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SURFACE TOPOGRAPHY AND ENERGY DISPERSIVE X-RAY OF RESIN MATRIX CERAMICS CONDITIONED WITH AN ULTRASHORT PULSE LASER, TRI-BIOCHEMICAL SILICA, AND HYDROXYAPATITE - AN IN-VITRO STUDY

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The effect of different surface modifiers, i.e., a tri-biochemical silica coating (TBC), an ultrashort pulsed laser (UPL), nano-hydroxyapatite (HA) on the surface roughness (Ra), and shear bond strength (SBS) of hybrid ceramics (HBCs) bonded to the resin luting cement was elucidated upon. Eighty-four squared HBC discs were prepared and randomly allocated into four groups based on the different surface pre-treatment: Group 1 (SB), Group 2 (TBC), Group 3 (UPL) and Group 4 (Nano-HA). A surface topography analysis was performed on one pair of samples from each group using scanning electron microscopy (SEM) followed by an EDX analysis. Forty discs underwent an Ra analysis using a profilometer. Luting cement was bonded to the HBC discs followed by artificial ageing on ten samples from each group. The SBS was measured using a universal testing machine and the failure was examined under a stereomicroscope. Data normality was assessed using the Kolmogorov-Smirnov test, and the Ra and SBS data were analysed using a one-way analysis of variance and Tukey's post-hoc test. ($p \leq 0.05$). The maximum Ra (1075.21 ± 0.5 µm) and SBS (9.6 ± 0.1 MPa) scores were obtained in the Group 4 (HAp coating) specimens. Nevertheless, the Group 3 (UPL) treated discs demonstrated the lowest outcomes of the Ra (999.42 ± 1.1 µm) and bond strength scores (8.9 ± 0.2 MPa). The nano-hydroxyapatite and tri-biochemical silica coating can be used as an alternative to sandblasting to condition hybrid ceramics.

INTRODUCTION

Hybrid ceramics (HBCs) also known as resin matrix ceramics (RMCs) have been developed by combining the beneficial features of composite and conventional ceramics. This was undertaken to augment the properties and durability of indirect restorations [1]. HBCs, which have replaced the glass matrix of conventional ceramics with a polymer matrix demonstrates, improved the crack resistance and mechanical properties that are comparable to enamel and dentin [2]. This also reduces the shortcomings of conventional ceramics as they abrade the opposing natural teeth. Despite these advantages, the adhesion of HBC to the tooth substrate is a critical step that has a substantial impact on the longevity and durability of the treatment [3].

The pre-treatment of the intaglio surface of HBC restorations with various conditioners has gained widespread acceptance due to the resulting improvements in the mechanical properties when bonded with resin-luting cement [4]. Based on the available evidence, sandblasting (SB), also known as alumina air abrasion, is recognised as one of the most effective initial surface conditioning methods for HBC. It increases the surface roughness (Ra), which enhances the surface energy and wettability, thereby promoting micromechanical interlocking and improving the shear bond strength (SBS) of resin cement [5, 6]. However, the surface indentation pattern on sandblasted ceramics can vary depending on factors such as the particle size, air pressure, and duration of the process [7]. Despite its excellent results, SB has some drawbacks, including the need for a micro-blaster and a rubber dam to protect against potential respiratory side effects [8].

In addition to sandblasting (SB), another technique that has attracted scholarly attention is Tri-biochemical Silica Coating (TBC). This method provides ultrafine mechanical retention by utilising silica-coated 30-µm Al_2O_3 particles [9]. A laboratory study by Wu and colleagues found that TBC is equally as effective as SB when used as a ceramic surface modifier [10]. However, limited data are available regarding its effects on the

surface roughness (Ra) and shear bond strength (SBS) of hybrid ceramic (HBC) restorations with a resin-luting cement, highlighting the need for further investigation.

