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Experimental Evaluation of Different Dentin Sandblasting Protocols in Restorative Dentistry

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Abstract*—The long-term stability and the aesthetic enhancement of dental restorations are primary concerns in clinical practice, driving ongoing research into advanced dental materials and clinical procedures. This study presents a novel approach to evaluate the effects of different sandblasting protocols on dentin surface properties in restorative dentistry. Human dentin samples were treated with aluminum oxide (Al_2O_3) or calcium carbonate ($CaCO_3$) abrasives and analyzed at three key stages of surface preparation for dental restorative treatments: post-sandblasting, post-rinsing, and post-etching with orthophosphoric acid. A multi-analytical approach, including Scanning electron microscopy (SEM), Raman spectroscopy, and surface roughness analysis was used to comprehensively assess morphological and chemical changes due to different treatments. Results demonstrated that Al_2O_3 significantly increased surface roughness, creating a highly textured morphology enhancing, thus, micromechanical interlocking with restorative materials. In contrast, $CaCO_3$ sandblasting resulted in a less pronounced increase in roughness, potentially offering a gentler approach to surface preparation. The application of orthophosphoric acid effectively etched the dentin surface, leading to further modifications in roughness and surface chemistry. Nonetheless, Al_2O_3 particles were not completely removed, contrasting with the complete absence of $CaCO_3$ residues. Raman spectroscopic analysis revealed changes in the intensities of characteristic peaks associated with hydroxyapatite and collagen, reflecting alterations in dentin composition. These findings provide insights into optimizing sandblasting protocols for improved dental restorations.

***Index Terms*—Raman spectroscopy, Material Characterization, Roughness, Restorative Dentistry, Dentin Treatment, sandblasting.**

I. Introduction

Restorative dentistry is the branch of dentistry that focuses on the diagnosis and treatment of conditions affecting the teeth and their supporting structures, with the goal of restoring function, aesthetics, and oral health, while preserving natural tooth structure and preventing further damage. The aesthetic enhancement and the long-term stability of restorations are primary concerns in clinical practice, driving ongoing research into advanced dental materials and clinical procedures [1]. Adhesive materials are now widely used in prosthetic and restorative dentistry due to their ability to improve restoration retention, enhance aesthetics, and reduce the need for more invasive procedures, demonstrating effective mechanical properties and adhesive capabilities [1], [2]. However, failures at the adhesive interface, manifesting as resin-dentin debonding, restoration detachment, or secondary caries, can compromise the longevity of bonded restorations [3]. These failures arise from a complex interplay of factors, including exposure to the oral environment, the specific adhesive protocol employed, the characteristics of the restorative material, the quality of the restoration, and the condition of the residual dentin [4]. Achieving durable adhesion requires careful preparation of the dental surface to create optimal bonding conditions. This typically involves mechanical and chemical treatments to remove damaged tissue and condition the dentin for bonding, such as rotary instrumentation (burs) and acid etching. While these are standard practices, they can alter the chemical and physical properties of the dentin [5]. Consequently, various adhesion protocols, alternative materials, and substrate pretreatment procedures have been investigated to enhance bond strength and improve the restoration outcome [6]. Both chemical and mechanical dentin pretreatments aim to increase surface roughness, potentially improving adhesion by expanding the contact area between the dentin and the adhesive [7]. Following these pretreatments, a bonding agent is applied, forming an intermediary layer between the dentin and the restorative material. Adhesion occurs through a complex exchange process in which minerals removed from dental tissues are replaced by resin monomers. Upon polymerization, these monomers chemically bond and micromechanically interlock within the created porosities, forming a stable hybrid layer [8]–[10]. Dentin bonding is particularly challenging compared to enamel due to its lower inorganic content [11]. Indeed, the enamel, the outermost layer of the tooth crown, is the hardest, most mineralized, and acellular tissue in the human body, mainly composed of over 96% inorganic hydroxyapatite (HA) crystals and approximately 4% water and organic material. In contrast to enamel, dentin is less mineralized (approximately 70% mineral by weight) and more elastic due to its composition of

