



# *Article* **Self-Etching Ceramic Primer Application Time Influences Durability of Bonding to Lithium Disilicate and Advanced Lithium Disilicate Ceramics**

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**Abstract:** Background: Monobond Etch & Prime (MEP; Ivoclar) has a material-dependent effect on dental ceramics. This study evaluated the impact of MEP application time on the resin–ceramic shear bond strength (SBS) of lithium disilicate (LD) and advanced lithium disilicate (ALD) ceramics. Methods: LD and ALD discs were distributed into four groups ( $n = 20$ ): no surface treatment; 5% HF etching followed by the application of a silane-containing primer (S); MEP applied for 40 s and maintained for 20 s; MEP applied for 2 min and maintained for 40 s. Resin–ceramic SBS was evaluated at 24 h and after thermocycling. The statistical analysis of SBS was performed by using the three-way ANOVA and Tukey's multiple comparisons tests. Additional ceramic samples were prepared to assess the surface topography. Results: There was no statistically significant difference in the SBS of the MEP groups (20 s and 2 min) and HF + S. However, only the HF + S and MEP (2 min) groups showed durable adhesion after thermocycling. MEP resulted in less distinct surface alterations compared with HF etching. Conclusion: Increasing the application time of MEP to 2 min resulted in more durable resin–ceramic adhesion compared with the application time (40 s) recommended by the manufacturer. The adhesion of resin–ceramic to LD and ALD did not significantly differ.

**Keywords:** ceramic; adhesion; etching; priming

#### **1. Introduction**

Several ceramic materials satisfy patients' and dentists' increasing expectations for esthetic restorations [\[1\]](#page-9-0). With the development of computer-assisted design (CAD)/computerassisted manufacturing (CAM) technology, which has improved in strength and marginal fit, dental ceramics have become more popular as restorative materials [\[2\]](#page-9-1). Currently, zirconia is one of the most common ceramic restorative materials, along with lithium disilicate ceramic (LD), which has higher translucency and lower mechanical strength than zirconia. LD is a glass-matrix ceramic that is composed of a matrix of silicon dioxide, generally silica or quartz, in which additional crystals are incorporated. The glassy matrix alone does not have sufficient strength to resist defects; therefore, to increase the strength and improve the mechanical properties, dispersion strengthening is usually required [\[3\]](#page-9-2). Dentsply Sirona has introduced the advanced lithium disilicate (ALD) CAD/CAM blocks, a new material for anterior and posterior CAD/CAM-fabricated restorations. ALD ceramic is characterized by its strength and highly esthetic nature. This type of ceramic has a better wear behavior than lithium disilicate [\[4\]](#page-9-3). ALD is composed of lithium disilicate crystals incorporated into a glassy matrix with lithium aluminosilicate [\[4\]](#page-9-3). LD is an etchable ceramic because of its glass-matrix content. Hydrofluoric acid (HF) etching is recommended as a surface treatment that can alter the surface morphology, increasing the surface roughness of LD and rendering the LD substrate more favorable for resin adhesion [\[5\]](#page-9-4). However, the toxicity



**Citation:** Alsolami, A.; Alzahrani, K.M. Self-Etching Ceramic Primer Application Time Influences Durability of Bonding to Lithium Disilicate and Advanced Lithium Disilicate Ceramics. *Coatings* **2024**, *14*, 1461. [https://doi.org/10.3390/](https://doi.org/10.3390/coatings14111461) [coatings14111461](https://doi.org/10.3390/coatings14111461)

Academic Editors: Lavinia Cosmina Ardelean and Laura-Cristina Rusu

Received: 22 September 2024 Revised: 10 November 2024 Accepted: 14 November 2024 Published: 17 November 2024



