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Assessment of effect of accelerated aging on interim fixed dental materials using digital technologies

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PURPOSE. This study assessed the physical and mechanical properties of interim crown materials fabricated using various digital techniques after accelerated aging. **MATERIALS AND METHODS.** Three groups of interim dental restorative materials (N = 20) were tested. The first group (CO) was fabricated using a conventional manual method. The second group (ML) was prepared from prefabricated resin blocks for the milling method and cut into specimen sizes using a cutting disc. The third group (3D) was additively manufactured using a digital light-processing (DLP) 3D printer. Aging acceleration treatments using toothbrushing and thermocycling simulators were applied to half of the specimens corresponding to three years of usage in the oral environment (N = 10). Surface roughness (Ra), Vickers microhardness, 3-point bending, sorption, and solubility tests were performed. A 2-way analysis of variance (ANOVA) and Fisher's multiple comparison test were used to compare the results among the groups. **RESULTS.** The mean surface roughness (Ra) of the resin after accelerated aging was significantly higher in the CO and ML groups than that before aging, but not in the 3D group. All groups showed reduced hardness after accelerated aging. The flexural strength values were highest in the 3D group, followed by the ML and CO groups after accelerated aging. Accelerated aging significantly reduced water sorption in the ML group. **CONCLUSION.** According to the tested material and 3D printer type, both 3D-printed and milled interim restoration resins showed higher flexural strength and modulus, and lower surface roughness than those prepared by the conventional method after accelerated aging. [J Adv Prosthodont 2022;14:360-8]

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INTRODUCTION

Interim, provisional, or temporary crowns are used during fixed prosthetic treatment to protect the prepared natural tooth and oral tissue from damage and restore esthetics and function.[1-3] Interim crowns are designed to be used for a short period between tooth preparation and fitting of the definitive dental crown.[3,4] However, interim crowns are sometimes used for a long period because of their acceptable aesthetics, functionality, and cost-effectiveness, especially if they are prepared with high-quality materials and precise marginal fit.[5,6]

Interim crowns can be fabricated directly on a prepared tooth or indirectly outside the mouth using a cast obtained from a dental impression.[3,6] The indirect procedure is safer, more comfortable for patients, and provides better-quality crowns than the direct procedure.[3,6] However, it increases the chair-side time because of the time required for manual fabrication in a dental laboratory. Interim crowns are commonly fabricated using self-cured polymethyl methacrylate (PMMA) resins because of their ease of fabrication, reliability and cost-effectiveness.[4,5] Insufficient mechanical properties, such as fractures, are common causes of interim restoration failures.[1]

Digital technologies, such as computer-aided design and computer-aided manufacturing (CAD-CAM) systems, have become increasingly popular for manufacturing interim crowns.[7-10] Digital technologies can rapidly process any 3D object with high quality and accuracy and reduce errors associated with manual fabrication.[8,9] CAD-CAM interim restorations can be fabricated by subtractive (milling) or additive (3D printing) manufacturing methods.[1,8] The subtractive method manufactured a designed object by grinding a block or disc of resin material into the desired shape.[3,9] These resin blocks undergo a high degree of conversion polymerization and are processed under pressure and heat, resulting in reduced porosity, which improves the strength and accuracy of the interim crown.[3] The high cost and wastage of milling devices, burs, and restorative materials as well as the limited motion range of the device to produce complex shapes are the main drawbacks of subtractive methods.[2]

The additive method processes a designed object in a layer-by-layer pattern.[3,10] This method is currently available for processing interim restorations using different types of technologies, such as stereolithography (SLA), digital light projection (DLP), and photopolymer jetting (PolyJet).[11-13] Among these methods, DLP is the most promising technology because it consumes fewer materials and processes objects with a higher speed, quality, and precision than other technologies.[8,9,11] The DLP processes an object upside-down by displaying the entire image of one object layer on the surface of a photopolymer resin vat using ultraviolet (UV) light that solidifies liquid resin.[2,8,11] The building process is repeated for the subsequent layers until object completion.[11] 3D printing technologies can quickly process precise dental prostheses with high-quality and minimal materials.[9,11]

The materials and processing techniques can influence the mechanical and physical properties of fabricated interim restorations. Several studies have evaluated the accuracy and properties of interim crown materials processed using digital technologies, which have superior precision and internal fit compared to interim crowns made manually.[3,5,7,9,10] Although the mechanical, physical and surface properties[2,4,10-17] of digitally fabricated interim crowns are suitable for intraoral applications, these properties after accelerated aging, such as toothbrush abrasion[18-21] and thermocycling,[22-24] to the best of our knowledge, have not been investigated. The objective of this study was to evaluate the physical and mechanical properties of interim crown materials fabricated using various digital techniques after accelerated aging. The null hypothesis was that there are no differences in the mechanical and physical properties of digitally fabricated and conventional interim crown materials after accelerated aging.