approximately 20% organic matrix (primarily type I collagen) and 10% water. While both tissues contain HA crystals, those in dentin are arranged in a less dense structure, contributing to its increased elasticity and fracture resistance [12]. The demineralized dentin collagen matrix plays a crucial role in resin infiltration and hybrid layer formation, both of which significantly impact bond strength [13]. Recent research has investigated air abrasion, an alternative technique that utilizes high-energy sandblasting to prepare cavities while preserving the chemical properties of dentin [14], [15]. Although this method effectively maintains substrate integrity, ensuring thorough and safe cleaning of the treated surface remains a critical challenge.

In restorative dentistry, achieving durable adhesion to dentin is crucial for the long-term success of dental restorations. This necessitates meticulous surface preparation to optimize bonding conditions. The preliminary study proposed a measurement approach to investigate the effects of sandblasting, a promising alternative to traditional methods, on dentin surface properties. Specifically, by coupling scanning electron microscopy (SEM), Raman spectroscopy, and roughness analysis, it compares the impact of sandblasting with two different abrasive media, namely aluminum oxide (Al_2O_3) and calcium carbonate ($CaCO_3$), on dentin.

Raman spectroscopy, a powerful technique for probing molecular vibrations, provides valuable insights into the chemical composition and structure of materials. This simple, reproducible, non-destructive, and non-invasive analytical method has diverse applications, ranging from established fields like chemistry and materials science to emerging areas such as biology [16]–[18]. Specifically, Raman spectroscopy offers precise information on dental tissues composition, including mineral concentration and crystallinity changes. It enables the detection and characterization of structural alterations in dentin, such as mineral loss or collagen property changes [19]. The Raman spectrum of dentin exhibits distinct vibrational bands corresponding to its mineral, carbonate, and organic components. Phosphate groups, the primary mineral component, are characterized by bending bands (ω_2 and ω_4 modes) at 430-450 cm^{-1} and 579-608 cm^{-1}, respectively. However, the most prominent peak at 960 cm^{-1} is assigned to the symmetric stretching vibration (ω_1) of the phosphate group (PO_4^{3-}) within HA ($Ca_{10}(PO_4)_6(OH)_2$). The organic components, primarily collagen, are identified by characteristic vibrational bands. The amide III peak, associated with peptide bond vibrations, appears around 1242 cm^{-1}, while the amide I peak, related to C=O stretching vibrations in peptide bonds, is found at 1660 cm^{-1}. These organic signatures are crucial for dentin's structural and functional integrity. In addition, in order to evaluate the structure integrity of the analyzed dentin, SEM images were collected. SEM is a fundamental tool for analyzing microstructural morphology and chemical composition, widely employed in different fields including medicine and dentistry. In particular, in dentistry, SEM is used to visualize dentinal tubules, smear layers, and root canal filling materials, as well as to assess enamel lesions and the dentin surface following different rotary instruments and techniques. It is particularly valuable for evaluating gaps between filling materials and dentin walls [20], [21]. When SEM is combined with energy-dispersive X-ray spectroscopy (EDS) it allows topographic and compositional analysis before and after treatment. For instance, EDS allows to study mineralized tissues, both normal and pathological, thus examining tissue responses to implants [15], [22]. The assessment of surface roughness is critical for evaluating material properties across various scientific fields. In dentistry, optical profilometry has been used to compare the effectiveness of different enamel abrasion polishing systems [23]. Similarly, SEM enables high-resolution roughness analysis, providing quantitative insights and visual analysis of surface morphology, including roughness. SEM can be employed to measure surface roughness, providing a rapid and reliable means to characterize surface type and quality [24]. The study evaluates dentin characteristics at three distinct stages: post-sandblasting, post-sandblasting with rinsing water, and post-application of orthophosphoric acid. By employing a multi-analytical approach integrating Raman spectroscopy, SEM, and surface roughness analysis, this study provides a more direct and comprehensive understanding of how dentin morphology and chemical stability evolve under controlled treatment conditions. Typically, studies focus on the clinical implications of sandblasting, indirectly evaluating surface effects through adhesion tests like shear and tensile bond strength. In contrast, our proposed protocol directly evaluates the effects of sandblasting on the dentin surface, offering a more fundamental understanding of the surface alterations beyond indirect clinical outcomes. Thus, this study contributes to the development of improved restorative techniques by providing insights into the effects of different air abrasion protocols on dentin, ultimately aiming to enhance the longevity, functionality, and aesthetics of dental restorations and improve patient outcomes.

