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Effect of Pre-Treatment Techniques Nd: YLF laser, Nano-HAp Particles on Surface Roughness and Repair Bond Strength of Different-Viscosity Composite Bonded to Hybrid Ceramics

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Abstract

Evaluating the effect of airborne particle abrasion (APA), Nd: YLF laser, hydroxyapatite nanoparticles (Nano-HAp) on surface roughness (*Ra*), topography, and repair bond strength (RBS) of different-viscosity composites bonded to hybrid ceramic (HBC). 78 CAD/CAM HBC discs were prepared and divided into three groups based on surface treatment (n = 26). Group 1: APA, Group 2: Nd: YLF laser, and Group 3: Nano-HAp coating. A surface profilometer measured the *Ra* scores (n = 5 each). SEM was used for surface topography (n = 1 each). On the remaining samples, an adhesive was applied and cured. Twenty samples from each group were further categorized into two subgroups based on the composite viscosity. Bond strength and failure mode were assessed. A one-way ANOVA and a Tukey test were conducted to identify significant differences among groups. The highest mean *Ra* score was exhibited by Group 2 (Nano-HAp). Group 3 (Nd:YLF laser)-conditioned samples demonstrated the lowest *Ra*. Maximum RBS values were observed for Group 2B (Nano-HAp+Injectable). The minimum bond strength was noted for Nd: YLF laser+Microhybrid. Lower-viscosity composite displayed better performance than conventional composite. The most successful method for surface preparation of HBC involves pretreating with Nano-HAp coating. The use of a low-viscosity injectable composite as the repair material results in enhanced bond strength.

Keywords: Hydroxyapatite, hybrid ceramic, roughness, bond strength, scanning electron microscopy

I. Introduction

CAD/CAM (Computer-aided Design/Computer-aided Manufacturing) restorations made from ceramics are praised for their strong fracture resistance, durability against wear, and attractive appearance [1]. However, it has been noted that traditional ceramics can cause wear on natural teeth [2]. To safeguard the opposing teeth, hybrid ceramics (HBCs) have been introduced [3]. These materials combine the durability and color retention of ceramics with the low-wear properties of resin composites [4]. The fact that HBC does not cause wear on opposing teeth has led to an increased preference for its use in clinical environments. Although CAD/CAM materials are known for their strong mechanical properties, they remain susceptible to breaking under chewing forces or due to distortions that can occur during the milling process [5]. In these cases, repairing damaged restorations with resin composites is preferred because it is both conservative and cost-effective [6]. Two important factors need to be considered when repairing the HBCs with the composite material, i.e. surface conditioning and the viscosity of the repair material.

Among the numerous surface conditioning agents available, the combination of airborne particle abrasion (APA) with a silane coupling agent (S) is often considered the benchmark for improving both the repair bond strength (RBS) and the surface roughness (*Ra*) of HBCs [7]. It utilizes aluminum oxide (Al_2O_3) particles which enhance the *Ra*, improve surface receptivity, and expand the adhesion area [8]. However, the pattern of indentations found on the surface of the sandblasted material changes depending on factors such as the size of the particles, pressure, and duration [6].

Alternative approaches that have gained attention include the use of lasers and nanotechnology. Light Amplification by Stimulated Emission of Radiation (LASER) has long been a method for altering *Ra* [9–11]. In dentistry, picosecond neodymium: yttrium lithium fluoride (Nd: YLF) lasers are utilized for both hard and soft tissue treatments. This system was initially introduced by Niemz et al. for medical purposes [12]. These lasers are renowned for their superior beam quality, especially in pulsed applications, and they provide a variety of lasing wavelengths, such as 1 047 nm and 1 053 nm. Hydroxyapatite nanoparticles (Nano-Hap) have attracted considerable interest from dental researchers due to their remarkable morphological and structural similarities to the apatite crystals in tooth enamel [12]. Research conducted by Almohareb and colleagues demonstrated that applying nano-HAp particles to HBCs enhances bond strength at the cement-ceramic interface. Nonetheless, there is still

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limited literature on the effect of nano-HAp particles on the RBS of HBC to composite restorations, warranting further investigation [13].

