Influence of Natural Bovine Enamel Roughness on Bond Strength after Etching

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ABSTRACT

Objective: To determine and compare the longitudinal and transverse roughness parameters of the enamel surface of bovine teeth and evaluate the influence of these parameters on bond strength.

Materials and Methods: Ninety bovine incisors were used. The surface roughness of enamel was measured with a profilometer. For each tooth, five readings were taken in the longitudinal plane and five were taken in the transverse plane of the long axis of the tooth in an area equal to the size of the bracket base. The metal brackets were bonded with Transbond XT, and bond strength was evaluated in a universal test machine.

Results: There was a statistical correlation ($P < .01$) between the longitudinal and transverse roughness measurements. There was no correlation between roughness measurements (longitudinal and transverse) and bond strength ($P > .05$), nor was there a correlation between total roughness (longitudinal Ra and transverse Ra) and bond strength. The Student's *t*-test showed that there was a statistically significant difference ($P < .05$) between longitudinal and transverse roughness.

Conclusion: The transverse roughness is greater than the longitudinal roughness, there is a strong correlation between longitudinal and transverse roughness, and there is no correlation between enamel roughness and bond strength. (*Angle Orthod.* 2010;80:562–569.)

KEY WORDS: Natural enamel roughness; Roughness; Bond strength; Bonding

INTRODUCTION

Direct bonding of orthodontic brackets to the labial face of teeth was presented by Newman[1] in 1965 with the purpose of eliminating the metal bands. This only became possible as a result of the concept of enamel etching, which had been introduced by Buonocore[2] in 1955 and which enabled orthodontic accessories to be bonded by means of an adhesive system with high bond strength. Since then, research has been conducted with the aim of analyzing the bond strength of direct bonding techniques in orthodontics by varying the adhesive system,[3–7] type and time of enamel etching,[6,8] and types of orthodontic brackets.[9,10]

Powers and Messersmith[11] showed that there are various factors that can interfere in the bond strength between the enamel and bracket, including the type of tooth, high fluoride concentration (fluorosis), concentration and time of enamel etching, adhesive system, and bracket base. Thus, in bond strength tests, great emphasis is placed upon the concentration and type of acid, etching time, adhesive system, and types of brackets, while little or no attention is paid to the characteristics of the enamel surface. Segura et al[12] referred to roughness as a characteristic of enamel that is closely connected with shininess, light reflection, and the accumulation and retention of bacterial plaque. Studies in the literature have evaluated the roughness of human primary and permanent teeth,[13] the effect of microabrasion on the surface roughness of restorative materials, dentin and enamel,[14,15] the correlation between enamel roughness and wettability,[16] and the influence of various methods of ceramic surface etching on roughness and the bond strength of metal brackets.[17,18]

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Roughness is defined as the set of irregularities, ie, small saliencies and re-entries, that characterize a surface and can be evaluated by means of electronic appliances, such as a roughness meter. Jung et al[19] raised the hypothesis of roughness resulting in a larger bonding area, facilitating the flow of the adhesive, but noted that deep and narrow irregularities could lead to bubbles between the enamel and the composite, weakening the bond strength. Thus, when considering that etching transforms the smooth and even surface of the enamel into an irregular surface that allows for penetration of the resinous monomers into the irregularities,[20] one must bear in mind that enamel surface roughness may influence the bond strength of brackets. Thus it is relevant to test this hypothesis, which could have a direct influence on bonding efficiency. Furthermore, as current experiments regarding the bond strength of brackets bonded to the enamel surface have not considered the natural roughness of enamel, depending on the results of this study, the results attributed to the types of brackets, adhesive systems, concentration, and time of acid etching could be compromised.

Therefore, the aim of the present study was to determine and compare the longitudinal and transverse roughness parameters of the enamel surface of bovine teeth and to evaluate the influence of these parameters on bond strength.

MATERIALS AND METHODS

The present study was submitted to the Ethics Committee on the Use of Animals (CEUA) of the Pontifical Catholic University of Paraná (registration no. 293) for evaluation and was approved (Report No. 214/07).