Recent advancements in laser technology have resulted in the development of ultrashort pulsed lasers (UPLs), which offer the advantage of minimising the energy delivered to the surface, thereby preventing thermal or mechanical damage [9, 11]. A laboratory study conducted by Ruja and colleagues reported that the UPL treatment increases the Ra of zirconia ceramics, ultimately improving the adhesion strength with a resin-luting cement [12]. However, there is limited data on its effects on the Ra and SBS of HBC restorations, indicating the need for further investigation.

In addition to lasers, advancements in nanotechnology have highlighted the potential applications of nano-hydroxyapatite (HA) in dentistry, with crystal sizes ranging from 50 to 1000 nm. Nano-HA is known for its bioactive and biocompatible properties, and it closely resembles the crystal structure and morphology of tooth enamel [13]. These nano-HA particles form a thin, uniform coating on ceramic surfaces, which contributes to the improved SBS [13, 14]. However, there is currently a lack of research specifically examining the impact of nano-HA on the Ra and SBS of HBC, indicating the need for further investigation in this area.

Based on the available literature, it can be assumed that there will be no significant difference in the Ra of the HBC when the latest surface modifiers are used as compared to the SB. Moreover, it was also predicted that there would be comparable SBS of the luting resin cement to HDC when the surface was pre-treated with a modern conditioning regime as compared to the control. Thus, the current inquiry aimed to explore the effect of different conditioners on the Ra and SBS of HBC on the luting cement used.

EXPERIMENTAL

Specimen preparation

A computer-aided design and computer-aided manufacturing (CAD/CAM) HBC material (Vita Enamic (VE) Vita Zahnfabrick in BadSackingen, Germany) was used in the existing study. Using a water-cooled diamond wafering blade with a low-speed cutting saw (Isomet 4000, Buehler Ltd, Lake Bluff, IL, USA), eighty-four squared specimens (10 × 10 × 3 mm) were carefully sectioned from the VE blanks. Various grades of silicon carbide papers (400, 600, and 1500-grit) were utilised to meticulously polish the exposed surfaces of the specimens while ensuring a constant flow of water. The specimens underwent a thorough 5-minute ultrasonic cleaning using an Ultrasonic Cleaner (Baioden Ultrasonic Cleaner, Beijing, China). The samples were allowed to air dry and were then allocated into four groups on the type of surface pre-treatment regime (n = 21 each group) [15].

Grouping of HBC discs according to the different surface treatments

Group 1 (SB): The samples were blasted with 50 μm aluminium oxide (Al_2O_3) particles using the Mestra sandblast apparatus (TALLERES MESTRAITUA S. L., Spain) at 2.5 bar for 15 sec. This was performed by placing the tip at a 10 mm distance and 45° angle from the surface. After alumina blasting, the samples were cleaned with an air stream to remove any remaining alumina particles from the surface [16].

Group 2 (TBC): The samples in this group were blasted with 30-μm coated alumina particles (Cojet sand, 3M ESPE, Neuss, Germany) at 2.5 bar with the tip being held at a 10 mm distance inclined at approximately 45° for 15 sec. After treatment, the samples were cleaned using an air stream to remove any remnants of particles on the surfaces. This was followed by a silane coupling agent application (Bisco Inc., Schaumburg, IL, USA) for 60 seconds followed by air drying [17].

Group 3 (UPL): The HBC surfaces in this group were irradiated with a solid-state Neodymium-doped yttrium orthovanadate (Nd: YVO_4) ultrafast laser (Lumera, Hyper Rapid 50, Coherent, IL, USA). Its wavelength was set at 1064 nm with a 10 picosecond (ps) pulse duration. The LSD dots are arranged in a pattern with a spacing of 2.5 m between each dot in all directions. The dots are subjected to multiple pulses with an energy of 8 millijoules and a frequency of 10 kilo-hertz. Following UPL ablation, the samples underwent a 10-minute ultrasonic cleaning process in distilled water to eliminate any surface debris. Afterward, the samples were left to dry in the air [12].