II. Materials and Methods

Single-rooted human teeth, extracted within the past year, were selected for this study. Six teeth, obtained from the Department of Cariology and Operative Dentistry, University of Turin (Italy), (protocol DS 00071 2018) were free of dental fillings, sealants, cracks, lesions, or caries. Following extraction, they were carefully debrided, cleaned, and stored in distilled water at room temperature to prevent dehydration and preserve structural integrity for subsequent analyses. The study followed a stepwise approach to assess the effects of different surface treatments on dentine. At baseline (T0), the dentine crowns were prepared, to ensure standardized experimental conditions, circular dentine crowns were cut using a water-cooled MICROMET circular cutting machine equipped with a diamond blade. Cross-sections with a uniform thickness of 1.5 mm – 1.6 mm were obtained, maximizing the exposed dentine surface for morphological and chemical characterization. Two different commercially available air-abrasion sands, i.e. Krugg Aluminum Oxide (50 μm), and PROPHYpearls by KaVo made by calcium carbonate (60-70 μm) spheres, were

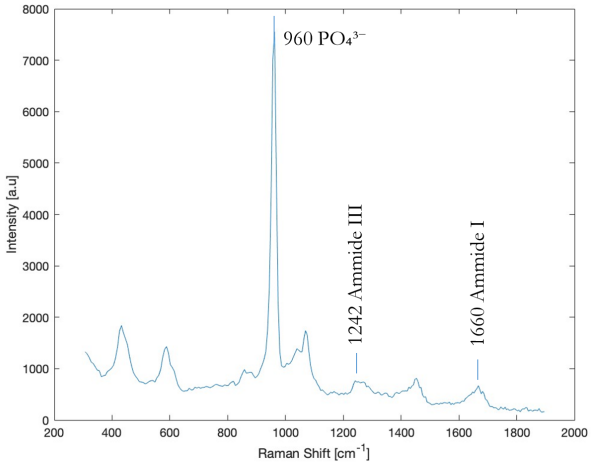


Fig. 1: Representative indexed Raman spectrum of dentin in the range 300 cm^{-1}- 1900 cm^{-1}

used in this study. At T1, samples underwent sandblasting with either aluminum oxide (Al_2O_3) or calcium carbonate ($CaCO_3$) for 10 s with pressure of 1,8 - 2 bar, to induce controlled surface roughness, which is critical for optimizing adhesion in restorative procedures. At T2, sandblasted samples were rinsed with distilled water to eliminate residual abrasive particles. Finally, orthophosphoric acid was applied for 10 s to remove the smear layer and enhance surface porosity, improving interaction with adhesive materials. At T3, after acid treatment, samples were thoroughly rinsed with distilled water to eliminate any residual acid. Before and after each treatment phase, dentine surfaces were characterized using Raman spectroscopy to assess chemical modifications, SEM to evaluate morphological changes, and roughness analysis to quantify surface texture. This integrated measurement approach ensured a comprehensive and reproducible evaluation of dentine surface modifications, contributing to a deeper understanding of their impact on adhesive performance and restorative treatments.

