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EFFECT OF SHADING TECHNIQUES AND CHEMICAL AGING ON TRANSLUCENCY AND SURFACE ROUGHNESS OF MONOLITHIC ZIRCONIA

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ABSTRACT

Aim: Evaluation of different shading techniques and aging effect on translucency (contrast ratio) and surface roughness of monolithic zirconia ceramic.

Materials and Methods: Forty zirconia (Zolid High translucent) samples were constructed in the form of discs. Samples were divided according to the technique of shading into two groups as follows: The first group: Pre shaded zirconia, was used as a control (n=20). The second group: Un-shaded zirconia, colored by immersion in a coloring liquid (n=20). Each group was then divided into 2 subgroups (n=10) according to the shade of samples (A2, A4). Each subgroup was furtherly subdivided into 2 subgroups(n=5) for measuring translucency (contrast ratio) & surface roughness before & after aging. Data were collected, tabulated & statistically analyzed.

Results: Results recorded no significant difference in CR value between the two shading techniques (pre-shaded & liquid-shaded) before & after aging. Whereas there was a significant difference in CR value between the two shades (A2, A4). Regarding the surface roughness, there was a non-significant difference between the pre-shaded & liquid-shaded zirconia groups, while there was a significant difference between both shades (A2 & A4) before & after aging.

Conclusions: Shade significantly affected translucency while shading technique & aging had no significant effect. Aging & shade significantly affect surface roughness, while there was no significant effect of the shading technique.

KEYWORDS: shading technique, translucency, surface roughness, zirconia

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INTRODUCTION

Dental ceramics, due to their exceptional esthetic properties, biocompatibility & wear resistance, are progressively used as anterior & posterior restorations. Zirconia (ZrO_2) is a polycrystalline ceramic of the transition metal zirconium (Zr) that has various dental applications attributable to the CAD/CAM technology advancement[1,2]. Owing to its exceptionally outstanding mechanical properties, it is usually chosen in situations where high occlusal forces are anticipated. Also, it is excellent biologically; it is low cytotoxic & has high corrosion resistance and minimal bacterial adhesion.

Restorations' optical properties, such as reflectance & light transmittance highly affect their esthetic outcome [3]. Material translucency is a major participant in the natural realistic lifelike appearance. However, this esthetic success depends mainly on the restoration's color stability. For long-term clinical success, color stability is as important as mechanical properties [4,5]. Therefore, restorative material selection in different cases seems crucial [6].

To overcome the esthetic inconvenience of zirconia restorations, 2 methods were advocated for their coloring. Pigments are either incorporated into zirconia powder before block pressing, where the block color is established at the instance of its fabrication, hence called pre-colored zirconia. The second method adopts the immersion of the restoration in differently shaded coloring liquids before sintering [7]. To get different shades, a variety of oxides are added by manufacturers to the coloring liquids. These oxides infiltrate into pores of milled green zirconia.

Ceramic restorations' surface roughness is a characteristic feature that might enhance surface discoloration, and wear of opposing teeth or ceramic restoration. Dental restorations with high surface roughness are proven to increase plaque accumulation & subsequently dental caries. [5] It was recorded that smooth restorations enhance oral comfort &patients' hygiene [6]. Stylus & optical profilometers are used for the measurement of surface roughness. stylus profilometers concept depends on a probe physically tracing the surface, while optical profilometry is non-contact and usually uses a light beam or laser to scan the surface.

Literature about coloring liquids' effect on the physical properties of zirconia is very limited & there is no consensus about their effect. So, the aim of this research is to assess the effect of different shading techniques on translucency & surface roughness of zirconia ceramic before & after aging.

The null hypothesis

The null hypothesis advocates that different shading techniques will have no effect on the translucency & surface roughness of zirconia ceramic before & after aging.

MATERIALS AND METHODS

The number of samples per group was determined according to a power calculation based on previously attained data in an earlier study [10]. Mean translucency before & after aging was 11.66±0.73 & 10.8±0.67, respectively, having a 0.75 correlation coefficient. The sample size of 5 samples was determined to provide 80% power for paired samples T-test at the level of 0.05 significance using G Power 3.1 9.2 software.

Forty disc-shaped samples were constructed from zirconia (Zolid High translucent, Amann Girrbach, Austria). Samples were divided according to the shading technique into two groups as follows: The first group was pre-shaded zirconia, used as a control (n=20). The second group: Un-shaded zirconia, colored by immersion in a coloring liquid (n=20). Each group was then divided into 2 subgroups (n=10) according to the shade of samples (A2, A4). Each subgroup was furtherly subdivided into 2 subgroups(n=5) for measuring translucency (contrast ratio) & surface roughness before & after aging.

