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ORIGINAL RESEARCH

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Correlation between the enamel-changed surface roughness, micro-hardness and the depth of demineralization after orthodontic bracket use

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Abstract

Background: Use of orthodontic bracket changes enamel surface properties and demineralization penetration (i.e. rougher surface, reduced hardness, and deeper demineralization). This study evaluated the correlation between the changed enamel surface roughness, micro-hardness and demineralization depth after orthodontic bracket use. **Methods**: Data were obtained from a previous research project involving 198 extracted human premolar teeth, which underwent standardized bonding and debonding procedures. Ninety-nine specimens were assessed for surface roughness before and after the bracket, measured as Ra (the arithmetic mean height in microns), and then underwent micro-hardness testing using the micro-Vickers hardness test. The remaining 99 specimens were exposed to a demineralization solution, and the demineralization depth was analyzed using scanning electron microscopy and ImageJ software. Pearson's correlation coefficient was used to evaluate the relationship between enamel surface roughness, micro-hardness, and demineralization depth. **Results**: The findings revealed a weak positive correlation between enamel surface roughness and demineralization depth, which was not statistically significant (r = +0.151, p = 0.134). However, a significant moderate negative correlation was found between enamel surface micro-hardness and demineralization depth ($r = -0.504$, $p < 0.01$). Additionally, a significantly weak negative correlation was found between enamel surface roughness and micro-hardness ($r = -0.289$, $p = 0.004$). **Conclusions**: The impact of orthodontic bracket use on the enamel surface roughness and hardness mutually influence each other and contribute to deeper demineralization. Enamel surface roughness is inversely correlated with hardness, and its hardness is inversely correlated with demineralization depth. These findings have clinical implications as the effect on enamel surfaces and demineralization penetration complicates the reversal and management of lesions. The study highlights the need to explore less invasive alternatives to bonding and debonding procedures, conduct further research in this area and improve dental materials.

Keywords

Dental enamel; Tooth demineralization; Dental white spot; Orthodontic brackets

1. Introduction

Dental enamel is composed of a high percentage of minerals and a well-packed crystalline structure, which imparts the enamel its unique physical properties and protects the dental substrate [1, 2]. Among oral diseases, dental caries is the most common [3–6], a result of complex and multifactorial interactions between cariogenic bacteria, fermentable carbohydrates and the host [5, 6]. White Spot Lesions (WSLs) represent enamel demineralization at an early stage of lesion development, having a soft texture and appearing rough, opaque and milky-white when dried [6–10].

The incidence and prevalence rates of WSLs among orthodontic patients are considerably high (45.8% and 68.4%, respectively) [10]. Orthodontic appliance-retentive areas contribute to these inevitable rates, which complicate oral hygiene maintenance and salivary and muscle cleansing, causing changes in oral biofilm composition, disruptions in the demineralization–remineralization cycle and WSLs development [11–15].

Cariogenic bacteria (most commonly Streptococcus mutans and Lactobacillus) metabolize fermentable carbohydrates producing hydrogen ions that lower the oral pH, leading to enamel dissolution, mineral loss and surface porosity [6, 16, 17]. This initial demineralization may be presented as enamel outer surface softening due to the removal of the interprismatic substance, creating a surface lesion [10, 18]. If some remineralization of the outermost layer occurs while the deeper

surface is still affected, the continuous loss of the deeper interprismatic substance leaves a porous, fragile and intact outer layer, resulting in a subsurface lesion [10, 18].

The demineralization complex cycle is influenced by various factors such as teeth characteristics, oral environment and dental materials (which may affect dental substrate characteristics and integrity). The impact of fixed orthodontic appliances on the enamel surface starts from surface treatment for bracket bonding (for example, through excessive enamel etching), which alters enamel surface properties like roughness and hardness [10, 19]. It also affects the amount of resin tag penetration for bracket placement and the extent of enamel demineralization, which is influential in understanding how to prevent iatrogenic enamel damage during debonding [19].

Surface roughness refers to irregularities or height deviations at each point from the average surface (arithmetical mean or roughness average (Ra)) [20]. Plaque accumulation and bacterial adhesion increase with a rougher enamel surface, contributing to enamel demineralization [20–23]. The use of orthodontic brackets can result in a rougher enamel surface compared with non-bonded control surfaces [24–27]. However, some studies found no difference in enamel surface roughness between bonded and non-bonded teeth [28–31].

