Clinical Oral Investigations (2021) 25:1953–1965
https://doi.org/10.1007/s00784-020-03504-0

ORIGINAL ARTICLE

Effect of a single-component ceramic conditioner on shear bond strength of precoated brackets to different CAD/CAM materials

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Received: 15 April 2020 / Accepted: 5 August 2020 / Published online: 15 August 2020

Abstract

Objectives To compare the shear bond strength (SBS) of the CAD/CAM material-bracket interface using three surface treatments: following manufacturers' instructions (MI), Monobond Etch & Prime (MEP) and 9.6% hydrofluoric acid plus silane (9.6% HF), after 24 h of water storage (24 h) and 10,000 cycles of thermocycling (TC).

Materials and methods A total of 126 crowns with four identical buccal surfaces were fabricated using seven different CAD/CAM materials: CEREC Blocs unglazed (CBU), CEREC Blocs glazed (CBG), IPS Empress CAD (EMP), IPS e.max CAD (EMA), VITA SUPRINITY PC (SUP), inCoris TZI (TZI) and VITA ENAMIC (ENA). A total of 504 APC Flash-Free (APC FF)-precoated brackets were bonded applying three surface treatments: (1) MI; (2) MEP and (3) 9.6% HF. SBS was performed after 24 h and TC. Results were analyzed by Kruskal-Wallis and Mann-Whitney *U* tests ($p < 0.05$).

Results MEP conditioning yielded lower SBS results compared with MI and 9.6% HF for CBG (24 h and TC) and EMA (TC) materials. EMP conditioning with MEP after 24 h obtained lower SBS values compared with MI; however, after TC, SBS was similar to MI group and higher than with 9.6% HF. After TC for TZI ceramic, MI protocol (sandblasting) obtained higher SBS scores than MEP, but similar than 9.6% HF. Treatment of ENA with MI and MEP produced higher results than 9.6% HF after TC. SBS results were similar for CBU and SUP, regardless of the treatment.

Conclusions Although each CAD/CAM material requires specific surface treatment to obtain the highest SBS of APC FF brackets, the treatment with MEP is a valid orthodontic alternative for most of the materials tested. TC significantly decreased SBS for most of the materials.

Clinical relevance MEP can be considered a valid and promising product to condition most of the CAD/CAM ceramics evaluated for APC FF bracket bonding purposes, allowing a faster and safer procedure.

Keywords CAD/CAM · Monobond Etch & Prime · APC Flash-Free · Dental ceramics · Orthodontics · Shear bond strength

Introduction

At present, the orthodontic treatment of adult patients is becoming more and more frequent [1–3] due to the great importance of facial esthetics in the society [4] and the evolution of orthodontics over the last few decades. This fact entails the need to treat adult patients, who often present crowns and other ceramic restorations, performed prior to the orthodontic treatment. Thus, bonding of different orthodontic devices such as buccal or lingual brackets, tubes, attachments and buttons mainly to ceramic surfaces is becoming ever more frequent.

Nowadays, the tendency is to use computer-aided design/computer-aided manufacturing (CAD/CAM) materials to restore the worn dentition [5, 6]. Due to the fact that many CAD/CAM materials are available in the market for its use, different conditioning protocols should be performed according to the composition of the material involved [7–9]; otherwise, the mechanical properties of the ceramic could be compromised.

The treatment of choice to condition glass-matrix ceramics and polymer infiltrated ceramic networks (PICN) is the use of

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hydrofluoric acid (HF) on a concentration between 5 and 12%; being 5% and 9.6% concentrations the most commonly used [10, 11]. Traditionally, a feldspathic ceramic has been the material of choice to develop veneers or to cover the metal structure of porcelain fused to metal crowns. This fact explains why orthodontists have usually used HF at a concentration around 10% during 1 or 2 min to etch ceramic for direct bracket bonding. Nevertheless, for the treatment of CAD/CAM materials, a lower concentration of HF is recommended, being the use of HF at 5% concentration and at different etching times, together with silane application, the gold standard method to condition CAD/CAM glass-matrix ceramics and PICN materials [10–12]. The etching with HF dissolves the superficial layer of the ceramic and creates a more irregular surface that promotes a micromechanical retention [13] with different dissolving patterns for each ceramic [9, 13]. On the other hand, silanes work as bifunctional coupling agents allowing a chemical union with hydroxyl groups of ceramic and with methacrylates [14, 15]. Ultimately, for zirconium oxide, sandblasting with aluminum trioxide followed by the application of a silane containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) is the protocol of choice for bonding [16].

The use of HF represents a challenge for the professional due to the causticity and corrosiveness of the acid, being critical its careful use to avoid lesions in eyes and skin, either for the patient or the clinical staff [17]. In addition, for orthodontic purposes, ceramic etching with HF takes place inside the patient's mouth, thus increasing the risk of injuring soft tissues. Other drawbacks of this technique are its higher bracket debonding rate [8] and that it requires more clinical steps compared with the conventional enamel bonding method, which makes the conditioning process more complex and more technically sensitive. Apart from the importance of the conditioning process, the bond strength of the ceramic-bracket interface is one of the keys of a successful orthodontic treatment, where neither bracket debonding during the treatment nor ceramic chipping or fractures when debonding, due to an excessive bond strength, are desirable situations [18, 19]. Thus, the optimal bond strength would be situated somewhere in between of those two scenarios.

Recently, a single-component ceramic conditioner (Monobond Etch & Prime, Ivoclar Vivadent AG, Schaan, Liechtenstein) (MEP) based on an ammonium polyfluoride (tetrabutylammonium dihydrogen trifluoride) was made available. Presumably, this novel ceramic conditioner allows treating the glass-ceramic materials in one step, by etching and silanating simultaneously, reducing bonding time and the toxic potential of HF [20]. Several studies have been published since the apparition of MEP in 2015 [21–24], finding heterogeneous results. To the best of our knowledge, there are only two published studies evaluating this product for orthodontic bonding purposes, which could be another indication of MEP. However, MEP was only tested in zirconia [25] and leucite-reinforced glass ceramic [26] blocks; thus, the performance on other CAD/CAM materials for bracket bonding is still unknown.