To conduct this research, 90 bovine mandibular permanent incisors with whole enamel and no decalcification, cracks, or fractures were used. After being extracted, the teeth were kept in a 0.5% Chloramine T solution for disinfection for 1 week. During this period, the soft tissues, the root section at the middle third, and the dental pulp were removed. After this, the teeth were stored in distilled water, which was periodically changed in accordance with ISO/TS 11405.[21]

To evaluate the difference in enamel surface roughness, each tooth was numbered and the area to be evaluated on the tooth was delimited for the purpose of standardizing the tests. Metal brackets for mandibular incisors with a 0.022- $\times$ 0.025-inch rectangular slot were used (3M Unitek, Monrovia, Calif). The area at the base of the bracket (9.35 mm^2) was demarcated with a waterproof pen in the incisal third of each tooth (Figure 1A,B). With a profilometer (Taylor Hobson, Form Talysurf series 2, Leicester, England), the roughness measurements of the delimited surface were made in the following manner: five longitudinal lines parallel to the long axis and five transverse lines perpendicular to the long axis of the tooth, for a total of 10 lines (Figure 1C,D). The roughness parameter analyzed was the average roughness (Ra), which is the arithmetical mean of the absolute values of the ordinates of the distance from the points of the roughness profile in relation to the midline, within the measurement run.

Afterward, in accordance with all the procedures adopted for bracket bonding in the clinic, the future bonding region was submitted to prophylaxis with pumice stone paste and water for 10 seconds using a rubber cup driven by a low-speed handpiece. Each cup was used on only five teeth to prevent its excessive wear. After this, the teeth were rinsed with jets of distilled water with a triple syringe for 20 seconds and dried with compressed air, free of humidity or oil, for 20 seconds. Next, the enamel surface was etched with 37% phosphoric acid (Dentsply, Petrópolis, Brazil) for 30 seconds, rinsed for 20 seconds with jets of water, and dried for 20 seconds with compressed air free of humidity or oil.

The brackets were bonded with Transbond XT resin cement (3M Unitek) in accordance with the manufacturer's instructions. The brackets were placed in the area previously demarcated on the surface of the incisal third of the labial face of the tooth, with the slot parallel to the long axis of the tooth, so that the factor of "wing deformity" would be minimized during the shear test. Force corresponding to 400 gf was applied on the bracket with a dynamometer (ETM, Monrovia, Calif), to standardize the bonding material thickness. Any excess material was removed with an exploratory probe. With the dynamometer still in place, light polymerization was performed with the Optilux 500 (Demetron Kerr, Danbury, Conn) with an irradiance of 600 mW/cm^2 for 20 seconds, 10 seconds of which were on the mesial and 10 seconds on the distal interface of the bracket, at a distance of 5 mm between the light beam and bracket. The light intensity was checked with the radiometer of the device.

To prepare the test specimens, a stainless steel metal device was used to guarantee the parallelism of the bracket slot in relation to the vertical plane and also to ensure that the bonding surface remained perpendicular to the horizontal plane and parallel to the direction of the force to be applied in the shear test, after the tooth was embedded in acrylic resin. This device was made with a rectangular stainless steel wire measuring 0.021 $\times$ 0.025 inch (3M Unitek) welded at an angle of 90° to its base. The bracket was fixed to this wire with an elastic ligature (3M Unitek). After this, the tooth root was embedded in Ortoglass self-polymerizing acrylic resin

