Effect of Food-Simulating Liquids on the Mechanical Properties of 3D-Printed Provisional Restoration Materials

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Purpose: To evaluate the effect of food-simulating liquids (FSLs) on the mechanical properties of provisional restoration materials fabricated by 3D printing, milling, and traditional fabricating methods. ***Materials and Methods:*** The bar specimens were fabricated with traditional, milling, and 3D-printing methods according to ISO 10477 specifications. Each group of specimens was randomly subdivided into four groups to be immersed in various FSLs: distilled water (control group), n-heptane, 50% ethyl alcohol, and 0.02 mol/L citric acid for 7 days at room temperature (n = 19 per group). The Knoop hardness (KHN) was evaluated, and the specimens were subjected to a three-point bending (3PB) test to evaluate flexural strength (FS) and flexural modulus (FM). One-way ANOVA and Tukey tests were used to analyze the data. ***Results:*** Fabrication methods had a significant effect on the mechanical properties of the materials being tested. FSLs had no effect on the FS and FM of materials being tested. The 50% ethyl alcohol solution significantly decreased the hardness of traditional group specimens, and the n-heptane and 50% ethyl alcohol solutions increased the hardness of the 3D-printed specimens significantly ($P \leq .05$). Scanning electron microscopy (SEM) revealed that while traditional and milling group specimens showed a ductile fracture type, 3D-printed specimens showed a brittle fracture type. ***Conclusions:*** Production methods affected the mechanical properties of provisional restoration materials. Immersion in 50% ethyl alcohol solution decreased the KHN of the traditional specimens. FSLs had no negative effect on the mechanical properties of the milled and 3D-printed specimens. *Int J Prosthodont 2024;37(suppl):s71–s77. doi: 10.11607/ijp.8869*

Fixed provisional restorations are transitional prostheses that are used until the definitive restorations are delivered to the patient.[1] Although provisional restorations are used only temporarily, they should exhibit functional, biologic, and esthetic properties that are similar to those of definitive restorations. Although the expected clinical service time for provisional restorations is limited in most cases, prolonged clinical service may be indicated depending on the rehabilitation planning.[2–6] Thus, improved mechanical properties of provisional restorations are beneficial for the treatment process.[7]

Various materials and techniques have been described for fabricating provisional restorations.[5] Generally, they are fabricated chairside with different kinds of polyacrylate monomers and impression molds as a template.[5] With the introduction of digital dentistry to clinical practice, provisional restorations fabricated with CAD/CAM techniques have become popular.[6,8,9] There are two main CAD/CAM methods: milling (subtractive manufacturing) and 3D printing (additive manufacturing).[10] 3D printing

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Table 1 Food-Simulating Liquids

FSLs	Simulated foods
Distilled water	Saliva in the oral environment
N-heptane	Butter, fatty meats, and vegetable oils
0.02 mol/L citric acid	Vegetables, fruits, and acidic beverages
50% ethyl alcohol	Alcoholic beverages

of provisional restorations has been only recently introduced to clinical practice, and there are very limited data about the mechanical properties of restorations produced according to this technique.[11,12]

Food-simulating liquids (FSLs) are chemical solvents used by researchers to investigate the effects of food ingredients on various materials in in vitro studies.[13,14] These liquids are approved by the FDA and are listed in Table 1 according to the type of food they simulate. Food ingredients can play an especially significant role in the failure of polymer-based dental materials by causing them to age in the oral cavity.[15] The majority of the studies in the literature regarding the effects of FSLs on dental materials are about dental composites, and significant changes have been reported in terms of mechanical properties.[16–19] However, studies regarding the effects of FSLs on the mechanical properties of provisional restoration materials are scarce.[14,20] Yap et al[14] reported significantly lower Knoop hardness (KHN) values in test groups compared to the control group when various provisional restorative materials were exposed to FSLs. Akova et al[20] investigated the effects of FSLs on the mechanical properties of methyl methacrylate, ethyl methacrylate, and bis-acryl composite resin provisional restorative materials and reported that the mechanical properties of provisional restorative materials were strongly influenced by FSLs.

Although 3D-printed provisional restorations are becoming increasingly popular, there is no study in the dental literature about the effect of FSLs on their mechanical properties. Therefore, the purpose of this study was to investigate the effect of FSLs on the mechanical properties of 3D-printed provisional restoration material and compare them to the effects on specimens fabricated by milling and traditional methods. The null hypothesis is that FSLs have no effect on the microhardness (KHN), flexural strength (FS), or flexural modulus (FM) of provisional restoration materials.