Therefore, the objectives of this study were (1) to compare the shear bond strength (SBS) of the CAD/CAM material-bracket interface using three surface treatments: following manufacturers' instructions (MI), Monobond Etch & Prime (MEP) and 9.6% hydrofluoric acid plus silane (9.6% HF); and (2) to determine the influence of thermocycling (TC) on the SBS for the three surface treatments. The two null hypotheses were that the surface treatment does not affect the SBS of precoated brackets obtained for the different CAD/CAM materials tested and that thermal aging has no effect on the SBS neither.

Materials and methods

A maxillary first human premolar was selected and prepared following the prosthetic guidelines for an all-ceramic crown preparation and digitally scanned (TRIOS 3, 3Shape, Copenhagen, Denmark). Subsequently, that preparation was digitally customized to allow the fit of a crown with four buccal surfaces, together with the incorporation of a cubic base to enable the fixation of the structure to perform the SBS test. To conclude with the digital design, a crown with four identical first premolar buccal surfaces was created, allowing it to fit properly on the abutment previously designed (Fig. 1).

Then, 126 abutments based on the preparation performed above were printed by the same 3D printer (Form 2 3D printer, FormLabs, Somerville, MA, USA) in acrylic resin (Standard grey resin, FormLabs). In order to mill all the crowns included in the study, the acrylic crown printed previously was scanned with an intra-oral scanner (Omnicam, Dentsply Sirona, York, PA, USA) and digitally designed using the appropriate software (CEREC 4.4.5, Dentsply Sirona).

Thereafter, 126 crowns, fabricated from seven different CAD/CAM blocks, were milled with the CEREC inLab MC XL (Dentsply Sirona) milling machine. The crowns that needed to be crystallized were sintered according to the manufacturers' instructions for each material (Table 1). The CAD/CAM materials tested were:

(1) Feldspathic ceramic unglazed: CEREC Blocs (Dentsply Sirona) (CBU).
(2) Feldspathic ceramic glazed: CEREC Blocs + IPS e.max CAD Crystall./Glaze Spray (Ivoclar Vivadent) (CBG).
(3) Leucite-reinforced glass ceramic: IPS Empress CAD (Ivoclar Vivadent) (EMP).
(4) Lithium disilicate-reinforced glass ceramic: IPS e.max CAD (Ivoclar Vivadent) (EMA).

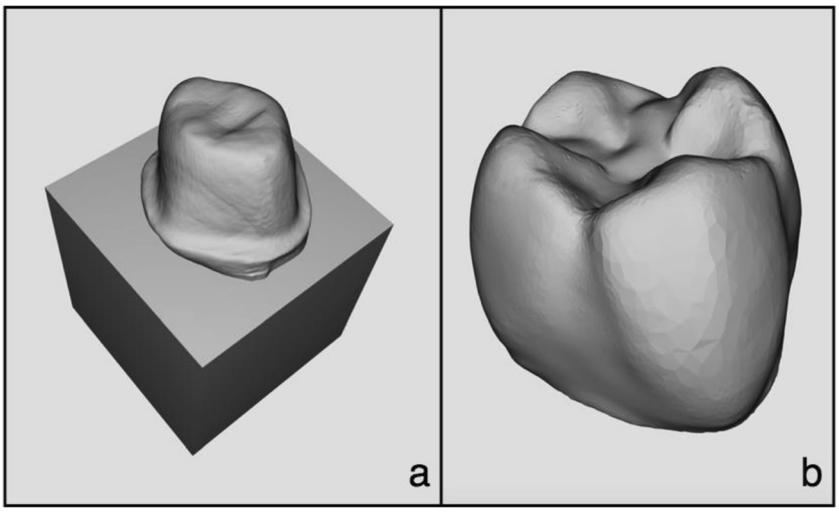

Fig. 1 Images obtained from the original STL files: **a** abutment together with the cubic base and **b** crown design with four identical buccal surfaces

(5) Zirconia-reinforced lithium monosilicate glass-ceramic: VITA SUPRINITY PC (VITA Zahnfabrik, Bad Säckingen, Germany) (SUP).
(6) Monolithic zirconia: inCoris TZI (Dentsply Sirona) (TZI).
(7) Polymer infiltrated ceramic network (PICN): VITA ENAMIC (VITA Zahnfabrik) (ENA).

Then, all the resin abutments were sandblasted with 50 μm aluminum oxide powder (Airsonic Alu-Oxyd, Hager & Werken, Duisburg, Germany) for 20 s at a pressure of 2 bars and then cleaned with the use of an ultrasonic bath with distilled water for 10 min. The crowns were adhesively luted with a self-adhesive resin cement (RelyX Unicem 2 Automix, 3M Oral Care, St Paul, MN, USA). Once all the crowns were

Table 1 Manufacturers, products, chemical composition and production of all the ceramics included in the study

