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Research Article / Araştırma Makalesi

The Effect of Different Surface Treatments and Accelerated Artificial Aging on the Color Stability of Lithium Disilicate Materials

Farklı Yüzey İşlemleri ve Hızlandırılmış Yapay Yaşlandırmanın Lityum Disilikatların Renk Kararlılığı Üzerine Etkisi

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ABSTRACT

Background: The aim of this study is to investigate the clinical acceptability of the color change that may occur in time about full ceramic materials used for aesthetic restorations, which are bonded after different surface conditioning processes, by measuring with spectrophotometer.

Methods: The lithium disilicate CAD material we used in our work was prepared with isomet in the form of 1-millimeter-thick specimens. Three random groups of 21 specimens were selected from the specimens. One of these groups was conditioned with phosphoric acid, which is recommended by the manufacturer. Er: YAG laser was applied to the second group. The third group was conditioned by Nd: YAG laser. Surface changes in the groups were evaluated by scanning electron microscope (SEM) and atomic force microscope (AFM). The specimens were bonded with the adhesive resin cement to the background material for color change measurement. The first color measurement of the bonded specimens was done by spectrophotometer. The specimens were aged at 10.000 cycles in the thermal cycle device. The second color measurement after aging was done with the same spectrophotometer.

Results: After aging with thermal cycle, there was a clinically acceptable color change in all groups. The Nd: YAG laser group presented better color stability ($p<0.05$). The SEM and AFM images presented different surface morphology on laser groups.

Conclusion: It has been determined that the color change in the lithium disilicate material bonded after different surface conditioning processes is clinically acceptable. The specimens conditioned with Nd: YAG have the best color stability.

Keywords: Lithium disilicate, Atomic force microscopy, Lasers, Aging, Color

ÖZ

Amaç: Bu çalışmanın amacı, farklı yüzey işlemlerinden sonra yapıştırılan lityum disilikat restorasyonlarda zamanla oluşabilecek renk değişiminin spektrofotometre ile ölçülerek klinik olarak kabul edilebilirliğini araştırmaktır.

Gereç ve Yöntemler: Çalışmamızda kullandığımız lityum disilikat materyali 1 milimetre kalınlığında numuneler şeklinde hazırlandı. Hazırlanan örneklerden, her biri rastgele seçilmiş 21 örnek içeren üç grup oluşturuldu. Birinci grup, üretici tarafından tavsiye edilen fosforik asit ile işlem gördü. İkinci gruptaki örnekler Er: YAG lazere tabi tutuldu. Üçüncü gruptaki örneklere ise Nd: YAG lazer uygulandı. Gruplardaki yüzey değişiklikleri taramalı elektron mikroskobu (SEM) ve atomik kuvvet mikroskobu (AFM) ile değerlendirildi. Numuneler, renk değişimi ölçümü için adeziv rezin siman ile arka plan malzemesine yapıştırıldı. Yapıştırılan numunelerin ilk renk ölçümü spektrofotometre ile yapıldı. Numuneler, termal döngü cihazında 10.000 döngü ile yaşlandırıldı. Yaşlandırma sonrası ikinci renk ölçümü aynı spektrofotometre ile yapıldı.

Bulgular: Termal döngü ile yaşlandırma sonrasında tüm gruplarda klinik olarak kabul edilebilir bir renk değişimi olmuştur. Nd: YAG lazer ile işlem gören grup daha iyi renk kararlılığı göstermiştir ($p<0.05$). SEM ve AFM görüntüleri incelendiğinde fosforik asit uygulanan grubun lazer gruplarına göre farklı yüzey morfolojisine sahip olduğu gözlendi.

Sonuç: Farklı yüzey işlemlerinden sonra yapıştırılan lityum disilikat örneklerde renk değişiminin klinik olarak kabul edilebilir olduğu belirlenmiştir. Nd: YAG lazer ile işlem görmüş örnekler en iyi renk stabilitesine sahiptir.