MATERIALS AND METHODS

Bar-shaped test specimens were fabricated using three different manufacturing methods: traditional molding (TM), milling (MM), and 3D manufacturing (3DM). Each type of specimen was then exposed to three different FSLs: n-heptane, 50% ethyl alcohol, and 0.02 mol/L citric acid solutions, as well as distilled water as the control group. In accordance with the the power analysis (effect size = .4, α =.05, power = 0.8, number of groups = 12), a total of 228 test specimens were fabricated (n = 19 per group).

First, a digital bar satisfying ISO 10477 requirements (25 mm × 2 mm × 2 mm) was designed with a design software program (AutoCAD, Autodesk) in STL format. This design was used for fabricating the specimens in the MM and 3DM groups. MM group specimens were milled with a computer-controlled milling device (inLab MC X5, Dentsply Sirona) from a prefabricated polymethyl methacrylate (PMMA) disk (Tempo-CAD, On Dent). A light-sensitive liquid resin (Temporary CB resin, Formlabs) was used for fabricating specimens in the 3DM group with a 3D dental printer (Form 3, Formlabs) according to manufacturer instructions. The 3DM specimens were horizontally oriented on the built platform of the 3D printer and fabricated with a layer thickness of 50 µm. The printing direction of the layers was perpendicular to the applied force executed in the three-point bending (3PB) test procedure. A metal mold was fabricated for the TM group specimens with a fiber laser cutting machine (XT Laser, Jinan Xintian Technology). PMMA powder (Imident, Imicryl Dental) was measured on an electronic balance (Shimadzu AX120) and mixed with liquid according to the manufacturer's instructions. Then it was poured into the metal mold until polymerization was complete. No surface conditioning was executed on the specimens other than cutting off the supports in the 3DM and MM groups. All test specimens were inspected for surface irregularities and porosities with a magnifier.

Next, each group of specimens (TM, MM, and 3DM) was randomly separated into four subgroups for testing with the different FSLs (distilled water, n-heptane, 50% ethyl alcohol, and 0.02 mol/L citric acid). Thus, 12 groups were used in the study (n = 19 per group). Specimens were subjected to the FSLs for 7 days (Fig 1). After exposure, the specimens were washed under running water and air dried. A randomization chart was prepared with a free internet program to determine the test sequence.[21] Before the 3PB test, a KHN test (Buehler MMT-3) was performed. Each specimen was subjected to the KHN test three times, from both the end and center of the bar beam, with 100 gf and 15 seconds of dwell time. The mean of the three measurements was recorded as the KHN of that particular specimen. Following the KHN test, specimens were subjected to the 3PB test with a universal testing device (M270, Testometric) with a cross-head speed of 1 mm per minute until fracture (Fig 2). Care was taken to keep the KHN testing surface of the specimens facing upward during the 3PB test.

The FS (MPa) and FM (MPa) were calculated according to the following formulas, were L is the distance between the supports (mm), w is the width of the connector (mm), h is the thickness of the connector (mm),

d is the deflection due to the load applied at the middle of the beam (mm), and *P* is the peak load at the point of fracture (*N*)[22]:

$$FS = 3PL/2wh^2$$
$$FM = L^3P/4wh^3d$$

Following the 3BP test, one specimen from each group was selected randomly for scanning electron microscopy (SEM) fracture surface analysis. One-way ANOVA followed by Tukey test was used for statistical analysis ($\alpha = .05$) with statistical software (SPSS Statistics version 21.0, IBM).

Fig 1 Immersion of the specimens in the FSLs.

RESULTS

The mean and SD values for the hardness (KHN), FS, and FM of the test specimens are presented in Table 2. The FSLs had no effect on the KHN of the MM group specimens, 50% ethyl alcohol decreased the KHN of the TM group specimens ($P = .026$), and 50% ethyl alcohol ($P < .001$) and n-heptane ($P < .001$) increased the hardness of the 3DM specimens. The 3DM group in n-heptane solution had significantly lower FS values compared to the 3DM groups in the 50% ethyl alcohol ($P = .027$) and 0.02 mol/L citric acid ($P = .002$) solutions. TM group specimens in 50% ethyl alcohol had significantly lower FS values than the TM group specimens in the 0.02 mol/L citric acid solution ($P = .02$). The FSLs had no effect on the FM of the MM and 3DM group specimens. However, TM group specimens placed in 50% ethyl alcohol had significantly lower FM values than TM group specimens placed in the n-heptane solution ($P = .039$)

SEM graphs of the fracture surfaces of the test specimens are shown in Figs 3 to 5. It was seen that while TM and MM specimens showed a ductile type of fracture, 3DM specimens showed a brittle type of fracture. Fracture origins have been distinguished.

