Journal of
Ceramic Science
and Technology

J. Ceram. Sci. Technol., **16** [4] 239-245 (2025)
DOI: 10.4416/JCST2025-00003
available online at: http://www.ceramic-science.com

Effect of Pretreatment Methods and Composite Repair Materials on the Repair Bond Strength of Resin Nanoceramics: An *In Vitro* SEM Analysis

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received February 14, 2025; received in revised form May 13, 2025; accepted May 18, 2025

Abstract

AIMS To investigate the effect of different conditioning regimes, that is diamond bur (DB) grinding, sandblasting (SB), and Er, Cr: YSGG laser (ECL) at different pulse modes (long and short), on the surface roughness (*Ra*) and repair bond strength (RBS) of resin nanoceramics (RNCs) to different viscosity composites.

METHODS 104 RNC discs were prepared and classified into four groups based on the surface pretreatment protocol (n = 26) Group 1 DB, Group 2 SB, Group 3 (ECL-short pulse), and Group 4 (ECL-long pulse). Five discs from each group underwent Ra assessment. SEM was used to assess the surface topographic changes. Twenty samples from each cohort were divided into two subgroups based on the composite types: micro-hybrid composite and low-viscosity injectable composite. ANOVA and Tukey HSD test were used to analyze the statistical difference among different groups tested for *Ra* and RBS.

RESULTS The highest value for *Ra* was observed in the SB group. The lowest *Ra* was indicated in the ECL-long pulse group. Maximum RBS was observed in Group 2B (SB+Injectable). The lowest scores were displayed by Group 4A (ECL (long pulse)+micro hybrid)

CONCLUSION SB and ECL along with low-viscosity injectable composite can be considered suitable surface conditioners and restorative materials for repairing fractured resin nano-ceramic.

Keywords: Diamond bur, sandblasting (SB), surface roughness, repair bond strength, SEM

I. Introduction

In the dental field, contemporary ceramics, primarily categorized as resin nano ceramics (RNCs), have attracted significant attention due to their remarkable flexibility and superior load-absorbing properties [1,2]. Research by Suksuphan *et al.* has demonstrated that RNCs can endure forces up to 2000 N without exhibiting surface cracks or restoration failure [3]. However, Yli-Urpo and associates have indicated that the load-bearing capacity of RNCs is relatively low compared to other traditional ceramics, such as lithium disilicate ceramics, hybrid ceramics, and feldspathic ceramics [4] Current literature suggests that repairing indirect restorations is preferable to replacement, as it is more cost-effective and economical [5,6]. This finding has underscored the crucial role of surface treatment techniques in successfully repairing RNC restorations [6,7].

Among the various methods employed, mechanical surface pre-treatment techniques such as diamond bur (DB) grinding and sandblasting (SB) are considered the most commonly used approaches for conditioning resin-based nanocomposites (RNCs) [8]. DB mechanical preparation enhances the shear bond strength (SBS) of hybrid ceramic bonded to resin by improving substrate wettability and roughness through the creation of micro-retentive grooves, resulting in increased resin cement interlocking with the surface [9,10]. Research suggests that grinding diminishes the fracture strength of dental ceramics [11,12]. Conversely, sandblasting (SB) using aluminum oxide (Al_2O_3), developed in 1945 to reduce the pain and discomfort associated with caries removal, has also found applications in prosthodontics. It improves surface roughness (*Ra*) for better bonding with various types of cement and restorative materials [13,14]. SB devices generate a high-speed stream of Al_2O_3 particles that impact dental struc-

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tures and materials [15]. The efficacy of this process depends on the mechanical properties of the target structure and the operational parameters of the device, including air pressure, particle size and hardness, application angle, distance from the target, and duration [13,16].

Research in this domain has shown that Er, Cr: YSGG laser (ECL) treatment could be a practical clinical alternative for chairside restoration of RNC with composite. A laboratory investigation examined the influence of ECL irradiation on the RBS of RNCs with composite, producing favorable outcomes [17,18]. Nonetheless, it has been emphasized that laser parameters are crucial for achieving successful results [19,20] At present, there is no consensus regarding the optimal parameters for conditioning hybrid ceramics. An investigation by Alperen *et al.* demonstrated that employing ECL in long-pulse and short-pulse modes results in differing bond strengths between composite materials [21].