Zirconia blank was positioned on the tray of the electric IsoMet micro saw 4000, where it was sectioned by a saw diamond disk (Buehler, USA). Sections were then sent to a metal turning machine to be shaped into cylinders. Cylinders were installed on a specially designed metal holder, using self-acrylic resin (IMICRYL, Konya, Turkey) to be able to install them for sectioning in the form of discs. Using the electric IsoMet 4000 micro-saw at a speed of 2500 rpm under water coolant & a feeding rate of 5mm/min, discs were cut from the constructed cylinders.

All discs were machine finished & any sharp edges were removed using silicon carbide papers (grits 400,600) (Microdent, 3R Ind, Com. Brazi). They were polished sequentially following the manufacturer`s instructions, using wet 320, 400, 600, 800, 1000 &1200 silicon carbide zirconia polishers (Zirkonzahn,Gais,Italy). The thickness of each disc was checked on 5 different sites (center & 4 points on borders) using a digital micrometer with 1um accuracy (Praecimeter S, 0.01mm; Renfert Gmb H, Hilzingen, Germany), as the samples were cut larger than the required dimensions [(11,12)]. Then all discs were soaked in distilled water & ultrasonically cleaned (Cavitron, Dentsply Intl, York, Pa) from any surface residues then air dried. The samples were held under the infrared drying lamp using Ceramill therm 3 (Amann Girrbach, Austria) for 60 minutes, to protect the sintering machine holding tray from rust [(13)].

Group 2 discs were air cleaned for the removal of any residual dust on their surfaces. An adequate amount of A2 & A4 staining liquids were poured into clean immersion containers. Group 2 subgroups 1 & 2 were immersed for 20 seconds in coloring liquids A2 & A4 (Cera mill liquid, LOT 3489, Amann Girrbach, Austria), respectively. Then plastic forceps were used to remove the samples from the liquid & carefully dabbed off using a clean paper cloth for to remove excess liquid. The colored samples were put onto a sintering plate as a clean, non-absorbent, temperature-resistant surface. They were then dried in a domestic oven directly after coloring for around 30 minutes at 90°C.

The sintering program with pre-drying was selected following the instructions of the manufacturer using a ceramill therm3 (Amann Girrbach, Austria), where the discs were dried for 60 minutes. The firing cycle included a temperature raise-up to 1450°C, 2 hours dwell time & heating rate 10°C/min., then slow cooling. The thickness of the samples was assessed where the final dimensions of each disc were 12mm in diameter & 1mm thick. Glazing of the discs was done using a glazing liquid (Ceramil liquid "new formula)" up to 850^{0}c, rate 40°C/minute & sustained for 1 minute.

Measurements of translucency

For each sample, three translucency contrast ratio (CR) measurements were recorded on white & black backgrounds using a spectrophotometer (Cary5000, USA) before & after aging. CR is calculated from the material's reflectance measurement over white & black backgrounds according to the equation: CR = Yb/Yw. where Yb is the material's reflectance over the black background, while Yw is the material's reflectance over the white background. [(14)] CR =1.00 for an opaque material.

Measurements of surface roughness

A contact profilometer (SJ-210 surface roughness tester Mitutoyo Japan) was used for roughness measuring. Each sample was horizontally placed & fitted to the specimen holder so that the surface which will be subjected to the roughness test was in the horizontal direction. The profilometer was first calibrated & with settings of 12mm measuring distance, 0.5mm/s speed & 1mm/s returning time with 0.75 mN force. The specimen holder moves in a vertical direction till the specimen surface touches the measuring tip angled at 60^{o} (radius=2microns)

Aging process

As per hydrolytic resistance of dental ceramic materials standards; ISO 6872, the samples were washed 3 times with ethyl alcohol & dried, then immersed in a laboratory prepared 4% acetic acid solution at a temperature of 80°C for 16 hours in a reflux system. A reflux system is an apparatus used in chemistry laboratories to prevent solvent loss in a heated environment. After 16 hours, samples were removed from the acetic acid, rinsed with alcohol & distilled water, then dried.

Translucency (CR) & surface roughness were measured after aging where all the steps were the same as before aging.

