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AAO Foundation Final Report Form (a/o 6/30/2020)

Type of Award

Biomedical Research Award

Name(s) of Principal Investigator(s)

F. Kurtis Kasper

Institution

The University of Texas Health Science Center at Houston

Title of Project

Characterization of 3D-Printed Esthetic Orthodontic Brackets

Period of AAOF Support

07-01-20 to 06-30-21

No Cost Extension 07-01-21 to 06-30-22

Amount of Funding

\$30,000

Summary/Abstract

Objectives: A variety of considerations drive patient and clinician decisions associated with orthodontic treatment. Patients often present concern regarding the esthetics associated with fixed appliances during treatment, which has motivated considerable investment over the years in the development of “esthetic brackets,” including plastic and ceramic brackets. Recent advances in additive manufacturing technologies present the potential for fabrication of esthetic orthodontic brackets in-office via 3D-printing using materials cleared for intraoral use. Direct fabrication of esthetic brackets via 3D-printing could revolutionize orthodontics by enabling clinicians to design and fabricate customized brackets that satisfy patient demands for esthetics in an on-demand fashion. At the same time, in-office design and fabrication of brackets via 3D-printing would support increased clinician control and operational efficiency in orthodontic practices. As clinical cases of 3D-printed brackets emerge in the literature, a clear and urgent need exists to investigate

key properties of 3D-printed brackets, including their mechanical properties and color stability, to inform the orthodontic community regarding potential advantages and limitations. The overall objective of the project was to evaluate the mechanical properties and color stability of 3D-printed orthodontic brackets fabricated with a filled biocompatible resin composite. Overall, the information gained through this project will inform the orthodontic community regarding key mechanical and color properties of 3D-printed esthetic brackets to guide appropriate use of the emerging approach.

Specific Aim 1 – Shear Bond Strength of Orthodontic Brackets Fabricated via 3D-Printing Using Filled Biocompatible Resins: Specific Aim 1 involved investigation of the shear bond strength of orthodontic brackets fabricated via 3D-printing using filled biocompatible resins when bonded to extracted human teeth. It was hypothesized that the initial shear bond strength of 3D-printed brackets would not differ significantly from that of corresponding commercially available plastic brackets, and that the shear bond strength of the 3D-printed brackets would increase with treatment of the bracket pad to increase surface area. To this end, 20 brackets of each of 4 biocompatible resins marked for dental applications (GR- 17.1 A1, GR- 17.1 A2, GR- 17.1 A3, and GR-10 Guide) were printed with an Asiga Max UV 3D-printer using a single master standard tessellation language (STL) file matching the American Orthodontics Silkon Plus™ design. Twenty Silkon Plus™ brackets were also obtained. Brackets were bonded to 100 mounted extracted human premolars and placed in a 37°C distilled water bath for 36 hours to simulate the oral environment. Brackets were then debonded using an Instron universal testing system. Maximum load was recorded and used to calculate shear bond strength. The buccal surface of each tooth was examined and photographed to evaluate the amount of adhesive remaining via adhesive remnant index (ARI) scoring. Statistical analysis included a generalized linear model with post-hoc Tukey contrasts to evaluate the effect of bracket material on shear bond strength. A Kruskal-Wallis rank sum test with post-hoc pairwise comparisons was used to evaluate the effect of bracket material on ARI score.

Mean shear bond strength of the 3D-printed brackets ranged from 10.033 ± 1.761 to 12.766 ± 1.666 MPa, while the shear bond strength of the conventionally manufactured brackets was statistically significantly lower at 7.467 ± 1.024 MPa ($p < 0.001$ for all pairwise comparisons; see Figure 1; Appendix). The GR-10 Guide group displayed statistically significantly lower shear bond strength than the GR-17.1 A1, A2, and A3 groups ($p < 0.001$, $p < 0.001$, and $p < 0.006$, respectively). There were no statistically significant differences in shear bond strength between the GR-17.1 A1, A2, and A3 groups. The conventionally manufactured group demonstrated significantly lower ARI scores than all other groups ($p < 0.001$ for all pairwise comparisons). There were no statistically significant differences in ARI scores between the 3D-printed groups (see Figure 2; Appendix). 3D-printed orthodontic brackets fabricated with GR-10 Guide and GR-17.1 (shades A1, A2, and A3) resins demonstrated clinically acceptable shear bond strengths under the conditions used in this study. 3D-printed brackets demonstrated higher ARI scores compared to conventionally manufactured brackets, indicating that less composite remained on the tooth after debond of the bracket. Overall, the results suggest that 3D-printed brackets appear to have promise for clinical applications in orthodontics, but more research is indicated to elucidate additional properties.

An additional study was completed to investigate the effect of air abrasion of bracket pads on the shear bond strength of 3D-printed plastic orthodontic brackets when bonded to the enamel of extracted human teeth. To this end, 125 deidentified extracted human premolars were divided into

6 groups of 20 premolars each. Eighty premolar brackets were 3D-printed using an American Orthodontics (AO) Silkon Plus™ bracket STL file from the manufacturer in 2 different commercially available biocompatible 3D-printing resins marketed for dental applications: Dental LT Resin (n = 40) and Dental SG Resin (n = 40). The 40 brackets 3D-printed in each resin plus 40 commercially manufactured brackets were divided into 2 groups of 20 brackets each, and 1 of the 2 groups of each resin was air abraded using 50 µm aluminum oxide at 80 pounds per square inch (psi). All brackets were bonded to the extracted premolars, and shear bond strength tests were performed on all samples with an Instron universal testing system. The failure types of each sample were classified using a 5-category modified ARI scoring system. A generalized linear model using the glm function with gamma specified error distribution was applied to evaluate effects of bracket material and air abrasion on shear bond strength, since data were not normally distributed. The POLR function was used to examine effects of bracket and air abrasion on ARI, as ARI is an ordered categorical variable.

Bracket material and bracket pad surface treatment presented statistically significant effects for mean shear bond strengths ($p=0.004$ and $p=0.005$, respectively), and a significant interaction effect between bracket material and bracket pad surface treatment was observed ($p<0.001$). The non-air abraded (NME) SG group (8.87 ± 0.64 MPa) had a statistically significantly lower shear bond strength than the air abraded (ME) SG group (12.09 ± 1.23 MPa; see Figure 3; Appendix) ($p<0.05$). In the manufactured brackets and LT Resin groups, the NME and ME groups were not statistically significantly different within each resin group ($p>0.05$). None of the samples in the study received an ARI score of 4 or 5. A significant effect of bracket material and bracket pad surface treatment on ARI score was observed ($p<0.001$ and $p<0.001$, respectively), but no significant interaction effect between bracket material and bracket pad surface was found ($p=0.067$). Within each bracket material, the group with ME surface treatment demonstrated ARI scores of 3 with greater frequency than the NME group (see Figure 4; Appendix). The traditionally manufactured brackets demonstrated low ARI scores with greater frequencies than the 3D-printed brackets. Based on the results of this study, follow-up clinical studies may further examine printing mediums and bracket pad optimization for clinically acceptable shear bond strengths to inform best practices. Overall, 3D-printed orthodontic brackets based on a Silkon Plus™ design printed in resins marketed for intraoral use (SG and LT) presented clinically sufficient shear bond strengths both with and without air abrasion prior to bonding. The effect of bracket pad air abrasion on shear bond strength depends on the bracket material. If clinicians are utilizing SG resin to print brackets, air abrasion of bracket pad bases prior to bonding could help increase shear bond strengths.

Specific Aim 2 – Investigation of Color Stability of 3D-Printed Orthodontic Brackets: Specific Aim 2 involved investigation of the color stability of 3D-printed orthodontic brackets. It was hypothesized that the composite resins applied in the fabrication of 3D-printed brackets would present initial color and translucency within acceptable limits, but that accelerated aging and staining solutions would each induce changes in the color and translucency beyond acceptable limits. To this end, GR-17.1 (shades A1, A2, and A3) and GR-10 Guide resins were printed on an Asiga MAX UV printer into discs 2 mm thick, with a diameter of 10 mm, and then post-processed as per manufacturer's instructions. Discs were immersed in 5 mL of coffee, tea, red wine, or distilled water for 1 week. Another group was subjected to artificial accelerated aging as per ISO Standard 4892-2. Ten samples were produced per resin, per treatment condition. Color measurements were taken on the discs before and after treatment using a spectrophotometer against

white and black reference tiles to assess color and translucency. Statistical analysis included a generalized linear model with post-hoc Tukey contrasts to evaluate the effect of bracket material and treatment condition on total change in color (ΔE_{00}) and translucency (ΔTP_{00}).

A statistically significant effect of the treatment ($p < 0.001$) and the disc material ($p < 0.001$) were found for ΔE_{00} , while only the treatment produced a significant change in ΔTP_{00} ($p < 0.001$), with the type of resin having no significant effect on the change in translucency parameter. An interaction effect between the treatment effects and the material was present for ΔE_{00} ($p < 0.001$), but not for ΔTP_{00} . Among the treatment conditions, immersion in red wine produced the greatest change in color (ΔE_{00}), except in shade A2 resin, which experienced the greatest change with coffee (see Figure 5; Appendix). Red wine also produced the greatest change in translucency parameter (ΔTP_{00}) for all materials except the A3 resin, where coffee also had a greater effect (see Figure 6; Appendix). Qualitatively, red wine and coffee had the largest effect on resin color. Immersion in tea caused staining in the resin samples to a lesser degree than coffee or red wine, but more than exposure to artificial aging, which had the least effect on color of any experimental treatment tested in this study (see Figure 7; Appendix). Artificial aging was not shown to have a significant effect on translucency. The control for the study was immersion in distilled water, which itself produced a mild color change for GR-17.1 filled resin samples, and a moderate color change for GR-10 Guide resin. The effects of distilled water on the samples were not apparent qualitatively. The three GR-17.1 resin materials – shade A1, A2, and A3 – had similar magnitudes of ΔE_{00} in response to the treatment conditions. The unfilled GR-10 Guide resin underwent significantly more color change than the GR-17.1 resins for all treatment conditions except for immersion in coffee. This resin was also the most variable in its response to treatment condition, with the standard deviations much wider than that of the other groups. Overall, while initial color of the printed resin discs was acceptable, all resin groups underwent significant color change during the experiment. Red wine and coffee produced the greatest color and translucency change, followed by tea, with artificial aging producing the least change in color and translucency. The 3D-printed resins tested underwent significant changes in color and translucency following exposure to endogenous and exogenous sources of staining, and are not recommended for esthetic orthodontic bracket applications.

Respond to the following questions:

1. Were the original, specific aims of the proposal realized?

Yes

2. Were the results published?

a. If so, cite reference/s for publication/s including titles, dates, author or co-authors, journal, issue and page numbers

Hanson, M. S. (2021). Effect of Pad Abrasion on Shear Bond Strength of 3D-Printed Orthodontic Brackets (Order No. 28650314). Available from Dissertations & Theses @ University of Texas School of Dentistry at Houston. (2562790237). Retrieved from <https://libdb.db.uth.tmc.edu/login?url=https://www.proquest.com/dissertations-theses/effect-pad-abrasion-on-shear-bond-strength-3d/docview/2562790237/se-2?accountid=7127>

b. Was AAOF support acknowledged?

Yes, as appropriate, AAOF support was acknowledged in each publication.

c. If not, are there plans to publish? If not, why not?

Yes, the project involved contributions from 3 residents in partial completion of the requirements of the degree of Masters of Science in Dentistry. Some project results were published in a thesis detailed above and other results will be published in planned theses, as follows:

1. Siller J. Shear Bond Strength of Orthodontic Brackets Fabricated via 3D-Printing Using Filled Biocompatible Resins. Masters of Science in Dentistry Thesis, Department of Orthodontics, The University of Texas School of Dentistry at Houston, Houston, Texas. (in preparation).
2. Wallach R. Color Stability of 3D-Printed Orthodontic Brackets Using Filled Resins. Masters of Science in Dentistry Thesis, Department of Orthodontics, The University of Texas School of Dentistry at Houston, Houston, Texas. (in preparation).

In addition, three research manuscripts based on the results of the project are presently under preparation for submission to peer-reviewed journals. In each case, the submissions will acknowledge AAOF support, as appropriate.

3. Have the results of this proposal been presented?

a. If so, list titles, author or co-authors of these presentation/s, year and locations

Siller J, Hanson M, English JD, Harrington D, Ontiveros J, Wirthlin J, Cozad B, Kasper FK. Effect of Pad Abrasion on Shear Bond Strength of 3D-Printed Orthodontic Brackets. American Association of Orthodontists Annual Meeting, Miami, FL. May 21-24, 2022. (E-Poster Presentation)

b. Was AAOF support acknowledged?