Mineral content and crystal lattice arrangement influence the enamel's solubility [32]. High-mineral enamel with a well-packed crystalline structure has fewer surface impurities and is more resistant to acid [33]. Surface hardness refers to the surface resistance to penetration and deformation [33–35]. High enamel hardness values indicate lower demineralization susceptibility and higher remineralization potential [36–40]. Attin *et al.* [41] (2003) and Tostes *et al.* [42] (2013) reported that demineralized enamel surface hardness increases after remineralization, but it remains lower than that of the intact enamel, demonstrating the importance of preserving the original integrity and intactness of the enamel. In several studies, hardness was found to be reduced after orthodontic bracket use compared with non-bonded surfaces [24, 43, 44], however, this difference is minimal and not significant according to Iijima *et al.* [19] (2010).

Many studies have found that orthodontic brackets demineralize enamel more than teeth without brackets [45–49]. Various factors contribute to this, including the materials' physical properties, their effects on the enamel surface and their interaction with the oral environment [49]. Furthermore, the debonding process contributes to the resultant enamel damage; higher debonding forces increase enamel damage risk [50]. Debonding force and its impact on the enamel are cumulatively affected by the etching/conditioning procedure, adhesive system, bracket material/design, bracket base architecture and debonding method [51–53].

To the best of our knowledge, there is a lack of studies examining the relationship between changes in enamel surface properties (roughness and hardness) and the depth of demineralization after orthodontic bracket use. This raises the question of whether changes in enamel roughness and hardness caused by orthodontic brackets could contribute to and correlate with enamel demineralization, which is crucial in providing evidence-based knowledge toward a more conservative clinical practice, material improvement and future research, regarding bracket-bonding (such as surface preparation *i.e.*, etching) and debonding steps to minimize impact on enamel surface properties and consequent demineralization.

Therefore, this study aimed to determine the correlation between the enamel changed surface roughness, micro-hardness and demineralization depth after orthodontic bracket use (regardless of the etchant type, adhesive system and bracket material). Based on the null hypothesis, there is no correlation between the changed enamel surface roughness, micro-hardness and demineralization depth after orthodontic bracket use. There is, according to the alternative hypothesis, a correlation between the changed enamel surface roughness, micro-hardness and demineralization depth after orthodontic bracket use.

2. Materials and methods

2.1 Sample/Data

Using the G*Power calculator (version 3.1.9.6, Franz Faul, Universität Kiel, Kiel, SH, Germany), a minimum of 96 samples were deemed sufficient at a 0.05 level of significance with an effect size of 0.35 and 95% power for a correlational assessment [54]. This study is a continuation of a doctoral thesis project involving two *in-vitro* studies that were approved by the Institutional Review Board of Health Sciences Colleges Research on Human Subjects, College of Medicine (Reg. No. E-21-5917) and the College of Dentistry Research Center (Reg. No. PR 0117) [24, 45].

The data utilized from a total of 198 anonymous human premolar teeth extracted for orthodontic purposes (99 sample size for each study), with standard inclusion criteria (teeth free from cracks, caries, and demineralization with no root canal treatments). Each sample had standardized simple randomization using Excel (version 16.0.12624; Microsoft, Redmond, WA, USA) with Kutools (version 22.00; ExtendOffice, Haikou, Hainan, China), grouping methods and bonding and debonding protocols.

2.2 Variables measured

2.2.1 Study I

Originally, it assessed the effect of different etchants, adhesive systems, and bracket materials on the enamel surface roughness and micro-hardness. Ninety-nine extracted human premolar teeth specimens were selected, and 88 were assigned to eight experimental groups (11 specimens for each). Following manufacturing instructions, they were prepared with total- or self-etchants before being bonded to pre-coated or flash-free adhesive metal or ceramic brackets. Eleven specimens were used as controls for micro-hardness without bracket bonding.

