Ceramics-Silikáty 69 (1), 93-100 (2025)
www.ceramics-silikaty.cz doi: 110.13168/cs.2024.0068

LITHIUM DISILICATE CERAMICS SURFACE PRE-TREATMENT USING LOW-LEVEL LASER THERAPY-ACTIVATED RIBOFLAVIN, AND Ti: Al_2O_3 LASER ON THE COLOUR CHANGE, SURFACE ROUGHNESS, AND SHEAR BOND STRENGTH TO ADHESIVE CEMENT: AN *in vitro* SEM VALUATION

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Submitted September 6, 2024, accepted October 4, 2024

Keywords: Lithium Disilicate Ceramics, Sandblasting, Titanium Sapphire laser, Low-level laser therapy, Shear bond strength

The present study aimed to examine the effectiveness of surface conditioners sandblasting (SB), Titanium Sapphire (Ti-Sapphire) laser, a low-level laser therapy activated riboflavin photosensitiser (LLLT-RFP) on the colour change (ΔE), surface roughness (Ra) and shear bond strength (SBS) of Lithium Disilicate Ceramic LDC. Eighty LDC discs were prepared and categorised into four different groups based on the pre-treatment. (n = 20) Group 1: HF acid (S), Group 2: SB, Group 3: Ti-sapphire laser Group 4: LLLT activated RFP. Five samples from each group underwent a ΔE assessment using a spectrophotometer. Surface Ra analysis was conducted on five specimens from each group using a profilometer. The bonding of a luting cement to the LDC discs was performed followed by the thermocycling of the specimens. An SBS and failure mode analysis were then performed using a universal testing machine and stereomicroscope, respectively. An analysis of variance was conducted to determine the mean and standard deviation (SD) of the ΔE, Ra, and SBS. Tukey's post hoc multiple comparison test was used for the intergroup comparisons while maintaining a margin of error of 0.05. The highest outcomes of ΔE were witnessed in the Group 1 specimens (HFA(S) + LDC). The minimum score of ΔE was displayed by the Group 4 (LLLT activated RFP) + LDC) specimens. The highest scores of Ra and SBS were detected in Group 3 (Ti: Al_2O_3 laser + LDC). Whereas Group 4 (LLLT activated RFP) + LDC exhibited the lowest Ra scores and bond integrity. The Ti-Sapphire laser proved to be a suitable alternative as it positively influences the mechanical properties (Ra, SBS) of the LDC. However, LDC conditioned with the Ti-Sapphire results in a considerable colour change which limits its application to posterior teeth only.

INTRODUCTION

The use of ceramic restorations has gained considerable attention due to the growing aesthetic demands of dental patients. Lithium disilicate-based ceramics (LDCs) have become the preferred material for indirect restorations, owing to their exceptional fracture toughness and superior aesthetic qualities [1, 2]. To enhance the bond strength between the ceramic and the luting material, various mechanical and chemical surface conditioning techniques have been developed [3]. Among these, hydrofluoric acid (HFA) etching followed by the application of a silane coupling agent is widely regarded as the gold standard, providing favourable outcomes in terms of the mechanical performance of the restoration [4]. The application of HFA etching increases the surface roughness (Ra), thereby promoting the micromechanical retention. Subsequently, the silane coupling agent facilitates the formation of a chemical bond between the ceramic surface and the luting material [5]. Despite the efficacy of this protocol, the use of HFA poses significant risks, including immediate hazards such as skin and nail burns, as well as long-term systemic effects like eye damage and respiratory issues [6]. Consequently, there is a pressing need to explore alternative conditioning methods that can achieve improved clinical outcomes, particularly in terms of the shear bond strength (SBS) of LDCs to resin cement, while minimising potential health risks.

Another method employed for conditioning ceramics is sandblasting (SB) with aluminium oxide (Al_2O_3). Research has shown that SB with Al_2O_3 modifies the surface texture of lithium disilicate-based ceramics (LDCs) by increasing the surface roughness (Ra), surface energy, and wettability [7]. This enhancement contributes to improved SBS by creating micromechanical undercuts for the luting cement [8]. However, sandblasting is considered a double-edged sword. While it effectively roughens the surface, it also removes a substantial amount of material from the restoration, potentially compromising the clinical fit [9]. Additionally, the increase in the surface roughness can alter the optical properties of the ceramics, leading to undesirable colour changes (ΔE) in the restoration [10].