A. Scanning Electron Microscopy (SEM)

1) Morphological characterization: SEM was used to analyze dentin surface modifications at each treatment stage, including after sandblasting, rinsing with water or ethanol to assess residual microparticles, and following orthophosphoric acid application. This allowed for a detailed evaluation of morphological changes induced by different surface treatments. SEM imaging was performed using a Thermo Scientific Phenom XL G2 Desktop SEM, operating under low vacuum, with backscattered electron detector (BSD), thus precluding the necessity for sample metallization, thereby preserving sample integrity and preventing degradation during imaging.Finally, the elemental composition was determined using energy-dispersive spectroscopy (EDS), which analyzes the characteristic X-rays emitted by the sample's constituent atoms. EDS analysis was performed using the following parameters: an accelerating voltage of 15 kV, a vacuum level of 60 Pa, a field width of 104 μm, a backscattered electron detector (BSD full), a magnification of 2600x, and a working distance of 7.69 mm.

2) Roughness: Roughness analysis was performed using the Thermo Scientific Phenom XL G2 Desktop SEM in 3D Roughness Reconstruction (3DRR) mode, enabling high-resolution topographic mapping of dentine surfaces and providing detailed roughness characterization. After thorough sample preparation, surface roughness parameters, including Ra (average roughness) and Rz (peak-to-valley height), were calculated to quantify texture variations. Ra represents the arithmetic mean of surface height deviations, offering an overall texture assessment, while Rz reflects extreme height variations, crucial for evaluating adhesive bonding properties [25]. The roughness measurements were collected after each step of the experimental protocol.

B. Raman Spectroscopy

In this study, Raman spectroscopy was performed using a portable modular BWTEK spectrometer equipped with a 1064 nm monochromatic laser and a BTC284N spectrometer. The spectrometer's range spans between 100 cm^{-1} and 2500 cm^{-1} with a 10 cm^{-1} resolution. The instrument was coupled with a compact BAC151 microscope, facilitating both visualization of the region of interest and laser beam focusing onto the sample surface.

Raman measurements were performed directly on the dentin surface using the following parameters: laser power 30% - 50% - 70% of nominal power, i.e. 450 mW; integration time of 40 s, and 4 repetitions for each area. Four spectra per sample were acquired at different points on the dentin surface of each sample at every phase. Subsequently, baseline correction to remove background signals was performed to observe the most prominent peaks and to assess the chemical composition of dentin, focusing on the integrity of HA and collagen. A typical Raman spectrum of human dentin is represented in Fig. 1, the key peaks analyzed in this study are shown. The Raman spectrum of Al_2O_3 air-abrasion sand, as well as $CaCO_3$ were acquired for comparison.

III. Results

The initial step, T0, involved morphological characterization of the dentin. As shown in the first image on the left of Fig. 2, the SEM image of an untreated dentin sample revealed a generally smooth surface with a uniform distribution of dentinal tubules, a typical characteristic of healthy, untreated dentin. SEM images were subsequently acquired following the sandblasting phase (T1) using both Al_2O_3 and $CaCO_3$. In both cases, SEM images clearly demonstrated the presence of embedded abrasive particles. EDS analysis confirmed the nature of these observed nanoparticles, as shown in the spectra of Fig. 4. The rinsing phase (T2) represents a critical step in the experimental procedure, aimed at evaluating the effectiveness of water in removing residual particles from the dentin surface. SEM images of samples treated with Al_2O_3 following rinsing (Fig. 2b) revealed that rinsing still left detectable Al_2O_3