Yes, as appropriate, AAOF support was acknowledged in each presentation.

c. If not, are there plans to do so? If not, why not?

The results will continue to be included in presentations, as appropriate, with proper acknowledgement of support from AAOF for the work. Planned presentations include the following:

Kasper FK. Leaving the Stone Age: Applying Biomaterials and 3D Printing to Meet Clinical Needs. American Institute of Oral Biology 79th Annual Meeting, Palm Springs, CA. October 21-23, 2022. (Invited Oral Presentation)

4. To what extent have you used, or how do you intend to use, AAOF funding to further your career?

As a bioengineer, I am thrilled to explore exciting new frontiers at the intersection of engineering and orthodontics, and funding from the AAOF has been vital to enable my investigations in these areas that traditionally are not targets for funding from federal sources. The funding from AAOF

provides me with opportunities to expand my exposure to the challenges of clinical orthodontics, to collaborate with clinicians and researchers in the field, to increase my research profile, and to broaden my professional network. Indeed, support from the AAOF has been instrumental in my career advancement through the academic ranks from an Assistant Professor to a recently approved promotion to Professor (Tenured). The benefits enabled by AAOF support provide a firm foundation upon which I plan to continue to build my research program in topics of relevance to orthodontics.

Accounting for Project

Approximately \$26,004 of the \$30,000 project budget was expended or encumbered to-date in completion of the project.

Appendix

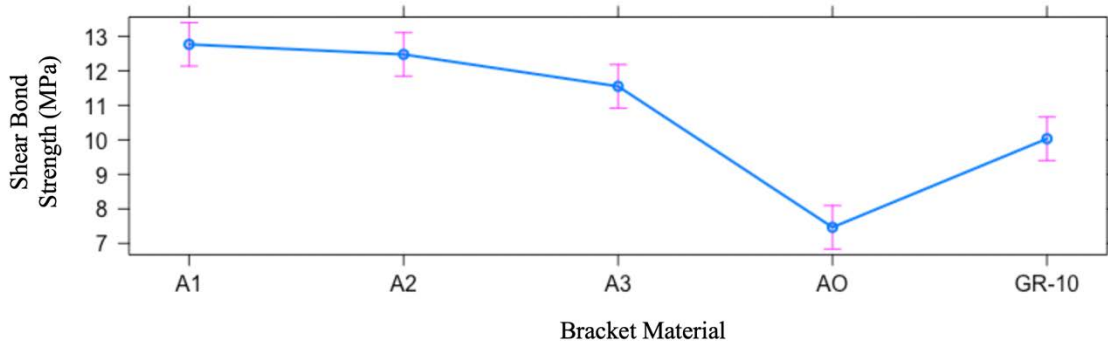


Figure 1. Mean shear bond strength and 95% confidence interval of each bracket material. A1 = GR-17.1 A1 resin group; A2 = GR-17.1 A2 resin group; A3 = GR-17.1 A3 resin group; GR-10 = GR- 10 Guide resin group; AO = American Orthodontics conventionally manufactured bracket group.

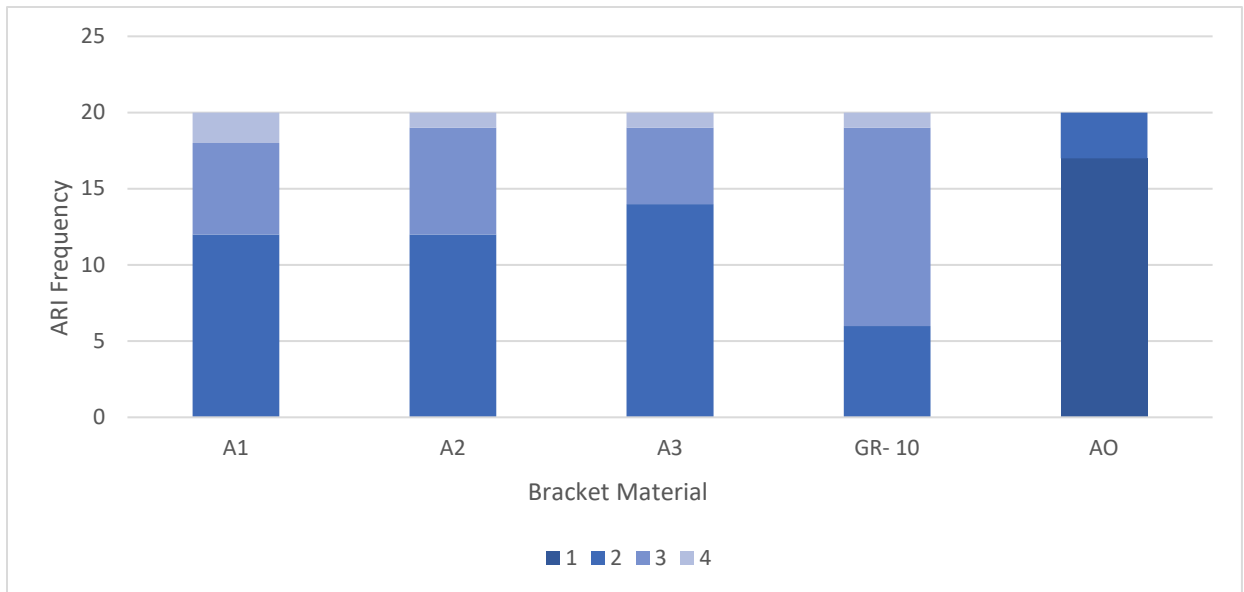


Figure 2. Frequency of ARI score by bracket material. A1 = GR-17.1 A1 resin group; A2 = GR-17.1 A2 resin group; A3 = GR-17.1 A3 resin group; GR-10 = GR-10 Guide resin group; AO = American Orthodontics conventionally manufactured bracket group.

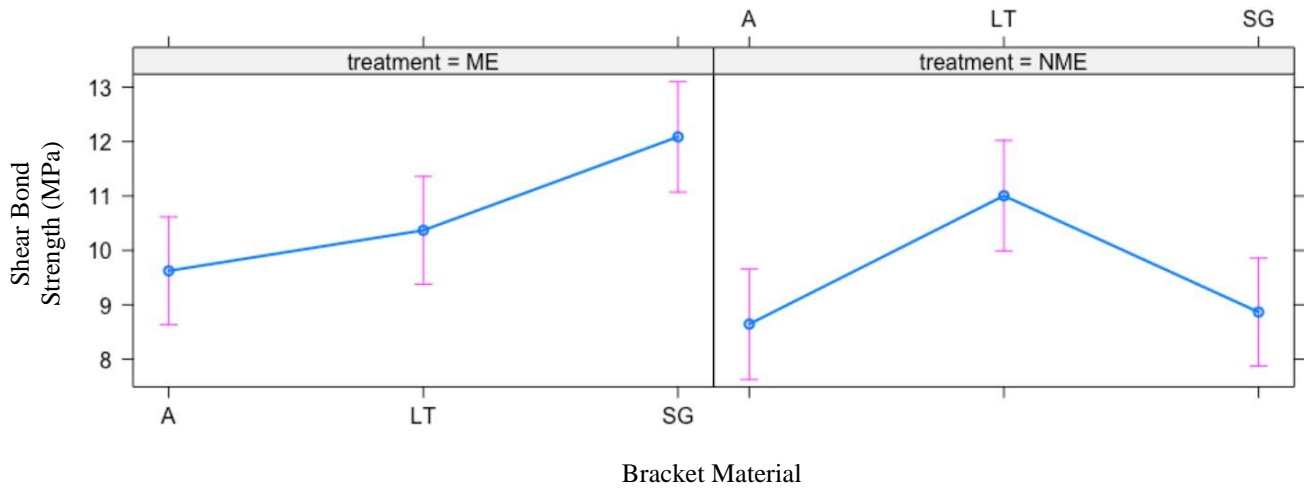


Figure 3. Mean shear bond strength and 95% confidence interval of each bracket material with air abrasion (ME) and without air abrasion (NME) of the bracket pad. A = American Orthodontics conventionally manufactured bracket group; LT = Dental LT Resin group; SG = Dental SG Resin group.

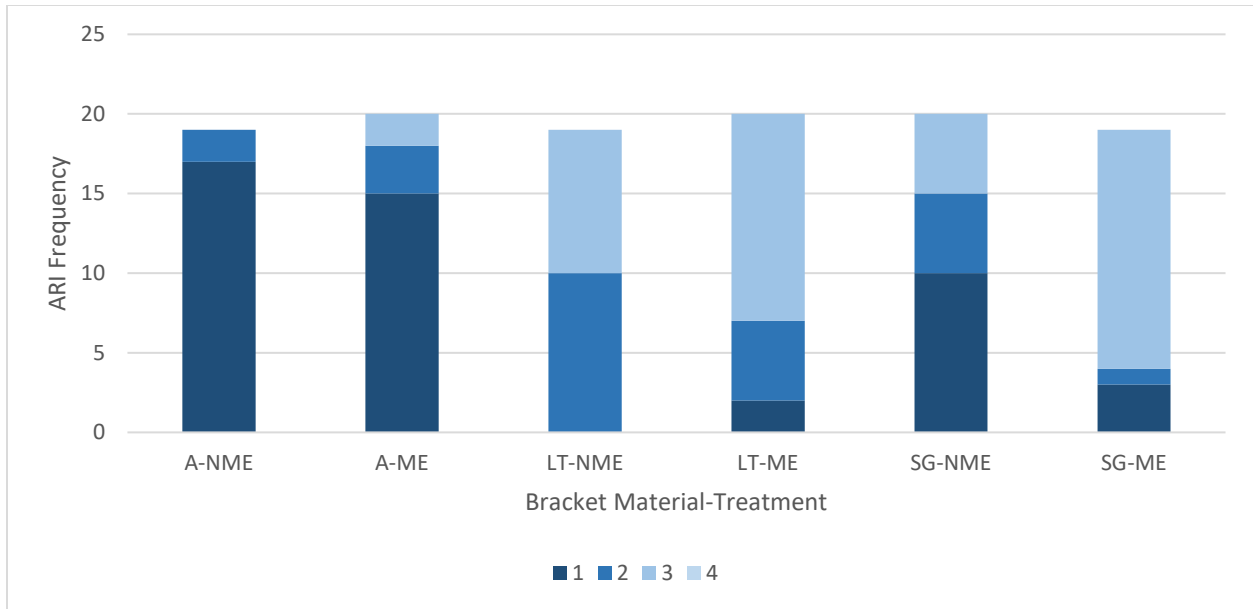


Figure 4. Frequency of ARI score by bracket material. A = American Orthodontics conventionally manufactured bracket group; LT = Dental LT Resin group; SG = Dental SG Resin group. NME = no air abrasion of the bracket pad; ME = air abrasion of the bracket pad.

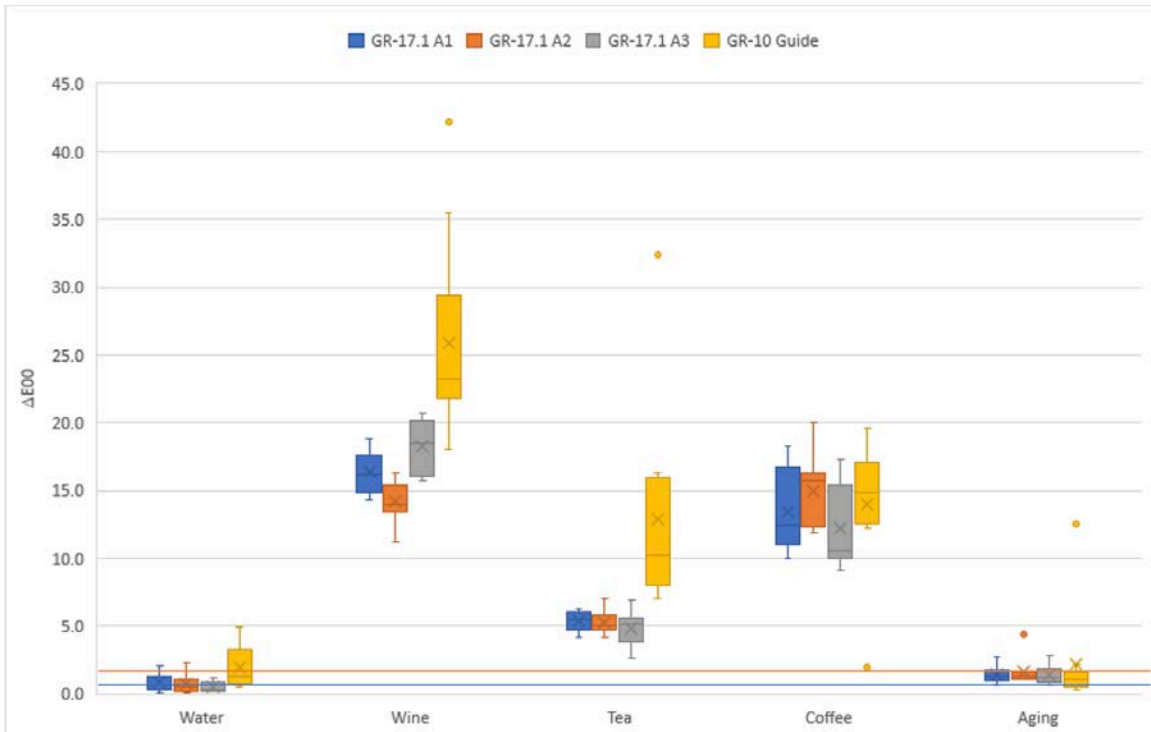


Figure 5. Box and whisker plot showing the distribution of color change (ΔE_{00}) observed for each material under experimental conditions. Blue line indicates the perceptibility threshold, and the orange line indicates the acceptability threshold.