In addition to the previously mentioned chemical and mechanical methods, laser irradiation has emerged as a promising technique for the surface pre-treatment of LDCs [11]. The use of a titanium sapphire (Ti-Al_2O_3) laser has demonstrated high efficacy in achieving precise micromachining across various dental materials with minimal collateral damage [12]. This laser technology is notable for its innovation and ability to generate ultrashort light pulses, making it versatile for numerous applications [13]. A study by Alrabiah and colleagues reported that Ti Al_2O_3 lasers resulted in satisfactory surface roughness (Ra) and bond strength between resin cement and hybrid ceramics [14]. Moreover, Photodynamic Therapy (PDT) represents another advanced approach, utilising low-level laser therapy (LLLT) to activate various dyes or photosensitisers (PS) for disinfection and conditioning in dental applications [15]. Among the different PS options, Riboflavin (RFP), a well-researched chromophore and natural water-soluble vitamin, has shown promise as an effective root canal disinfectant [16]. However, there is limited data on its effects on the Ra, SBS, and ΔE of LDC indirect restorations, indicating a need for further research in this area

The present study was conducted with the underlying assumption that there will be no notable difference in the ΔE of the LDC following surface conditioning with various contemporary surface modifiers, namely SB, LLLT-activated RFP, and Ti-Al_2O_3 laser in comparison to conventional HFA. Moreover, it has also been hypothesised that the application of contemporary surface conditioners to the LDC, when compared to the control, will not result in any significant difference in the Ra and SBS of the LDC to the resin adhesive cement. Therefore, the objective of the present study was to assess the effectiveness of various surface modifiers on the ΔE, Ra, and bond strength of LDC to resin cement.

EXPERIMENTAL

Specimen preparation guidelines

The present study complied with the specifications given in the checklist for reporting in vitro studies (CRIS). A total of eighty discs were carefully prepared from LDC blocks (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) using an Isomet machine (Buehler, Illinois, USA) along with water-cooling. These discs have dimensions of 2 mm in diameter and 10 mm in height. The sample surface was rendered flat and smooth by employing automated polishing equipment (Aropol 2 V, Arotec) operating at a speed of 450 rpm. After a comprehensive disinfection process using 96 % isopropanol (Mitsubishi chemicals, Japan) for 3 min, the produced discs were left to dry in the air. The discs were subsequently classified into four groups based on the surface pre-treatment they received (n = 20) [17].

Group 1: HF acid (S)

Within this experimental cohort, the LDC surface underwent a conditioning process using a 9.6 % HF acid solution (Bisco, Schaumburg, IL, USA) using a micro brush (Dentsply, New York, NY, USA) for 90 sec. The pre-treated surfaces were subjected to a water spray and then air-drying. Subsequently, a silane coupling agent (Monobond Plus, Ivoclar Vivadent, Schaan, Liechtenstein) was applied using a micro brush (Dentsply, New York, NY, USA) and left for 60 sec. The excess was removed by blowing air for 10 seconds [18].

Group 2: SB

The SB on the specimens was performed using 50 μm Aluminium oxide (Al_2O_3) particles (Korox, 110#46,014; BEGO) with Renfert Basic Master sandblaster (Renfert dental, Hilzingen, Germany) keeping it at a distance of 1 cm in the vertical direction. The procedure was conducted at a pressure of 2.8 atm for 15 seconds using a fine tip in a circular path. The surface was then washed using a constant flow of water, followed by air drying [19].

Group 3: Ti-Al_2O_3 laser

In this group, the discs were conditioned using a Ti-Al_2O_3 laser (Fotona Fidelis Plus, Ljubljana, Slovenia) set at an 800 nm wavelength, a power output of 2 W, a pulse energy of 120 $mJ.pulse^{-1}$, and a pulse frequency of 15 Hz. The radiation was administered using a 0.4-micron optical fibre tip, moving in a circular motion for 40 sec. This process was repeated four times, with each cycle lasting for 10 seconds with an interval of 15 seconds in between to avoid any increase in temperature [12].

