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Original Article

Effect of Aqueous and Acid-based Coloring Solutions on Microhardness and Translucency of Monolithic Zirconia

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Abstract

Introduction: Zirconia has encountered a rapid development of metal-free dentistry because of its biological, mechanical, and esthetic properties. The white opaque color of zirconia limits the possibility of natural-looking restoration. Pre-colored blocks and green stage coloring liquids are used to obtain shaded zirconia. Coloring may affect mechanical and optical properties.

Purpose: The purpose of the study was to evaluate the effect of aqueous and acid-based coloring solutions on microhardness and translucency of monolithic zirconia.

Materials and Methods: A total of 30 specimens (15 × 12 × 0.5 mm) were divided into three groups. Non-colored zirconia specimens, Group A, were used as a control group. Acid-based coloring liquid treated zirconia, Group B, and aqueous coloring liquid treated zirconia, Group C, as experimental groups. Coloring liquids were applied to the specimens 1 time using a brushing technique. A micro Vickers hardness tester was used to evaluate the surface microhardness. Translucency values were measured by UV visible spectrophotometer. The results were analyzed using a one-way analysis of variance (ANOVA) and 2-way ANOVAs, followed by post hoc comparison by Tukey's method.

Results: The mean values of surface microhardness (in HV) of all three groups, Group A, B, and C, were 1077.34, 1192.51, and 1222.64, respectively. The mean values of translucency (in %) of all three groups, Group A, B, and C are 40.06, 70.41, and 48.71, respectively. There was a statistically significant improvement in surface microhardness and translucency of monolithic zirconia after coloring with acid based and aqueous coloring solutions.

Conclusion: The surface microhardness and translucency of both monolithic zirconia treated with acid based and aqueous coloring solution was significantly increased as compared with non-colored monolithic zirconia. Monolithic zirconia treated with acid-based coloring solution has the highest translucency. Aqueous colored monolithic zirconia has the highest surface microhardness.

Key words: Monolithic zirconia, Prettau A3 coloring liquid, Prettau Aquarell A3, Surface microhardness, Translucency

INTRODUCTION

A good face is a letter of recommendation. Media projected perfect appearance has a strong impact on the behavior and thinking of our beauty-conscious society. Moreover, it has led to an increased demand for esthetic treatment from public.[1] Good dental appearances are thought to be a requirement of prestigious occupations among some professional groups.[2]

Metal-ceramic restoration has been used successfully in dentistry. Although it provides favorable results in terms of strength, form, and function, it shows unesthetic appearance from metal underneath, especially at the cervical part of the restoration or from the opaque porcelain layer.[3] Ceramic materials, also named bioceramics, are especially

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developed for dental use to solve the esthetic problems related to metal-ceramic fixed dental prosthesis.[3,4]

The modern era, which is encountering an ever-increasing assortment of ceramic material, could be characterized as the "ceramic age."[5] Over the last decade, zirconia has encountered a rapid development of metal-free dentistry because of its biological, mechanical, and esthetic properties. The mechanical properties of zirconia are the highest ever reported for any dental ceramics.[6] The high strength and fracture toughness of zirconia result from transformation toughening.[7,8] Due to its white opaque color, it has to be veneered with feldspathic porcelain, but cohesive failure of feldspathic porcelain has been identified as the main complication.[8] Clinical studies have reported that the use of weak veneering porcelain, differences in the coefficient of thermal expansion, low core veneer bond strength, and undesirable zirconia framework design are the main reason for chipping or fracture on veneering porcelain.[9-12] One approach to overcome this problem is to fabricate complete contour zirconia without any veneering. Since zirconia is opaque, coloring must be done for zirconia.

Two main coloring techniques are additive and painting technique.[13] In additive techniques, metal oxides are mixed with zirconia powder at a stage of zirconia block manufacturing and the colored block is sintered.[13] In the painting technique, color is superficially applied with a brush before being dried and sintered.[13] Two commonly used zirconia coloring liquids are acid based and aqueous coloring solutions. Acid-based coloring solution imparts color to zirconia through acid-base reaction,[14] whereas aqueous coloring solution imparts color by filtering metal cations into a porous zirconia framework.