Group 4 (Nano-HA): The process involved using the HBC discs that were coated using the HA thermal protocol. A slurry solution was prepared by adding 10 g of a nano-particle HA powder (Merck, Berlin, Germany) having a particle size less than 100 nm with 50 cc of distilled water. Additionally, 1 g of polyvinyl alcohol (Merck, Berlin, Germany) was included as the binder for the suspension. The mixture was then heated on a magnet stirrer at 100 °C and 1000 rpm for 60 min to ensure a consistent suspension. Finally, the HBC discs were immersed in the slurry at a 45° angle for 5 seconds [18].

Surface topography and EDX analysis

The blended cement materials were prepared by re-placiOne pair of samples from each group was affixed to metallic stubs and then coated with a layer of gold using a gold sputter coating system (Polaron SC7620; Quorum Technologies). These samples were then examined under a scanning electron microscope (SEM) (Apreo S, Thermo Fisher Scientific) at a magnification of 2000× to observe any changes in the surface topography resulting from the surface treatment. An energy-dispersive X-ray

spectroscopy (EDS) examination was conducted to determine the constituents of the specimens [19].

Measurement of surface roughness

A total of ten specimens were randomly selected from each group to assess surface roughness using a profilometer (Surftest SJ-201, Mitutoyo Corp., Japan). The probe was positioned in the centre of each disc and measurements were taken in three distinct directions. The traversing length was set at 0.8 mm, and the measuring speed remained constant at 0.5 mm·sec^{-1}. The average surface roughness (Ra) for each specimen was computed [20].

Adhesion of the luting cement to the HBC discs

For the experiment, a total of ten specimens from each group were fixed in self-cure acrylic resin which was enclosed in silicone moulds. The positioning of each disc was carefully assured to be perpendicular to the long axis of the mould. The base and catalyst of the dual-cure resin luting cement (Multilink N, Ivoclar Vivadent) were combined following the manufacturer's recommendations. The cement mixture was then injected into silicone microtubes which were placed in a perpendicular orientation concerning HBC discs having a height and an internal diameter of 2 mm. The cement was then subjected to LED photo polymerisation (Valo, Ultradent Products) at the intensity of 1000 mW·cm^{-2} and a wavelength ranging from 395 to 480 nm for 20 sec. The discs were then submerged in deionised water for 24 hours at 37 ℃ [20].

Artificial ageing of the samples

The samples were subjected to thermal cycling (Ethik Technology, São Paulo, Brazil) with temperature variations ranging in water baths between 5 and 60 ºC with a dwell time of 15 sec each. A total of 10,000 cycles which is equivalent to one year of exposure in a living body was performed.

SBS assessment and failure assessment

The SBS was measured using a universal testing machine (Autograph AGS X, Shimadzu, Kyoto, Japan), Turkey). The knife-edge chisel was used to apply a shear force at the cement disc interface at a crosshead speed of 0.5 mm·min^{-1} till the time failure occurred. The load required to cause failure of the bond was measured in megapascals (MPa). To ascertain the type of failure, all the discs were examined under a stereomicroscope (Discovery Z-20, Zeiss, Jena, Germany) at a magnification of ×50. The failure modes were categorised as adhesive, cohesive, and admixed [21, 22].

Statistical analysis

Following the verification of data normality using the Kolmogorov-Smirnov test, the Ra and SBS data were analysed using a one-way analysis of variance (ANOVA) and Tukey's post-hoc tests ($p \leq 0.05$).

RESULTS AND DISCUSSION

Ra analysis

Figure 1 presents the Ra score of the HBC discs pre-treated using different surface modifiers. The maximum Ra score was obtained in the Group 4 (HAp coating) specimens (1075.21 ± 0.5 µm). Nevertheless, the Group 3 (UPL) treated discs demonstrated the lowest Ra outcomes (999.42 ± 1.1 µm). The intergroup comparison analysis unveiled that the Group 1 (SB) (1055.21 ± ± 1.5 µm), Group 2 (TBC) (1060.13 ± 0.9 µm), and Group 4 treated discs displayed no significant difference in their Ra values ($p > 0.05$).

