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# The effect of post-processing washing time on the physical and chemical properties of 3D printable resin materials

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## Abstract

**Purpose** This study aimed to evaluate the effects of post-processing washing time on the mechanical properties (flexural strength, Vickers hardness) and chemical properties (water sorption, water solubility, degree of conversion, and elution of residual monomers) of different 3D printable resin materials.

**Materials and methods** Two different 3D printable resin materials were used: Formlabs 3D Permanent Crown (F) and Saremco Print Crowntech (S). Washing procedures were applied to the control groups according to each manufacturer's instructions and to the experimental groups with increasing washing times ($T_1 = 2 \times 3$ min, $T_2 = 2 \times 5$ min, and $T_3 = 2 \times 10$ min). A total of 296 specimens were evaluated for flexural strength (FS), Vickers hardness (VH), water sorption (Wsp), water solubility (Wsl), degree of conversion (DC), elution of residual monomers (RM) and scanning electron microscopy (SEM) combined with energy dispersive X-ray spectroscopy (EDS) was used for element analysis and surface characterization. Two-way ANOVA test was used for analyses, and Tukey test was used for post-hoc.

**Results** No significant differences were found in pairwise comparisons of FS values. ($p > 0.05$). VH values were significantly higher in the F resin material than in the S resin material at all washing times ($p < 0.001$). No significant interaction was found between resin type and washing time ($p = 0.255$), and Wsp values were not significantly different between materials ($p = 0.639$). A significant interaction was found between resin type and washing time for Wsl ($p < 0.001$), indicating material-dependent effects. No significant differences in DC values were observed between materials or washing times ($p > 0.05$). Residual monomer release was significantly influenced by both the resin type and the washing duration, especially for BPA, TEGDMA, UDMA, and Bis-GMA ($p < 0.05$).

**Conclusions** Washing time and resin type influenced specific physical and chemical properties of the materials. A $2 \times 5$ min ($T_2$) washing protocol appears optimal for the F resin material, balancing mechanical integrity with reduced monomer release, while longer washing time for S resin material did not provide additional benefits.

**Keywords** Degree of conversion, Flexural strength, Residual monomer, Vickers hardness, Water sorption and solubility

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## Introduction

The transition from traditional manufacturing techniques to digital technologies has significantly transformed the production paradigm in dentistry. One of the earliest implementations of this digital shift was the adoption of Computer-aided design (CAD) and Computer-aided manufacturing (CAM) based subtractive manufacturing methods for fabricating dental restorations. In these methods, restorations are milled from prefabricated blocks using computer-controlled milling machines. However, subtractive techniques present several limitations, including material waste, tool wear, and difficulty in fabricating complex geometries. In contrast, additive manufacturing (AM) technologies, such as three dimensional (3D) printing, enable the layer-by-layer construction of restorations directly from digital designs. This approach allows for greater design flexibility, enhanced customization, and more efficient material usage. Consequently, resin materials suitable for 3D printing have gained increasing popularity in clinical practice, particularly for the fabrication of fixed dental crowns [1–3].

Permanent crown resins utilized in three-dimensional (3D) printing through additive manufacturing (AM) are essential materials for producing partial and full crown restorations. Although there are different 3D printing technologies in the production of these restorations, the most established and widely used are stereolithography (SLA) and digital light processing (DLP) [1].

In SLA technology, an ultraviolet (UV) laser beam polymerizes the liquid photopolymer resin surface layer by layer, following the digital design point by point. This method offers high accuracy and excellent surface quality for detailed applications. In contrast, DLP technology uses a digital light projector to light-polymerize the entire layer of resin simultaneously. This system offers a much faster print time compared to SLA while providing a sufficient level of accuracy and surface quality for clinical use [4].

Additive manufacturing varies in terms of methods and materials used. One of these, Lithography-based ceramic manufacturing (LCM) processes, enables complex 3D shaping through additive manufacturing. LCM uses the same principle of layer-by-layer photopolymerization as Lithography-based manufacturing, but is optimized for ceramic suspensions, enabling the production of ceramic parts with high density, precision and superior mechanical properties [5].

Independent of the technology used, 3D printed objects are treated with a two-stage post-processing procedure as recommended by the manufacturers. To eliminate unpolymerized resin, immediately after printing, post-washing is performed with alcohol (Alc), and the post-curing process is completed using heat and ultraviolet (UV) light to obtain the mechanical and biological properties [6].

3D printable materials are generally formed from methacrylate (or di-methacrylate) monomers, inorganic fillers, initiators, silane coupling agents, and pigments [2].

A certain viscosity is required in each curing cycle in the 3D production process for the resin to flow between the platform and resin vat. Therefore, the total volume of filler particles (i.e., the inorganic %) is kept at low rates to provide flowability [3, 7].

Thus, with an increase in the organic matrix ratio after printing all the monomers cannot be fully converted to polymers and residual acrylic monomers are removed with a post-washing procedure [8]. This process is a washing procedure to remove resin remnants that have stuck to the printed surface, and traditionally, solvents such as isopropyl alcohol or tri-propylene glycol monomethyl ether are used [9–11]. If the washing is insufficient, the remaining resin polymerizes and can reduce the accuracy and therefore the fit of the print [12]. In addition, there are concerns that intra-oral release of residual monomers (RM) have cytotoxic and allergic effects on human cells [13, 14].

A prolonged washing procedure is known to increase cell vitality and reduce cytotoxicity [15, 16]. However, extending the washing procedure with alcohol weakens the mechanical properties of the material by reducing RM [17]. This is because Alc causes irreversible loosening of the polymer network by dissolving polymers and monomers [16, 18]. In a very recent study it was reported that compared to Alc, longer washing with deionized water showed better results in 3D printed materials in respect of mechanical and surface properties, degree of conversion (DC), and biological performance [19]. Saremco Print Cleaning Concentrate, described as a "Water-based alternative to conventional solvents for 3D printing" was recently launched on the market. It was stated that this solvent was a superior alternative to isopropanol, ethanol and butyl di-glycol for printed objects produced with SLA and DLP. Previously, after printing for Saremco Print Crowntech, cleaning with a cloth or brush wet with Alc (96%) without full submersion in the Alc was recommended, whereas now a 2 × 3 min ultrasonic bath with a cleaning concentrate (CC) solution is recommended [20]. Another recent study recommended a two-step washing protocol (2 × 3 min) for Formlabs 3D Permanent Crown resin (F), using fresh 99.9% isopropyl alcohol for both pre-cleaning and final cleaning steps [7].

