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# Evaluation of the extrinsic characterization layer of zirconium-infiltrated lithium silicate glass ceramic

Avaliação da camada de caracterização extrínseca de um vitrocerâmica de silicato de lítio infiltrada com zircônia

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## ABSTRACT

**Objective:** To evaluate the effects of the extrinsic characterization process on the optical properties and roughness of zirconium-infiltrated lithium silicate glass-ceramic. **Material and Methods:** Twenty-four Disc-shaped samples (Ø=12 mm and 1.2 mm in thickness) were divided into two groups (n=12): "Without Characterization" (C) – control group, and "With Characterization" (CC). Surface topography (Ra) was analyzed using a contact profilometer, color and translucency were evaluated using a spectrophotometer. The samples were subjected to aging in red wine for 15 minutes over 30 days. After aging, the tests were repeated. Two-way ANOVA, and Tukey's 5% test were applied to roughness and translucency. The Kruskal-Wallis and Dunn's tests were used for color analysis. **Results:** Roughness (Ra), color variation, and translucency were statistically affected only by the "characterization" factor ($p<0.001$). The CC group exhibited the highest Ra values and the greatest color variations, before and after aging. The C group, with and without aging, presented the highest translucency values. **Conclusion:** It was observed that characterization influenced color variations, translucency and increased the surface roughness before and after aging. Aging in red wine did not significantly affect color variation or translucency; however, it resulted in a color alteration that can be clinically perceptible. Clinical relevance: While the characterization process enhances the aesthetic customization of restorations, clinicians should be aware that it may lead to increased roughness and greater susceptibility to clinically perceptible color changes over time. These findings highlight the need for careful selection and application of characterization protocols to balance aesthetics and material longevity.

## KEYWORDS

Ceramics; Color; Dental Materials; Glass ceramics; Surface roughness.

## RESUMO

**Objetivo:** Avaliar os efeitos do processo de caracterização extrínseca nas propriedades ópticas e na rugosidade de vitrocerâmicas de silicato de lítio infiltradas com zircônia. **Material e Métodos:** Vinte e quatro amostras em formato de disco (Ø = 12 mm e 1,2 mm de espessura) foram divididas em dois grupos (n = 12): "Sem Caracterização" (C) - grupo controle, e "Com Caracterização" (CC). A topografia da superfície (Ra) foi analisada usando um perfilômetro de contato; a cor e a translucidez foram avaliadas usando um espectrofotômetro. As amostras foram submetidas ao envelhecimento em vinho tinto por 15 minutos ao longo de 30 dias. Após o envelhecimento, os testes foram repetidos. ANOVA-2 fatores e o teste de Tukey a 5% foram aplicados à rugosidade e à translucidez. Os testes de Kruskal-Wallis e teste de Dunn foram usados para análise de cor. **Resultados:** A rugosidade (Ra), a variação de cor e a translucidez foram estatisticamente afetadas apenas pelo fator "caracterização" ($p<0,001$). O grupo CC apresentou os maiores valores de Ra e as maiores variações de cor, antes e após o envelhecimento. O grupo C, com e sem envelhecimento, apresentou os maiores valores de translucidez. **Conclusão:** Observou-se que a caracterização influenciou as variações de cor, a translucidez e aumentou a rugosidade da superfície antes e após o envelhecimento.

O envelhecimento em vinho tinto não afetou significativamente a variação de cor ou a translucidez; no entanto, resultou em uma alteração de cor que pode ser clinicamente perceptível. Relevância clínica: Embora o processo de caracterização aprimore a personalização estética das restaurações, os clínicos devem estar cientes de que ele pode levar ao aumento da rugosidade e à maior suscetibilidade a alterações de cor clinicamente perceptíveis ao longo do tempo. Esses achados destacam a necessidade de seleção e aplicação cuidadosas de protocolos de caracterização para equilibrar a estética e a longevidade do material.

## PALAVRAS-CHAVE

Cerâmica; Cor; Materiais Dentários; Cerâmicas Vitrocerâmicas; Rugosidade de Superfície.