Group 4: LLLT activated RFP

In this group, a concentration of 100 mg·L^{-1} of RFP was utilised in the LLLT approach. The photosensitiser (PS) was activated by exposing it to light emitted by a FlashMax P3 460, which emits light in the blue spectrum with a peak power of 460 nm. The LED blue lamp was fitted with small, cone-shaped conductive tips having a diameter of 4 mm at the cone end [15].

Evaluation of ΔE

Five samples from each group underwent an initial colour measurement which was performed in the CIE L*a*b* colour space using a spectrophotometer (GretagMacbeth, Color-Eye 7000A) against a white and black background. The spectrophotometer was set up to function in the mode specifically designed for measuring fundamental shades. To maintain precision, the spectrophotometer was calibrated at regular intervals as prescribed by the manufacturer. A 2 mm mid-labial section of each disc was examined. Each disc is positioned at a distance of 4 mm, 45 degrees angulated to the tip of the spectrophotometer. As part of the spectrophotometric measurement process, a small amount of distilled water was added to improve the optical contact and minimise the light loss around the edges of the disc. After the surface conditioning, the colour shift of all the samples was assessed using the standardised method mentioned earlier [20, 21]:

$$\Delta E = [(\Delta L^*)\ 2 + (\Delta a^*)\ 2 + (\Delta b^*)\ 2]\ 1/2$$

Surface Ra analysis

The surface Ra analysis was conducted on five specimens from each group using a Profilometer (PLμ 2300, Sensofar). The device was calibrated to enable the stylus point to precisely scan 0.75 mm. Three measurements were obtained from each sample to compute the average Ra value, expressed in micrometres (μm) [22].

SEM Evaluation

The surface characterisation of the LDC were evaluated under SEM (Zeiss Leo 1430, Carl Zeiss, Oberkochen, Berlin, Germany) at different magnifications following different surface pre-treatment methods. The samples were coated with a layer of gold using a sputter coater (SPI-Sputter Coater, IL, USA). All the specimens were evaluated under SEM.

Bonding of the luting cement to the LDC discs

For the experiment, ten specimens from each group were meticulously placed inside polyvinyl pipes that were filled with acrylic resin. It was ensured that the condition of each disc was positioned perpendicularly to the long axis of the polyvinyl pipe. The base and catalyst of the dual-cure resin luting cement RelyX (3M ESPE, St. Paul, MN, USA) were mixed following the instructions provided by the manufacturer. The cement mixture was subsequently introduced into silicone microtubes (Norton Performance Plastic; Cleveland, OH, USA) which had a height of 2 mm and an internal diameter of 2 mm. The microtubes were positioned in a perpendicular orientation relative to the surface of the LDC. The cement was subsequently exposed to photo polymerisation using an LED curing light (Demetron LC, Kerr, USA) with a wavelength ranging from 350 to 520 nm. The light guide tip was positioned as close as feasible to the tube's opening. The teeth were then subsequently immersed in deionised water for 24 hours while maintaining a constant temperature of 37 °C [23].

Thermocycling of the specimens

All forty specimens were subjected to 1000 artificial ageing cycles using a thermocycling machine (Nova Inc., Konya, Istanbul, Turkey). The cycles consisted of temperature changes ranging from 5 °C to 55 °C in two deionised grade 3 water baths. Each temperature was maintained for 30 seconds, followed by 10 seconds of transition to the next temperature. This process effectively simulated the ageing impact of 1 year [24].

SBS and failure mode analysis

The SBS of each sample was determined using a universal testing machine (UTM) (Autograph, model AG-IS, Shimadzu). The LDC cement interface was positioned perpendicular to the long axis of the jig (Bencor Multi-T shear assembly, Danville Engineering Inc., San Ramon, CA, USA) and the load was applied at a crosshead speed of 1 mm·min^{-1}. The samples were loaded until the ceramic disc detached from the luting cement and the maximum load was measured. The SBS was determined in Mega Pascals (MPa). Following the debonding process, the samples underwent evaluation under a stereomicroscope, with magnifications set at ×40. The failure modes can be classified into three categories: adhesive, cohesive, and admixed [25, 26].

Statistical analysis

The data were tabulated using the Statistical Program for Social Science (SPSS) version 21. An analysis of variance (ANOVA) was conducted to determine the mean and standard deviation (SD) of the ΔE, Ra, and

SBS. Tukey's post hoc multiple comparisons test was used for the intergroup comparisons while maintaining a margin of error of 0.05.

