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Original research

A study on the discoloration of different dental porcelain systems

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Purpose
This study aimed to investigate the discoloration of four types of dental porcelain systems (feldspathic, monolithic zirconia, lithium disilicate, and leucite glass-ceramic) with various surface treatments (glazed and mechanically polished) after undergoing chewing simulation against a natural tooth antagonist.

Materials and Methods
Disc-shaped porcelain groups (9x3mm) were produced for the following categories: feldspathic glazed (FG), feldspathic mechanically polished (FM), zirconia glazed (ZG), zirconia mechanically polished (ZM), lithium disilicate glazed (ExG), lithium disilicate mechanically polished (ExM), leucite glass-ceramic glazed (EG), and leucite glass-ceramic (EM). In vitro chewing simulation was conducted on all porcelain groups, and a spectrophotometer was used to compare the color changes between their initial states.

Results
The ΔE values (discoloration) of all porcelain samples were statistically different ($p<0.05$), and the ΔE values of the glazed samples were higher than those of the mechanically polished samples.

Conclusion
All the glazed samples are more likely to show more color change than the mechanically polished sample groups.

Keywords: *Dental ceramics, color stability, discoloration, glaze, mechanical polish*

Introduction

The fundamentals of planning and applying current dental prosthetic treatments are based on restoring the integrity of lost tissue, regulating function and phonation, and meeting the patient's aesthetic preferences to the highest degree (1, 2). In recent decades, with technological advances and patients' preference for fully natural products, polycrystalline ceramic and glass-ceramic monolithic porcelain restorations have gained importance in dentistry due to their edge and biological compliance with tissues, superior aesthetic properties, color stability, chemical durability, and corrosion resistance (3-6). However, ceramic compounds have different chemical-physical and optical characteristics influenced by crystal structures, elemental compounds, and the chemical composition of the matrix and particle positions (7). Some studies have investigated the translucency of ceramics, one of their optical characteristics, and have found that the thickness, chemical structure of crystal particles, and particle sizes in the matrix affect translucency for all ceramics, including glass-ceramics and polycrystalline ceramics (7, 8).

The glaze structure is another factor affecting the optical characteristics of ceramics, as the presence of glaze increases their translucency (9).

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Additionally, the glaze layer increases the surface energy of ceramics, reduces plaque retention, and makes the cleaning process easier (10-12). To maximize the surface characteristics of dental ceramics, mechanical polishing is another surface finishing technique that enhances the material's mechanical and optical features (13-15).

However, it is evident that during most cementation processes, the glaze layer is removed by occlusal adjustment. On the other hand, clinical studies have reported that the glaze layer is removed by daily usage within 6 months (16). Once the glaze layer fades, dental porcelains become rough and less attractive in terms of aesthetics. It has been reported that an increase in surface roughness of restorative materials has negative effects on their discoloration (13-15). Bollen *et al.* (12) reported in their study that when the roughness level exceeds 0.2 μm, the plaque accumulation also increases, in addition to discoloration.

The ΔE values of the CIE L*ab** system, which is a standard and reliable method, are used in literature and studies to calculate color changes. CIE L* measures the lightness of the material, CIE a* measures the redness (positive value) or greenness (negative value), and CIE b* measures the yellowness (positive value) or blueness (negative value). In this calculation system, ΔE values define the level of color change. It has been reported that a color change can be imperceptible to the human eye when ΔE values are lower than 1; when the values are between 1.0 and 3.3, changing the color is hardly noticeable and is generally considered clinically acceptable. However, when ΔE value is higher than 3.3, the color change can be easily detected and is considered clinically unacceptable (17-19).

The purpose of our study is to evaluate the discoloration of four different dental porcelains, including Feldspathic (Super Porcelain EX-3, Kuraray Noritake Dental Inc.), Zirconia (Katana Zirconia UTML, Kuraray Noritake Dental Inc.), Leucite (IPS Empress CAD, Ivoclar VIVADENT), and Lithium Disilicate (IPS e.max CAD, Ivoclar VIVADENT), in their glazed and mechanically polished versions after undergoing chewing simulator tests. The null hypothesis of this study is that there will be no difference in the optical properties of the dental ceramics between their mechanically polished and glazed versions after undergoing the chewing simulation.