## INTRODUCTION

The development of computer-aided design and computer-aided manufacturing (CAD-CAM) technology, combined with the increasing patient demand for aesthetic dental restorations, has greatly influenced the advancement of ceramic restoration materials with various microstructures aimed at achieving excellent mechanical and optical properties [1]. In recent years, lithium disilicate and zirconia ceramics have gained popularity for monolithic restorations, with lithium disilicate offering higher translucency and lower mechanical properties compared to zirconia [2]. The continuous advancement in ceramic materials to combine high optical and mechanical properties in a single material has led to the development of lithium silicate glass ceramic reinforced with zirconia [3]. This material has a significant phase of lithium metasilicate (Li2SiO3) and a smaller phase of lithium orthophosphate crystals (Li3PO4). The glassy matrix is supplemented with approximately 10% zirconium dioxide (ZrO2), which, after the final crystallization process, results in the formation of fine-grained lithium silicate with zirconia (Li2O-ZrO2-SiO2) [4]. Thus, this ceramic combines a glass portion (Li2SiO3) with a crystalline portion of zirconia (ZrO2) [5]. The glassy portion provides the optical aesthetic characteristics of the material, such as translucency and a natural tooth-like appearance. However, it is highly susceptible to fracture and crack propagation due to its inherently low fracture toughness. This limitation arises from the brittle nature of ceramic materials, which are highly sensitive to stress concentrations around pre-existing flaws, such as pores or surface defects formed during processing, handling, or finishing. When subjected to tensile loading, the stress intensity at the tip of these flaws increases, and once it reaches the material's critical stress intensity factor ($K_{Ic}$), crack propagation becomes unstable, ultimately leading to failure [6]. According to the manufacturer (Dentsply. CeltraDuo), the amount of zirconium dioxide incorporated into the ceramic is up to ten times greater than that incorporated into other glass ceramics, so its small grains can result in a more resistant material with better surface polishing [7]. However, zirconia can compromise the translucency of ceramic materials due to the high refractive index mismatch between the zirconia crystals and the glass matrix. This mismatch increases light scattering at the crystal–matrix interfaces, resulting in greater opacity. [8].

For better aesthetic results in restorations, the use of extrinsic characterization of the ceramic material is recommended. In addition to providing a better appearance, extrinsic characterization enhances biocompatibility by reducing biofilm retention and wear on the opposing tooth [9]. Extrinsic characterization involves the application of stain and glaze to the surface of the material to achieve the desired color of the prosthetic restoration [10]. A previous study demonstrated that polishing performed after crystallization reduces surface roughness and increases translucency and opalescence parameters, while the application of glaze increased surface roughness and decreased translucency and opalescence parameters [9]. Thus, studies that test the ability of glaze to alter the surface smoothness of a ceramic are relevant, as they are used for finishing these prosthetic restorations along with stains. [9,11].

In a previous study, Oliveira et al. [12] analyzed the optical properties and roughness of zirconia after immersion in a beverage. The results demonstrated that wine significantly altered the translucency and color but did not affect surface roughness. Only one study assessed the color stability of lithium silicate specimens containing zirconia and lithium disilicate, which were immersed in coffee and black tea after receiving surface characterization (stain and glaze). The specimens with extrinsic characterization showed greater color stability compared to those that were only polished [13]. Despite extensive research on discoloration of restorative materials, studies correlating color aging with artificially stained and glazed ceramics represent a gap in literature. Therefore, understanding the behavior and longevity of this characterization process (stain and glaze) when exposed to daily food coloring is valuable, as certain pigments might tend to alter the optical parameters of a restorative material even if it remains intact [12,14-16].

Therefore, understanding the effects of crystallization firing and characterization on the optical properties, such as color and translucency, is crucial to determining their influence on the aesthetic outcome. This study aimed to evaluate the effects of crystallization and characterization on the optical parameters and surface roughness of a zirconia-reinforced lithium silicate glass ceramic, as well as its effects after aging in red wine. The tested hypotheses were: (1) color and translucency will change after characterization; (2) surface alterations will occur after the application of the characterization layer; and (3) color and translucency will change after aging in red wine.

## MATERIAL AND METHODS

The experimental groups are presented in the flowchart in Figure 1.