MATERIALS AND METHODS

Three groups of interim crown materials were prepared using three different manufacturing techniques: CO, conventional manual method; ML, CAD-CAM milling method; and 3D, CAD-CAM 3D printing method. Twenty rectangular specimens (2 × 2 × 25 mm) for each group were fabricated according to the

ISO10477 standard for the 3-point bending test.[3,7] The chemical compositions of the interim crown materials provided by the manufacturers are listed in Table 1.

The specimens for the CO group were fabricated using a self-cured interim material (Bosworth Trim Plus; Bosworth, Skokie, IL, USA) and polymerized according to the manufacturer's instructions in a custom mold duplicated from 3D printed sample using duplicate silicon materials (Adisil; Siladent, Munich, Germany). For the ML group, specimens were prepared from prefabricated resin blocks (Ceramill temp; Amann Girrbach AG, Koblach, Austria) and cut into specimens using a cutting disc (IsoMet 5000 Linear Precision Saw; Buehler Ltd., Lake Bluff, IL, USA). The 3D group specimens were designed using CAD software (FreeCAD v.18) and saved in the standard stereolithography language (STL) format. The file was then transferred to an in-office digital light projection (DLP) 3D printer (NextDent 5100; 3D Systems, Soesterberg, The Netherlands) for printing using a photopolymer (Crown & Bridge NextDent®; 3D Systems, Soesterberg, The Netherlands) with a layer thickness of 50 µm and a printed angle of 0°. The supporting structures were removed from the printed specimens before cleaning with isopropyl alcohol. Finally, a post-curing unit (LC-3D Print Box; 3D Systems, Soesterberg, The Netherlands) was used for post-processing polymerization for 30 min according to the manufacturer's instructions.

All specimens were polished with the aid of custom-designed polishing holders that were 3D printed (M200; Zortrax SA, Olsztyn, Poland) with filament material (Z-ABS; Zortrax SA, Olsztyn, Poland). One side of each specimen from each group was ground and polished using a polishing machine (EcoMet/AutoMet 250; Buehler, Lake Bluff, IL, USA) under water cooling using three types of silicon carbide papers (800, 1000, and 1500 grit), followed by a final polishing cloth with polishing paste (Abraso-Starglanz asg; Bredent, Senden, Germany). This polishing procedure is similar to that used for interim restorations in dental laboratories.

Ten specimens from each group (CO, ML, and 3D) were subjected to accelerated aging using a toothbrushing simulation followed by a thermocycling process. Accelerated aging simulates the mechanical wear and hydrothermal cycle in the oral environment in accordance with the ISO11405 standard.[22,25] First, a toothbrushing simulator device (ZM 3; SD Mechatronik GMBH, Feldkirchen Westerham, Germany) equipped with 12 removable brush heads was used. Six soft toothbrushes (Oral-B Classic Care 40 M; Procter & Gamble, Surrey, UK) were fixed at brushing stations. The brushing slurry was prepared with toothpaste (Signal; Unilever, London, UK) and distilled water at a ratio of 1:2 and supplied every 5000 cycles. The specimens were fixed horizontally on station holders using custom-designed 3D-printed connectors. A brushing speed of 30 sec/min under a vertical load of 200 g and with a 10 mm cycle movement was set for 27500 strokes that corresponded to 3 years of toothbrushing time.[19] Then, a thermocycler (Huber 1100; SD Mechatronik GmbH, Feldkirchen-Westerham, Germany) equipped with cold and hot baths (5 and 55°C, respectively) at alternating temperatures with an immersion time of 15 sec. Thermocycling was performed for 3500 cycles to estimate