RESULTS

Evaluation of the ΔE

The mean and SD of the ΔE among the different experimental groups when treated with the contemporary surface conditioners are presented in Figure 1. The highest ΔE outcomes were witnessed in the Group 1 specimens (HFA (S) + LDC) (4.09 ± 1.11). Nonetheless, the minimum ΔE score was displayed by the Group 4 (LLLT activated RFPS) + LDC) (1.67 ± 1.20) specimens. The intergroup comparison analysis presented that the Group 1, Group 2 (SB + LDC) (3.77 ± 0.06), and Group 3 (Ti: Al_2O_3 laser + LDC) (3.99 ± 0.07) treated discs displayed comparable ΔE scores ($p > 0.05$).

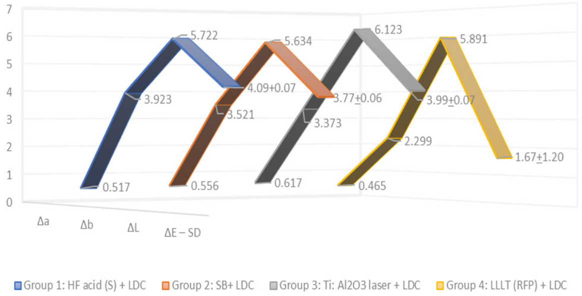


Figure 1. Mean and standard deviation (SD) of the colour change (ΔE) among the different study groups after using different pre-treatment regimes.

SEM assessment

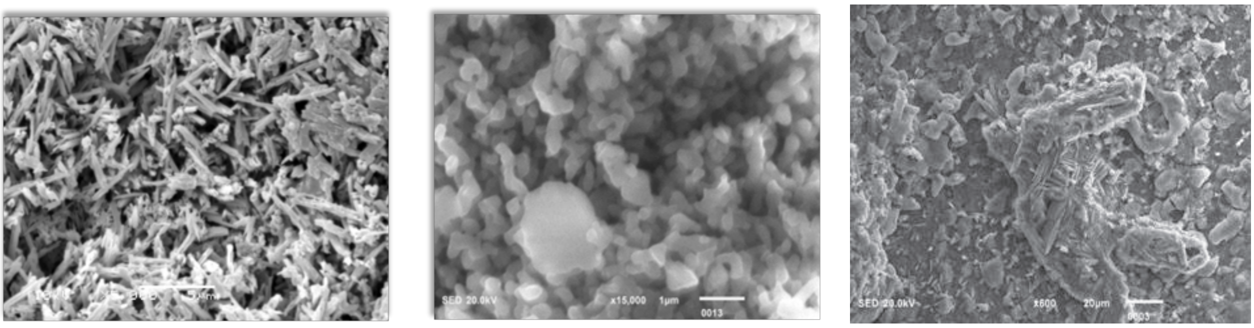
Figure 2A - The LDC surface conditioned with a Ti-Al_2O_3 laser. The SEM micrograph shows a needle-like appearance with clusters of crystals in certain areas. Figure 2B - The LDC surface treated with alumina particles via sandblasting. The SEM image again reveals a needle-like structure with alumina particles embedded on the surface. Overlapping crystals and lacunae are also evident. Figure 2C - The LDC surface pre-treated with LLLT-activated RBP. The SEM image displays a fusion of crystals and some damage to the superficial layer, but no matrix dissolution is observed.

Evaluation of Surface Ra

The mean and SD of the Ra after utilising various surface conditioners on the LDC discs are displayed in Figure 3. The highest Ra scores were detected in the Group 3 (Ti: Al_2O_3 laser + LDC) (1225.50 ± 0.035 μm) treated specimens. Whereas the Group 4 (LLLT activated RFPS) + LDC) pre-treated samples exhibited the lowest Ra scores (845.65 ± 0.017 μm). The comparison among the different investigated groups established that the Group 1 (HFA(S) + LDC) (1130.11 ± 0.025 μm) and Group 3 samples displayed no significant difference in their Ra outcomes ($p > 0.05$). The Group 2 (SB + LDC) samples, on the other hand, presented significantly lower values of roughness when compared to the Group 1 and 3 samples yet higher than the Group 4 conditioned LDC discs. ($p < 0.05$)