Surface roughness was initially assessed before bonding using non-contact surface metrology and an imaging optical microscope (Contour GT-K 3D; Bruker, Tucson, AZ, USA) and recorded as Ra in μm [24]. This measurement served as a control of surface roughness [24]. Subsequently, the brackets were bonded and the specimens were immersed in distilled water at room temperature for 24 hours. Following this, the brackets were debonded using a removing plier (Unitek Debonding Instrument 804-175; 3M, St. Paul, MN, USA),

and any remaining resin was cleaned using a non-cutting, large round tungsten carbide bur attached to a slow-speed handpiece (FX23 Contra Angle, NSK, Kanuma, Tochigi, Japan) at 20,000 rpm. The cleaning process was conducted with air and without water cooling in an occlusal-gingival direction [24].

The specimens were then scanned again for surface roughness (post-debonding). Eventually, enamel surface micro-hardness was assessed using quasi-static indentation (Hysitron TI 750; Innovatest, Nihonbashi, Horidomecho Chuoku, Tokyo, Japan) with a 200 g force for 10 s, as indicated by the micro-Vickers hardness number (VHN) [24].

2.2.2 Study II

Originally, it assessed the effect of different etchants, adhesive systems, and bracket materials on the enamel demineralization depth. Ninety-nine extracted human premolar teeth specimens were assigned into 9 groups (8 experimental and 1 control), with 11 specimens for each. A total-or self-etchant with a pre-coated or flash-free adhesive system was used to bond the experimental groups with either metal or ceramic brackets.

All specimens were immersed in a freshly prepared artificial demineralization solution composed of 2.2 mM of calcium chloride, 2.2 mM of monosodium dihydrogen orthophosphate, and 0.05 mM of acetic acid adjusted to pH 4.5 using 1 M of potassium hydroxide, at 37 °C for 7 days [45]. Brackets were then debonded, and the remaining resin was cleaned completely following the previously mentioned protocol [24, 45].

The specimens were sectioned buccolingually at the right and left sides of bracket base margins, representing the right and left sides of the bonded enamel. Enamel demineralization images were captured using scanning electron microscopy (JEOL 6060 LV Scanning Electron Microscope; JEOL, Tokyo, Japan), and demineralization depth was measured using ImageJ software (in μm) from the buccal surface to the deepest detection point for the right and left sections [45].

The details of the grouping, bonding, and debonding protocols are illustrated in Fig. 1. The methodology sequence is outlined in Fig. 2.

2.3 Statistical analyses

A total of 296 readings (99 readings for each assessment of surface roughness, micro-hardness and demineralization) were obtained as secondary quantitative data for this correlational assessment. Surface roughness, surface micro-hardness and demineralization depth data from studies I and II were pooled and linked by the grouping between the two samples (the grouping according to etchant type, adhesive system and bracket material was used as a link variable). Although these variables did not influence the correlation outcome, they were not included to simplify the interpretation.

The statistical test was performed using SPSS version 26 (IBM Corp., Armonk, NY, USA). The correlation between the changed enamel surface properties (surface roughness and microhardness) and demineralization depth was assessed using Pearson's correlation. $p \leq 0.05$ indicates statistically significant differences.

3. Results

Each variable's cut-point (enamel surface roughness, surface micro-hardness, and demineralization depth) by averaging the pooled data mean values. Pearson's correlation suggested a weak positive correlation between the enamel surface roughness and enamel demineralization depth, demonstrating that higher enamel surface roughness contributed to deeper enamel demineralization. This correlation, however, was statistically insignificant ($r = +0.151, p = 0.134$) (Table 1). This is reflected in Fig. 3A by the randomly scattered data around the roughness cut-point value.

There was a statistically significant and moderate negative correlation between enamel surface micro-hardness and enamel demineralization depth, with higher enamel surface micro-hardness resulting in shallower enamel demineralization ($r = -0.504$, $p < 0.01$) (Table 1). A negative correlation is indicated in Fig. 3B by the deepest demineralization readings that are mostly below the hardness cut-point value and *vice versa*.

Additionally, a statistically significant and weak negative correlation was observed between enamel surface roughness and enamel micro-hardness, with higher enamel surface roughness resulting in lower enamel surface micro-hardness ($r = -0.289, p = 0.004$) (Table 1). The roughest readings in Fig. 3C are mostly below the hardness cut-point value and *vice versa*, showing a negative correlation.

