



ORIGINAL ARTICLE

Experimental study of brackets adhesion with a novel enamel-protective material compared with conventional etching

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Abstract *Introduction:* A reliable adhesion between fixed devices and dental surfaces is a key factor for the clinical success of any orthodontic treatment. Adhesion preparation is associated with damages related to abrasive cleaning, enamel structure defacing caused by etching, enamel loss when removing resin remnants at orthodontic treatment finishing stage or when conditioning surface for adhesive failure and fractures at bracket removal.

Aim: The objective of this study was to compare the shear bond strength of metallic brackets to enamel adhered with a novel non-damaging and remineralizing material for enamel versus the traditional 37% phosphoric acid etching.

Material and methods: 75 Premolars collected from 15- to 40-years old healthy donors requiring extraction were collected. The teeth were then randomly divided into three groups (n = 25). One group was used for the experimental new method (EX), the second for the conventional phosphoric acid etching (PA) method and the third group was left without any treatment (NT). The metallic brackets were fixed with Transbond® XT adhesive and composite resin polymerized for 40 s with a halogen photocuring lamp. The shear bond strength was quantified by means of a universal testing machine at 1 mm/min crosshead speed and a load cell of 1 kN.

Statistical analysis used: Tests of normality, adjustment of the data to a root square, a one-way ANOVA and Tukey tests were performed.

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Results: Statistically significant differences between the NT (1.4 MPa), PA (32.1 MPa) and EX (9.7 MPa) groups were observed.

Conclusions: The experimental material for conditioning human enamel induces calcium phosphates crystals on the enamel surface and improves the bond strength in comparison to the NT group.

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1. Introduction

A reliable adhesion between fixed devices and dental surfaces is a key factor for the clinical success of any orthodontic treatment (Verma et al., 2013). Debonding of brackets is closely related to lengthier treatment duration (Skidmore et al., 2006).

Enamel etching of dental structures increases adhesion to brackets (Amm et al., 2008). Several etching acids such as phosphoric, polyacrylic, maleic, and itaconic-oxalic acids mixtures have been used for this purpose (Fjeld and Øgaard, 2006; Zhu et al., 2014). Other surface treatments include Nd: YAG laser (Sallam and Arnout, 2018) and aluminum oxide air-abrasion (Canay et al., 2000). Etching with 37% phosphoric acid for 15 s renders superior bond strength than Er: YAG laser or air-abrasion (Hamamci et al., 2010).

Adhesion is not the sole property that defines the quality of a surface treatment. Dental enamel is a highly mineralized tissue characterized by its years-long slow formation, limited quantity and its loss irreversibility (Fukae, 2009; Dorozhkin, 2013). Adhesion preparation is associated with several procedures such as damages related to abrasive cleaning, enamel structure defacing caused by etching, enamel loss when mechanically removing resin remnants or when failure and fractures at bracket removal are generated (Arhun and Arman, 2007).

A 37% aqueous solution of phosphoric acid when applied for a brief lapse (15 to 30 s) dissolves enamel minerals creating roughness with a wide extent of coverage that is microscopically visible from 3, 10 and even 170 μm (Øgaard and Fjeld, 2010). Er: YAG laser is aggressive for dental surface and sand-blasting erodes the enamel inducing shear bond strength quite smaller than those of phosphoric acid or self-etching systems (Türköz and Ulusoy, 2012).

Options to avoid harming enamel include removing cement and resin traces with sof-lex® discs (Grocholewicz, 2014), enamel protectors such as fluoride varnishes, infiltration resins (Montasser and Taha, 2014), or remineralizing agents as bioactive calcium silicate among others (Li et al., 2014). This article contains the proposal of a novel material and conditioning method to create an enamel remineralizing coating that protects the tissue and promotes adhesion. This is done by comparing the shear bond strength of metallic brackets on enamel adhered by the proposed method versus the traditional 37% phosphoric acid etching.