SBS analysis

The mean and SD of the SBS of the LDC discs to the resin cement after utilising different surface conditioning protocols are exhibited in Figure 3. The maximum and minimum bond strength scores of the dual-cure resin cement to the LDC discs were recorded in Group 3 (Ti:



a) b) c)

Figure 2. LDC surface conditioned with the Ti-Al_2O_3 laser. The SEM micrograph depicts a needle-like appearance with a congregation of crystals in some areas. 2B- LDC surface conditioned with alumina particles via sand blaster. Again, the SEM image shows a needle-like appearance with alumina particles integrated on the surface. Lacunae with overlapping crystals are visible from the SEM image. 2C- LDC pre-treated with LLLT activated RBP. From the SEM image, a fusion of crystals with damage to the superficial layer, but with no dissolved matrix, can be observed.

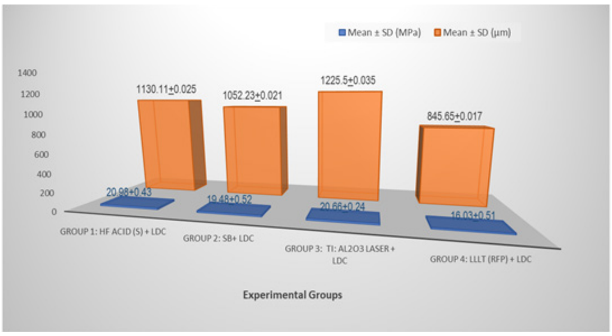


Figure 3. Ra scores and SBS of the LDC discs after utilising various surface conditioners.

Al_2O_3 laser + LDC) (20.66 ± 0.24 MPa) and Group 4 (LLLT activated RFPS) + LDC) (16.03 ± 0.51 MPa), respectively. Through the intergroup comparison, it was found that Group 1 (20.18 ± 0.13 MPa) and Group 3 established no significant difference in the SBS scores ($p > 0.05$). Furthermore, the Group 2 (SB + LDC) treated discs presented significantly lower outcomes of bond integrity scores than the Group 1 and 3 samples yet higher than the Group 4 conditioned LDC discs ($p < 0.05$).

Failure mode analysis

The percentage of the fracture distribution among the different study groups is presented in Figure 4. The outcomes revealed that Groups 1, 2, and 3 predominantly presented cohesive failures. However, the Group 4 treated discs offered adhesive and admixed fractures the most.

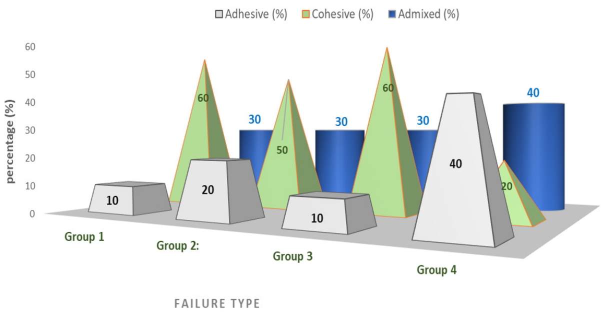


Figure 4. Percentage distribution of the failure mode among the different study groups.

DISCUSSION

The current study was based on the premise that there would be no significant difference in the colour change (ΔE) of the LDC after surface conditioning with various modern surface modifiers, specifically SB, LLLT-activated RFP, and Ti-Al_2O_3 lasers, compared to the traditional HFA method. Additionally, it was hypothesised that the use of these contemporary surface conditioners on LDCs, when compared to the control, would not lead to significant differences in the Ra and SBS to the resin adhesive cement. This primary hypothesis was partially validated, as the discs conditioned with SB and Ti-Al_2O_3 lasers demonstrated ΔE scores comparable to those of the control. Similarly, the secondary hypothesis was also partially validated, as only the Ti-Al_2O_3 laser-conditioned LDC discs showed Ra and SBS scores comparable to those obtained with HFA. The evaluation of SBS was conducted using a Universal Testing Machine (UTM) due to its unmatched precision, accuracy, and versatility in automated data collection, analysis, and reporting.

