Effect of conventional and digital methods and aging on the shear bond strength of orthodontic brackets with temporary crowns based on aged PMMA

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Abstract: Shear bond strength (SBS) of orthodontic brackets bonded to temporary crowns (TC) was investigated. TC were manufactured using conventional and digital methods (CAD/CAM milling, 3D printing) and then subjected to cyclic aging (5 000 cycles). Surface roughness (Ra) and contact angle of polished and abrasive blasted TC were determined before and after aging. Data analysis was performed using one-way ANOVA and repeated measures, and LSD in post-hoc tests (α =0.05). After surface treatment, there was a significant increase in Ra of conventional and milled TC (p<0.001), while 3D printed showed a slight increase in Ra (p=0.073). The contact angle of polished TC surfaces was significantly different (p>0.05), and after surface treatment the differences in contact angle were small (p<0.001). Aesthetic orthodontic brackets bonded to temporary crowns, regardless of the production method, have adequate bond strength before and after aging, with the exception of 3D printed TCs after aging. **Keywords:** Shear bond strength, temporary crowns, sandblasting, 3D printing, CAD/CAM.

Wpływ konwencjonalnych i cyfrowych metod otrzymywania i starzenia na wytrzymałość na ścinanie zamków ortodontycznych z koronami tymczasowymi na bazie starzonego PMMA

Streszczenie: Zbadano wytrzymałość na ścinanie (SBS) zamków ortodontycznych przyklejonych do koron tymczasowych (TC). TC wykonano metodami konwencjonalnymi i cyfrowymi (frezowanie CAD/ CAM, druk 3D), a następnie poddano cyklicznemu starzeniu (5000 cykli). Oznaczono chropowatość powierzchni (Ra) i kąt zwilżania polerowanych i poddanych obróbce strumieniowo-ściernej TC, przed i po starzeniu. Do analizy danych zastosowano metodę ANOVA jednokierunkową i powtarzanych pomiarów oraz LSD w testach post-hoc (α =0,05). Po obróbce powierzchniowej nastąpił istotny wzrost Ra konwencjonalnych i frezowanych TC (p<0,001), a drukowane w 3D wykazały niewielki wzrost Ra (p=0,073). Kąt zwilżania polerowanych powierzchni TC różnił się istotnie (p>0,05), a po obróbce powierzchni różnice kąta zwilżania były niewielkie (p<0,001). Estetyczne zamki ortodontyczne przyklejone do koron tymczasowych niezależnie od metody otrzymywania mają odpowiednią wytrzymałość wiązania przed i po starzeniu, z wyjątkiem TC drukowanych w 3D po starzeniu.

Słowa kluczowe: wytrzymałość połączenia na ścinanie, tymczasowa korona, piaskowanie, druk 3D, CAD/CAM.

A person's social, functional, and psychological wellbeing, as well as their facial aesthetics, are impacted by malocclusion, which is the second most prevalent oral health problem [1]. In recent years, orthodontists have faced specific challenges due to an upsurge in adults seeking orthodontic care [2-5]. Adult patients make up 20% of the average patient population for an orthodontist, and their primary reason for seeking treatment is to enhance esthetic appearance and function [6]. Orthodontic care is a part of a multidisciplinary treatment strategy. Many adult orthodontic patients have restored and replaced teeth, which makes bonding orthodontic brackets challenging on these surfaces [6, 7]. In certain scenarios, a temporary crown (TC) is essential to preserve the ther-

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apeutic, aesthetic, and functional role of the teeth in the oral cavity [8, 9]. Definitive restorations are not advised before orthodontic treatment due to occlusal relationship changes and the potential of compromising the definitive restoration surface [2, 3, 8]. Therefore, a TC may have to remain during the entire duration of orthodontic treatment. In several orthodontic procedures, orthodontic brackets must be bonded on TCs. However, like natural crowns, the brackets must have a strong bond strength to withstand functional and orthodontic forces [8, 9].

The recent and swift expansion of digital technology has increased the efficiency of clinical dentistry [10]. The accuracy, effectiveness, and general quality of dental treatments and processes are improved using digital technologies in dental practices [11]. Temporary restorations are directly or indirectly fabricated using conventional or digital methods either in the dental office or laboratory [12]. Compared to conventional technique, the digital approach lessens several challenges, including poor structural qualities, high surface porosity, color instability, high water absorption, polymerization shrinkage, marginal integrity, and low strength, while creating direct temporary crowns [9, 13].

Digital fabrication techniques include computer-aided design/manufacturing (CAD/CAM) milling and threedimensional (3D) printing. In CAD/CAM milling or subtractive manufacturing, a resin block is milled to fabricate the digitally planned design. These temporary restorations are stronger and more accurate than those fabricated using conventional approaches, which is owed to the high monomer-to-polymer conversion rate during the resin block polymerization. However, the drawbacks of this approach include material loss, a limited range of motion, and a milling bur diameter that prevents exact reconstruction in certain regions. In 3D printing or additive manufacturing, the prosthesis is fabricated by layerby-layer deposition of photopolymerized resins [15]. The 3D printing system facilitates fabricating complicated structures with less material, thereby addressing the flaws of CAD/CAM milling [11, 13, 15, 16].

The bonding procedure involves physical and chemical forces, but the underlying concept is based on mechanical interlocking between the treated bonding surface and the low-density polymer bonding adhesive [17]. High failure rates and ineffective orthodontic treatment, as measured by cost and efficacy, are imposed by brackets with inadequate bonding resistance [18]. Temporary restorative materials should have adequate physical and mechanical qualities to minimize failures under prolonged functional loading in clinical circumstances when temporization is necessary for longer periods [19]. Clinicians may fabricate TCs using various materials with different chemical, physical, and clinical characteristics. However, the most widely utilized material is poly(methyl methacrylate) (PMMA) [2, 3, 6].