Manufacturer	Product	Chemical composition	Production
CEREC Blocs (Dentsply Sirona) (CBU)	Feldspathic ceramic	SiO_2 (56–64 wt%), Al_2O_3 (20–23 wt%), Na_2O (6–9 wt%), K_2O (6–8 wt%), CaO (0.3–0.6 wt%), TiO_2 (0.0–0.1 wt%).	Milling (CEREC inLab MC XL, Dentsply Sirona)
CEREC Blocs (Dentsply Sirona) + IPS e.max CAD Crystall./Glaze Spray (Ivoclar Vivadent) (CBG)	Feldspathic ceramic + glazing spray	SiO_2 (56–64 wt%), Al_2O_3 (20–23 w%, Na_2O (6–9 wt%), K_2O (6–8 wt%), CaO (0.3–0.6 wt%), TiO_2 (0.0–0.1 wt%) + 40–60% powder [SiO_2 (60–65 wt%), K_2O (15–19 wt%), Al_2O_3 (6–10.5 wt%), other oxides and pigments (5.5–30 wt%)], propanolol (15–20%), isobutane (20–40%), butanediole.	Milling + sintering (CEREC inLab MC XL + CEREC SpeedFire, Dentsply Sirona) *Glazing spray applied at a distance of 10 cm for 1s per surface.
IPS Empress CAD (Ivoclar Vivadent) (EMP)	Leucite-reinforced glass-ceramic	SiO_2 (60–65 wt%), Al_2O_3 (16–20 wt%), K_2O (10–14 wt%), Na_2O (3.5–6.5 wt%), other oxides (0.5–7 wt%), pigments (0.2–1 wt%).	Milling (CEREC inLab MC XL, Dentsply Sirona)
IPS e.max CAD (Ivoclar Vivadent) (EMA)	Lithium disilicate--reinforced glass-ceramic	SiO_2 (57–80 wt%), Li_2O (11–19 wt%), K_2O (0–13 wt%), P_2O_5 (0–11 wt%), ZrO_2 (0–8 wt%), ZnO (0–8 wt%), Al_2O_3 (0–5 wt%), MgO (0–5 wt%), colouring oxides (0–8 wt%).	Milling + sintering (CEREC inLab MC XL + CEREC SpeedFire, Dentsply Sirona)
VITA SUPRINITY PC (VITA Zahnfabrik) (SUP)	Zirconia-reinforced lithium monosilicate glass-ceramic	SiO_2 (56–64 wt%), Li_2O (15–21 wt%), ZrO_2 (8–12 wt%), La_2O_3 (0.1 wt%), pigments (< 10 wt%), various (> 10wt%).	Milling + sintering (CEREC inLab MC XL + CEREC SpeedFire, Dentsply Sirona)
inCoris TZI (Dentsply Sirona) (TZI)	Monolithic zirconia	ZrO_2+HfO_2+Y_2O_3 (≥ 99.0 wt%), Al_2O_3 (≤ 0.5 wt%), other oxides (≤ 0.5 wt%).	Milling + sintering (CEREC inLab MC XL + CEREC SpeedFire, Dentsply Sirona)
VITA ENAMIC (VITA Zahnfabrik) (ENA)	Polymer-reinforced ceramic	Inorganic portion (86 wt%): SiO_2 (58–63 wt%), Al_2O_3 (20–23 wt%), Na_2O (9–11 wt%), K_2O (4–6 wt%), B_2O_3 (0.5–2 wt%), ZrO_2 (< 1 wt%), CaO (< 1 wt%). Polymers (14 wt%): Urethane dimethacrylate and triethylene glycol dimethacrylate.	Milling (CEREC inLab MC XL, Dentsply Sirona)

bonded, the three surface treatments described in Table 2 were performed (n = 18; 6 crowns per treatment).

Thereafter, a uniform coat of a light-cure adhesive primer (Transbond XT Primer, 3M Oral Care, Monrovia, CA, USA) was applied prior to bracket cementation. A total of 504 maxillary first human premolar metallic brackets without hook (Victory Series Low Profile Bracket System, 3M Oral Care) were bonded by the same operator (C.G-S.) following the same bonding protocol in all groups. As illustrated in Fig. 2, four brackets were bonded per crown (one on each buccal surface) using a non-excess precoated orthodontic adhesive system (APC Flash-Free Adhesive Coated Appliance System, 3M Oral Care) (APC FF). This adhesive does not generate excesses around brackets, so no flash removal was needed prior the polymerization step.

Brackets were positioned through an orthodontic bracket tweezer at a distance of 4 mm to the buccal cusp of each surface, following the longitudinal axis of the crown. Once positioned, a constant force of 200 g was applied and maintained using a laboratory press during the polymerization process. The same LED unit (Elipar S10, 3M Oral Care) was used to photopolymerize the flash-free orthodontic adhesive. The time of polymerization was 40 s per bracket (20 s on each proximal side) with an intensity of 1200 mW/cm^2. A LED radiometer (Bluephase Meter II, Ivoclar Vivadent) was used to verify the intensity of the curing unit for every crown.

Once all the brackets were bonded, half of them were stored in distilled water for 24 h at 37 °C (24 h), whereas the other half were submitted to 10,000 cycles TC (SD Mechatronik GMBH, Feldkirchen-Westerham, Germany) before SBS test. TC was performed applying water baths at 5 °C and 55 °C with a dwelling time of 30 s between each bath. Thus, three crowns of each CAD/CAM material (12 brackets) were included per surface treatment and per thermal procedure, to make a total of 126 crowns (504 brackets).

Subsequently, SBS test was performed with a universal testing machine (Instron 5965, Instron Corp., Canton, MA, USA). A knife-edge chisel was mounted in the movable crosshead of the testing machine, positioned perpendicular to the edge of the brackets' base. The test was performed at a crosshead speed of 1 mm/

Table 2 Description of the surface treatments used for the different experimental groups

Type of ceramic	MI	MEP	9.6% HF
CBU, EMP and ENA	Application of 5% HF (IPS Ceramic Etching, Ivoclar Vivadent) for 60 s, then rinsed for 60 s and dried for 30 s to continue with the application of a silane primer containing 10-MDP (Monobond Plus, Ivoclar Vivadent) following manufacturer's instructions (60 s reaction and then dried for 5 s with free-moisture air to disperse any remaining excesses).	Application of MEP according manufacturer's instructions. Firstly, it was gently applied with a microbrush, agitating it into de buccal surface for 20 s, allowing it to react for other 40 s (total: 60 s exposure). Then, it was rinsed and dried for 60 s and 30 s, respectively.	Application of 9.6% HF (Porcelain Etchant, Premier Dental) for 60 s, then rinsed for 60 s and dried for 30 s, followed by the application of Monobond Plus as explained in MI group.
CBG	Application of 9.6% HF (Porcelain Etchant, Premier Dental) for 90 s, then rinsed for 60 s and dried for 30 s, followed by the application of Monobond Plus as previously mentioned.		
EMA and SUP	Application of 5% HF (IPS Ceramic Etching, Ivoclar Vivadent) for 20 s, then rinsed for 60 s and dried for 30 s, followed by the application of Monobond Plus as previously mentioned.		
TZI	Sandblasting with 50 μm grain sized aluminium trioxide powder for 20 s, with a pressure of 2 bars and at a constant distance (2 cm). Then, powder excesses were removed with the use of an ultrasonic bath with distilled water for 10 min, dried for 30 s, followed by the application of Monobond Plus as previously mentioned.		

Fig. 2 One of the crowns included in the study with one metal bracket bonded to each of the four buccal surfaces

min and with an occlusogingival direction (Fig. 3). The force required to dislodge the brackets was registered in megapascals (MPa), considering the area of the brackets given by the manufacturer (9.03 mm^2).