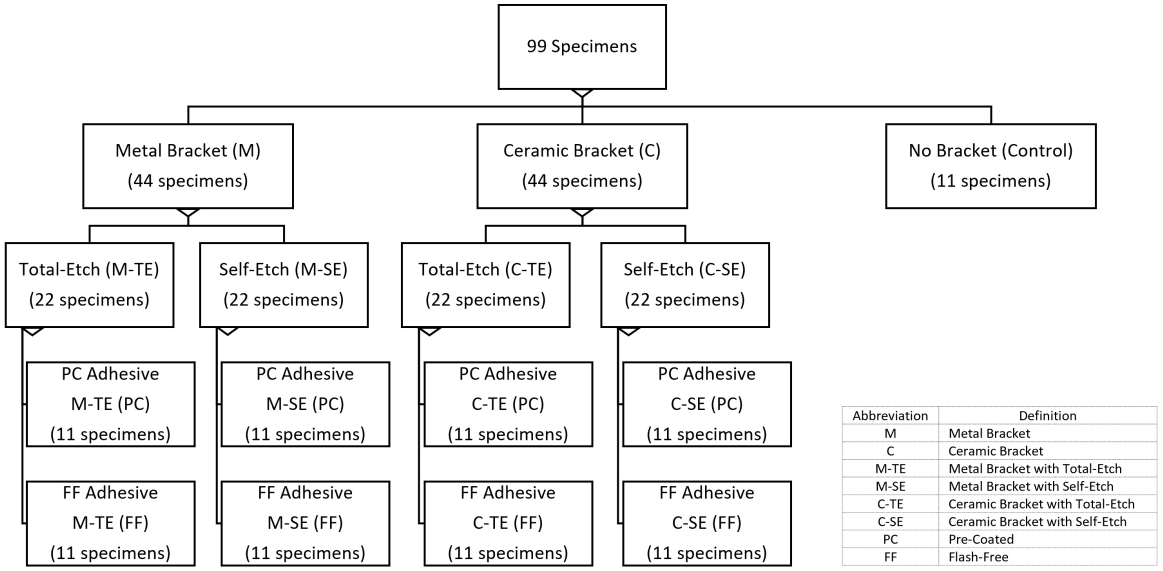
4. Discussion

Enamel surface properties changes and demineralization are risk factors for orthodontic patients [19]. The use of orthodontic bracket found to introduce rougher surface, reduced hardness and deeper demineralization [24, 43–49]. However, the correlation between the altered enamel surface properties (roughness and hardness) and demineralization depth after bracket use is scarcely explored. This study aimed to examine the correlation between the changed enamel surface properties (roughness and micro-hardness) and the depth of demineralization after orthodontic bracket use (regardless of the etchant type, adhesive system and bracket material).

Enamel surface roughness influences bacteria's adhesion and plaque build-up [19]. Bacteria adhere more readily to rough surfaces, which may lead to plaque build-up, acid production and demineralization [19–23, 44, 55]. Total-etch and self-etch are common acid etching techniques, which create a rougher surface than non-etched surfaces, while a total-etch produces deeper irregularities than self-etch [24, 56, 57]. The commonly used methods for brackets removal (straight debonding plier or instrument Lift-Off) and adhesive remnants cleaning (long adhesive removing plier and tungsten carbide bur at low- and high-speed) can make the enamel surface rougher and cause surface cracks, affecting the overall surface smoothness [24, 58, 59].

The adhesive system used affects the amount of adhesive remaining after bracket removal. Adhesive systems without a flash-free feature tend to leave more adhesive remanent on the enamel surface, requiring a greater cleaning area and potentially affecting the enamel's properties [60–62]. However,

(A)



Abbreviation	Definition
M	Metal Bracket
C	Ceramic Bracket
M-TE	Metal Bracket with Total-Etch
M-SE	Metal Bracket with Self-Etch
C-TE	Ceramic Bracket with Total-Etch
C-SE	Ceramic Bracket with Self-Etch
PC	Pre-Coated
FF	Flash-Free

(B)