The bond strength between the bracket and the temporary restorations is influenced by a number of parameters, including surface preparation, adhesive type, time after bonding, material type, fabrication method, and aging [20, 21]. In clinical environment, it is very unlikely that brackets are bonded to a newly fabricated crown. The dental literature and clinical experience have demonstrated that it is possible to achieve a satisfactory bond by utilizing a variety of materials and procedures in newly fabricated restorations. However, bond strength to aged substrates are scarce and needs to be continually investigated [22]. The assessment of the shear bond strength (SBS) to the aged restorative surface will provide a comprehensive knowledge of the bond strength as existing restorations in oral cavity degrade chemically and mechanically [23]. It has been demonstrated that aesthetic ceramic brackets have higher SBS compared to metallic brackets due to their greater ability to adhere. The light transmittance of ceramic brackets, which promotes better photopolymerization and reduced strain at the adhesive-bracket interface, may be a contributing factor to their higher bond strength [24].

Previous studies have investigated the SBS between the bracket and temporary crowns fabricated by different techniques. However, there is a lack of information regarding the SBS between aesthetic bracket and aged temporary crown materials fabricated by different techniques and the roughness and wettability of the bonding surface of these materials.

Consequently, this study aimed to evaluate the SBS of aesthetic orthodontic brackets bonded to aged temporary crowns fabricated by conventional, CAD/CAM milling and 3D-printing with particular emphasis on the roughness and wettability of the bonding surface.

EXPERIMENTAL PART

Materials

A total of seventy-two temporary crowns (TCs) were fabricated by conventional 3D printing and CAD/CAM milling to have 24 crowns from each material. The sample size was determined per DIN 13990-1 standard specification, which recommends a minimum sample of 10 specimens per group [25]. However, we used twelve crowns to compensate for any experimental loss. The crowns were fabricated using the materials detailed in Table 1.

Fabrication of TC by conventional and digital methods

All the crowns were prepared as the upper first premolar and were the same size, thickness, and design. In the conventional method, a putty impression (Coltene/ Whaledent AG, Altstätten, Switzerland) of the prepared upper right first premolar typodont was taken, and the cast was poured using dental stone (Type III, Whipmix, Louisville, KY, USA). A total of 24 TCs were made on the plaster model obtained using self-cured PMMA resin

T a b l e 1. Materials characteristics

Materials	Composition	Manufacturer				
Temporary crown materials						
NT Newton Aycliffe	<i>Powder:</i> cadmium free PMMA and catalyst (<1%) <i>Liquid:</i> MMA, TEGDMA, DMA and catalyst (<1%)	Toros Dental, Antalya, Turkey				
Telio CAD	PMMA (99.5%) and pigments (<1.0%)	Ivoclar Vivadent, Schaan, Liechtenstein				
NextDent C&B MFH	7,7,9(or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane- 1,16-diyl bismethacrylate (50-75 wt%); 2-hydroxyethyl methacrylate (< 25 wt%); silicon dioxide (1-5 wt%); diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide (1-5 wt%); ethoxylated bisphenol A dimethacrylate (<10 wt%); ethylene dimethacrylate (<10 wt%); titanium dioxide (<0.1 wt%); 4-methoxyphenol (<0.1 wt%)	NextDent B.V., AV Soesterberg, The Netherlands				
Adhesives						
Transbond XT Primer	bis-GMA, TEGDMA, 4-(dimethylamino)-benzeneethanol, camphorqui- none, hydroquinone	3M Unitek, Monrovia, CA, USA				
Transbond XT Paste	nsbond XT Paste silane treated quartz (70–80 wt%); BADGE-DMA; bisphenol A bis (2-hydroxyethyl ether)-DMA. silane treated silica; diphenyliodonium					

PMMA - polymethyl methacrylate; MMA – methylmethacrylate; TEGDMA – triethylene glycol dimethacrylate; DMA – dimethacrylate; BADGE – bisphenol A diglycidyl ether;

(NT Newton Aycliffe, Toros Dental, Antalya, Turkey). The TC remained on the model until the final setting was completed to reduce polymerization shrinkage.

The TC fabricated by the conventional method was scanned (KaVo Everest Scan pro, Biberach an der Riss, Germany) to obtain a virtual image that was saved as an STL file. The resulting STL file was imported into the PreForm print preparation software (Formlabs, Inc, Somerville, MA, USA). A total of 26 TCs were printed with a stereolithography (SLA) 3D printer (Formlabs, Inc, Somerville, MA, USA) using photopolymer resin (NextDent C&B MFH, NextDent B.V., AV Soesterberg, The Netherlands). The TCs were printed horizontally to the build platform, with the occlusal plane facing the build platform. The printed TCs were cleaned in an ultrasonic bath containing 90% isopropyl alcohol for 3 minutes to remove any excess resin and rigorously dried with compressed air. Next, the crowns were air-dried for at least 10 minutes in a well-ventilated area to confirm that the printed surfaces were free of isopropyl alcohol. Then, the crowns were placed in an oven (Zirlux, Zahn Dental Labs, Henry Schein, USA) at 60°C for 40 minutes with the occlusal plane facing upwards to allow for final polymerization followed by careful removal of the print supports.