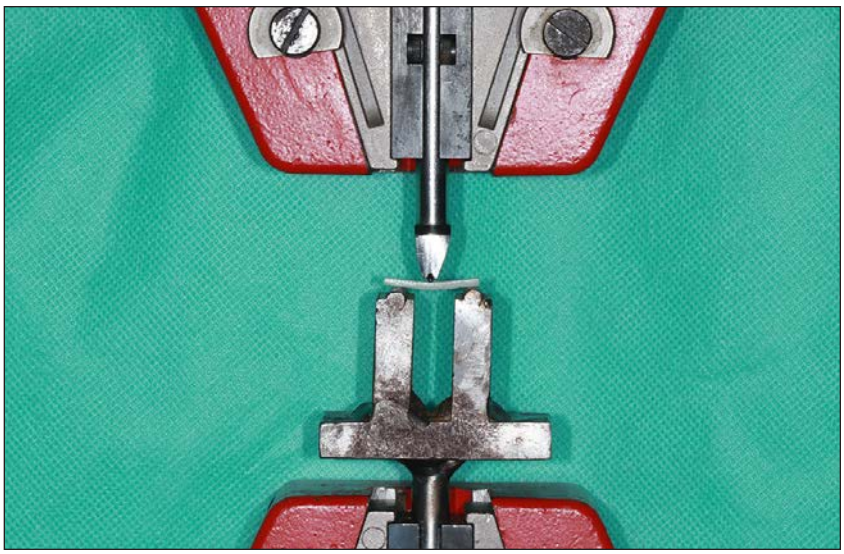

Fig 2 3PB test assembly.

DISCUSSION

In the current study, the mechanical properties of provisional materials fabricated via various production methods were evaluated after exposure to FSLs. According to the study results, the null hypothesis about microhardness (KHN) was rejected for the TM and 3DM groups.

Studies in the literature vary concerning the storage solution used for control group specimens.[9–14] In some studies, authors preferred to store control group specimens in dry air at room temperature, whereas other authors preferred to store the control specimens in distilled water. This is a critical decision with regard to the final results.[9,14] Because provisional restorations are constantly exposed to oral fluids in the mouth, the authors of the present study were convinced that air would be insufficient to simulate the conditions encountered in clinical practice.

Table 2 Values for KHN, FS, and FM

	Traditional	Milled	3D-printed
Microhardness (KHN)			
Distilled water	13 ± 1.2$^{A\text{-}a}$	19.1 ± 2$^{B\text{-}a}$	16.4 ± 4.6$^{C\text{-}a}$
N-heptane	12.9 ± 1.4$^{A\text{-}a,b}$	18.8 ± 3.1$^{B\text{-}a}$	24.5 ± 3.9$^{C\text{-}b}$
50% ethyl alcohol	11.8 ± 1$^{A\text{-}b}$	17.5 ± 2.2$^{B\text{-}a}$	23 ± 2.6$^{C\text{-}b}$
0.02 mol/L citric acid	13.2 ± 1$^{A\text{-}a}$	18 ± 2.7$^{B\text{-}a}$	17.7 ± 4.3$^{B\text{-}a}$
FS, MPa			
Distilled water	91.3 ± 11.8$^{A\text{-}a,b}$	118.7 ± 15.2$^{B\text{-}a}$	137.1 ± 15.7$^{C\text{-}a,b}$
N-heptane	92.1 ± 17.1$^{A\text{-}a,b}$	112.6 ± 11.7$^{B\text{-}a}$	127.7 ± 10.2$^{C\text{-}b}$
50% ethyl alcohol	81.3 ± 9.4$^{A\text{-}a}$	115.3 ± 12.4$^{B\text{-}a}$	139.7 ± 7.4$^{C\text{-}a}$
0.02 mol/L citric acid	93.7 ± 11.3$^{A\text{-}b}$	114.3 ± 9.4$^{B\text{-}a}$	143.5 ± 15.8$^{C\text{-}a}$
FM, MPa			
Distilled water	2502 ± 271.5$^{A\text{-}a,b}$	2472 ± 240.8$^{A\text{-}a}$	4162 ± 396.1$^{B\text{-}a}$
N-heptane	2701 ± 612.9$^{A\text{-}a}$	2544 ± 741.2$^{A\text{-}a}$	4268 ± 449.5$^{B\text{-}a}$
50% ethyl alcohol	2326 ± 445.1$^{A\text{-}b}$	2412 ± 278.7$^{A\text{-}a}$	4218 ± 307.4$^{B\text{-}a}$
0.02 mol/L citric acid	2601 ± 269.4$^{A\text{-}a,b}$	2415 ± 211.1$^{A\text{-}a}$	4425 ± 317$^{B\text{-}a}$

