Original Article

# Comparison of the Water Sorption and Solubility of Four Reline Acrylic Resins after Immersion in Food-Simulating Agents

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ABSTRACT

**Objectives:** Water sorption and water solubility adversely affect the mechanical properties and biocompatibility of the denture material. This study aimed to evaluate the water sorption and solubility of three direct hard reline acrylic resins and a heat-curing one after immersion in food-simulating agents.

**Materials and Methods:** This study was performed on four groups of samples ($n$ = 10 per group). The samples were made of three direct hard reline acrylic resins (TDV Cold Liner Rebase, Tokuyama Rebase II Fast, GC Reline Hard) and a heat-curing one (Meliodent). Each group was divided into four subgroups ($n$ = 10) to undergo 7-day immersion in distilled water, 75% ethanol/water, 0.02 N citric acid, and heptane. Water sorption and solubility were calculated according to Oysaed and Ruyter formula. The statistical analyses were done by using SPSS software (version 22). Kruskal–Wallis H Test and Dunn's test were used to detect any significant difference among the groups ($P < 0.05$).

**Results:** The median range of water solubility and water sorption values were −0.87–4.92 and 3.75–27.25 μg/mm$^3$, respectively. The median solubility and sorption values of different resins differed significantly in the same solution ($P < 0.05$). Besides, immersion in different solutions caused significant differences in the median solubility and sorption values of each reline material ($P < 0.05$), except for Meliodent whose solubility was not significantly affected by different solutions ($P = 0.16$).

**Conclusions:** Water sorption and solubility values of the tested hard reline resins were within the range of International Standards Organization 1567:1999. Given the low sorption and solubility values, these hard reline materials can be safely used in clinical situations.

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## INTRODUCTION

The ongoing bone resorption in edentulous patients leads to a space between the denture base and, which jeopardizes the denture retention and stability. Ridge resorption of the removable partial dentures also causes tissue-ward rotation of the distal extension base around the fulcrum line which transmits the detrimental torquing forces on the abutment.[1] Therefore, the significance of improving denture fit necessitates periodic services such as reline in denture wearers. The conjunction of indirect or compression molding technique and heat-curing acrylic resin is the standard method for relining. Proper color stability, low water sorption and solubility values, low toxicity (ideal biocompatibility), and simple processing technique are some benefits of heat-curing acrylic resins.[2]

Among the shortcomings of indirect method are dimensional changes and therefore inaccuracy of the denture base fitness which are the results of highly

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stressful compression polymerization technique.[2,3] Direct method by application of chemically-activated acrylic resins is faster and less expensive than the laboratory reline systems. Nor does it require the patient to discontinue using the denture for a while. Moreover, the direct relining procedure does not result in dimensional instability of the denture.[1] This technique is increasingly demanded by patients, and it has given rise to the evolution of new direct reline materials in recent years. However, when employing the direct method with these autopolymerizing resins, close attention should be paid to the physical and mechanical properties such as their water sorption and solubility.[1,4]

Based on the previous studies, the absorbed water diminishes the mechanical properties such as hardness, transverse strength, and fatigue limit due to the plasticizing effect of water.[5-7] In addition, water sorption affects the dimensional stability through a three-dimensional volumetric expansion and causes occlusal changes.[8] The dimensional change predisposes the appliance to internal stresses and negative consequences such as crack formation and denture fracture.[9,10] Water sorption into polymer is the result of molecular polarity in the polymer, unsaturated bonds of the molecules, or unbalanced intermolecular forces in the polymers.[11] It causes reversible rupture of ill-qualified interchain bonds and irreversible flaws in the polymer matrix.[12] Water sorption in moderate amounts releases the internal stresses induced by polymerization shrinkage, compensates for the shrinkage, and consequently, improves the marginal seal.[13-16]

