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REPAIR BOND STRENGTH, SURFACE ROUGHNESS, AND TOPOGRAPHIC ASSESSMENT OF NANOCOMPOSITE CERAMICS WHEN SURFACE MODIFIED WITH A NANOSECOND LASER; LOW-LEVEL LASER THERAPY ACTIVATED MALACHITE GREEN AND TRI-BIOCHEMICAL SILICA

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The impact of different surface modifiers, i.e., tri-biochemical silica coating (TBC), low-level laser therapy (LLLT) using malachite green (MG) and a nanosecond (ns) laser on the surface roughness (Ra) and repair bond strength of a nanocomposite ceramic (NCC) to a composite was elucidated upon. NCC discs were manufactured by utilising prefabricated blocks and underwent disinfection. These discs were subsequently categorised into four groups at random, applying various surface modifiers (n = 16) - Group 1: HFA+S; Group 2: TBC + S; Group 3: LLLT (MG) + S and Group 4: ns laser + S. A topographic evaluation following surface modification was performed using Scanning Electron Microscopy (SEM). An Ra analysis was performed on five specimens using a surface profilometer. A composite was applied to repair the ceramic surface, where the repair bond strength and failure mode assessment was performed utilising a universal testing machine and stereomicroscope, respectively. The data were analysed using a one-way analysis of variance (ANOVA), and Tukey's post-hoc test, with $p < 0.05$ deemed significant. The highest Ra values (1558.38 ± 0.022 µm) and repair bond strength (18.22 ± 0.19 MPa) of the NCC were observed in Group 2 (TBC) samples. However, the Group 3 LLLT-MG pre-treated samples exhibited the lowest roughness (1009.24 ± 0.021 µm) and bond strength (13.18 ± 0.32 MPa) values. A tribochemical silica coating and nanosecond laser can be used as an alternative conditioning regime for nanocomposite ceramics as they positively influence the repair bond strength and Ra.

INTRODUCTION

Dental restorations and crowns are commonly employed to rehabilitate damaged or decayed teeth. Recently, computer-aided design and computer-aided manufacturing (CAD/CAM) nanocomposite ceramics (NCCs) have gained significant popularity in the field of prosthodontics [1, 2]. This can be ascribed to their exceptional aesthetics, higher hardness, and superior toughness provided by the resin content which contributes to the improved tolerance against masticatory forces [3]. Nonetheless, any restoration derived from ceramic material is prone to fracture during clinical use [4]. Therefore, it is essential to identify methods that contribute to the easy and long-lasting repair of the fractured segment [4]. To repair ceramics, the role of surface conditioning is of utmost importance [5]. It has been asserted from previous studies that NCC materials establish a robust chemical bond with resin materials owing to the elevated polymer component [3].

Hydrofluoric acid (HFA), in conjunction with a silane coupling agent (S), is recognised as the standard NCC conditioning regime for improving the shear bond strength (SBS) and surface roughness (Ra) [6]. However, researchers have indicated that HFA exhibits corrosive properties and can harm human tissues [7]. Therefore, it becomes necessary to find better alternatives that are safe for human use. Another method that has gained widespread acceptance is a tri-biochemical silica coating (TBC). It was developed for the surface conditioning of dental restorative materials, including ceramics, metals, and metal alloys [8,9]. This system utilises silica-modified 30-µm Al2O3 particles to achieve ultrafine mechanical retention. A laboratory-based analysis conducted by Wu and colleagues concluded that TBC, as a ceramic surface modifier, enhances the Ra and bond integrity of NCCs [10]. However, data are still scarce regarding TBC's ability to repair a fractured NCC and hence needs future inquiry.

Lasers and low-level laser therapy (LLLT) utilising a photodynamic treatment (PDT), represent non-invasive techniques that are progressively employed in various fields of dental sciences. Malachite green (MG), a photosensitiser (PS), commonly referred to as brilliant green, has attracted significant interest from various researchers [11,12]. This positive charge dye unveils a high absorption rate in the red region of the visible spectrum [13]. However, data are limited regarding its effectiveness for ceramic surface modifiers. Similarly, nanosecond (ns) lasers have also gained significant attention in the past few years in the field of dentistry [14]. A study conducted by Garcia-de-Albeniz through a laboratory-based analysis demonstrated that linear microgrooves with a periodicity of 50 µm were formed on a zirconia (3Y-TZP) surface which results in enhanced mechanical properties [15]. Concerning the impact of an ns laser and LLLT-(MG) on the Ra and SBS to NCC is limited and needs further investigations.