In this context, the present study was conducted to evaluate the effect of microhardness and translucency of monolithic zirconia after the application of acid based and aqueous coloring solution as compared with non-colored monolithic zirconia.

MATERIALS AND METHODS

Fabrication of Test Specimens

Monolithic zirconia blocks (Alpha Z 0; DMAX) were used in this study. Green stage zirconia blocks were milled into 15 × 12 × 0.5 mm using a milling machine [Figure 1]. All the specimens were polished with 1000 grit silicon carbide paper to produce a uniform surface profile [Figure 2]. A total of 30 specimens were divided into three groups. Non-colored zirconia specimens (Group A) were used as a control group. Acid-based coloring liquid (Group B) treated zirconia and aqueous coloring liquid treated (Group C) zirconia as experimental groups. Prettau A3 coloring liquid was used for acid based and Prettau Aquarell A3 for the aqueous coloring techniques [Figure 3]. Coloring liquids were applied to the specimens 1 time using the brushing technique [Figure 4]. The specimens were then dried for 30 min in an oven at 130°C and sintered in a sintering furnace according to manufactures instructions [Figures 5-7].

Figure 1: Zirconia milling machine

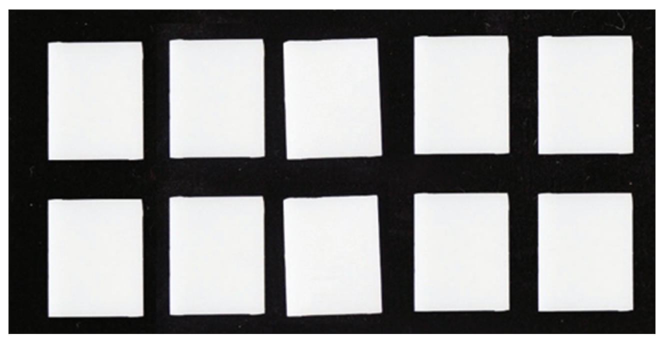


Figure 2: Finished zirconia blocks

Figure 3: Prettau A3 and Prettau Aquarell

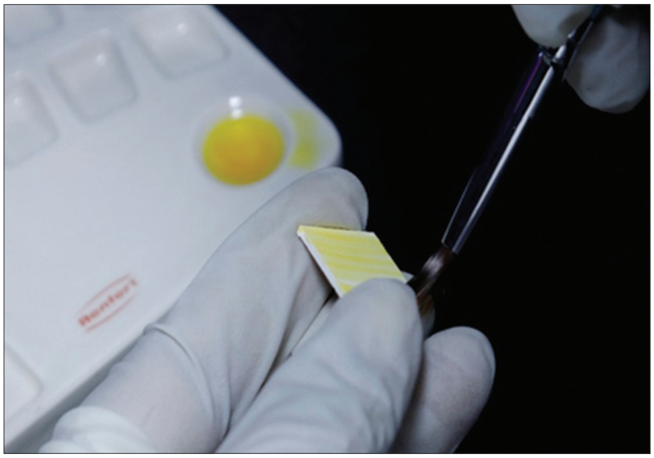


Figure 4: Coloring of zirconia using brushing technique

Figure 5: Sintering unit

Figure 6: Sintering

Testing of Specimens for Surface Hardness

A micro Vickers hardness tester (HMV SCHIMADZO, model HMV 2T ADW) was used to evaluate the surface hardness of specimens for each treatment [Figures 8 and 9]. Surface hardness was measured using the indentation

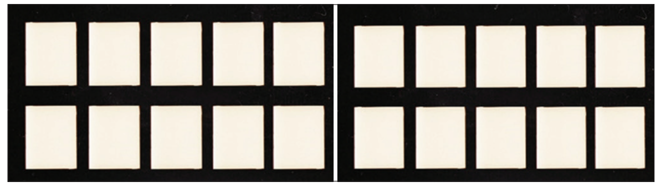


Figure 7: Monolithic zirconia colored with acid-based coloring solution (A3 Shade) and aqueous coloring solution (A3 Shade)