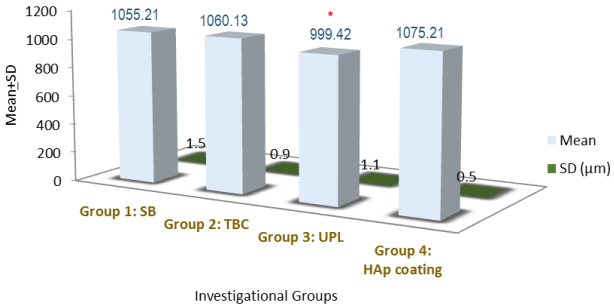

Figure 1. Ra of the hybrid ceramics discs pre-treated using different surface modifiers.

SEM analysis after the surface modifiers

The SEM analysis showed that the surfaces of the SB and TBC groups exhibited irregular and non-uniform notches. This variation is attributed to the inconsistent and imprecise manual application of alumina in SB and TBC, leading to inconsistencies in the depth of the surface indentations. In contrast, the UPL treatment produced a uniform engraving pattern and a smoother surface compared to the other treatment groups (Figures 2a and 2b). The EDX analysis shows major constituents of calcium, silica, oxygen, and carbon in hybrid ceramics (Figure 3).

SBS analysis

Figure 4 displayed the SBS of the HBC discs to resin luting cement pre-treated using different surface modifiers. The maximum bond strength score was

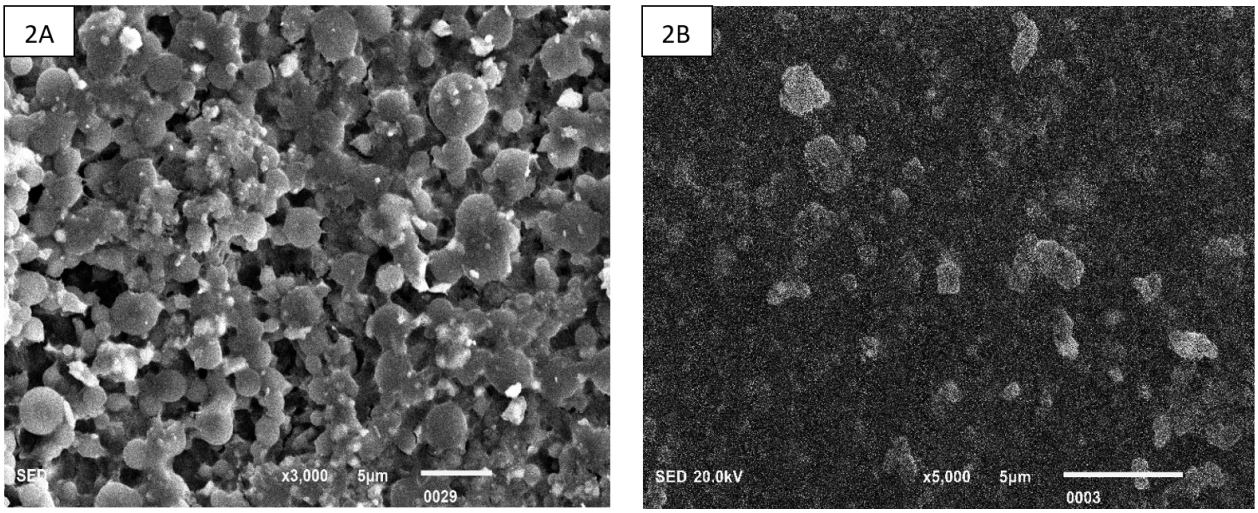

Figure 2. SEM image depicts the hybrid ceramic surface treated with SB and TBC. 2A) Loss of matrix is visible with irregular non-uniform notches. 2B) hybrid ceramics treated with a UPL uniform engraving pattern and a smoother surface with no loss of matrix.