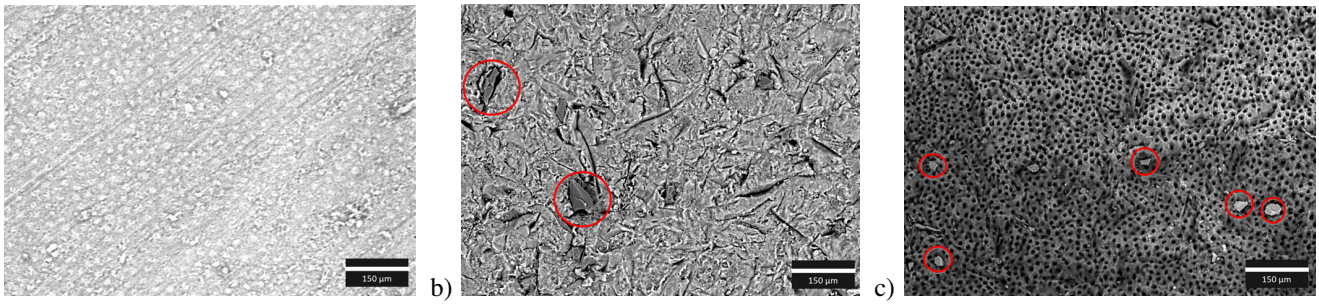


Fig. 2: Dentin SEM images collected in BSD before the sandblasting T0-a; after the sandblasting with Al_2O_3 and water rinsed T2-b, and finally after acid etching T3-c.

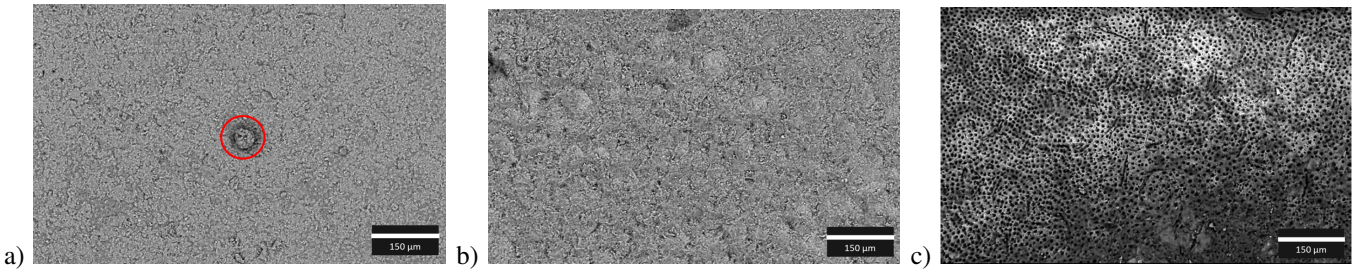


Fig. 3: Dentin SEM images collected in BSD after the sandblasting with $CaCO_3$ T1-a, after water rinsing T2-b, and finally after acid etching T3-c.

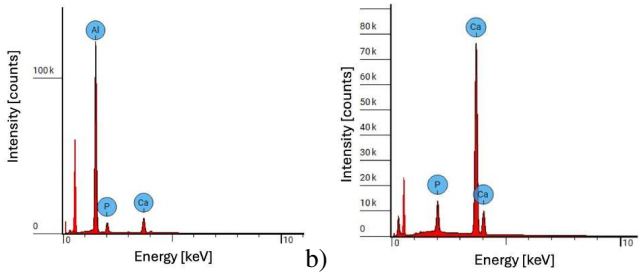


Fig. 4: EDS spectra at T1 of a dentin sample treated with Al_2O_3 a), and dentin sandblasted with $CaCO_3$, b).

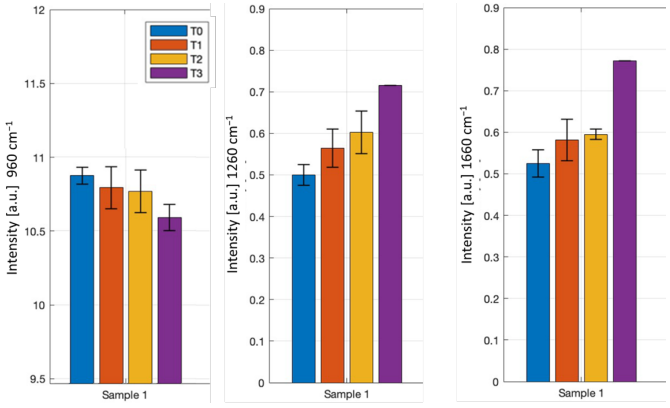


Fig. 5: Raman intensity of samples sandblasted with Al_2O_3 and rinsed with ethanol: 960 cm^{-1} (left), 1260 cm^{-1} (middle) and 1660 cm^{-1} (right) at different phases (T0, T1, T2, and T3). The error bars represent the standard deviation.

fragments on the dentin surface, indicating its strong adhesion to the dentin. The latter remained into the dentin also post-acid etching as shown in Fig. 2c. In contrast, no $CaCO_3$ microparticles were observed in the SEM images of samples rinsed as shown in the second picture of Fig. 3b. This suggests that calcium carbonate was effectively removed during the rinsing phase, indicating lower adhesion to the dentin surface compared to Al_2O_3.