Besides the influence of conditioning, the viscosity of composite materials plays a crucial role in determining bond strength during HBC repairs, as it affects how well the material can flow and fill surface irregularities [14]. Earlier studies have indicated that while composite resins with low viscosity can improve marginal sealing when paired with specific adhesive systems, they do not consistently enhance tensile bond strength or prevent leakage at the interface [15, 16] However, there is still no consensus on which viscosity level of composite is ideal for achieving the best performance and increasing patient satisfaction.

The current research endeavor is designed to thoroughly evaluate and contrast the effect of various pre-treatment techniques on *Ra* and RBS of different-viscosity composite repair materials bonded to HBCs. It was anticipated that there would be no significant disparity in the *Ra* scores, surface topography, and RBS of HBC specimens that received surface pretreatments with Nd: YLF laser and nano-HAp particles when compared to those that underwent APA conditioning. Furthermore, it was also predicted that the different-viscosity composite would exhibit no substantial difference in the *Ra* and RBS when used on the same conditioning group.

II. Materials and Methods

1. *Specimen preparation*

The present study encompasses the use of 78 CAD/CAM-manufactured Vita Enamic (Vita Zahnfabrik, Bad Säckingen, Germany) discs [17]. A total of 15 blocks, each with precise dimensions of 10 mm × 10 mm × 6 mm, were meticulously sliced to create the 78 discs utilizing a water-cooled diamond blade paired with a low-speed cutting saw (Isomet, Buehler, Düsseldorf, North Rhine, Germany) [18]. The specimens were then polished using 1 200-grit silicon carbide paper within a polishing apparatus (EcoMet 300 Pro, Buehler). This process was subsequently followed by ultrasonic cleansing (Toption Digital Ultrasonic Cleaner, Shaanxi, China) in distilled water for 5 mins. To facilitate handling and support, each ceramic specimen was encased in a block of self-curing acrylic resin (Acrostone, Cairo, Egypt) [19].

(a) *Group 1: APA*

Specimens were exposed to APA employing 50 µm powder (BEGO Easyblast, Bremen, Germany) under an applied pressure of 290 kPa for a time frame of 20 secs at a distance of 10 mm. Subsequently, all specimens underwent washing followed by air drying [20].

(b) *Group 2: (Nano-HAp coating)*

The protocol for applying nano-HAp thermal coating was executed for the pretreatment of HBCs. A slurry was created by combining 10 g nano-HAp powder (Merck, Berlin, Germany) with 50 ml distilled water. To act as a binder for the suspension, 1 g polyvinyl alcohol (Merck, Berlin, Germany) was utilized. Subsequently, the solution was heated on a magnetic stirrer at a temperature of 100 C while maintaining a rotational speed of 1 000 rpm for 1 min to achieve a uniform suspension. The discs were then immersed in the slurry at a 45-degree angle for 20 sec [13].

(c) *Group 3 Nd: YLF laser*

In this group, a Nd: YLF laser (model 204A, Quantronix, Darmstadt, Germany) was used to condition the HBC disc. The parameters were set at a wavelength of 1.053 µm, pressure of 25 ps, pulse energy of 0.2 nJ, and repetition rate of 160 MHz. The laser was used in contact mode for 20 sec [12].

Following surface treatment, all the samples were treated with silane coupling agent (Ultradent Silane, Ultradent) with the help of a micro brush and allowed to-air dry for 1 min adhering closely to the guidelines provided by the manufacturer [12].

2. *Surface roughness (Ra) analysis*

The *Ra* (n = 5 in each group) of HBC discs was evaluated using a surface profilometer (Surfanalyzer 4000, Federal Products Corp., Providence, RI). The probe of the device operated at a velocity of 2 mm/s, and data was collected from five distinct points at intervals of 1 mm. Subsequently, the arithmetic mean was computed to ascertain the average *Ra* value in µm [21].