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of HF is well documented and known [\[6](#page-9-5)[,7\]](#page-9-6). In addition, considering the aggressiveness of HF, prolonged etching might deteriorate the mechanical properties of LD restorations. HF etching is followed by a deactivation step and/or a rinsing step. The inadequate cleaning of the etching residue can impair resin–ceramic adhesion. Thus, several alternative ceramic surface treatments have been attempted [\[8](#page-9-7)[–10\]](#page-9-8). However, none of these have yielded comparable results to HF etching. Despite the altered surface topography of HF-etched LD, a priming step using a silane-containing primer (S) is essential to promoting bonding between the resin cement and LD surface. Silane is a bifunctional monomer that bonds to the ceramic surface via its silanol group and to resin cements via its methacrylate group [\[11\]](#page-9-9). Silane primers contain large amounts of organic solvents; thus, an efficient air-drying step is required to evaporate such solvents [\[12\]](#page-9-10). Otherwise, a loose silane layer can be formed on the LD surface, resulting in inferior bonding [\[13\]](#page-9-11). This drying step can be controversial for some clinicians, particularly for restorations of complex shape and geometry [\[14\]](#page-9-12). In an attempt to avoid clinical errors associated with HF etching and silane priming steps, Monobond Etch & Prime, Ivoclar (MEP), a self-etching ceramic primer, has been introduced as a simplified approach that combines the etching effect and silane priming capacity in one step [\[13](#page-9-11)[,15\]](#page-9-13). MEP is composed of a tetrabutylammonium dihydrogen trifluoride (TADF) as an etchant, silane methacrylate, methacrylate phosphate monomer, alcohol and water as solvents, and colorant as a pigment to enhance its visibility [\[16\]](#page-9-14). Several studies have assessed the influence of MEP on adhesion to dental ceramics. It was found that the etching (application) duration of MEP might influence the obtained resin–ceramic adhesion. Up until now, the effect of MEP on the surface topography and adhesion of resin to advanced LD has not been investigated yet. This study involved the evaluation of the resin–ceramic shear bond strength (SBS) of LD and advanced LD following MEP surface treatment. The hypothesis were as follows: (1) there would be no significant difference in SBS between lithium disilicate and advanced lithium disilicate materials; (2) there would be no significant differences in SBS between the use of 5% HF followed by the application of S and that of MEP; (3) there would be no effect of increasing the application time of the self-etching ceramic primer (MEP) on SBS; (4) there would be no significant difference in SBS before thermocycling at 24 h and after 10 k cycles of thermocycling.

#### **2. Materials and Methods**

#### *2.1. Specimen Preparation*

Lithium disilicate and advanced lithium disilicate ceramic blocks (Table [1\)](#page-2-0) were sectioned under running water by using a low-speed prevision cutting saw (Isomet Low Speed, Buehler, Lake Bluff, IL, USA) into a total of 168 smaller blocks (Figure [1\)](#page-2-1). The dimensions of each prepared block were approximately 6 mm  $\times$  6 mm  $\times$  3 mm. This was followed by an ultrasonic cleaning step for 5 min in distilled water (Sonicer, Yoshida Dental Manufacturing, Tokyo, Japan) and an air-drying step. The IPS e.max CAD and CEREC Tessera polished blocks were sintered and glazed and then embedded in self-curing orthodontic resin. Figure [1](#page-2-1) shows a flowchart of the study design. The samples were cleaned, polished under running water by using 600-grit silicon carbide polishing paper and a grinder–polisher machine (MetaServ® 250 grinder-polisher machine; Buehler, Lake Bluff, IL, USA) at 150 rpm, ultrasonically cleaned as described above, and air-dried.



<span id="page-2-1"></span><span id="page-2-0"></span>**Table 1.** The materials utilized and their composition.

**Figure 1.** Study design. **Figure 1.** Study design.

# **Table 1.** The materials utilized and their composition. *2.2. Surface Treatment*

The blocks of each ceramic material were allocated to a total of four groups (n = 20 per  $\mathcal{S}$  -1 material) based on the following protocol:

Group 1 (NT): No surface treatment was performed.

 $\sum_{i=1}^{n}$ instructions. HF was applied for 20 s for LD and 30 s for ALD and rinsed thoroughly. One Group 2 (HF + S): Surface treatment with 5% HF etching following the manufacturer's coat of a silane-containing universal primer (S) was applied to the etched ceramic surface, left to react for 1 min according to the manufacturer's instructions, and then thoroughly air-dried.

Group 3 (MEP 20 s + 40 s): MEP was applied following the manufacturer 's instructions (rubbed for 20 s and left on the surface for 40 s), rinsed thoroughly with water, and then air dried for 10 s. air-dried for 10 s.

Group 4 (MEP 2 min + 40 s): MEP was applied with modifications with respect to the manufacturer's instructions (rubbed for 2 min and left on the surface for 40 s), rinsed thoroughly with water, and then air-dried for 10 s.