2. Materials and methods

The teeth collected with the approval of the Ethics Committee of the Faculty of Dentistry at Universidad Nacional de Colombia, after delivering an informative sheet and signing

the written informed consent of the donors. Thus, 75 premolars obtained from healthy donors between 15 and 40 years of age requiring extraction for orthodontic treatment were collected. The samples excluded teeth with an associated diagnosis of periodontal disease, pulpal pathology, caries or fractures. Teeth with moderate or severe pre-eruptive defects in the enamel, amalgam, adhesive restorations, rehabilitation and internal or external whitening were also excluded. The specimens were deposited in high density polyethylene (HDPE) containers with aqueous 0.5% Chloramine-T solution (Jaffer et al., 2009).

The collected teeth were randomly divided into three 25 specimen groups. One group for the assessment of experimental new method, the second for the conventional phosphoric acid etching method and the third group assigned to undergo no treatment (negative control). Every tooth was cleaned with a toothbrush (Oral-B Prosalud, P&G, Cali, Colombia) and USP (United States Pharmacopoeia) purity grade aqueous sodium lauryl sulfate surface active agent (solution 1%wt, Sigma-Aldrich 1614363 USP, San Luis, Missouri, USA) to avoid introducing fluoride from dental pastes and then thoroughly rinsed with distilled water and blow-dried with oil-free air.

2.1. Surface treatments

The teeth in the conventional group (PA group) were etched with 37% orthophosphoric acid (Super Etch®, Southern Dental Industries – SDI, Bayswater, Victoria 3153, Australia) for 15 s, washed for 20 s with distilled water followed by a micro-brush applied layer of adhesive (Transbond™ XT 3 M Unitek, Maplewood, Minnesota, USA) and polymerized during 20 s. Finally the metallic brackets (Miniature Twin, 3M Unitek, Maplewood, Minnesota, USA) were fixed with a composite resin (Transbond® XT, 3M Unitek, Maplewood, Minnesota, USA) polymerized for 40 s by means of a halogen photocuring lamp (Sunlite 1275, Omedis LTDA, Medellín, Colombia) endowed with a 450 mW/cm^2 power. In the group with no treatment or negative control (NT) the teeth did not receive any surface treatment. The adhesive and resin were applied similarly to the PA group but, without any etching. For the experimental group, the teeth were covered with wax except for a square window on the vestibular side aimed for the conditioner. Within this window on the enamel, a blanket of powdered solid conditioner was set and a few drops of the liquid part of the conditioner was also placed to obtain a paste that seated on the enamel for 48 h at room temperature (20 °C) (Fig. 1). After this, every tooth was thoroughly washed with deionized water, dried and studied under the scanning electron microscope to ensure that each tooth had been coated. No phosphoric acid etching was used in the experimental group.

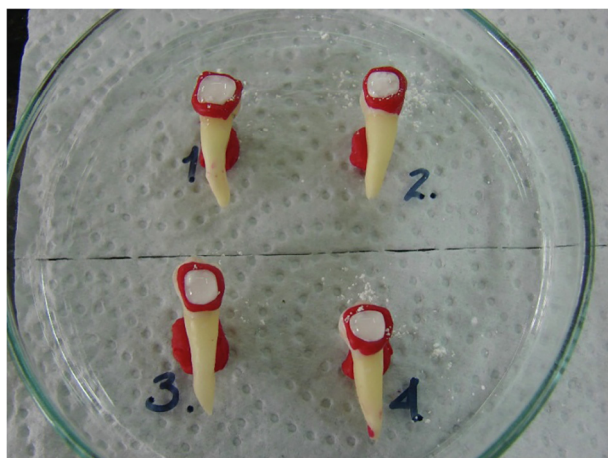


Fig. 1 Applied substance, experimental group.

The lapse of 48 h was determined by test runs observing the surface coatings by SEM (Fei, Quanta 200, Hillsboro, Oregon 97124, The USA). A layer of adhesive (Transbond™ XT 3M Unitek, Maplewood, Minnesota, the USA) with microbrush was applied followed by 20 s of polymerization and placement of premolar metallic brackets (Miniature Twin, 3M Unitek, Maplewood, Minnesota, USA) with composite resin (Transbond® XT, 3M Unitek, Maplewood, Minnesota, USA) polymerized during 40 s with the above mentioned halogen photocuring lamp.