A limited number of studies conducted with a washing procedure applied after AM have shown that in addition to material selection, post-washing and

post-cure processing have an effect on in-vitro cytotoxicity [21], that the temperature and time washing affect the mechanical and biological properties of printed resin [22], and it has been emphasized that a longer period of washing increases cell vitality and reduces cytotoxicity [15]. It has also been reported that the bath (ultrasonic bath or rotary washer) [23], and adjustment of the solvent and UV curing time [13] specific to each printed resin are important in respect of obtaining optimum mechanical and aesthetic results in restorations.

Although there has been previous research of flexural strength (FS) and Vickers hardness (VH) values in F and S resin materials [24–27], there are no studies in the literature that have examined increasing washing times with different washing solutions. To the best of our knowledge, there has also been no research related to the water sorption (Wsp) and water solubility (Wsl) of printable permanent crown resin materials. The DC values of printable resins used for different purposes in AM have been investigated [28–31] but not those used for permanent crowns. In a previous study [32], although RM release of printable crown resins was researched, the effect of washing time was not evaluated.

The aim of this study was to evaluate the effects of a prolonged washing procedure on the physical (FS, VH, Wsp, Wsl) and chemical (DC, RM) properties of 3D printable resin materials (F and S). The null hypothesis ($H_0$) of the study was that a prolonged washing procedure would not affect the physical and chemical properties of 3D printable resin materials.

## Materials and methods

Two different 3D printable resin materials were used in this study: Formlabs 3D Permanent Crown Resin (Formlabs, Somerville, MA, USA) and Saremco Print Crowntech Resin (Saremco Dental AG, Switzerland) (Table 1).

### Sample size

The sample size for the analyses performed in this study was calculated using G*Power 3.1 software (Heinrich-Heine-Universitat Dusseldorf, Germany). Based on the Gan et al. study [23], the between-group effect size was determined to be Cohen's d = 1.86. Considering this effect size, a power analysis was conducted at a 95% confidence level ($\alpha$ = 0.05) and 80% statistical power ($1-\beta$ = 0.80), resulting in a minimum sample size of 5 per group.

In total, 296 specimens were prepared to evaluate the effects of washing time on the physical and chemical properties of two different 3D printable permanent crown resins (Formlabs Permanent Crown [F] and Saremco Print Crowntech [S]). Each material group was divided into four subgroups based on different washing protocols (Control, $T_1$, $T_2$, and $T_3$) (Fig. 1).

### Specimen preparation

In accordance with the ISO 4049 and ISO 10,477 standards, three different files were designed in standard tessellation language (STL) format with the following dimensions and purposes: 25 mm (length) × 2 mm (width) × 2 mm (thickness) for the FS test, 15 mm (length) × 2 mm (width) × 2 mm (thickness) for the Wsp and Wsl tests, 6 mm (length) × 6 mm (width) × 2 mm (thickness) for the other tests [33]– [34]. Using these STL files, Formlabs 3D Permanent Crown resin specimens were prepared with 50 micron (µm) layer thickness (Formlabs). The specimens underwent washing at four different time intervals (FC = 3 min; F-$T_1$ = 2 × 3 min; F-$T_2$ = 2 × 5 min; F-$T_3$ = 2 × 10 min) using 99% pure isopropyl alcohol in a Form Wash device (Formlabs, Somerville, MA, USA) (Fig. 1). Before the second washing, the Alc was emptied from the device and replaced with a fresh solution, then the washing procedure was repeated. These washing durations were not continuous; each consisted of two sequential sub-washing steps with fresh solution replacement in between. After completion of the washing procedures, the specimens were polymerized for 20 min at 60 °C in a Form Cure device (Formlabs, Somerville, MA, USA). The supports were cut, and the specimens were turned upside down, then the polymerization was repeated under the same conditions.

Using the prepared STL files in an Asiga max UV printer (Asiga Sydney, Australia), Saremco Print Crowntech resin specimens were prepared with the DLP production method using 50 µm layer thickness. In accordance with the manufacturer's instructions, the specimens in the SC group (control group) were wiped several times with a cloth soaked in alcohol until the

**Table 1** 3D printable resin materials used in this study and their properties

| | Manufacturer/Lot number | Production Method | Composition |
|---|---|---|---|
| Formlabs 3D-Permanent Crown (F) | Formlabs, Somerville, MA/ 600,163 | Stereolithography (SLA) | Esterification products of 4,40-isopropylidenediphenol, ethoxylated and 2-methylprop-2-enoic acid 50–75%. Diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide (< 2.5%) |
| Saremco Print Crowntech (S) | Saremco Dental AG; Rebstein, Switzerland/ E 308 | Digital light processing (DLP) | Ethoxylated bisphenol-A di-methacrylate (50–70%), dental glass silica |