Table 1. Chemical composition of interim crown materials provided by the manufacturers of conventional (CO), milling (ML), and 3D printing (3D) groups

Group	Chemical composition	Manufacturers
Conventional (CO)	powder - Polymethylmethacrylate (PMMA) and Benzoyl peroxide liquid - Methylmethacrylate (MMA) and N, N-Dimethylp-toluidine	Bosworth Company, Skokie, IL, USA
Milling (ML)	Polymethylmethacrylate (PMMA) and cross-linked polymers based on methacrylic acid esters, colorants, dibenzoyl peroxide, and Methylmethacrylate (MMA)	Amann Girrbach AG, Koblach, Austria
3D printing (3D)	Methacrylic oligomers, methacrylate monomer, phosphine oxides, pigment	Nextdent, Soesterburg, The Netherlands

nearly three years of usage in the oral environment.[22]

The surface roughness of the specimens for each group (N = 10) was measured using a non-contact optical profilometer (Contour GT; Bruker, Tuscon, AZ, USA) operated with the principle of vertical scan interferometry. Three individual measurements at different locations were recorded for each specimen (N = 5) with a threshold of 4%, length of 90 µm, speed of × 2, and VSL measurement type. The mean Ra of the 15 measured values was calculated in micrometers (µm).

The microhardness of all groups was measured using a Vickers microhardness indenter (Nova 130; Innovatest Europe BV, Maastricht, The Netherlands) under a 50 g indentation load with a 10 sec dwell time. Three specimens from each group were selected randomly and indented five times at different points, and the mean microhardness values were calculated from images captured by a built-in camera at the indentation site.[14]

Three-point bending tests were conducted at room temperature using an Instron Universal Testing Machine (Instron Corp., Canton, MA, USA) with a 500 N load cell at a constant speed of 1 mm/min. Each specimen (N = 10) from each group was placed on two supporting pins 18 mm apart. Force-deflection curves for each test were obtained using Bluehill software (v.2; Instron Corp., Norwood, MA, USA). The flexural strength (F) and modulus (E) were calculated using the following equations:[14] $F = 3 F_{max} L / 2 b d^2$, and $E = F_y L^3 / 4 \delta b d^3$, where F_{max} is the maximum applied force, F_y is the yield force, L is the distance between the supports, b is the width of the tested specimen, d is the height of the tested specimen, and δ is the deflection of the tested specimen.

For the water sorption and solubility measurements, cubic specimens (2 × 2 × 2 mm) were prepared for each group (N = 10), and the specimen dimensions were measured using a digital caliper. First, the specimens were placed in a desiccator for 24 h at 23 ± 1°C and their weights were measured (m_0). The specimens were then placed in a tube containing 15 ml of distilled water and placed in an incubator at 37 ± 1°C for 24 h. The specimens were then removed from the water, swabbed, weighed, and returned to the water (m_1). Finally, the specimens were removed from the water, dried in a desiccator at 23 ± 1°C for

24 h, and weighed for the final time (m_2). Water sorption (A), water solubility (S), and density (D) were calculated using the following equations:[24] $A = m_1 - m_2 / V$, $S = m_0 - m_2 / V$, and $D = mass / V$, where m_0 is the specimen weight before immersion, m_1 is the specimen weight after immersion, m_2 is the specimen weight after immersion and desiccation, V is the specimen volume, and D is the specimen density.

Qualitative surface roughness and microstructure analyses were performed on gold-sputtered replicas of the specimens using a field-emission scanning electron microscope (SEM) (JSM-5900 LV; JEOL, Tokyo, Japan) at an operating voltage of 10 kV. The aged and fractured areas of the specimens were observed at 50 and 200 µm magnifications at the end of the aging acceleration and flexural tests.[18]

The sample size was calculated using G*Power software (v.3.01; Kiel, Germany) based on a pilot study comparing two means with 80% power and an alpha of .05, with an estimated effect size of .52.[17] Statistical analyses of the surface roughness, microhardness, mechanical test, and sorption data were performed using the Origin software (v.9.0; Origin Lab, Northampton, MA, USA). Means and standard deviations (SD) were calculated, and the data were checked for normality using a histogram. A 2-way analysis of variance (ANOVA) and Fisher's multiple comparison test were used to test for statistical differences among the CO, ML, and 3D groups. The significance level was set at $P < .05$.