Materials and Methods

Sample characteristics

The present study analyzed the color change patterns of four different dental porcelain systems: Super Porcelain EX-3 (Kuraray Noritake Dental Inc., Tokyo, Japan), Katana Zirconia UTML (Kuraray Noritake Dental Inc., Tokyo, Japan), IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein), and IPS empress CAD (Ivoclar Vivadent, Schaan, Liechtenstein).

Study groups

The porcelain groups were divided into two sub-groups and treated with different surface processes, namely glaze and mechanical polish (Table 1). The total number of porcelain samples was 64. Sixty-four premolar and canine teeth, which were extracted due to orthodontic and periodontal indications, had no tissue loss from mechanical and chemical factors, and had no cavities or fillings, were prepared as antagonists for the porcelain samples. The samples were cleaned of debris using low rev polish, and prior to enamel surface processing, the sample teeth were soaked in a 0.1% thymol solution to prevent possible corrosion of the enamel tissue.

Table 1. *Porcelain samples stratified by the the composition and surface treatment.*

No	Porcelain Material	Manufacturer	Composition	Surface Treatment Method	Code
1	**Super Porcelain EX-3**	Kuraray Noritake Dental Inc.	Feldspathic	Glaze	FG
				Mechanical Polish	FM
2	**KATANA Zirconia UTML**	Kuraray Noritake Dental Inc.	Zirconia	Glaze	ZG
				Mechanical Polish	ZM
3	**IPS e.max CAD**	Ivoclar VIVADENT	Lithium disilicate	Glaze	ExG
				Mechanical Polish	ExM
4	**IPS Empress CAD**	Ivoclar VIVADENT	Leucite	Glaze	EG
				Mechanical Polish	EM

Preparation of porcelain samples

Dental porcelain samples were fabricated in standardized cylindrical forms with a width of 9 mm and a height of 3 mm. The dimensional controls were performed using a digital caliper.

Preparation of feldspathic porcelain samples

Super Porcelain EX-3, a low-temperature feldspathic porcelain system, was prepared using the conventional manual method. As recommended by the manufacturer and due to known material shrinkage during heat treatment, the samples were molded into 10% larger PEEK (Polyether ether ketone) molds. The manually fabricated feldspathic porcelain was heat-treated for 7 minutes at 600°C as per the manufacturer's instructions. Then, the vacuum process was initiated, and the process was stopped at 920°C. The entire heat treatment process was completed at 930°C with a heating rate of 45°C per minute.

A total of 16 feldspathic porcelain samples were prepared. Within the subgroups, 8 samples were glazed and the other 8 were mechanically polished. For glaze treatment of FG samples, a mixture of Super Porcelain External Stain Glaze powder and ES LIQUID was used (Kuraray Noritake Dental Inc., Tokyo, Japan). The process was initiated with 5 minutes of dehumidification, followed by the application of the glaze material to the porcelain surfaces using a porcelain brush. The heat treatment process was then started at 650°C and completed at 910°C with a heating rate of 50°C per minute. The heat treatment and glaze processes were carried out using Ivoclar Vivadent Programat ep 3000 (Ivoclar Vivadent, Schaan, Liechtenstein).

Preparation of monolithic zirconia ceramics

The monolithic zirconia CAD/CAM porcelains were prepared from Katana Zirconia UTML blocks. The zirconia discs were milled using a CAD/CAM machine Yenadent D43 milling machine (Yenadent, Istanbul, Turkey) and then sintered following the manufacturer's guidelines. The sintering process was performed with a heating rate of 10°C per minute, and the temperature in the oven was stabilized at 1550°C for two hours before being decreased to room temperature at a rate of 10°C per minute.

For the zirconia samples, 8 of them were glazed and the other 8 were mechanically polished. For the ZG group, a mix of CERABIEN ZR VC Glaze Powder and liquid porcelain was used (Kuraray Noritake Dental Inc., Tokyo, Japan). The procedure was done according to the guidelines of the manufacturer. After 5 minutes of dehumidification, the glaze heat treatment process was initiated at 600°C under vacuum (72cm/Hg), and the heat was increased to 850°C at a rate of 65°C per minute.