The STL dataset used to print 3D printed TCs were imported to the milling control program (KaVo Everest engine control, Biberach an der Riss, Germany) for milling of TCs. All twenty-four crowns were milled from a single PMMA blank (Telio CAD) in a 5-axis milling machine (KaVo Everest Engine, Biberach an der Riss, Germany) attached with a milling bur (KaVo Everest, Biberach an der Riss, Germany). The fabrication process was continuously monitored with the aid of the milling control program.

All the crowns were individually embedded in clear self-curing acrylic resin (Orthoplast, Vertex-Dental, AV Soesterberg, The Netherlands) to have the occlusal surface parallel to the acrylic resin base (Fig. 1). All the crowns were finished and polished using rotary polishing kit (Sof-Lex[™] Disc, 3M ESPE, Germany) and were examined under a stereomicroscope to detect any circumferential defects or cracks.

Methods

Aging simulation by thermocycling

A thermocycler device (Huber 1100, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany) was used for the aging simulation. Aging was performed for 5000 cycles at temperatures between 5°C and 55°C, a dwell time of 30 s, and a transfer time of 10 s. The 5000 cycles represented six months of intraoral usage [26].

Surface treatment

The aged TCs' bonding surface was conditioned by grit blasting using a hand-held device (LEMAT, Wassermann, Hamburg, Germany). Grit blasting was performed from 10 mm between the grit blaster nozzle and bonding surface using 50 μ m alumina oxide particles (Korox[®] 50, BEGO Canada Inc., Québec, Canada) at 4 psi for 10s (Fig. 1a). The sandblasted TCs were cleaned for 10 minutes in an ultrasonic bath containing distilled water to remove any remnants and then air-dried using oil-free compressed air.

Surface roughness

A non-contact optical profilometer (Contour GT, Bruker, CA, USA) was used to measure the roughness of the surface-conditioned TCs. The crown was placed and stabilized on the automated X-Y stage of the profilometer. The surface was scanned using white light interferometry without contacting the crown surface. Scanning was performed using a 5× nano-lens, 1× speed, 1×1 mm² viewing field, and 0.1 mm/s X-Y stage speed. The X-Y stage movements were controlled by a Vision 64 program (ver. 5.30, Bruker, Billerica, MA, USA), which was also utilized to precisely measure the selected surface area. Three evenly spaced regions of the surface were scanned, and the meaning of these values represented the roughness of that crown. Roughness was expressed as the arithmetic average of heights measured through the surface [27, 28].

SEM

One representative crown from each material was imaged for surface topographic changes before and after grit blasting using SEM (JEOL JSM-6610LV, Tokyo, Japan). The TCs were gold sputtered coated in a vacuum chamber (Q150R, Quorum tech, East Sussex, UK) before imaging. The SEM was operated at 15 kV, in vacuum, at \times 500 magnification (50 μ m scale).

Wettability

The contact angle measurement to determine surface wettability of the TC was evaluated by a camera based optical tensiometer (Theta Lite, Dyne Technology, and Staffordshire, UK) using sessile drop technique. A 2 μ L drop of distilled water was dispensed on to the bonding surface of the crown and the dynamic contact angle was measured after 20–30 s, when the droplet was stabilized. The contact angle image was obtained using a built-in digital camera attached to the tensiometer and displayed onto the computer monitor via an inbuilt software [29].

Orthodontic bonding

The bonding surface of TC were etched with 37% H₃PO₄ for 30 s, rinsed with copious amount of water for 15 s and air-dried with oil-free compressed air for 20 s. Premolar aesthetic orthodontic brackets (FLI[™] signature CLEAR brackets, Rocky Mountain Orthodontics, Denver, CO, USA) with a mesh area of 10.8 mm² were used for orthodontic bonding. The orthodontic adhesive resin (Transbond[™] XT, 3M Unitek, Monrovia, CA, USA) was applied to the bracket base, and the bracket was positioned using bracket positioning gauge and pressed firmly on to the crown surface to minimize adhesive thickness. The excess adhesive resin around the bracket



Fig. 1. Graphic representation: a) sandblasting, b) shear bond testing set up

T a ble 2. Adhesive remnant index (ARI) scores and description

ARI scores	Interpretation
Score 0	No adhesive remaining on crown surface
Score 1	< 50% adhesive remains on the crown surface
Score 2	> 50% of adhesive remains on the crown surface
Score 3	Adhesive remains on the crown surface with a clear imprint of the bracket mesh.

was carefully removed with a sharp scaler before polymerization. The proximal surfaces of the bracket edges were light-cured for 20 s using a handheld light curing unit (Elipar Free Light 2, 3M ESPE, Seefeld, Germany) with a wavelength between 420–540 nm and an output power of 1505 mW/cm². The light output was monitored using Managing Accurate Resin Curing system (MARC, Blue Light Analytics, Halifax, Canada). After bonding, all the specimens were stored in distilled water at 37°C for 24 h. Twelve TCs from each group were evaluated for adhesion strength (baseline or dry values), and the remaining 12 specimens were subjected to further aging by thermocycling.

Shear bond strength (SBS)

For SBS testing, each crown was oriented with the buccal bonded surface parallel to the shearing rod in a custom-made jig of a universal testing machine (Instron Corporation, Canton, MA, USA). The chisel-shaped rod attached to the testing machine and with a load cell of 2 kN was directed towards the crown-bracket interface near the bracket base at a constant crosshead speed of 0.5 mm/min until fracture (Fig. 1b). The force required to debone the orthodontic bracket from the crown surface was recorded, and SBS was calculated and presented in MPa by desktop attached to the testing machine.