According to the International Standards Organization (ISO) 1567:1999, water sorption must not exceed 32 μg/mm$^3$ for both heat-cure and self-cure materials.[17,18] Water solubility of the denture base acrylic resins is the result of free monomers and water-soluble additives percolating into oral fluids.[19] The material weight loss which is called solubility is the result of this leaching process.[20] Furthermore, chemical toxic materials such as formaldehyde and methacrylic acid are released through this process.[21,22] The released monomers sometimes induce soft-tissue reactions such as erythema, erosion, and mucosa irritation which affect the material biocompatibility.[23] Solubility must not exceed 1.6 μg/mm$^3$ for heat-curing and 8 μg/mm$^3$ for self-curing materials.[17,18]

Although, water sorption and solubility of dental materials such as composite resins, compomers, glass ionomer, resin-modified glass ionomer, luting cements, and denture base, acrylic resins has been the area of interest for many researchers up to now,[24-26] the authors could not find any published data about water sorption and water solubility of hard reline materials in food-simulating agents (FSAs). Therefore, the present study was designed to assess the water sorption and water solubility of hard reline materials after immersion in distilled water, 75% ethanol/water, 0.02 N citric acid, and heptane. The null hypothesis was that FSA could not affect the water sorption and water solubility of direct hard reline materials.

## Materials and Methods

This *in vitro* study was conducted on four types of frequently used hard reline acrylic resins, three of which were self-curing and one was heat-polymerizing. Table 1 displays the properties of the acrylic resins tested in this study.

Brass molds (1 mm × 5 mm, thickness × diameter) were used to prepare the wax patterns (Polywax, Bilkim Chemical Company, Izmir, Turkey). Forty wax patterns were invested in dental stone (Dental Model Stone, TARA 250, Kheyzaran co. Ltd, Isfahan, Iran) to form molds for preparing the Meliodent specimens. Meliodent was mixed, packed, and cured according to

**Table 1: Detailed properties of the tested acrylic resins**

| Material | Polymerization mode | Composition | | Powder/liquid ratio (g/ml) | Manufacturer |
|---|---|---|---|---|---|
| | | Powder | Liquid | | |
| TDV Cold Liner Rebase | Self-cure | PEMA | DMPT, BHT, IBMA | 1.2/0.94<br>5 min in 37°C | TDV Dental Ltd, Pomerode, Brazil |
| Tokuyama Rebase II fast | Self-cure | PEMA | AAEM, ND | 2.4/1.0<br>5 min in 37°C | Tokuyama Dental Corp., Tokyo, JAPAN |
| GC Reline hard | Self-cure | PEMA | BE, BME, HDMA, TDEA | 1.8/1<br>5-6 min in 37°C | GC America, incorporation, Alsip IL, USA |
| Meliodent | Heat-cure | PMMA | EGDMA | 35/1<br>90 min in 73°C<br>30 min in 100°C | Heraeus Kulzer, Hanau, Germany |

DMPT=Dimethyl-p-toluidine, BHT=Butylated hydroxytoluene, IBMA=Isobutyl methacrylate, AAEM=2-(acetoacetoxy) ethyl methacrylate, ND=1,9-nonanediol dimethacrylate, BE=Butoxy ethyl, BME=Benzoyl methacrylate, HDMA=1,6-hexanediol dimethacrylate, TDEA=P-Tolyldiethanolamine, EGDM=Ethylene glycol dimethacrylate, PEMA=Poly (ethyl methacrylate), PMMA=Poly (methyl methacrylate) the manufacturer's instruction. Then, the specimens were retrieved from the flasks.

Having mixed the self-curing materials according to the manufacturer's instruction, they were placed into prefabricated plastic molds and pressed between two glass slabs by hand pressure to remove the air bubbles and excess material. The glass slabs were separated from the filled molds using a plastic sheet. After polymerization, the specimens were finished and polished using 240-grit silicon carbide paper (Pace Technologies, Tucson, USA). They were set to be 5 mm in diameter and 1.0 mm in thickness by means of a digital caliper (Altas 905; Gedore-Altas, Istanbul, Turkey) with 0.01-mm accuracy.

All of the specimens were placed in a desiccator (glass vacuum; 25 cm, Isolab, Germany) containing fresh colloidal silica gel ($SiO_2$) at 37°C for 48 h. Then, they were stored in a desiccator at 23°C for 1 h and weighed with a calibrated electronic balance of 0.002-g accuracy (GR-300; A and D Company Ltd., Japan). The drying procedure was repeated until a constant mass (m1) for each specimen was gained. The specimens of each resin material were randomly divided into four subgroups ($n$ = 10).