The composite type plays a crucial role in addition to the surface pretreatment method. Soares *et al.* identified a notable gap in the current research on repairing RNCs [22]. Their study emphasized that while surface treatment received extensive focus, the potential impact of the repair material was largely neglected. Their findings revealed that newer injectable composites demonstrated superior performance compared to conventional composites in achieving higher RBP of the bond to the hybrid ceramic [22]. Nevertheless, data on this topic remains scarce, necessitating further investigation.

This research aims to comprehensively investigate and compare the effect of different pre-treatment methods and composite repair materials on the RBS of RNCs. The researchers of the present research have hypothesized that no significant variations would be observed in the *Ra* and bond strength of RNC discs treated with ECL and SB when compared to those conditioned with DB. Additionally, it was expected that the RBS of the micro-hybrid composite would be comparable to that of the low-viscosity injectable composite within the same conditioning group.

II. Materials and Methods

(1) Preparation of samples

104 RNC discs were prepared by sectioning Vita Enamic CAD-CAM blocks (VITA Zahnfabrik H. Rauter GmbH & Co. KG). Each disc had dimensions of 2 mm in diameter and 8 mm in thickness, respectively. The discs were prepared using a diamond blade in a slow-speed saw (Isomet, Buehler, Düsseldorf, Germany) under constant water irrigation. The samples were rooted in self-cure acrylic resin within a polyvinyl pipe, which was followed by polishing using 600 – 1200 grit SiC papers. Based on the conditioning regimes the discs were classified into four groups (n = 26) [11].

Group 1 (DB): The discs in this group were ground using a 30-µm grit diamond bur applying ten strokes at 40 rpm under constant water cooling [23].

Group 2 (SB): 110-µm Al_2O_3 particles (Ney; Blastmate II, Yucaipa, CA) were used to SB the discs at 2.5 bar of pressure for 10 secs, maintaining a distance of 10 mm for 15 secs [15].