Anahtar Kelimeler: Lityum disilikat, Atomik kuvvet mikroskopisi, Lazerler, Yaşlanma, Renk

1. INTRODUCTION

Nowadays, Computer Aided Design-Computer Aided Manufacturing (CAD-CAM) ceramic materials have been preferred due to their easy manufacturability, biocompatibility, and also aesthetic properties for restoring lost tooth tissues ([1]). The lithium disilicate CAD-CAM material (LD) is one of the CAD-CAM ceramic material types that contains homogeneously distributed polyvalent and colorant ions for aesthetic purposes. In this way, LD can have the appearance of natural teeth by imitating the optical properties, like translucency of the enamel, which is called the chameleon effect. Although translucency is an advantage of LD in terms of color ([2]), this property may be a disadvantage for the long-term success of restorations in terms of visibility of the polymerized resin cement color from the surface ([3-6]).

Resin cements are commonly used in cementing the LD due to their low solubility, high bonding strength, and different color options. Dual-cure resin cement is one of the resin cement varieties that has both two properties at once: it is auto-polymerized on lightless zones and polymerized on transparency zones using light ([7]). However, in the long run, the color of dual-cure resin cement may change as it is intrinsically due to oxidation of the amine which is responsible for auto-polymerization, thereby causing a non-aesthetic appearance in thinner LDs ([3]). In order to overcome this problem, amine-free, dual-cure, resin cement has been developed. But still, it is reported that these types of developed cement may result in discoloration by absorbing water due to their chemical contents, such as bisphenol A-glycidyl methacrylate (Bis-GMA) ([8, 9]).

Another way to prevent aesthetic problems in LDs caused by resin cement discoloration is by changing the optical properties by altering the surface texture of LD during bonding procedures ([5, 6]). Altering the surface texture of LDs is one of the many phases in bonding LDs and teeth, which is primarily used for increasing micromechanical retention of the cement by creating a rougher surface ([10]). Various methods are available for this, including applying aluminum oxide particles, acid etching, and laser treatment([3-6, 10]). Aluminum oxide particles have been proven effective in ceramic surface roughening, but extreme applications may result in microcrack formation on ceramic surfaces ([11]). Although acids are considered the most effective method for pretreatment of ceramic surfaces ([12]), they may also be caustic and hazardous for users and the environment ([13]). Recently,

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using lasers as a surface pretreatment method has gained popularity. For this purpose, Nd:YAG lasers ([14-17]), CO^2 lasers ([18]), Er:YAG lasers ([5, 6, 19]), and Er;Cr:YSGG ([4]) have been researched widely. Lasers with certain parameters may affect surface roughness positively ([4, 14, 15]). However, there is very limited information concerning the alteration in time of optical properties of cemented ceramics ([20]) after different surface pretreatment processes ([5, 6]). Safe and useful ceramic surface roughening methods can contribute to preventing aesthetic problems caused by cement discoloration.

The aim of this study is to investigate the clinical acceptability of the color change that may occur in time from LDs which were bonded after different surface pretreatment processes. The null hypothesis tested that there would be no difference with time in the color properties of LDs subjected to different surface treatments and bonded by dual-cure resin cement.

2. MATERIAL AND METHODS

2.1. Specimen Preparation

A total of 63 rectangular-shaped specimens (1 mm thickness) in shade A2-high translucency (HT) were prepared from the lithium disilicate (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) blocks by using a cutting device under constant irrigation (MKC-100, Mod Dental, Ankara, Turkey) ([21]). All specimen surfaces were polished with wet #400 grit silicon carbide paper under standardized conditions on a grinding device (Minitech 233, Grenoble, France) and cleaned in distilled water for 5 minutes using an ultrasonic cleaner (Skymen JP-060s, China) ([14]). They were then dried with oil-free air and one side was glazed with a layer of neutral-shade glaze in a ceramic furnace (Ivoclar Vivadent Programat, Liechtenstein) to mimic the in-vivo condition ([6]). Three random groups were obtained according to the surface treatments applied to the non-glazed side (n = 21).

- The specimens in phosphoric acid group were subjected to 35% phosphoric acid (K-Etchant Syringe; Kuraray Panavia V5, Sakazu, Japan) in 5 seconds according to the dual-cure resin cement manufacturer's instructions for surface conditioning, rinsing for 5 minutes, and then drying with air (22).
- The specimens in ER: YAG group were treated with Er: YAG laser irradiation (Fotona, Fidelis, Slovenia) with a power setting of 400 mJ and a 20 Hz repetition rate (23). Laser energy was delivered to the ceramic surfaces at a distance of 1 mm with a 20 s in a sweeping motion over the entire surface. The same experienced researcher achieved all interventions in order to avoid scanning differences.
- The specimens in Nd: YAG group were prepared with Nd: YAG (Fotona, Fidelis, Slovenia) laser with the following laser specifications: energy parameter = 100 mJ and repetition rate = 20 Hz ([14]). The other the procedures were similar to those in group 2.