A silicon mold was used for building cylinders (3 mm in diameter and 2 mm in height) of a dual-cure resin cement (Variolink esthetic DC, Ivoclar). While the mold was placed, the light curing of the resin cement was performed for 1 min by using a light-curing device  $(Elipar<sup>TM</sup> S10; 3M ESPE, St. Paul, MN, USA)$  operated at an intensity of approximately 1100 mW/cm<sup>2</sup>. Additional light curing for 1 min was performed at approximately 1 mm after the silicone mold was carefully removed. A total of 10 specimens of each group were tested for resin–ceramic SBS at 24 h. The other 10 samples of each group were artificially aged through 10k thermal cycles by using a thermocycler machine (THE-1100; SD Mechatronik GmbH, Feldkirchen-Westerham, Germany). Each cycle involved fully dipping the specimens in a distilled water bath at a temperature of  $5 \pm 1$  °C for 30 s followed by fully dipping them in another water bath at a temperature of 55  $\pm$  1 °C for 30 s each, with a 5 s transfer time between the two baths.

#### *2.3. Evaluation of Resin–Ceramic SBS*

The cross-sectional area of the resin–ceramic interface was determined by using a digital caliper with a surface area range of  $3 \pm 0.03$  mm. A stainless steel chisel connected to a universal testing machine (Instron 5965; Instron Corporation, Canton, MA, USA) equipped with a load cell of 1 kN was utilized to shear the specimens at the resin–ceramic interface until failure. The cross-speed was 0.5 mm/min. SBS was obtained by diving the load reported at specimen failure by the premeasured resin cement cross-sectional surface area.

#### *2.4. Failure Mode Analysis*

All the tested specimens were investigated by using a digital microscope (Hirox Co., Ltd., Tokyo, Japan) at  $\times$ 50 magnification to determine the failure mode patterns that had occurred. The failure modes were classified as adhesive (type 1), with specimen failure (debonding) at the resin–ceramic interface; cohesive in the ceramic (type 2), in which the failure (fracture) had occurred within the ceramic material (substrate) with no adhesive failure; cohesive in the resin cement (type 3), with whole specimen failure (fractured) within the resin cement build-up with no adhesive failure; and mixed failure (type 4), which combined both adhesive and cohesive failure. Samples that presented failure before testing (pre-test failures) were recorded and accounted as 0 MPa in the statistical analysis.

#### *2.5. Statistical Analysis*

The SBS values were checked for data normality by using the Shapiro–Wilk statistical test. The effect of the three independent variables identified (ceramic material, surface treatment, and thermocycling), as well as their interactions, on resin–ceramic SBS was statistically analyzed by using three-way analysis of variance (ANOVA) test. Further, Tukey's multiple comparisons test was used for the inter-group comparisons. The distribution of failure modes among the groups was determined by using a chi-squared test.

#### *2.6. Assessment of Surface Topography*

Additional ceramic samples were prepared and treated as described for SBS evaluation except for the silane priming step. Ceramic samples were cleaned and then dehydrated in ascending concentrations of ethanol. Dried ceramic samples were fixed on the sample stub with the treated surface facing upwards and then sputter-coated with gold by using a sputter coater (Q150RS; Quorum Technologies, Laughton, East Sussex, UK). The ceramic samples were observed by using a scanning electron microscope (SEM) (EVO LS10; Carl Zeiss SMT, Cambridge, UK) at  $10,000 \times$  magnification operated at 15 kV EHT voltage.

## **3. Results**

*3.1. SBS*

The SBS data showed a normal distribution ( $p > 0.05$ ). The SBS results are illustrated in Figure [2.](#page-4-0) The results of the three-way ANOVA are illustrated in Table [2.](#page-4-1)

 $\blacksquare$  10 k TC

 $\blacksquare$  24 h

<span id="page-4-0"></span>

**Figure 2.** Bar chart of mean and standard deviation of resin–ceramic shear bond strength (SBS) lowing different surface treatments of lithium disilicate (LD) and advanced lithium disilicate (ALD) following different surface treatments of lithium disilicate (LD) and advanced lithium disilicate (ALD) ceramics. Different letters indicate statistically significant differences between the groups. \* indicates ceramics. Different letters indicate statistically significant differences between the groups. \* indicates a statistically significant difference between immediate (24 h) and aged (TC) bond strength. a statistically significant difference between immediate (24 h) and aged (TC) bond strength.