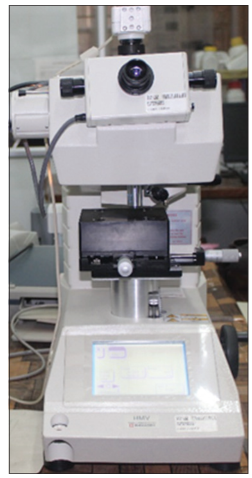


Figure 8: Micro Vickers hardness tester (HMV SCHIMADZO, model-HMV 2T ADW)

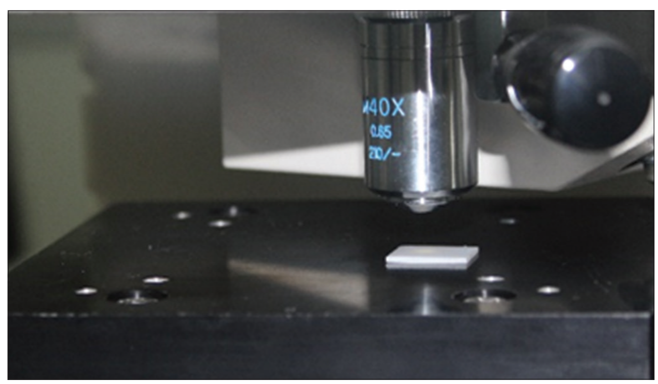


Figure 9: Specimen *in situ*

technique to determine the Vickers hardness number of specimens. Specimens were mounted in the horizontal stage of the tester, then the indenter was lowered under a load of 9.8 N for 15 s. Three indentations will be placed in the center of each specimen. For each tested sample, all indentations were measured and an average Vickers microhardness number HV was determined.

Testing of Specimens for Translucency

Light transmittance was measured using UV visible spectrophotometer (Varian, model CARY100BIO) [Figures 10 and 11].

Since translucency is the characteristic of a substance that is partially able to allow light to pass through it, light transmittance was considered as a translucency parameter. UV/VIS spectroscopy is based on the absorption of light by a sample. Depending on the amount of light and its wavelength absorbed by the sample, valuable information can be obtained.

The spectrophotometer consisted of two instruments: A spectrometer with a double prism monochromator to produce the light of any selected wavelength, a photometer with a silicon photodetector to measure the intensity of light and a sample holder. The instruments were arranged so that specimens could be placed between the spectrometer beam and the photometer at the entrance port of the integrating sphere in order that the total amount of light transmitted and scattered through it could be measured.

Fabricated specimens were placed in a spectrophotometer with the help of a holder and working wavelength ranged from 400 to 800 nm of the visible light spectrum was arranged. The readings were obtained on the computer connected to spectrophotometer. The amount of light transmittance was recorded in percentage and a UV/VIS spectrum is graphically represented as absorbance as a function of wavelength.

RESULTS

Data were analyzed using computer software, Statistical Package for the Social Sciences version 16.0. Data were expressed in its mean and standard deviation. Analysis of variance (one-way ANOVA) was performed as a parametric test to compare different groups. To facilitate multiple comparisons between groups, Tukey's method was employed as a post hoc test along with ANOVA. For all statistical evaluation, a two-tailed probability of value <0.05 was considered significant.

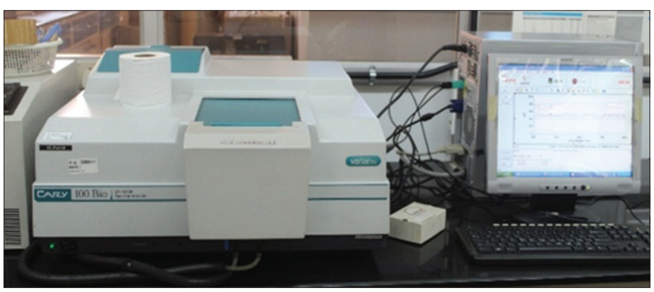


Figure 10: UV visible spectrophotometer (Varian, model CARY 100 BIO)