RESULTS

The mean and standard deviations of surface roughness (Ra ± SD) and surface profile images of conventional (CO), milled (ML), and 3D printed (3D) interim crowns before (Pre) and after (Post) toothbrushing simulations are presented in Figure 1 and Figure 2. The mean Ra values of the CO and ML groups after toothbrushing simulation were significantly ($P < .05$) higher (0.52 ± 0.22 and 0.27 ± 0.12 µm, respectively) than the Ra before toothbrushing simulation (0.40 ± 0.20 and 0.14 ± 0.04 µm). Whereas, the mean Ra of the 3D group after the toothbrushing simulation (0.15 ± 0.04 µm) was not statistically different ($P = .09$) than before toothbrushing simulation (0.24 ±
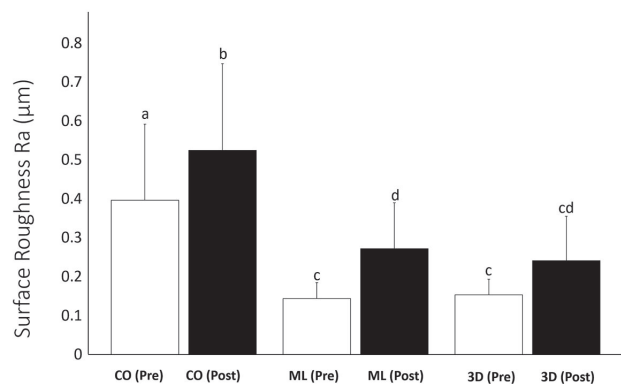
Fig. 1. Chart showing the surface roughness (Ra) of the interim crown materials before (Pre) and after (Post) brushing simulation in the conventional (CO), milled (ML), and 3D printed (3D) groups. The same letter indicates no significant differences between the groups.
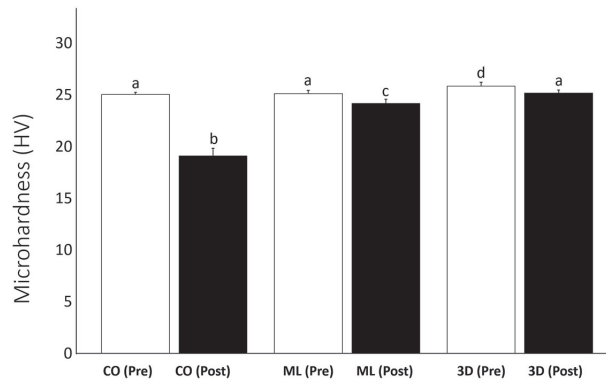

Fig. 3. Chart showing the microhardness values (HV) of the interim crown materials before (Pre) and after (Post) accelerated aging process of conventional (CO), milled (ML), and 3D printed (3D) groups. The same letter indicates no significant differences between the groups.
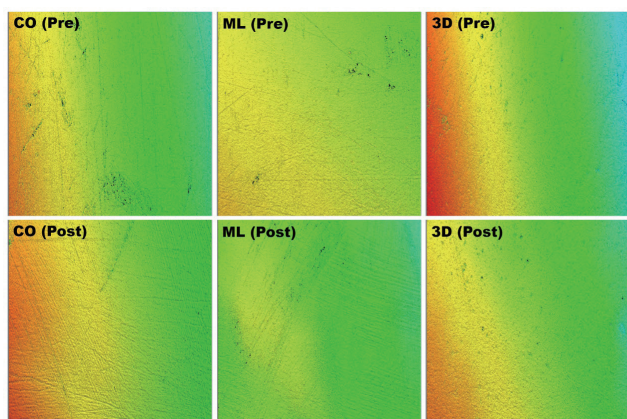

Fig. 2. Surface profile images of conventional (CO), milled (ML), and 3D printed (3D) interim crown materials before (Pre) and after (Post) brushing simulation. The color variation corresponds to the surface depth.

0.11 μm). Both the ML and 3D groups exhibited lower Ra values ($P < .05$) than the CO group before and after the toothbrushing simulation.