2.2. Bond strength test

The three teeth groups were included in self-curing acrylic cylinders with the treated enamel flush with the surface to measure the shear bond strength in a universal testing machine (Shimadzu AG-IS 5 kN), calibration certificate (No. 1289-2008, CONCRELAB). A device with a steel blade shape descended and applied a force to the bracket base until separation was achieved (this force was expressed in newtons, N) (Fig. 2). The adhesion area (9.87 mm^2) was used to divide the force of rupture or failure between the constant adhesive area and

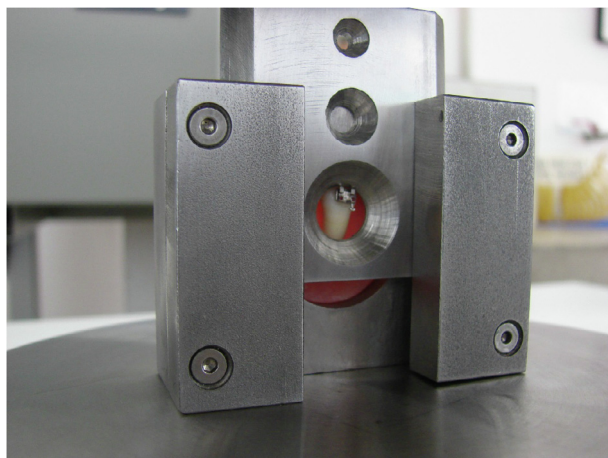


Fig. 2 Device with a steel blade shape.

convert the results in megapascals (MPa). A load cell of 1 kN to 1 mm/min was used (Scribante et al., 2016).

2.3. Statistical analysis

In order to assess the normality of the bond strength data, a Q-Q plot was drawn for an Anderson-Darling test. In the absence of normality, a model of mean square root adjustment was performed whereby the normality was achieved (see Figs. 4 and 5). Afterwards a one-way ANOVA and Tukey test were run (MINITAB version 17.2.1, Minitab Inc, Pennsylvania State University, Pennsylvania, USA).

Window limited by wax and conditioning on the vestibular surface. The paste seated on the enamel for 48 h at 20 °C.

3. Results

The highest bond strength was observed in the phosphoric acid etching group (Fig. 3 and Table 1). The verification of normality parameters was performed (Fig. 4), followed by the adjustment of the model by the square root of bond strength (Figs. 5 and 6). All three groups reported statistical differences according to the ANOVA and Tukey tests (Tables 2 and 3).

4. Discussion

In this study the bond strength of the three groups was deemed statistically different. The experimental and acid-etched groups were superior to the group without treatment.

In 1979, Reynolds stated that the minimum bond strength should be between 5.9 y 7.9 MPa. This range was exceeded by the experimental and acid etching groups. The Reynolds minimum values range is to be reconsidered under the light of advances in adhesion technology up to these days. Recent information provided by Reicheneder et al. (2009) evaluated

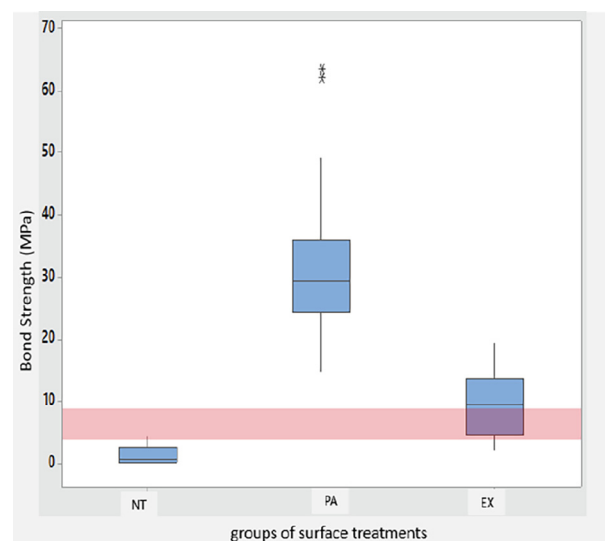


Fig. 3 Descriptive results of bond strength: Box-plot of bond strength (MPa) for the three groups. The red band highlights an acceptable range of bond strength values, 5.4-8 MPa (Reynolds, 1979; Reicheneder et al., 2009., Kumar et al., 2011).

