poured into the clean, dry dipping jar to ensure that the samples were covered by at least one mm of liquid when in the center of the jar. [2,5,13-15]

In the dipping process, a dental technician submerged the milled zirconia ceramic framework in a colored liquid and allowed it to sinter for a predetermined time. Manufacturer-marketed liquid color shades have the ability to penetrate the surface structure of zirconia and impart color to the material. Zirconia's mechanical qualities can be altered by immersing it in liquid color. (Mahshid et al., 2015). [16]

In this study, after the white zirconia samples were shaded, all samples were mounted into the center of the polypropylene holders with acrylic resin to be ready for surface microhardness tests. [2]

Among its properties, zirconia exhibits high Vickers hardness around (1300 VHN) and must comply with criterion (F1873) of the American Society for Testing and Materials (ASTM), which suggests values above (1200 HV3). [17]

A micro-Vickers hardness tester (Tukon 1102 Wilson, Buehler) was used to evaluate the surface hardness of specimens. Surface hardness was measured using the indentation technique to determine specimens' Vickers hardness number (VHN) by applying a (9.81 N) load. [18] Three indentations were placed in the center of each specimen. An average hardness value (HN) was calculated from the three measurements by Donmez MB [18] and others. [6,19,20]

The resistance of materials to producing surface cracks can be predicted using surface hardness as an index. Reduced fatigue strength can cause surface cracks, leading to early fractures. Thus, employing a (9.81 N) stress, this study examined the surface hardness of shaded zirconia specimens. [6]

Also, Donmez MB et al (2021) [18] examined the effects of coloring liquid immersion of different durations on flexural strength, Vickers hardness, and zirconia color. The result showed that Immersing zirconia specimens in coloring liquid decreased the flexural strength and hardness values.

The null hypothesis that the coloring technique will not affect the surface hardness of monolithic zirconia blocks was rejected.

Regarding the microhardness, in this study, the highest Vickers hardness number was in group (ISZ), in which zirconia samples were pre-colored (A2). The lowest values were in the group (ESZ), in which samples were immersed in coloring liquid, and there was no significant difference between group (ISZ) and group (ESZ). These results agreed with Nam J-Y et al (2017) [6], who concluded that the coloring liquid group, including acid, exhibited the lowest average hardness values, ranging from 1220 ±45 to 1311 ±23 HV. There were no notable variations in mean hardness between the preshaded zirconia and aqueous coloring liquid groups (P>.05).

According to the previous study, zirconia's strength and surface hardness significantly dropped when diluted with a pink liquid containing Er and Nd.ions. However, other research found no discernible shift in the hardness or flexural strength. [6]

According to Liu Q, there was no discernible change in microhardness between the two shading times. Longer shade times reduced the zirconia ceramic's biaxial flexural strength but did not influence the surface microhardness. [20]

In this study, scanning under an electron microscope showed that surface morphology was modified after coloring zirconia samples. Any coloring method applied revealed several deep, multidirectional scratches with an inhomogeneous texture.

CONCLUSION

Within the limitation of this in vitro study, it can be concluded that coloring techniques affect the surface hardness of monolithic zirconia blocks—the highest Vickers hardness number in pre-colored zirconia samples.

CLINICAL IMPLICATIONS &LIMITATIONS

Precolored zirconia is more preferred than white zirconia in dental use. Great caution must be taken in the coloring procedure of zirconia crowns.

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