The results of the microhardness tests for all the groups are shown in Figure 3. All groups showed reduced microhardness values ($P < .01$) after accelerated aging. Before accelerated aging, the microhardness values of the CO (25.0 HV) and ML (25.1 HV) groups were lower ($P < .01$) than that of the 3D (25.9 HV) group. After accelerated aging, microhardness values were significantly different ($P < .01$) among the

CO (19.1 HV), ML (24.2 HV), and 3D (25.2 HV) groups.

The mean and standard deviation values of the flexural strengths and moduli of the CO, ML, and 3D groups are shown in Figure 4 and Figure 5, respectively. The flexural strength values before and after accelerated aging for the 3D group (151 ± 7 and 114 ± 8 MPa, respectively) were significantly higher ($P < .05$) than those for the CO (84 ± 14 and 76 ± 12 MPa, respectively) and ML (128 ± 12 and 94 ± 19 MPa, respectively) groups. The flexural strength of the ML group was higher ($P < .001$) than that of the CO group before accelerated aging. However, the ML and 3D groups exhibited reduced ($P < .001$) flexural strength values after aging, but not in the CO group ($P = .20$). The flexural moduli of the ML and 3D groups (3440 ± 232 and 3603 ± 305 MPa, respectively) were higher (P < .05) than that of the CO group (2288±471 MPa) before accelerated aging. After aging, the 3D group (3677 ± 270 MPa) was significantly higher ($P < .01$) than the CO (914 ± 266 MPa) and ML (2390 ± 624 MPa) groups. Only the ML group exhibited reduced ($P < .001$) flexural modulus values after accelerated aging.

The mean and standard deviation results for the water sorption, water solubility, and water density are shown in Table 2. There were no variations in the water sorption among the different groups under the same conditions before and after accelerated aging.
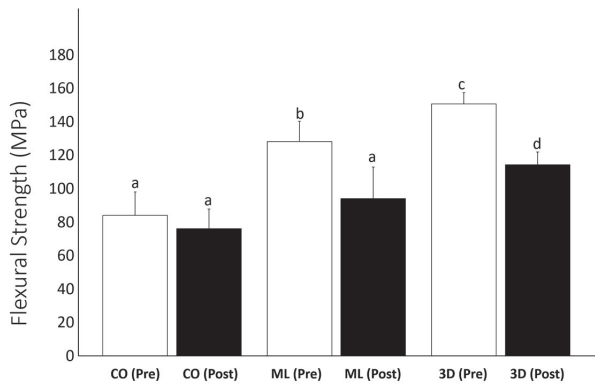
Fig. 4. Charts showing the flexural strength (MPa) of different interim crown materials on conventional (CO), milled (ML), and 3D printed (3D) groups before (Pre) and after (Post) aging processes. The same letter indicates no significant differences between the groups.

Fig. 5. Charts showing the flexural modulus (MPa) of different interim crown materials on conventional (CO), milled (ML), and 3D printed (3D) groups before (Pre) and after (Post) aging processes. The same letter indicates no significant differences between the groups.
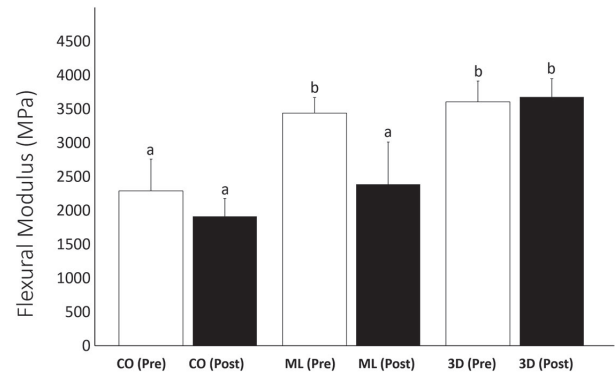
Table 2. Water sorption and solubility of CO, ML, and 3D groups of different interim crown materials before and after accelerated aging

Property	Aging effect	CO	ML	3D
Sorption ($\mu g/mm^3$)	No	44.8 ± 18.1 [a]	45.0 ± 22.3 [a,*]	45.3 ± 15.7 [a]
	Yes	38.0 ± 16.8 [a]	26.2 ± 23.6 [a,*]	37.1 ± 13.2 [a]
Solubility ($\mu g/mm^3$)	No	33.1 ± 13.7 b [a]	17.7 ± 16.6 [b]	22.1 ± 10.3 [a]
Density (mg/mm^3)	No	1.1 ± .1 [a]	1.1 ± .1 [a]	1.2 ± .1 [b]

The same superscript in the row indicates no significant difference between the groups ($P < .05$).
* indicates a significant difference between columns under the same property ($P < .05$).
CO, conventional; ML, milled; 3D, 3D printed.