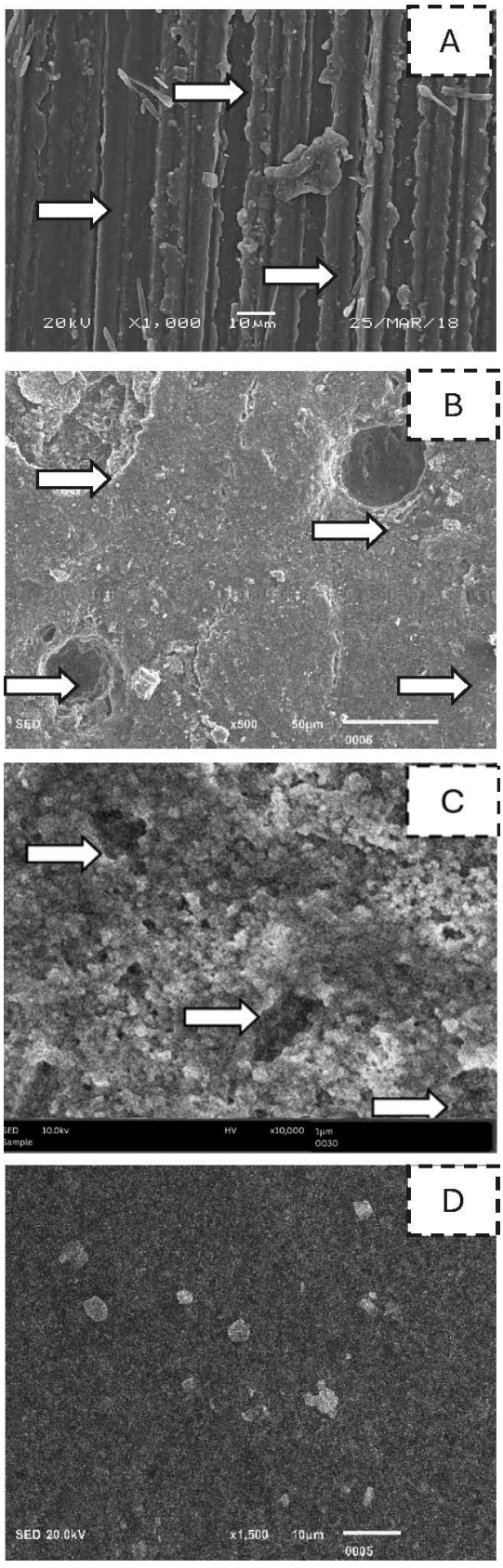

Fig. 1: The SEM image in Fig. 1A shows RNC treated with a diamond bur, revealing grooves (indicated by arrows). Fig. 1B's micrograph illustrates significant surface irregularities and hollowness resulting from sandblasting the RNC (see arrows). In Fig. 1C, RNC discs pretreated with short-pulse laser mode exhibit notable roughness and surface void (see arrows). Conversely, the SEM micrograph in Fig. 1D displays ECL treated with long-pulse laser mode, showing an unchanged surface.

Group 3 (ECL-short pulse): The ECL 600-µm-diameter fiber tip was used in H mode at 60 µs pulse duration at a power output of 6 W, with a frequency set at 20 Hz for 20 secs. The distance between the tip and the discs was maintained at 1 mm [24].

Group 4 (ECL-long pulse): The ECL 600-µm-diameter fiber tip was used in S mode at 700 µs pulse duration at the power output of 6 W, with a frequency set at 20 Hz for 20 secs. The distance between the tip and the discs was maintained at 1 mm [25,26]. The discs in all the groups were cleaned in an ultrasonic bath, and then carefully dried using a paper towel.

(2) Ra assessment

Five discs from each group underwent *Ra* assessment using a profilometer (Suftest SJ-201, Mitutoyo Corp. Japan). Measurements were conducted at three distinct locations using a probe positioned at the center of each disc. The traversing length was set to 0.8 mm, and the measuring speed was maintained at a constant rate of 0.5 mm/sec. The average *Ra* for each specimen was determined [27].

(3) SEM analysis

The surface topography was assessed using one disc from each pretreatment group under SEM (Keyence VK-X100, Osaka, Japan). The discs were gold-sputtered (SPI-Moduletm, SPI Supplies, USA) and observed under SEM at 2000x magnification to determine any changes [28,29].

(4) Adhesive application

Universal adhesive (G-Premio Bond) was smeared on 20 samples from each group using a micro-brush and air-dried with light air pressure. Photo-polymerization was performed using LED curing light (D-Light Pro, GC Corporation, Tokyo, Japan), with the tip being kept 10 mm away from the substrate for 10 secs. Twenty samples from each cohort were divided into two subgroups based on the composite used. ($n = 10$). Microhybrid composite (A) (G-Aenial Universal Posterior, GC Corp) and low-viscosity injectable (B) composite (G-Aenial Universal Injectable GC Corp) were built in 2-mm thickness using a silicon mold followed by curing for 20 secs. The restored discs were subsequently placed in distilled water inside a 37 °C incubator for 24 hours before the repair bond strength test was conducted.

(5) RBS and failure mode assessment

The Universal Testing Machine (Lloyd instruments Ltd, Fareham, UK) was used for evaluating the RBP. The discs were placed perpendicular to the long axis of the jig. The load cell of 5 kN was used to apply force at a rate of 1.0 mm/min until the time fracture occurred. The load was measured in Megapascals (MPa). The failure mode was evaluated under a stereomicroscope (TM-505, Mitutoyo, Tokyo, Japan) which was categorized as adhesive, cohesive, or admixed.

(6) Data analysis

The Shapiro-Wilk test was performed to assess the data normality. One-way analysis of variance (ANOVA) and the Tukey HSD test were used to analyze the statistical difference among different groups tested for *Ra* and RBS *($p = 0.05$)*

III. Results

(1) Ra analysis

Ra scores of RNCs following treatment with different surface conditioners are reported in Table 1. The highest values were observed in Group 2 (SB) (1341.32 ± 0.076 µm). However, the lowest *Ra* was indicated in Group 4 (Group 4: ECL- long pulse) (1086.67 ± 0.091 µm). Comparative analysis among investigated groups revealed that Group 1 (DB) (1321.28 ± 0.045 µm), Group 2, and Group 3 (ECL-short pulse) (1337.22 ± 0.054 µm) exhibited comparable scores for Ra. *($p > 0.05$)* Group 4 on the other hand exhibited significantly lower values for *Ra ($p < 0.05$)* (Fig. 2).