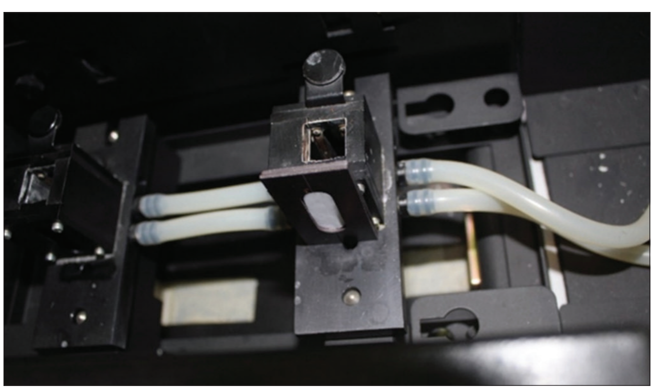
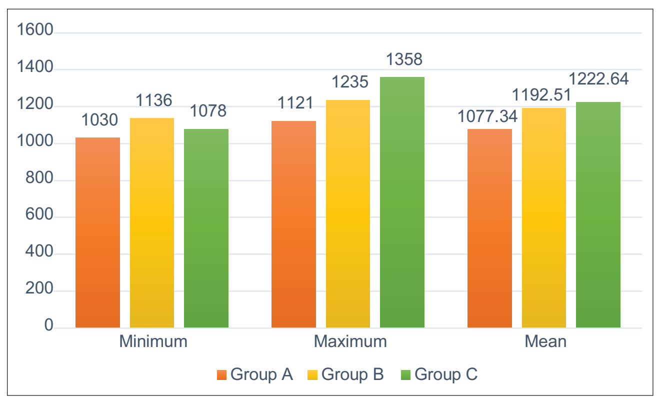


Figure 11: Specimen *in situ*

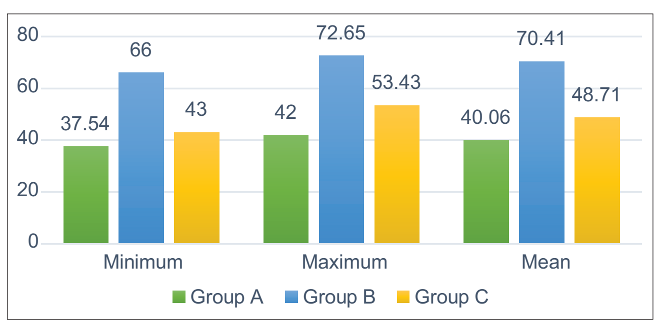
Graphical representation of the mean values of surface hardness and translucency, comparison of surface microhardness and translucency among groups is shown in Graphs 1-4, respectively.

Mean and standard deviation values of the surface microhardness are presented in Table 1. As per this study, maximum mean surface microhardness was obtained for monolithic zirconia colored with aqueous-based coloring liquid (Group C) and non-colored monolithic zirconia is minimum (Group A) [Table 1]. One-way ANOVA showed a statistically significant difference between mean values [Table 2]. Multiple comparisons with post hoc by Tukey's method revealed that comparison between groups (A and B) and (A and C) were statistically significant as $P < 0.001$. Since P-value is 0.529 (>0.005), comparison between Groups B and C was not statistically significant [Table 3].

Mean and standard deviation values of the translucency are presented in Table 4. As per this study, maximum mean