<span id="page-4-1"></span>



\* indicates a statistically significant difference.

different storage conditions (24 h vs. 10k thermocycling) (*p*-value = 0.000). The interactions There was no significant difference in SBS between lithium disilicate and advanced lithium disilicate materials in all surface treatment groups at 24 h and after thermocycling<br>
end a FeT M  $(p$ -value = 0.587). However, there was a statistical difference among different surface treatments and different storage conditions (24 h vs. 10k thermocycling) (*p*-value = 0.000). The interactions among study factors (material, surface treatment, and thermocycling) were tested, which showed that there were no interactions among the test factors. When comparing surface

treatments, both MEP and HF + S (groups 2, 3, and 4) presented significantly increased SBS compared with group 1 (no surface treatment) at 24 h and after thermocycling (*p*-value = 0.000). However, HF + S showed comparable SBS to MEP in both materials (*p*-value > 0.05). Group 4 (MEP 2 min  $+40$  s) showed relatively higher SBS than group 3 (MEP 20 s  $+40$  s) in both materials, regardless of the evaluation time; however, the difference was not statically significant (*p*-value > 0.05). Regarding the storage condition, thermocycling significantly decreased SBS for most of the surface treatment groups ( $p < 0.05$ ), but in group 2 and group 4, the difference was not statistically significant (*p* > 0.05) for both materials. compared with group 1 (no surface treatment) at 24 h and after the <sup>24</sup> h and after the <sup>24</sup> h and after the <sup>24</sup>

# *3.2. Failure Mode Analysis*  $\overline{\phantom{a}}$

The distribution of failure modes in terms of frequency and percentage is shown in Table [3.](#page-5-0) Cohesive failure within resin (Figure [3B](#page-5-1)) and mixed failures (Figure [3C](#page-5-1)) were the most common among surface treatment groups, whereas the adhesive failure type (Figure [3A](#page-5-1)) and pre-test failure were most common in the control group (group 1). However, the failure mode cohesive in ceramic was not detected in any of the study samples. Pre-test failures occurred in the control group. rial distribution of tandic modes in terms of nequency and percentage is shown

![](_page_5_Picture_442.jpeg)

<span id="page-5-0"></span>**Table 3.** The distribution of failure modes among study groups.

<span id="page-5-1"></span>LD: lithium disilicate ceramic; ALD: advanced lithium disilicate ceramic; TC: thermocycling. LD: lithium disilicate ceramic; ALD: advanced lithium disilicate ceramic; TC: thermocycling.

![](_page_5_Figure_7.jpeg)

Figure 3. Representative images of different failure modes examined under digital microscope (A–C): **C**): (**A**) adhesive failure, (**B**) cohesive failure within resin, and (**C**) mixed failure. (i) Resin–ceramic (A) adhesive failure, (B) cohesive failure within resin, and (C) mixed failure. (i) Resin–ceramic interface, (re) resin cement, and (ce) ceramic surface.

## *3.3. Surface Topography*

Application of either HF or MEP yielded distinct surface topographic changes in both the ceramic materials compared with no treatment, which showed a smooth, more homogenous surface texture without any craters or pits in all ceramic materials (Figure [4\)](#page-6-0). A more prominent etching pattern with micro-porosities, grooves, and striations was observed with HF application due to the dissolution of the slightly larger vitreous matrix in both materials compared with the MEP surface treatment in both groups 3 and 4, which showed a milder etching pattern and limited change in roughness. Increasing the application time to 2 min in group 4 (MEP 2 min +40 s) led to an increase in micro-porosities on the ceramic surface and more topographic changes, resulting in a slightly more prominent etching pattern compared with group 3 (MEP 20 s+ 40 s).

<span id="page-6-0"></span>![](_page_6_Figure_4.jpeg)

**Figure 4.** Representative SEM micrographs (10,000×) of lithium disilicate and advanced lithium disilicate: (a,e) control (no treatment); (b,f) hydrofluoric acid etching; (c,g) Monobond Etch & Prime (MEP) applied following the manufacturer's instructions (rubbed for 20 s and left on ceramic surface extended application time (rubbed for 2 min and left on ceramic surface for 40 s). for 40 s); (**d**,**h**) MEP extended application time (rubbed for 2 min and left on ceramic surface for 40 s).