Raman measurements were collected at four distinct points on each sample at each experimental stage (T0: untreated, T1: post-sandblasting, T2: post-rinsing, T3: post-acid etching). Preliminary characterization of sound dentin revealed characteristic peaks associated with the main components of dentin tissue, which were used as reference points for subsequent evaluation of chemical changes induced by surface treatments, specifically at 960 cm^{-1}, 1260 cm^{-1}, and 1660 cm^{-1}. While SEM imaging (Fig. 2) confirmed the presence of Al_2O_3 particles on the dentin surface after sandblasting, and acid-etching reliable detection of Al_2O_3 using Raman spectroscopy under these experimental conditions proved challenging. After sandblasting with $CaCO_3$ (T1), Raman spectroscopy results also did not reveal any detectable $CaCO_3$ signals after the rinsing phase (T2). Notable changes were anticipated in the peaks located at 960 cm^{-1} (characteristic of HA, the main component of dentin), 1242 cm^{-1}, and 1660 cm^{-1} (associated with the peptide bonds of dentin collagen) after the application of the orthophosphoric acid (T3). Analysis of the Raman spectra revealed variations in the intensities of the

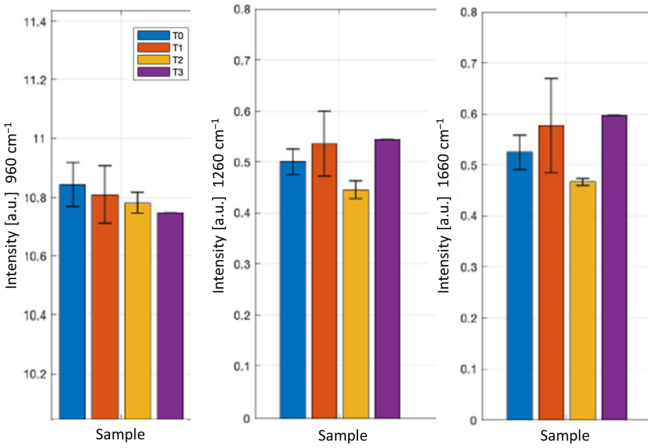


Fig. 6: Raman intensity of samples sandblasted with $CaCO_3$ and rinsed with ethanol: 960 cm^{-1} (left), 1260 cm^{-1} (middle) and 1660 cm^{-1} (right) at different phases (T0, T1 T2, and T3). The error bars represent the standard deviation.

peaks at 960 cm^{-1}, 1242 cm^{-1}, and 1660 cm^{-1} across the analyzed samples, as evidenced by the histograms of Fig. 5 and Fig. 6. A noticeable decrease of the intensity of the peak at 960 cm^{-1} was observed after the acid-etching in most samples, indicating a substantial interaction between the acid and the HA layer, leading to partial dissolution or surface etching, both for samples treated with Al_2O_3 and $CaCO_3$ as shown in the first histogram block of Fig. 5 and Fig. 6. For the peaks at 1242 cm^{-1} and 1660 cm^{-1}, an increase in intensity was observed in some samples after acid etching. The roughness evaluation throughout the entire treatment process for the samples treated with Al_2O_3 is shown in Tab. I, which compares Ra and Rz values at four different stages of the experimental procedure. The roughness increased after the sandblasting, but after the application of orthophosphoric acid, both the mean roughness (Ra) and the maximum roughness depth (Rz) decreased significantly, indicating a pronounced smoothing effect of the acid. This suggests that the acid effectively etched the surface, reducing surface irregularities and creating a more uniform texture.