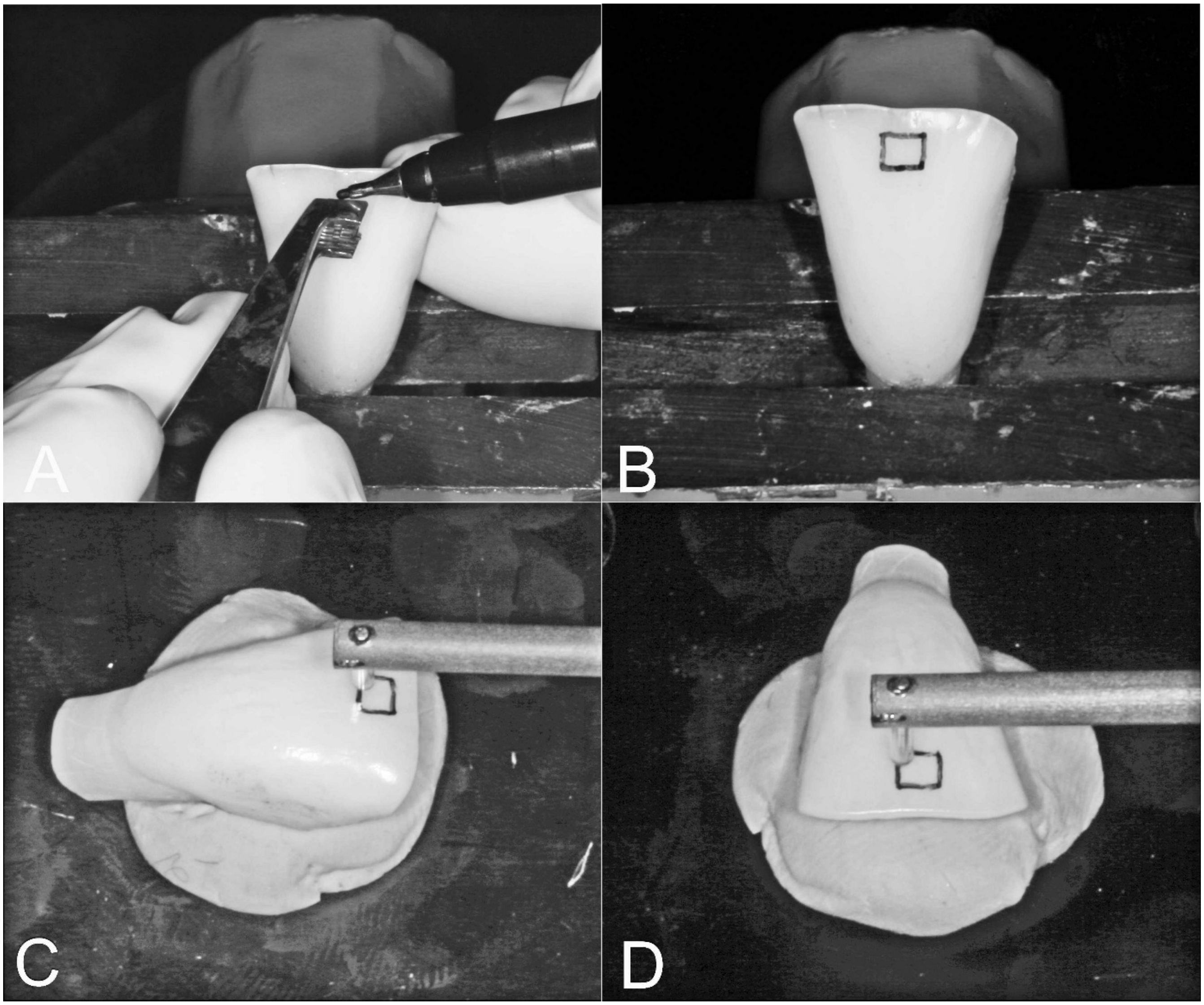

Figure 1. Sequence of roughness measurement. (A) Delimitation of the area to be measured. (B) Delimited area. (C and D) Measurements of longitudinal and transverse roughness in the long axis of the tooth.

(Clássico, São Paulo, Brazil) in a metal ring 2.5 cm in diameter and 2.0 cm high (Figure 2A through E). The test specimens were stored in distilled water at a temperature of 37°C for 24 hours. The brackets were then debonded.

To evaluate the bond strength, the test specimens were submitted to a shear test in an EMIC DL 500 universal test machine (Equipamento de ensaio, São José dos Pinhais, Brazil) with a 50 kg-f load cell at a speed of 0.5 mm/min in the occlusal-gingival direction until the bracket ruptured (Figure 2F). A computer connected to the machine recorded the rupture results, in megapascals, of each tooth submitted to testing in relation to the area of each bracket. Because all the teeth were numbered, it was possible to correlate the bond strength of the orthodontic brackets with the surface roughness of the enamel after the mechanical test.

Representative images of the bracket bonding area were obtained by scanning electronic microscopy (SSX 550 Superscan, Shimadzu, Kyoto, Japan) after the teeth were gold coated. Images were captured with a photographic camera (EOS Rebel XTi, Canon, Tokyo, Japan) fitted with a 50-mm f/2.8 EX DG macro objective (Figure 3).

Statistical analysis was performed with the Statistical Package for Social Sciences 13.0 for Windows software (SPSS Inc, Chicago, Ill) using the Kolmogorov-Smirnov, Pearson's correlation, and Student's *t*-tests.

RESULTS

The observed roughness values are shown in histograms (Figure 4A through C). Descriptive statistics for the roughness and bond strength are shown in Table 1.