The resin materials were immersed in four different storage media including distilled water, 75% ethanol/water, 0.02 N citric acid, and heptane [Table 2] and placed in digital incubator (ES250; 264 litter, NUVE, Turkey) at 37°C for 7 days. Each specimen was periodically weighed until a constant weight (m2) was obtained. Then, the specimens were removed from the storage media and reinserted in the desiccator at 37°C until a constant weight was obtained. Finally, they were dried at 60°C for 24 h and reweighed (m3).

The water sorption and solubility were calculated through the Oysaed and Ruyter formula[27] as follows:

$$\text{Water solubility} = \frac{m1 - m3}{\text{v}}$$

Where m1 was the sample weight before immersion in solutions, m3 was the sample final weight after being removed from the solution and drying in desiccator, and V was the sample volume. Meanwhile, the water sorption was calculated for each sample according to the following formula:

$$\text{Water sorption} = \frac{m2 - m3}{v}$$

Where m2 was the sample weight after immersion in solutions and m3 and V were as previously defined. The sample volume was calculated according to the formula V = $\pi r^2 h$ in which r was the sample radius, h was the sample thickness, and π was 3.14.

**Table 2: The employed food-simulating solutions**

| Food-simulating solutions | Formula | Manufacturer |
|---|---|---|
| Distilled water | $H_2O$ | SKG, Sina, Shiraz, Iran |
| Ethanol | $C_2H_5OH$ | Nasr Alcohol, Khoram Aabad, Iran |
| Citric acid | C3H4(OH)(COOH) 3H2O | Shimiran, Kimia Mavad, Tehran, Iran |
| Heptane | CH3(CH2) 5CH3 | Daejung Chemicals and Metals Co., Ltd, Korea |

The obtained data were fed into SPSS software (version 22; SPSS Inc., Chicago, IL, USA), for statistical analysis. Kruskal–Wallis H test was performed to compare the water sorption and water solubility among the groups. Dunn's test was used for pairwise comparison of the groups. The significance level was set at 0.05.

## Results

The median solubility values of hard reline materials ranged from −0.87 to 4.92 μg/mm³. The median solubility values (μg/mm³) and interquartile range are represented in Table 3. Figures 1 and 2 display the median solubility values (μg/mm³) for the tested acrylics in different and same solutions, respectively. As shown in Table 4, the median sorption values of the tested reline resins were 3.75–27.25 μg/mm³. The median sorption values (μg/mm³) for the tested acrylics in the same and different solutions are shown in Figures 3 and 4, respectively.

Regarding the pairwise comparison of the groups, Dunn's test revealed the median solubility and sorption values to be significantly different among the four acrylic resins in the same solution ($P$ < 0.05). Significant differences were also observed in the median solubility and sorption values of each tested acrylic resin after storage in different FSAs ($P$ < 0.05). Meliodent Heat Cure was the only exception, that is, no significant difference was detected in its solubility after storage in different solutions ($P$ = 0.16). The highest and lowest median solubility values were observed for TDV in citric acid and GC Reline Hard in ethanol, respectively. Besides, the highest and lowest median sorption values were found in Tokuyama Rebase II and Meliodent, respectively, both in heptane. GC Reline Hard showed the least solubility after storage in FSAs compared with other tested resins.

## Discussion

Several studies have investigated the water sorption and water solubility of the materials used in fabricating the prosthesis. Khaledi *et al*.[7] showed that FSA could influence the hardness and bond strength of silicon soft liners to denture base. In another study, it was