2.2. Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) Analysis

The morphological changes of the ceramic surface after treatments were examined using a scanning SEM (QUANTA FEG-250 Field Emission Scanning Electron Microscope, FEI Company, USA) and AFM (NanoMagnetic Instrument, Turkey) with six specimens, thus performing their descriptive analysis. Three treated ceramic surfaces were gold-sputtered and evaluated under SEM. The representative micrographs were recorded at 500× magnifications. The other three treated ceramic surfaces were examined with an AFM. Fields of view at a scan size of 30×30 μm were considered and recorded with 2.5 μm/s scan rate. AFM images were prepared using NanoMagnetic software.

2.3. Bonding Procedure

A dual-cure cement system was used (Panavia V5, Kuraray; Tokyo, Japan) for the study. Specimens used for surface analysis with SEM and AFM were excluded from the bonding procedure (n = 15). The tooth and ceramic primers in the system were applied to the white background material (durable acetal polyoxymethylene) ([24]) and the non-glaze surface of the ceramic specimens, respectively, as the manufacturer recommended. The dual-cure cement (Clear type) was mixed using an auto-mix syringe, then inserted into the non-glaze surface of the ceramic specimens, which were seated on Delrin using finger pressure. After a five kg weight was applied ([25]), the tack cure was conducted using the light-curing unit (Ly-A180, Anyang Zongyan Dental Material, China) for 5 seconds with an output of 1600 milliwatt/centimeter2 (mW/cm2) on the glazed surfaces. The excess cement was removed, and polymerization continued using the light-curing unit for 60 seconds.

2.4. Assessment of Color Stability

A spectrophotometer device (Vita Easyshade Advance 4.0, Vita Zahnfabrik, Germany) was used for color stability measurement before and after aging. The numerical notation of the color of each specimen was obtained according to the Commission Internationale de l'Eclairage (CIE) system: b* (blue-yellow chromatic coordinate), L* (lightness, in which 100 represents white and 0 represents black), and a* (red-green chromatic coordinate) ([3, 5, 6]). After the initial color measurements, the specimens were stored in distilled water at 37°C for 24 h. A thermocycle device (Gokceler Makine, Sivas, Turkey) was used for an accelerated aging procedure with 10,000 cycles (5°C-55°C) and a dwell time of 5 s. Following the aging procedure, the color of each specimen was measured again and ΔE was calculated using a calculation formula ([6]).

2.5. Statistical Analysis

In this study, when values were determined as α=0.05 β=0.10 (1-β) =0.90, it was decided to take 15 samples for each group. The power of the test was P = 0.90849. The data obtained from this study was loaded into the program (SPSS Ver: 22.0 Chicago, USA). The groups' ΔE values were compared via the one-way ANOVA and the Tukey test. The Kruskal Wallis test and the Mann–Whitney U test were used for the other parameters in the groups. Differences were considered statistically significant at $p < 0.05$.

3. RESULTS

Table 1 presents the results of color values before the aging procedure. There are no significant differences among a1 values obtained from each group ($p > 0.05$). The same trends also occurred for b1 and L1 values ($p > 0.05$).

Table 1. a_1, b_1, L_1 values in the bonded specimens

	a_1	b_1	L_1
	Mean (SD)	Mean (SD)	Mean (SD)
Phosphoric acid	-1.38 (0.34)	8.71 (1.02)	80.64 (3.21)
Er: YAG	-1.63 (0.29)	8.79 (0.78)	80.82 (2.04)
Nd: YAG	-1.48 (0.20)	8.60 (0.95)	82.09 (1.86)
P value	P=0.052	P=0.757	P=0.297

SD: Standart Deviation. (P<.05; Mann Whitney U test).

Δa value in the Nd: YAG group was the highest among the groups (p = 0.001), whereas the other groups were comparable with each other. In the Er: YAG group, the Δb value was the lowest (p = 0.006) and ΔL value was the highest (p = 0.001), but there were no significant differences between the other groups for Δb and ΔL parameters.

The Nd: YAG laser group showed better color stability in all groups (p = 0.001) with the lowest ΔE value, but there are no significant differences between other groups ($p > 0.05$). In addition, all of the groups did not show any visible discoloration (ΔE < 3.7) after the aging procedure.