Then, the type of failure and adhesive remnant index (ARI) were determined using a stereomicroscope (Olympus SZX7, Hamburg, Germany) at a magnification of 10× and 25× for borderline cases. The scores were categorized as [27]: 0 = no adhesive remaining on the buccal surface; 1 = less than half of the adhesive remaining on the buccal surface; 2 = more than half of the adhesive remaining on the buccal surface; 3 = all adhesive remaining on the buccal surface.

Specimens with representative failures from each experimental group were sputter-coated with gold (Bal-Tec Sputter Coater SCD 005, Witten, Germany) and observed under a scanning electron microscope (Phillips XL30 ESEM, FEI Company, Hillsboro, OR, USA).

The effect of the three surface treatments on the topography of the CAD/CAM materials tested was analyzed under scanning electron microscopy (SEM) (JSM 6400, JEOL, Tokyo, Japan) and compared with a non-treated specimen (Fig. 4). Sections of 2 mm thick were obtained from each CAD/CAM block and polished under water cooling with decreasing granulation polishing discs (Buehler, Lake Buff, IL, USA): P 320; P 500; P 800; P 1200; P 2500 and P 4000. Then, they were sputter-coated with gold (Bal-Tec Sputter Coater SCD 005) prior analyzing them.

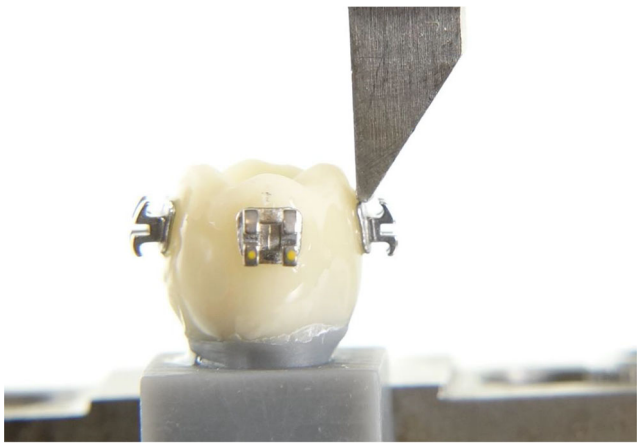

Fig. 3 Image of the SBS test performed on one APC FF metallic bracket

Results were analyzed by Kruskal-Wallis and Mann-Whitney *U* tests using the Bonferroni correction, as they did not follow a normal distribution (confirmed by Shapiro-Wilk test). All statistical tests were performed at a pre-set alpha of 0.05 using SPSS 22 for Windows software (IBM Corporation, Armonk, NY, USA).

Results

The results of descriptive statistics are summarized in Table 3. After 24 h, the surface treatment influenced the SBS of the brackets bonded to CBG ($p = 0.002$), EMP ($p = 0.014$) and EMA ($p = 0.036$) materials. For CBG, MEP treatment obtained lower SBS compared with MI and 9.6% HF groups. In connection with EMP ceramic, the results obtained were statistically lower with the use of MEP conditioner, but only compared with the MI group, as no significant differences were found between MEP and 9.6% HF surface treatments. However, when applying Bonferroni correction, no statistically significant differences were found for EMA material after 24 h.

When TC was performed, significant differences were found for the following materials: CBG ($p < 0.001$), EMP ($p = 0.013$), EMA ($p < 0.001$), TZI ($p = 0.017$) and ENA ($p = 0.005$). The same behaviour was found for CBG and EMA, in which MEP treatment decreased SBS scores in comparison with MI and 9.6% HF conditioning. However, for EMP and ENA materials, the surface treatment with MEP showed higher SBS values than 9.6% HF and similar results than MI group. TZI ceramic obtained higher SBS with MI conditioning method (sandblasting) than with MEP treatment.

When the SBS values obtained were compared between 24 h and the presence of TC for each CAD/CAM material, the following results were obtained: in MI group, a significant decrease on the SBS was observed for the bracket-ceramic interface with the following materials: CBG ($p = 0.033$), EMP ($p = 0.028$) and EMA ($p = 0.038$). With the use of MEP, CBG ($p = 0.005$), EMA ($p < 0.001$) and TZI ($p = 0.043$) were the materials significantly influenced by TC. Finally, when 9.6% HF was used, CBU ($p = 0.006$), CBG ($p = 0.009$), EMP ($p = 0.021$), SUP ($p = 0.011$) and ENA ($p = 0.001$) materials experienced a significant decrease on the SBS of their respective interfaces.

The ARI scores obtained for the seven groups of CAD/CAM materials are detailed in Table 4. Overall, after 24 h,