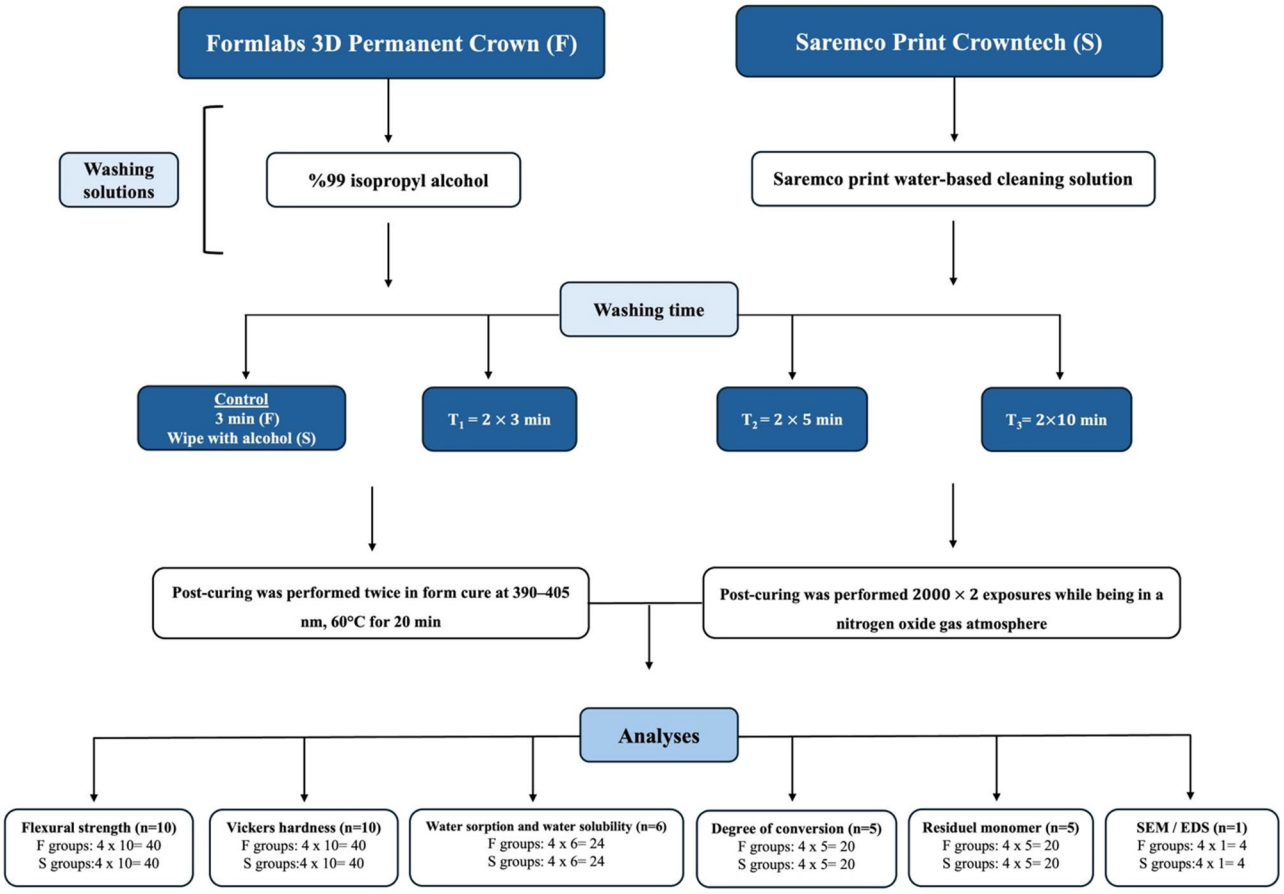



**Fig. 1** Experimental workflow of post-processing and analyses of Formlabs 3D Permanent Crown and Saremco Print Crowntech resin materials

surface became matte. Specimens in the experimental groups were washed for 3 different time periods (S-$T_1$: 2 × 3 min, S-$T_2$: 2 × 5 min, S-$T_3$: 2 × 10 min) with Saremco print cleaning concentrate in an ultrasonic bath (Fig. 1). Before the second washing, all specimens were washed in warm water, dried with pressurized air, then the solution in the washing device was replaced and the washing procedure was repeated. These washing durations were not continuous; each consisted of two sequential sub-washing steps with fresh solution replacement in between. After completion of the washing procedures, the specimens were polymerized in an Otoflash G171 device (NK Optik, Baierbrunn, Germany) in nitrogen oxide gas atmosphere set at 2000 × 2 pulses. All prepared specimens were polished with a felt cloth (IMIPOMZA, Imicryl Konya, Turkey) for 90 s at a low-speed of 1500 rpm using a manual rotary device. After these processes, the specimens were cleaned in an ultrasonic bath for 1 min and then left to dry in the open air.

### Flexural strength (FS) test

The prepared specimens (25 × 2 × 2 mm) ($n$ = 10) were stored at room temperature for 24 h prior to testing. Before starting the tests, the dimensions of each specimen were measured with digital calipers (HSL 246–15, Karl Hammacher GmbH, Solingen, Germany.). The FS test was performed with the three-point bending method using a universal test device (Testometric M500, 25kN, Testometric Co., Rochdale, UK) at a crosshead speed of 1 mm/min, in accordance with the procedure defined in ISO 10477:2004 [33]. The FS formula was was used to calculate the FS in megapascal (MPa) units (Table 2).

### Vickers hardness (VH)

The specimens (6 × 6 × 2 mm) ($n$ = 10) were left for 24 h in an incubator at 37 °C. The micro-hardness measurement was performed using a Vickers hardness device (Micro Hardness Tester, Wilson; Buehler, Germany) with 300 gf loading (2.941 N) for 15 s [34]. The mean of three measurements performed at random from the top surface of each specimen — the surface exposed to ambient conditions during 3D printing and post-processing — was determined as the VH value of that surface. Care was taken to leave a sufficient distance (at least 5-fold the diagonal length) between the measurements performed on each sample.

**Table 2** Formulas used in analysis

| Analysis | FS | V | Wsp | Wsl | DC |
|---|---|---|---|---|---|
| Formula | $\frac{3FL}{2wh^2}$ | $\left(\frac{d}{2}\right)^2 \times \pi \times t$ | $\frac{M2-M3}{V}$ | $\frac{M1-M3}{V}$ | $\left(1-\frac{\left(\frac{A_{aliphatic}}{A_{aromatic}}\right)_{cured}}{\left(\frac{A_{aliphatic}}{A_{aromatic}}\right)_{uncured}}\right)\times 100$ |

*FS* flexural strength (MPa), *V* volume ($mm^3$), *Wsp* Water sorption,*Wsl* Water solubility, *DC* Degree of conversion, *F* the loading force applied at the fracture point (N), *L* distance between supports (mm), *w* sample width (mm), *h* sample height (mm), *d* sample diameter (mm), *t* sample thickness (mm), *M1* initial mass (mg), *M2* second mass (mg), *M3* third mass (mg)

### Water sorption (Wsp) and water solubility (Wsl)

To accelerate the drying procedure, the prepared specimens (15 × 2 × 2 mm) ($n = 6$) were transferred to a desiccator with silica on the base. The desiccator was placed in an incubator at 37 °C for 22 h, then left for 2 h at room temperature. The weight of each specimen was then measured on electronic scales (MS105DU, Mettler-Toledo Group, Switzerland). This procedure was repeated until the mass loss of each specimen did not exceed 0.1 mg throughout 24 h. When the mass loss of the specimens fell below 0.1 mg, the first weighing was performed and recorded as M1, which represents the initial dry mass after storage in a desiccator at 37 °C for 24 h.

The volume of each specimen was calculated using digital calipers based on the geometric dimensions and the formula provided in Table 2.

Specimens were then completely immersed in distilled water and stored in an incubator at 37 °C for 7 days. After removal, the specimens were gently blotted with absorbent paper until no visible moisture remained on the surface. The mass was immediately measured and recorded as M2, representing the wet mass after water immersion. To remove any residual moisture, specimens were again placed in a desiccator at 37 °C for an additional 24 h, after which the final weighing was performed and recorded as M3, corresponding to the final dry mass.

These three values (M1, M2, and M3) were used to calculate water sorption (Wsp) and water solubility (Wsl) in accordance with ISO 10477:2004 [33], which defines M1 as the initial dry mass, M2 as the wet mass after immersion, and M3 as the final dry mass after re-drying. The Wsp and Wsl values were calculated using the formula provided in Table 2.