### Specimens preparation

A2 color shade zirconia-reinforced lithium silicate glass ceramic blocks (Celtra Duo – Dentsply Sirona) were shaped into cylinders and sectioned into 12 mm diameter and 1.2 mm thickness discs using a precision cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA). Each sample was polished with progressively finer silicon carbide paper (220, 300, 400, 600, 800, 1200, 2500#) [5]. The samples were then cleaned in an ultrasonic bath with distilled water for 6 minutes and randomly divided into two groups: without characterization (C) – control group, and characterization (CC).

For the without characterization group, the samples were crystallized in a Vita Vacumat 40 furnace (Vita Zanhfabrik, Germany), following to the manufacturer's recommendations in Chart I. For the characterization group, the samples were crystallized in the same furnace with the same settings as in Chart I. After that, samples received a stain layer (Universal Stain CeramCo color rose – Dentsply Sirona) and were fired according to the manufacturer's recommendations in Chart II. To complete the characterization group, the samples received a glaze layer (Pigmento DS Universal Overglaze High Flu) and were subjected to another firing following the manufacturer's recommendations in Chart II.

**Chart I -** Crystallization Firing (Celtra Duo)

| Temperature and Heating Rate | |
|---|---|
| Initial chamber temperature | 400° |
| Closing time | 8 min |
| Temperature rate increase | 55°C/min |
| Crystallization temperature | 830° |
| Holding time | 10 min |
| Ending time | 700°C |

**Chart II -** Characterization Firing (stain and glaze)

| Temperature and Heating Rate | |
|---|---|
| Initial chamber temperature | 500° |
| Closing time | 2 min |
| Temperature rate increase | 55°C/min |
| Crystallization temperature | 820° |
| Holding time | 1 min 30 s |
| Ending time | 3 min |

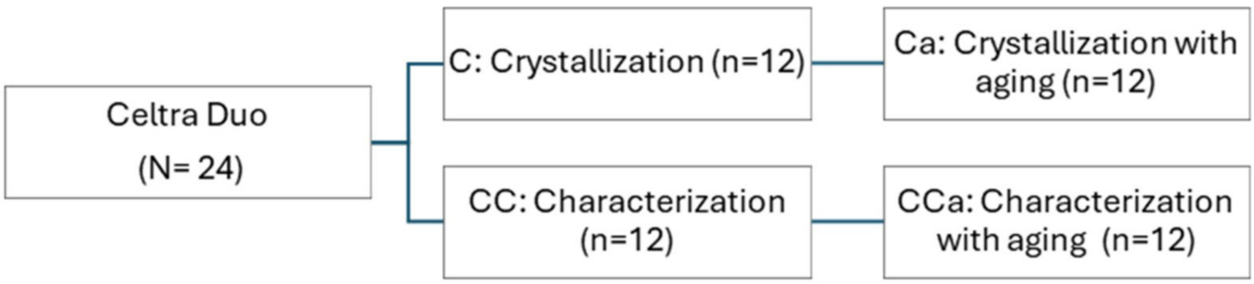


**Figure 1 -** Experimental groups.

### Surface roughness evaluation

Surface roughness (Ra) was evaluated using a Surftest 211 roughness tester, series 178 (Mitutoyo, Suzano, São Paulo, Brazil). Each sample underwent three measurements, after which the mean was calculated. The parameters used for the roughness analysis were ISO 1997, 6.0 mm traverse length, 800 $\mu$m range, Gaussian filter, 0.8 mm cut-off λc, and a speed of 0.5 mm/s.

### Color measurement

The color evaluation of the material was conducted using a spectrophotometer (VITA Easyshade Advance 4.0) with a white background. All samples were assessed before and after each treatment using the CIE Lab* color system (Commission Internationale de l'Eclairage - CIE). The CIE Lab* color system comprises three coordinates that define color: L*, a*, and b*. These coordinates are: L*, which indicates the lightness of the object, ranging from black to white (0 = black, 100 = white); a*, which measures the chroma along the red-green axis (-120 = green, +120 = red); and b*, which measures the chroma along the yellow-blue axis (-120 = blue, +120 = yellow). Color differences were determined by numerical comparison for samples in the no crystallization, crystallization, and characterization groups using Delta (Δ) values. The Deltas for L* (ΔL), a* (Δa), and b* (Δb) can be negative (-) or positive (+), and the total difference is represented by Delta E (ΔE). ΔL* indicates the difference in lightness (+ = lighter, - = darker); Δa* indicates the difference in red and green (+ = more red, - = more green); Δb* indicates the difference in yellow and blue (+ = more yellow, - = more blue); ΔE* represents the total color difference. For each specimen, in a temperature- and humidity-controlled room with daylight, color coordinates were measured three times on each background, which were then averaged.

