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EFFECT OF DIFFERENT SHADING TECHNIQUES ON COLOR, TRANSLUCENCY AND FLEXURAL STRENGTH OF ZIRCONIA CERAMIC AFTER AGING

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ABSTRACT

Purpose: The ultimate appearance of all ceramic restorations is significantly influenced by color and translucency. The performance of the restoration is significantly influenced by flexural strength. This study's objective was to assess how various shading methods affected the color, translucency, and flexural strength of monolithic zirconia ceramic as it aged.

Materials and Methods: Thirty zirconia discs with a diameter of 12mm and thickness of 1mm were divided into two groups of 15 discs each according to the technique of shading, the first group was constructed from A2 pre-shaded zirconia while the second group was constructed from un-shaded zirconia that was shaded by immersion in A2 coloring liquid. Aging was done using acetic acid 4% for both groups. Color and translucency were measured using a spectrophotometer before and after artificial aging. Flexural strength was measured using a universal testing machine before and after aging. Data were collected, tabulated, and statistically analyzed.

Results: Color change of both pre-shaded and liquid-shaded zirconia were within the clinically acceptable range (1.7±0.3) and (1.9±0.3) respectively and there was no significant difference in translucency between both groups' P value (0.253). Flexural strength increased from (921.9±73.3) before aging to (1025.1±178.5) after aging with pre-shaded zirconia while it decreased from (938.6±32.7) before aging too (899±54.5) after aging with liquid shaded zirconia but this difference was statistically non-significant.

Conclusion: The shading technique had no noticeable effect on color, translucency, or flexural strength before or after aging.

KEYWORDS: Zirconia ceramic, shading techniques, Aging.

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INTRODUCTION

The strength and esthetic of the restoration are the most crucial variables that could impact the ultimate decision. Although ceramic-metal restorations are sufficient, doctors prefer to use all-ceramic restorations because of the metal framework's opaque construction, the veneering porcelain's tendency to chip, and the corrosion of non-precious metals **(1,2)** due to their natural appearance and pleasing esthetic **(3)**.

Because all-ceramic restorations have poorer mechanical qualities, there are few uses for them. In order to address this issue, reinforced dental porcelain materials like lithium disilicate, aluminum oxide, and zirconium oxide have been created. (4)

Due to the advancement of CAD/CAM technology, zirconia stands out among these new materials in terms of its excellent mechanical qualities, making it the material of choice for scenarios involving significant loading forces. (5)

The restoration's esthetic is significantly influenced by its optical characteristics such as light transmittance and reflectance (3). The material's translucency is crucial in creating a realistic look. (, 6, 7, 8)

Zirconia restorations are more esthetic than ceramo-metallic, although the white and opaque appearance is still unfavorable. In order to achieve the good color matching, zirconia can be tinted. (3)

To solve the esthetic issues, two techniques for coloring zirconia restorations have been devised. In order to get the right shade, zirconia restorations are either immersed in coloring liquids before sintering, or pigments are added to zirconia powder before pressing the milling blocks, where the color of the block is predetermined at the time of its creation. (4)

The manufacturers of the coloring liquids mix different oxides into them to create a variety of shades (9, 10) These oxides have entered the pores of the milled green zirconia (4). Dental restorative materials' color stability is just as crucial to long-term clinical success as their mechanical qualities. The color stability of the material is what determines whether restorations that mimic natural tooth color look good on the outside. (11, 12)

Flexural strength is a crucial mechanical characteristic of a material that helps anticipate its performance and is significantly influenced by the extent of flaws and faults on its surface. (13)

Dental literature on the effects of coloring solutions on the mechanical properties of zirconia is limited, and opinions are varied (4)

So, the aim of this research was to evaluate the effect of different shading techniques on the color, translucency, and flexural strength of zirconia ceramic after aging.

The null hypothesis of this study: different shading techniques would have an effect on the color, translucency, and flexural strength of zirconia ceramic after aging.

MATERIALS AND METHODS

Thirty zirconia samples in the form of discs (12mm diameter x 1mm thickness) were constructed from two partially sintered green stage monolithic zirconium dioxide blocks, one was A2 pre-shaded (Ceramill Zolid HT+ PS A2 98X14, Amanngirrbach, Austria, Lot: 2004003) and the other was un-shaded which was colored by immersion in A2 coloring liquid (Ceramill Zolid HT+ white 98X16, Amanngirrbach, Austria, Lot: 1902000).