Representative SEM images of the ceramic surfaces in the groups are shown in **Figure 1 (a-c)**. A few melting points were seen in the Nd: YAG treated surface, whereas irregular areas were observed in the Er:YAG laser group. The phosphoric acid treated surfaces were regular. Representative AFM views of ceramic surfaces treated with the different techniques are presented in **Figure 2 (a-c)**. The ceramic surfaces in laser groups presented moderate irregularity surface topography.

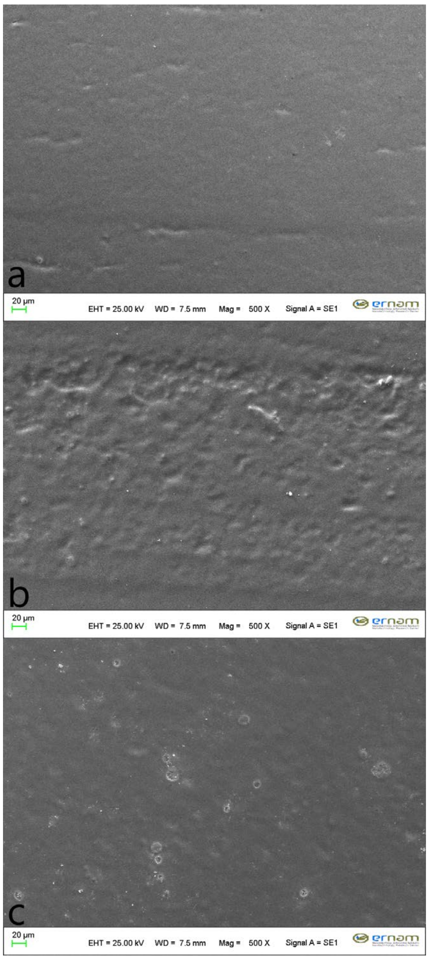

Figure 1. Representative scanning electron microscopy (SEM) images (500X) of the phosphoric acid (a), Er: YAG (b) and Nd: YAG (c) treated ceramic surfaces.

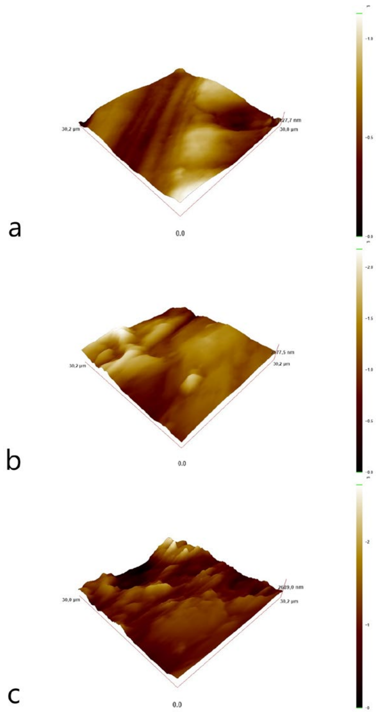

Figure 2. Representative atomic force microscopy (AFM) images (30 μm x 30 μm) of the phosphoric acid (a), Er: YAG (b) and Nd: YAG (c) treated ceramic surfaces.

4. DISCUSSION

This in vitro study measured the changing in time of the optical properties of cemented LDs after different surface pretreatment procedures. The results of this study do not support the hypothesis which there would be no difference with time in color properties of LDs subjected to different surface treatments and bonded by dual-cure resin cement. There were significant differences in color changes among the groups. Under the current study's conditions, the lowest color changes (ΔE value) were recorded from the Nd: YAG laser group (**Table 2**).

Table 2. Differences color in the bonded specimens

	Δa	Δb	ΔL	ΔE
	Mean (SD)	Mean (SD)	Mean (SD)	Mean (SD)
Phosphoric acid	-0.01 (0.09)	0.79 (1.30)	0.24 (2.35)	2.70 (0.41)
Er: YAG	0.0067 (0.07)	-0.66(0.37) *	-2.69 (0.11) *	2.79 (0.16)
Nd: YAG	0.29 (0.05) *	0.78 (1.32)	-0.61 (0.08)	1.63 (0.23) *
P value	P=0.001	P=0.006	P=0.001	P=0.001