The ΔE* was calculated by comparing the different experimental groups. Color changes ($\Delta E_{00}$) for all phases were calculated using the CIEDE2000 formula:

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \cdot \left(\frac{\Delta C'}{k_C S_C}\right) \cdot \left(\frac{\Delta H'}{k_H S_H}\right)} \quad (1)$$

### Measurement of translucency

All samples from each group (n=12) were measured for translucency using the spectrophotometer (VITA Easyshade Advance 4.0) and color measurement system. An optical contact, polyethylene glycol 400 (LabSynth, São Paulo, SP, Brazil), was used between the specimen and the black or white background. This optical contact had a refractive index of 1.47, similar to tooth [14]. The device was set to take three consecutive readings, which automatically calculate the average values of L*, a*, and b*. Translucency was evaluated using the translucency parameter (TP), which is the color change (ΔEab) between the readings on the white and black backgrounds. This difference was calculated using the following equation:

$$TP = \begin{bmatrix} (Lp^* - Lb^*)^2 + \\ (ap^* - ab^*)^2 + \\ (bp^* - bb^*)^2 \end{bmatrix}^{1/2}. \quad (2)$$

The subscript letters "p" refers to the color coordinates on the black background, and the subscript letters "b" refer to those on the white background.

### Aging of samples in red wine

After all initial tests, all specimens were immersed in the wine (Salton Tinto Chalise, Vinícola Salton S.A) for 15 minutes per day at 37°C for 30 days [15]. After immersion, the specimens were rinsed with distilled water, dried with gauze, and stored in distilled water in an incubator at 37 ± 1°C, 5% $CO_2$ for 24 hours, and 100% humidity until the next immersion. After aging, all tests were repeated.

### Statistical analysis

The data of roughness, color, and translucency were tabulated and subjected to normality and homoscedasticity tests (Shapiro-Wilk and Levene's test). The power of the sample was estimated with the website www.openepi.com with 95% confidence interval. Roughness data and translucency data were analyzed using two-way ANOVA and Tukey's test at a 5% significance level. Color data were analyzed using the Kruskal-Wallis test followed by Dunn's test.

**Table I -** Mean (±SD) of surface roughness (Ra) found for the groups in μm

| crystallization and characterization | Aging | |
|---|---|---|
| | Without | With |
| C | 0.03 ± 0.01 $^{B,b}$ | 0.03 ± 0.01 $^{B,b}$ |
| CC | 0.80 ± 0.23 $^{A,a}$ | 0.71 ± 0.24 $^{A,a}$ |

Different uppercase letters indicate statistically significant differences between rows, while different lowercase letters indicate statistically significant differences between columns. Tukey's test, 5%.

**Table II -** Mean (±SD) of color differences (ΔE00) found for the groups

| crystallization and characterization | Aging | |
|---|---|---|
| | Without | With |
| C | 0.0 $^{B,b}$ | 3.94 ± 1.69 $^{B,b}$ |
| CC | 16.76 ± 2.78 $^{A,a}$ | 14.17 ± 2.42 $^{A,a}$ |

Different uppercase letters indicate statistically significant differences between rows, while different lowercase letters indicate statistically significant differences between columns. Dunn's test.

**Table III -** Mean of translucency data (TP00) found for the groups

| crystallization and characterization | Aging | |
|---|---|---|
| | Without | With |
| C | 14.40 ± 4.95 $^{A,a}$ | 15.90 ± 5.52 $^{A,a}$ |
| CC | 8.66 ± 3.45 $^{B,b}$ | 8.75 ± 3.41 $^{B,b}$ |

Different uppercase letters indicate a statistically significant difference between rows, and different lowercase letters indicate a statistically significant difference between columns. Tukey's test, 5%.