Table 1: Evaluating the impact of various conditioning methods on the surface roughness (*Ra*) of resin-based nanoceramic materials (RNCs).

Tested groups	*Mean ± SD (µm)*	*p- value!*
Group 1: DB	1 321.28 ± 0.045[a]	*< 0.05*
Group 2: SB	1 341.32 ± 0.076[a]	
Group 3: ECL-short pulse	1 337.22 ± 0.054[a]	
Group 4: ECL- long pulse	1 086.67 ± 0.091[b]	

! ANOVA
Diamond bur (DB), Sandblasting (SB), Er,Cr:YSGG laser (ECL)
Different superscript small alphabet denotes statistically significant differences! (Tukey Post Hoc Test)

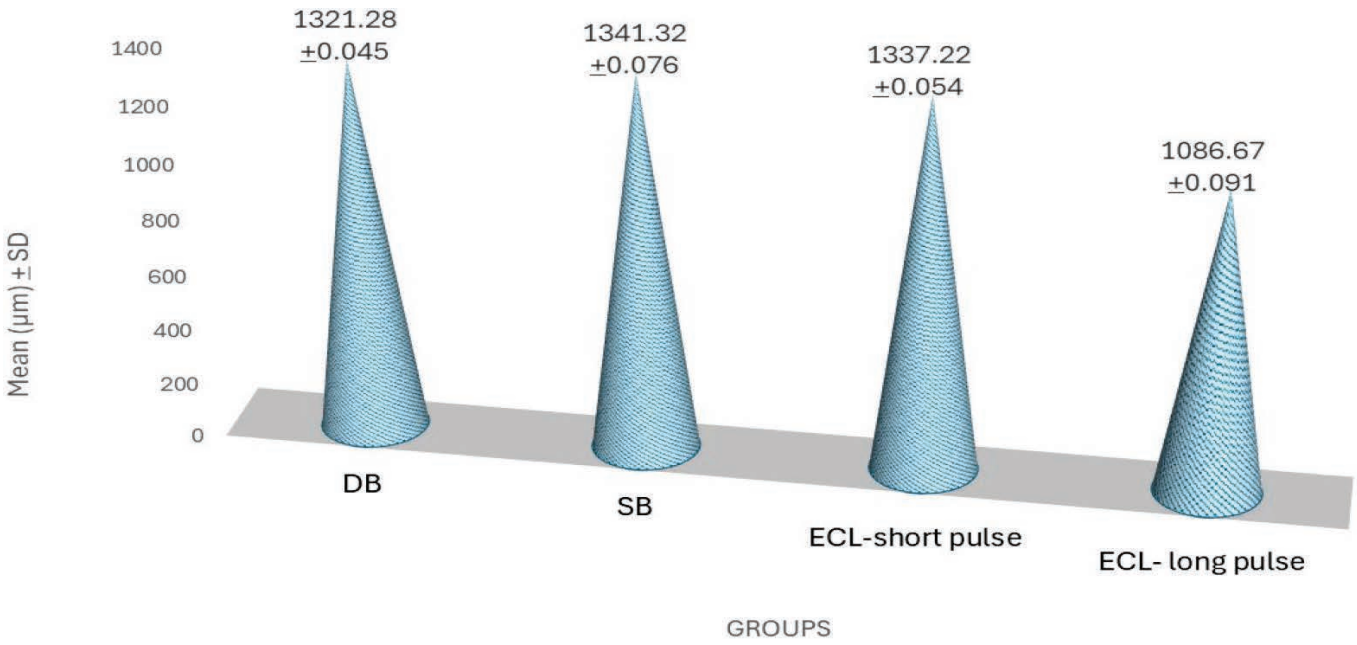

Fig. 2: *Ra* of resin nanoceramics after conditioning with different regimes.