The water sorption values before and after accelerated aging were significant ($P = .049$) only for the ML group. The water solubility in the CO group before accelerated aging was significantly different from that in the ML group ($P = .038$).

DISCUSSION

The null hypothesis was rejected based on the statistical analysis of the results, indicating that interim crown materials fabricated using digital technologies would exhibit superior mechanical and physical properties after accelerated aging compared to the conventional method. 3D-printed interim restorations exhibit acceptable mechanical and physical properties, with excellent accuracy and marginal fit, making them a suitable option for long-term dental crowns.[7,8,10,13,15,23,25] To the best of the author's knowledge, there is a lack of scientific data to evaluate the effect of accelerated aging on the mechanical and physical properties of 3D-printed interim restoration materials compared with other fabrication methods. This study is clinically important for evaluating restoration longevity and its effect on oral hygiene, as the surface roughness and hardness of dental resins play crucial roles in plaque accumulation and periodontal health condition.[4,9,22]

In the oral environment, interim restorations are exposed to thermal changes, repeated toothbrushing, and occlusal loads.[25] The aging of resin-based materials causes matrix softening degradation and deformation, resulting in crack initiation and growth in porous resin areas that can affect the mechanical and physical properties of dental materials.[4,25] Material prop-

erties, such as polymerization shrinkage, thermal expansion, and water sorption, can change. Thermocycling is a testing technique for simulating natural aging process of dental materials by exposing them to recurrent temperature exposures.[22] Also, toothbrushing simulation is considered an established model in the literature that causes abrasions on the surface caused by applied brushing forces.[18,21]

In this study, the higher surface roughness (Ra) value found in the CO group than in the ML and 3D groups might be related to the fabrication method.[2] The conventional method of fabricating interim restorations involves the preparation and polymerization of resins in an uncontrolled environment that often presents surface irregularities and defects.[18] While, the ML and 3D groups were polymerized in controlled environments in which ML was prepared by manufacturers and 3D was processed using 3D printing technology.[18] In addition, the surface smoothness of a resin is highly influenced by its inherent material composition including the initiator, pigment, accelerator, matrix, and inorganic fillers.[15,23] The increases in Ra of the CO and ML groups after the toothbrushing simulation were related to the brushing effects on the surface, as shown in Figure 2 and Figure 6. However, the brushing simulation did not significantly affect the Ra of the 3D group, which might be related to the higher microhardness of the 3D printed samples compared with the other groups.

The 3D group showed a significantly higher Vickers microhardness than the CO and ML groups. These findings are in agreement with those presented in other studies done by Al-Qahtani et al.[2] and Kim et al.[12] The fact that the 3D group material is formed out of cross-linked monomers and inorganic fillers, causing decreased polymerization shrinkage and increased abrasion resistance, could also explain the increased flexural strength of the 3D group.[2] The reduction in microhardness following thermocycling could be attributed to the physical properties of the resin affected by thermocycling, which causes water molecules to penetrate the resin, resulting in resin expansion, and degradation of the polymeric matrix.[16,22] There is a positive relationship between the hardness of a material and abrasiveness against natural teeth; however, the microhardness values of all groups were below the hardness of the enamel surface of the tooth.[14,15,17]

The flexural strengths and moduli of the 3D and ML groups were higher than those of the CO group, which may be due to the homogenous microstructure of the 3D printed and milled materials resulting from controlled polymerization process, as explained earlier. Conversely, the lower flexural strength and moduli of the CO group are probably related to defects on the surface during the auto-polymerization of the resin material.[2,5] Defects and open porosities can initiate cracks, thereby reducing the strength of a material.[20] The SEM images (Fig. 6) show an organized fracture