Table 1 Descriptive results of bond strength.

Groups	Mean	Standard deviation	Median	Maximum values	Minimum value
NT	1.42	1.38	0.78	4.54	0.00
PA	32.10	12.41	29.48	63.43	14.81
EX	9.66	5.55	9.58	19.53	3.07

Data expressed in megapascals (MPa).

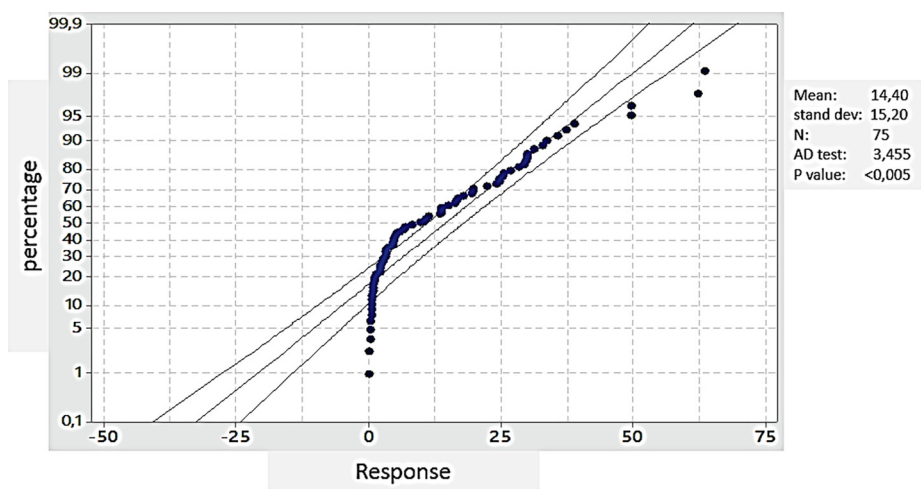


Fig. 4 Q-Q plot to determine normality: Q-Q Plot of the three groups initials data where a non-normality behavior is observed.

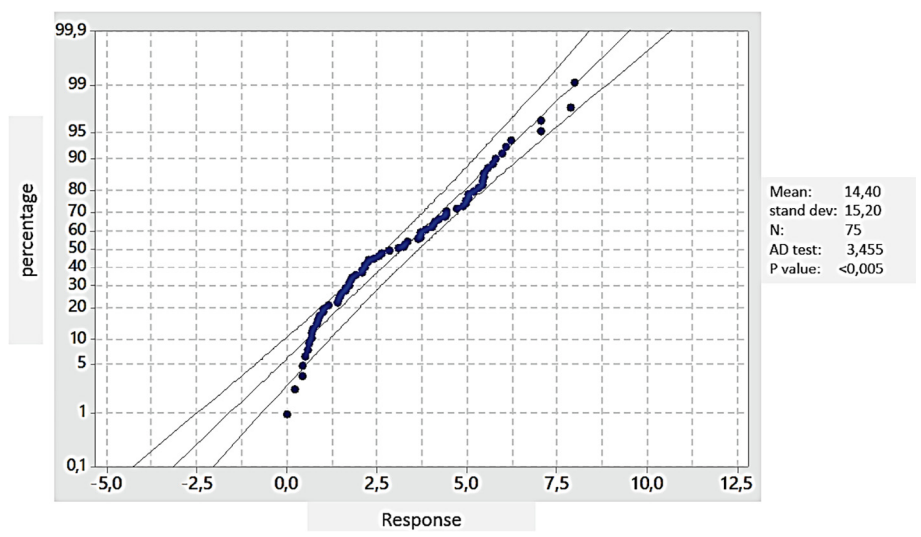


Fig. 5 Q-Q plot of adjusted model: Q-Q Plot of square root adjusted model.

the brackets to bovine enamel shear bond strength of eight adhesive systems arriving at a range of means between 5.47 and 7.24 MPa and medians between 5.8 y 7.75 MPa as clinically acceptable. The results for the acid etching group as well as the experimental group lie within bond strength acceptable limits.