**Table 3: The median solubility values (μg/mm³) of the tested acrylic resins in different solutions (the interquartile range shown in parenthesis)**

| Acrylic resins | Distilled water | 75% ethanol-water | Citric acid | Heptane |
|---|---|---|---|---|
| TDV Cold Liner Rebase | 4.50[a,1] (1.10) | −0.15[b,1,2] (2.46) | 4.92[a,1] (2.07) | −0.24[a,b,1,2] (8.62) |
| Tokuyama Rebase II Fast | 0.79[a,2] (0.62) | −0.05[a,b,1,2] (0.65) | 0.41[a,b,2] (1.05) | −0.26[b,1,2] (0.31) |
| GC Reline hard | 0.48[a,2] (1.04) | −0.87[b,1] (0.71) | 0[a,b,2] (1.09) | −0.41[a,b,1] (0.30) |
| Meliodent | 1.60[a,1,2] (2.16) | 1.37[a,2] (2.25) | 0.86[a,1,2] (1.86) | 0.48[a,2] (0.99) |

Different letters and numbers show significant differences between the solutions for each material and between the materials in the same solution, respectively ($P<0.05$)

**Table 4: The median sorption values (μg/mm³) of the tested acrylic resins in different solutions (interquartile range shown in parentheses)**

| Acrylic resins | Distilled water | 75% ethanol-water | Citric acid | Heptane |
|---|---|---|---|---|
| TDV Cold Liner Rebase | 7.54[a,1] (0.86) | 26.06[b,1] (7) | 8.91[a,c,1] (4.90) | 20.74[b,c,1,2] (6.43) |
| Tokuyama Rebase II fast | 11.33[a,2,3] (0.68) | 12.22[a,c,1,2] (12.65) | 11.28[a,1,2] (3.69) | 27.25[b,c,1] (3.79) |
| GC-Reline hard | 9.08[a,1,2] (1.59) | 13.40[b,1,2] (18.02) | 10.59[a,b,1,2] (0.84) | 17.28[b,2,3] (5.99) |
| Meliodent | 20.83[a,3] (2.48) | 15.23[a,b,2] (16.65) | 18.38[a,b,2] (4.85) | 3.75[b,3] (0.48) |

Different letters and numbers show significant differences between the solutions for each material and between the materials in the same solution, respectively ($P<0.05$)