Data are presented as mean ± SD. Different uppercase letters represent a statistically significant difference in the same row. Different lowercase letters represent a statistically significant difference in the same column.

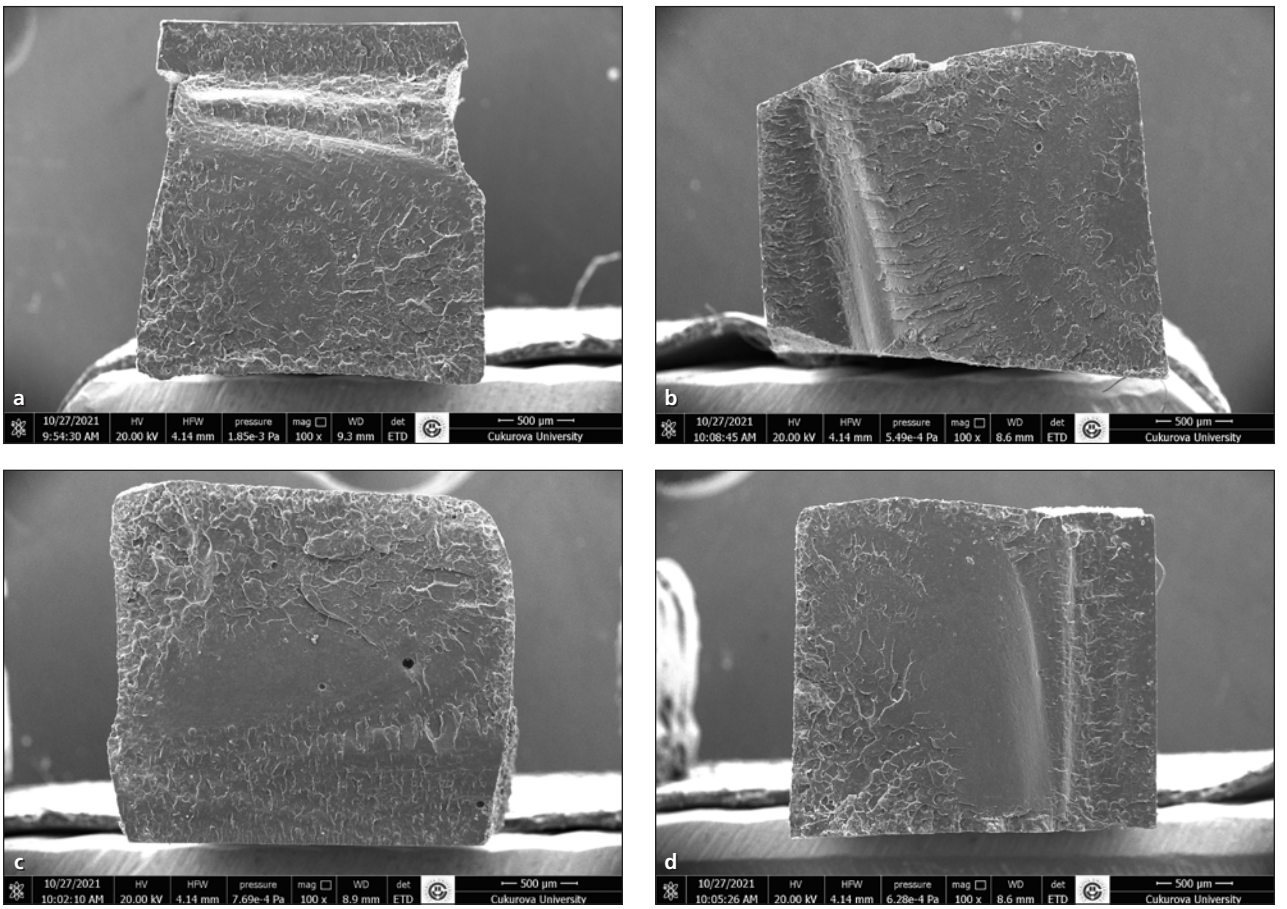

Fig 3 SEM images (×100) of the TM specimens in *(a)* distilled water, *(b)* 0.02 mol/L citric acid, *(c)* n-heptane, and *(d)* 50% ethyl alcohol.