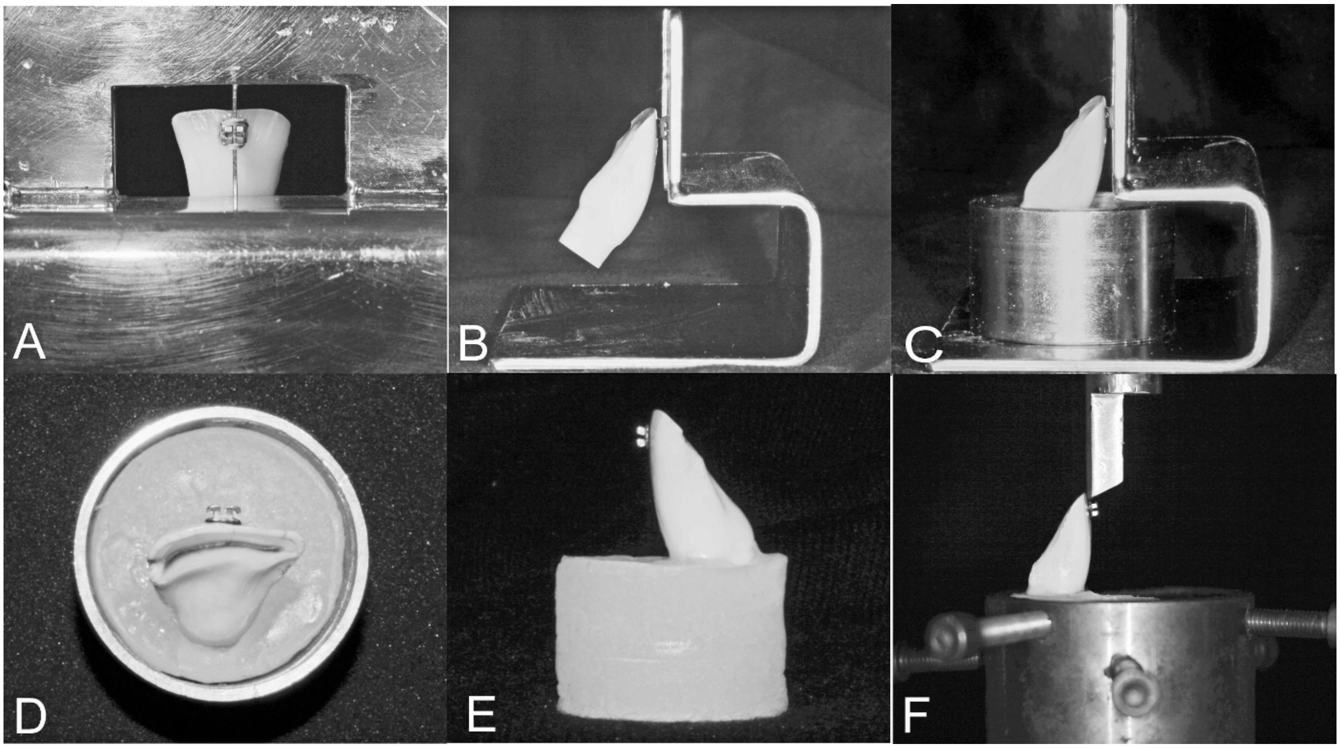

Figure 2. Sequence of mounting test specimens. (A and B) The tooth is fixed to a 0.021- × 0.025-inch wire with an elastic ligature. (C) Device with inclusion ring. (D) The root embedded in chemically cured acrylic resin. (E) Test specimen with bracket perpendicular to base and parallel to the direction of shear force. (F) Shear bond strength test.

To determine whether there was a correlation between the longitudinal and transverse roughness measurements along the long axis of the tooth for a sample size of n = 90, the normality of the data for both variables was tested using the Kolmogorov-Smirnov test at a significance of 5%. Both variables showed a normal distribution ($P > .05$).

Pearson's linear correlation test showed a correlation between the mean longitudinal and transverse roughness measurements of r = 0.632 ($P < .01$), indicating that this correlation was strong and statistically different. For the correlations between the mean longitudinal and transverse roughness measurements and shear bond strength, values of r = 0.048 ($P =$ .656) and r = –0.064 ($P = .547$), respectively, were obtained, indicating that the variable shear bond strength was not correlated with the longitudinal and transverse roughness. The same result was observed for the correlation between total mean roughness (longitudinal Ra and transverse Ra) and bond strength, showing that there was no statistically significant correlation ($P > .05$). The results of Pearson's linear correlation are shown in Table 2.