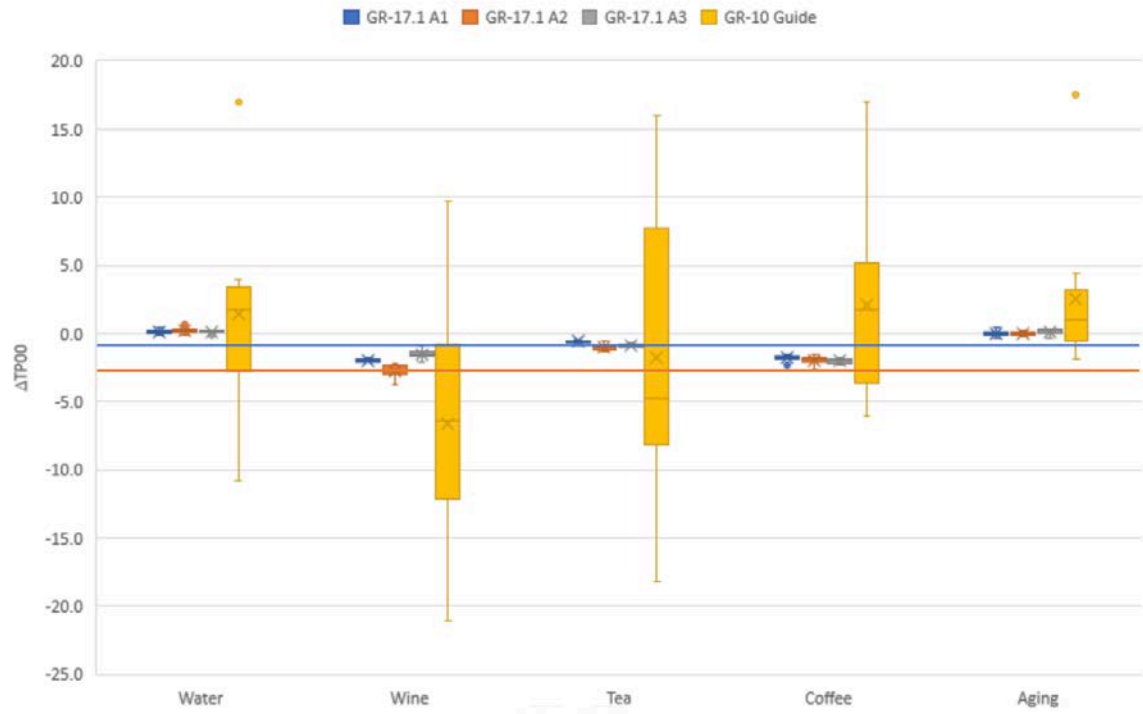


Figure 6. Box and whisker plot showing the distribution of translucency parameter change (ΔTP_{00}) observed for each material under experimental conditions. Blue line indicates the translucency perceptibility threshold, and the orange line indicates the translucency acceptability threshold.

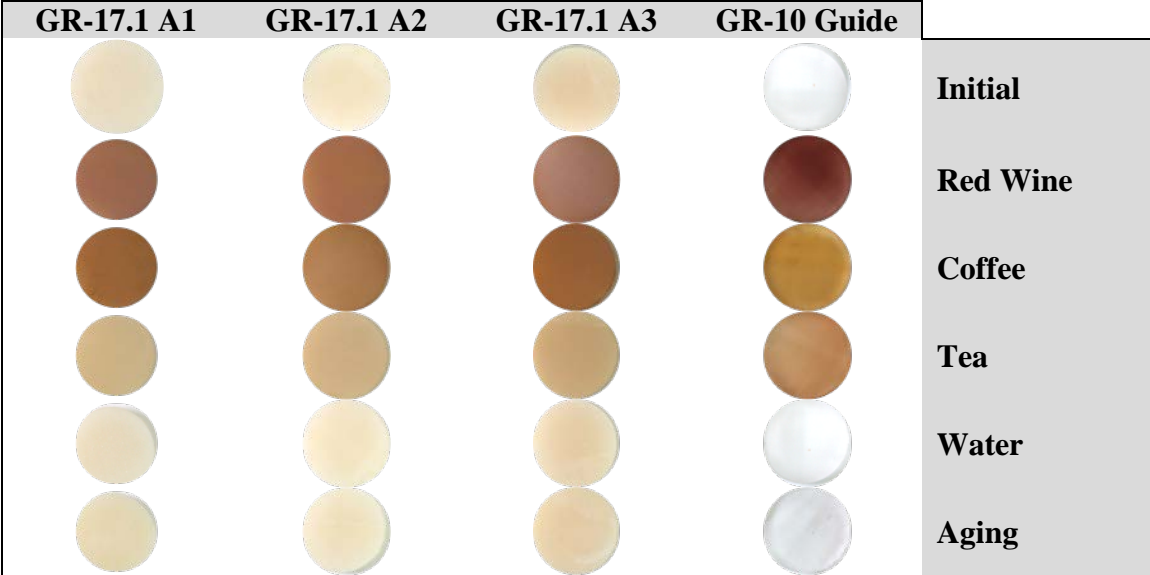



Figure 7. Representative photographs of 3D-printed resin samples pre- and post-treatment.

Colour stability of 3D-Printed orthodontic brackets using filled resins

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Funding information

American Association of Orthodontists Foundation

Abstract

Objective: To determine the effect of common beverages and accelerated aging on the colour stability of filled resins, which could potentially be used for fabrication of 3D-printed orthodontic brackets.

Materials and Methods: GR-17.1 (shades A1, A2, and A3), and GR-10 Guide resins (pro3dure medical, Eden Prairie, MN) were printed on an Asiga MAX UV printer into discs 2mm thick, with a diameter of 10mm, and then post-print processed as per manufacturer's instructions. Discs were immersed in 5mL of coffee, tea, red wine, or distilled water for 7 days. Another group was subjected to accelerated aging in accordance with ISO Standard 4892-2. Ten samples were produced per resin, per treatment condition. Colour measurements were taken on the discs before and after treatment using a spectrophotometer against white and black reference tiles to assess colour and translucency differences with the CIEDE2000 colour difference formula.

Results: While initial colour of the printed resin discs was acceptable, all resin groups underwent significant colour change during the experiment. Red wine and coffee produced the greatest colour and translucency change, followed by tea, with accelerated aging producing the least change in colour and translucency.

Conclusion: The 3D-printed resins tested underwent significant changes in colour and translucency following exposure to endogenous and exogenous sources of staining, which may affect their acceptability for fabrication of aesthetic orthodontic brackets.

KEYWORDS

aesthetics, dental, orthodontic brackets, printing, three-dimensional

1 | INTRODUCTION

Aesthetics play an integral part in nearly every orthodontic treatment plan, and it has previously been shown that the primary motivation for seeking orthodontic treatment for most patients is an improvement in dentofacial appearance.¹ As such, it is increasingly apparent that patients desire not just an attractive smile at the completion of treatment, but to undergo orthodontic treatment in the

most aesthetically pleasing manner possible. A variety of aesthetic bracket options have emerged in the fixed appliance space to meet this demand.

The earliest iterations of aesthetic brackets were made from unfilled polycarbonate,² but were prone to breakage under stress, slot distortion, increased friction compared to metal brackets, and discoloration.³⁻⁵ Subsequent advances in manufacturing allowed the creation of ceramic brackets, which have superior mechanical

and optical properties, but have their own limitations, including brittleness and the potential for iatrogenic damage to enamel during debonding,^{6,7} or to the opposing dentition during function.⁸ For practitioners who prefer plastic, manufacturers have introduced several modifications to bracket composition and design to overcome their mechanical shortcomings. These modifications include different matrix compositions, such as polyurethane or methacrylate matrices, the introduction of silica or fibreglass fillers for additional strength, and the insertion of a metal slot, which is particularly effective in increasing a plastic bracket's ability to resist creep during torqueing movements and reducing friction.^{4,9-11} Additionally, bond strength of plastic brackets has reached a level of clinical acceptability.¹² Given the viability of plastic brackets to be utilized for orthodontic treatment, several groups are now using emergent additive manufacturing methods, namely 3D printing, to produce brackets that not only have the aesthetics of plastic but the potential for individual customization of bracket prescription and design for each patient.¹³⁻¹⁶

While significant improvements in mechanical performance of plastic orthodontic brackets have been achieved, aesthetic issues, such as discoloration over time, remain a shortcoming.¹⁷ Colour stability, a material's ability to resist both endogenous and exogenous staining over time, can be quantitatively analysed via a number of methods. The most common method in dentistry uses the CIELAB colour space, first introduced in 1976, which defines the gamut of colours perceptible to the naked eye on a three-dimensional Cartesian coordinate system defined by the axes L^* - light/dark, a^* - red/green, and b^* - yellow/blue. Amount of discoloration is determined by the absolute distance between points representing the final and initial colour.¹⁸ $L^*a^*b^*$ coordinates can be transformed into $L^*C^*h^*$ coordinates, which define the same space using cylindrical, instead of Cartesian coordinates. In this system, L^* describes lightness/darkness, C^* describes chroma, the intensity of a colour, and h^* is a polar variable describing a colour's hue. Use of $L^*C^*h^*$ coordinates facilitates the determination of colour stability using the CIEDE2000 colour difference formula, an update to the CIELAB formula, first introduced in 2000, which improves perceptual uniformity of colour differences,¹⁹ a distinction which has been shown to be relevant in determination of clinically acceptable colour differences in dentistry.²⁰ Staining can occur endogenously from within the resin itself, or exogenously from external agents such as food dyes.²¹ Endogenous staining of resins is the result of oxidation of residual carbon-carbon double bonds following matrix polymerization, and typically leads to an increase in yellowness.²² Exogenous staining is due to absorption of staining agents into the matrix. Previous studies have shown that an increase in filler content decreases endogenous staining due to a reduction in the number of carbon-carbon double bonds available for oxidation. However, contrary to expectations, increasing filler content was shown to increase exogenous staining of some resin materials.²³ The proposed mechanism by which this occurred is that the interface between the filler and the matrix presents a point at which staining molecules are able to more easily penetrate. This effect has not been shown for all filled resins, and

has not previously been shown for filled resins used in 3D printing applications.

The objective of this study was to determine the colour stability of 3D-printed, commercially available, filled resins marketed for dental applications. It was hypothesized that when exposed to either exogenous staining from common beverages, or endogenous staining from accelerated aging, filled 3D-printed resin discs would undergo a significant degree of colour and translucency change.