Fig. 6. SEM images of brushed and fractured surfaces of conventional (CO), milled (ML), and 3D printed (3D) groups of interim crown materials after (post) aging processes. The images show that the fracture surface (arrow) were smoother and more organized in the ML and 3D groups than in the CO group.
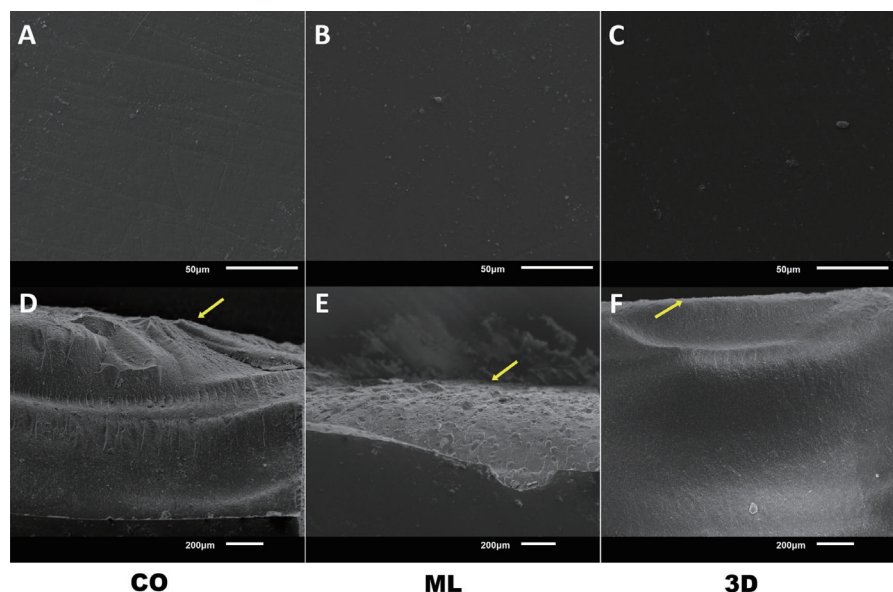
path for ML and 3D groups but not for CO group. The reduced flexural strength values after accelerated aging may be due to softening and degradation of the resin matrix.[4,16,22] However, the flexural strength values of the interim crown materials for all groups were considered suitable for interim restorations because they surpassed the minimum ISO10477 standard for flexural strength of 50 MPa.[7] The flexural modulus of the 3D groups after accelerated aging was not affected, which is an excellent characteristic for interim crowns to resist stress, occlusal load, and deformation during chewing.[25]

Acrylic resins tend to absorb water over time owing to the polarity and nature of the resin molecules.[22,24] This can cause resin softening and chemical degradation, creating internal stress and crack deformation over long periods of time.[24] This study found that thermocycling changed the water sorption of the ML resin but not that of the CO and 3D groups. This may be related to the chemical composition of the resin, in which the cross-linking agents and initiators in the ML group led to high water sorption.[24] In addition, the high solubility in the CO group was probably due to the polymerization technique because the low polymerization degrees of the CO resin resulted in unreacted monomers that could be dissolved in water.[22] The solubility of the resin after accelerated aging was not calculated because the loss of material was due to the brushing and thermocycling processes, which are considered confounding variables.

3D-printed resins are promising materials for clinical applications. The smoother surface of the 3D printed resin for interim restorations would be clinically beneficial, as less time is required for manufacturing the prostheses and less bacterial and plaque accumulation follow.[4,22] Additionally, strong and stiff materials would be valuable for longevity of treatment. The results of this study may influence the selection of the appropriate products for patient treatment. Therefore, 3D-printed interim restoration materials can exhibit superior mechanical and surface properties for long-term use in the clinical setting.

This study had some limitations because it was performed *in vitro* using flat specimens that did not fully mimic *in vivo* conditions. Moreover, the results were limited to a single type of 3D printed material from a single manufacturer, which may not be generalizable to the method. Future studies are needed to assess the color stability, microbial adhesion, and additional mechanical measurements of interim crown materials. In addition, with the findings of this study, further characteristic studies could be conducted on a variety of 3D printed interim crowns, as various manufacturers and technologies are currently available.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn: toothbrushing and thermocycling affected the surface roughness, microhardness, and flexural strength of the tested interim resin restorations, depending on the material and fabrication method. Despite the aging process, both 3D-printed and milled resins tend to have higher flexural strengths and moduli, and smoother surfaces than those prepared by the conventional method. According to the tested material and 3D printer type, 3D printed interim resin restorations showed higher flexural strength and modulus after accelerated aging than those of the milling and conventional groups.

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