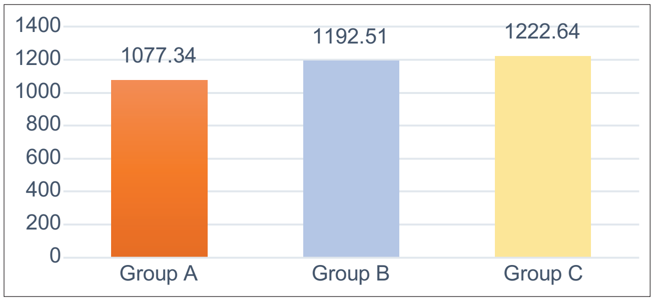


Graph 1: Surface microhardness in HV unit

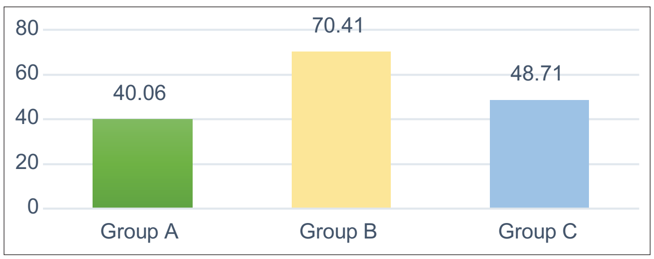


Graph 2: Transmittance in percentage

translucency was obtained for monolithic zirconia colored with acid-based coloring liquid (Group B) and non-colored monolithic zirconia is minimum (Group A) [Table 4]. One-way ANOVA showed a statistically significant difference between mean values [Table 5]. Multiple comparisons with post hoc by Tukey's method revealed that comparison between Groups (A and B), (A and C), and (B-C) were statistically significant as $P < 0.001$ [Table 6].



Graph 3: Comparison of surface microhardness in HV unit



Graph 4: Comparison of transmittance in percentage

Table 1: Surface microhardness in HV unit

Variable	Groups	*n*	Minimum	Maximum	Mean	Std. Deviation
Surface micro hardness in HV unit	Group A	10	1030.00	1121.00	1077.34	34.19
	Group B	10	1136.00	1235.00	1192.51	35.95
	Group C	10	1078.00	1358.00	1222.64	94.93

Table 2: Comparison of surface microhardness in HV unit among the three groups using one way ANOVA

Groups	*n*	Mean	Std. Deviation	Std. Error mean	*P*-value
Group A	10	1077.34	34.19	10.81	0.001* (significant)
Group B	10	1192.51	35.95	11.37	
Group C	10	1222.64	94.93	30.02	

*Significant at the 0.05 level using one way ANOVA

Table 3: Pairwise comparison of surface microhardness in HV unit among the three groups using Tukey post hoc test

Groups	Groups	Mean Difference (I-J)	Sig.
Group A	Group B	–115.17200*	0.001* (significant)
	Group C	–145.30200*	0.001* (significant)
Group B	Group C	–30.13000	0.529 (not significant)

*Significant at the 0.05 level using Tukey post hoc test

DISCUSSION

The use of an esthetic material for the restoration of missing tooth structure has been a long-standing goal in dentistry. Patients are expecting to have several options for esthetically pleasing, functional, and durable restorations in all areas of the dentition. To remedy this, new materials that combine strength, esthetics, and biocompatibility have been developed and brought to market.[15]

Metal ceramic restorations are a type of ceramic system for fixed prosthetic rehabilitation that has been widely used since the early 1960s.[16] Light-reflecting from the opaque porcelain used to mask the metal, particularly at the cervical third of the restoration, causes a light gray appearance of the adjacent gingival tissue. This phenomenon led to the development of metal-free ceramics. Some of the mechanical properties of these materials, such as brittleness, crack propagation, fracture toughness, low tensile strength, wear resistance, marginal accuracy, and difficulty of repair, have limited their clinical use.[7]

Zirconia was introduced in dentistry in the early 1990s and has been used as a core material to support more esthetic ceramic materials. Zirconia shows similar mechanical properties to stainless steel[10] and the highest ones among

Table 4: Transmittance in percentage

Variable	Groups	*n*	Minimum	Maximum	Mean	Std. Deviation
Trans mittance in percentage (%)	Group A	10	37.54	42.00	40.06	1.64
	Group B	10	66.00	72.65	70.41	2.32
	Group C	10	43.00	53.43	48.71	2.88

Table 5: Comparison of transmittance in percentage among the three groups using one way ANOVA

Groups	*n*	Mean	Std. Deviation	Std. Error mean	*P*-value
Group A	10	40.06	1.64	0.52	0.001*
Group B	10	70.41	2.32	0.73	
Group C	10	48.71	2.88	0.91	

*Significant at the 0.05 level using one way ANOVA

Table 6: Pairwise comparison of transmittance in percentage among the three groups

Groups	Groups	Mean difference (I-J)	Sig.
Group A	Group B	–30.35100*	0.001* (significant)
	Group C	–8.65200*	0.001* (significant)
Group B	Group C	21.69900*	0.001* (significant)