2 | MATERIALS AND METHODS

2.1 | Disc fabrication

Two types of resin were tested, GR-17.1 shade A1, shade A2, shade A3, and GR-10 Guide (pro3dure medical, Eden Prairie, MN). Marketed for the additive manufacturing of long-term temporary crowns and bridges, GR-17.1 is composed of a matrix of methacrylate derivatives with up to 50% silicon dioxide filler. Filler size is between 0.4 and 3 μm .²⁴ GR-10 Guide is an unfilled resin produced from the esterification products of 4,4'-isopropylidenediphenol, and ethoxylated and 2-methylprop-2-enoic acid. It is marketed for the production of intraoral surgical guides for implant placement.²⁵ The resins were printed into discs 2mm in height and 10mm in diameter on an Asiga Max UV printer (Asiga, Alexandria, NSW, Australia) with a layer height of 100 μm . Discs were chosen as an analog for orthodontic brackets because the complex geometry of the brackets could present a confounding variable to the colour measurements if they were measured directly. The discs were printed with the long axis of the cylinder parallel to the build plate of the printer to prevent any effects that scraping the face of the disc off of the build plate would have on the surface texture and associated optical properties of the discs. In clinical use, the geometry of brackets would necessitate build supports during additive manufacturing, so this orientation also produces discs that more closely resemble the brackets for which they are analogs. Following printing, samples were processed as per protocols provided by the manufacturer: 4 minutes in a CLD-1 cleaning system (pro3dure medical LLC) with >97% isopropyl alcohol,²⁶ drying with compressed air, and post-print curing in a CD-2 light polymerization unit (pro3dure medical LLC) with an inert atmosphere of nitrogen gas.²⁷ The GR-10 Guide resin was cured for 4 minutes, and the GR-17.1 was cured for 10 minutes, per manufacturer's instructions.^{28,29}

2.2 | Exposure to staining agents

A total of 50 samples of each resin were produced, 10 for each of 3 exogenous staining treatments, 10 for endogenous staining, and 10 as a control. To represent various food dyes that may cause exogenous staining of intraoral appliances, samples were immersed in red wine (Frontera, Concha y Toro, Santiago, Chile), coffee (Folgers Classic Roast Medium, The Folger Coffee, Orrville, OH), or tea

(Unsweet black tea, Gold Peak Tea, Coca-Cola Company, Atlanta, GA) for a period of 7 days, with the solution changed daily. Coffee was prepared by mixing 30g of coffee grounds in 600mL of boiling water (1:20 ratio). The control group was immersed in distilled water. Staining via endogenous sources was produced via accelerated aging with a flatbed Suntest XXL+ xenon lamp weathering and lightfastness test chamber (Atlas Material Testing Technology LLC, Mount Prospect, IL) using light/dark and wet/dry cycles in accordance with conditions set forth in ISO Standard 4892-2 to a total light irradiant exposure of 300kJ/m². Cycles consisted of 102 minutes of light exposure under artificial daylight (CIE D65 illuminant) followed by 18 minutes of water spraying with a relative humidity of 50%, black panel temperature of 65°C with an ambient constant temperature of 37°C, and irradiance control in the 300-400nm interval of 60W/m².³⁰

2.3 | Colour measurement

Initial colour measurements were taken immediately after post-print processing, and final colour measurements were taken after rinsing and drying discs after 7 days in solution, or at the conclusion of accelerated aging. Each sample was measured once prior to and again after the respective treatment. Measurements were taken using a bench top spectrophotometer Ci7600 (X-Rite, Grand Rapids, MI) with CIE D65 standard illumination and 2° standard observer, against white ($L^* = 96.3$, $a^* = -0.6$, $b^* = 0.9$) and black ($L^* = 24.4$, $a^* = 0.2$, $b^* = -0.7$) calibration tiles. Spectral data were converted to CIEDE2000 colour coordinates, describing each sample using three parameters: L' – lightness, C' – chroma, and h' – a polar coordinate denoting hue. The total change in colour (ΔE_{00}) was calculated from the CIEDE2000 coordinates using the following formula:

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{K_L S_L} \right)^2 + \left(\frac{\Delta C'}{K_C S_C} \right)^2 + \left(\frac{\Delta H'}{K_H S_H} \right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C} \right) \left(\frac{\Delta H'}{K_H S_H} \right) \right]^{\frac{1}{2}}$$

$\Delta L'$, $\Delta C'$, and $\Delta H'$ represent the change in the sample's lightness, chroma, and hue. S_L , S_C , and S_H represent weighting functions improving perceptual uniformity. K_L , K_C , and K_H are parametric factors adjusted for different viewing parameters, such as textures or background, which were all set to 1 due to the use of standardized viewing conditions with a D65 illuminant. The translucency parameter determined by measuring the colour of a disc against both a white and a black background and calculating the colour difference between the two, as shown in the following equation, derived from the CIEDE2000 colour difference formula:

$$TP_{00} = \left[\left(\frac{L'_B - L'_W}{K_L S_L} \right)^2 + \left(\frac{C'_B - C'_W}{K_C S_C} \right)^2 + \left(\frac{H'_B - H'_W}{K_H S_H} \right)^2 + R_T \left(\frac{C'_B - C'_W}{K_C S_C} \right) \left(\frac{H'_B - H'_W}{K_H S_H} \right) \right]^{\frac{1}{2}}$$

Change in translucency is the magnitude of difference between final and initial translucency.

2.4 | Statistical analysis

Generalized linear models were applied using `glm()` in R statistical software (R Core Team 2018, R Foundation for Statistical Computing, Vienna, Austria) to examine the effects of the explanatory variables (type of resin and treatment) on the response variables ($\alpha = 0.05$).

3 | RESULTS

Colour for each sample was measured prior to and again immediately following immersion in staining solution or accelerated aging, and the change in L' , H' , and C' , as well as the overall change in colour (ΔE_{00}) and the translucency parameter (ΔTP_{00}) are shown in Table 1. As summarized in Table 2, statistically significant effects of the treatment ($P < .001$) and the disc material ($P < .001$) were found for ΔE_{00} , while only the treatment produced a significant change in ΔTP_{00} ($P < .001$), with the type of resin having no significant effect on the change in translucency parameter. An interaction effect between the treatment and the material was present for ΔE_{00} ($P < .001$), but not for ΔTP_{00} .

Among the treatment conditions, immersion in red wine produced the greatest change in colour (ΔE_{00}), except in shade A2 resin, which experienced the greatest change with coffee. Red wine also produced the greatest change in translucency parameter (ΔTP_{00}) for all materials except the A3 resin, where coffee also had a greater effect. Qualitatively, red wine and coffee had the largest effect on resin colour (Figure 1). Immersion in tea caused staining in the resin samples to a lesser degree than coffee or red wine, but more than exposure to accelerated aging, which had the least effect on colour of any experimental treatment tested in this study. Accelerated aging was not shown to have a significant effect on translucency. The control for the study was immersion in distilled water, which itself produced a mild colour change for GR-17.1 filled resin samples, and a moderate colour change for GR-10 Guide resin. The effects of distilled water on the samples were not apparent qualitatively.

The three GR-17.1 resin materials – shade A1, A2, and A3 – had similar magnitudes of ΔE_{00} in response to the treatment conditions. The unfilled GR-10 Guide resin underwent significantly more colour change than the GR-17.1 resins for all treatment conditions except for immersion in coffee. This resin was also the most variable in its response to treatment condition, with the standard deviations much wider than that of the other groups.

4 | DISCUSSION

The objective of this study was to evaluate the colour stability of commercially available filled resins marketed for intraoral dental applications, which could be candidates for in-office 3D-printed orthodontic bracket fabrication. Previous studies have evaluated the feasibility of unfilled resins for 3D-printed orthodontic brackets, both in terms of mechanical and aesthetic properties,³¹ but

TABLE 1 Mean and standard deviation of the change in lightness ($\Delta L'$), chroma ($\Delta C'$), hue ($\Delta H'$), colour change (ΔE_{00}), and translucency parameter (ΔTP_{00}) for each 3D-printed resin material before and after treatment.

Test	Resin	$\Delta L'$	$\Delta C'$	$\Delta H'$	ΔE_{00}	ΔTP_{00}
Coffee	GR-17.1 A1	-13.1±2.7	13.7±3.5	-17.4±5.7	13.4±3.0	-1.8±0.2
	GR-17.1 A2	-14.7±2.5	13.7±2.9	-23.8±3.9	15.0±2.6	-2.0±0.3
	GR-17.1 A3	-12.5±2.7	12.4±3.5	-8.2±4.3	12.2±2.9	-2.0±0.2
	GR-10 Guide	-5.8±5.2	20.2±8.5	-21.8±11.2	13.9±4.9	2.0±6.7
Wine	GR-17.1 A1	-14.9±2.8	7.2±2.7	-64.4±10.9	16.3±1.6	-2.0±0.1
	GR-17.1 A2	-11.3±1.4	7.4±1.5	-66.8±6.3	14.2±1.5	-2.8±0.5
	GR-17.1 A3	-18.7±3.2	5.4±3.3	-53.3±10.8	18.2±2.0	-1.5±0.3
	GR-10 Guide	-22.2±8.6	22.0±2.5	-87.9±20.9	25.9±7.5	-6.6±9.6
Tea	GR-17.1 A1	-4.8±0.6	5.6±1.0	-8.5±1.6	5.3±0.8	-0.6±0.1
	GR-17.1 A2	-5.1±0.6	4.7±1.3	-10.5±1.1	5.3±0.8	-1.0±0.2
	GR-17.1 A3	-4.7±1.1	5.3±1.5	-1.5±1.9	4.9±1.2	-0.9±0.1
	GR-10 Guide	-8.1±10.3	12.7±3.9	-38.1±19.9	12.9±7.6	-1.7±10.8
Aging	GR-17.1 A1	0.9±0.7	-1.1±0.7	6.3±4.2	1.4±0.6	0.0±0.2
	GR-17.1 A2	1.1±1.2	-1.4±0.9	7.1±4.7	1.6±1.0	0.0±0.2
	GR-17.1 A3	1.3±0.8	-1.2±0.7	3.2±2.3	1.4±0.7	0.1±0.2
	GR-10 Guide	0.1±2.7	1.5±4.7	-8.7±12.2	2.1±3.7	2.5±5.6
Water	GR-17.1 A1	0.5±0.9	-0.2±1.0	0.2±1.2	0.8±0.7	0.1±0.2
	GR-17.1 A2	0.2±0.9	0.3±0.9	-0.6±1.6	0.7±0.7	0.2±0.2
	GR-17.1 A3	0.1±0.6	0.1±0.5	-0.4±0.8	0.5±0.3	0.1±0.1
	GR-10 Guide	-0.9±3.2	-0.6±0.4	-2.3±2.7	1.9±1.6	1.5±7.0

TABLE 2 Summary of analysis of deviance for ΔE_{00} and ΔTP_{00} values related to resin (R), treatment (T) and their interactions (R × T).

	Effect	Likelihood Ratio χ^2	dF	P
ΔE_{00}	R	60.88	3	3.81×10^{-11}
	T	982.51	4	$<2.2 \times 10^{-16}$
	R × T	73.79	12	6.20×10^{-11}
ΔTP_{00}	R	0.62	3	0.89
	T	23.18	4	1.17×10^{-4}
	R × T	20.16	12	0.06

no known reports have investigated filled resins for this purpose. The GR-17.1 resin was chosen for this study based on its current marketed application for 3D-printed provisional restorations for dental crowns, which have similar requirements to orthodontic brackets, including dimensional accuracy, bond strength, torsional strength, initial shade matching, and colour stability. The GR-10 Guide resin, which is marketed for fabrication of surgical guides, was chosen as a control to evaluate the effects of exogenous staining and accelerated aging on a 3D-printed resin with no filler or predefined shade.

Prior to assessment of colour stability, a practitioner must know if the initial colour of the brackets is acceptable to the patient. A 2006 study by Cho et al. found considerable variation in the range of colour of natural teeth, both between patients and between different

colour measurement devices.³² The overall range found for L' was 39.0–83.2, for C' was 0.4–29.9, and for h' was 57.8–90.0. By this standard, the initial values of the resins used in this study presented acceptable lightness and chroma values for at least some patients within the range. Hue values were slightly out of range. However, qualitative analysis of the water treated samples in [Figure 1](#) shows that the resin colours are very similar to the expected colour of natural teeth, and brackets made from these resins would thus likely be initially accepted by patients.

In order to put the magnitude of colour differences given in [Table 1](#) in context, it is necessary to determine the minimum threshold of colour difference an observer is able to perceive, and the threshold above which an observer finds a colour difference clinically unacceptable. A multicentre study published in 2015 determined the 50:50 perceptibility threshold (PT) and 50:50 acceptability threshold (AT) for tooth coloured shades – the points at which 50% of participants were able to perceive a change in colour between two samples, and at which 50% of participants found a colour difference acceptable, respectively. The PT was found to be $\Delta E_{00}=0.8$, and AT was found to be $\Delta E_{00}=1.8$.²⁰ Similar testing has been conducted on the translucency parameter to determine the 50:50 translucency perceptibility threshold (TPT) and 50:50 acceptability threshold (TAT), which are $\Delta TP_{00}=0.6$ and $\Delta TP_{00}=2.6$, respectively.³³

Staining from exogenous sources produced aesthetically unacceptable levels of colour change in all resins used in this study. Red wine and coffee were the two most potent staining agents. Red wine effected a colour change (ΔE_{00}) ranging from 14.2 ± 1.5 to 18.2 ± 2.0

FIGURE 1 Representative photographs of the 3D-printed resin samples after treatment.