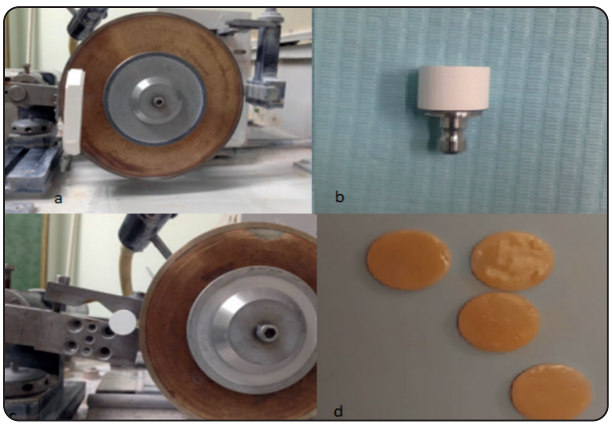

Fig. (1) a: zirconia slice cutting, b: zirconia cylinder, c: mounted zirconia cylinder, d: zirconia discs

Statistical method:

Coding & tabulation of the collected data as well as statistical analysis using **SPSS program (Statistical Package for Social Sciences) software version 25** was performed. Mean, Standard deviation (SD) & range were calculated. **Shapiro-Wilk test** was used for the detection of the normality of data distribution**. Three Way ANOVA test** was done to determine the impact of combining aging, shading technique, and shade on different variables. Analysis was performed for quantitative data between the 2 groups using the **Independent Samples T-test** & within each group using **Paired Samples T-test**. The level of significance was taken at (**P value ≤ 0.05**)

RESULTS

I - Contrast Ratio (CR)

TABLE (1) Three-Way ANOVA test for determining the effect of aging, shading technique, color & interaction among them on translucency (CR).

Contrast ratio	F	P value
Aging	0.441	*0.511*
Shading technique	1.683	*0.204*
Shade	**158.809**	***<0.001****
Aging * Shading technique	0.178	*0.676*
Aging * Shade	0.982	*0.329*
Shading technique * Color	0.205	*0.654*
Aging * Shading technique * Shade	0.002	*0.965*
R Squared = 0.835		

* *Indicates significant difference at* ***P value < 0.05***

By studying the factors that can affect the CR of zirconia ceramic using a three-way ANOVA test, results displayed that the shade factor significantly affected the CR values, while there was no significant effect for the shading techniques. Aging did not significantly affect the CR. All the interactions of shade* technique, color* aging, technique* aging & shade* technique* aging had no significant effect on CR.

TABLE (2) Comparison of contrast ratio between different shading techniques of different shades before & after aging

Aging	Shade	Contrast ratio				P value
		Pre shaded (N=5)		Liquid shaded (N=5)		
		Range	*Mean±SD*	*Range*	*Mean±SD*	
Before aging	A2	(0.59-0.74)	0.68±0.06	(0.63-0.78)	0.7±0.06	*0.657*
	A4	(0.84-0.88)	0.86±0.02	(0.85-0.94)	0.89±0.04	*0.201*
After aging	A2	(0.68-0.73)	0.71±0.02	(0.66-0.76)	0.72±0.04	*0.818*
	A4	(0.83-0.93)	0.86±0.04	(0.85-0.92)	0.88±0.03	*0.457*

Independent Samples T test ***Significant level at P value < 0.05***

TABLE (3) Comparison of contrast ratio between different shades with different shading techniques before & after aging

Aging	Shading technique	Contrast ratio				P value
		A2 (N=5)		A4 (N=5)		
		Range	*Mean±SD*	*Range*	*Mean±SD*	
Before aging	Pre shaded	(0.59-0.74)	0.68±0.06	(0.84-0.88)	0.86±0.02	***<0.001****
	Liquid shaded	(0.63-0.78)	0.7±0.06	(0.85-0.94)	0.89±0.04	***0.001****
After aging	Pre shaded	(0.68-0.73)	0.71±0.02	(0.83-0.93)	0.86±0.04	***<0.001****
	Liquid shaded	(0.66-0.76)	0.72±0.04	(0.85-0.92)	0.88±0.03	***<0.001****

Independent Samples T test ****: Significant at P < 0.05***

The results showed that when studying the effect of the different shading techniques (pre-shaded & liquid-shaded), no significant difference was recorded in the translucency CR value between the two techniques. (p>0.05). Also, there was no significant difference in CR value before & after aging. Whereas there was a significant difference in CR value between the two colors (A2, A4).