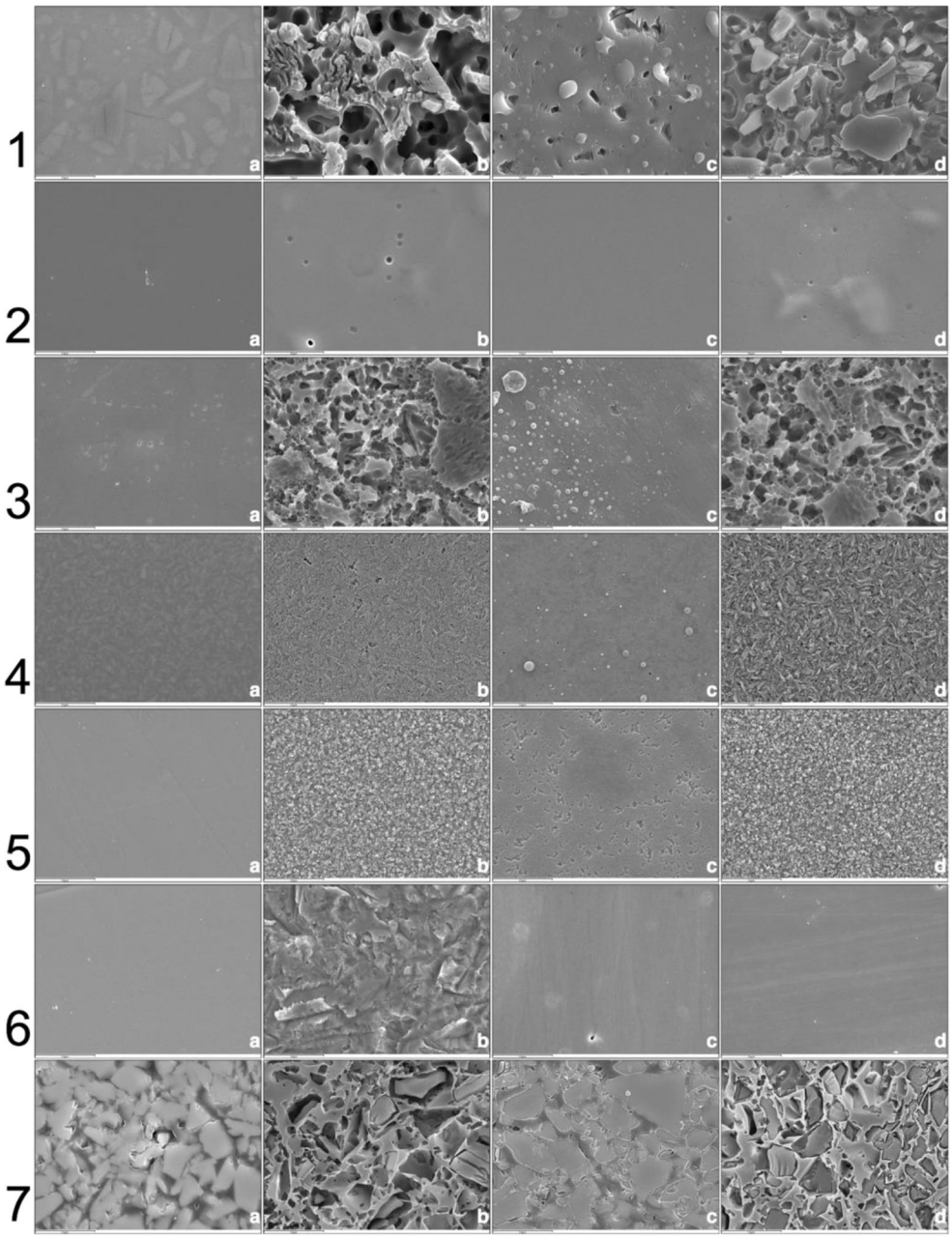

Fig. 4 SEM images of all the CAD/CAM materials (3000×) with the three surface treatments performed and without treatment as control: (a) No treatment, (b) MI, (c) MEP and (d) 9.6% HF. CAD/CAM materials: (1) CBU, (2) CBG, (3) EMP, (4) EMA, (5) SUP, (6) TZI and (7) ENA

adhesive remnants were observed in the majority of the cases, regardless of which CAD/CAM material and surface treatment were used. When TC was applied, for the MEP conditioning group, a prevalence of 83.3% and 66.7% in ARI 0 score was found for CBG and EMA, respectively. In general, with the use of MEP, the tendency was to find less adhesive remnants compared with the other two surface treatments.

Figure 4 shows the SEM micrographs of CAD/CAM materials tested after application of the surface treatments. The control group specimens showed smooth and homogeneous surfaces. When HF was applied in MI and 9.6% HF groups, clear morphological changes were observed for CBU, EMP, EMA and SUP ceramics. The dissolution of the glass phase produced a protrusion of the crystals, generating a strong etching pattern with an irregular surface with deep pores, more evident in CBU and EMP materials. These irregularities were less pronounced on the surface of EMA and SUP ceramics, showing a more homogeneous pattern. Treatment of EMA with HF revealed the spindle shape crystals characteristic of lithium disilicate, and these crystals were more evident with defined cavities between them when the concentration used of HF was 9.6% in comparison with 5%. In contrast, the application of HF in SUP ceramics exhibited a distinct surface with smaller, rounded and granular crystals being this pattern also

Table 3 Median, minimum (min), maximum (max), interquartile range (IQR) of SBS expressed in MPa, for all the CAD/CAM materials tested (n = 12)

Material	Aging	Surface treatments															
		MI					MEP						9.6% HF					
		SBS (MPa)															
		Median		Min	Max	IQR		Median		Min	Max	IQR		Median		Min	Max	IQR	
CBU	24 h	34.0	a	11.3	42.4	31.0		32.3	a	26.4	39.7	13.3		34.4	a	24.4	41.3	16.9	*
	TC	32.2	a	22.5	36.0	13.5		33.7	a	25.1	39.8	14.7		27.9	a	20.4	35.2	14.8	
CBG	24 h	34.6	a	15.6	42.4	26.9	*	19.5	b	10.7	39.4	28.8	*	36.2	a	29.3	40.0	10.7	*
	TC	27.5	a	13.9	35.9	22.1		11.4	b	0.0	31.8	31.8		31.7	a	20.2	37.4	17.2	
EMP	24 h	36.5	a	24.6	44.0	19.4	*	26.7	b	15.0	39.2	24.2		31.4	ab	23.0	40.9	17.8	*
	TC	33.4	a	20.3	38.2	17.9		34.1	a	18.7	38.9	20.2		21.4	b	12.1	36.9	24.8	
EMA	24 h	36.6	a	11.1	41.2	30.1	*	28.6	a	14.2	40.0	25.8	*	34.9	a	24.3	38.5	14.2	
	TC	30.5	a	16.4	37.0	20.6		10.1	b	5.4	16.0	10.6		33.1	a	12.9	39.4	26.5	
SUP	24 h	36.1	a	25.4	40.7	15.4		31.6	a	15.1	40.0	24.9		38.5	a	14.6	41.5	26.9	*
	TC	32.8	a	18.0	38.1	20.0		28.7	a	17.7	34.8	17.1		24.9	a	16.0	41.6	25.7	
TZI	24 h	32.1	a	18.2	39.0	20.9		29.3	a	13.4	33.9	20.5	*	29.4	a	12.5	35.6	23.1	
	TC	32.2	a	13.2	37.5	24.3		22.3	b	9.5	32.3	22.7		21.4	ab	12.6	33.7	21.0	
ENA	24 h	34.3	a	19.2	41.0	21.8		34.2	a	14.4	39.9	25.6		33.6	a	29.2	40.5	11.3	*
	TC	33.0	ab	19.4	35.6	16.2		32.2	a	28.7	38.1	9.4		24.1	b	17.2	41.2	23.9	