*SD: Standart Deviation; * mean significant statistical differences inside the same column. Tukey honest significant difference test for ΔE, Mann Whitney U test for others, P<.05.*

The color properties of CAD-CAM ceramic materials may be affected by extrinsic and intrinsic factors in time. The effect of extrinsic factors (e.g., acidic solutions, coffee, smoking) depends on the physicochemical properties of CAD-CAM ceramic materials ([26, 27]), while the intrinsic factors are affected by certain properties in resin cements, such as chemical components ([3, 8]). In CAD-CAM ceramic materials, dual-cure resin cements are recommended to ensure the intended mechanical and biological properties ([28]). However, it is known that the amine content of the dual-cure resin cement causes discoloration ([8]). Therefore, the amine-free, dual-cure, resin cement was preferred in this study. However, the color of all specimens changed after aging, even though the values were under the clinically unacceptable limit (ΔE < 3.7) ([29]) in the current study. This result may be due to the Bis-GMA content of the amine-free, dual-cure, resin cement ([8, 9]). In addition, the intrinsic discoloration may be affected by the thickness of the ceramic and the color of the cementation area in the background ([30]). Ceramics with a greater thickness exhibit higher color stability ([8, 31]). The color stability of dental materials should measure on a white background according to ISO standardization ([32]). The ceramic thickness of 1 mm and Delrin (white colored and durable acetal polyoxymethylene) ([24]) were used to ensure standardization in the current study. Also, there were no statistical differences among groups when considering a_1, b_1, and L_1 parameters in this study. These results may prove that initial standardization is achieved for the color measurement of the specimens.

The properties of CAD-CAM ceramic surfaces before cementation play an important role in the clinical success of restorations. Resin cements bond to the surface mechanically and chemically. The mechanical bonding occurred from the roughening of the surface and infiltration of the resin cement on the surface ([10]). As recommended by resin cement manufacturers, increasing the ceramic surface roughness is possible thanks to both hydrofluoric and phosphoric acids. However, there are certain controversial reports about which one is more effective on bond strength between ceramic and resin cement. In one study ([33]), the bond strength between ceramics and orthodontic brackets cemented with a composite resin-based bonding system was analyzed after hydrofluoric and phosphoric acid treatments, revealing similar bond strength values between the groups. In another study ([34]), the bond strength of a lithium disilicate-based ceramic and stainless steel rods cemented with a self-adhesive resin was examined, and phosphoric acid-treated surfaces had a weaker bond strength than those treated with hydrofluoric acids. Generally, using hydrofluoric acid in terms of providing bond strength between resin cement and ceramic is the most favored method ([4]). It has been shown in a study that there is no change in the optical properties of LDs roughened with hydrofluoric acid and aged only half as much as in our study ([20]). After a hydrofluoric acid treatment, the glassy matrix of the ceramic is selectively removed, crystalline structures are exposed, and an abundant porosity is formed on the ceramic surface thereby contributing to the bond strength ([13, 35]). However, hydrofluoric acids have inherent flaws, such as being caustic and hazardous ([36]). Also, insoluble silica-fluoride salts may weaken the bond strength between the ceramic and the ceramic primer when hydrofluoric acids are used. Whereas, phosphoric acids are relatively safer and do not produce any compounds that decrease the bond strength and are non-washed easily ([34]). Phosphoric acid may minimally roughen surfaces ([34, 37]), but can neutralize the alkalinity of the absorbed water layer on the ceramic surface, thus contributing to the chemical activity of the ceramic primer that is subsequently applied ([33]). It has been reported that bond strength between ceramic and cement may increase when phosphoric acid is used with ceramic primer ([34]). Also, in the current study, phosphoric acid was used with ceramic primer as the manufacturer recommended.

The SEM and AFM images of the phosphoric acid group presented a surface that was superficially modified in the current study. In previous studies ([14, 15]), the effect of Er: YAG and Nd: YAG lasers on the roughness of the lithium disilicate-based ceramics were examined. It was reported that there were no significant differences between Er: YAG and Nd: YAG lasers-treated surfaces with similar topographic AFM images (**Figure 2**). This result was similar to those of the current study. In the SEM images of the current study, irregular areas were observed in the Er: YAG laser group, while the Nd: YAG laser-treated surface presented certain melting points. It may be related that the Nd: YAG laser causes surface roughness by melting ([10, 17]) and random re-crystallization, which increases the strength of the resin-ceramic bond (**Figure 1**) ([10]).