Preparation of lithium disilicate ceramics

The lithium disilicate discs were carved via CAD/CAM device Yenadent D43 milling machine (Yenadent, Istanbul, Turkey) and heat-treated according to the manufacturer's guidelines. After the 6-minute dehumidification process at 403°C, the heat treatment was initiated, and the heat decreased 90°C per minute until the target temperature of 820°C was reached. The first vacuum was applied between 550°C and 820°C during the initial heat treatment. After waiting for 10 seconds at 820°C, the second stage of the heat treatment was started. The temperature was increased up to 840°C at a rate of 30°C per minute during the second heat treatment process. The vacuum was applied at each stage of the process. After the samples were stabilized at 840°C for 7 minutes, they were left to cool down. 8 of the samples were glazed and the other 8 were mechanically polished.

For the ExG group, a mix of IPS Ivocolor Glaze Powder and Liquid material was used (Ivoclar Vivadent, Schaan, Liechtenstein). After the samples were dehumidified at 403°C for 6 minutes, they were heat-treated with an increase of 60°C per minute up to 725°C, and the glazing process was completed by keeping the samples at this temperature for one minute. The vacuum was applied between 450°C and 724°C during the firing process.

Preparation of leucite glass ceramics

Leucite glass-ceramic was prepared via CAD/CAM device Yenadent D43 milling machine. 8 of the samples were glazed and the other 8 were mechanically polished. For the EG group, the IPS Empress Universal Glaze Liquid was applied with a porcelain brush (Ivoclar Vivadent, Schaan, Liechtenstein), and then the dehumidification process was carried out at 403°C for 6 minutes. After the samples were heated at an increased rate of 55°C per minute up to 790°C and kept at this temperature for a maximum of 2 minutes, the glazing process was completed. The vacuum was applied between 450°C and 789°C during the firing process.

Application of glaze and mechanical polish

Under the heading of preparation of samples above, the glaze materials used for each group and the protocols followed are explained. For all samples, the glaze application process was carried out as follows: first, each sample was dehumidified, then a uniform layer of overglaze porcelain liquid was evenly applied on one side of the samples with a porcelain brush by one operator. After this process, the samples were fired as recommended by the manufacturers.

The mechanical polishing was carried out using the EVE Diapol three-stage porcelain polishing kit (EVE Ernst Vetter GmbH, Keltern, Germany) and Renfret Polish All in One polishing paste (Renfrent, Hilzigen, Germany). According to the manufacturers, these polish kits and paste can be used on all porcelain samples in our study (zirconia, feldspathic, lithium disilicate, and leucite porcelains). In addition, a single mechanical polishing system was chosen for standardization for all porcelains in our study.

The mechanical polishing was carried out as follows: All polishing processes were applied by one operator. Each of the mechanical polishing steps included coarse, medium, and fine rubber discs (EVE Diapol Rubber Discs), and polishing paste (Renfret Polish All in One polishing paste) with cotton buff, respectively. In the polishing kit, blue rubber discs were used for coarse polish, then medium polish was applied with pink rubber discs, and finally, a third step was carried out with white rubber discs for a fine finish. Each step was performed with a 10,000-rpm rotary instrument, lasting a minimum of one minute. This polish kit was chosen because, according to its manufacturer, it can be used for all types of porcelain (zirconia, feldspathic, lithium disilicate, and leucite porcelains) as well as for the standardization of mechanical polish kits for all porcelains. After mechanical polishing with rubber discs, polishing paste (Renfert Polish All in One) was applied to all porcelains with a cotton buff for at least 60 seconds with a 7,000-rpm rotary tool for a spotless and high-gloss polish finish.

The chewing simulation

The chewing simulation test on porcelain samples of our study was carried out via the SD Mechatronic Chewing Simulator CS-4.8 Biaxial Fatigue Testing system (SD Mechatronik GMBH, MiesbacherStrasse 34 D-83620 Feldkirchen-Westerham, Germany). For the simulation, the sample porcelains were embedded into acrylic and placed into retainers. The antagonist's teeth were also embedded into acrylic and placed into retainers. Samples and teeth were placed into the simulator and calibrated. The samples were subjected to 600,000 chewing cycles at 50N chewing pressure and a frequency of 1Hz, while thermal cycles were applied to fatigue the dental ceramics. The ceramics underwent 10,000 thermal cycles from 5°C to 55°C.

Color assessment

Color values of each porcelain sample were measured after surface finishing applications and after chewing simulation with thermal cycle using the Vita Easyshade Advance (Vita Zahnfabrik Bad Sackigen Germany) spectrophotometer system. A black background was used to mimic the oral environment, and for the standardization of measurements. The ΔE values of the CIE Lab* system were used to calculate the color changes.