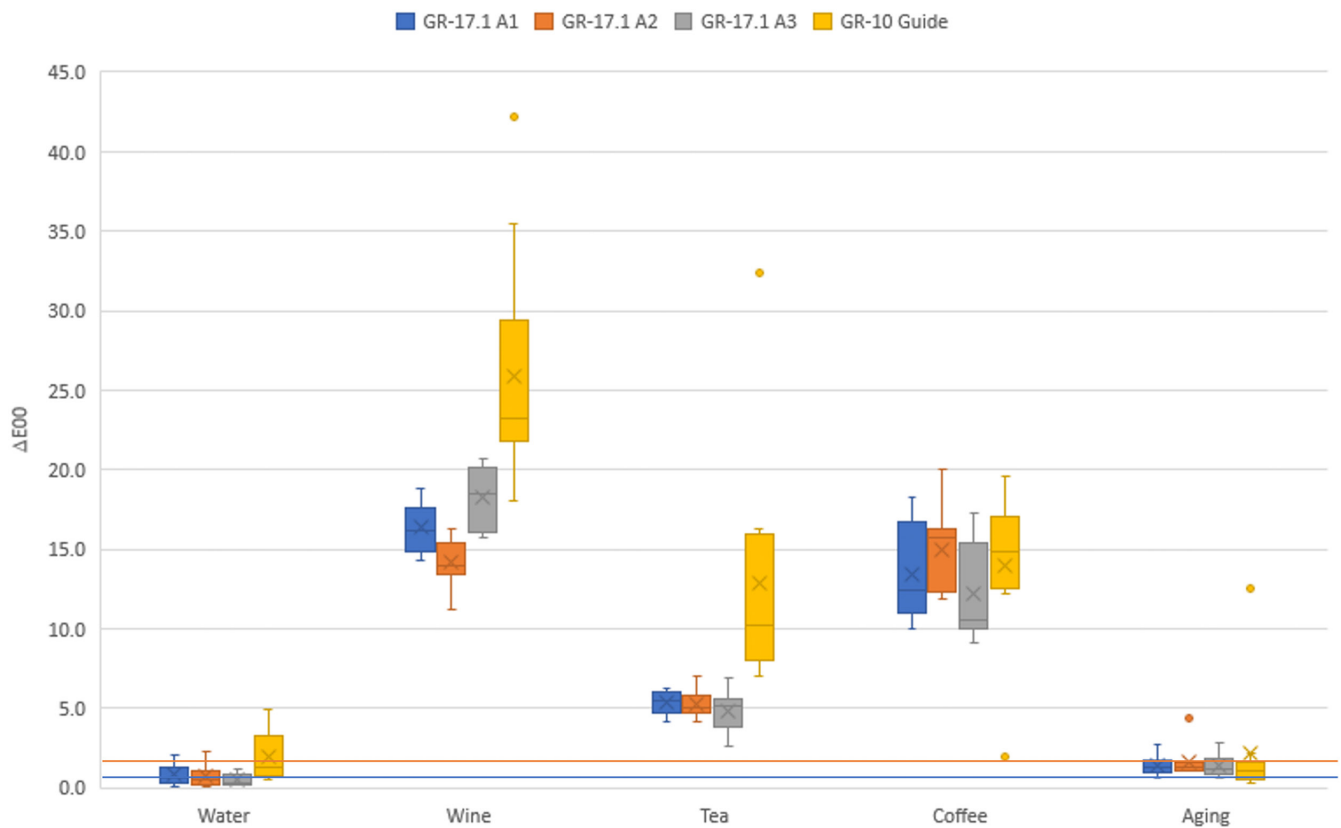
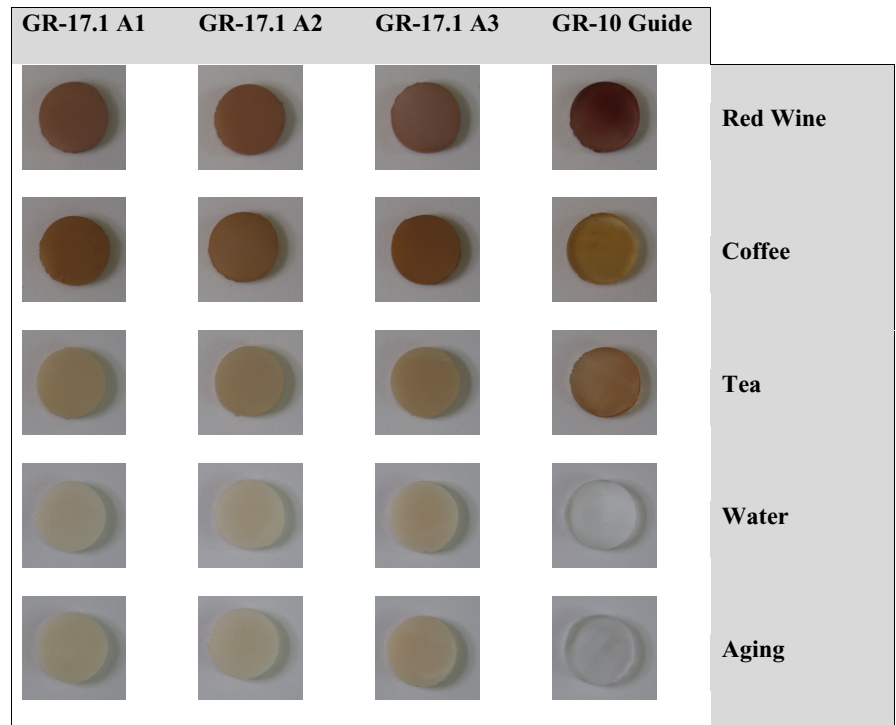


FIGURE 2 Box and whisker plots showing the distribution of colour change (ΔE_{00}) observed for each 3D-printed resin material under experimental conditions. Blue line indicates the 50:50 perceptibility threshold (PT), and the orange line indicates the 50:50 acceptability threshold (AT). Whiskers indicate the range of data, with the box illustrating the first and third quartiles, the line indicating the median, the X indicating the mean, and dots representing outliers.

for GR-17.1 resins, and 25.9 ± 7.5 for GR-10 Guide, and coffee caused ΔE_{00} of 12.2 ± 2.9 to 15.0 ± 2.6 for GR-17.1 resins, and 13.9 ± 4.9 for GR-10 Guide resin. The mean value for the smallest colour difference

after immersion in either red wine or coffee, shade A3 GR-17.1 resin following immersion in coffee, was still six times greater than the acceptability threshold. This is in line with previous studies evaluating

colour stability of dental materials, which have found coffee and red wine to produce the greatest difference in colour.²¹ Compared to a previous study applying similar treatment protocols, the 3D-printed filled resins showed a similar degree of staining from red wine, and significantly more staining from coffee immersion compared to 3D-printed unfilled resins.³¹ However, caution should be taken in directly comparing these materials, as the matrix composition, resin manufacturer, and post-print processing protocols also differed. For exogenous staining treatments tested, tea caused the smallest colour difference, but was still greater than the AT for all materials.

Endogenous staining, produced via accelerated aging in accordance with ISO Standard 4892-2, caused less colour change than exogenous staining, with ΔE_{00} ranging from 1.4 ± 0.6 to 1.6 ± 1.0 for GR-17.1 resins and 2.1 ± 3.7 for the GR-10 Guide resin. The mean ΔE_{00} of all filled resins was below AT but above PT (Figure 2). For the unfilled GR-10 Guide resin, ΔE_{00} was above PT. Yellowing of dental resins with a bisphenol A-glycidyl methacrylate matrix has been shown to be a result of oxidation of residual carbon-carbon double bonds in an incompletely cured polymer. Despite the different matrix composition for 3D-printed resins, the effect appears consistent that ultraviolet light-induced aging increases yellowness of resins.²² Further, as would be expected from studies showing a decrease in this yellowing if filler content is increased, all materials in the present study underwent less endogenous staining than exogenous.²³

Changes in translucency parameter (ΔTP_{00}) for all specimens immersed in coffee were above the TPT, but below the TAT (Figure 3). Red wine caused clinically unacceptable levels of ΔTP_{00} for GR-17.1 shade A2 and GR-10 Guide, and perceivable, but clinically acceptable ΔTP_{00} for remaining groups. Tea caused a ΔTP_{00} that was below TPT for shade A1 GR-17.1, and above TPT but below TAT for all other groups. Accelerated aging did not cause a ΔTP_{00} greater than TPT for any GR-17.1 resin, and was below TAT for GR-10 Guide resin. Interestingly, both the accelerated aging and control groups underwent an increase in the translucency parameter, indicating that the material became more translucent over the course of the experiment. A possible mechanism for this effect is that the fully cured polymer is more translucent than its monomeric building blocks, and that the resins cured over the course of the experiment, enhanced by the ultraviolet light exposure in the accelerated aging group.

No statistically significant difference in colour stability was observed between shades A1, A2, or A3 of GR-17.1 resin. The specific compositional difference between these resins is proprietary, but given that the manufacturer only reports a single safety data sheet for all GR-17.1 resins as a class,²⁴ it can be inferred that the materials are very similar. Thus, the lack of specific material effects between shades of GR-17.1 resins ($P = .94$) is reasonable.

While the colour changes of all materials tested were found to exceed acceptable limits for aesthetics based on previously

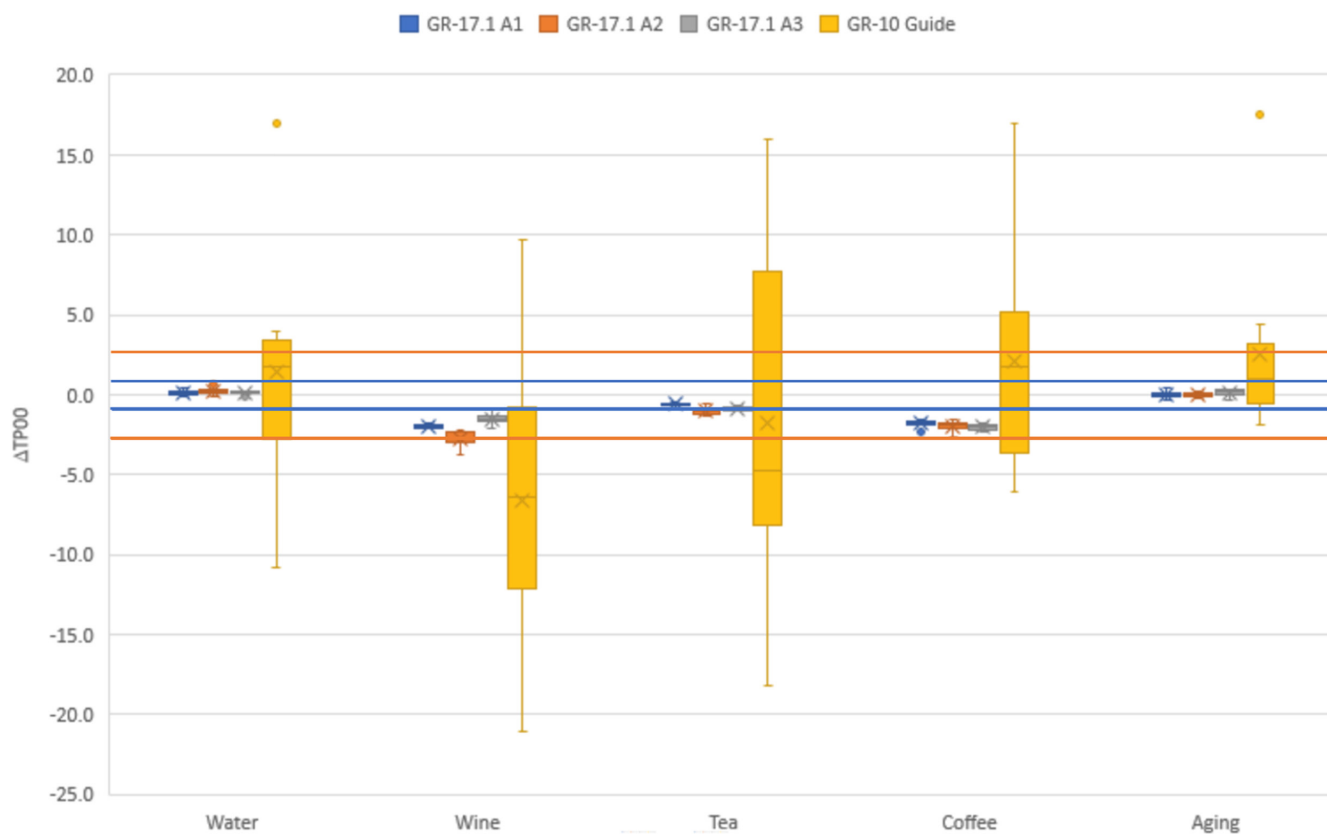


FIGURE 3 Box and whisker plots showing the distribution of change in translucency parameter (ΔTP_{00}) observed for each 3D-printed resin material under experimental conditions. Blue lines indicate the translucency perceptibility threshold (TPT), and the orange lines indicate the 50:50 translucency acceptability threshold (TAT). Whiskers indicate the range of data, with the box illustrating the first and third quartiles, the line indicating the median, the X indicating the mean, and dots representing outliers.