## RESULTS

### Surface roughness

The power of sample reported was 100%. Two-way ANOVA revealed that the factor "characterization" was statistically significant ($p < 0.001$) for surface roughness (Ra), but the factor "aging" the interaction between them were not significant ($p > 0.05$). When comparing the groups (Table I), Tukey 5% test indicated that the characterization group exhibited the highest roughness values, both with and without aging, differing significantly from the other groups. The groups without characterization were statistically similar, both with and without aging (C and Ca).

### Color analysis

The power of sample reported was 100%. Kruskal-Wallis revealed a statistical influence for the "characterization" factor ($p < 0.001$) and no influence for the "aging" factor for color variation (ΔE00); significant differences were found for the interaction between the factors ($p < 0.05$). When comparing the groups (Table II), Dunn's test showed that the characterization group (CC) exhibited the highest color variation, both with and without aging. The group without characterization (C) showed the least color variation among the aging groups.

### Translucency analysis

The power of sample reported was 93,37%. Two-way ANOVA showed a statistical influence for the "characterization" factor ($p < 0.001$); however, no influence was observed for the "aging" factor or for the interaction between the factors ($p > 0.05$). The Tukey 5% test revealed that the group without characterization, both with and without aging, presented the highest translucency values (Table III).

## DISCUSSION

This study aimed to evaluate the effect of crystallization and characterization on the optical parameters and surface roughness of a zirconium-infiltrated lithium silicate glass ceramic (Celtra Duo – Dentsply Sirona), with and without aging in red wine. Understanding the optical properties, such as color stability and translucency, of a commercially available ceramic restorative material, as well as topographic analyses, is important because it allows for the understanding and prediction of behavior when applied to functional demands [16,17], guiding the indication for use with a view to the longevity of the treatment outcome and patient satisfaction in aesthetic terms [1].

The first hypothesis, stating that there would be changes in color and translucency after characterization, was accepted. The results showed a color change in the characterization group (CC) before and after aging in wine. This result is consistent with previous studies, in which greater color variation was observed after characterization with ceramic stain compared to samples that were only crystallized [18]. This can be attributed to the polychromy of pigments obtained through different concentrations of metal salts and oxides, which form the basis for stain formulation and color development, being responsible for altering the original surface color of the ceramic where they are applied [19]. This study observed that the CC group exhibited the greatest color variation and the lowest level of translucency. The study conducted by Eldwakhly et al. [17] on staining in different ceramics established an inversely proportional relationship between color variation and translucency, which corroborates with the present study. The authors explained this negative correlation by suggesting that color variation, due to changes in particle clustering, water or pigment absorption, and surface alterations, would also be accompanied by changes in a material's light scattering properties, such as its degree of translucency.

Regarding surface characteristics, these also influence optical properties, as a smoother surface allows the angle of light incidence to more closely match the angle of reflection. In contrast, on rough surfaces, light is incident and reflected at different angles [20]. Therefore, a surface topography analysis was performed to evaluate surface roughness (Ra). The results showed a significant increase in roughness in the CC group, consistent with the study by Habib et al. [18], which compared the behavior of different ceramic stains (VITA and Ivoclar) and attributed the increased roughness to the stains' composition. These stains contain multiple colored oxide pigments, including Fe, Zn, Sn, Al, Co, and Cu, incorporated into low-fusion glass [19]. During the characterization firing process, the dissolution of silica and the subsequent loss of ions may have compromised the surface smoothness previously achieved by polishing. These findings support the second hypothesis, confirming that surface alterations occur in the material following the application of the characterization layer.