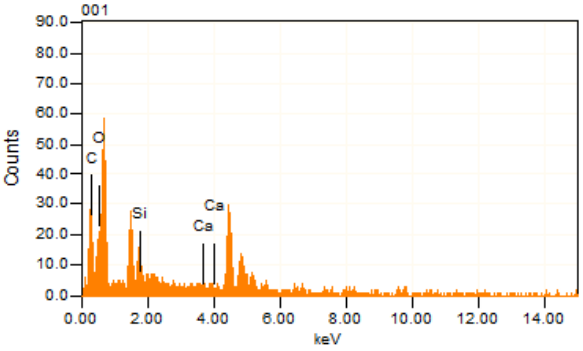

Figure 3. EDX analysis of the hybrid ceramics.

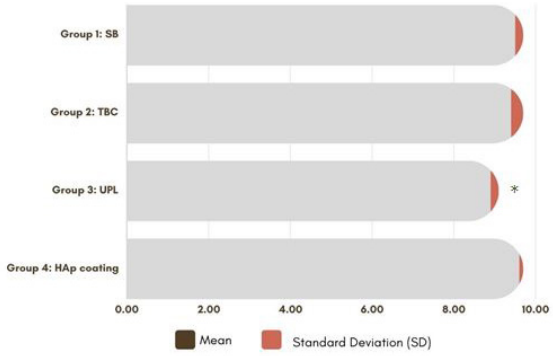

Figure 4. Shear bond strength (SBS) of the hybrid ceramic (HBC) discs to the resin luting cement discs pre-treated using different surface modifiers.

exhibited by the Group 4 (HAp coating) (9.6 ± 0.1 MPa) specimens. Nevertheless, minimum bond integrity values were recorded by the Group 3 (UPL) (8.9 ± 0.2 MPa) treated discs. The intergroup comparison analysis discovered that Group 1 (SB) (9.5 ± 0.2 MPa), Group 2 (TBC) (9.4 ± 0.3 MPa), and Group 4 demonstrated comparable SBS outcomes ($p > 0.05$). Whereas Group 3 displayed significantly lower scores than the other treated groups ($p < 0.05$).

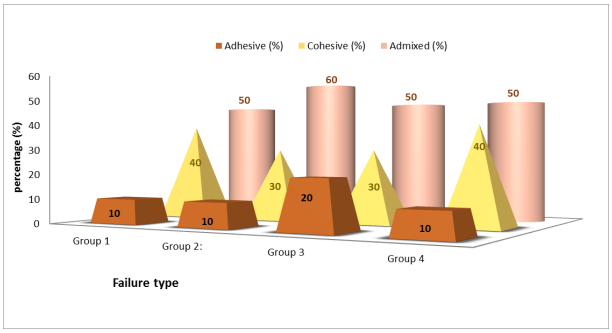

Figure 5. Percentage distribution of the failure modes.

Failure mode analysis

Figure 5 displays the percentage distribution of the failure modes among all the investigated groups. The admixed failure is the most predominant fracture pattern among all the tested groups followed by adhesive and cohesive failures.

DISCUSSION

The present research was based on the prediction that TBC, UPL, and HAp coating treated HBC discs would exhibit similar Ra scores to that of SB. Furthermore, it was expected that there would be no substantial disparity in the SBS between the TBC, UPL, and HAp coating pre-treated discs in comparison to the control group. However, both the stated hypotheses were partially invalidated since the Ra and SBS scores of the UPL-treated HBC were lower as compared to the SB-

conditioned samples. The SBS test is a highly prevalent technique employed to assess the adhesive strength of a substance. Furthermore, the process is simple and can be easily replicated [23, 24].