3. *Surface topography analysis using SEM*

One specimen from each group underwent surface topographic analysis. The discs were sputtered-coated using gold powder (JFC – 1100 fine coat ion sputter, Osaka, Japan) at 40 mA for 250 s followed by examination under SEM (EVO MA 10, Carl Zeiss, London, UK) at different magnifications [22].

4. *Adhesive and repair resin composite application*

The SE adhesive with the brand name Single Bond Universal adhesive (3M ESPE) was carefully applied to the surface of the discs of 20 specimens from each cohort using a micro brush for a precise duration of 20 secs, after which any excess universal adhesive was meticulously removed with the aid of a new micro brush. The solvent contained within the universal adhesive was removed by allowing the adhesive to air-dry gently for approximately 5 secs. The specimens were then allowed to air-dry until a glossy finish appeared on the surface, indicating that all liquid movement had come to a halt. Following this, the specimen underwent a light curing process for 20 secs using an LED curing light (VALO Ultradent, Utah, USA) [23].

5. *Subgrouping based on composite viscosity*

Each group was further categorized into two subgroups based on the type of composite resin employed (n = 10). Microhybrid composite (A) (G-aenial Universal Posterior, GC Corp, Tokyo, Japan.) and low-viscosity injectable composite (B) (G-aenial Universal Injectable, GC Corp, Tokyo, Japan) were both built on the HDCs discs to a height of 2 mm utilizing a silicone mold, which was subsequently followed by a curing period of 20 secs.

The bonded discs were then immersed in distilled water at a controlled temperature of 37 °C for 24 h.

6. *Artificial ageing*

All samples underwent a thermocycling process in distilled water utilizing a thermocycling apparatus (Julabo FT200, Julabo, Seelbach, Germany) for a total of 5 000 cycles. Each cycle consisted of alternating immersion of samples in water baths maintained between temperatures of – 5 °C and 55 °C for 20 secs. The transfer time between the two immersions was 15 secs [24].

7. *RBS and failure mode analysis*

The bonding strength of the HBC discs that had been repaired with composite was assessed with a universal testing machine (UTM, Dahometer, China). The samples were arranged in such a way that the force applied by the chisel was perpendicular to the composite ceramic interface. The force was gradually applied at a crosshead speed of 1 mm/min until the repair material became detached from the HBC discs. The samples that had debonded were analyzed with a stereomicroscope (Leica EZ4D, Leica Microsystems Ltd, Switzerland; × 40) to determine the nature of the fracture. The different modes of failure were classified into three categories: adhesive, admixed, and cohesive [25,26].

8. *Statistical analysis*

The mean and standard deviation (SD) related to *Ra* and bond strength underwent a one-way analysis of variance (ANOVA) along with a Tukey test to determine the statistically significant differences among the various groups tested ($p = 0.05$). Data normality was assessed using the Kolmogorov-Smirnov test.

III. Results

1. *Surface topography*

Fig. 1: 1A - The application of a HAp coating on HBC results in the formation of micro-retentive regions on the surface, which are likely instrumental in enhancing the mechanical bond strength. Fig. 1B - The use of APA with coarser Al_2O_3 particles increases surface irregularities, thereby enlarging the surface area and creating a crater-like texture on the HBC. The employment of the Nd: YLF laser on HBC does not lead to matrix loss, while surface cracking contributes to a reduction in the *Ra* (Fig. 1C)