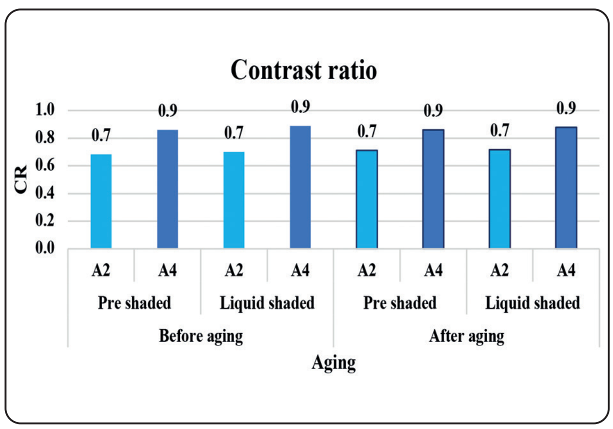

Fig. (2)

II - SURFACE ROUGHNESS

TABLE (4) Three Way ANOVA test for determining the effect of aging, shading technique, shade & interaction among them on surface roughness.

Surface roughness	F	P value
Aging	39.328	<0.001*
shading technique	1.894	0.178
Shade	16.927	<0.001*
Aging * shading technique	26.075	<0.001*
Aging * Shade	3.337	0.077
shading technique * Color	15.133	<0.001*
Aging * shading technique * Shade	4.851	0.035*
R Squared = 0.771		

Three Way ANOVA test

Significant level at P value < 0.05

By studying factors affecting the surface roughness of the samples using the three-way ANOVA test, results showed that aging & shade factors had a significant effect on surface roughness. Different shading techniques had no significant effect on surface roughness value (Ra). All the interactions either shade*technique, technique*aging & shade* technique* aging had a significant effect on surface roughness while the shade*aging had a non-significant effect.

Ra of the pre-shaded zirconia, both shades (A2 & A4) showed statistically significant higher mean (Ra) values after aging than before aging. While liquid-shaded zirconia, both shades (A2 & A4) showed higher but statistically non-significant mean (Ra) values after aging than before aging. While there was a significant difference between both shades (A2 & A4) subgroups.

TABLE (5) Comparison of surface roughness between different shades of different shading techniques before and after aging

Aging	**Shading technique**	**Surface roughness**				**P value**
		A2 (N=5)		**A4 (N=5)**		
		Range	***Mean±SD***	***Range***	***Mean±SD***	
Before aging	**Pre shaded**	(0.28-0.48)	0.37±0.09	(0.33-0.85)	0.53±0.24	*0.194*
	Liquid shaded	(0.67-0.91)	0.79±0.1	(0.29-0.65)	0.43±0.14	***0.002****
After aging	**Pre shaded**	(0.79-1.19)	1.03±0.17	(0.79-0.89)	0.85±0.05	***0.045****
	Liquid shaded	(0.67-0.91)	0.82±0.1	(0.38-0.66)	0.5±0.1	***0.001****

Independent Samples T test ****: Significant level at P value < 0.05***

TABLE (6) Comparison of surface roughness before & after aging of different shading techniques and different shades.

Shading technique	**Shade**	**Surface roughness**				**P value**
		Before aging (N=5)		**After aging (N=5)**		
		Range	***Mean ± SD***	***Range***	***Mean ± SD***	
Pre shaded	**A2**	(0.28-0.48)	0.37±0.09	(0.79-1.19)	1.03±0.17	***0.004****
	A4	(0.33-0.85)	0.53±0.24	(0.79-0.89)	0.85±0.05	***0.037****
Liquid shaded	**A2**	(0.67-0.91)	0.79±0.1	(0.67-0.91)	0.82±0.1	*0.374*
	A4	(0.29-0.65)	0.43±0.14	(0.38-0.66)	0.5±0.1	*0.172*

Paired Samples T test ****: Significant level at P value < 0.05***

TABLE (7) Comparison of surface roughness between different shading techniques of different colors before and after aging

Aging	Shade	Surface roughness				P value
		Pre shaded N=5		Liquid shaded N=5		
		Range	*Mean ± SD*	*Range*	*Mean ± SD*	
Before aging	**A2**	(0.28-0.48)	0.37±0.09	(0.67-0.91)	0.79±0.1	***<0.001****
	A4	(0.33-0.85)	0.53±0.24	(0.29-0.65)	0.43±0.14	*0.443*
After aging	**A2**	(0.79-1.19)	1.03±0.17	(0.67-0.91)	0.82±0.1	***0.043****
	A4	(0.79-0.89)	0.85±0.05	(0.38-0.66)	0.5±0.1	***<0001****