Treatment interval	Conventional	Milled	3D-printed	p-value
Polished, µm	0.95±0.01 ^{A, a}	1.01±0.08 ^{A, a}	0.92±0.01 ^{A, a}	0.313
Surface treatment, μm	2.96±0.39 ^{A, b}	2.66±0.75 ^{A, b}	$1.31 \pm 0.64^{B_{r,a}}$	< 0.001*
p-value	< 0.001*	< 0.001*	0.073	

T a b l e 3. Surface roughness of the materials

Failure mode analysis

After debonding, a light stereomicroscope (Nikon SM2-10, Tokyo, Japan) operating at a magnification of ×20 was used to visually analyze the TC surface and the bases of the brackets to determine the site of predominant bond failure. The failure modes were categorized per the adhesive remnant index (ARI) as previously proposed by Årtun and Bergland (Table 2)[30]. Figure 2 presents the flow chart illustrating the study procedure and specimen distribution.

Statistical analysis

All data analysis was performed using IBM Statistical Package for the Social Sciences (v.22, IBM[®] SPSS[®] Inc., Chicago, IL, USA). Data followed normal distribution (Shapiro-Wilk Test; α =0.05) and hence parametric tests were applied. One-way ANOVA followed by post-hoc Tukey HSD test was used to analyze the difference in roughness, contact angle measurement and shear bond strength between the three materials. Repeated measure ANOVA was used to analyze the difference in roughness



Fig. 3. Profilometer images of TCs before and after (1) surface treatment: a) conventionally polished, b) milled, c) 3D-printed



Fig. 4. SEM images of TCs before and after (1) surface treatment: a) conventionally polished, b) milled, c) 3D-printed; magnification ×500

and contact angle measurement between polished and surface treated TCs. The significance level was set at p<0.05.

RESULTS

Surface roughness

The surface roughness (Ra) of the polished and surface treated TCs of the three materials are presented in Table 3. The roughness of the polished TC surfaces did not vary significantly between the three materials (p=0.313). After surface treatment, there was a significant increase in Ra of conventional (2.96 \pm 0.39; p< 0.001) and milled (2.66 \pm 0.75; p< 0.001) TCs but 3D-printed materials demonstrated non-significant increase in Ra (1.31 \pm 0.64; p=0.073). The profilometer 3D images of the representative polished and surface treated TCs of three materials are presented in Figure 3. Different upper cases in





a row indicate significant difference between the materials groups at different measurement intervals (posthoc Tukey HSD test, p <0.001), while different lower case in a column indicates significant difference within the materials at different measurement intervals (Repeated measure ANOVA, p<0.001). The * indicates statistically significant p-values (p<0.001)

Wettability

The difference in contact angle measurements of the polished TC surfaces varied significantly between the three materials (p< 0.05). After surface treatment, the repeated measure ANOVA showed significantly decrease in contact angle for milled ($63.43\pm9.20^\circ$; p=0.03) and 3D-printed (71.61±4.51; p=0.029) materials but did not vary significantly for the conventional material ($67.42\pm3.88^\circ$; p=0.058). Furthermore, the comparison of the surface treated materials showed no significant differences in contact angle (p>0.05). The decreased contact angle from polished to surface treated indicated increased wettability of the surfaces. The outcome of contact angle measurement analysis of the polished and surface treated TC fabricated from three materials are presented in Figure 5 and Table 4. Different upper cases in a row indicate significant difference between the materials groups at different measurement intervals (post-hoc Tukey HSD test, p <0.001), while different lower case in a column indicates significant difference within the materials at different measurement intervals (Repeated measure ANOVA, p<0.001). The * indicates statistically significant p-values (p<0.001)

Shear bond strength

The shear bond strength (SBS) initially and after thermal cycling is shown in Table 5, where \pm denotes a value below the optimal shear bond strength used in orthodontics (5.8–7.8 MPa). The highest SBS at the baseline, 8.20±1.38 MPa, was demonstrated by conventional TCs followed by milled (7.16±1.22 MPa) and 3D-printed TCs (6.92±1.02 MPa). The difference in SBS between the three TCs materials was not statistically significant (*p*=0.187). Following aging, the highest SBS was demonstrated by conventional TCs (6.39±1.51 MPa) followed by milled (6.30±1.35 MPa) and 3D-printed TCs (5.59±0.78 MPa). Like baseline specimens, the difference in SBS between the three TCs materials was not statistically significant (*p*=0.496). Furthermore, there was no significant difference in SBS between the baseline and aged crowns of the tested materials (*p*>0.05).

Fracture mode analysis

Following the debonding process, the TC surface and the bracket bases were visually inspected using a light stereomicroscope (×20) to identify the bond site failure. The number and frequencies of predominant bond failures of the baseline and aged TCs are presented in Table 6. The ARI score was distributed between 0 and 1 for the TCs at baseline, suggesting more adhesive failures. After aging, the ARI scores were between 0 and 2, suggesting adhesive and mixed (adhesive-cohesive) failures. However, there were no instances involving fractures of the TCs with any material groups.

Fig. 6 presents the representative optical microscopy images of the deboned TCs used to assign the ARI scores.