established thresholds, it is important to note that these thresholds were developed for use in restorative dentistry, not orthodontics.^{20,34} Further studies are needed to determine whether patients interested in orthodontic treatment with aesthetic brackets are similarly discriminating about colour difference. While perceptibility threshold is likely to be the same for these patients as it is for the general population, the acceptability threshold may be higher, given that patients interested in undergoing fixed orthodontics are already tolerating the aesthetic compromise of a metal wire. Further, this study was conducted in vitro, and further investigation is required to determine the colour stability of aesthetic bracket materials in the oral cavity. Despite the laboratory setting of the investigation, clinically relevant conclusions can still be drawn, as the methods used are based on standardized protocols used in determination of colour stability of a variety of dental materials.^{17,21,35}

5 | CONCLUSION

Filled 3D-printed resins showed significant changes in colour and translucency upon staining and accelerated aging. There were significant differences in the level of colour change both between resins and treatment conditions. Coffee and red wine caused the greatest change in colour, followed by tea, followed by accelerated aging. The colour changes observed with these materials under the conditions investigated present potential implications on the suitability of these resins for aesthetic orthodontic bracket applications.

AUTHOR CONTRIBUTION

Richard Wallach: investigation, validation, data curation, formal analysis, writing – original draft; Jeryl D. English: writing – review and editing, project administration; Audrey Moon: writing – original draft, review, editing, and visualization; Ralph A. Brock II: writing, original draft, review, editing, and visualization; Rade D. Paravina: methodology, formal analysis, resources, writing – original draft, review, editing, and visualization; F. Kurtis Kasper: conceptualization, methodology, resources, formal analysis, writing – review and editing, funding acquisition.

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CONFLICT OF INTEREST

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Effect of Material and Pad Abrasion on Shear Bond Strength of 3D-Printed Orthodontic Brackets

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Abstract

Objective: To investigate the effect of printing material and air abrasion of bracket pads on the shear bond strength of 3D-printed plastic orthodontic brackets when bonded to the enamel of extracted human teeth.

Materials and Methods: Premolar brackets were 3D-printed using the design of a commercially available plastic bracket in two biocompatible resins: Dental LT Resin and Dental SG Resin ($n=40/\text{material}$). 3D-printed brackets and commercially manufactured plastic brackets were divided into two groups ($n=20/\text{group}$), one of which was air abraded. All brackets were bonded to extracted human premolars, and shear bond strength tests were performed. The failure types of each sample were classified using a 5-category modified adhesive remnant index (ARI) scoring system.

Results: Bracket material and bracket pad surface treatment presented statistically significant effects for shear bond strengths, and a significant interaction effect between bracket material and bracket pad surface treatment was observed. The non-air abraded (NAA) SG group ($8.87 \pm 0.64 \text{ MPa}$) had a statistically significantly lower shear bond strength than the air abraded (AA) SG group ($12.09 \pm 1.23 \text{ MPa}$). In the manufactured brackets and LT Resin groups, the NAA and AA groups were not statistically significantly different within each resin. A significant effect of bracket material and bracket pad surface treatment on ARI score was observed, but no significant interaction effect between bracket material and pad treatment was found.

Conclusion: 3D-printed orthodontic brackets presented clinically sufficient shear bond strengths both with and without AA prior to bonding. The effect of bracket pad AA on shear bond strength depends on the bracket material.

1 | INTRODUCTION

3D-printing has disrupted the orthodontic space, allowing for in-house fabrication of many orthodontic supplies that have traditionally been available only from manufacturers. The technology has progressed such that 3D-printing brackets in orthodontic offices is rapidly approaching reality, with several case reports published

in the literature indicating that 3D-printed brackets can be bonded to the dentition and function to align the teeth.^{1,2} Companies are taking 3D-printed bracket fabrication a step further and offering custom, 3D-printed brackets designed to fit a patient's dentition.^{3,4}

With the increasing prevalence of 3D-printers in orthodontic practices, it is crucial that clinicians have a thorough understanding of the different variables affecting the quality of their 3D-printed

brackets and subsequent bonding to enamel surfaces. Some of these variables include the type of printer, printing medium, post-print processing methods and bonding protocol, among others.⁵⁻⁷ Bonding an orthodontic appliance to enamel requires the success of three components: the attachment base, the tooth surface and its preparation and the bonding material itself. The attachment base of metal brackets is designed to create mechanical interlock between the base and the bonding material, as stainless steel typically does not chemically bond with orthodontic adhesives. This mechanical interlock generally increases as the bondable surface area of the attachment base increases,⁸ which is why a metal mesh is often braised onto metal bracket pads following bracket fabrication.⁹

With the introduction of more diverse bracket materials as well as 3D-printing, the bracket bonding mechanism to the enamel surface becomes more complex and may be diversified beyond pure mechanical interlock of pad to adhesive. For example, acrylic-based 3D-printed brackets may form chemical bonds with bracket adhesives via residual carbon-carbon double bonds in the acrylic with the composite. In addition, in 3D-printed brackets, the 3D-printing process leads to a stair-stepping effect on smooth sloped edges whereby the XY resolution of the printer and print layer thickness create roughness on printed surfaces designed to be smooth.⁷ This surface roughness on a printed bracket pad may aid bonding via mechanical retention due to increased surface area. It has been demonstrated that air abrading restorative surfaces roughens the surfaces,¹⁰ and air abrasion of the enamel surface of a tooth increases the shear bond strength of metal brackets bonded to the teeth.¹¹ However, little research has been reported examining the effect of air abrasion of plastic bracket pads on shear bond strengths, especially in the context of 3D-printed brackets.

This study seeks to investigate the effect of printing material and the effect of air abrasion of bracket pads on the shear bond strength of 3D-printed plastic orthodontic brackets when bonded to the enamel of extracted human teeth. It was hypothesized that the shear bond strength of 3D-printed orthodontic brackets would not differ significantly from that of corresponding commercially manufactured plastic brackets 24h after bonding. Further, it was hypothesized that air abrasion of bracket pads would not affect shear bond strength of 3D-printed orthodontic brackets bonded to enamel.

2 | MATERIALS AND METHODS

2.1 | 3D-printing orthodontic brackets

A single stereolithography format file (STL) of an American Orthodontics (AO) Silkon Plus™ universal premolar bracket (002-959M, American Orthodontics, Sheboygan, WI, USA) was utilized in this study. Eighty copies of this bracket were 3D-printed using a desktop stereolithography printer (Form 2, Formlabs, Inc., Somerville, MA, USA), with 40 brackets printed in each of 2 commercially available 3D-printing resins: Dental LT Resin (LT; $n=40$) and Dental SG Resin (SG; $n=40$) (Formlabs, Inc., Somerville, CA,

USA). The rationale for selecting these resins was twofold: previous research demonstrated clinically sufficient shear bond strengths with these resins when used to print a metal bracket file from AO,¹² and these two resins are marketed as being biocompatible and approved for intraoral use.^{13,14} The SG and LT brackets were printed in 1 print job per resin type set up using Preform Software (Version 3.12.1, Formlabs, Inc., Somerville, MA). The brackets were printed with a layer height of 100µm.^{5,7} Following removal from the build platform, all 3D-printed brackets were post-print processed according to the manufacturer's instructions,^{15,16} including washing in an isopropyl alcohol (IPA, >99%) ultrasonic bath for 2min, followed by a second ultrasonic bath for 3min. Brief exposure to compressed air accelerated drying of the brackets. Following at least 30min of additional air drying, the brackets were polymerized in a curing unit (Form Cure, Formlabs, Inc., Somerville, MA) for 20min at 80°C and 30min at 60°C for the LT and SG resins, respectively. All brackets were stored in a dark cabinet in the lab prior to bonding. In addition, 40 traditionally manufactured premolar brackets (Silkon Plus™) were obtained from AO.

2.2 | Bracket pad treatment

The 40 brackets 3D-printed in each resin and the 40 commercially manufactured brackets were divided into 2 groups ($n=20$ /group), and 1 of the 2 groups of each type of bracket was air abraded with a dental intraoral sandblasting machine (Microetcher™ II, Danville Materials, San Ramon, CA, USA) using 50µm aluminium oxide (TruEtch™ Aluminium Oxide 50 Micron White, Ortho Technology, Inc., Tampa, FL, USA) at 80psi. Air abrasion was completed by holding the wings of each bracket with anterior bracket placing forceps such that the bracket pad surface was 10mm from the Microetcher™ II nozzle. A single operator air abraded in a back-and-forth motion for 2s per bracket pad to facilitate equal distribution of aluminium oxide abrasion across the bracket pad surface.

2.3 | Bonding brackets to extracted premolars

A schematic overview of the study workflow appears in Figure 1. One hundred and twenty de-identified, extracted human premolars were stored in 0.05% sodium azide solution until use. The study was determined by the Institutional Review Board of The University of Texas Health Science Center at Houston to qualify as non-human subjects research (HSC-DB-18-0253). The premolars were divided into 6 groups ($n=20$), with maxillary and mandibular premolars divided equally between groups. Premolars with clinically visible caries, decalcification, fractures, fluorosis, enamel defects, anomalous crown morphology, and/or restorations were excluded from the study.

The 120 premolars were mounted in a 1:1 mixture of powder: liquid acrylic resin (SampleKwik Powder and Liquid Fast Cure Acrylic; Buehler, Lake Bluff, IL, USA) using mounting cups (SamplKups™,

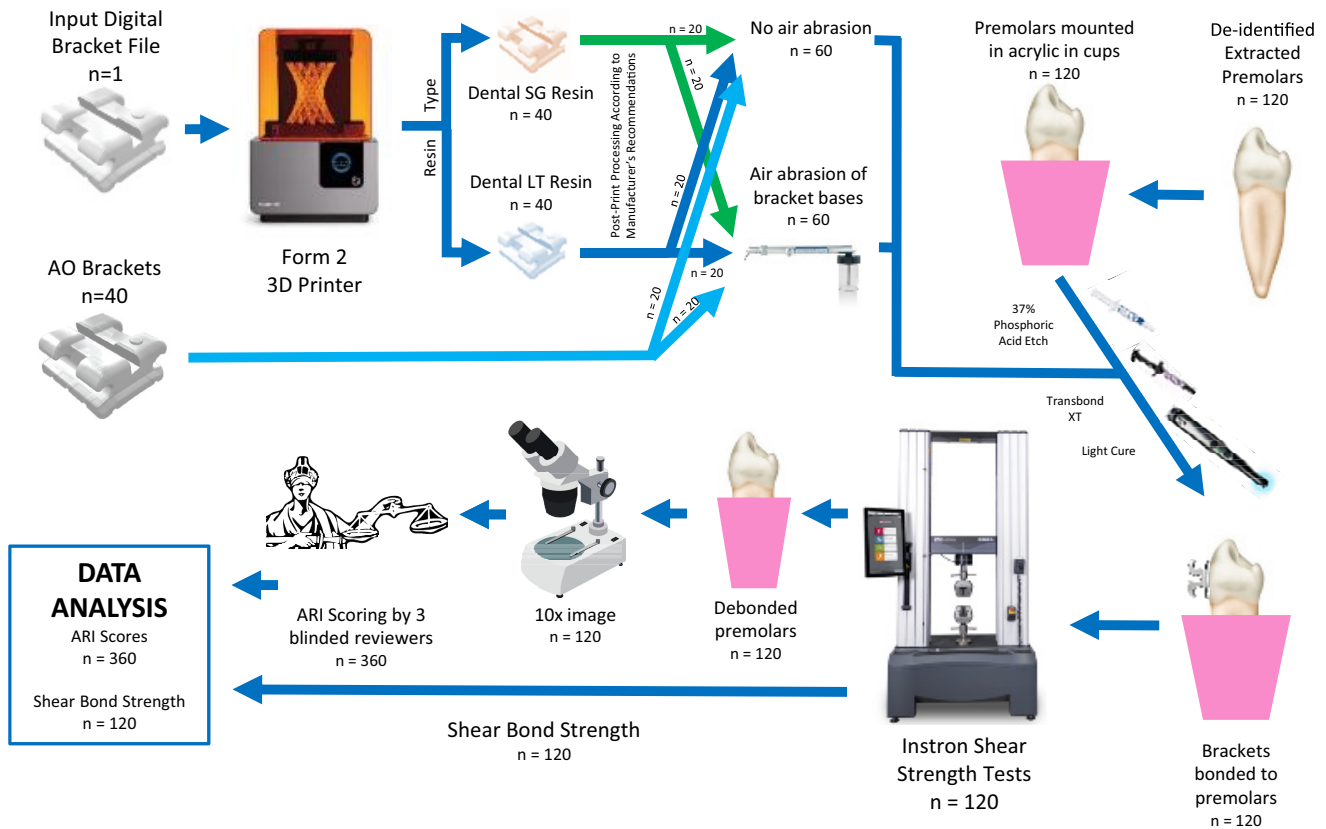


FIGURE 1 Schematic overview of the study design and workflow.