The application of glaze is a common surface finishing procedure for ceramic restorations [21]. It is known that the surface characteristics of a material affect its optical behavior. Kilinc and Turgut [22] observed that manual polishing helped reduce surface roughness and maintained the color stability of different ceramics. In the study by Vichi et al. [21], different finishing methods for ceramic materials—zirconia-reinforced lithium silicate (VITA Suprinity) and lithium disilicate (IPS e.max CAD)—were investigated. Each ceramic group was divided into subgroups according to the finishing technique, which included mechanical polishing using dedicated ceramic polishing systems for 30 or 60 seconds, as well as glaze application either by brush or spray. The authors observed that there was a significant difference only in the application method, and the brush application in zirconia-reinforced lithium silicate —also the method used in the present study—resulted in a more uniform application and smoother glaze coat. This improved uniformity may have contributed to a more consistent superficial refractive index, thereby minimizing light scattering at the surface. Consequently, it is suggested that the effectiveness of glaze application may depend on the operator's technique, highlighting the importance of applying the glaze evenly to optimize the material's optical properties. In another study by Kurt et al. [9], the application of glaze increased roughness and decreased translucency, which was also observed in the present study when comparing the C and CC groups. This inverse correlation between translucency and roughness was established in the present study and in several others [9,17,20], demonstrating that higher roughness levels are associated with lower translucency, although the converse is not always proven. In the present study, an increase in roughness was observed with the addition of the characterization layer (stain and glaze), and both glaze application and the combined factors also tended to reduce surface smoothness in previous studies [9,18]. Thus, we hypothesize that both stain and glaze tend to alter the surface topography, making it less smooth. Although this may increase surface roughness, the clinical application of stain and glaze is essential for achieving aesthetic outcomes, as it allows the restoration to match the shade and appearance of adjacent teeth. Therefore, despite the impact on surface smoothness, their use remains justified to ensure visual harmony within the oral environment.

The third hypothesis, which proposed that aging in wine would alter both color and translucency, was rejected, as no statistically significant differences were observed in these parameters. A staining scenario was simulated by immersing the samples in red wine for 15 minutes daily over 30 days [15]. The tests showed no statistically significant color variation across all groups, which contrasts with the findings of Eldwakhly et al. [17]. In their study, crystallized Celtra Duo and other ceramics were exposed to staining agents such as coffee, Coca-Cola, and ginger, resulting in noticeable color changes. This effect was attributed to the acidity of these substances, which react with ceramic components, leading to increased infiltration. This analysis is particularly relevant, as the consumption of various foods and beverages—and the consequent exposure to food pigments and pH variations—can impact the aesthetic properties of restorations over time [23], potentially affecting treatment longevity and patient satisfaction. In the present study, although the non-characterized group exhibited color variation before and after aging, the difference was not statistically significant. This aligns with the findings of Choi et al. [24], who reported that ultraviolet aging did not significantly affect the optical properties of Celtra Duo ceramic. However, in our study, the mean color variation ($\Delta E00$) after aging was 3.94, which, according to previous studies, is clinically unacceptable [25,26] as variations exceeding 3.3 are perceptible [27]. Given that the aging process in this study involved immersion in wine with an acidic pH (pH = 3.55), we hypothesize that the observed color variation is likely attributed to this acidity. This aligns with the findings of Rodrigues et al. [15], who conducted similar staining experiments on resin materials. A significant gap exists in the literature regarding the behavior of ceramics characterized by stains and subjected to artificial aging, making it challenging to compare our results with previous research.

While this study contributes to the understanding of the effects of crystallization and characterization processes on the optical properties and roughness of a zirconia-reinforced lithium silicate, further research is needed to correlate these processes with different ceramic stain colors and to assess color stability after aging in various substances with differing pH levels, such as soft drinks, coffee, and acidic juices. Additionally, since this is an in vitro study, clinical follow-ups evaluating the stability of optical properties and surface changes of this restorative material will provide further insight into the material's limitations, benefits, and clinical applications.

## CONCLUSION

Based on our results, we conclude that the characterization process influenced color and translucency variations in zirconia-reinforced lithium silicate. Regarding surface topography, characterization led to an increase in surface roughness, both before and after aging. Additionally, aging in red wine did not cause a statistically significant change in color or translucency among the groups. However, it did lead to a color change that may be noticeable in a clinical setting.

## Author's Contributions

NRR, TJAPJ: Conceptualization. LVFT, KBS, LALB, AMD, NRR, TJAPJ: Methodology. KBS, AMD: Formal Analysis. LVFT, LALB: Investigation. TJAPJ: Resources. LVFT, KBS: Data Curation. LVFT, KBS: Writing – Original Draft Preparation. KBS, AMD, TJAPJ: Writing – Review & Editing. LVFT, KBS, LALB: Visualization. TJAPJ: Supervision. KBS, LALB: Project Administration. LVFT, TJAPJ: Funding Acquisition.

## Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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## Regulatory Statement

This study did not involve the use of participants, data, human tissue, or other animals. Therefore, ethical approval and informed consent were not required.

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