To evaluate the mean differences between the variables of longitudinal roughness and transverse roughness, Student's *t*-test was used for paired samples. This showed that there was a statistically significant difference between the variables ($P < .05$), with the mean transverse roughness being greater than the longitudinal roughness.

DISCUSSION

To conduct this research, bovine mandibular permanent incisors were used, because the enamel of these teeth is similar in composition and physical properties to human enamel, making them a valid substitute for research on human teeth.[22,23] To evaluate the bond strength, the shear test was used because this mechanical test exerts forces on the brackets similar to those exerted during orthodontic movements.[24]

Following ISO/TS 11405,[21] the extracted teeth were kept in 0.5% Chloramine T for disinfection and were stored in distilled water. Because teeth from humans and bovines may be contaminated, it is important that they be decontaminated before any laboratory testing.[25] Several studies[25–27] investigated whether the disinfection and/or storage media might influence the bond strength on dentin surfaces; Chloramine T and distilled water had no effect on bond strength. These findings can be extrapolated to enamel surfaces, considering that adhesion to dentin is much more complex than bonding to enamel because of the nature of this substrate.[28]

One of the requirements for obtaining good bonding is the formation of microporosities caused by etching. Nevertheless, studies can be found in the literature

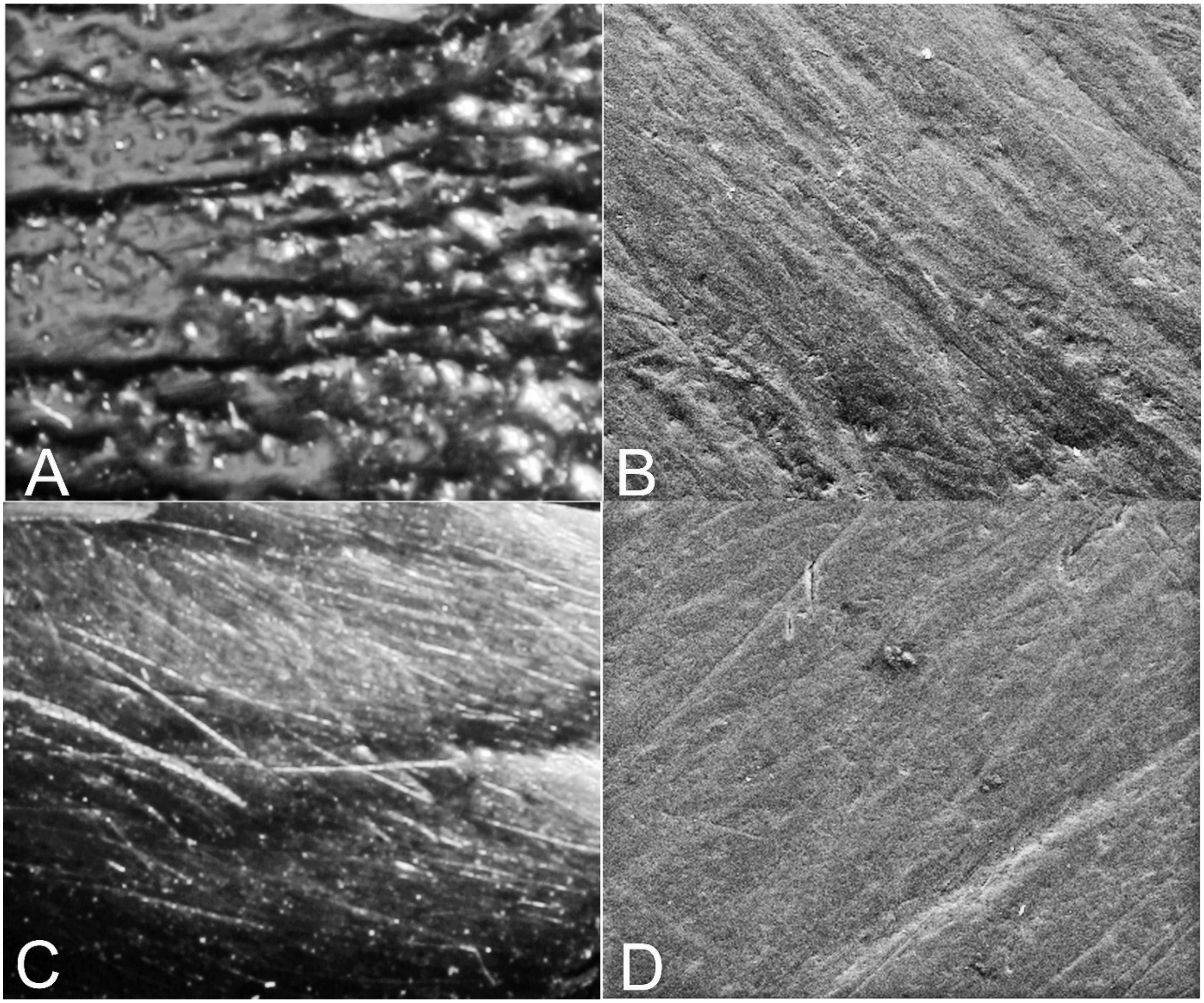

Figure 3. Images obtained with camera and scanning electron microscopy (×500). (A and B) Higher surface roughness. (C and D) Lower surface roughness.