### Degree of conversion (DC)

The prepared specimens (6 × 6 × 2 mm) ($n$ = 5) were incubated at 37 °C for 24 h. The Fourier-transform infrared (FTIR) spectrometer (Bruker Vertex70, Bruker Optics Inc., Ettlingen, Germany) was used to evaluate the DC values. ATR crystal was placed in close contact and FTIR spectrums between 400 and 4000 $cm^{-1}$, performed at 4 $cm^{-1}$ resolution were recorded with 16 scans. To measure the bonds in a non-cured scenario, a drop of each of the non-cured resins was used. In the spectrums obtained, the peak elevations of the absorption bands of the C networks, equal to aliphatic and aromatic C, were measured at 1638 $cm^{-1}$ and 1608 $cm^{-1}$, as recommended by Rueggeberg et al. [35]. The DC values were calculated according to the formula (Table 2).

### Residual monomer (RM)

The prepared specimens (6 × 6 × 2 mm) ($n$ = 5) were placed in amber-colored bottles containing 5 mL 5% ethanol/water solution, then kept at 37 °C in an incubator for 24 h. The specimens were removed from the solution after 24 h and the solution was analyzed with high-performance liquid chromatography-ultraviolet (HPLC-UV; Shimadzu Nexera X2, Tokyo, Japan) [36]. Diode Array (PDA/DAD) was used as detector and Inertsil ODS-4 C18 (4.6 × 250 mm, 5 µm, GL Sciences) as the column.

In the analysis of 2-hydroxyethyl methacrylate (HEMA), bisphenol A (BPA), tri-ethylene glycol di-methacrylate (TEGDMA), urethane di-methacrylate (UDMA), and bisphenol A-glycidyl methacrylate (Bis-GMA), a 30:70 water-acetonitrile solution was used as the mobile phase. The standard solutions of monomers were prepared in methanol. The flow rate of the mobile phase was 1.0mL/min and column temperature were 25 °C, and absorbance readings were taken at 210 nm. The volume of liquid injected to the column from each specimen was defined as 20µL. The concentrations of RM were determined by forming calibration curves at low, moderate, and high concentrations (Fig. 2).

### Scanning electron microscope (SEM) and energy distribution X-ray spectroscopy (EDS) analysis

An additional specimen from each group, selected at random, was fixed with aluminum pins, then coated in gold using the Q150R Plus Sputter Coater Combined System (Quorum Technologies, Lewes, United Kingdom). For detailed examination and element analysis, an SEM device combined with EDS (FEI, Quanta 650 FEG, Eindhoven, Netherlands) was used. SEM images of each specimen were taken at × 500, × 1000, and × 10,000 magnifications. A specific region of the specimens (20 × 20 µm) was examined at × 500 magnification. An EDS spectrum was obtained showing the peak of elements in the basic composition of the materials (both in wt % and at % of the groups).

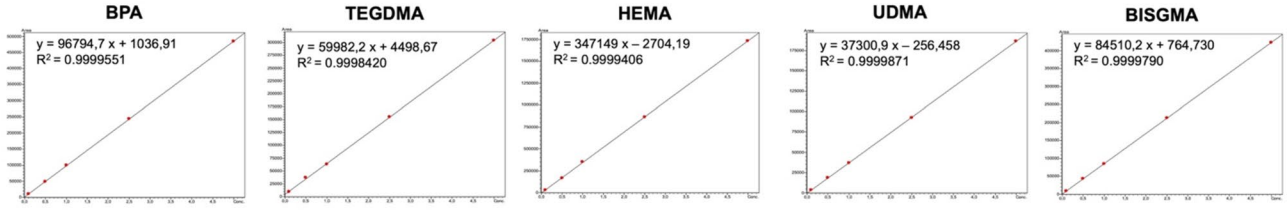



**Fig. 2** The retention time of HPLC peaks of BPA, TEGDMA, HEMA, UDMA and Bis-GMA

### Statistical analysis

Data obtained in the study was analyzed statistically using SPSS vn. 28.0 software. Normality of the data was assessed using the Shapiro–Wilk test, supported by visual inspection of Q–Q plots and evaluation of skewness and kurtosis values (Thresholds of ±1 considered acceptable). Once normality was confirmed, two separate two-way ANOVAs were conducted to examine the effects of washing time and material type on mechanical performance and residual monomer release, respectively.Where statistically significant differences were observed, post-hoc pairwise comparisons were performed using Tukey's HSD test. Results were given in a 95% confidence interval and the level of statistical significance was accepted as $\alpha = 0.05$.

## Results

The mean, standard deviations, and statistical analysis results of FS, VH, Wsp, Wsl, and DC values of the F and S resin materials are shown in Table 3. According to the Two-Way ANOVA results for FS, a statistically significant difference was observed between materials ($p = 0.001$); however, this finding was not confirmed by post-hoc analysis. Therefore, no significant pairwise differences could be detected (Table 3).

VH values showed statistically significant differences depending on the type of material ($p < 0.001$). The F resin material exhibited significantly higher VH values compared to the S resin material at all washing times. In the F resin material, VH values tended to decrease as washing time increased, with the exception of $T_1$ ($p < 0.001$).

There was no statistically significant difference in the interaction between resin material type and washing time ($p = 0.255$) and also in Wsp values between the two resin materials ($p = 0.639$). However, the effect of washing time on Wsp was statistically significant ($p = 0.011$). Post-hoc comparisons showed that some washing times resulted in significantly different Wsp values, particularly within the Saremco resin material groups.

A statistically significant interaction between resin type and washing time was found ($p < 0.001$), indicating that the effect of washing time on Wsl differed depending on the material. Moreover, both resin type and washing time individually had significant main effects ($p < 0.001$ and $p = 0.033$, respectively). While no significant difference was found among the F resin material subgroups, all Saremco resin material subgroups showed significantly higher Wsl values compared to the F resin material groups.

A representative graph of the DC of the F and S resin material subgroups are presented in Fig. 3. No statistically significant difference was observed in the DC values between different resin materials ($p = 0.423$), or washing times ($p = 0.198$).

Residual monomer release was significantly influenced by both the resin type and the washing duration, especially for BPA, TEGDMA, UDMA, and Bis-GMA ($p < 0.05$) (Table 4).