For each row, different letters indicate significantly different SBS values among the surface treatments performed for each material

*Significant differences between SBS median after 24 h and TC for the same material and surface treatment

Table 4 ARI scores obtained for each of the groups after debonding

Material	Aging	Conditioning method												*p*
		MI				MEP				9.6% HF				
		ARI 0	ARI 1	ARI 2	ARI 3	ARI 0	ARI 1	ARI 2	ARI 3	ARI 0	ARI 1	ARI 2	ARI 3	
CBU	24 h	0 (0%)	1 (8.3%)	3 (25%)	8 (66.7%)	0 (0%)	1 (8.3%)	1 (8.3%)	10 (83.3%)	1 (8.3%)	7 (58.3%)	1 (8.3%)	3 (25%)	0.003
	TC	0 (0%)	6 (50%)	0 (0%)	6 (50%)	0 (0%)	7 (58.3%)	3 (25%)	2 (16.7%)	0 (0%)	0 (0%)	4 (33.3%)	8 (66.7%)	0.014
CBG	24 h	1 (8.3%)	7 (58.3%)	2 (16.7%)	2 (16.7%)	3 (25%)	8 (66.7%)	0 (0%)	1 (8.3%)	0 (0%)	4 (33.3%)	1 (8.3%)	7 (58.3%)	0.006
	TC	1 (8.3%)	10 (83.3%)	1 (8.3%)	0 (0%)	10 (83.3%)	2 (16.7%)	0 (0%)	0 (0%)	1 (8.3%)	7 (58.3%)	2 (16.7%)	2 (16.7%)	0.000
EMP	24 h	0 (0%)	2 (16.7%)	8 (66.7%)	2 (16.7%)	0 (0%)	3 (25%)	2 (16.7%)	7 (58.3%)	0 (0%)	5 (41.7%)	2 (16.7%)	5 (41.7%)	NS
	TC	0 (0%)	4 (33.3%)	0 (0%)	8 (66.7%)	0 (0%)	5 (41.7%)	0 (0%)	7 (58.3%)	0 (0%)	0 (0%)	4 (33.3%)	8 (66.7%)	NS
EMA	24 h	0 (0%)	5 (41.7%)	1 (8.3%)	6 (50%)	1 (8.3%)	9 (75%)	1 (8.3%)	1 (8.3%)	0 (0%)	10 (83.3%)	2 (16.7%)	0 (0%)	0.017
	TC	0 (0%)	5 (41.7%)	6 (50%)	1 (8.3%)	8 (66.7%)	4 (33.3%)	0 (0%)	0 (0%)	0 (0%)	5 (41.7%)	4 (33.3%)	3 (25%)	0.005
SUP	24 h	0 (0%)	2 (16.7%)	4 (33.3%)	6 (50%)	0 (0%)	0 (0%)	3 (25%)	9 (75%)	0 (0%)	5 (41.7%)	1 (8.3%)	6 (50%)	0.005
	TC	0 (0%)	1 (8.3%)	3 (25%)	8 (66.7%)	0 (0%)	7 (58.3%)	3 (25%)	2 (16.7%)	0 (0%)	1 (8.3%)	3 (25%)	8 (66.7%)	NS
TZI	24 h	0 (0%)	3 (25%)	5 (41.7%)	4 (33.3%)	0 (0%)	9 (75%)	1 (8.3%)	2 (16.7%)	0 (0%)	8 (66.7%)	3 (25%)	1 (8.3%)	NS
	TC	0 (0%)	3 (25%)	2 (16.7%)	7 (58.3%)	1 (8.3%)	9 (75%)	1 (8.3%)	1 (8.3%)	4 (33.3%)	8 (66.7%)	0 (0%)	0 (0%)	0.000
ENA	24 h	0 (0%)	3 (25%)	1 (8.3%)	8 (66.7%)	0 (0%)	2 (16.7%)	1 (8.3%)	9 (75%)	0 (0%)	2 (16.7%)	1 (8.3%)	9 (75%)	NS
	TC	0 (0%)	1 (8.3%)	1 (8.3%)	10 (83.3%)	0 (0%)	0 (0%)	2 (16.7%)	10 (83.3%)	0 (0%)	0 (0%)	0 (0%)	12 (100%)	NS

The total of the percentages for a given group is 100% and differences from this total arise from the rounding process

more relevant after application in a concentration of 9.6%. Concerning TZI material, sandblasting produced an irregular and a marked topographical pattern on the surface, while a very smooth and homogeneous surface was observed after the other two treatments (MEP and 9.6% HF), similar to the control image.

The surface of ENA showed also a dissolution of the crystalline particles after conditioning with HF, although with a different pattern compared with the other materials, as the polymer network is maintained to a larger extent.

When MEP was applied, the morphology obtained for the different materials tested was similar to the control group. Only small, irregular and shallow isolated defects were observed on the surface of CBU, EMP, SUP and ENA materials.

Discussion

According to literature, bond strengths between 6 and 8 MPa are adequate for an orthodontic treatment [28]; therefore, the results obtained in the present study for all the CAD/CAM materials tested using three surface treatments seem to be valid for orthodontic bonding purposes. However, the use of MEP to condition CBG and EMP ceramics produced significant lower immediate SBS values in comparison with the application of the protocol recommended by manufacturers. Moreover, surface treatment affected SBS after TC aging procedure for CBG, EMP, EMA, TZI and ENA materials. A significant decrease on the SBS after TC was found for all the materials in at least one of the treatments. Hence, the first and second hypotheses were rejected.