### **4. Discussion**

This laboratory study tested the impact of MEP and HF on the surface topography and resin–ceramic adhesion of two ceramic materials. Although the correlation between adhesion strength studies and clinical performance of ceramic restorations is still debatable, in vitro studies can provide a reliable evaluation of new adhesive systems and/or techniques using standardized testing conditions that cannot be provided by clinical studies [\[17\]](#page-9-15). The SBS test utilized in this study has been extensively used due to uncomplicated specimen preparation. In addition, bonded resin–ceramic specimens do not necessitate additional preparation, i.e., micro-sectioning, which might influence the resin–ceramic interface. However, this method might be associated with inhomogeneous stress distribution at the resin–ceramic interface [\[18\]](#page-9-16). A dual-cure resin cement was applied to the ceramic materials in all the study groups. Although constructing the resin cement cylinders might not be the main clinical application of the resin cement used, this study followed the protocol applied in previous similar studies [\[19,](#page-9-17)[20\]](#page-10-0). The SBS results of the current study showed no statistically significant differences between the two ceramic materials tested. Thus, the first null hypothesis of this study was accepted. This can be explained by the similar amount of glassy matrix in both ceramic materials which reacts with the ceramic etchant (HF) or MEP. Different surface treatments, including MEP and HF, followed by S priming were evaluated in this study. The second null hypothesis was accepted, since there was no statistically significant difference between the experimental groups (HF +S and MEP). Resin–ceramic adhesion relies on two main aspects. First, micromechanical retention of resin-based cement or inside micro irregularities created by the dissolution of silica matrix. Second, a chemical bond (adhesion) between the ceramic surface and methacrylate-based resin cement must be achieved through a silane priming step. The lack of both aspects well explains the reduced SBS means of the control group [\[21\]](#page-10-1). Interestingly, there was no difference between the HF and the MEP groups (groups 2, 3, and 4). Such findings come in accordance with those of other studies that showed comparable resin–ceramic adhesion following MEP application and 5% HF. The application and reaction times of MEP reported in the literature varies between 20 and 60 s for application time and between 40 s and 10 min for reaction time [\[13\]](#page-9-11). In the present study, increasing the application time of MEP to 2 min followed by 40 s of reaction time (group 4) did not significantly affect SBS. Therefore, the third null hypothesis was accepted. However, increasing the application time enhanced the durability of the resin–ceramic bond strength compared with that achieved by the 20 s application time, which significantly deteriorated after artificial aging by TC. A previous study reported similar results to our study, with no significant difference in SBS when MEP was applied either following the manufacturer's instructions (20 s of application and 40 s of reaction time) or by increasing the application and reaction times to 40 s and 80 s, respectively [\[22\]](#page-10-2). Another study by Alshihri et al. reported no significant differences in SBS between MEP-etched samples with standardized application time (20 s) and various reaction times (40, 80, and 120 s) [\[23\]](#page-10-3). However, this contradicts with the results of a previous study in which higher bond strength was associated with MEP ceramic surface treatment compared with HF when the application time of MEP was increased to 60 s and 40 s of reaction time. The variation in the results could relate to the different bond strength tests used, i.e., the macro-shear bond test used in the present study as opposed to the micro-shear test used in the study by Cardenas et al. [\[24\]](#page-10-4).

Various techniques have been utilized in in vitro studies to simulate the physiological aging of adhesively bonded material, including water storage and thermocycling. Thermocycling involves subjecting samples to alternating temperatures (usually 5 and  $55 \degree C$ ) to simulate thermal changes that occur in the oral cavity. The interfacial stresses generated by temperature alterations can negatively impact resin–ceramic adhesion [\[25\]](#page-10-5). A total of 10 k thermal cycles were applied in this study. This might be equal to 1 year of clinical function [\[26\]](#page-10-6). Thermocycling caused a significant decrease in SBS in most of the treatment groups, except for HF for both materials and MEP 2 min + 40 s for lithium disilicate. Thus, the fourth null hypothesis was partially rejected. This coincides with the

results of a recent study which indicated stable adhesion to MEP-treated glass ceramic after thermocycling [\[27\]](#page-10-7). Similarly, Azevedo et al. reported stable SBS with glass-ceramic samples treated with 5% HF for 20 s over 16 months of water storage [\[28\]](#page-10-8).