DISCUSSION

The current study evaluated the SBS of orthodontic aesthetic brackets bonded to aged temporary crowns

T a bl e 4. Contact angle of the tested materials at different measurement intervals

Treatment interval	Conventional	Milled	Milled 3D-printed	
Polished, °	77.14±5.45 ^{A, a}	95.69±7.63 ^{B, a}	80.56±4.93 ^{A, a}	< 0.05*
Surface treatment, °	67.42±3.88 ^A , a	63.43±9.20 ^{A, b}	71.61±4.51 ^{A, b}	> 0.05
p-value	0.058	0.03*	0.029*	

T a b l e 5. SBS of the tested materials

Materials	Baseline	Aged	p-value
Conventional, MPa	8.20±1.38	6.39±1.51	0.145
Milled, MPa	7.16±1.22	6.30±1.35	0.831
3D-printed, MPa	6.92±1.02	$5.59 \pm 0.78^{+}$	0.446
p-value	0.187	0.496	

T a b l e 6. Adhesive remnant index (ARI) scores with values

Materials	Baseline			Aged				
	ARI scores (n=12), %							
	0	1	2	3	0	1	2	3
Conventional	7(58.34)	2(16.66)	3(25)	0(0)	4(33.33)	3(25)	4(33.33)	1(8.34)
Milled	5(41.66)	6(50)	1(8.34)	0(0)	6(50)	3(25)	2(16.66)	1(8.34)
3D-printed	2(16.68)	8(66.64)	2(16.68)	0(0)	5(41.66)	4(33.33)	3(25)	0(0)





Fig. 6. The representative optical microscopy images of the deboned TC surface used to assign the ARI scores: a) 0, b) 1, c) 2, d) 3

fabricated by conventional polishing, CAD/CAM milling and 3D-printing with particular emphasis on roughness and wettability of the bonding surface.

In interdisciplinary orthodontic treatments, orthodontists frequently require bonding brackets to TC-restored teeth; this is essential to preserve the teeth's therapeutic, esthetic, and functional roles within the dental arch [31]. It is highly improbable that orthodontic brackets are bonded to newly fabricated TC. Instead, they are typically bonded to pre-existing TC that has already undergone mechanical and chemical deterioration. The orthodontist must assess the aged TC surface to choose the suitable TC material to obtain optimal SBS [23]. Nevertheless, the ideal orthodontic bond strength between the TC and the brackets should be robust enough to withstand the stresses of orthodontics and chewing throughout the entire orthodontic therapy while preventing the fracture of the crown surface during debonding after orthodontic treatment [23].

The literature contains few scientifically-based advocacies for the optimum SBS of orthodontic brackets, and as per these literature, the optimal SBS recommended for orthodontic purposes is 5.8 to 7.8 MPa [32, 33]. Accordingly, the optimal SBS values were used to compare the SBS values obtained in this study and to determine which TC material fabrication produced the optimal SBS while causing the least surface damage after debonding. The SBS values ranged from 5.5 to 8.2 MPa, and there were no statistically significant differences in the SBS data between the study materials at baseline and after aging. Furthermore, the SBS obtained in this study was within the optimal limit except for aged 3D-printed TCs (5.5 MPa). This outcome led to the rejection of the null hypothesis that stated significant differences in the SBS of orthodontic brackets bonded to the TCs fabricated by conventional and digital methods.

Previous studies evaluating the SBS of orthodontic brackets to milled and 3D-printed TCs have demonstrated values ranging from 1.53 to 43.77 MPa [2, 3, 6-8, 20, 34]. Goracci *et al.* [7], demonstrated non-significantly different SBS values for two different types of CAD/CAM milled TCs (CAD Temp-5.95 MPa and Telio CAD-5.23 MPa), while Garces *et al.* [34], reported SBS values of aged (2.7 MPa) and non-aged (3.2 MPa) milled TCs specimens and Haber *et al.* [8], reported SBS values of 4.21 MPa. All these reported values were below the optimal SBS. On the contrary, Baser *et al.* [20], demonstrated SBS values of 15.85 MPa and 15.61 MPa for Vita CAD and Temp CAD, respectively.

Regarding the comparison of SBS between the three TC fabrication materials, Alijani et al. [2], reported SBS values of 1.53 MPa, 4.69 MPa and 4.18 MPa for milled, 3D-printed and conventional materials, respectively. Fotovat et al. [3], reported SBS values of 15.96 MPa, 43.77 MPa and 43.02 MPa for milled, 3D-printed and conventional materials, respectively. On the contrary, Biadsee et al. [6], compared SBS between milled and 3D printed materials and reported values of 9.11 MPa and 10.95 MPa, respectively. The significant variations in SBS test values between the studies can be attributed to the various approaches employed, including the location of the knife-edge rod in the bracket, specimen storage time after bonding, surface treatment of specimens, the base area of the bracket, type of bracket, aging of the specimens, the duration of adhesive polymerization, and the crosshead speed [6, 8, 35]. Although, it is imperative to consider the underlying limitations of the SBS tests, specifically concerning the non-uniform stress distribution and consequent failure mode, the SBS test appears useful in simulating the kind of service load encountered in orthodontics, particularly when assessing the bonding of orthodontic brackets to various dental substrates [23].

In the current study, thermocycling was employed to determine if temperature variations impacted the bond between the TC and the bracket. It is speculated that hydrolysis-induced deterioration of the adhesive interface results in a reduction in bonding efficacy [36, 37]. The resin bond's mechanical properties can be reduced by the continuous action of water or by the abrupt decrease in temperature on bonded materials with different expansion coefficients and thermal conductivities, which causes thermal stress at the interface [37]. A minimum of 500 cycles between 5°C and 55°C in water has been recommended by the International Organization for Standardization (ISO TR-11405) to assess the bond strength after aging [26]. However, many researchers recommend a higher number of cycles to simulate the oral environment accurately [38]. Accordingly, the specimens in the current study were exposed to 5000 cycles, representing 6 months of aging per Gale and Darvell's recommendations [39]. The data outcome showed no significant difference in the SBS after aging, which agrees with other studies [6, 36, 40]. On the contrary, few studies report a significant effect of thermocycling on the SBS [37, 41–44].