#20-9178, Buehler, Lake Bluff, IL, USA), such that the crown remained exposed and vertically oriented.¹⁷ Each exposed labial tooth surface was cleaned with a moist prophy head (Non-Latex Disposable Prophy Angle, #754031, Prophy Perfect Inc., Osseo, WI, USA) and prophy paste (Ortho Technology Prophy Paste, #15486, Ortho Technology®, Lutz, FL, USA) for 5 s, rinsed with water for 5 s and air dried completely. Next, teeth were etched with 37% phosphoric acid (Gel Etching Agent, #18-EGSS, Lot #171205, Reliance Orthodontic Products Inc., Itasca, IL, USA) for 15 s, rinsed with water for 10 s and air dried with compressed air for 10 s. Self-etching primer was applied to the labial surface of each tooth (Transbond™ Plus Self-Etching Primer, Lot #7353898, 3M™, Saint Paul, MN, USA), followed by gently air drying with compressed air from a dental unit. A thin coat of plastic conditioner (Plastic Conditioner, Reliance Orthodontic Products Inc.) was applied with a microbrush to each bracket pad surface of the 3D-printed LT and SG brackets and air dried. A single operator held each bracket with anterior bracket placing forceps to place light-cure adhesive paste (Transbond™ XT Adhesive Paste, Lot #N942962, 3M Unitek Dental Products, Monrovia, CA, USA) on each bracket pad surface and subsequently place the bracket on the central portion of the labial crown surface of each of the mounted premolars. An elastic remover (Elastic Remover and Square Band Pusher, #0158-B, Ortho-pli, Philadelphia, PA, USA) was used to push each bracket onto the enamel surface with at least 300 g of force and to remove excess adhesive expelled

around the sides of the bracket pad. The adhesive was cured for 3 s through the bracket per the manufacturer's instructions¹⁸ using a blue LED curing unit (Ortholux™ Luminous Curing Unit, 3M™, Saint Paul, MN, USA).

2.4 | Shear bond strength testing

Less than 24 h after bonding, shear bond strength tests were performed on all samples using a universal testing machine (Model #4465, Instron®, Norwood, MA, USA) with previously established software parameters for shear bond strength testing (Bluehill® 2 Software, Instron®).¹² Each mounted tooth was held vertically (occlusal surface facing up) by its acrylic mounting cup. Prior to each test, the tooth was positioned such that the shearing attachment of the testing machine was just above the bracket vertically, but not touching the bracket. Horizontally, the shearing attachment was placed as close to the bracket base as possible, with the aim for the force of the testing machine to be applied to the bracket base, rather than on the wings of the bracket. Next, each bracket was debonded via the shearing attachment of the testing machine, which descended vertically at a crosshead speed of 0.5 mm/min until the bracket debonded from the tooth. The software automatically recorded the highest force experienced by the crosshead during testing. The resultant shear bond strength of each bracket was

calculated by dividing the maximum force from the shear testing by the surface area of the bracket pad as previously described.^{19,20}

2.5 | ARI scoring

Following shear bond strength testing, each tooth was photographed (Digital Sight DS-Fi2, Nikon, Tokyo, Japan) under 10× magnification with a reflected light microscope (SMZ800, Nikon) to visualize the quantity of adhesive paste and/or bracket fragments remaining on each tooth. A 5-category modified adhesive remnant index (ARI) scoring method was utilized in concordance with previous research^{12,21,22} to guide analysis of the bond failure mode and associated implications on enamel integrity. An ARI score of 1 indicated that 100% of adhesive was left on the tooth with a bracket base impression visible. ARI scores of 2, 3, and 4 indicated that ≥90%, 10–90%, and ≤10% of adhesive was left on the tooth, respectively. An ARI score of 5 indicated that no adhesive was left on the enamel surface. Three reviewers scored each sample blindly and independently. In cases of differing ARI scores by the reviewers, a consensus score was developed. The actual tooth samples were also kept on hand for reviewers to reference when desired.

Three samples were not included in data analysis following testing. One sample was removed from analysis due to errors in shear bond strength testing, and two samples were removed from analysis due to crown enamel fracture during the shear testing, rather than debonding of the bracket from the enamel surface.

2.6 | Statistical analysis

All analyses were performed using R statistical software (R Core Team 2018, R Foundation for Statistical Computing, Vienna, Austria). A Generalized Linear Model using the *glm* function with Gamma specified error distribution was applied to evaluate effects of bracket material and air abrasion on shear bond strength, since

data were not normally distributed. The POLR function was used to examine effects of bracket and air abrasion on ARI, as ARI is an ordered categorical variable.

3 | RESULTS

3.1 | Shear bond strength

The shear bond strengths of each of the 6 bracket groups are presented in Figure 2. Bracket material and bracket pad surface treatment presented statistically significant effects on shear bond strength ($p=0.004$ and $p=0.005$, respectively), and a significant interaction effect between bracket material and bracket pad surface treatment was observed ($p<0.001$). The untreated (NAA) SG group (8.87 ± 0.64 MPa) had a statistically significantly lower shear bond strength than the air abraded (AA) SG group (12.09 ± 1.23 MPa) ($p<0.05$). In the manufactured brackets and LT Resin groups, the NAA and AA groups were not statistically significantly different within each resin group ($p>0.05$).

3.2 | ARI scores

Figure 3 presents the ARI scores for each of the 6 groups. Of note, none of the samples in the study received an ARI score of 4 or 5, meaning that for all samples at least 10% of adhesive remained on the tooth surface following bracket debonding. A significant effect of bracket material and bracket pad surface treatment on ARI score was observed ($p<0.001$ and $p<0.001$, respectively), but no significant interaction effect between bracket material and bracket pad surface was found ($p=0.067$). Within each bracket material, the group with air abrasion surface treatment demonstrated ARI scores of 3 with greater frequency than the group with no surface treatment. The traditionally manufactured brackets demonstrated low ARI scores with greater frequencies than the 3D-printed brackets.

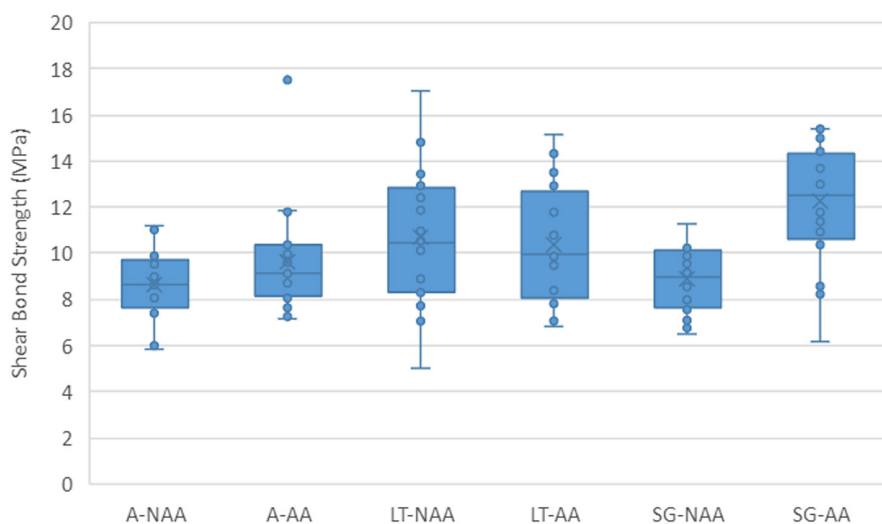
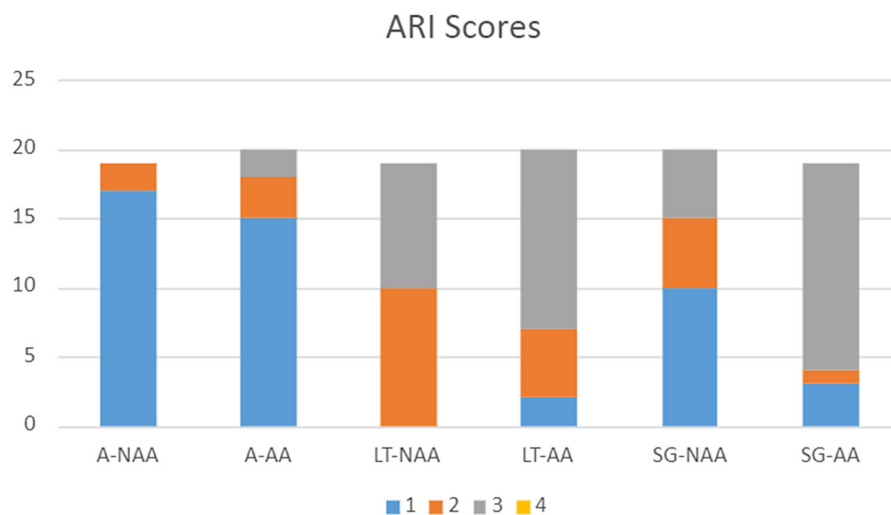


FIGURE 2 Box and whisker plot of shear bond strength values of each bracket material both with and without surface treatment. A, Manufactured bracket; AA, Air Abraded; LT, Dental LT Resin; NAA, Not Air Abraded; SG, Surgical Guide Resin.

FIGURE 3 Frequency of each adhesive remnant index (ARI) score for the test groups. A, Manufactured bracket; AA, Air Abraded; LT, Dental LT Resin; NAA, Not Air Abraded; SG, Surgical Guide Resin.



4 | DISCUSSION

4.1 | Shear bond strength

It could be an easy assumption to make that higher shear bond strengths are always more desirable in orthodontics, because brackets need to remain bonded to patients' teeth during daily speech, chewing and parafunctional activity. However, if bracket bond strengths are too high, they can surpass the strength of the tooth and cause tooth fracturing during the debonding process at the end of treatment, as has been reported in the literature.^{23,24} The issue is multifactorial, as there are many variables affecting the debonding of brackets and the interaction with the enamel surface, including the etching and bonding agents utilized, bracket material, bracket base design and tooth anatomy. Even the method of debonding may have an impact,²⁵ so the desired shear bond strength in orthodontics may be comparable to the fairy tale of Goldilocks and the Three Bears: not too high or enamel fracture could result, not too low or brackets will debond prior to the desired time, but just right in the middle is the ideal bond strength. Where is this ideal middle?

A minimum range for the shear bond strength of brackets according to Reynolds is 5.9–7.9 MPa,²⁶ and this range has been widely accepted in the literature. Gange recommended that adhesives withstand forces in the range of 20 MPa,²⁷ and biting force research by Proffit supports Gange's statement.²⁸ Guan et al.²⁹ reported significantly lower shear bond strengths (3–6 MPa) of plastic brackets compared to metal brackets. Applying Reynolds' widely accepted range of 5.9–7.9 MPa to the present study, as in previous research,¹² all groups demonstrated clinically sufficient shear bond strengths.