Before the application of a resin cement, specific surface pre-treatments can significantly enhance the micromechanical retention by increasing the Ra, thereby yielding a stronger bond [9, 25]. In the present study, the comparable highest SBS was achieved on surfaces treated with sandblasting (SB), the tri-biochemical silica coating (TBC), and the hydroxyapatite (HAp) coating. There are various coating methods available to harness the advantages of nano-hydroxyapatite (nano-HA). Previous studies have documented several techniques for applying a nano-HA layer onto bioinert metallic implant surfaces, such as plasma spraying and sol-gel methods [26-28]. In this study, we employed the thermal coating method due to its favourable mechanical properties and practical feasibility. The HBC discs modified with the HAp coating exhibited the highest Ra and SBS to the resin-luting cement. These findings are consistent with the laboratory analysis by Sagsoz et al., who observed that micro-retentive areas under SEM may significantly contribute to enhancing the mechanical bond strength [29]. Moreover, a similar in vitro study conducted by Atri et al. on zirconia concluded that the thermal coating technique might alter the bonding characteristics of zirconia [30]. Additional in vitro experiments and clinical investigations are necessary to further explore the applicability of this method.

Previously, SB has been specifically recommended for conditioning HBC due to its ability to remove the smear layer produced after milling [31, 32]. This technique enhances the surface area by increasing the Ra and exposes the inner filler aggregates. Additionally, roughening the surface improves the surface energy and wettability of the restorative materials, thereby promoting the micromechanical interlocking and enhancing the bond strength [33]. Mao and co-authors revealed that pre-treatment with SB resulted in the highest Ra values among all the tested materials, characterised by a micro-retentive ridge and groove patterns, which led to satisfactory SBS outcomes at the same time [34]. However, contrary findings were observed in a laboratory investigation conducted by Campos and colleagues [35]. In the authors' opinion, the duration of use, particle size and shape, and study design may have contributed to the contradictory outcomes. TBC, which utilized 30 μm aluminium oxide (Al_2O_3) particles modified with silica for mechanical grinding displayed outcomes comparable to the control. The slight differences in the observed Ra and bond integrity may be attributed to the varying grit sizes used in the TBC and SB groups. Loomans et al. reported that CoJet® positively influenced the SBS of HBC CAD/CAM restorations [36]. Similarly, a study by Tatar and Ural found that CoJet® was the most effective method for enhancing the repair bond strength of resin matrix ceramics [37]. Some studies have presented contradictory findings, indicating that TBC was ineffective as a surface conditioner for HBC [32, 35]. However, the observed increase in the Ra in the TBC group is consistent with the in vitro results of Porojan and co-workers [38].

This study found that using an ultrashort pulsed laser (UPL) on HBC resulted in a lower Ra and SBS to the luting cement compared to the control group. This contrasts with the findings of Mahmood and colleagues, who, in an ex vivo analysis, suggested that UPL, when applied in a dot pattern, could be a promising alternative to mechanical blasting for zirconia surface treatment [39]. The main effect of the laser is the conversion of radiant energy into heat, creating a thermo-mechanical response. This heat induces ruptures on the ceramic surface, increasing the Ra and ultimately improving the bond strength of the luting resin [9, 40, 41]. While the laser treatment's effects on ceramics are well known, this study is the first to investigate UPL as a surface modifier for HBC, signalling the need for further research into its potential applications.

The limitations of the present study are inherent to the in vitro design, where only controlled variables are considered. Obtaining a consistent HA coating on the HBC discs is a delicate and intricate process. The most frequently employed test method for assessing adhesive bonding between two materials is SBS. However, this method displays the drawbacks of having an uneven stress distribution. Furthermore, variations in the particle size and laser parameters could have led to diverse outcomes, therefore, more studies are required to generalise the findings of the present investigation.

CONCLUSION

A nano-hydroxyapatite coating and tri-biochemical silica coating can be used as an alternative to sandblasting for the surface modification of hybrid ceramics.

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