that, in addition to using etching, used other forms of adherent surface preparations before bonding with the goal of increasing surface roughness and evaluating its influence on bond strength.[19,29,30] Powers and Messersmith[11] confirmed that the bond strength between enamel and brackets could be influenced by the type of tooth, high concentration of fluorides (fluorosis), concentration and time of etching, adhesive system, and bracket base, but they did not consider enamel surface roughness as a factor that could also interfere in this bond.

Eick et al[31] presumed that rougher and more irregular surfaces could promote greater adhesiveness because they presented a larger surface area and, consequently, that the bond strength between the adherent surface and the adhesive would be greater. Nevertheless, excessive roughness could impede total wetting of the adhesive and cause the formation of air bubbles at the adhesive/enamel interface. Therefore, the topography of the tooth could have an important role in bonding, since the presence of bubbles between the adhesive and the tooth surface could weaken the bond. This hypothesis should be tested, because the current study found a significant difference between the roughness characteristics in the longitudinal and transverse directions, and the mean bond strength was 13.3 MPa, a value that is much higher than that recommended by Reynolds[32] (5.9 to 7.8 MPa) for clinical bond strength requirements.

Jung et al[19] evaluated the influence of enamel roughness on the shear bond strength of a composite using different rotary instruments. The surface with the highest roughness values did not show a higher bond strength, and there was no correlation between the