The evaluation of the colour disparity in this study was conducted using the CIE Lab* system, selected for its ability to correlate clinical relevance with visual perception. Previous research has shown that colour variations within a clinical setting can be complex, with changes of less than 3.7 units often being imperceptible to the human eye [27, 28]. The findings of this investigation revealed that discs treated with HFA, SB, and Ti-Al_2O_3 lasers exhibited noticeable colour changes, as their ΔE scores exceeded this threshold. Conversely, samples conditioned with RFP demonstrated ΔE scores below this critical value, indicating that their colour change is likely unnoticeable. Additionally, the treatments with HF acid, air-abrasion using Al_2O_3, and Ti-Al_2O_3 lasers are known to effectively eliminate the vitreous matrix and enhance the crystalline phase of ceramic materials [29, 30]. As a result, these methods produced a rougher surface texture on the LDC, as evidenced by the outcomes of this research. Therefore, the findings of this study are consistent with previous laboratory investigations, supporting the idea that surfaces with higher Ra tend to exhibit more noticeable colour variations compared to smoother surfaces [31, 32].

According to Dr. Georgios Siviloglou, a laser can be described as a type of Airy Beam that contains a finite amount of energy and can bend and propagate without dispersion [33]. The Ti-Al_2O_3 laser is a high-powered, rapid system that emits beams with picosecond and femtosecond pulse widths, ensuring no additional thermal damage occurs [34]. The Ti-Al_2O_3 laser has demonstrated the ability to achieve the highest surface roughness, suggesting it is a potential alternative treatment for ceramic surfaces to enhance the bond strength, as noted by Erdur et al. [35]. In the present study, the scanning electron microscopy (SEM) of the ceramic surface treated with the Ti-Al_2O_3 laser have shown significant irregularities characterised by deep valleys, pronounced round peaks, and a uniform Ra. This surface configuration is likely to enhance the mechanical retention, thereby improving the bond strength between the composite and ceramic surfaces. In contrast, Peixin

Hu and colleagues observed that surfaces treated with a Ti-Al_2O_3 laser exhibited a considerably smoother profile with reduced average Ra [36]. However, the evidence concerning the impact of the Ti-Al_2O_3 laser on the LDC is still inconclusive, indicating a need for further investigation

On the other hand, HF acid displays a unique capability to create a pronounced etching pattern by selectively dissolving the silica glass phase, which enhances the Ra of the used ceramic [37]. Moreover, this process exposes the hydroxyl groups on the ceramic surface, leading to a significant increase in surface energy [6]. Subsequently, the application of a silane coupling agent initiates a chemical reaction that forms covalent bonds with the exposed hydroxyl groups. This interaction results in a decrease in the surface energy and the creation of a durable hydrophobic layer, which improves the bonding with the resin-luting cement [38].

Furthermore, it was observed that the SB testing group likewise yielded satisfactory Ra scores and SBS. Nevertheless, it exhibited a notable decrease compared to the HFA and Ti-Al_2O_3 laser. According to the available indexed literature, the effect of SB on the roughness and bond strength outcomes is controversial. Some studies concluded that SB is not an effective way to strengthen the resin cement bond as the surface micromorphology remains unchanged. However, this process did produce the propagation of tiny cracks around the crystal phase which leads to restoration failure [39, 40]. However, there is much research available that has proclaimed that SB enhances the surface irregularities and improves the bond integrity scores [41, 42]. Regarding LLLT-activated RFP, the lowest Ra and SBS outcomes can be elucidated on behalf of the lab-based analysis conducted by Basunbul and colleagues which follows the outcomes of the present inquiry. They explained that LLLT-activated RFP leads to the formation of an O_2 inhibition layer on the LDC discs which interacts with the resin cement polymerisation and eventually compromises bond strength [16].

The current investigation is subject to certain inherent limitations. The findings should be interpreted with caution in clinical settings, as the research was predominantly conducted as a lab-based trial. The use of different PS activation parameters, types of PS, and laser may have led to different outcomes. Furthermore, the understanding of the conditioned surfaces and their interactions with the luting cement with different viscosities is limited and needs further probing. Also, other mechanical testing methods should be analysed following the conditioning of LDCs in a dynamic oral environment.

CONCLUSION

Ti-Al_2O_3 laser proved to be a suitable alternative as it positively influences the shear bond strength and surface roughness. However, it results in a considerable colour change which limits its application to posterior teeth only.

Acknowledgment

This work was funded by the researchers supporting project number (RSP2024R261) King Saud University, Riyadh, Saudi Arabia

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