First, the zirconia discs were installed on the tray of the electric isoMet micro saw 4000 where it was cut into slices using a saw diamond disk of 0.6mm thickness (Buehler, USA). After that, the slices were sent to a metal turning machine which converts them into cylinders. The cylinders were installed on a specially designed metal using self-acrylic resin.

The discs were then cut from the constructed cylinders using the electric isoMet 4000 micro saw. Cutting the discs was done using a saw diamond disk of 0.7 mm thickness (Buehler, USA) at a speed of 2500 rpm under water coolant and a feeding rate of 5mm/min.

The discs were divided into two groups of 15 discs each according to the technique of shading of the blocks which were used for their construction; the first group was A2 pre-shaded zirconia while the second group was unshaded zirconia that was shaded by immersion in A2 coloring liquid.

All the discs were polished using zirconia polishers (Zirkonzahn, Gais, Italy). The discs were machine polished in a consistent procedure using wet 320, 400, 600, 800, 1000, and 1200 silicone carbide sheets (Microdent, 3R Ind, Com. Brazil). The order of the polishers was done in accordance with the manufacturer's recommendations.

After finishing all the discs were subjected to ultrasonic cleaning (Cavitron, Dentsply Intl, York, Pa) on both sides in distilled water (El fath for drug and cosmetic industry, Borg el Arab new city, Cairo) to remove any residues on the surface and air dried. Discs were put for 20 minutes under the infrared drying lamp using a Ceramill Therm 3 machine (Amann Girrbach, Austria).

The discs were carefully measured using a digital caliper (Praecimeter S, 0.01mm; Renfert Gmb H, Hilzingen, Germany) to ensure similar thicknesses. The discs were cut larger than the required dimensions by 20% to compensate for sintering shrinkage.

The un-shaded zirconia discs were immersed into A2 coloring liquid (Cera mill liquid, LOT 3489, Amanngirrbach, Austria) according to the vita classic scale (Vita Zahnfabrik, H Rauter GmbH & CO. Bad Sackingen, Germany). using a plastic tweezer and held for 20 seconds and then dried for 30 minutes under an infrared drying lamp using a Ceramill Therm 3 machine (Amann Girrbach, Austria)

The pre-shaded and the liquid-shaded zirconia discs were sintered according to the manufacturer`s instructions following the protocol. The firing cycle was done at TABEO sintering machine (MIHM-VOGT GmbH, Stutensee Blankenloch, Germany). The firing cycle was controlled as followings: the temperature was raised to 950^0c for 1.5 hours and maintained for 3 hours, and then raised to 1550^0c for 1.5 hours and maintained for 3 hours.

Glazing of the discs was done in a vacuum in Programat P310 (Ivoclar Vivadent AG, Ser.NO. 350371, Liechtenstein, Austria) ceramic furnace using a glazing paste (Cera Motion one touch, Denta aurum, Germany) following the protocol: the temperature was raised to 950^0c at the firing rate of 30^0c/minute and maintained for 30 seconds, and then cooled down to 300^0c at 15^0c/minute as shown in figure (1).

To estimate the color parameters (hue, value, chroma) and the translucency parameter, a reflection spectrophotometer (Cary 5000, USA) was used according to the CIE 1976 L*a*b* color scale relative to the CIE standard illuminant D65 (as defined by the International Commission on Illumination) which corresponds to average daylight.

The translucency parameter of each sample was obtained by calculating the difference between the sample against the white background and the black background using the following equation:

$$Tp= \{(Lb^*-Lw^*)^2+(ab^*aw^*)^2+(bb^*bw^*)^2\}^{1/2}$$

L*: refers to lightness.

a*: refers to redness to greenness.

b*: refers to yellowness to blueness

The flexural strength for all the discs which were used for measuring the flexural strength before aging was measured using a universal testing machine

Fig. (1) a): Steps of construction of zirconia discs. b): The constructed discs

(Instron Industrial Products, Norwood, MA, USA) in accordance with the ISO standard 6872.

The samples were tested dry at room temperature where each sample was rested on three symmetrically based steel balls with a diameter of 3.2 mm and positioned 120 degrees apart on a circle with a diameter of 10 mm and the load was applied to the center of the top surface by the piston until fracture occurred where the cross-head speed of the piston was 1mm/min as shown in figure (2).