Kumar et al., 2011 studied the metallic brackets shear bond strength of three light cure adhesive-Resins (Admira, Grandio, and Transbond XT) on groups of fifteen human premolars and

obtained mean values 8.50 ± 5.22 , 8.13 ± 4.35 MPa and 5.59 ± 1.91 MPa (Transbond XT, Grandio and Admira respectively). The recommended materials were Transbond XT and Admira and therefore strengths between 6 and 8 MPa are regarded suitable. Most of the experimental group results are better than this acceptable range. It should also be noted that Kumar results show great dispersions.

Establishing the optimum adhesion value for an ideal clinical performance by means of in-vitro trials poses many

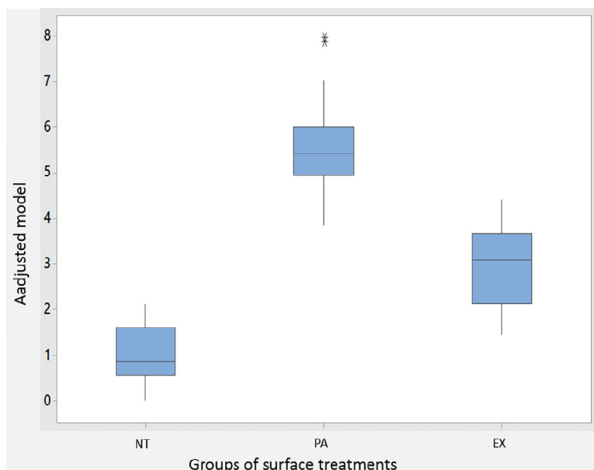


Fig. 6 Descriptive results of bond strength (adjusted model): Box-plot of square roots adjusted model.

Table 2 Variance analysis (ANOVA).

Source	DF	SC adjust.	MC adjust.	F value	p value
Treatment	2	258.75	129.377	169.19	0.000
Error	72	55.06	0.765		
Total	74	313.81			

Model summary

S	R-square.	R-square. (adjusted)	R-square. (pred.)
0.874457	82.46%	81.97%	80.96%

hindrances (Verma et al., 2013). There are many parameters that should be standardized such as, adhesive system (Hattar et al., 2015), type of tooth, adhesion surface (vestibular or lingual), enamel chemical composition, bracket base shape (Scribante et al., 2013), surface materials (Hattar et al., 2014) or surface contamination (Al Qahtani and Al Shethri, 2010), direction of debonding applied force on bracket, spot where applied force on bracket is exerted, debonding time, storing conditions (water, air), light-curing time, and universal testing machine crosshead speed. All these variables affect the results of in-vitro tests (Finnema et al., 2010; Lou et al., 2009; Klocke and Kahl-Nieke, 2006; Yamamoto et al., 2006). These many parameters might explain the variability displayed among studies and even within treatment groups as evidenced in the present work. Therefore the results of the present investigation represent data that should be confirmed with further studies

that assess enamel protecting materials both in vitro and in vivo.

It is worth emphasizing that nowadays it is almost impossible to collect teeth without fluoride since the use of fluoridated toothpastes is well extended and there are many other fluoride sources such as water in some municipalities, salt, tea, and many others. Even those teeth that are apparently fluoride free because of the absence of white spots may display substantial fluoride concentration on the surface that, as it is well known, inhibit adhesion. Some authors such as Wiltshire and Noble in 2010 reported that fluorosed enamel does not affect bond strength while others such as Mendes et al. (2014) experimentally proved that fluorosed enamel reduces bond strength when compared to healthy enamel.

A more reasonable approach that dodges the dispersion of results consist in minimum adhesion strength values assessed in in-vitro experiments via bond shear strength studies. Clinically suitable strengths should be above 3 or 4 MPa (Wiltshire and Noble, 2010). The average value for the experimental group (near 10 MPa) is above this limit as stated in Ref. Pont et al. (2010).