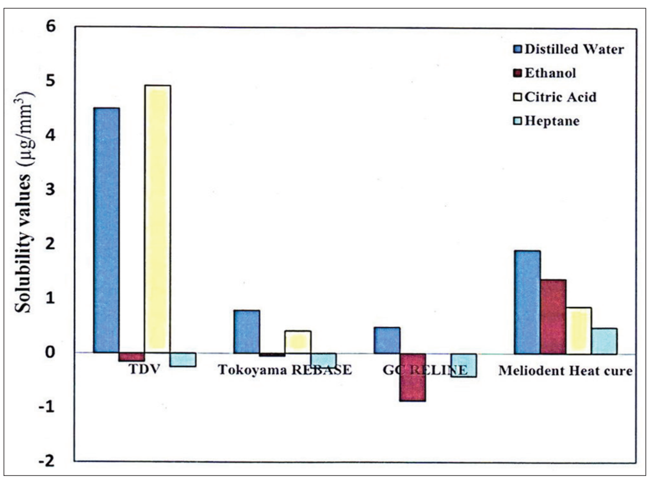


**Figure 1:** Comparison of median solubility values (μg/mm³) of dental acrylics in different solutions

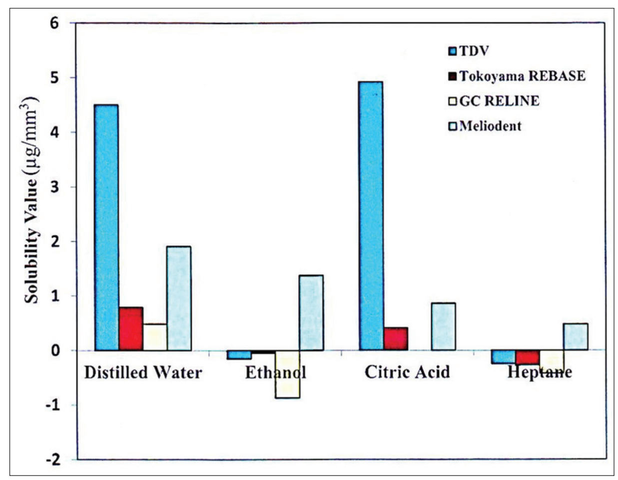


**Figure 2:** Median solubility values (μg/mm³) of dental acrylics in each solution

demonstrated that FSA affected the flexural strength and surface hardness of denture acrylic resins.[9] Cucci *et al*.[5] showed that water sorption and the solubility of reline acrylic resins, after storage in water for 7 days were within specification limits. However, no study had evaluated the effect of FSA on the water sorption and solubility of direct reline materials. Therefore, the present study evaluated the effects of four FSAs on water sorption and solubility of three direct reline materials and one heat-curing resin. The null hypothesis was rejected as the FSAs significantly changed the water sorption and water solubility of the hard reline materials. Significant differences were found in the median solubility and sorption values of the tested reline resins in the same solution. Moreover, immersion in different solutions caused significant differences in the median solubility and sorption of each resin material.

Reline of removable prosthesis is a necessary inevitable procedure in denture wearers. Self-curing reline acrylic resins are easy to use and time- and cost-effective for the patients. However, exposure of restorative and prosthodontic materials to food, saliva, and beverages could affect the physical and mechanical properties of these materials. Therefore, interaction of dental materials with different FSAs should be highly considered.[7] If self-curing acrylic resins are used to save time and money, their water sorption and water solubility should be meticulously evaluated because these features have the potential to negatively affect the material durability. Water sorption decreases the mechanical properties and water solubility and negatively affects the material biocompatibility. Water sorption is measured according to the mass gain per

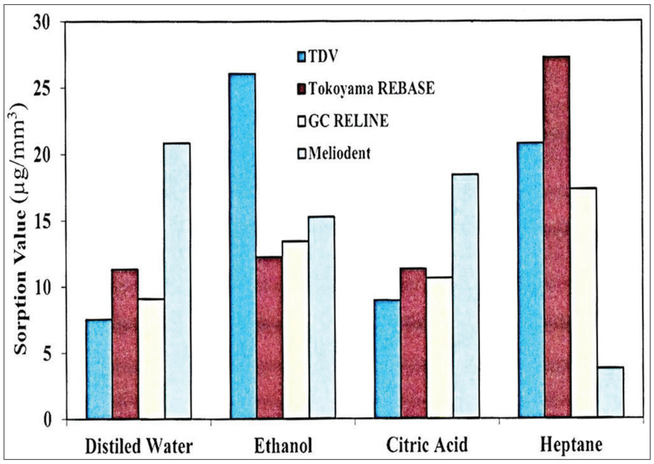


**Figure 3:** Comparison of median sorption values (µg/mm$^3$) of dental acrylics in each solution

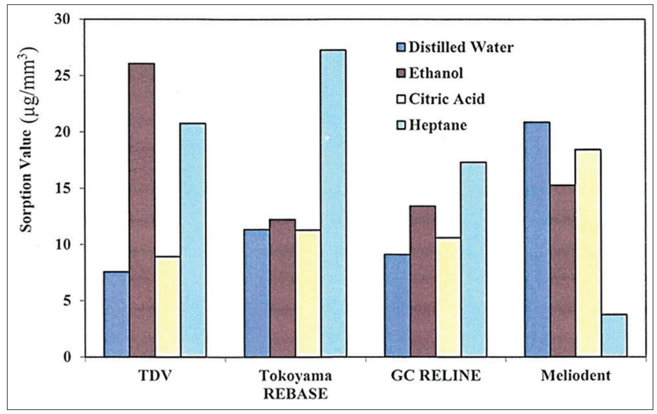


**Figure 4:** Comparison of median sorption values (µg/mm$^3$) of dental acrylics in different solutions

unit volume and water solubility is determined by measuring the polymer mass loss.[17,18]

In the current study, the testing solutions were chosen according to the Food and Drug Association guidelines. Distilled water was used to simulate saliva and water, heptane represented butter, fatty meat, and vegetable fats; meanwhile, the aqueous ethanol and citric acid solution simulated the environment created by alcoholic drinks, vegetables, fruits, candy, and therapeutic syrups.[3,28] The reline acrylic resins in the oral environment are exposed to these solutions constantly or sporadically. Occasional exposures occur during eating and drinking and go on as long as the denture surface is clean. Meanwhile, chemical solutions are absorbed by adherent food particles and calculus that are attached to the denture, then released over time and expose the acrylic resins constantly. In addition, chemical media can be held around the margins, into acrylic porosities, under the dentures, or be produced during bacteria disintegration of debris.[29-31]