Based on the available evidence, it can be inferred that there will be no substantial disparity in the Ra of the NCC when contemporary surface conditioners (TBC, ns laser, and LLLT-activated MG) are utilised in comparison to the HFA. Furthermore, it was also predicted that the repair bond strength of the composite to the NCC would be comparable when the surfaces were pre-treated with a contemporary conditioning regimen in comparison to the control (HF+S). Thus, the existing study aimed to explore the influence of different surface conditioners on the Ra and repair bond strength of the NCC to the composite.

EXPERIMENTAL

The present investigation conformed to the stipulations established by the checklist for reporting in vitro studies (CRIS) guidelines. Sixty-four discs were made from a 3M™ Lava™ Ultimate CAD/CAM NCC material. The discs were manufactured by utilising prefabricated blocks having measurements of 6 mm in diameter and 2 mm in thickness. Each disc underwent a disinfection process employing 96 % isopropanol for 3 minutes succeeded by an air-drying phase to guarantee the maximum cleanliness and disinfection. The discs were subsequently categorised into four groups at random, applying various surface modifiers (n = 16).

Group 1: HFA+S

The samples were conditioned using a 9.6 % HFA gel (IPS Ceramic Etching Gel, Ivoclar Vivadent, Schaan, Lichtenstein) for 1 min. The surface was subsequently rinsed with a powerful water jet, followed by air-drying. A silane coupling agent (Ultradent, IL, USA) was applied for 1 min [16].

Group 2: TBC

NCC discs were blasted at 2.5 bar using 30-µm Al2O3 particles modified with silica (Cojet sand, 3M ESPE, Neuss, Germany) for 15 sec. The blasting tip nozzle was positioned 10 mm away and tilted at an estimated 45° angle. Post-treatment, the samples underwent a cleaning process utilising an air stream to eliminate any residual particles present on the surfaces. A silane coupling agent (Bisco Inc., Schaumburg, IL, USA) was then applied for 1 min and air dried [17,18].

Group 3: LLLT-MG

The NCC discs were treated with a 0.01 % MG solution for 5 min, which is designated as the pre-irradiation period. The surface was subjected to laser irradiation for about 3 minutes, utilising an energy density of 5.4 $J \cdot cm^{-2}$ [19].

Group 4 (ns laser)

The surface patterning was performed utilising the Spectra-Physics Explorer One 349–120 laser source, which operates at a wavelength of 349 nm, with a pulse duration of less than 5 ns and an energy output of 120 µJ per pulse [20].

Surface characteristics assessment

Scanning Electron Microscopy (SEM, LEO 1530VP, Oberkochen, Germany) analysed the surface topography of the pre-treated samples. The substrate was cleaned using ethanol followed by drying. Sputter coating was performed on the specimens using gold powder (JFC – 1100 fine coat ion sputter) in 6 nm thickness at a current of 40 mA for 250 sec. The specimens were then affixed in the chamber of the SEM with the help of carbon tape and analysed under magnification of ×2000. An energy dispersive spectrometer (EDS; INCAx-sight, Oxford Instruments, UK) was employed to analyse the elemental distributions [3,21].

Surface roughness (Ra)

Five samples from each cohort underwent an Ra analysis. On each sample, five distinct measurements at various positions were obtained using a surface profilometer (Surftest 211 model, Bruker, Billerica, MA, USA). The Ra threshold was set at 0.8 mm, with the stylus moving at a distance of 4.0 mm. The radius of the tracing diamond tip was measured at 5 μm, utilising a measuring force of 4 mN (0.4 gf) and a speed of 0.5 m s−1, respectively. The mean roughness value of a single disc was calculated by averaging the five Ra values [22]. Application of the composite to repair the ceramic surface

The peak universal bond adhesive (Ultradent Products Inc., South Jordan, UT, USA) was meticulously applied to the ceramic surface using a single brush stroke and subsequently, light cured for 15 sec with Bluephase G2 (Ivoclar, Vivadent). The treated surfaces were then bonded using a composite material (Clearfil Majesty Aesthetic; Kuraray, Medical, Tokyo, Japan) in incremental layers of 2 mm with the help of a Teflon mould and cured for 40 seconds [23].Thermocycling of the prepared samples

All the specimens were aged artificially by exposing them to 10,000 thermal cycles (Suzhou Weier Lab ware Co. Ltd., China). The temperatures maintained were 5 °C and 60 °C in two water baths. The samples were immersed in these water baths for 60 seconds each with a transfer time of 5 seconds. Afterwards, all the specimens underwent a 24 hour-storage period in distilled water maintaining the temperature at 37 °C [24].