The traditional method with 37% phosphoric acid etching for 15 s far exceeded the minimum standards (Reynolds, 1979). Some fractures have been reported at values as low as 9.7 MPa (Wiltshire and Noble, 2010), which may imply that it is not recommended to have bond strength as high as (63.44 MPa). It is well known that the phosphoric acid creates a rough surface on enamel with more or less deep porosity as a result of demineralization by the withdrawal of calcium phosphates from the enamel surface therefore destroying its hierarchical structure (Gandhi et al., 2018) in an irreversible way. The situation is worsen by the removal of the brackets that further spoil the enamel as well as the damage caused by the final step of polishing for eliminating adhesives remnants (Pont et al., 2010).

The idea of protecting the enamel is not new. The protection arises from the growth of crystals on the surface to act as an adhesion substrate for cementing agents for orthodontics as an alternative to elude crystals dissolution inherent to phosphoric acid etching (Øgaard and Fjeld, 2010).

Majjer and Smith, 1979, reported that a mixture of polyacrylic acid and 40% ammonium persulfate as initiator used for 2–6 min was effective for inducing the precipitation of salts crystals on upper premolars enamel yielding adhesions tensile strength similar to the values attained with one minute of 36% phosphoric acid.

The same researchers published another study in 1986 carried out with patients with ages between 10 and 32 years using the “divided mouth methodology”. This time one group had a sulfated polyacrylic acid for 30–90 s and the second group had 37% phosphoric acid for 60 s prior to bonding the brackets

Table 3 Bond strength results of the adjusted model.

Groups	n	Mean (standard deviation)	Confidence interval 95%	min	Median	max
NT	25	1.04 (0.59) ^A	(0.69–1.39)	0.00	0.88	2.13
PA	25	5.57 (1.03) ^B	(5.22–5.92)	3.84	5.43	7.96
EX	25	2.97 (0.93) ^C	(2.62–3.32)	1.43	3.09	4.42

Different superscript capital letter indicates statistical differences according to Tukey test ($p < 0,05$). Data expressed in megapascals (MPa).