**Table 3** Evaluation of flexural strength (MPa), Vickers hardness (VH), water sorption (μg/mm$^3$), water solubility (μg/mm$^3$), and degree of conversion (%) variables between groups

| 3D printable resin materials | Washing time | FS (Mpa) | VH | Wsp | Wsl | DC (%) |
|---|---|---|---|---|---|---|
| F | C | 181.6 ± 18.6 | 37.6 ± 7.7$^{ab}$ | 1.3 ± 0.15$^{ab}$ | 0.44 ± 1.2$^{a}$ | 73.2 ± 2.0 |
| | $T_1$ | 174.3 ± 15.9 | 47.3 ± 2.4$^{a}$ | 1.5 ± 0.3$^{ab}$ | −0.97 ± 0.6$^{a}$ | 73.8 ± 4.4 |
| | $T_2$ | 183.2 ± 24.8 | 34.9 ± 3.9$^{b}$ | 1.8 ± 1.0$^{ab}$ | −0.6 ± 0.4$^{a}$ | 72.7 ± 0.8 |
| | $T_3$ | 161.1 ± 16.1 | 29.5 ± 1.5$^{c}$ | 1.2 ± 0.3$^{ab}$ | −1 ± 1.1$^{a}$ | 73.1 ± 0.8 |
| S | C | 172.5 ± 14.1 | 21.3 ± 0.9$^{d}$ | 1.0 ± 0.3$^{a}$ | 0.5 ± 0.4$^{a}$ | 71.4 ± 1.1 |
| | $T_1$ | 155.1 ± 32.5 | 17.8 ± 2.0$^{e}$ | 1.1 ± 0.3$^{ab}$ | 1.6 ± 0.6$^{b}$ | 70.3 ± 0.2 |
| | $T_2$ | 147.1 ± 24.5 | 20.5 ± 2.4$^{de}$ | 2.0 ± 0.6$^{b}$ | 3.1 ± 0.8$^{b}$ | 75.6 ± 3.1 |
| | $T_3$ | 162.0 ± 16.4 | 18.8 ± 3.1$^{de}$ | 1.6 ± 0.3$^{ab}$ | 2.5 ± 0.8$^{b}$ | 73.0 ± 3.1 |
| *p* -value | Group | 0.001 | < 0.001 | 0.639 | < 0.001 | 0.423 |
| | Time | 0.118 | < 0.001 | 0.011 | 0.033 | 0.198 |
| | Group*Time | 0.052 | < 0.001 | 0.255 | < 0.001 | 0.051 |

p -values were obtained by Two-Way Anova test. Post-hoc comparisons were made by Tukey test. Data are presented as mean ± standard deviation. F: Formlabs resin material, S: Saremco resin material, C: Control, $T_1$: 2 × 3 min, $T_2$: 2 × 5 min, $T_3$: 2 × 10 min

*: The same lowercase letters indicate that there is no statistically significant difference between the washing-times of each resin material

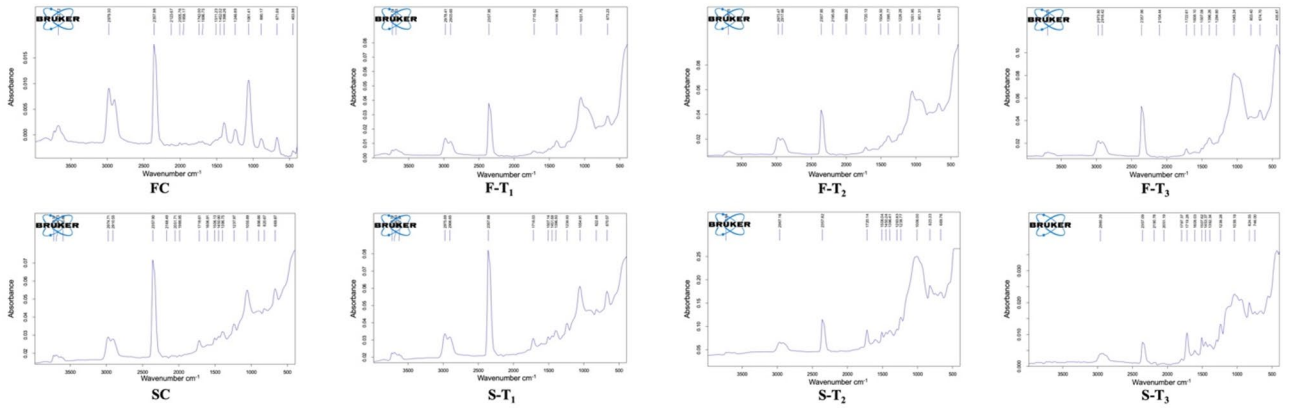



**Fig. 3** Representative *Fourier-Transform Infrared Spectroscopy* (FTIR) spectra (Absorbance *versus* Wavenumber) of 3D-printed permanent crown resin subgroups used for degree of conversion (DC) analysis. *(F: Formlabs, S: Saremco, C: Control, $T_1$: 2 × 3 min, $T_2$: 2 × 5 min, $T_3$: 2 × 10 min.)

In the F resin material, all monomer levels decreased with increasing washing time. Although lower residual monomer levels were initially observed in the Saremco resin material, the decrease in these levels was limited over time.

Washing time had no significant effect on HEMA release for both resin materials ($p = 0.502$). Furthermore, the interaction between resin material type and washing time was at the threshold of statistical significance ($p = 0.050$). Regardless of washing time, BPA and Bis-GMA concentrations were consistently higher in the F resin material than in the S resin material ($p < 0.001$). Conversely, UDMA levels were significantly greater in the S resin material at all time points, except for the control group.

When the SEM images of the materials were examined (Fig. 4), it can be said that an irregular organic matrix appearance with residual resin remnants was seen to remain on the surface in both the FC and SC groups. The surface solvent effect of the CC washing solution on the S resin material was not seen to progress as dramatically as the effect of Alc on the F resin material.

The EDS spectrum (Fig. 5) was evaluated, and it was determined that the wt and at % of elements was similar in both the F and S resin material groups and the small differences seen were due to the filler particles not showing absolute homogeneity in all areas.

## Discussion

Crowns produced with 3D printing are currently often used in clinics, and adherence to the manufacturer's instructions when treating these resin-based materials with the post-washing and post-curing procedures is very important. While prolonged washing with Alc improves the biological properties of resin crowns, concerns remain regarding its potential to diminish the mechanical properties. Therefore, a CC has been introduced to the market as an alternative to the traditional washing process. The results of this study demonstrated that an increased washing time, independently of the washing solutions, did not change the FS and DC values, lowered the VH values, increased the Wsl, and decreased RM. In accordance with these findings, the null hypothesis of the study was rejected.