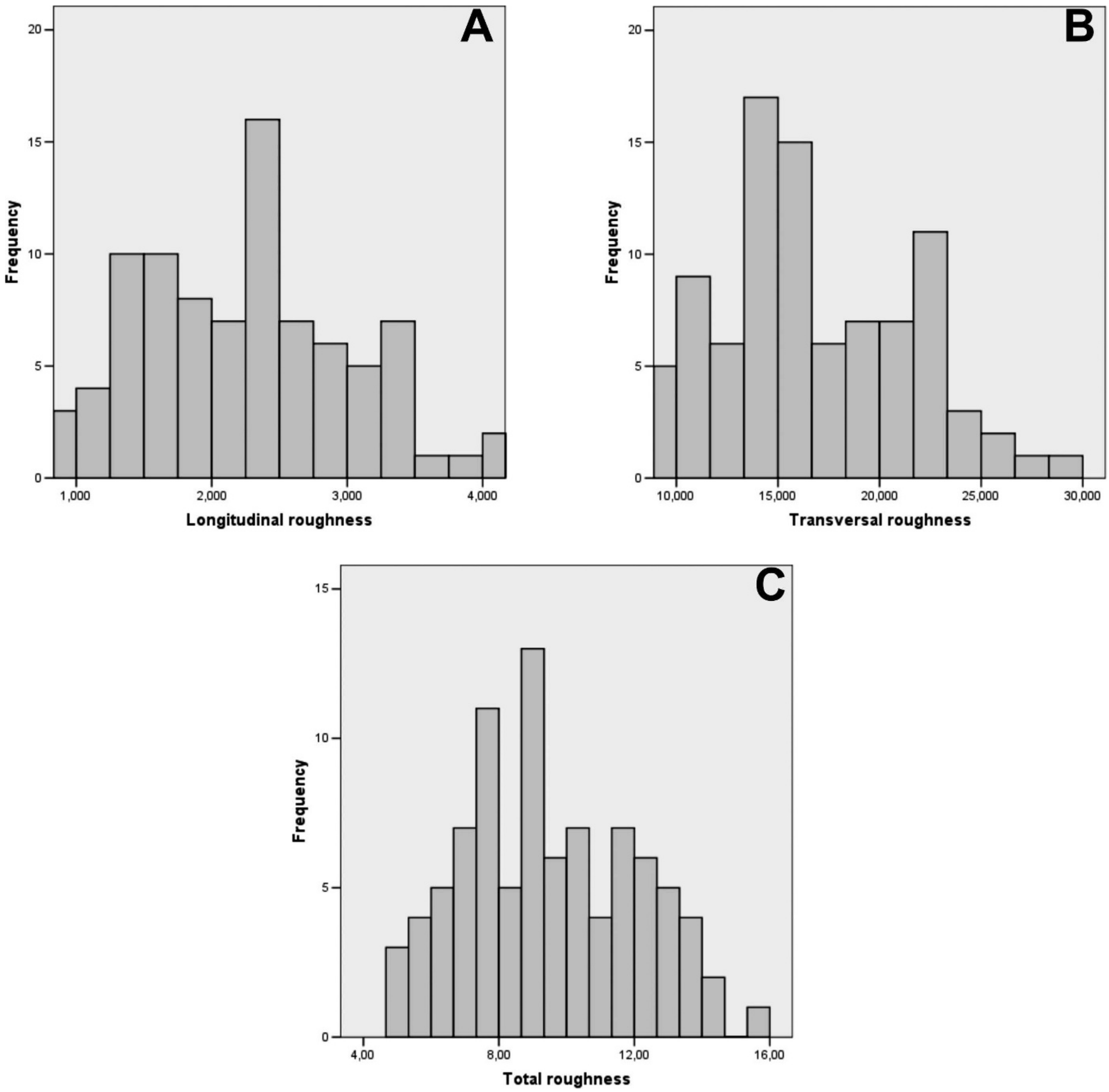

Figure 4. Histograms of (A) longitudinal roughness, (B) transverse roughness, and (C) total roughness variables.

roughness created by rotary instruments and shear bond strength. A similar result was found by Ariyaratnam et al,[29] who studied the correlation of enamel morphology with the bond strength of a composite, using an Nd:YAG laser at different parameters and 37% phosphoric acid to prepare the enamel surface and concluded that there was no correlation between roughness and bond strength.

Table 1. Descriptive Statistics for Roughness (μm) and Bond Strength (MPa)

Groups	n	Mean	Median	Minimum	Maximum	Standard Deviation
Longitudinal roughness	90	2.287	2.295	0.702	4.372	0.831
Transverse roughness	90	16.756	16.020	8.372	28.526	4.640
Total roughness	90	9.521	9.157	4.855	20.364	2.902
Bond strength	90	13.317	11.997	3.401	25.706	5.415

Total roughness was obtained by calculating the mean of the longitudinal and transverse values.

Table 2. Pearson Correlation for Longitudinal and Transverse Roughness and Bond Strength Variables

Variables	Correlation	P
Longitudinal roughness × transverse roughness	0.632	.000
Longitudinal roughness × bond strength	0.047	.656
Transverse roughness × bond strength	–0.064	.547
Total roughness × bond strength	–0.045	.669

$P < .05$ indicates statistically significant correlation between the variables.

Total roughness was obtained by calculating the mean of the longitudinal and transverse values.

The results of the present study were no different, showing that there was no correlation between the roughness of the enamel surface and bond strength and that surface roughness did not influence bond strength. This result is in agreement with the studies of Jung et al[19] and Ariyaratnam et al,[29] who varied the roughness of enamel before bonding by means of different rotary instruments, lasers, and etching. These studies did not support the hypothesis raised by Eick et al[31] that the topography of the adherent surface could affect bonding of an adhesive system. As in the present study, Pearson's linear correlation test showed that there was no correlation between roughness and bond strength; thus, there is no justification for the comparison of rougher or less rough enamel surface with bond strength.

It should be mentioned that a high standard deviation is an indication that it is not statistically confirmative that the different variations in enamel mineralization and structure could influence the bond strength values.[19] However, the present research also had a high standard deviation. Thus, the difference in roughness cannot be used as an argument, since the results showed that there was no statistically significant correlation between roughness and bond strength.

CONCLUSIONS

- The amount of transverse roughness is greater than the longitudinal roughness.
- There is a strong correlation between longitudinal and transverse roughness.
- There is no correlation between enamel roughness and bond strength.

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