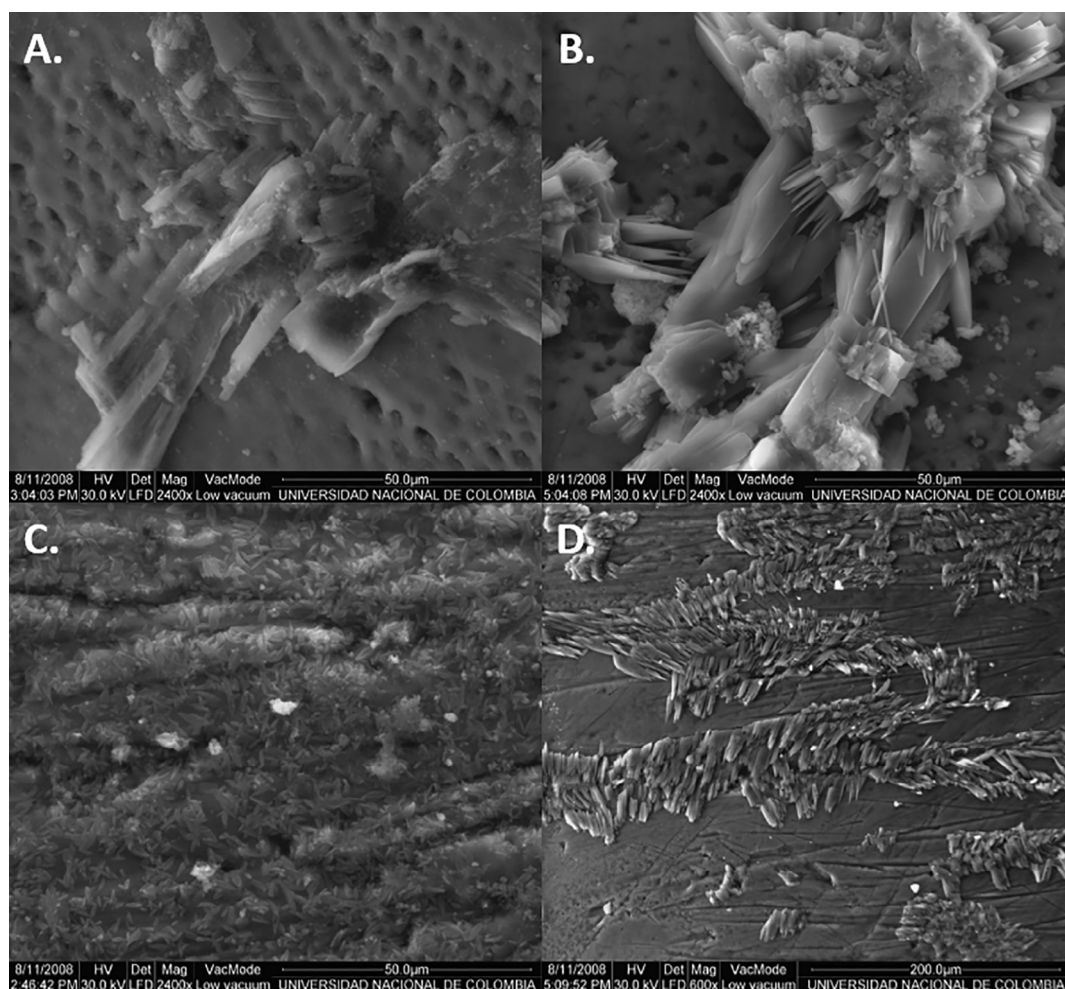


Fig. 7 Scanning electron microscopy (SEM): four different samples observed with the scanning electron microscope (SEM): A and B show, at the same magnification, details of newly deposited mineral on the enamel surface. C. exhibits a profuse deposit while D at a lesser magnification displays larger crystals originated along surface defects.

with light cure adhesive and resin composite. The number of debonded brackets within 22 months was the evaluation meter and no meaningful differences were found.

It can be argued that the acid etching in both studies was applied much longer (2–6 min. and 1 min, respectively) than the time currently recommended (15 s). These results should be scrutinized under contemporary advanced adhesive systems as well as careful standardization since new different conclusions could be withdrawn.

The Surface crystals formation was demonstrated, by Maijers and Smith and also in the present work, (Fig. 7A–D), however the results are different due to the fact that the chemical approach is different. In Maijers' work, the material was an organic polymer that is cytotoxic and inexistent in living beings, in contrast with the present paper where the material comprises a biomimetic blend of various phosphates that closely imitates the natural inorganic composition of enamel. The experimental material application time needs more research to reduce it to a clinically suitable lapse. It would also be important to study the approach to obtain only one type of crystal topography on the enamel surface. Some insight can be gained by de use of the adhesive remnant index ARI to make clear the debonding pattern (Artun and Bergland, 1984).

The original purpose of the proposed method is to protect enamel and to remineralize and strengthen it instead of destroying it, making it more porous and prone to acid attack therefore facilitating the rise of sensibility. This makes it worthwhile to continue the study of the novel material and its application.

5. Conclusions

The present work as described leads to the following statements:

- The experimental material for conditioning human enamel induces calcium phosphates crystals on the enamel surface and these foster the brackets adhesion.
- The concept of the 37% phosphoric acid etching as the gold-standard for bracket adhesion is reinforced as shown by the high bond strengths well above the minimum recommended values.
- Although the experimental adhesion values are not high enough for substituting the phosphoric acid procedure for brackets, the results indicate they are acceptable and a possible alternative that requires further research.

- The adhesive mechanism by means of formation of new crystal structures is not thoroughly understood and therefore requires a deeper look into it to clarify it. The advantages of a surface treatment that protects the integrity of enamel as a non-renewable resource are of the utmost importance.

Declaration of Competing Interest

The authors declare there are no potential conflicts of interest with the materials involved in the present investigation.

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