The flexural strength for each sample was calculated from the following equation as follows:

$$S=0.2387P(X-Y)/d^2$$

Where:

S: bi-axial flexural strength (MPa)

P: fracture load (N)d: sample disc thickness at fracture origin (mm).

$$X=(1+v)\text{in } (B/C)^2+\{(1-v)/2\}\ (B/C)^2$$

$$Y=(1+v)\text{in } (A/C)^2+\{(1-v)/2\}\ (A/C)^2$$

Where:

v: piston coefficient (ceramic=0.25) according to ISO standardization 6872.

A: radius of support circle (mm).

B: radius of loaded area (mm).

C: radius of sample disc

All the discs which were used for measuring color, and translucency and those which were used for measuring flexural strength after aging were subjected to aging using acetic acid 4% (Vosol, England).

According to ISO 6872 standards for hydrolytic resistance of dental ceramic materials, the samples were first washed three times with ethyl alcohol,

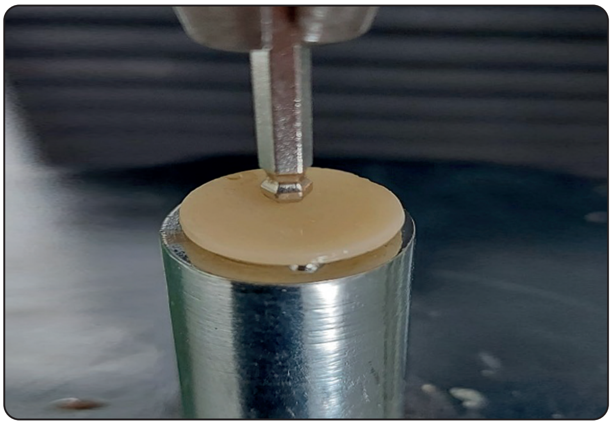

Fig. (2) A disc mounted in the jig of the universal testing machine ready for fracture

dried, and then immersed in a 4 percent acetic acid solution at a temperature of 800°C and held for 16 hours.

After 16 hours of immersion, the samples were removed, rinsed with distilled water and alcohol then dried

The color parameters (hue, value, chroma) and translucency parameters for the discs were measured using a spectrophotometer (Cary 5000, USA) after aging where all the steps during using a spectrophotometer were the same as before aging.

The total color difference between the samples before aging and after aging was calculated as ΔE from the equation

$$\Delta E = ([\Delta L^*]^2 + [\Delta a^*]^2 + [\Delta b^*]^2)\ ½$$

Where:

ΔL =L before aging – L after aging

Δb = b before aging – b after aging

Δa = a before aging – an after aging

The flexural strength was measured for the discs which were used for measuring the flexural strength after aging where all the steps using a universal testing machine were the same as before aging.

RESULTS

The results of the mean color change values of the A2 pre-shaded zirconia and the A2 liquid shaded zirconia showed that the color change that occurred with A2 liquid shaded zirconia (1.9±0.3) was more than the A2 pre-shaded zirconia (1.7±0.3).

The color change that occurred in both groups was in the clinically acceptable range. The color change between the two groups was statistically non-significant, P value (0.253) as shown in table (1) and Figure (3).

TABLE (1): Independent Samples T-test for quantitative data between every two groups. Color change (ΔE) for the A2 pre-shaded and the A2 liquid-shaded zirconia after aging

ΔE	A2		P value
	Pre shaded	Liquid shaded	Pre vs Liquid
	N=5	N=5	A2
Range Mean ± SD	(1.4-2.2) 1.7±0.3	(1.5-2.3) 1.9±0.3	0.253

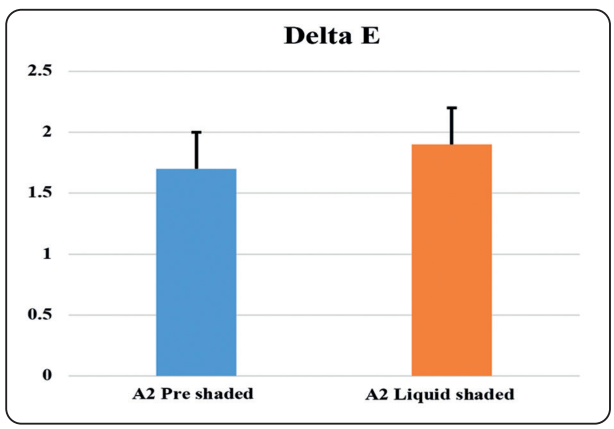

Fig. (3): Color change of the A2 pre-shaded zirconia versus the A2 liquid shaded zirconia after aging

The comparison of the mean translucency values of A2 liquid shaded zirconia and A2 pre-shaded zirconia before and after aging revealed that the pre-shaded zirconia had a lower translucency than the liquid shaded zirconia before aging. The two groups' differences in translucency were statistically insignificant P values (0.556). P value (0.916), indicates that there was no change in translucency between the two groups after age as shown in table (2) and Figure (4).