For $CaCO_3$, roughness analysis revealed a smaller increase in roughness after sandblasting compared to alumina, as shown in Tab. I. This increase was slightly reduced after rinsing, likely due to the removal of some $CaCO_3$ particles. However, a significant increase in surface roughness was observed following acid etching compared to previous stages, possibly attributed to the less abrasive nature of $CaCO_3$ particles. This suggests that orthophosphoric acid treatment effectively created a more irregular surface with a more pronounced pitted texture in the case of $CaCO_3$ sandblasted samples.

IV. Discussion

Aluminum oxide, used as an abrasive medium, exhibited a high abrasive capacity on the dentin surface. Despite rinsing procedures and subsequent treatment with orthophosphoric acid, residual microparticles were observed under SEM. In contrast, calcium carbonate particles were more easily removed during the rinsing phase. The spherical shape of calcium carbonate particles likely contributed to their reduced adhesion to the dentin surface. While SEM provided visual confirmation of particle presence, Raman spectroscopy proved less effective in directly detecting these particles, suggesting limitations in its sensitivity for this specific application. However, Raman spectroscopy revealed structural alteration in dentin, mainly linked to mineral loss and collagen, as shown in the decrease of the intensity peak associated with the HA. Whilst, the increase in peak intensity at 1242 cm^{-1} and 1660 cm^{-1}, after the acid etching may be attributed to increased exposure and denaturation of collagen, rendering it more susceptible to Raman scattering. The observed variability in Raman spectral responses across samples highlights the influence of factors such as pre-existing surface roughness induced by sandblasting. This variability, reflected in increased standard deviations, could be attributed to several factors, including the effects of orthophosphoric acid treatment, which may have degraded both the dentin and collagen structures.

TABLE I: Surface Roughness Parameters (µm) at different phases both for samples treated with Al_2O_3 and $CaCO_3$.

Treatment	Al_2O_3		$CaCO_3$	
	Ra (µm)	Rz (µm)	Ra (µm)	Rz (µm)
T0	0.30 ± 0.04	1.33 ± 0.24	0.25 ± 0.05	1.01 ± 0.17
T1	0.95 ± 0.16	1.33 ± 0.24	0.39 ± 0.08	1.82 ± 0.47
T2	1.15 ± 0.26	4.00 ± 0.62	0.25 ± 0.02	1.06 ± 0.15
T3	0.85 ± 0.17	2.87 ± 0.39	1.40 ± 0.42	4.35 ± 1.13

The initial surface roughness induced by Al_2O_3 sandblasting was significantly higher compared to calcium carbonate. Subsequent treatment with orthophosphoric acid led to a marked decrease in the surface roughness of Al_2O_3-treated samples, indicating effective surface etching and smoothing. For $CaCO_3$-treated samples, while the initial increase in roughness was less pronounced, the subsequent acid treatment resulted in a significant increase in surface roughness. This suggests that the less abrasive nature of calcium carbonate particles combined with the etching effect of phosphoric acid created a more irregular surface texture. Thus, $CaCO_3$ sandblasting, while resulting in a less pronounced increase in roughness, offers a potentially gentler approach to surface preparation.

V. Conclusion

This study proposes a new procedure to evaluate the effects of different surface treatment protocols for dental restorative using an integrated measurement approach to systematically characterize dentin surfaces. Specifically, the research investigates the impact of sandblasting with two distinct abrasive materials—aluminum oxide (Al_2O_3) and calcium carbonate ($CaCO_3$)—followed by rinsing and the application of orthophosphoric acid. The analysis focuses on key parameters, including smear layer removal, alterations in surface roughness, and chemical composition changes throughout the treatment process.

By employing a multi-analytical approach, this study provides a comprehensive understanding of how dentin morphology and chemical stability evolve under controlled treatment conditions. The integration of Raman spectroscopy, SEM, and surface roughness analysis enables a robust evaluation of surface modifications. This research aims to contribute to the optimization of clinical protocols, ultimately enhancing the adhesion properties and longevity of dental restorations.

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