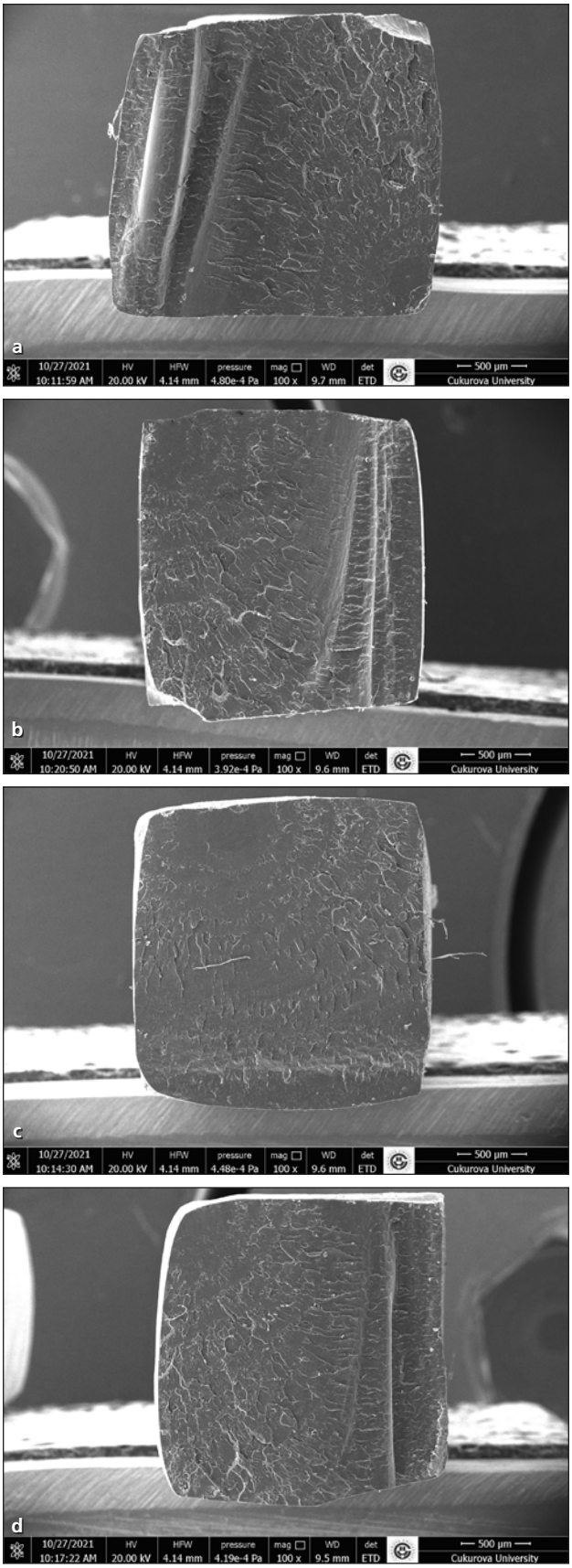

Fig 4 SEM images (×100) of the MM specimens in *(a)* distilled water, *(b)* 0.02 mol/L citric acid, *(c)* n-heptane, and *(d)* 50% ethyl alcohol.