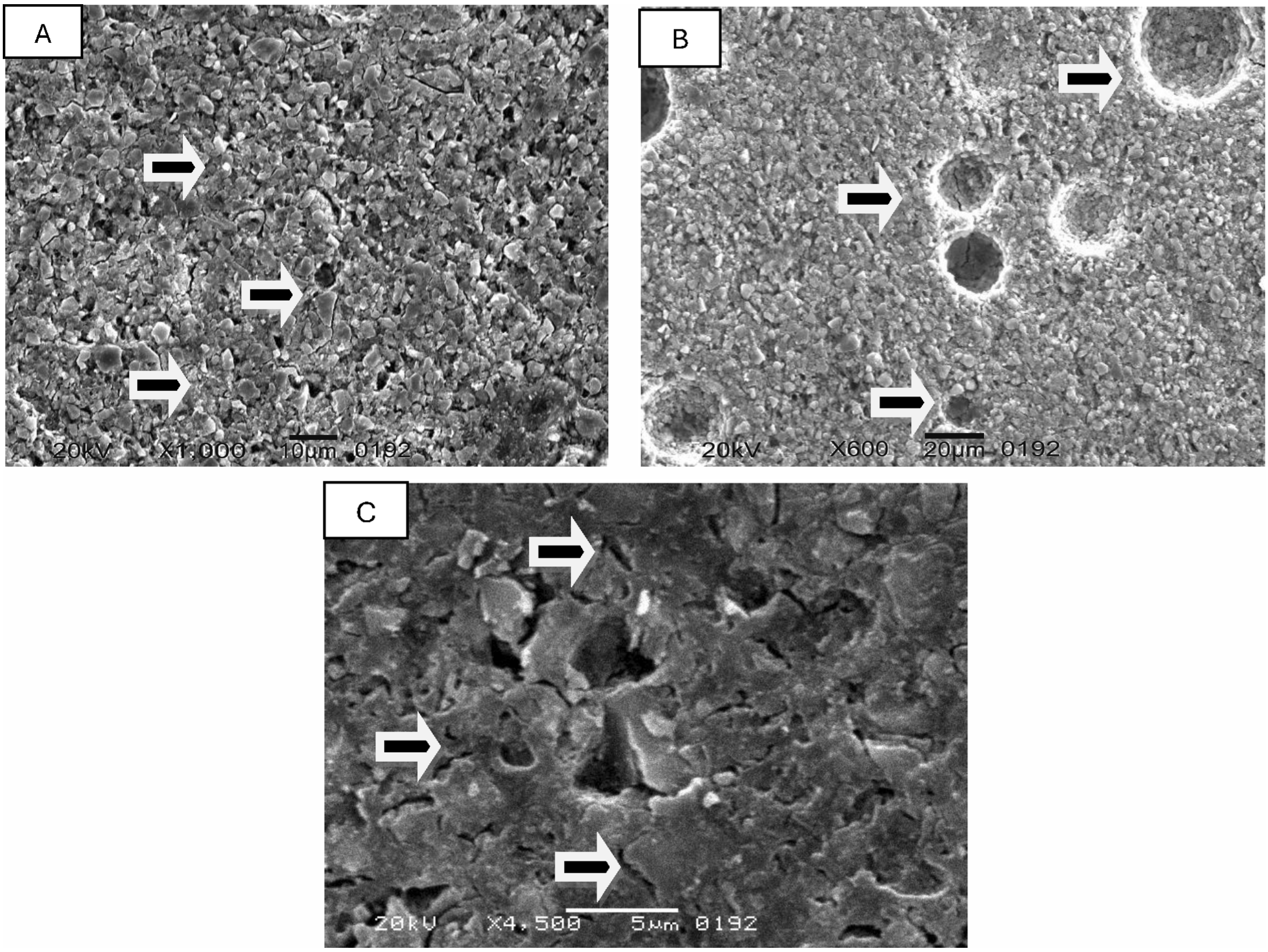

Fig. 1: 1A- HAp coating on HBC creates micro-retentive regions on the surface (shown by arrows), which likely played a significant role in improving the strength of the mechanical bond. 1B- APA employing coarser Al_2O_3 particles led to an enhancement in surface irregularities, which subsequently augmented the surface area giving a crater-like impression on HBC (shown by arrows). Nd: YLF laser on HBC shows no loss of matrix along with surface cracking (shown by arrows) decreasing *Ra*.