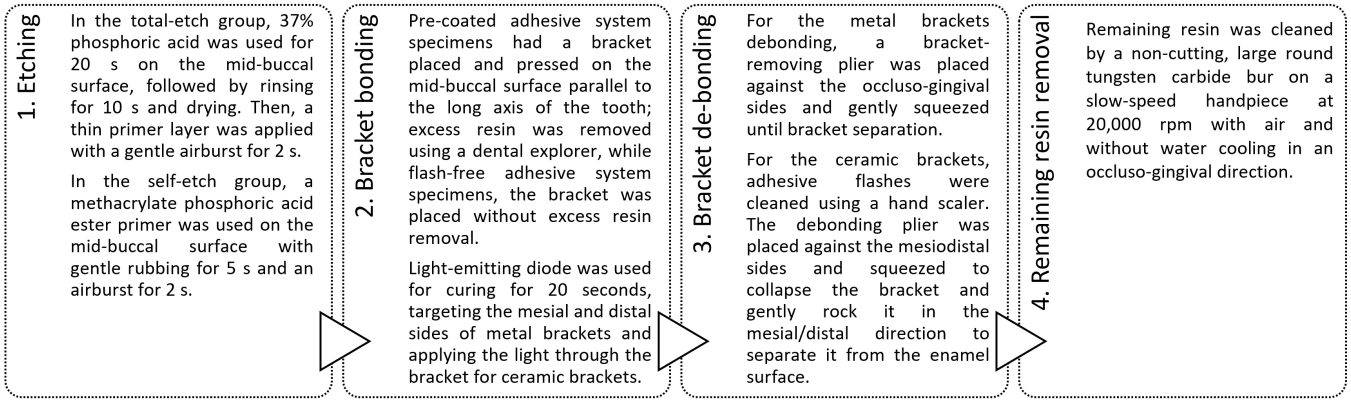


FIGURE 1. Original studies grouping, bonding, and debonding protocols. (A) The grouping performed in each study. (B) The standardized bonding and de-bonding protocols in studies I and II.

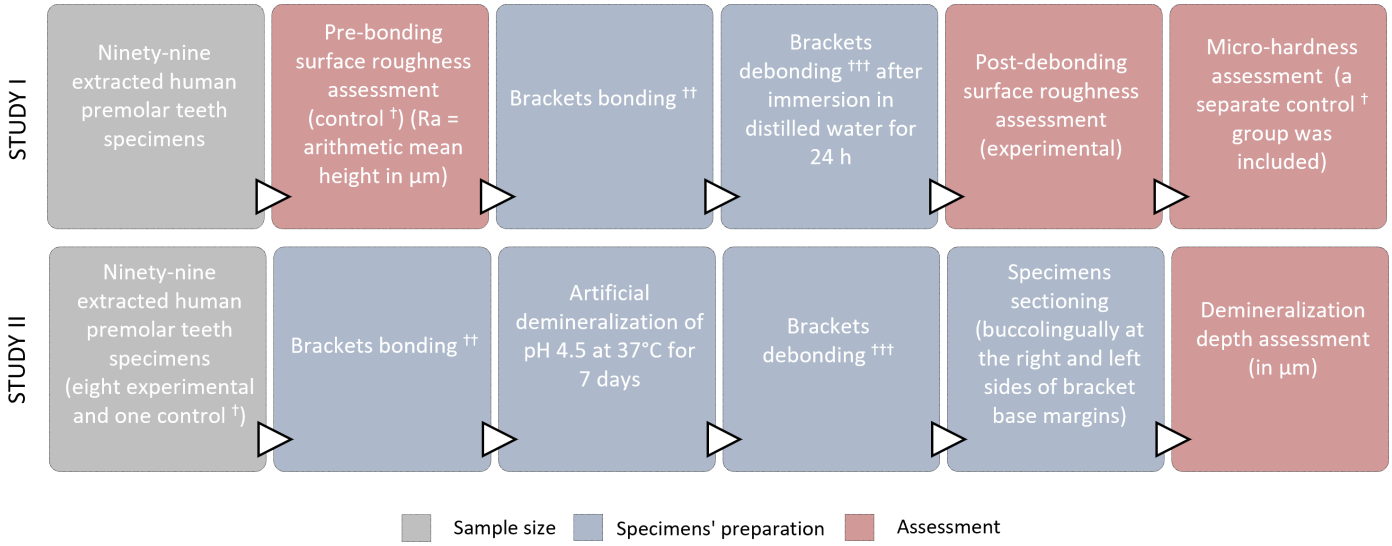


FIGURE 2. Original studies method sequence. (†: Control group refers to non-bonded specimens, ††: Standardized bonding materials and protocol, †††: Standardized debonding instruments and protocol).