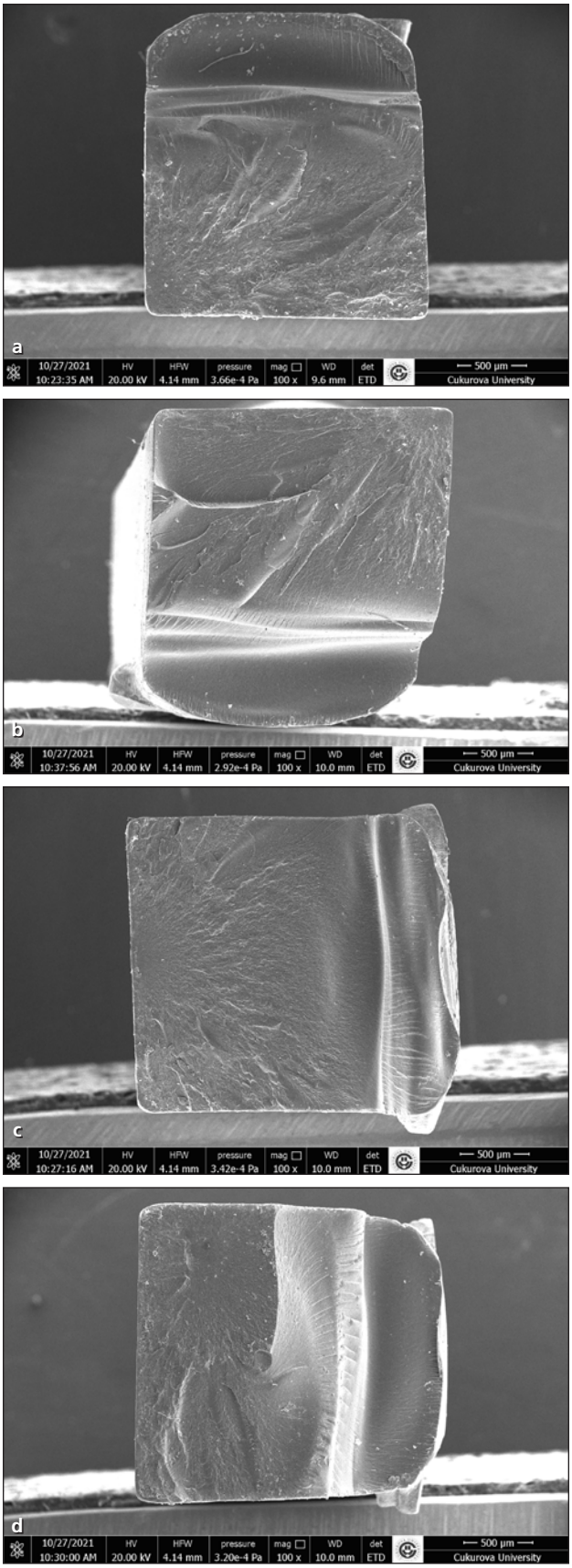

Fig 5 SEM images (×100) of the 3DM specimens in *(a)* distilled water, *(b)* 0.02 mol/L citric acid, *(c)* n-heptane, and *(d)* 50% ethyl alcohol.

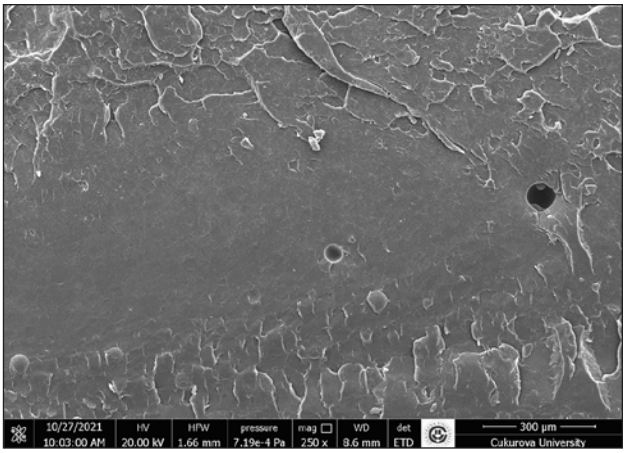

Fig 6 Porosities in the fractured surface of a specimen from the TM group.

The other critical question that needed to be decided by the authors was the exposure time in the FSLs. The studies in the literature also vary with regard to the immersion time of test specimens for artificial aging. The immersion time varies from 1 day to 1 month in studies intending to simulate mid- to long-term aging.[9,13,19] Furthermore, the immersion of test specimens in chemical solutions might not be a good way of simulating the conditions of prostheses in clinical use because restorations come into contact with food or beverages only when the patient is eating and drinking until the teeth are cleaned. Despite this fact, immersion in different chemical solutions is very common in the literature. In the current study, the specimens were exposed to FSLs for 7 days. It has been shown previously that the greatest changes in the mechanical properties of composite resins occurs within the first 7 days after immersion in FSLs.[13]