Statistical analysis

The statistical package for Social Sciences (SPSS) software version 22.0 (IBM Corp. Released 2013. IBM SPSS Statistics for Windows, Version 22.0. Armonk, NY, USA) was used for data evaluation. The normality of the dataset was checked with Kolmogorov-Smirnov test. Multiple and pairwise comparisons were made, respectively, with analysis of variance and Tukey's Honestly Significant Difference (HSD) tests. The confidence interval was set to 95% and p values less than 0.05 were considered significant.

Results

After analyzing the color changes of glazed and mechanically polished porcelains with different chemical structures, it was found that porcelains with the same surface finish process exhibited significant differences in ΔE values among samples with different compositions ($p<0.05$, Table 2). Among the glazed porcelains, group EG had the highest ΔE values, while the least color change was observed in group FG ($p<0.05$). For samples treated with mechanical polishing, the highest ΔE values were found in group EM, similar to the glazed porcelains (Figure 1). However, the porcelains that underwent mechanical polishing with the least color change were in group ZM ($p<0.05$).

Table 2. *ΔE and standard deviation values of the porcelain specimens according to surface treatments.*

Porcelain	N	Average	Std. Deviation	Result
FG	8	2,7388	,01246	F=8266,17 P=0,001*
ZG	8	3,0613	,00835	
ExG	8	3,1863	,00518	
EG	8	3,4313	,00835	
FM	8	1,2100	,00756	F=9285,44 P=0,001*
ZM	8	,7663	,01302	
ExM	8	2,0263	,02615	
EM	8	2,4563	,03335	

*p<0,05 significant

As shown in Table 3, the ΔE values of all glazed samples were higher than those of the mechanically polished samples when comparing post-simulation values of samples with the same composition in different surface treatment groups. Mean values in group EG were above the apparent and unacceptable limit of 3.3, while values in glaze groups other than FG were above 3 ($p<0.05$).

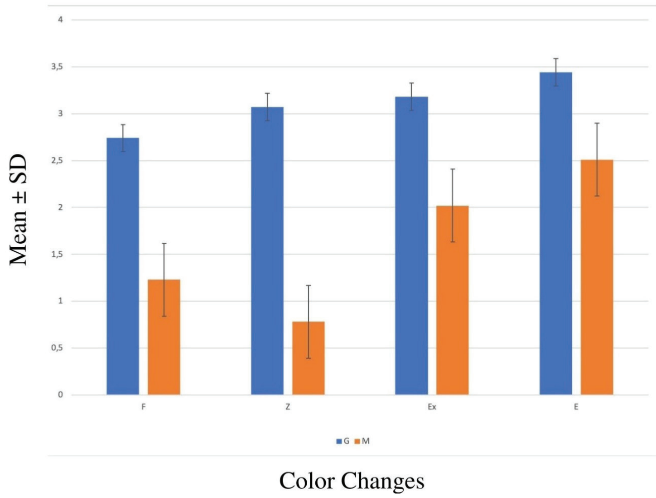

Figure 1. *Color changes of the porcelain samples.*

Table 3. *ΔE and standard deviation value of glaze and mechanical sub-groups of porcelain groups according to their contents.*

Porcelain	N	Average	Std. Deviation	Result
FG	8	2,7388	,01246	F=1,46 P=0,001*
FM	8	1,2100	,00756	
ZG	8	3,0613	,00835	F=1,87 P=0,001*
ZM	8	,7663	,01302	
ExG	8	3,1863	,00518	F=5,57 P=0,001*
ExM	8	2,0263	,02615	
EG	8	3,4313	,00835	F=14,51 P=0,001*
EM	8	2,4563	,03335	

*p<0,001 significant

Discussion

The null hypothesis of the present study was that there would be no difference in optical properties of dental ceramics after the chewing simulation on surfaces that were mechanically polished and glazed, but this hypothesis was rejected. The results of the study indicated that the initial surface treatments, including different surface preparation, mechanical polishing, and glazing methods, affected the color changes after the chewing simulation.

The ceramics used in this study are commonly known and used in routine dental practice. Manufacturers provide various options for color selection, including different hues, chroma, and translucency, such as high translucency (HT) or low translucency (LT). For standardization, A1 color low translucency blocks were selected in this study.