Independent Samples T-test ****: Significant level at P value < 0.05***

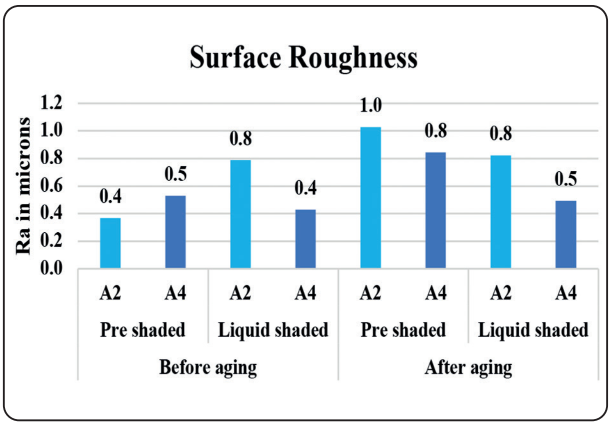

Fig. (3)

DISCUSSION

Monolithic zirconia enabled the production of unveneered esthetically successful zirconia restorations that require minimally invasive preparations & exhibit high flexural strength combined with minimal occlusal wear of opposing natural dentition. Different CAD/CAM systems are used for milling different restorations in a very short time. The absence of veneering porcelain eliminates the risk of unwanted chipping [15,16].

As the strength of translucent zirconia differs according to the brand [17], blanks & coloring solutions used in this study were produced by the same manufacturer. Discs dimensions were selected to ensure ceramic has adequate mechanical properties, good aging resistance as well as adequate esthetic properties. The 12mm diameter was selected corresponding to the size of the sample compartment of the spectrophotometer for easy & accurate readings.

Tooth-colored resin & ceramic restorations were greatly affected by the CAD/CAM technology allowing accuracy & rapid production. These systems use industrial blocks prefabricated under optimum & controlled milling conditions, thus delivering a high-quality restoration with improved mechanical and optical properties[19,20].

Iso Met 4000 micro saw was used to cut the ceramic blocks to the desired shape due to its reliability in cutting with minimum sample deformation and lower kerf loss that can ensure samples' standardized thickness to overcome any optical alterations that might occur due to changes in thickness [21].

The two approved approaches for coloring monolithic zirconia restorations are either the incorporation of metal oxides into the zirconia powders prior to the industrial pressing of blocks or surface infiltration of pre-sintered zirconia restorations using solutions containing metal salts [22]. Two techniques are available for coloring un-shaded zirconia, the first can impart color to the sample by immersion which allows coloring liquid to penetrate all parts of the sample while the second can impart color to the outer surface of the sample by using a brush. The immersion technique is preferred in coloring zirconia ceramic *[23]*. In this study, immersion of samples in an acid-based coloring liquid for 20 seconds was done due to its ability to penetrate deep within the restoration & as prolonged dipping time may reduce the fracture strength & deteriorate the color *[24]*. A sintering program with the preheating function was selected to ensure complete dryness of the pre-shaded samples & to avoid homogenization of the liquid-colored samples. For the aging process, to control the heating of the acetic acid & to ensure constant temperature; a reflux system was used to keep the specimens. This system operates under a dynamic balance between evaporation & condensation rates of the acetic acid so that its amount remains constant throughout the whole process [25].

The worth value of materials' translucency can be attributed to their role in achieving a natural-looking appearance *[26]*. Assessment of the materials' translucency can be achieved by measuring & calculating the translucency parameter (TP), contrast ratio (CR), and transmittance. According to ISO 6872, the aging of samples was performed by immersion in a weak organic acetic acid *[27]*, which is corrosive to ceramics due to its chelating effect**.** Samples immersion in 4% acetic acid at a temperature of 80°C for 16 hours corresponds to 2 - 3 years of clinical use.

Surface roughness tests usually present clinically reliable data about the expected mechanical performance of ceramic restorations, due to their influence on the creation & propagation of surface micro-cracks which reduce restorations' fracture resistance. It also acts a major role in the adhesion of bacteria, resulting in caries development & various periodontal inflammation. Additionally, rough surfaces might augment restorative materials' liability to be stained. Longstanding restoration gloss, smoothness & stability are important from a hygienic & esthetic point of view. Surface roughness as well as a fixed restoration surface treatment affects its color stability **[29,30]**.