**Table 4** Evaluation of residual monomers values (in parts-per-million) between groups

| 3D printable resin materials | Washing time | HEMA | BPA | TEGDMA | UDMA | Bis-GMA |
|---|---|---|---|---|---|---|
| F | C | 0.2 ± 0.04 | 1.1 ± 0.3$^{a}$ | 0.29 ± 0.03$^{a}$ | 1.2 ± 0.06$^{a}$ | 3.7 ± 0.9$^{a}$ |
| | $T_1$ | 0.18 ± 0.02 | 0.7 ± 0.04$^{b}$ | 0.2 ± 0.02$^{ab}$ | 0.86 ± 0.06$^{b}$ | 2.36 ± 0.1$^{b}$ |
| | $T_2$ | 0.17 ± 0.02 | 0.6 ± 0.04$^{b}$ | 0.18 ± 0.02$^{b}$ | 0.77 ± 0.5$^{b}$ | 2.03 ± 0.1$^{b}$ |
| | T3 | 0.17 ± 0.01 | 0.5 ± 0.02$^{b}$ | 0.17 ± 0.02$^{b}$ | 0.57 ± 0.04$^{b}$ | 1.98 ± 0.1$^{b}$ |
| S | C | 0.14 ± 0.04 | 0.38 ± 0.06$^{c}$ | 0.45 ± 0.09$^{c}$ | 1.59 ± 0.42$^{a}$ | 1.19 ± 0.2$^{c}$ |
| | $T_1$ | 0.16 ± 0.02 | 0.33 ± 0.05$^{c}$ | 0.49 ± 0.04$^{c}$ | 1.59 ± 0.25$^{a}$ | 1.9 ± 0.2$^{b}$ |
| | $T_2$ | 0.2 ± 0.02 | 0.29 ± 0.02$^{c}$ | 0.48 ± 0.07$^{c}$ | 1.42 ± 0.16$^{a}$ | 1.84 ± 0.11$^{b}$ |
| | $T_3$ | 0.18 ± 0.02 | 0.29 ± 0.03$^{c}$ | 0.46 ± 0.06$^{c}$ | 1.47 ± 0.31$^{a}$ | 2.1 ± 0.23$^{b}$ |
| *p* -value | Group | 0.096 | < 0.001 | < 0.001 | < 0.001 | < 0.001 |
| | Time | 0.502 | < 0.001 | 0.096 | 0.004 | 0.020 |
| | Group*Time | 0.050 | < 0.001 | 0.015 | < 0.001 | < 0.001 |

p-values were obtained by Two-Way Anova test. Post-hoc comparisons were made by Tukey test. Data are presented as mean ± standard deviation. F: Formlabs resin material, S: Saremco resin material, C: Control, $T_1$: 2 × 3 min, $T_2$: 2 × 5 min, $T_3$: 2 × 10 min

*: The same lowercase letters indicate that there is no statistically significant difference between the washing-times of each resin material

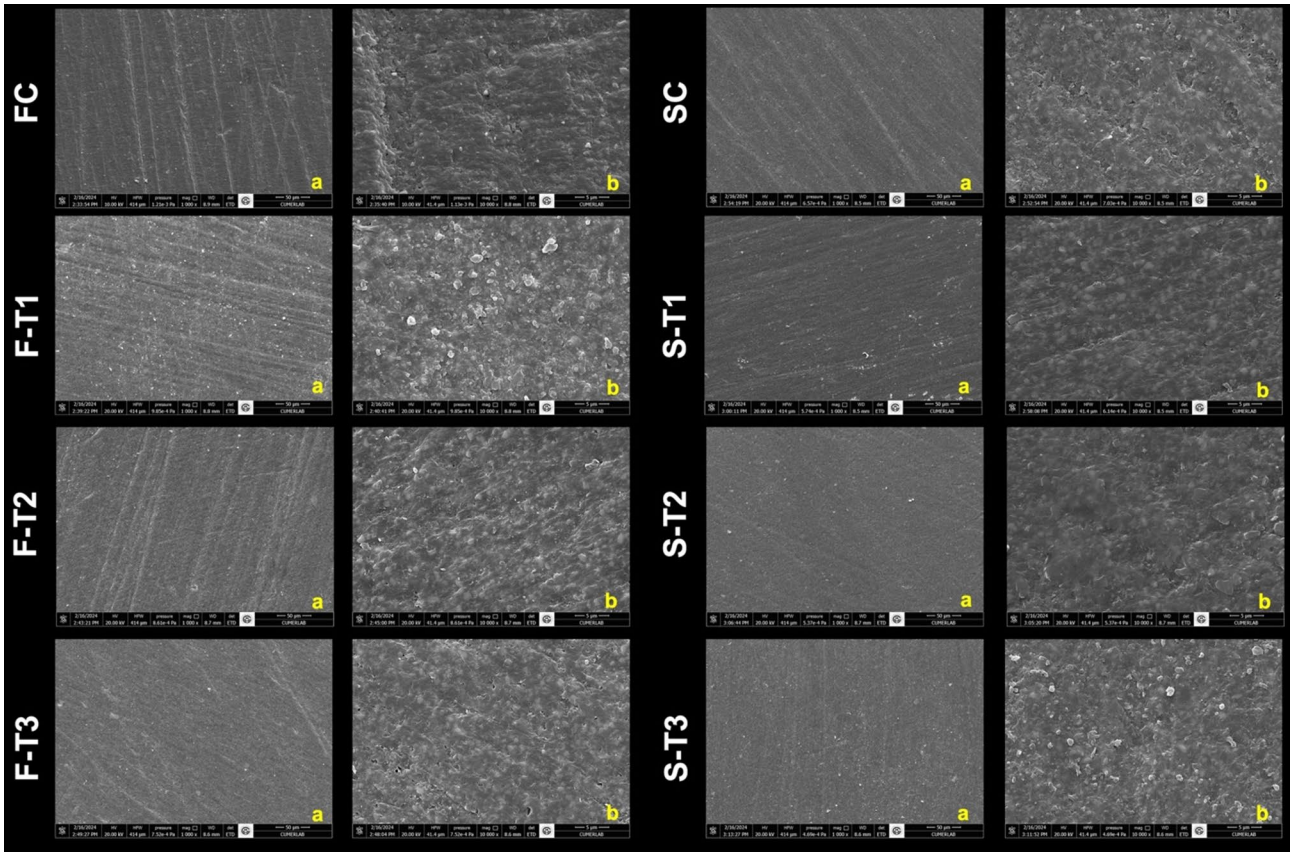
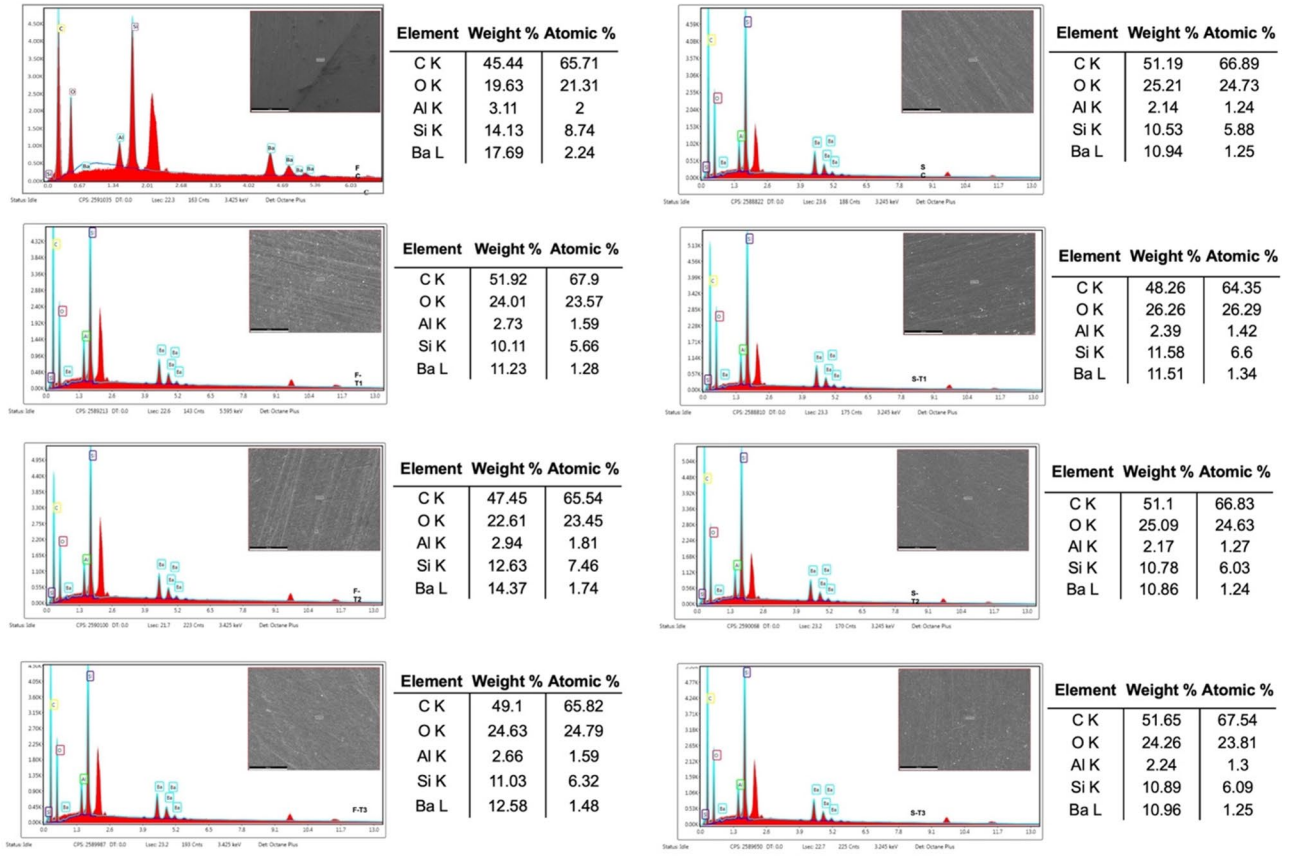