TABLE (2): Independent Samples T-test for quantitative data between every two groups. The translucency of the A2 pre-shaded group versus the A2 liquid shaded group before and after aging

Translucency	Before aging		After aging		P value	
	A2 Pre shaded	A2 Liquid shaded	A2 Pre shaded	A2 Liquid shaded	Pre vs Liquid	
	N=5	N=5	N=5	N=5	A2	A2
Range Mean ± SD	(12.8-13.8) 13.3±0.4	(12.4-14.4) 13.6±0.9	(12.6-14.4) 13.1±0.7	(13-13.4) 13.1±0.2	0.556	0.916

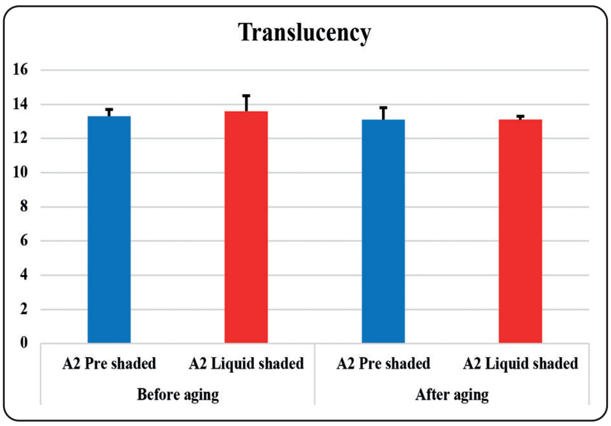

Fig. (4): Translucency of the A2 pre-shaded group versus the A2 liquid shaded group before and after aging

The findings of comparing the mean flexural strength values of A2 pre-shaded zirconia and A2 liquid shaded zirconia before and after aging revealed that the flexural strength of the latter was greater before aging. The two groups' flexural strength differences were not statistically significant, P value (0.654). After aging, the A2 pre-shaded group's flexural strength increased, whereas the A2 liquid shaded group's flexural strength decreased. The difference in flexural strength between the two groups was statistically non-significant, P value (0.169) as shown in table (3) and Figure (5).

TABLE (3): Independent Samples T-test for quantitative data between every two groups. Flexural strength of the A2 pre-shaded group versus the A2 liquid shaded group before and after aging

Flexural strength	Before aging		After aging		P value	
	A2 Pre shaded	A2 Liquid shaded	A2 Pre shaded	A2 Liquid shaded	Pre vs Liquid	
	N=5	N=5	N=5	N=5	A2	A4
Range Mean ± SD	(811-1004.5) 921.9±73.3	(906.2-989.9) 938.6±32.7	(730.2-1199.2) 1025.1±178.5	(816.8-961.1) 899±54.5	0.654	0.169

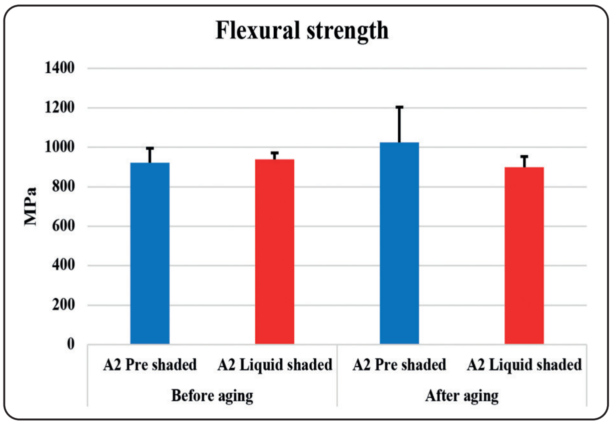

Fig. (5): Flexural strength of the A2 pre-shaded group versus the A2 liquid shaded group before and after aging

DISCUSSION

In this study, pre-shaded and liquid-shaded monolithic translucent zirconia ceramics were compared in terms of their optical and mechanical characteristics. Another objective was to see whether these qualities change with age.

Translucency is crucial for achieving a natural-looking appearance and is a consideration when choosing materials [14, 15].

There are two varieties of zirconia available today: core and monolithic. The major objective of both monolithic and multilayer restorations is to restore form, function, and aesthetics while causing the least amount of harm and extending the life of the remaining natural dentition.