TABLE 1. Pearson correlation test.

Assessment	Pearson Correlation Coefficient (*r*)	*p*-value
Enamel Surface Roughness and Demineralization Depth	0.151	0.134
Enamel Surface Micro-hardness and Demineralization Depth	−0.504	0.001**
Enamel Surface Roughness and Micro-hardness	−0.289	0.004**

*(**: Statistically Significant with $p < 0.01$).*

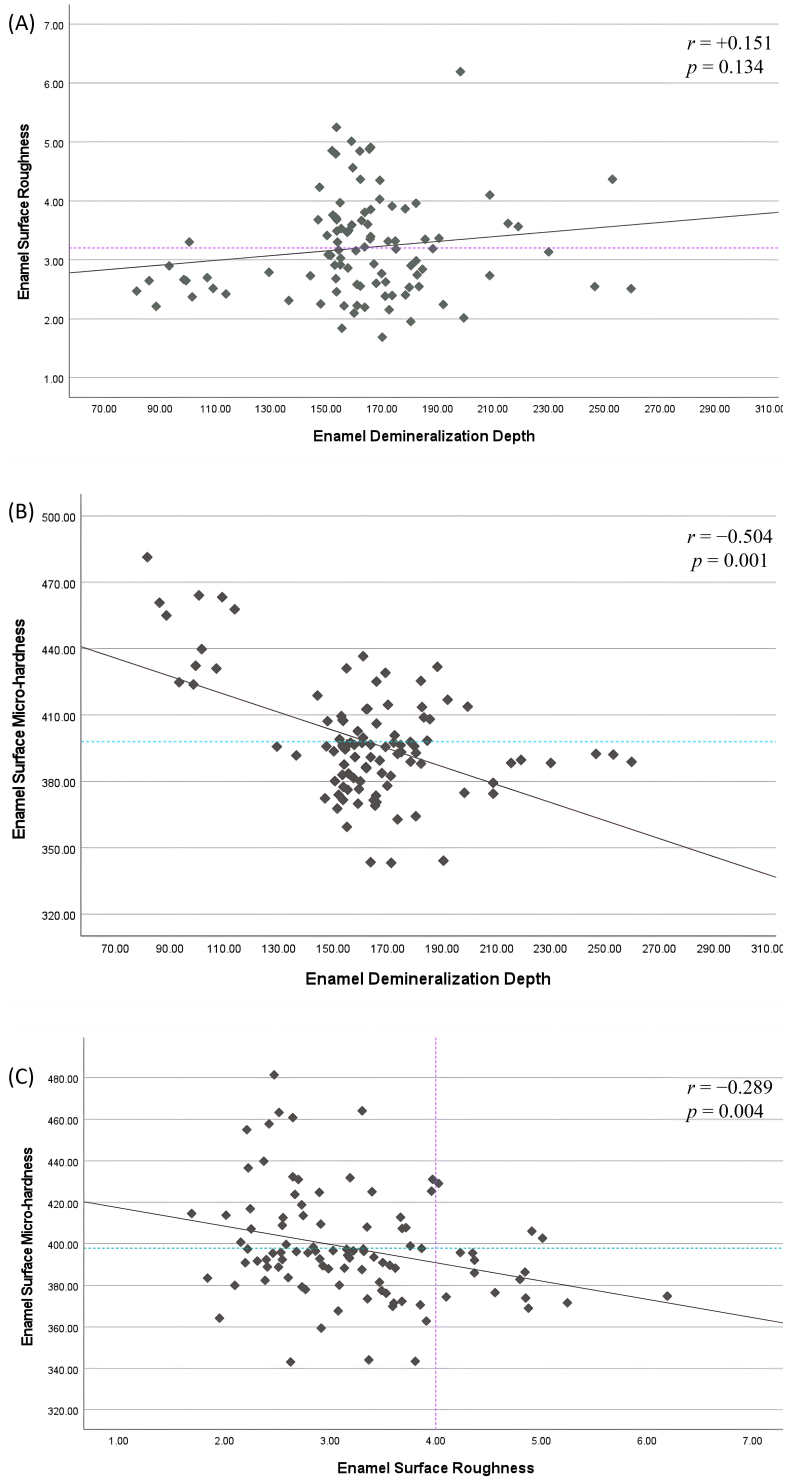


FIGURE 3. Correlation scatter plot. (A) Scatter plot of enamel demineralization depth by surface roughness. (B) Scatter plot enamel demineralization depth by surface micro-hardness. (C) Scatter plot of enamel surface roughness by surface micro-hardness. Dotted line represents axis cut-point mean values, purple: enamel surface roughness, and blue: enamel surface micro-hardness.

other studies have not discovered any difference between the two systems [24, 63]. A recent systematic review and meta-analysis concluded that current evidence is not sufficient to show that the flash-free system reduces lesion development [64].

Limited studies have examined the correlation between enamel surface roughness and demineralization depth after orthodontic bracket use. This study's findings demonstrated a weak positive correlation between enamel surface roughness and demineralization depth, which was statistically insignificant. Teutle-Coyotecatl *et al*. [59] (2022) reported higher bacterial adhesion in deciduous teeth (which are smoother) compared with permanent teeth and postulated that enamel roughness could not be used to determine bacterial adhesion. However, their study was done without orthodontic brackets to compare deciduous teeth and permanent teeth [59]. Kapur *et al*. [65] (1961) concluded that roughened enamel surfaces increased acidic buffer penetration by 27%; however, a variety of methods were used to intentionally roughen the enamel, including sandpaper disks, carborundum stones and diamond disks. A similar finding of higher acid penetration with a rougher surface was reported in another study, but their evaluation was conducted after interproximal enamel reduction without brackets [66].

Enamel surface roughness and demineralization are primarily related to plaque and bacterial accumulation [21, 67]. This could explain the lack of significant correlation between enamel surface roughness and demineralization observed in this study, which used data from *in-vitro* studies (without a dental biofilm model). Because bacterial adhesion is influenced by several factors other than surface roughness, it is expected that the positive correlation between enamel