Aging procedures can cause color change in resin cements ([38]) as resin-based materials tend to cause yellowing after aging ([25]). After cementation, the final shade of CAD-CAM materials is clinically acceptable, and ceramics may mask most of the color change caused by resin cement ([3]). Also, in the current study, the color change of all specimens is found as being under the clinically unacceptable limit ($\Delta E < 3.7$) ([29]). Using phosphoric acid as the manufacturer recommended caused brighter specimens after aging (positive ΔL) in the current study. The same result, an increase in L* value after aging, was reported by a study that used dual-cure resin cement, as in the current study ([8]). On the other hand, the alterations at the surface texture may affect the color properties of the ceramics ([5, 6]). In the current study, the L* value decreased in the laser groups and laser treatment with the Er: YAG laser caused the ceramic veneers to become the darkest and more bluish. However, previous studies have reported that laser treatment with the Er: YAG laser caused the LDs to become more yellowish and brighter ([6]). These differences may be related to different adhesive system used, the testing conditions, and the use of different laser parameters during the etching procedure. The least difference in color change among groups was in the Nd: YAG laser-treated group in the current study. The laser-treated ceramic surface may become more opaque ([5]), thus the light that passed through the ceramic would no longer pass through the matter with the same incidence and direction. The observed melting areas in the SEM image of the Nd: YAG group may be related to this result.

The results of the present in vitro study are of clinical relevance because they provide information related to the effect of surface pretreatment on the optical properties of cemented lithium disilicate ceramics. Dentists applying LDs may use Nd: YAG laser before cementation to protect LD restoration from color changes that may occur over time, according to this study's results. However, the current study had a few limitations based on its in vitro methodology. First, only one type and color of resin cement and also one type of LDs ceramic material that has one color with a certain thickness were evaluated for standardization. The results cannot be applied to other LDs ceramic materials which are cemented with other resin cements that have different characteristics. Second, the shear bond strength parameter was not evaluated, even if using phosphoric acid that the manufacturer recommended and laser parameters were selected from those that created the best bond strength in the literature, according to the authors' knowledge. Third, translucency of treated-LD, which gives information about the color masking capacity, was not evaluated. Fourth, the phosphoric acid application time was applied according to the resin cement manufacturer's instructions, but different acid dwells that the ceramic material manufacturer may suggest should also be considered. Further research is needed to better understand the possible effects of surface treatments on the color stability of CAD-CAM ceramic materials. In future studies, various CAD-CAM ceramic materials with different properties and resin cement in different structures should be evaluated by applying according to both materials manufacturers' instructions. Also, different parameters such as resin cement-ceramic bond strength, translucency, shade, different thickness should be investigated in these materials.

CONCLUSION

Within the limitations of the current study, the following can be concluded:

1. There was no clinically unacceptable color change (Delta E > 3.7) in all groups.
2. The Nd: YAG laser group presented better color stability.
3. Among the groups, Δa value of the Nd: YAG group was the highest, whereas the Δb value of the Er: YAG group was the lowest and the ΔL value of the Er: YAG group was the highest.
4. The SEM and AFM evaluations revealed irregular surface morphology in laser groups when compared to the phosphoric acid group.

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Değerlendirme / Peer-Review

İki Dış Hakem / Çift Taraflı Körleme

Etik Beyan / Ethical statement

Bu çalışmanın hazırlanma sürecinde bilimsel ve etik ilkelere uyulduğu ve yararlanılan tüm çalışmaların kaynakçada belirtildiği beyan olunur.

It is declared that during the preparation process of this study, scientific and ethical principles were followed and all the studies benefited are stated in the bibliography.

Benzerlik Taraması / Similarity scan

Yapıldı – ithenticate

Etik Bildirim / Ethical statement

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Çıkar Çatışması / Conflict of Interest

Yazarlar çıkar çatışması bildirmemiştir. | The authors have no conflict of interest to declare.

Yazar Katkıları / Author Contributions

Çalışmanın Tasarlanması | Design of Study: ODD (%60), GAD (%40)
Veri Toplanması | Data Acquisition: ODD (%100)
Veri Analizi | Data Analysis: ODD (%100)
Makalenin Yazımı | Writing up: ODD (%10), AK (%90)
Makale Gönderimi ve Revizyonu | Submission and Revision: AK (%100)

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