*Significant at the 0.05 level using Tukey post hoc test ceramics used in dentistry. As the strongest and toughest of all dental ceramics,[17] zirconia has 900–1200 MPa flexural strength.[18] Since of their lack of translucency, zirconia cores are generally veneered with porcelain. Differences in the coefficient of thermal expansion between the zirconia substructure and the veneering ceramic improper framework design, rapid cooling rates, and low fracture toughness and flexural strength of veneering ceramic compared to the zirconia core have been considered as the cause of this cohesive failure.[19] Recently, there has emerged a trend of fabrication of full-contour zirconia restoration to avoid veneering failure. To increase the translucency and esthetics of full-contour zirconia, coloring liquids have been applied. Two main coloring techniques are additive and painting technique.[13] In additive techniques, metal oxides are mixed with zirconia powder at a stage of zirconia block manufacturing and the colored block is sintered.[13] In the painting technique, color is superficially applied with a brush before being dried and sintered.[13] Two commonly used zirconia coloring liquids are acid based and aqueous coloring solutions. Acid-based coloring solution imparts color to zirconia through acid-base reaction,[14] whereas aqueous coloring solution imparts color by filtering metal cations into porous zirconia framework. The coloring procedure may affect the mechanical and optical properties of zirconia.[20]

The present *in vitro* study investigated the effect of aqueous and acid-based coloring solutions on translucency and microhardness of monolithic zirconia. The first property evaluated in this study was surface microhardness. A micro Vickers hardness tester (HMV SCHIMADZO, model HMV 2T ADW) was used to evaluate the surface hardness of specimens for each treatment. There was a statistically significant increase in the surface microhardness of monolithic zirconia colored with aqueous-based coloring liquid and monolithic zirconia colored with acid-based coloring liquid as compared with non-colored monolithic zirconia was obtained. Among these, monolithic zirconia colored with aqueous-based coloring solution showed the highest value of surface microhardness.

Surface hardness can be used as an index to predict the resistance of materials to surface crack formation. Surface cracks can be due to reduced fatigue strength and produce early fractures of restorations.[12] This difference in surface microhardness is due to the chemical composition of coloring liquids and the mechanism of imparting color to zirconia. The addition of metal oxides for zirconia coloring may cause crystallographic and microstructural modifications which in turn could affect the mechanical properties of zirconia. Nature of grain form and concentrations of the coloring liquids can have a considerable effect on the mechanical properties of zirconia materials.

The coloring solution contains a solvent, coloring agents, and additives. The coloring agents contain rare earth metals and transition metals. The coloring agents are a combination of two or more rare earth metal compounds such as praseodymium (Pr) ions, erbium (Er) ions, cerium (Ce) ions, and neodymium (Nd) ions. Solvent can dissolve the coloring agents (water and alcohol). The additives preferably are organic additives that do not leave any harmful residue after sintering. Additives are polyethylene alcohol and polyethylene glycol-600.

Acid-based coloring liquids typically contain 0.1 wt% HCl (pH 1 to 3), a strong acid which imparts colors to zirconia through an acid-based reaction, whereas aqueous coloring solution imparts color by filtering metal cations into porous zirconia framework. Certain metal salts can only be liquefied in acid, and the acidification of coloring liquid helps them to infiltrate deeper into the zirconia framework. Scanning electron micrograph analysis revealed that the entire surface of the acid-based coloring liquid specimens was coated with coloring liquid. Hence, the metal ions can enter into the crystal lattice of the zirconia. Coloring liquid was observed mainly at the grain boundary for the aqueous coloring liquid. This difference in the distribution of metal ions in the crystal lattice of colored zirconia is the reason for increased surface microhardness of aqueous colored zirconia than acid colored zirconia.

Various authors have a different opinion regarding microhardness of colored zirconia.