surface roughness and demineralization would be amplified in an *in-vivo* setting [68]. These factors include surface electrostatic interactions, hydrophobic ion bonding, van der Waals forces and host-related factors including saliva characteristics and oral hygiene [68]. Therefore, it is important to determine which of these factors contributes most to bacterial adhesion to the enamel surface.

Enamel surface hardness represents the enamel mineral content. Higher mineral loss is associated with lower hardness values [36, 37, 39, 40, 69–72]. Orthodontic bracket bonding and debonding can significantly reduce enamel surface hardness [24, 43, 44]. Hardness reduction was found to be related mostly to the etching step, regardless of whether total- or self-etchants were used [19, 24, 73].

Several studies have examined the relationship between enamel surface hardness and its demineralization (without orthodontic bracket use) and concluded that harder enamel surfaces have shallower demineralization [37, 71, 74, 75]. However, studies examining the correlation between the enamel surface hardness and its demineralization depth after orthodontic bracket use are rare.

This study found a statistically significant negative correlation between enamel surface micro-hardness and demineralization depth. This indicates that after orthodontic bracket use, enamel hardness decreases, resulting in deeper enamel demineralization. This finding is crucial because enamel etching in preparation for bracket bonding significantly reduces hardness, including total- and self-etch techniques [24, 76–79]. Alterations in the chemical composition caused by enamel apatite mineral reduction reduce the enamel surface hardness and permit rapid and deeper enamel demineralization.

This study also evaluated the correlation between enamel surface roughness and micro-hardness. The availability of studies that assess this aspect is not known. Statistically significant negative correlation was found between enamel surface roughness and micro-hardness, implying that higher surface roughness causes lower surface hardness. The enamel roughness introduced by the etching procedure could result in mineral loss and a porous enamel surface with dissolved prisms that would be less resistant to penetration [56, 57, 73, 80–82], thus lowering the hardness [83]. The correlation between surface roughness and surface hardness would indirectly affect demineralization penetration. Thus, maintaining enamel surface properties preserves enamel integrity and limits demineralization penetration.