No significant differences were found on the SBS for CBU ceramic regardless the surface treatment performed, either after 24 h or aging through TC. Similar to our findings, Liebermann et al. [23] and Tribst et al. [29] reported similar bond strength values for Vita Blocs Mark II (VITA Zahnfabrik) after application of 9–10% HF combined with Monobond Plus as silane and MEP. As mentioned before, we neither found differences with 5% HF treatment. However, El-Damanhoury et al. [20] and Prado et al. [22] found that statistically higher bond strength values were obtained when 5% HF + Monobond Plus was used compared with MEP. Prado et al. [22] also observed this behaviour after 12,000 cycles TC. Although the absence of differences in SBS values, the etching pattern was completely different after applying HF and MEP (Fig. 4, 1a–d). The treatment with HF clearly produced a deeper and more irregular etching pattern on the feldspathic ceramic while MEP application produced a much more superficial pattern and a less irregular surface (Fig. 4, 1c). This higher retentive surface created by HF could be in connection with the higher prevalence of adhesive remnants after TC observed on failed surfaces (ARI 3). These findings are in accordance with El-Damanhoury et al. [20], who also found that HF produced a higher mean surface roughness compared with the use of MEP. After TC, they found a high percentage of cohesive failures for both treatments (HF and MEP).

In the present study, the effect of ceramic glaze on surface treatment efficacy was tested applying a thin coat of glazing on top of the same feldspathic ceramic (CBG group). According to our results, CBG was the only material where TC significantly decreased SBS values obtained for the three treatments evaluated. The most remarkable issue was the effect of MEP on the SBS results, as it was the only material where the median values obtained either after 24 h or TC were lower than 20 MPa (19.5 MPa and 11.4 MPa, respectively). This could be explained due to the fact that the gloss may act as an insulation layer when a mild acid as MEP was used, producing a significant decrease on the SBS compared with the values obtained in the unglazed group (CBU). Moreover, as studied by Hammad et al. [30], glazed ceramic surfaces appeared to be more resistant to fluoride agents than just polished ceramic surfaces, being fluoride one of the main components of MEP. In addition, these findings are associated to the type of failure found, where ARI 1 was the most prevalent failure after 24 h and ARI 0 with an 83.3% prevalence after TC (Table 4).

Although the mechanism of action of MEP is not very clear, another explanation of these low SBS values obtained on CBG material might be related to the silanating capacity of MEP. As exposed by El-Damanhoury et al. [20], the silane system in MEP leaves a chemically bonded thin layer of silane that remains even after the thorough washing after MEP application. However, as it can be demonstrated in Fig. 4, 2c, a flat and smooth surface without any type of roughness was obtained after the application of MEP. Thus, this scenario could be due to two different reasons: the silane system contained on MEP, which is based on a trimethoxypropyl methacrylate, could have been removed after rinsing with water, as no penetration of the glaze appears to have taken place, neither achieving an optimal mechanical nor chemical union; or the etching capacity of MEP is not enough to allow the function of the silane, which could remain on the ceramic surface even after rinsing with water. Therefore, the complete or partial removal of the thin superficial glazed layer should be considered for the orthodontic bonding process; sandblasting or roughening with a bur could be considered and evaluated in future studies for this purpose.

Regarding EMP material, the three surface treatments evaluated obtained adequate SBS values, although significantly higher results were yielded with MI and MEP after TC than applying 9.6% HF, in agreement with Murillo-Gómez et al. [31]. This could be attributed to an over etch effect of the higher HF concentration (9.6%), which may cause a deep fragmentation of the microstructure of the ceramic, reducing the bonding values obtained. Murillo-Gómez et al. [32] also

described that EMP underwent greater morphological alterations than EMA and ENA after the application of HF at different concentrations and times, and MEP produced lower surface roughness and fewer morphological alterations than HF etching [31], according to our SEM observations (Fig. 4, 3a–d). Miersch et al. [26] found that with the use of an experimental ceramic primer with ammonium polyfluoride and trimethoxysilylpropyl methacrylate (same composition as MEP), acceptable SBS capable of withstanding orthodontic forces were achieved. In their study [26], they found that SBS values were significantly higher when roughening the ceramic surface before applying the ceramic conditioner.

Although no differences were found among the three treatments for EMA material after 24 h, with TC, the use of MEP showed statistically lower SBS results compared with HF either at 5% or 9.6% concentrations. In fact, after TC, the treatment of EMA material with MEP obtained the lowest median value (10.1 MPa) among all the materials tested. These findings are correlated with the SEM images obtained (Fig. 4, 4a–d), as the irregularities and morphology of the surface obtained after the use of HF were not observed after MEP application showing a less pronounced etching pattern, in agreement with other authors [9, 20, 32]. Strasser et al. [9] found that HF achieved the highest surface energy values for all the glass ceramics evaluated. However, similar to what we found, Strasser et al. [9] described the effect of MEP on EMA surface as a primer covering of the surface, effecting only slight changes on the roughness of the material, and increasing the surface energy. Moreover, it has to be highlighted that the etching pattern observed in the 9.6% HF group was more marked and with more crystals exposed than with the use of 5% HF in the MI group. These findings are correlated with the ARI indexes obtained, as for MEP, ARI 0 and ARI 1 were the most prevalent (tendency to an adhesive failure), whereas ARI 2 and ARI 3 (tendency to a cohesive failure) were the most prevalent scores for MI treatment.

Lithium disilicate has been the most studied ceramic where MEP has been tested with heterogeneous results in the literature [9, 20, 24, 29, 32–35]. Some authors [9, 20, 34] described that the combined use of HF and Monobond Plus showed higher bond results than MEP application. On the other hand, Tribst et al. [29] and Lyann et al. [33] reported no statistically significant differences between the use of HF and MEP regardless of TC aging, recommending MEP as a potential substitute for the combination of HF and Monobond Plus for the treatment of lithium disilicate ceramics. Maier et al. [35] obtained similar results between MEP and the use of HF plus a silane, considering MEP as an acceptable alternative to HF for the treatment of EMA. Nevertheless, for some of the adhesives tested, conventional pretreatment (HF + silane) obtained higher and more stable mean bond strengths.

The three surface treatments tested showed good SBS results to SUP either after 24 h water storage or 10,000 cycles TC. When analyzing the topographical surface under SEM (Fig. 4, 5a–d), the observed pattern was very similar between the two HF concentrations (5% and 9.6%), perhaps observing more marked irregularities with 9.6% HF treatment. With the use of MEP, some porosities were observed compared with the control image, but much less marked than with HF. Liebermann et al. [23] in their study did not find any significant differences between using 9% HF for 30 s together with Monobond Plus and MEP treatment, obtaining high mean tensile bond strength values (33.8 and 35.2, respectively) with both treatments for Celtra Duo, which is almost identical to SUP, after 20,000 cycles TC. Strasser et al. [9] included CAD/CAM blocks of SUP and Celtra Duo and examined the effects of different surface pre-treatment on them, observing that HF etching led to strong and homogeneous etching patterns. However, with the use of MEP, the primer covered the surface and effected only slight changes on the surface of both glass ceramics. It has to be highlighted that similar to what happened with EMP, the SBS obtained in 9.6% HF treatment after TC decreased drastically and after analyzing the etching pattern of this treatment (Fig. 4, 5d) could be due to an excessive etching after applying 9.6% HF with a partial destruction of the crystals of the ceramic.