Repair bond strength assessment

A universal testing machine (UTM) (Model 3365; Instron Corp., Canton, MA, USA) was used to determine the SBS of the NCC disc bonded to the composite resin at a crosshead speed of 1 $ml \cdot min^{-1}$ at a load of 2.5 kN. The specimens were carefully assembled and secured within a jig, onto which a force was applied perpendicularly to the bonded surface until the point of repair failure was achieved. The SBS was measured in Megapascals (MPa) [25].

Failure mode assessment

Following the completion of the SBS testing, the failure mode for each specimen was recorded using a stereomicroscope at 40 X. The failure modes were classified into three categories: adhesive, cohesive, and admixed [26].

Statistical analysis

The bond strength and Ra score were compared using a one-way analysis of variance (ANOVA) and Tukey's test. A significance threshold of $P < 0.05$ was employed to ascertain the outcomes.

RESULTS

Topographic Assessment and Elemental Analysis

The SEM findings indicate that the TBC air abrasion techniques produced rough surface morphology, resulting in the exposure of internal filler aggregates which enhances the mechanical interlocking of the repair material used and eventually the bond strength (Figure 1). The ns-laser enabled the formation of distinct and reproducible grooves on the NCC discs as seen in the SEM analysis. The observed pattern formation encompassed the melting and resolidification of the surface material (Figure 2). The elemental analysis via EDX shows three main elements abundant in the NCC. Carbon (C), Oxygen (O_2) and Flouride (F)

Ra Evaluation

Figure 4 presents the mean Ra score of the NCC after applying different surface modifiers. The highest Ra

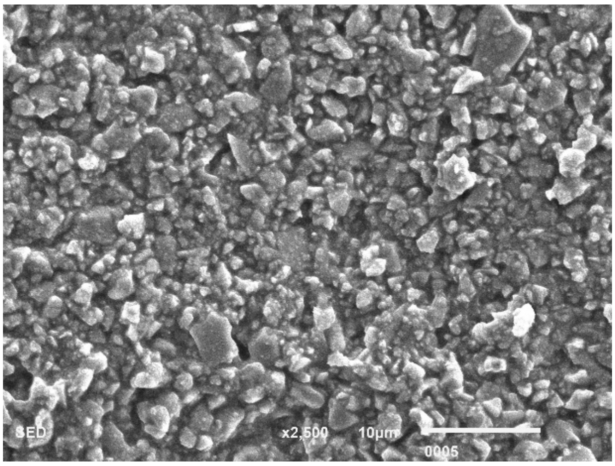

Figure 1. SEM surface modified with the TBC. Loss of silica in the observed increasing Ra.

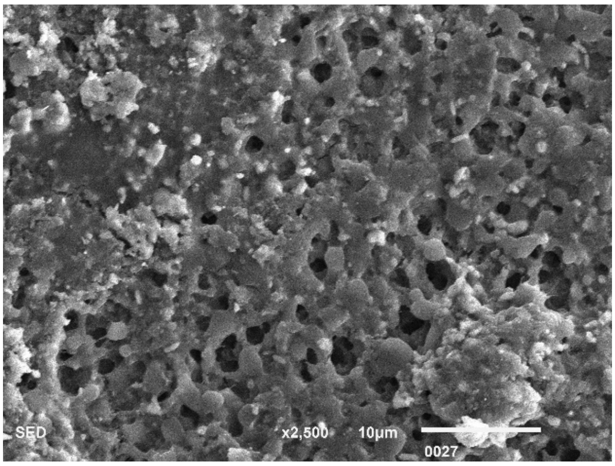

Figure 2. SEM surface depicts the melting and resolidification of the material when the surface is modified with a nanosecond laser.