In a study conducted by Nam and Park,[12] a statistically significant difference in hardness was found between acid-colored zirconia and aqueous colored zirconia. They also found that increasing the number of applications decreased the hardness value of acid colored zirconia but had no effect on aqueous colored liquid zirconia. This can be attributed to the change in the distribution of metal ions of coloring solution in the crystal lattice of zirconia. According to Hjerppe *et al*.,[21] biaxial strength and surface microhardness of zirconia framework were reduced after shading. The reduction was due to a difference in shading time and composition of coloring solution. Investigation done by Ban *et al*.[22] reported that various ions in the coloring liquid may act as impurities in the sintered zirconia block and can affect its properties. Their studies concluded that coloring liquids containing Er and/or Nd should be avoided for dental prostheses. Furthermore, coloring with Fe and Co showed no remarkable changes in mechanical property, indicating little reaction with zirconia.

The second property assessed in this study was translucency. Translucency was measured using a UV visible spectrophotometer (Varian, model CARY100BIO). Optical properties such as light transmittance and reflectance have important roles in the esthetics of restorations, and the translucency of the material can ensure the natural appearance of the restorations. There was a statistically significant increase in the translucency of monolithic zirconia colored with acid-based coloring liquid and monolithic zirconia colored with aqueous-based coloring liquid as compared with non-colored monolithic zirconia was obtained. Among these, monolithic zirconia colored with acid-based coloring solution showed the highest value of translucency. This difference in translucency is due to the chemical composition of coloring liquids and the distribution of metal ions in the crystal lattice of zirconia. Since the entire surface of the acid-based coloring liquid specimens was coated with coloring liquid, the metal ions can enter into the crystal lattice of the zirconia. Coloring liquid was observed mainly at the grain boundary for the aqueous coloring liquid specimens. Hence, the metal ions maintained at the crystal boundary may scatter the incident light. This difference in the distribution of metal ions in the crystal lattice of colored zirconia is the reason for increased translucency of acid colored zirconia than aqueous colored zirconia.

An *in vitro* study conducted by Nam and Park[12] found that aqueous coloring liquid on zirconia produced a greater redness or yellowness compared to acid-coloring liquid. They also concluded that the coloring of zirconia lowered its brightness and imparted a red/yellow hue to zirconia. They attributed that this can be due to a change in the composition of coloring liquids. However, many of the researchers found that coloring procedures can decrease the translucency of zirconia. According to Sulaiman *et al.*[14] staining decreased the transmittance of zirconia due to change in chemical nature of the coloring liquid. This lead to more absorption of light energy and thus decreasing the amount of light transmission.

The result of the present study states that monolithic zirconia colored with acid based and aqueous coloring solution increased translucency and surface microhardness. Monolithic zirconia colored with aqueous coloring solution has the highest microhardness and monolithic zirconia colored with acid-based coloring solution has the highest translucency.

According to Nam and Park,[12] aqueous coloring liquids have long-term stability without the need for pigment-stabilizing agents, as high pH values are required for hydrolysis of rare earth metal ions. Compared with acid-based zirconia coloring liquids, aqueous coloring liquids are safer and more convenient for dental technicians because less acidic fumes are emitted during the application and sintering processes of aqueous coloring solution. Furthermore, they have more enhanced drying time. Therefore, aqueous coloring liquids could be a good substitute for acid-based coloring liquids.

The results of the study were limited to 1-time application of aqueous colorant (A3 shade) and an acid-based colorant (A3 shade). Therefore, it is necessary to study the effect of various coloring agents and multiple applications on the coloring of zirconia. Furthermore, the study evaluated only the changes in two properties that are translucency and surface microhardness. The change in other esthetic and physical properties of the material should be evaluated. The effect of coloring on various components of the zirconia block among the manufacturers also be evaluated. Moreover, as per the present study, no correlation exists between microhardness and translucency of colored specimens. Hence, further investigations are required.

CONCLUSION

The present study was conducted to evaluate the effect aqueous and acid-based coloring solutions on microhardness and translucency of monolithic zirconia.

Within the limitations of the study, the following conclusions were drawn

1. The surface microhardness and translucency of both monolithic zirconia treated with acid-based coloring solution and aqueous coloring solution were significantly increased as compared with non-colored monolithic zirconia
2. Monolithic zirconia treated with aqueous coloring solution has the highest surface microhardness
3. Monolithic zirconia treated with acid-based coloring solution has the highest translucency.

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