Metal brackets usually adhere to tooth enamel via mechanical retention with adhesive paste, while the adhesive chemically bonds with the enamel surface following either traditional etching of the enamel or self-etching primer application.^{8,30–32} Typically, this mechanical retention results from the penetration of adhesive into a metal mesh that is braised onto the metal bracket pad following

bracket fabrication.⁹ Plastic brackets cannot be braised with a mesh in a similar fashion, but manufacturers still desire similar levels of mechanical retention in their plastic bracket designs, so plastic bracket pads often incorporate protrusions and/or canals as retentive features.³³ The pad of the Silkron Plus™ bracket examined in this study presents a built-in grid pattern and retentive canals, which are referred to by AO on their website as, 'a reliable mechanical lock base.'³⁴ The surface area of the bracket pad was measured to be 16.56 mm² using the STL bracket file from AO and Geomagic Control 3D metrology comparison software (3D Systems, Inc., Rock Hill, SC, USA) using previously reported methods.^{12,35} For comparison, the surface area of the AO .018 twin master series maxillary 1st premolar bracket (#390–0060, American Orthodontics, Sheboygan, WI, USA) examined in a previous study¹² was 11.71 mm². This difference in surface areas between the two brackets likely reflects the Silkron Plus™ bracket's built-in grid pattern compared to the smooth surface of the metal bracket STL file previously examined.

The formula for the calculation of shear stress is $\tau = F / A$, where τ is shear stress, F is the applied shear force, and A is the cross-sectional area to which the force is applied. For this study, shear bond strength was defined as the maximum shear stress experienced by each bracket during debonding from its enamel surface. Since the definition of a shear force is one which is parallel to the material's cross-section, the goal was for the entirety of the force of the universal testing machine chisel to be applied to the bracket base as a shear force (Figure 4). This goal, however, was lofty, as any angulation incisogingivally of the bracket during bonding or displacement of the shearing attachment to touch a bracket wing could have resulted in a portion of the testing machine force being applied in a non-perpendicular fashion to the bracket pad, resulting in the shear force experienced by the bracket being less than the measured applied force (Figure 4). This phenomenon would likely result in higher maximum shear stress measurements than are truly reflective of the shear bond strength of the brackets. Addressing these limitations of the test method could be an area of exploration for future studies. For this study, the protocol of past lab studies was utilized,¹² with as much care taken to maximize the shear force component of the force as possible.

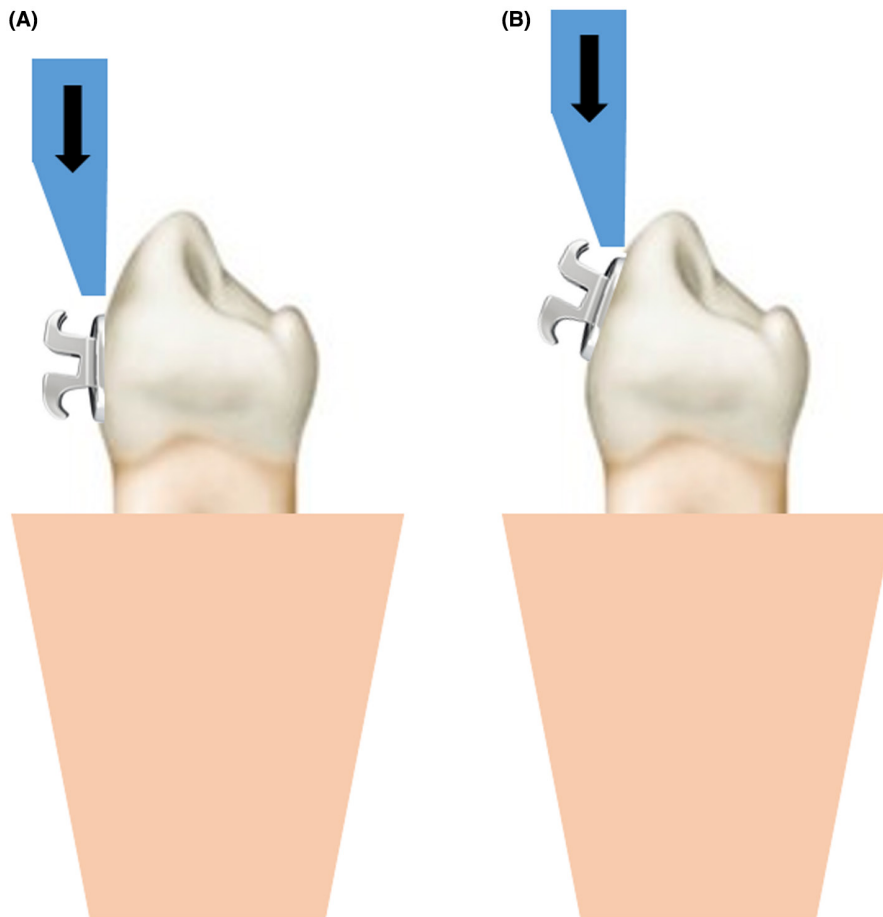


FIGURE 4 (A) Ideal vertical positioning of bracket during shear bond strength testing. (B) Non-ideal vertical positioning of bracket, leading to loss of crosshead force into the tooth rather than directed towards shearing of the bracket.

4.2 | ARI results

As a complement to the shear bond strength data, the ARI scores quantify the sites of bond failure of the various bracket groups. All of the ARI scores in this study ranged from 1–3, indicating that in every case at least 10% of the adhesive remained on the tooth following bracket debonding.

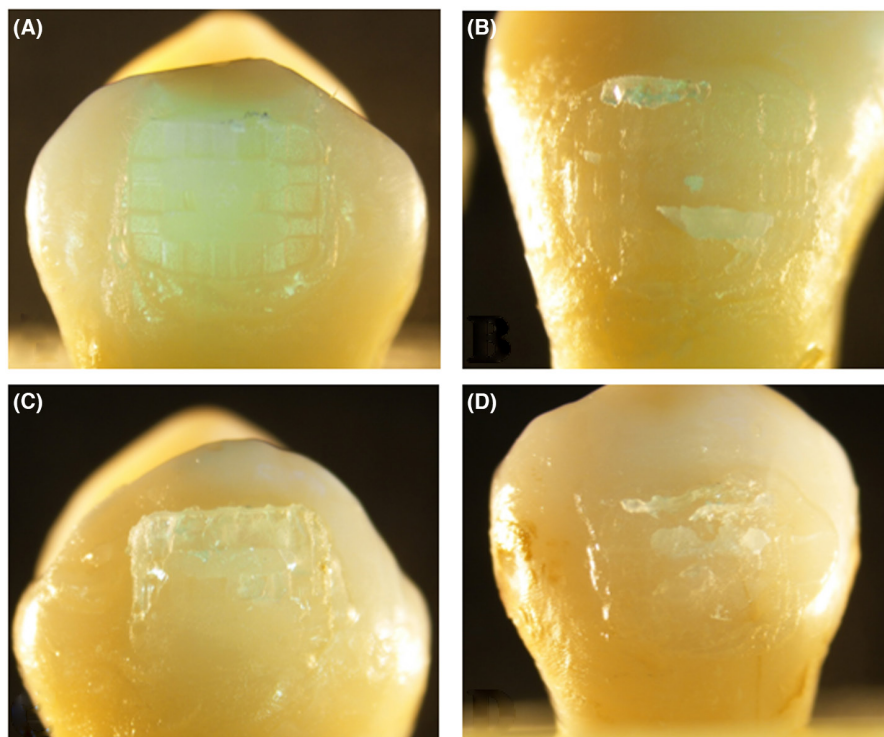
There are five possible locations where failure can occur during a bracket debonding procedure: failure within the enamel, failure between the enamel and bonding agent, failure within the bonding agent, failure between the bonding agent and the bracket and failure within the bracket. According to Proffit, bonded brackets should be removed in a method that avoids damaging the enamel, by fracturing either within resin bonding material or between the bracket and the resin, and subsequently removing remaining bonding material from the enamel surface.³⁶ Proffit did not include failure between the enamel and resin bonding material as a desirable option for debonding, and indeed, a review of ceramic brackets by Bishara suggests that an increased incidence of enamel fractures may be associated with failure at the enamel-adhesive interface.³⁷ Upon the introduction of ceramic brackets, the previously rare occurrences of fracturing enamel or fracturing brackets during debonding became much more common.²⁴ Enamel fracture is undesirable for obvious reasons, and when ceramic bracket fragments remain on the teeth

and require removal with a diamond bur and handpiece, it is considered an undesirable complication due to increased chair time and possible damage to the enamel.³⁶

The ARI scoring system utilized in this study did not enable quantification of enamel fracture or when fragments of bracket were left on the bonding surface due to failure of the bracket. The presence of small portions of bracket material adherent to the bonding surface was a common feature throughout the photographed teeth, but it was very hard to visually detect bracket versus bonding material during indexing, as demonstrated in Figure 5B–D. Previous case reports have also cited issues with brackets fragmenting rather than debonding,² therefore, continued research could attempt to develop a modification to the ARI index scoring method to include indices for bracket fragments remaining.

Fragments of bracket remaining on the enamel surface in this study indicated that the internal strength of the bracket material was less than the bond strength between the adhesive and the bracket, as well as less than the bond strength between the adhesive and the enamel. Intra-bracket failure is not one of the two debonding methods suggested by Proffit,³⁶ so practitioners who wish to print and utilize brackets as described in this study should keep this potential complication in mind. However, this complication may be outweighed by the potential benefits of 3D-printing plastic brackets including customization, aesthetics and

FIGURE 5 Representative photographs of enamel surface following bracket debonding. (A) Resin bonding agent remaining on enamel. (B–D) Resin bonding agent and bracket fragments remaining on enamel.



lower inventory requirements. In general, the large quantity of ARI 1 scores in the manufactured bracket group indicates that these brackets broke cleanly off of the teeth, whereas the 3D-printed groups' lower number of ARI 1 scores indicates higher complexity in the debonding process and potentially more bracket fragments remaining on the teeth.

Within each bracket material, the group with air abrasion surface treatment demonstrated ARI scores of 3 with greater frequency than the group with no surface treatment, indicating that 10%–90% of the adhesive remained on the tooth. Previous studies found that air abrasion increases the surface roughness of restorative materials,¹⁰ so one possible explanation for the increased adhesive being removed from the enamel surface at debonding is that the air abrasion increased the surface area of the abraded pad surface and, therefore, increased the bond strength between pad and adhesive. Since the present study did not quantify the effect of air abrasion on bracket pad surface area, this could be an avenue of future investigation. In this study, three diverse materials were air abraded, and the same trend of increased ARI scores in the air abraded group was consistent across all materials, suggesting that the air abrasion affected each material. Since there was no quantification of intra-bracket fracture in this study, it is difficult to determine whether the pad/adhesive bond is getting stronger leading to an increased ARI score, or the increased ARI score is due to decreased internal bracket strength, leading to bracket fracture. While the ARI scores indicate that air abrasion makes a difference in the bonding of plastic brackets to enamel, the lack of statistical significance in the shear bond strengths of the Silkon™ and LT groups suggest that only the SG group supports air abrasion to have a statistically significant impact on shear bond strength.

4.3 | Limitations

It is worth noting that following printing and post-print processing per the manufacturer's instructions, the SG, LT and AO manufactured Silkon Plus™ brackets appeared to be slightly different sizes. This was despite the fact that the exact same bracket file was utilized in 3D-printing all of the brackets, and the file reflects the Silkon Plus™ bracket design. For the purposes of this study, we assumed that all brackets had the same surface area. While not quantified in the present study, the potential differences in bracket dimensions call into question the dimensional accuracy of the 3D-printed brackets. Additionally, while maxillary and mandibular premolars were evenly distributed between groups in the study, potential mismatch between the curvature of the bracket pad and the specific tooth anatomy could confound the results of shear bond strength testing. Although beyond the scope of the present study, characterization of 3D-printed bracket slot accuracy, friction and strength could be the subject of a future investigation, given the importance of slot properties on bracket prescriptions and resultant tooth movements. Other bracket characteristics beyond shear bond strength are important for clinicians to consider when choosing their bracket fabrication modality or designing their 3D-print settings for optimal clinical performance, including dimensional accuracy, durability, slot friction and colour stability.³⁸

5 | CONCLUSIONS

3D-printed orthodontic brackets based on a Silkon Plus™ design printed in resins approved for intraoral use (SG and LT) presented

clinically sufficient shear bond strengths both with and without air abrasion of the bracket pad prior to bonding. The effect of bracket pad air abrasion on shear bond strength depends on the bracket material. If clinicians are utilizing SG resin to print brackets, air abrading the bracket pad bases prior to bonding could help increase shear bond strengths.

AUTHOR CONTRIBUTIONS

MSH involved in investigation, validation, data curation, formal analysis, writing the original draft and editing. JCO involved in methodology, writing the original draft, review, editing and visualization. JDE involved in review and editing and project administration. JOW involved in writing the original draft, review, editing and visualization. BEC involved in writing the original draft, review, editing and visualization. DAH involved in methodology, writing the original draft, review, editing and visualization. FKK involved in conceptualization, methodology, resources, formal analysis, review and editing and funding acquisition.

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CONFLICT OF INTEREST STATEMENT

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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