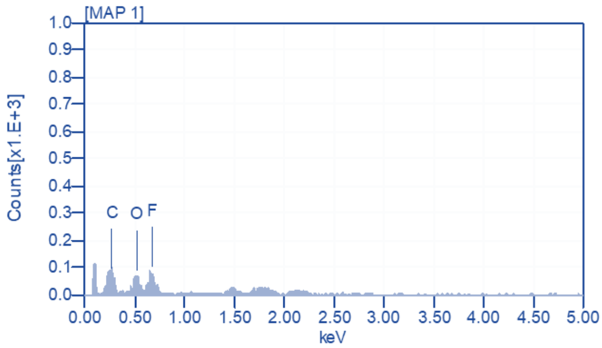

Figure 3. The elemental analysis of the NCC shows Carbon (C), Oxygen (O2), and Flouride (F).

values of the NCC were observed in the Group 2 (TBC) (1558.38 ± 0.022 µm) samples. However, the Group 3 (LLLT -MG (1009.24 ± 0.021 µm) pre-treated group exhibited lower Ra values. The intergroup comparison analysis indicated no statistically significant difference in the Ra levels between Group 2 and Group 4 (ns laser) (1536.31 ± 0.085 µm) ($p > 0.05$). However, it was noticed that the Group 1 (HFA + S) (1395.61 ± 0.021 µm) pre-treated samples exhibited lower SBS scores than Group 2 and 4 yet higher than Group 3 ($p < 0.05$).

Repair bond strength analysis

Figure 4 presented the mean and standard deviation (SD) of the repair bond strength among the NCC pre-treated discs bonded to a composite. The highest bond integrity outcomes were exhibited by the Group 2 (TBC) (18.22 ± 0.19 MPa) pre-treated samples. Whereas the lowest bond strength scores were documented in the Group 2 LLLT-MG (13.18 ± 0.32 MPa) conditioned

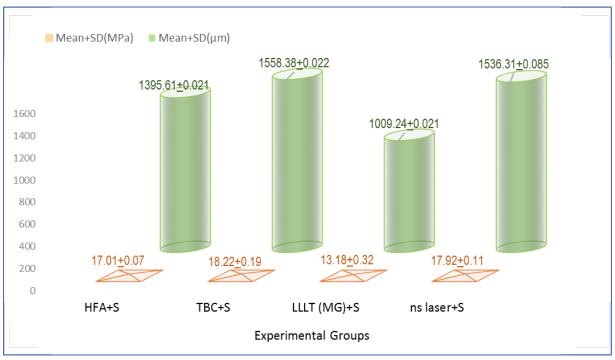

Figure 4. Mean and (SD) of the surface Ra and SBS in the NCC after applying different surfaces. A strong direct correlation exists between the Ra and the repair bond strength.

NCC discs. The comparative evaluation among the different experimental groups discovered that Group 2 and Group 4 (ns laser) (17.92 ± 0.11MPa) achieved comparable bond strength results ($p > 0.05$). Similarly, it was noted that the Group 1 (HFA + S) (17.01 ± 0.07 MPa) conditioned discs exhibited lower repair bond scores than Group 2 and 4 yet higher than Group 3 ($p > 0.05$)

Nature of the fracture

Figure 5 presents the percentage distribution of the modes of failure among different study groups. Groups 1, 2, and 4 exhibited cohesive fracture patterns the most. Whereas, the Group 3 treated specimens displayed all three types of failures.

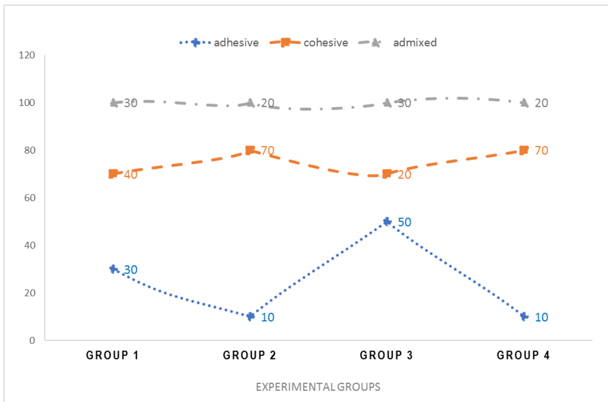

Figure 5. Percentage distribution of the modes of failure.

DISCUSSION

The present-day research was grounded in the assumption that there would be no substantial disparity in the Ra of the NCC when contemporary surface modifiers (TBC, ns laser, and MG-LLLT) were used in comparison to the HFA. Furthermore, it was also predicted that the repair bond strength of composite to the NCC would be comparable to the surface pre-treated with contemporary surface modifiers in comparison to the control. According to the outcomes attained, both the stated suppositions were completely rejected as the TBC and ns laser-treated discs exhibited better Ra and repair bond strength scores than the HFA. Whereas, the LLLT-MG conditioned samples exhibited lower mechanical outcome values.