The adhesive remnant (ARI) index is one of the most popular indices in orthodontic studies to determine the amount of adhesive present on the enamel surface following bracket debonding. The index evaluates the predominant bond failure site after assigning a score from 0 to 3 for each tooth. The TC clean-up is safer, and the risk of surface damage is reduced when there is less adhesive left on the crown following the debonding process [45]. The ARI score was distributed between 0 and 1 for the TCs at baseline, suggesting more adhesive failures. After aging, the ARI scores were between 0 and 2, suggesting adhesive and mixed (adhesive-cohesive) failures. However, there were no instances involving fractures of the TCs with any material groups.

The clinician relies on numerous surface treatment methods to enhance the bond between two materials [6, 20, 41, 46-48]. Nevertheless, the current study applied grit blasting using alumina particles as this surface treatment method has yielded optimal SBS [4, 5, 8, 41, 46, 49]. Furthermore, the bonding surface was etched with 37% orthophosphoric acid, although, in actuality it does not affect the surface of TC, unlike normal teeth, but is limited to removing the contaminants left behind by the grit blasting process [8]. The surface treatment of an aged TC's enhances the surface area with the related surface flaws and eliminates the saliva-damaged superficial layer to expose a surface that has high energy and is fresh [15]. The two steps involved in strengthening the bond between two materials is increasing the surface roughness by surface treatment to improve mechanical retention and then applying a bonding agent to improve chemical bonding [6].

Surface roughness is an important parameter for micromechanical retention of orthodontic adhesives [5]. The surface roughness data showed that the Ra of the crowns did not vary significantly before treatment (p=0.313). However, after surface treatment by grit blasting, conventional and milled crowns demonstrated a sig-

nificant increase in roughness, but no significant increase was found with 3D-printed crowns (p>0.001). The SEM images showed significant differences in the surface topography of the treated crowns, dominated by irregular specks and grooves pertinent to the micromechanical retention of orthodontic adhesives. Nevertheless, the 3D printed crown surface was less rough than conventional and milled surfaces, which explains the non-significant increase in roughness of 3D printed crowns.

The main prerequisite for achieving effective adhesion in adhesive dentistry is proper surface wetting of the adhesive on the dental substrate. A surface contact angle (°) is often used to convey wettability, a physical characteristic that describes a liquid droplet's ability to sustain or spread on a solid surface [50]. A low contact angle indicates good surface wetting, which promotes close contact between the adherend and adhesive and improves mechanical interlocking. Therefore, one of the factors that could be used to predict bonding efficacy is surface wettability [51]. Numerous approaches can be used to measure wettability, including the drop-shape analysis method, the Wilhelmy balance method, and the traditional telescope-goniometer method [52]. The sessile drop method, which allows a liquid on a solid surface to maintain its contact angle in a dry environment, was applied in the current study [53].

It has been shown that the parameter resulting from the surface chemistry and roughness of the substrate determines the contact angle [51]. The contact angle for a hydrophilic surface may decrease with increased surface roughness [54]. Nonetheless, the contact angle diminishes as surface roughness increases to a point where a greater improvement in wetting is not anticipated for extremely rough surfaces [55]. The liquid wets the substrate if the contact angle is < 90°, a surface that is not wet is indicated by an angle > 90° and a contact angle of 0° signifies complete wetting. Consequently, wettability and contact angle have an inverse relationship; the greater the wettability, the lower the contact angle, and vice versa [51, 53]. The difference in contact angle measurements of the polished TC surfaces varied significantly between the three materials (p < 0.05). After surface treatment, milled and 3D-printed materials showed significantly decreased differences in contact angle, whereas the conventional material showed a non-significant decrease in contact angle. The decreased contact angle from polished to surface treated indicated increased wettability of the surfaces. Among the materials, 3D-printed materials demonstrated high contact angle, which could be related to the low roughness.

According to the authors' knowledge, this is the first study to evaluate the SBS between aesthetic brackets and aged temporary crowns fabricated by conventional and digital methods. The specimens used in the study were crowns rather than flat surfaces, used in most SBS studies. The test conditions and aging were applied to simulate the clinical conditions as closely as possible. Despite the study's strengths, the limitations are also worth mentioning. The inability to completely replicate the conditions of an individual's oral environment is a primary limitation of this *in-vitro* study. The orthodontic brackets and the interfacial bond are vulnerable to varying temperatures, moisture content, and forces acting in different directions in the *in-vivo* environment, which was not considered in this study. In terms of the oral environment's factors, such as fatigue loading, dynamic forces of chewing, oral hygiene and dietary intake, in-vitro data cannot be related to clinical practice despite thermocycling and shear testing simulating these conditions. Thus, it is recommended that future research be conducted in environments that mimic the actual oral environment using various temporary crown materials and surface treatments. Furthermore, it would be interesting to see how this adhesive bond reacts to dynamic masticatory forces, acidic drinks and toothbrushing.

CONCLUSIONS

Aesthetic orthodontic brackets bonded to aged conventional and digitally fabricated temporary crowns demonstrate adequate bond strength at baseline and after aging, except for 3D-printed temporary crowns after thermocycling. Surface roughness and wettability data of the three-method temporary crowns were consistent with the SBS values.