Digholkar et al[12] evaluated the FS and microhardness of provisional restorative materials fabricated by different methods without conditioning any chemical solution. The group reported 27.36 KHN for materials fabricated via a conventional method, 25.33 KHN for materials fabricated via milling, and 32.77 KHN for materials fabricated via 3D printing. The results found in that study are approximately two-fold greater than the results of the current study. Digholkar et al adjusted the microhardness indenter to apply 50 gf for 15 seconds in contrast to 100 gf for 15 seconds in the current study.[12] This difference in methodology may result in different hardness values. However, Digholkar et al[12] reported similar values in terms of FS in the traditional (95.58 MPa) and milled (104.20 MPa) materials, with lower values in the 3D-printed group (79.54 MPa) compared to the current study. Digholkar also used a different 3D-printing technology for fabricating test specimens, which may have caused the difference in FS.[12]

Taşın et al[11] compared the effect of 2,500 thermocycles on the mechanical properties of provisional restorative materials fabricated by different techniques. They reported no significant decrease in FS or FM in 3D-printed (FS: 125 to 125 MPa; FM: 3,357 to 3,214 MPa), milled (FS: 127 to 122 MPa; FM: 3,107 to 3,039 MPa), or conventionally fabricated (FS: 68 to 62 MPa; FM: 2,284 to 2,045 MPa) provisional materials. Although different aging methods were used, the results of the current study are in accordance with the results of the Taşın et al study, as no significant changes were observed in terms of FS and FM values in the test groups compared to the control groups.[11]

It is worth mentioning that according to the study results, exposure to n-heptane (24.5 KHN) and 50% ethyl alcohol (23 KHN) solutions seemed to increase the hardness of the 3DM group specimens compared to the control group specimens (16.4 KHN). The authors suggest two possible explanations for this phenomenon. First, Yap et al[19] reported that distilled water and 0.02 mol/L citric acid solution can soften composite resins by degrading the resin-filler interface, resulting in the complete or partial debonding of fillers, which decreases KHN values.[19] The material for fabricating the 3DM group specimens was a light-sensitive liquid composite resin with filler contents, so exposing that material to distilled water or 0.02 mol/L citric acid may soften the 3DM specimens, as Yap et al[19] described in their study. Thus, it may be speculated that there was no increase in the KHN values of the 3DM specimens placed in the n-heptane and 50% ethyl alcohol solutions, but there was a decrease in KHN values for the 3DM specimens placed in the distilled water and 0.02 mol/L citric acid solutions. Second, according to manufacturer instructions, the specimens must be washed in isopropyl alcohol prior to final curing to clean the residual monomer on the surface. It may be speculated that 50% ethyl alcohol plays a similar role and further cleaned the surfaces of the specimens of residual monomers, resulting in increased KHN values. N-heptane solution, on the other hand, may act as an oil barrier on the surface of the specimens, thus impeding the formation of an oxygen-inhibited layer during post-curing and eliminating the leaching of fillers, resulting in increased KHN values.[19]

When the KHN, FS, and FM values of the restoration materials produced via the various methods were compared, values for the conventionally produced specimens were found to be lower than the values for both the milled and 3D-printed group specimens. The greatest cause for this is undoubtedly the human-dependent factors in the conventional production method. Voids and porosities resulting from this production technique are frequently observed.[7] A SEM image (×250) of a specimen from the TM group is shown in Fig 6. Because the conventional group specimens were fabricated by

hand, they are prone to human-introduced errors, with air bubbles, voids, and porosities frequently observed. These defects inevitably risk affecting the strength of the material. Hence, Rayyan et al[7] have stated that provisional restorations produced with CAD/CAM methods have better color stability, mechanical properties, and biocompatibility than conventionally produced provisional restorations.

Finally, the temperature in the mouth changes constantly with eating and drinking, and the teeth come into contact with each other during eating and swallowing. Thus, when evaluating the effects of food contents on the mechanical properties of restoration materials, mastication forces should not be ignored. It would be more clinically relevant to test specimens in chew-simulating chambers where they are subjected to thermocycling with FSLs. This is both a limitation of the current study and a suggestion to consider for future studies. However, to the present authors' knowledge, there is no chewing simulator on the market that can function with any fluid other than distilled water.

CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn:

1. The fabrication method significantly affects the mechanical properties of provisional restoration materials.
2. Specimens in the 3DM group had the highest FS and FM values among the tested materials.
3. The FSLs had no negative effect on the mechanical properties of either the 3DM or MM group specimens.
4. Immersion of the TM group specimens in 50% ethyl alcohol had detrimental effects on KHN values.

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