In the analysis of Ra and bond strength, it was found that the NCC discs treated with the TBC and ns laser demonstrated the highest Ra and repair strength when bonded to the composite. TBC, a modified air abrasion technique, was employed utilising 30-μm silica-coated alumina grit to generate a rough surface pattern [27, 28]. Previous studies have reported that air abrasion can achieve better adhesion than acid etching in the NCC [29, 30]. These outcomes can be explained based on the surface topographic analysis via SEM in the present inquiry. The SEM findings indicated that TBC air abrasion techniques produced rough surface morphology, resulting in the exposure of internal filler aggregates which enhances the mechanical interlocking of the repair material used and eventually the bond strength. In addition to forming micro-interlocking, these unique grits were designed to encapsulate silica particles on the surface, thereby enhancing the surface content of silicon hydroxyl and boosting the efficacy of the silanization through chemical interaction [17]. This is in alignment with the outcomes of the lab-based investigation conducted by Wu and colleagues [31].

Apart from TBC, the ns laser has also demonstrated significant improvement in the Ra and repair bond strength. It has been documented that laser patterning has been shown to offer superior control over topography in comparison to alternative technologies by enabling the structuring and scalability of a variety of geometrical characteristics [8,31,32]. The ns-laser enabled the formation of distinct and reproducible grooves on NCC discs as seen in the SEM analysis. The observed pattern formation encompassed the melting and resolidification of the surface material, as evidenced by the melting tracks, pile-up structures, and the interconnected crack network that emerged across the treated surfaces [20, 32]. This pertains to the mechanism of the photothermal material removal from the surface [14, 33]. This is in concordance with the outcomes of the ex vivo analysis conducted by Pereira et al. [34]. They described a "melted like-topography" characterised by a dented morphology resulting from the thermal cracks that occurred during the melting and recrystallisation processes of ns laser exposure [34]. However, the ns laser is a new concept and more information is required to confirm the findings of existing investigation.

The group treated with HFA showed notably lower Ra and repair bond strength scores compared to both the TBC and ns laser, yet still higher than the LLLT (MG) group. This observation can be explained by the hexafluoro-silicate formation which happens when the HFA interrelates with the NCC matrix [35]. This reaction modifies the surface texture and enhances the surface energy, thereby augmenting the integrity of the adhesive bond [36]. Papadopoulos and co-authors in their in vitro investigation revealed that pre-treating NCC with HFA alters the surface micromorphology, resulting in a pattern of pores and grooves of varying widths [37]. Conversely, Salem and co-workers indicate outcomes that are in contradiction with the findings of existing research [38]. The LLLT alongside MG as a photosensitiser when used as a NCC surface conditioner resulted in lower Ra and repair bond strength scores. A lab-based analysis conducted by Maawadh and colleagues reported similar outcomes in their in vitro analysis [3]. They explained that MG being hydrophilic in nature results in water absorption on the surface of NCC discs which negatively impacts their bonding capacity of composite [39]. The results from the fracture pattern analysis showed that cohesive failures were most commonly observed in the TBC, ns laser, and HFA groups, suggesting that cracks started in a specific part of the material. Conversely, the samples exposed to LLLT (MG) demonstrated all three varieties of fracture patterns. The variety of fracture types indicates that the cement was primarily concentrated at the periphery of the bonding interface. This distribution suggests that stress concentration generally manifests in the central area of the interface between NCC and composite materials [40, 41].

This investigation underscores several inherent constraints. The repair bond strength results, although insightful, may not entirely encompass the intricacies of clinical conditions. Therefore, it is crucial to analyse these results with careful attention. Furthermore, atomic force microscopy (AFM) should have been used to analyse the surface topography of pre-treated NCC discs. In addition, thorough assessments are essential to ascertain the impacts of diverse concentrations of MG and various laser parameters on the Ra and SBS of NCCs.

CONCLUSIONS

In this study, the effect of ground slag and dune sand powA tribochemical silica coating and nanosecond laser can be used as an alternative conditioning regime for nanocomposite ceramics as they positively influence the repair bond strength and surface roughness.

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