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Authors contribution

M.M.A. and D.B. – conceptualization, methodology, formal analysis, investigation, writing-original draft, writing-review and editing; N.A. – investigation, visualization, writing-original draft; A.A.A. – methodology, formal analysis, investigation; T.E.-B. – conceptualization, supervision, writing-review and editing.

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Conflict of interest

The authors declare no conflict of interest.

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REFERENCES

- [1] Ajwa N., AlHammad A., AlAmmar L. *et al.*: *Healthcare* 2022, 10(11), 2153. https://doi.org/10.3390/healthcare10112153
- [2] Alijani S., Fotovat F., Rezaei Soufi L. *et al.*: *International Orthodontics* 2023, *21(3)*, 100790.
 https://doi.org/10.1016/j.ortho.2023.100790
- Fotovat F., Shishehian A., Alijani S. et al.: International Orthodontics 2022, 20(2), 100641.
 https://doi.org/10.1016/j.ortho.2022.100641
- [4] Kedia N.B., Bangar B., Rao S.J. et al.: Journal of Pharmacy and Bioallied Sciences 2023, 15, S1013. https://doi.org/10.4103/jpbs.jpbs_248_23
- Shahin S.Y., Abu Showmi T.H., Alzaghran S.H. et al.: International Journal of Dentistry 2021, 1, 9999933. https://doi.org/10.1155/2021/9999933
- [6] Biadsee A., Rosner O., Khalil C. et al.: Korean Journal of Orthodontics 2023, 53(1), 45. https://doi.org/10.4041/kjod22.098
- [7] Goracci C., Özcan M., Franchi L. *et al.*: *Korean Journal* of Orthodontics 2019, 49(6), 404. https://doi.org/10.4041/kjod.2019.49.6.404
- [8] 8] Haber, D., Khoury, E., Ghoubril, J. and Cirulli, N. *Dentistry Journal* **2023**,11, 38.
- Karaokutan I., Sayin G., Kara O.: The Journal of Advanced Prosthodontics 2015, 7(1), 27. https://doi.org/10.4047/jap.2015.7.1.27
- [10] Bangalore D., Alshehri A.M., Alsadon O. *et al.*: *Polymers* 2023, 15(9), 2164. https://doi.org/10.3390/polym15092164
- [11] Ahmed K.E.: *Primary Dental Journal* **2018**, 7(2), 30. https://doi.org/10.1177/205016841800700205
- [12] Alsarani M.M.: *The Saudi Dental Journal* 2023, 35(8), 939.
 - https://doi.org/10.1016/j.sdentj.2023.07.017
- [13] van Noort R.: Dental Materials 2012, 28(1), 3. https://doi.org/10.1016/j.dental.2011.10.014
- [14] Rendas P., Figueiredo L., Machado C. *et al.*: . *Progress in Biomaterials* 2023, *12*, 89. https://doi.org/10.1007/s40204-022-00214-6
- [15] AhmadAbadi M.N., Goharifar A., Mahabadi M.: Dental Research Journal **2023**, 20, 86.
- [16] Stansbury J.W., Idacavage M.J.: Dental Materials 2016, 32(1), 54.
 - https://doi.org/10.1016/j.dental.2015.09.018
- [17] Matasa C.G.: American Journal of Orthodontics and Dentofacial Orthopedics 1989, 95(4), 355. https://doi.org/10.1016/0889-5406(89)90171-6
- [18] Kafle D., Mishra R.K., Hasan R.: International Journal of Dentistry 2020, 2020(1), 8810946. https://doi.org/10.1155/2020/8810964
- [19] Jain S., Sayed M.E., Shetty M. et al.: Polymers 2022, 14(13), 2691.
 - https://doi.org/10.3390/polym14132691
- [20] Baser H., Ozel M.B., Baser B.: Annals of Medical Research 2023, 30(5), 621.

https://doi.org/10.5455/annalsmedres.2023.04.098

- [21] Chay S.H., Wong S.L., Mohamed N. et al.: American Journal of Orthodontics and Dentofacial Orthopedids 2007, 132(5), 577.e7. https://doi.org/10.1016/j.ajodo.2004.01.024
- [22] Iliadi A., Baumgartner S., Athanasiou A.E. et al.: American Journal of Orthodontics and Dentofacial Orthopedics 2014, 145(4), 425. https://doi.org/10.1016/j.ajodo.2013.11.020
- [23] Della Bona A., Kochenborger R., Di Guida L.: *Current Dentistry* 2019, 1(1), 40. http://doi.org/10.2174/2542579X01666180919121640
- [24] Mehmeti B., Azizi B., Kelmendi J. et al.: Acta Stomatologica Croatia 2017, 51(2), 99. https://doi.org/10.15644/asc51/2/2
- [25] Durgesh B.H., Hijji S.A., Al Kheraif A.A. et al.: International Journal of Adhesion and Adhesives 2015, 62, 101. https://doi.org/10.1016/j.ijadhadh.2015.07.006
- [26] Morresi A.L., D'Amario M., Capogreco M. et al.: Journal of the Medical Behavior of Biomedical Materials 2014, 29, 295. https://doi.org/10.1016/j.jmbbm.2013.09.013
- [27] Alshahrani I., Asiry M.A., Altwijry M.K. et al.: Polymers and Polymer Composites 2019, 27(2), 92. https://doi.org/10.1177/0967391118819703
- [28] Alsarani M., Alaida W., Ajwa N. et al.: Polimery 2023, 68(11-12), 607.
 - https://doi.org/10.14314/polimery.2023.11.4
- [29] Durgesh B., Alaqeel S., Ajwa N. et al.: Ceramics-Silikaty
 2020, 64(4), 469.
 https://doi.org/10.13168/cs.2020.0034
- [30] Årtun J., Bergland S.: American Journal of Orthodontics 1984, 85(4), 333.
 https://doi.org/10.1016/0002-9416(84)90190-8
- [31] Soon H.I., Gill D.S., Jones S.P.: *Journal of Orthodontics* 2015, 42(3), 192.
- https://doi.org/10.1179/1465313315y.0000000003 [32] Bourke B.M., Rock W.P.: *British Journal of Orthodontics* **1999**, *26*(4), 285.