Modern, cutting-edge technology that is accessible in both fields can produce results that are above average to superb in terms of esthetic, strength and whether the anterior or posterior portions are being restored can all influence the clinical decision between the two. The weakest link in all restorations is the multilayer porcelain that is layered over the core and gives under shear or flexural loads between 90 and 140 MPa.

Due to their great flexural strength, monolithic restorations are excellent for usage in stress-bearing areas (380–1,000 MPa). In the posterior zone or as anterior or posterior bridges with short spans, they can be employed as a single bulk material without the inferior outer layer of stacked porcelain [16]. It is advised to use translucent monolithic zirconia ceramic for anterior and/or posterior monolithic crowns [17].

Monolithic zirconia restorations have a number of benefits, including the need for only minimally invasive tooth preparation, little wear on neighboring teeth, and great flexural strength [18]. Besides, this restoration can be produced using a CAD/CAM system in a short time without the need for the addition of veneering porcelain [19,20] so lack the unwanted complications of chipping [21,22].

The discs utilized in this investigation have dimensions of 12mm in diameter and 1mm in thickness [23]. A ceramic disc with a thickness of 1mm was employed to balance strong aging resistance, appropriate cosmetic qualities, and adequate mechanical properties. The spectrophotometer sample chamber size of 12mm diameter was chosen to allow the testing device to capture the readings conveniently and precisely [24, 25].

Discs were constructed using Iso Met micro saw 4000 [23] because it enables cutting materials with little sample deformation and less kerf loss, which may provide uniform thickness for all samples and prevent any optical changes that may result from thickness changes [26].

One of the biggest issues with monolithic zirconia is accurately reproducing tooth color [27]. It has been difficult to create tooth-colored monolithic zirconia restorations due to the materials' semi-translucent and white opaque look. Zirconia can be tinted to resemble the color of tooth structures. [28, 29]

Monolithic zirconia restorations can be colored using one of two techniques: either adding metal oxides to the powders before pressing or infiltrating metal salt solutions onto the zirconia's surface once it has already been pre-sintered [27].

Due to market availability, acid-based coloring liquid was employed. Acidic or neutral liquid colorings are both acceptable. Due to its acidity (PH 1-3), liquids with an acidic base can permeate deeply through restorations. In addition, certain metal salts can be dissolved in acid. According to VITA traditional or VITA 3D Master hues, manufacturers have offered a variety of liquid shades. Zirconia ceramic's translucency is affected by the color of the color liquid. [3].

Zirconia ceramic is best colored using the immersion technique rather than the brush method, hence un-shaded zirconia discs were colored using this method [28, 30].

The samples were immersed in coloring liquid for 20 seconds [31, 32]. Prolonged dipping time of more than 2 minutes may dampen the fracture strength and deteriorate the color [4].

The color parameters and translucency parameters were assessed before and after aging because the use of a spectrophotometer during the creation of all ceramic crowns enables the dentist and technician to communicate critical information to one another about the shade of tooth preparation [33].

It is well known that the maximum tensile stress that occurs in the middle loading area is the biaxial flexural strength test [34, 35, 36]. Strength [37] is defined as the maximum strength needed to fracture a material or cause plastic deformation and can be used to assess the performance of brittle materials like ceramics [38]. Biaxial flexural strength was measured for the pre-shaded and the liquid-shaded discs using a universal testing machine [39] because it is a widely used machine in research work. [40]

The impact of long-term aging must be assessed with the rise in the usage of monolithic entire contour crowns and fixed partial dentures [41] because it might have an impact on the material's optical and mechanical properties, which directly affect the material's suitability for usage in the future [42].

The results showed that the color change after aging with A2 pre-shaded group and the A2 liquid shaded group was within the clinically acceptable range. This result is in agreement with several investigators, including ***Salah Eldine (2021)*** [43] who examined how indirect zirconia-containing restorative materials changed over time and came to the conclusion that zirconia ceramic displayed the best color stability.

Also, ***Colombo et al (2017)*** [44] investigated the A2 shade of CAD/CAM zirconia ceramic and came to the conclusion that zirconia ceramic maintains its color over time whether exposed to oral conditions or staining agents.

In addition, **Arocha et al (2013)** [45] also revealed that ceramic is color stable. **Gawriolek et al (2012)** [46] investigated the color and luminescence of certain dental materials and came to the conclusion that composite resin did not demonstrate the same color stability as ceramic materials.