(2) RBS analysis

The RBS of RNCs bonded to different-viscosity composites after conditioning with different regimes are shown in Table 2. Maximum RBS was observed in Group 2B (SB + Injectable) (15.98 ± 0.12 MPa). However, the lowest scores were displayed by Group 4A (ECL (long pulse) + Microhybrid) (13.31 ± 0.11 MPa). Comparative analysis among investigated groups revealed that Group 1B (DB + Injectable) (15.83 ± 0.09), Group 2B (SB + Injectable) (15.98 ± 0.12 MPa) and Group 3B (ECL-short pulse + Injectable) (15.82 ± 0.13 MPa) exhibited no significant difference in RBS. *($p > 0.05$).* Likewise, Group 1A (DB + Microhybrid) (14.29 ± 0.19 MPa), Group 2A (SB + Microhybrid) (14.49 ± 0.23 MPa), and Group 3A (ECL-short pulse + Microhybrid) (14.38 ± 0.13 MPa) also demonstrated comparable bond integrity *($p > 0.05$)* However, Group 4A (ECL-long pulse + Microhybrid) (13.11 ± 0.11 MPa) and Group 4B (ECL-long pulse + Injectable) (13.84 ± 0.18) presented significantly different values for bond strength *($p < 0.05$)* (Fig. 3).

(3) Failure mode analysis

Regarding the failure mode, it was noted that the ECL at short pulse and DB groups often demonstrated mixed types of failures. However, the SB group shows a significant prevalence of cohesive failures. ECL-long-pulse-treated samples exhibited adhesive failure predominantly (Fig. 4).

Table 2: Repair Bond Strength of resin nanoceramics (RNCs) attached to composites of varying viscosities following diverse conditioning protocols.

Tested groups	*Mean ± SD (MPa)*	*p- value!*
Group 1A: DB+ Microhybrid	14.29 ± 0.19 [b]	*< 0.05*
Group 1B: DB + Injectable	15.83 ± 0.09[a]	
Group 2A: SB + Microhybrid	14.49 ± 0.23[b]	
Group 2B: SB+ Injectable	15.98 ± 0.12[a]	
Group 3A: ECL (Short pulse)+ Microhybrid	14.38 ± 0.13[b]	
Group 3B: ECL (Short pulse)+ Injectable	15.82 ± 0.13[a]	
Group 4A: ECL (long pulse)+ Microhybrid	13.11 ± 0.11[c]	
Group 4B: ECL (long pulse)+ Injectable	13.84 ± 0.18[d]	

Diamond bur (DB), Sandblasting (SB), Er,Cr:YSGG laser (ECL)
Different superscript characters denote statistically significant differences!