A contact Profilometer was used for measuring surface roughness to get highly accurate readings compared to the non-contact devices which might lead to false readings when used with a shiny surface like ceramics due to the scattering effect of the reflected light. [31]

In the current investigation, translucency was not significantly affected by the shading technique for both shades before & after aging. These findings might be referred to that coloring agents are supplied in the form of oxides obtained by dissolving in hydrochloric acid, where the color can be damaged by being subjected to high temperatures during the sintering procedure 1600°C or more at which the color saturation & translucency might be significantly affected *[32]*. In the current study, the sintering was done according to the manufacturer's instructions at a temperature of 1450 °C so the coloring agents were not noticeably affected at this temperature. These results agree with several investigators, including[)], ***Yilmaz & Uluso (2014)*** *[34]* & ***Kim (2014)*** *[35]* **Sulaiman et al** ***(2015)*** *[33]*, where all concluded that coloring liquid had no significant effect on translucency.

Results showed that the shade itself A2 & the A4 significantly affected translucency. Shade A2 had a significantly higher translucency compared to A4 whether pre-shaded or liquid-shaded, before & after aging. This might be attributed to the fact that the yellow colorants in the dark coloring solution result in a noticeable decrease in the ceramic translucency associated with the color change *[36,]*. Also, it was reported that the optical properties of Y-TZP ceramic are influenced by [the] material's brand, composition & thickness *[33]*, type & quantity of additives, shade, coloring protocol (pre-shaded or colored by immersion in coloring liquids), & surface roughness. *[36,37]*

These results were in agreement with several investigators, Al ***Harbi et al (2012)*** [38]***, Awad et al (2015)*** [39]***, and Ataya et al (2009).***[40] who detected a significant difference in translucency between different shades. Also, this is in agreement with ***Abbasimoghaddam et al (2021)*** [41] found that aging had no statistically significant effect on translucency of the same shade but a significant difference was recorded between different zirconia shades.

Although there was a decrease in translucency due to aging regarding both shades, this decrease was statistically non-significant. This non-significant decrease might be related to the aging holding time as it was reported that the translucency of Y-TZP was not significantly affected when the duration of aging was lower than 20 hours [42]. Also, ***Kou et al (2019)*** [43] concluded that aging had no significant effect on the translucency of zirconia ceramic. However, these results contradict what ***Alamledin et al (2020)*** [44], reported in their research that aging improved the translucency of monolithic zirconia. However, differences in the aging protocol or differences in the material brand might cause such controversy.

Results of surface roughness (Ra) revealed that zirconia discs after aging in an acidic medium showed a higher statistically significant mean (Ra) value than those before immersion in the acid. This agrees with many investigations, which proved that surface roughness significantly increased when glazed zirconia was submerged in a corrosive medium. They observed slow & spontaneous transformation from tetragonal to monoclinic phase on zirconia surface in contact with body fluids at 37°C, water or water vapour; this process is called low-temperature degradation (LTD). This introduced about a 4% volume increase of the particles beneath the surface leading to stress formation around the monoclinic particles & consequently particles separation from the surface. This separation results in the increase of the roughness with the micro-cracks on the surface [45,46]. Monolithic zirconia does not require a veneering ceramic layer, so restorations are directly exposed to the intra-oral environment, which triggers LTD in zirconia. In monolithic zirconia, alumina which is responsible for the resistance to LTD is reduced to enhance translucency. Thus, monolithic zirconia might be more sensitive to LTD compared to conventional core-type zirconia. [47]

From the previous discussion, the hypothesis of this study was partially accepted.

The limitations of this study include that:

- The samples were only subjected to chemical aging, discarding the effect of thermomechanical aging, which simulates the oral environment.
- Only two shades were examined.
- The inability to correlate translucency & roughness results due to using different samples for each test as roughness was measured by contact profilometry.

Further investigations are needed to correlate the translucency & surface roughness of zirconia with different shades & shaded with both shading techniques & subjected to thermomechanical as well as chemical aging to simulate the clinical situation.

CONCLUSIONS

1. Translucency significantly differs according to the selected shade. Shading technique & aging do not significantly affect the translucency of monolithic zirconia.
2. Aging significantly increases the surface roughness of monolithic zirconia, while the shading technique does not have a significant effect.

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