Table 1: Surface *Ra* of HBC after conditioning with different surface pretreating protocols

Tested groups	Mean ± SD (μm)	p-value!
Group 1: APA	1 148.23 ± 0.054 [a]	
Group 2: Nano-HAp particles	1 176.44 ± 0.067 [a]	< 0.05
Group 3: Nd: YLF laser	822.18 ± 0.017 [b]	

!ANOVA
Airborne particle abrasion (APA), Nanohydroxyapatite (HAp), Neodymium: Yttrium Lithium Fluoride (Nd: YLF) laser
Different superscript characters denote statistically significant differences (Post Hoc Tukey Multiple Comparison Test)

Table 2: RBS of HBC bonded to different-viscosity composite after pretreating with different conditioning agents

Investigated groups	*Mean ± SD (MPa)*	*p- value!*
Group 1A: APA+ Microhybrid	11.01 ± 0.10 [b]	
Group 1B: APA + Injectable	11.45 ± 0.13 [a]	
Group 2A: Nano-HAp particles + Microhybrid	11.05 ± 0.11 [b]	< 0.05
Group 2B: Nano-HAp particles + Injectable	11.77 ± 0.16 [a]	
Group 3A: Nd:YLF laser + Microhybrid	9.32 ± 0.15 [c]	
Group 3B: Nd:YLF laser + Injectable	9.65 ± 0.18 [d]	

!ANOVA
Airborne particle abrasion (APA), Nanohydroxyapatite (HAp), Neodymium: Yttrium Lithium Fluoride (Nd: YLF) laser
Different superscript characters denote statistically significant differences (Post Hoc Tukey Multiple Comparison Test)

2. *Ra analysis*

The mean and SD of *Ra* among all tested groups are delineated in Table 1. The highest mean *Ra* score was exhibited by Group 2 (Nano-HAp particles) (1 176.44 ± 0.067 μm) pretreated discs. Conversely, Group 3 (Nd: YLF laser) (1 176.44 ± 0.067 μm) conditioned samples demonstrated the lowest roughness outcomes. A comparative analysis revealed that Group 1 (APA) (1 148.23 ± 0.054 μm) and Group 2 presented statistically comparable *Ra* outcomes ($p > 0.05$)

3. *RBS analysis*

The RBS of different-viscosity composite materials, following pretreatment with various conditioning agents are presented in Table 2. Maximum RBS values were observed for Group 2B (Nano-HAp particles + Injectable) (11.77 ± 0.16 MPa). In contrast, the minimum average bond strength was noted for Group 3A (Nd: YLF laser + Microhybrid) (9.32 ± 0.15 MPa). A comparative evaluation among the experimental cohorts revealed that Group 1A (APA + Microhybrid) (11.01 ± 0.10 MPa) and Group 2A (Nano-HAp particles + Microhybrid) (11.05 ± 0.11 MPa) exhibited no statistically significant differences in bond strength parameter ($p \leq 0.05$). Likewise, Group 1B (APA + Injectable) (11.45 ± 0.13 MPa) and Group 2B also demonstrated no significant difference in bond strength outcomes. ($p > 0.05$) Nevertheless, Groups 3A and 3B (Nd: YLF laser + Injectable) (9.65 ± 0.18 MPa) exhibited significantly dissimilar bond strength values ($p < 0.05$).

4. *Failure type analysis*

In the context of failure modes, it was discerned that the APA and Nano-HAp particles predominantly revealed a mixed failure type. In contrast, the Nd: YLF laser cohort demonstrated a significant prevalence of adhesive type of failure.

IV. Discussion

The present inquiry was based on the hypothesis that there will be no significant disparity in the *Ra* scores, surface topography, and RBS of HBC that received surface treatments with Nd: YLF laser and nano-HAp particles when compared to SB conditioning. Furthermore, it was also predicted that the different-viscosity composite would exhibit no substantial differences in the *Ra* and RBS when used on the same conditioning group. The findings derived from the current research indicated that the *Ra* score along with the bond strength of nano HAp coatings pretreated discs were similar to that of the control group when they were bonded using both high- and low-viscosity resin composite thereby partially disproving the first null hypothesis. However, Nd: YLF laser displayed significantly lower scores of *Ra* and SBS when compared to the control. Furthermore, it is essential to highlight that the second hypothesis was completely dismissed, as the groups utilizing low-viscosity resin composite to repair HBC presented the highest bond integrity outcomes.