https://doi.org/10.1093/ortho/26.4.285

- [33] Kukiattrakoon B., Samruajbenjakul B.: European Journal of Orthodontics 2010, 32(1), 87. https://doi.org/10.1093/ejo/cjp055
- [34] Garcés G.A., Rojas V.H., Bravo C. et al.: Dental Press Journal of Orthodontics 2020, 25(3), 31. https://doi.org/10.1590/2177-6709.25.3.031-038.oar
- [35] Degrazia Weidenbach F., Justino F.B., Grehs R.A. et al.: *Revista da Faculdade de Odontologia UPF* 2013, 18(1), 83.
 https://doi.org/10.5335/rfo.v18i1.3120
- [36] Yuasa T., Iijima M., Ito S. et al.: European Journal of Orthodontics 2010, 32(3), 285. https://doi.org/10.1093/ejo/cjp118
- [37] Jurubeba J.E.P., Costa A.R., Correr-Sobrinho L. *et al.*: *Brazilian Dental Journal* 2017, 28(2), 206. https://doi.org/10.1590/0103-6440201701217

- [38] El-Ramly M., Mowafy M.I., Abdel-Haffiez S. H.: Alexandria Dental Journal 2023, 48(2), 188. https://doi.org/10.21608/adjalexu.2022.142358.1284
- [39] Gale M.S., Darvell B.W.: *Journal of Dentistry* **1999**, 27(2), 89.
- https://doi.org/10.1016/s0300-5712(98)00037-2
- [40] Bishara S.E., Ostby A.W., Laffoon J.F. et al.: The Angle Orthodontist 2007, 77(2), 337.
 https://doi.org/10.2319/0003-3219(2007)077[0337:SBSC OT]2.0.CO;2
- [41] Al Jabbari Y.S., Al Taweel S.M., Al Rifaiy M. et al.: The Angle Orthodontist 2014, 84(4), 649. https://doi.org/10.2319/090313-649.1
- [42] De Abreu Neto H.F., Costa A.R., Correr A.B. et al.: Brazilian Dental Journal 2015, 26(6), 685. https://doi.org/10.1590/0103-6440201300416
- [43] de Fatima Fraga P., de Godoi A.P.T., Costa A.R. et al.: Brazilian Journal of Oral Sciences 2017, 15(2), 176. https://doi.org/10.20396/bjos.v15i2.8648779
- [44] Lopes G.V., Correr-Sobrinho L., Correr A.B. et al.: Brazilian Dental Journal 2020, 31(1), 52. http://doi.org/10.1590/0103-6440202003101
- [45] Nabawy Y.A., Yousry T.N., El-Harouni N.M.: BMC Oral Health 2021, 21, 306. https://doi.org/10.1186/s12903-021-01669-y
- [46] de Almeida J.X., Deprá M.B., Marquezan M. *et al.*: Dental Press Journal of Orthodontics **2013**, 18(4), 29. https://doi.org/10.1590/S2176-94512013000400006
- [47] Blakey R., Mah J.: American Journal of Orthodontics and Dentofacial Orthopedics **2010**, 138(1), 72.

https://doi.org/10.1016/j.ajodo.2008.08.030

- [48] Cardoso R.M., Godinho J., Jardim L.: Revista Portuguesa de Estomatologia, Medicina Dentária e Cirurgia Maxilofacial 2021, 62(1), 16. https://doi.org/10.24873/j.rpemd.2021.01.818
- [49] Hooman Z.N., Moradi M., Torkan S.: International Orthodontics 2019, 17(1), 89. https://doi.org/10.1016/j.ortho.2019.01.017
- [50] Ge D., Deng J., Duan R. et al.: Ceramics International 2019, 45(18, Part A), 24554. https://doi.org/10.1016/j.ceramint.2019.08.184
- [51] Wongsue S., Thanatvarakorn O., Prasansuttiporn T. et al.: Scientific Reports 2023, 13, 18249. https://doi.org/10.1038/s41598-023-45510-8
- [52] Yuan Y., Lee T. R.: "Contact Angle and Wetting Properties" in "Surface Science Techniques", (editors: Bracco G., Holst B.), Springer Berlin, Heidelberg 2013. p. 3. https://doi.org/10.1007/978-3-642-34243-1_1
- [53] Katyal D., Subramanian A.K., Venugopal A. et al.: International Journal of Dentistry 2021, 2021(1), 9457553. https://doi.org/10.1155/2021/9457553
- [54] Rupp F., Gittens R.A., Scheideler L. *et al.*: Acta Biomaterialia 2014, 10(7), 2894. https://doi.org/10.1016/j.actbio.2014.02.040
- [55] Kuznetsov G.V., Islamova A.G., Orlova E.G. et al.: Surface and Coatings Technology 2021, 422, 127518. https://doi.org/10.1016/j.surfcoat.2021.127518 Received 26 VIII 2024.

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