Also, **Shereen and Rasha (2020)** [47], **Vasiliu et al (2020)** [48], **and Haralur et al (2019)** [49] were concluded that there was no significant change in the color of the ceramic before and after aging.

This might be a result of the material's microstructure having an impact on its optical characteristics. Aging may cause the Y-TZP crystal structure to alter from tetragonal to monoclinic [50]. Aging causes more monoclinic phases to occur [51]. The opacity can vary depending on the optical characteristics of the monoclinic and tetragonal crystals [52].

Different refractive indices are produced by the anisotropic and refractive nature of the monoclinic and tetragonal phases [53]. One of the crucial elements that influences color is the level of birefringence [54]. However, the impact of aging on color is time-dependent, with aging having little to no impact on color for aging times of less than 20 hours [50]. A2 pre-shaded compared A2 liquid shaded before and after age, as well as A2 pre-shaded versus A2 liquid shaded after aging, all showed a decrease in translucency; however, this decline was statistically non-significant.

This result was in agreement with **Kou et al (2019)** [55] who investigated the impact of artificial aging on highly translucent zirconia and came to the conclusion that the translucency of zirconia ceramic was unaffected by aging.

The transition of zirconia from the t phase to the m phase may be the cause of the change in translucency that occurs during gingivitis. Micro cracks are created and surface porosity rises as a result of an increase in the m phase content brought on by aging, which also increases surface roughness, light scattering, and reflection [56].

Aging may also cause the coexistence of the t and m phases, which would reduce translucency by increasing the difference in refractive indices for an incident light beam [57].

The results indicated that the decrease in translucency was statistically non-significant; this could be explained by the relationship between translucency Y-TZP and the aging holding time, where translucency was not significantly impacted when the aging time was less than 20 hours [50].

However, this result was controversial with **Alamledin et al (2020)** [58] investigated how aging affected monolithic zirconia's translucency and came to the conclusion that aging affected the material's translucency, leading to an increase in the translucency parameter.

Regarding the flexural strength test results, the flexural strength of the A2 liquid shaded group was lower than that of the A2 pre-shaded group before aging but this decrease was statistically non-significant. This is in agreement with several investigators, **Ebied et al (2014)** [59] **and Hjerppe et al (2008)** [60] whom all concluded that coloring had a significant effect on the mechanical properties of zirconia.

Also, **Shah et al (2008)** [61] studied the influence of coloring using various metal oxides on the color and flexural strength of zirconia ceramic and concluded that coloring caused a decrease in the flexural strength and this decrease was significant. This result is controversial to **Sen et al (2018)** [62] who studied the influence of coloring and sintering on the translucency and flexural strength of zirconia ceramic and concluded that coloring liquids did not affect the flexural strength of zirconia ceramic.

The results of this study showed that the flexural strength of A2 pre-shaded zirconia was increased after aging. This is in agreement with **Kou et al (2019)** [55] who investigated how artificial aging affected highly translucent zirconia and came to the conclusion that it increased the zirconia ceramics mean flexural strength.

The volumetric expansion caused by the t-m phase shift at a restricted area around the superficial faults resulted in compressive stress focused there, which in turn prevented the crack from spreading and may have contributed to the increase in the BFS [63] which need more force to bring the sample to fracture.

This resulted in controversy with **Flinn et al (2012)** [64] who investigated the aging of yttria-stabilized zirconia polycrystal and came to the conclusion that the flexural strength significantly decreased over time. This can be because the aging media used differs.

The study's findings demonstrated that after aging, A2 liquid-shaded zirconia's flexural strength was lower than that of A2 pre-shaded zirconia, but this difference was statistically non-nsignificant. This is in agreement with **Agingu et al (2018)** [65] investigated the impact of various coloring techniques on the aging behavior of monolithic zirconia and came to the non-significant conclusion that aging and coloring techniques decreased flexural strength.

CONCLUSIONS

We can conclude that:

1. Liquid-shaded zirconia showed more color change than pre-shaded zirconia after aging.
2. The aging and technique of shading of zirconia ceramic have no significant effect on translucency.
3. There is an increase in the flexural strength of zirconia ceramic after aging with the pre-shaded samples and a decrease with the liquid-shaded samples.

Clinical recommendations

a. Pre-shaded zirconia ceramic is recommended than liquid-shaded due to:

1. The minimal color change that occurred with aging.
2. It maintains high flexural strength after aging.

b. The color of the material should be accurately selected as it affects the translucency.

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