The SEM results indicate almost no surface alterations in group 1 (no treatment) (Figure [4\)](#page-6-0) compared with the other groups. This could account for the low mean bond strength and frequent pre-test failure of this group. Group 2 produced the highest dissolution of the matrix, showing a porous and rough surface (Figure [4,](#page-6-0) HF). This could explain the high SBS and stable bond observed after thermocycling. The MEP surface treatment, according to the SEM analysis, resulted in a milder etching pattern and less distinct topographical changes compared with HF etching. This could be a result of the partial dissolution of the glass-ceramic matrix, which could have affected the durability and resulted in a significant decrease in bond strength after thermocycling [\[24\]](#page-10-4). The selfetching ceramic primer uses ammonium polyfluoride and trimethoxypropyl methacrylate for etching and priming, respectively. Ammonium polyfluoride is frequently used to etch glass-based ceramics, and the lower acidity of this etchant could explain the milder etching pattern and limited changes in surface topography observed (Figure [4\)](#page-6-0) [\[29\]](#page-10-9). However, more distinct surface topographic changes were noticed with extending the MEP application time to 2 min. This provides a plausible explanation for the durable adhesion of the MEP 2 min group compared with the 20 s group. On the other hand, the phosphate ester monomers of the universal primer or MEP utilized might make a minimal contribution to the adhesion of resin to glass ceramics [\[30\]](#page-10-10).

Failure mode analyses can offer important information for interpreting bond strength results and identifying weaknesses in testing procedures. Although the incidence of frequent cohesive failures within resin cement might be an indicator of inhomogeneous stress distribution during testing and can be considered a limitation of the testing procedure, omitting the bond strength values associated with cohesive failures can lead to biased conclusions [\[31\]](#page-10-11). This information can then be used to increase the reliability of these methodologies to ensure that the results accurately reflect the strengths of these bonds [\[18\]](#page-9-16). The present study showed an association between the failure mode and surface treatment, where the highest percentage of adhesive failure was observed in the no-surface-treatment group, while a high percentage of both cohesive failure and mixed failure was observed in the MEP and HF groups. The high frequency of cohesive and mixed failures in the surface treatment groups could be explained by the robust resin–ceramic adhesion following ceramic surface treatment using MEP or HF followed by silane priming. However, the absence of cohesive failure within ceramics could be due to the mechanical properties of the ceramic materials used [\[32\]](#page-10-12). Despite the promising results of the MEP-treated groups, the outcome of this study should be interpreted considering the limitations of this study, which include the lack of multi-factorial artificial aging utilizing mechanical loading and exposure to oral biofilm and different beverages. One more limitation of this study is that one resin cement was used for build-up on resin–ceramic specimens. Further in-depth chemical characterizations of MEP-primed ceramic surface are required to verify the possible effect of water rinsing on the hydrolysis of the silane molecules.

#### **5. Conclusions**

Within the confines of this in vitro study, it can be concluded that although the selfetching ceramic primer (MEP) resulted in the partial dissolution of the ceramic glass content and a less aggressive ceramic etching pattern compared with HF etching, MEP can be considered an alternative to the protocol including hydrofluoric acid etching and silane priming. Increasing the application time of MEP to 2 min resulted in more durable resin–ceramic adhesion compared with the application time (20 s) recommended by the manufacturer. The strength of the adhesion of resin–ceramic to lithium disilicate and advanced lithium disilicate ceramic did not significantly differ.

**Author Contributions:** Conceptualization, K.M.A.; methodology, A.A. and K.M.A.; software, A.A.; validation, A.A. and K.M.A.; formal analysis, A.A.; investigation, A.A.; resources, K.M.A.; data curation, A.A.; writing—original draft preparation, A.A.; writing—review and editing, K.M.A.; visualization, A.A.; supervision, K.M.A.; project administration, K.M.A.; funding acquisition, K.M.A. All authors have read and agreed to the published version of the manuscript.

**Funding:** This study was supported via funding from Prince Sattam bin Abdulaziz University, project number PSAU/2024/R/1446.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** The data presented in this study are available upon request from the corresponding author.

**Acknowledgments:** This study was supported via funding from Prince Sattam bin Abdulaziz University, project number PSAU/2024/R/1446.

**Conflicts of Interest:** The authors declare no conflicts of interest.

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