Aesthetic appearance is a critical success criterion for dental materials, which can be described as the compatibility of the materials with natural teeth and their color stability (20). Patients' expectations and the durability of aesthetical success are of great importance in dental restoration processes. Discoloration is usually at the top of the list of reasons for patients' dissatisfaction with dental restoration processes (21).

As mentioned earlier, the surface properties of ceramics, such as the thickness of the material and the chemical composition of the structure, also affect the optical characteristics (22, 23). The porosity of the surface structure or the increase in the roughness of the ceramics can lead to an increase in plaque uptake in the mouth. Furthermore, the rough surface can affect the reflection of light, thereby impacting the optical characteristics of the ceramic, such as gloss (13, 15).

In clinical practice, glazing and mechanical polishing (with or without polish paste) are the generally used procedures for ceramics surface treatment (24, 25). Studies have shown that the glaze layer directly affects the color characteristics of the materials. Glazed surfaces have higher success in reflecting light, and the glazing procedures on surfaces are very successful in achieving the desired surface properties of ceramics (24, 26). However, it has been reported that if the glaze material is not applied uniformly to the surface during the glaze surface treatment, it can affect the surface properties (25, 27).

After the cementation process of fixed restorations, occlusal and proximal adjustment or shape corrections are required. However, after these adjustments, the glaze layer is lost (28-31). It has been reported that faded glaze layers on rough ceramic surfaces reflect light less directly, resulting in a negative effect on the color values of the restorations (32). In the present study, we found that the glazed surface's color-changing parameters are higher than those of mechanically treated surfaces. Our theory is that the occlusal abrasion forces lift the glazed surface, causing more color changes than just mechanically treated surfaces in their initial state after simulation.

Studies suggest that mechanical polish kits with polish pastes are a successful alternative for removed glaze layers (24, 28, 33). Mohammadibassir *et al.* also claim that repeated firing procedures for repairing glazing treatment reduce ceramic surface quality (28). Vichi *et al.* (25) mention that mechanical polishing is a good alternative for glaze-treated surfaces and that the mechanical application duration is essential for surface smoothening. In their study, 60 s of mechanical-polished lithium disilicate and zirconia-reinforced lithium silicate ceramics showed the lowest roughness parameter. Nevertheless, ceramics mechanically polished for just 30 s showed no difference between their initial surface conditions (25). Can *et al.* state that mechanical polishing affects glaze-treated ceramic surfaces (34). In our study, we applied mechanical surface treatment via a polishing kit, and each procedure was applied for at least 60 s.

ΔE values are used to base the discoloration values on numerical values of different samples of the same processes or the same samples of different processes (35, 36). The human eye cannot notice the discoloration ΔE values that are below 1 (36-38). As previously mentioned, ΔE values between 1 and 3.3 are acceptable discoloration levels in clinical work, and these color changes are barely noticeable by the human eye. Change values over 3.3 are easily noticeable and called clinically unacceptable (17-19, 39). Studies regarded 3.3 as the limit to ΔE value (40, 41).

On repeated color measurements, there are studies that report that Vita Easyshade (Vita Zahnfabrik) spectro-photometer, which we used in our study, is a reliable device (42, 43). Moreover, other reports prove that the results are not affected under different types of light sources (42).

According to the data that we gathered from our study, except for the FG group, the ΔE levels of glaze groups are higher than 3. Also, it needs to be added that for those three-group color differences from the initial forms, they can be easily visible. All glazed-porcelain values are also higher than those of mechanically polished groups. We observed that the ΔE values of mechanically polished groups are lower than 3.3, which was previously mentioned to be the highest acceptable value (40, 41). Upon ΔE value evaluation of the porcelain groups exposed to the same surface processes, we found that the samples that were abraded more had higher ΔE values. We can conclude that the glazed group's worse discoloration level results from the removal of the glaze layer upon exposure to external effects, causing more wear on the surfaces of the samples.

In their study on porosity and discoloration of porcelains exposed to different polishing processes, Saraç *et al.* reported that materials with higher porosity levels also had higher ΔE levels (44). Similarly, in their study on the discoloration of porcelain systems containing various chemical compounds, Soygun *et al.* stated that the surface porosity value was directly proportional to the ΔE level (45). Our study's findings are in line with those of the previous work, indicating that the greater the surface structure change, the greater the discoloration. Specifically, we observed the highest levels of discoloration in the leucite and lithium disilicate groups.