According to our results, no significant differences were found between MI (sandblasting + Monobond Plus) and 9.6% HF treatments after TC (once applied Bonferroni correction), being the results obtained with MEP lower. We also observed that the SEM images obtained using MEP (Fig. 4, 6c) and HF (Fig. 4, 6d) are very similar to the control group (flat and smooth); however, the SBS values obtained, indicate that MEP treatment could be valid for an orthodontic treatment. Although MEP was not initially launched to condition zirconia, there are also studies evaluating its bonding properties for this material [25, 36]. Franz et al. [25] research is the only published study that used MEP to test TZI CAD/CAM zirconia for bracket bonding purposes. Similar to our results, they found that after 10,000 cycles TC, MEP is an effective treatment obtaining a mean value of 13.66 MPa when brackets were non-activated and 14.53 MPa when activated with a 0.14 nickel titanium wire. However, the SBS results after TC for TZI material were higher in our study (Table 3). Wille et al. [36] found a similar topographical pattern after sandblasting the zirconia to our study (Fig. 4, 6b), that did not change when they applied MEP afterwards. The high SBS values obtained in the present study and by Franz et al. [25] using MEP, might be related with a distinct increase in surface energy produced by ammonium polyfluoride, even with a minimal increase in roughness not identifiable on SEM images [9].

ENA, as a PICN material, represents an excellent substrate to be treated with any of the three treatments evaluated. However, once again, when applying 9.6% HF, the differences of the SBS obtained after 24 h and TC suggest that an excessive etching pattern was produced (Fig. 4, 7d). On the

other hand, either MI or MEP treatments performed excellent in this PICN material, as no significant differences were found regardless of aging. After TC, even the SBS obtained with MEP was higher than with the use of 9.6% HF. Similar to us, El-Damanhoury et al. [20] and Murillo-Gómez et al. [31] neither found significant differences between the use of HF at a 5% concentration plus silane and MEP. However, when the topographical analysis was performed under SEM (Fig. 4, 7a–d), a less marked dissolving pattern was observed with the use of MEP compared with HF, where the irregularities and the porosities observed were relevant in agreement with Murillo-Gómez et al. [32].

Finally, apart from the importance of the conditioning treatment used, the orthodontic adhesive is also one of the keys to success in an orthodontic treatment. The non-excess and pre-coated APC FF adhesive used in this study has been broadly studied to enamel either through in vitro studies [37, 38] or clinical trials [39, 40] showing excellent results and the advantage that it does not generate flash around the bracket once it is placed, reducing thus the time needed for the cementation process and the risk of appearance of white spot lesions or staining around the bracket. Nevertheless, there are no studies evaluating the behaviour of APC FF on ceramic materials, which according to our results, it is also a valid adhesive to bond brackets to different CAD/CAM materials showing reliable bonding properties to all the materials tested.

In the present study, we have tried to apply a methodology that replicates a clinical situation. Therefore, first human premolar APC FF metallic brackets were bonded to ceramic crowns fabricated to simulate buccal surfaces of the corresponding teeth instead of using flat surfaces. This generates a uniform cement thickness that allows a better stress distribution along the interface. However, in shear bond strength tests, stresses concentrate close to the loading area, especially when a knife-edge chisel is used. Therefore, results should be better expressed as shear debonding force than shear bond strength, but this term is widely assumed in literature. Moreover, tensile stresses are also generated during the test and are even responsible for failure initiation [41]. This stress distribution is also affected by the distance between the point of load application and the ceramic-bracket interface, being a reason for differences in bond strength results between the same experimental group [41]. Finally, masticatory forces are not accurately represented through laboratory methods, nor the action of biofilm on bonding properties of the interface, among other intrinsic characteristics of the oral environment. Thus, we encourage other researchers to perform clinical studies to analyse the influence of this variables on the bonding properties of APC FF brackets to different CAD/CAM materials and with different bonding protocols.

Conclusions

According to our findings, the following conclusions were drawn from this investigation:

- The three treatments evaluated (MI, MEP and 9.6% HF) obtained enough and acceptable results in terms of SBS of APC FF brackets to complete an orthodontic treatment for all the CAD/CAM materials tested, being the MI treatment the one that achieved the highest SBS results for most of the materials evaluated. MEP showed to be a valid orthodontic conditioner for most of the CAD/CAM materials evaluated, although the effectiveness of MEP decreased drastically for CBG and EMA compared with the use of HF. Thus, its indication to condition these two materials should be considered cautiously.

- Although TC significantly decreased the SBS of APC FF brackets for the three treatments in most of the CAD/CAM materials tested, the SBS results obtained are still enough to stand the required forces in an orthodontic treatment.

Acknowledgments Authors are indebted to Dr. Neimar Sartori and Claudia Pohlen (CIRO) for collaborating with the digital part of the study and to the SEM technician Ana Vicente for helping us with the SEM analysis. This study is part of a thesis to fulfill the requirements for the PhD degree of first author C.G-S.

Authors' contributions Carlos González-Serrano: conceptualization, writing (original draft), writing (review and editing), methodology, software, validation, investigation, resources, data curation, visualization, supervision, project administration and funding acquisition.

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Funding information The work was supported by a predoctoral grant given by the Madrid Regional Dental Council (Madrid, Spain) to first author C.G-S to afford some of the materials used in this investigation. The work was also supported by Rey Juan Carlos University (Móstoles, Madrid, Spain) by a second grant to pay part of the travel and accommodation expenses to perform the study in University of Southern California (Herman Ostrow School of Dentistry, Los Angeles, CA, USA), where the study was conducted. This fact will made C.G-S to opt for the International Mention in his PhD degree.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent For this type of study, formal consent is not required.

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