With regard to the *Ra* and RBS of different tested groups, the nano-HAp coating exhibited the highest scores for *Ra* and bond strength which was comparable to the control group. The use of HAp coating in implant dentistry has a long history, as it serves to improve the attachment of surrounding cells to the surface of the implant [27,28]. The favorable *Ra* results observed when HBC discs are treated with nano-HAp particles align with the conclusions drawn from a laboratory study carried out by Almohareb *et al.* [29] SEM images of the surface topography assessment after conditioning with Nano HAp particles showed results that correspond with the *in vitro* analysis performed by Jung and his team [30]. In their study they revealed that HAp coating creates micro-retentive regions on the surfaces of ceramics, which likely played a significant role in improving the strength of the mechanical bond (Fig. 1A). Likewise, the findings of the existing investigation confirmed that specimens subjected to APA using Al_2O_3 also exhibited higher bond strength values. This phenomenon can be elucidated by the fact that APA employing coarser alumina particles led to an enhancement in surface irregularities, which subsequently augmented the surface area available for adhesion with the restorative bonding material, thereby enhancing the micro-mechanical retention and eventually the RBS (Fig. 1B) [31,32]. The findings were further justified with an SEM image which indicated that APA creates a textured surface with visible pits, grooves, and craters (Fig. 1B). Apart from both techniques, Nd: YLF laser was also used in the existing inquiry and displayed significantly lower surface irregularities and bond integrity. This laser is most closely related to the Nd: YAG laser as both utilized neodymium-doped crystals. While they share a similar active medium and operational principles the ablation experiments conducted by Niemz and his colleagues utilizing the Nd: YLF laser demonstrate that the process of ablating tooth structure was approximately ten times more effective, establishing that Nd: YLF laser selectively targets caries [12] and its effect on ceramics is minimum. SEM analysis revealed that HBC discs had no surface cracks with no loss of matrix (Fig. 1C).

Regarding composite repair material, it has been documented in the existing body of evidence that healthcare professionals often choose low-viscosity composites due to their exceptional ability to thoroughly penetrate fracture surfaces [22]. Comparable findings were reported in the ongoing investigation, where low-viscosity injectable composites exhibited a notable enhancement in restoring the bond strength in contrast to micro-hybrid composites, irrespective of the pretreatment method employed [13,33]. Kemaloğlu and colleagues validated these findings with their experimental laboratory research analysis. The observed impact can be ascribed to the low viscosity, enhanced wettability, the creation of a broad free surface, and the minimal shrinkage at the adhesive interface. Collectively, these elements allowed the injectable composite to attain superior adaptation to the HBC discs, thereby facilitating a more effective repair procedure [26].

In the context of identifying failure modes, it was observed that the nano-HAp coatings and APA presented the highest frequency of mixed fracture occurrences. The diversity in fracture types implies that the composite was predominantly situated at the peripheral edges of the bonding interface. This arrangement indicated that stress concentration typically developed in the central region of the HBCs composite interface.[34] This finding is consistent with the outcomes of laboratory-centered investigations carried out by Kılınç *et al.* [35]. On the other hand, the Nd: YLF laser presented adhesive failures the most.

The present research exhibited some intrinsic limitations. Primarily, this examination was conducted within a controlled laboratory environment; therefore, any inferences made from the findings ought to be regarded with caution in the clinical scenarios. The lack of surface characterization methodologies, such as Raman spectroscopy and X-ray photoelectron spectroscopy could impede a more comprehensive understanding.

V. Conclusion

1. For the restoration of CAD/CAM hybrid ceramic materials, the most effective approach for surface conditioning involves pretreatment with hydroxyapatite nanoparticles.
2. Regardless of the chosen surface treatment option, an increase in bond strength is achieved when a low-viscosity injectable composite is utilized as the repair material.

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Ethical Statement: The research experiments conducted in this article were approved by the Ethical Committee and responsible authorities of our research organization(s) following all guidelines and regulations.

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