Conversely, the feldspathic and zirconia groups showed the least amount of discoloration in our study, potentially due to the smaller scale wear of these materials. Furthermore, the larger average particle sizes of lithium disilicate and leucite ceramics compared to zirconia ceramics may account for the higher amount of wear and discoloration observed on their surfaces (22, 25).

We also noted that the mechanically polished samples showed less discoloration compared to the glazed samples in our study. This difference may be attributed to the removal of the glaze layer over time. However, our study had several limitations, including the lack of fully standardized extracted teeth used as antagonists, the disc-shaped porcelains that do not imitate natural morphological crowns, and the changes in porcelain behavior against abrasive forces and their optical properties in the oral environment, making analysis challenging. As such, future studies could involve simulations with anatomically-shaped crowns and standard antagonists, and even clinical studies.

Conclusion

The glaze layer is removed due to occlusal adjustment and exposure over time. The surfaces then become rough, resulting in heavier wear and abrasion. Additionally, the removal of the glaze layer and the appearance of rough surfaces can cause aesthetic issues and affect patient satisfaction. In light of the aforementioned circumstances, dentists can perform routine mechanical polishing of surfaces to improve the aesthetic and mechanical features of the materials. It can also be argued that zirconia ceramics' applications may be more comfortable, especially for patients with bruxism, due to their corrosion resistance, smaller impact on their antagonists, and lower discoloration levels resulting from stabilized surface characteristics.

Türkçe özet*: Farklı dental porselen sistemlerinin renk değişimleri üzerine çalışma. Amaç: Bu çalışmada, doğal diş antagonistlerine karşı çiğneme simülasyonu sonrasında çeşitli yüzey işlemlerine tabi tutulan (glaze uygulanmış ve mekanik cila uygulanmış) dört tip dental porselen sisteminin (feldspatik, monolitik zirkonya, lityum disilikat ve lösit cam-seramik) renk değişimleri incelenmiştir. Gereç ve Yöntem: Yüzey bitim işlemleri uygulanan, feldspatik glaze uygulanmış (FG), feldspatik mekanik cila uygulanmış (FM), zirkonya glaze uygulanmış (ZG), zirkonya mekanik cila uygulanmış (ZM), lityum disilikat cam-seramik glaze uygulanmış (EXG), lityum disilikat cam-seramik mekanik cila uygulanmış (ExM), lösit cam-seramik glaze uygulanmış (EG), lösit cam-seramik mekanik cila uygulanmış (EM) porselen grupları 9x3 mm boyutlarında disk formunda hazırlandı. Bütün porselenler in-vitro ortamda çiğneme simülasyonuna tabi tutuldu. Simulasyon sonrası örneklerin başlangıç durumları ile renk değişimlerinin incelenmesi için spektrofotometre kullanıldı. Bulgular: Tüm porselen örneklerin ΔE değerleri (renk değişim değerleri) istatistiksel olarak farklı ($p<0,05$) ve glaze uygulanmış numunelerin ΔE değerleri mekanik cilalı numunelerin ΔE değerlerinden daha yüksek bulunmuştur. Sonuç: Tüm glaze işlemi uygulanmış örneklerin renk değişim değerleri mekanik cila uygulanmış örnekelere göre yüksek olduğu belirlenmiştir. Anahtar Kelimeler: dental seramik, renk stabilitesi, renk değişimi, glaze, mekanik cila*

Ethics Committee Approval: The study protocol has been reviewed and approved by the Sivas Cumhuriyet University Clinical Research Ethics Committee (Decision no: 2016-05/9).

Informed Consent: Participants provided informed constent.

Peer-review: Externally peer-reviewed.

Author contributions: MCU, GB participated in designing the study. MCU, GB participated in generating the data for the study. MCU, GB participated in gathering the data for the study. MCU, GB participated in the analysis of the data. MCU, MKA, GB wrote the majority of the original draft of the paper. MCU, MKA, GB participated in writing the paper. MCU, MKA, GB have had access to all of the raw data of the study. MCU, MKA, GB have reviewed the pertinent raw data on which the results and conclusions of this study are based. MCU, MKA, GB have approved the final version of this paper. MCU, MKA, GB guarantee that all individuals who meet the Journal's authorship criteria are included as authors of this paper.

Conflict of Interest: The authors declared that they have no conflict of interest.

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