This study emphasizes the multifactorial nature of dental enamel demineralization, which has clinical relevance. Orthodontic brackets use altered enamel surfaces, which affect demineralization depth negatively. A deeper demineralization complicates the reversal and management of resulting lesions. Considering this, it is crucial to find a less invasive alternative to bonding (such as surface preparation—etching) and debonding to minimize damage to enamel surface properties and consequent demineralization.

In practice and research, laser light has gained attention for more conservative enamel surface conditioning [83]. Erbium-doped yttrium aluminum garnet (Er:YAG) laser and total-etching introduce no differences in enamel demineralization development [60, 84]. Er:YAG laser introduces less mineral loss from the enamel surface than total-etch [85]. To standardize the recommended protocol and setting for clinical use, further studies are needed.

In regards to the debonding step, electrothermal method utilizes heat to soften the adhesive for bracket removal, avoiding physical contact with the enamel surface and its sequelae, but there is the risk of pulp necrosis from heat application [86]. The Er:YAG laser reduces ceramic bracket bond strength and enamel fracture with minimal thermal effect on the pulp, however, it is time-consuming and results in a rougher enamel surface than current methods [87, 88].

In the experimental studies of this project, the manual specimens' preparation and assessment might influence the performed procedure and the measured outcomes. However, having a well-trained single operator with high intra-rater reliability (who has undergone previous training and conducted a pilot study) could minimize the variations and produce consistent results, thus minimizing the impact on the outcomes.

In this study, correlations between enamel surface roughness, hardness and demineralization are depicted, but causality in the complex dynamic demineralization process is not predicted. Moreover, it utilizes secondary data from experimental studies and does not represent clinical conditions influenced by environmental factors. However, the study emphasizes the impact of the bracket-bonding and -debonding steps on enamel surface properties and subsequent demineralization. Undoubtedly, this correlation is expected to have an amplified

effect within the oral environment. Future studies are needed to evaluate these variables and the role of oral hygiene in bacterial adhesion, as well as to assess the influence of these approaches on enamel surface characteristics and the development of improved dental materials.

5. Conclusions

The enamel-changed surface roughness and hardness after orthodontic bracket use mutually influence each other. The resulting rougher surface and reduced hardness contribute to deeper demineralization, making the enamel more prone to lesion development and progression during or after orthodontic treatment. These relationships can be summarized as follows:

- Enamel surface roughness and demineralization depth are directly correlated, but this relationship is not significant in an *in-vitro* setting.
- Enamel surface hardness and demineralization depth are inversely correlated, *i.e.*, a higher hardness value results in lower demineralization depth.
- Enamel surface properties are associated; enamel surface roughness and its hardness are inversely correlated, *i.e.*, a higher roughness value implies a lower hardness.

Preserving the enamel surface properties is crucial for maintaining its integrity and limiting demineralization penetration; this would prevent complications in the reversal and management of the lesions, ultimately ensuring optimal oral health. It is recommended to utilize and explore less invasive alternatives to bonding and debonding procedures in orthodontics.

AVAILABILITY OF DATA AND MATERIALS

The original data of the secondary retrieved data studies are available in the published paper and cited accordingly.

AUTHOR CONTRIBUTIONS

RZ—authors contributed to conceptualization, data acquisition, interpretation, drafted the manuscript. RZ and NA—design, critically revised the manuscript. NA—revised data interpretation and supervised the project. All authors have read and agreed to the published version of the final manuscript.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

All methods of the secondary data studies were carried out in accordance with relevant guidelines and regulations and were approved by the Institutional Review Board for Health Sciences Colleges Research on Human Subjects, College of Medicine, King Saud University, Riyadh, Saudi Arabia (Reg. No. E-21-5917) and the College of Dentistry Research Center, Riyadh, Saudi Arabia (Reg. No. PR 0117). The use of anonymous extracted human teeth did not require participant consent, as stated by the Ethical Committee and the responsible authorities of this research organization, which adhered to all legal and ethical standards.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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