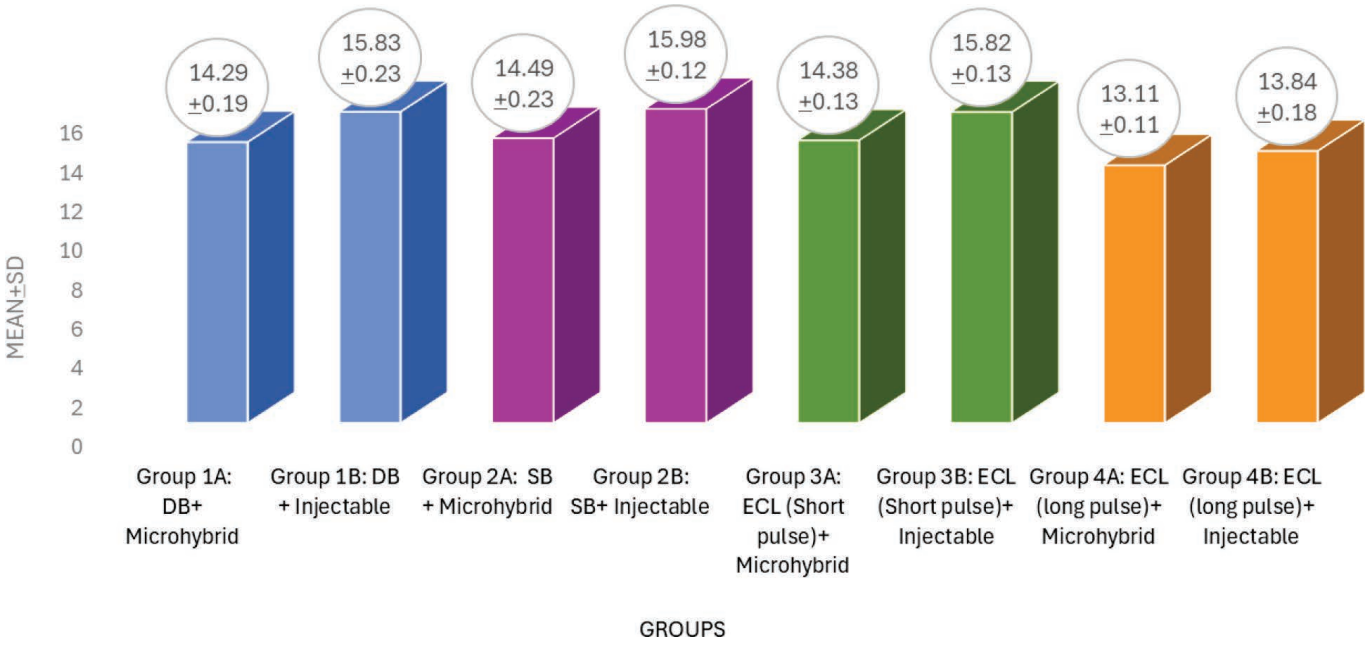

Fig. 3: RBS of resin nanoceramics bonded to different-viscosity composites after conditioning with different regimes.

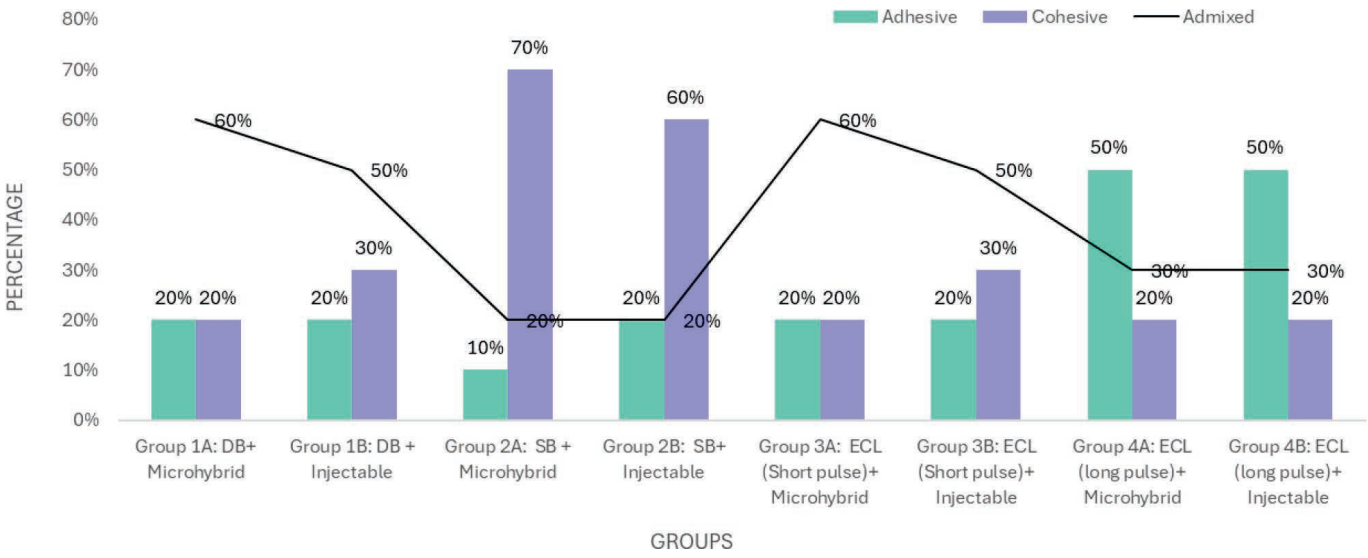

Fig. 4: Modes of failure distribution in different experimental groups.

IV. Discussion

This study assumed that no notable variations would be observed in the bond strength and *Ra* of ECL when using different pulse modes (long and short) and SB-treated RNC discs compared to DB-conditioned discs. Additionally, it was expected that the RBS of the micro-hybrid composite would be comparable to that of the low-viscosity injectable composite within the same conditioning group. The main proposed hypothesis was partially confirmed, as only ECL utilized in the long pulse mode showed notably reduced *Ra* scores and bond strength compared to DB. However, the secondary hypothesis was entirely disproven, as the low-viscosity injectable composite demonstrated significantly superior performance than its higher-viscosity micro-hybrid counterpart. The shear bond test was performed in the present study as it is commonly accepted as a standard technique for evaluating bond strength, given that shear stresses are believed to be important contributors to the failure of restorative materials' bonding in real-world conditions [30,31].