**Fig. 4** Representative SEM images of the materials. a: ×1000 magnification, b: ×10,000 magnification. The irregular organic matrix appearance seen on the surface in both FC and SC groups may be residual resin residues. In the F-$T_1$ group, it is observed that the top layer is removed with the effect of Alc and inorganic filler particles become visible. In the F-$T_2$ group, some of the filler particles are seen to be removed and, in the F-$T_3$ group, the amount of removed particles increases and the voids left behind are seen. In the S-$T_1$ group, it is observed that the surface becomes smooth after washing with CC. While no significant difference was observed between S-$T_1$ and S-$T_2$, the organic matrix may have moved away as filler particles became visible in the S-$T_3$ group

A 3D printable resin has a raw material based on methacrylate or di-methacrylate monomers together with inorganic fillers [2]. With exposure to light in the 3D printer the viscous liquid resin can be photopolymerized and cured, and a washing procedure is applied to remove RM that are not cross-linked to the post-printing matrix. Although other solutions have been tested, isopropyl alcohol is generally used in this washing procedure [10, 11]. The washing procedure is applied with an ultrasonic or rotary washer or by simply immersing in the solution. Washing the printed product for 3 min with isopropyl alcohol with an ultrasonic or rotary washer has been found to improve cell vitality compared to immersion washing [23]. The manufacturer's instructions for F resin are to wash with isopropyl alcohol for 3 min in a rotary washer, and for S resin, to wipe it with Alc. However, it is now recommended to apply a 3-min ultrasonic bath procedure twice with a new CC washing solution for S resin. The recommendation to also wash F resin twice (2 × 3 min) was the reason for the two repetitions of increasing time in this study.

Previous studies have reported that FS, DC, cell viability and VH are significantly affected because of prolonged washing with Alc [22, 23, 31]. The polymer matrix is affected by the chemical structure and concentration of the solvent used in washing. Thus, Alc can cause an excessive degree of removal of partially cured and uncured monomers, and this weakens the matrix structure [18]. However, if it is considered that many studies have reported positive effects of increased washing time, investigation of this time with a washing solution other than Alc can be seen to be extremely important [15, 21, 22].

In a study by Hwangbo et al., it was reported that cell viability increased with extended washing time and FS significantly decreased, especially at 60 min [15]. Therefore, the total washing periods in the current study were limited to 6 (2 × 3 min), 10 (2 × 5 min), and 20 (2 × 10 min) minutes. Like the results of the current study, Temizci et al. found that the mean FS and VH values of F resin material were higher than those of S resin material [25]. However, there are also studies in the literature showing



| Element | Weight % | Atomic % |
|---|---|---|
| C K | 45.44 | 65.71 |
| O K | 19.63 | 21.31 |
| Al K | 3.11 | 2 |
| Si K | 14.13 | 8.74 |
| Ba L | 17.69 | 2.24 |

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 51.19 | 66.89 |
| O K | 25.21 | 24.73 |
| Al K | 2.14 | 1.24 |
| Si K | 10.53 | 5.88 |
| Ba L | 10.94 | 1.25 |

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 51.92 | 67.9 |
| O K | 24.01 | 23.57 |
| Al K | 2.73 | 1.59 |
| Si K | 10.11 | 5.66 |
| Ba L | 11.23 | 1.28 |

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 48.26 | 64.35 |
| O K | 26.26 | 26.29 |
| Al K | 2.39 | 1.42 |
| Si K | 11.58 | 6.6 |
| Ba L | 11.51 | 1.34 |

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 47.45 | 65.54 |
| O K | 22.61 | 23.45 |
| Al K | 2.94 | 1.81 |
| Si K | 12.63 | 7.46 |
| Ba L | 14.37 | 1.74 |

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 51.1 | 66.83 |
| O K | 25.09 | 24.63 |
| Al K | 2.17 | 1.27 |
| Si K | 10.78 | 6.03 |
| Ba L | 10.86 | 1.24 |

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 49.1 | 65.82 |
| O K | 24.63 | 24.79 |
| Al K | 2.66 | 1.59 |
| Si K | 11.03 | 6.32 |
| Ba L | 12.58 | 1.48 |

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 51.65 | 67.54 |
| O K | 24.26 | 23.81 |
| Al K | 2.24 | 1.3 |
| Si K | 10.89 | 6.09 |
| Ba L | 10.96 | 1.25 |

**Fig. 5** EDS spectrum, SEM images of the focused region of the investigated materials and the percentage of weight and atomic weight of the groups (scale bar: 100 µm). Both F and S resin materials were found to contain carbon (C), oxygen (O), aluminum (Al), silicon (Si) and barium (Ba)

higher mean FS and VH values of S resin material [26, 27]. When this was examined in the study by Nam et al., the surface procedures applied to the 3D resin specimens were seen to have increased the mean mechanical strength [24]. This difference can be attributed to the technical sensitivity of the post-production procedures.

The present study found no significant difference between F and S resin materials in respect of the Wsp values ($p$ = 0.639), but the Wsl was seen to be significantly lower in F resin material ($p$ < 0.001). 3D printed permanent resins reinforced with ceramic (ceramic micro-filler content was higher for F resin material and dental glass silica was higher for S material) and with low inorganic filler (approximately weighted 50%; filler size 700 nm) have been reported [3, 7]. The reason for low Wsl of F resin material is thought to be associated with the higher filler ratio. In addition, despite the washing solution for S resin material being CC, Wsl significantly increased more as time increased. This result, which was not expected at the start of the study was thought to be due to the structure of the material. However, to see the clear effect of the washing solution, a future study should be designed by washing both resins in both solutions. This can be counted as a limitation of the study. However, as a first step the authors of this study had to go with the manufacturer recommendations. Within the limitations of this study, it can be said that a longer washing time is not appropriate for S resin material despite the use of a CC.

Although there have been investigations of the DC values of printable resins used in temporary crowns and bridges produced with 3D printing [6, 31], splint and orthodontic applications [28] denture base materials [29], and dental modelling [30], permanent crown resins have not been researched. It has been shown that DC, which is closely related to the mechanical properties and biocompatibility of 3D printable resin, is affected by the curing time and temperature [22]. The results of the current study showed that a prolonged washing time did not change the DC of permanent crown resins.

3D printable resin materials are generally produced with extremely cytotoxic materials such as polymethyl methacrylate (PMMA), and although the curing process reduces toxicity it does not eliminate it. Even if the post-processing procedures of these resins remove uncured monomers, the mechanical strength may be reduced [15]. The chemical content has not been fully disclosed by the manufacturers, but it has been stated that as organic

content they have small molecule monomers that reduce viscosity (TEGDMA for F resin material and ethoxylated bisphenol A di-methacrylate (Bis-EMA) for S resin material) [3, 7]. In the current study, there was no difference between the F and S resin materials in respect of HEMA, whereas there was seen to be greater release of BPA and Bis-GMA monomers in F material, and of TEGDMA and UDMA monomers in S resin material. Therefore, taking the potential toxicity of BPA and Bis-GMA into consideration [37], the importance of the washing procedure for F resin material must be emphasized again. With increasing washing time, RM decreased in both resins, and thus it can be predicted that the cytotoxic potential would also decrease. However, it has been reported that for RMs in the resin material to be able to create a toxic effect, there should be more than one-tenth of the methacrylic groups remaining (approximately >1.5–5.5%) [14]. Therefore, it can be stated that the tested 3D resins did not exhibit toxic potential, as the monomer release was well below 0.01% in almost all samples.

In a very recent study by Liu et al., with the thought that a washing process with organic solutions could damage 3D printed resin polymer, and there could be a possible risk to human health, different washing solutions (water, detergent, alcohol) were investigated for a 3D printed resin that could be washed with water. It was concluded that the use of deionized water for prolonged washing protected the mechanical properties better, created a smooth surface, improved the DC, and provided the same biological performance as washing for 20 min with organic solutions [19]. The results of the current study can be considered to contribute valuable information to the literature in terms of the development and use of water-based solutions.

Following the manufacturer's instructions in the preparation of the tested materials is of great importance in respect of both research and evidence-based dentistry. Therefore, this study was performed in adherence to the production and post-production procedures recommended by the manufacturers, and thus clinical suitability was taken into consideration. However, that both washing solutions were not used on both resins constitutes a limitation of this study. In addition, when evaluating the physical and chemical properties, it was only possible to evaluate the effects of the washing procedure on the surface properties. There is a need for further studies designed to overcome these limitations.

## Conclusions

These findings suggest that a 2 × 5 min ($T_2$) washing time may be a balanced option for the Formlabs resin material in terms of maintaining mechanical properties while significantly reducing the release of residual monomers, particularly BPA, UDMA, and Bis-GMA. For the Saremco resin material, shorter wash times appear sufficient, as longer wash times do not provide any additional benefit in terms of significant mechanical properties or residual monomer release.

### Abbreviations

| | |
|---|---|
| Alc | Alcohol |
| F | Formlabs 3D Permanent Crown |
| S | Saremco Print Crowntech |
| CC | Cleaning concentrate |
| FS | Flexural strength |
| VH | Vickers hardness |
| Wsp | Water sorption |
| Wsl | Water solubility |
| DC | Degree of conversion |
| RM | Residual monomers |
| SEM | Scanning electron microscopy |
| EDS | Energy dispersive X-ray spectroscopy |
| BPA | Bisphenol A |
| HEMA | 2-hydroxyethyl methacrylate |
| Bis-GMA | Bisphenol A-glycidyl methacrylate |
| TEGDMA | Tri-ethylene glycol di-methacrylate |
| UDMA | Urethane di-methacrylate |
| Bis-EMA | Ethoxylated bisphenol A di-methacrylate |
| AM | Additive manufacturing |
| SLA | Stereolithography |
| DLP | Digital light processing |
| STL | Standard tessellation language |
| LCM | Lithography-based ceramic manufacturing |
| HPLC-UV | High-performance liquid chromatography-ultraviolet |
| PMMA | Polymethyl methacrylate |

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Conceptualization: Z.S.Y., and S.G.B.; validation: Z.S.Y., Y.U., and S.G.B.; methodology: Z.S.Y. and S.G.B.; formal analysis and investigation: Z.S.Y. and S.G.B.; writing—original draft preparation: Z.S.Y. and S.G.B.; writing—review and editing: Z.S.Y., Y.U., and S.G.B.; resources: Z.S.Y., Y.U., and S.G.B.; supervision: Z.S.Y. and Y.U Final approval of the version to be published: All the authors have approved the final draft of the manuscript to be published.

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### Data availability

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request due to privacy reasons and large data size.

## Declaration

### Ethics approval and consent to participate

The samples utilized in this study were resin-based materials, and no living organisms were involved. Therefore, ethical approval was not required.

**Consent for publication**
Not applicable.

**Competing interests**
The authors declare no competing interests.

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