The current body of research consistently underscores the significance of creating irregularities on the RNCs' surface with diverse surface modification techniques or chemical processes [32,33]. In their research, Elkassaby and colleagues stressed the necessity of assessing the appropriate surface conditioning methods that are preferred by dentists in their routine clinical practice [34]. According to the findings of the present research analysis, it was found out that DB grinding, SB, and ECL in short-pulse mode resulted in the highest and comparable bond strength outcomes [34]. The satisfactory performance of DB can be better understood after examining the SEM images which

have shown the presence of vertical grooves on the RNC substrate. This increase in surface irregularities enhances the surface area for better composite adhesion and formation of resin tags. A similar presentation was observed in an *in vitro* investigation piloted by Bahadır *et al.* [35] Likewise, SB with 110 µm alumina particles resulted in elevated *Ra* and RBS measurements. This finding aligns with the *in vitro* study conducted by Ghorab *et al.* [9] The observed effect can be attributed to the use of larger alumina particles during air abrasion, which creates more pronounced surface irregularities. These increased surface irregularities effectively expand the area available for bonding with the cementing material, thus enhancing the micro-mechanical characteristics [36, 37].

ECL demonstrated significantly different outcomes when utilized in various pulse modes, with the short-pulse mode outperforming the long-pulse mode. This can be justified from the SEM images which show considerable roughness in the short-pulse laser mode pretreated discs. Bahadır and Bayraktar noted that "SEM images of short-pulse laser-irradiated surfaces display irregularities and features similar to those associated with melting" [35]. In contrast, ECL in long-pulse mode displayed lower scores for *Ra* and RBS than all the other tested groups. It can be seen in SEM images that ECL in long-pulse laser mode presented unaltered surface islands, which supports the RBS data obtained. This is in agreement with the findings of *in vitro* analysis by Degirmenci and coworkers [21].

In the field of dental restoration repair, various composites with differing viscosities are employed. Many practitioners prefer low-viscosity composites due to their superior ability to penetrate crack surfaces [38]. However, research on the impact of composite viscosity as a repair material for different ceramics is limited. The present study's findings revealed that low-viscosity injectable composite significantly outperformed micro-hybrid composite in RBS, regardless of the pretreatment method used. These results align with those of Kemaloğlu *et al.*, who concluded that lower-viscosity composites are the ideal choice for repairing RNCs, leading to substantially improved bond integrity scores [39]. The injectable composite's low viscosity and high wettability facilitated the creation of a larger free surface. Consequently, the reduced shrinkage at the adhesive interface and decreased shrinkage stresses allowed the injectable composite to better adapt to the restoration surface, resulting in a more effective repair process [39].

Regarding failure mode, it was noted that the ECL at short pulse and DB groups often demonstrated mixed types of failures. However, the SB group shows a significant prevalence of cohesive failures. ECL-long-pulse-treated samples on the other hand exhibited adhesive failure predominantly. The elevated incidence of cohesive failures in the SB group is presumably attributable to the augmented filler material content, leading to a more brittle performance of the evaluated composite [40]. Conversely, mixed failures are prevalent in the ECL-at-short-pulse- and DB-pretreated groups suggesting that laser can produce an adequate adhesive surface, as evidenced by other investigations [41, 42].

Several significant limitations are present in this research. Primarily, the study's execution in a controlled laboratory setting may restrict the generalizability of its findings to practical, real-world applications. The exclusive use of a single RNC type could have constrained the study's results. Additionally, the preparation of specimens should have better replicated clinically relevant anatomical structures. Moreover, the research fails to address the potential impact of saliva contamination on RBS during intraoral applications. As a result, additional *in vitro* and *in vivo* investigations are required to thoroughly evaluate all these aspects.

V. Conclusions

For repairing fractured resin nano-ceramic, appropriate surface conditioners and restorative materials may include SB and ECL@ short pulse, with low-viscosity injectable composite. These options can be deemed suitable for this purpose.

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