Guler *et al*.[32] reported that 7-day immersion in FSAs corresponded 7-month usage. Changes in the mechanical and physical properties of dental materials after immersion in water and other solutions depend on several factors including the chemistry of resin monomer, polymeric matrix polymerization percentage (residual monomer), the filler size, shape, and distribution, and the interfacial characteristics between the filler and resin matrix which relies on the contact area of resin matrix.[33-36] The extent and rate of water sorption are predominantly controlled by the resin polarity, dictated by the concentration of polar sites available to form hydrogen bonds with water and network topology.[37,38]

Dixon *et al*.[39] detected that the residual monomer affected water sorption and its consequent expansion. They reported that higher liquid to powder ratio could result in higher residual monomer and lower water sorption. On the other side, Jagger *et al*.[40] showed that the higher the residual monomer, the more the water sorption would be and vice versa. Although the present study showed that the water sorption of acrylic resins lies within the ISO accepted range, further studies are recommended to determine the amount of residual monomer in different resin materials after storage in different solutions and also their effect on water sorption, since it seems that the food materials could unpredictably affect the amount of residual monomer.

Craig *et al*.[19] demonstrated that the acrylic resin solubility was the result of unreacted monomer leakage into the oral fluids. Fletcher *et al*.[41] showed that the higher residual monomer of self-curing resins was the cause of their higher solubility compared with the heat-curing ones. In the current study, although Meliodent revealed higher solubility than the direct resin materials, the amount of its solubility was relatively the same after storage in different solutions. It can be concluded that in acrylic resins, both water solubility and water sorption can be influenced by some modifying factors such as food-simulating liquids. However, according to the current results, with less residual monomer, more constant water solubility is expected after immersion in different solutions (like Meliodent).

Further studies are also recommended to investigate the effects of different contents of direct reline materials on their mechanical and physical properties. Wady *et al*.[42] revealed that autopolymerizing acrylic resins which contained isobutyl methacrylate (IBMA) monomer in the liquid had the lowest impact strength among all the tested acrylic resins. In a study by Cucci *et al*.,[5] direct reline materials containing IBMA showed lower water solubility despite lower transverse bond strength compared with other resins. However, in the current study, TDV which contained IBMA had the highest solubility values in citric acid and water although within the ISO recommended range.

On the other side, it was reported that cross-linking agents could improve the mechanical properties of resins. In Arima *et al*. study,[43] 1,6-hexanediol dimethacrylate (1,6-HDMA) decreased the water sorption of reline material. In Azevedo *et al*.'s study,[44] the mean shear bond strength of Tokuso Rebase Fast and Ufi Gel Hard which contained HDMA, increased after 90 days of immersion in water. Wady *et al*.[42] observed an improvement in the impact strength of Ufi Gel Hard containing HDMA. In agreement with these studies, the present study detected the lowest water solubility value in GC Reline Hard which contained HDMA.

In the current study, some reline materials showed negative solubility values after immersion in some solutions. Tuna *et al*.[45] stated that the water absorbed by some acrylic resins could not be released and concluded that the components of these materials chemically bonded to water molecules. They believed that this fact would justify the negative values of water solubility of these materials.

Sometimes, it is impossible to make a direct and quantitative comparison between different studies about water sorption and solubility of resin materials since these studies have inevitably obtained different results over different time spans, different size of samples, and expression in different units.[46] Different size of samples allows for complete penetration of water into polymer matrix, different time elapses, and more water absorbed by the material necessitates more time for stabilization.[47]

A weakness of water sorption test is that it simply assumes that a weight gain in the sample corresponds to water gain; whereas, in reality, weight gain is the difference between water gain and dissolution of low molecular weight monomers. Therefore, the true water sorption values may be somewhat higher than those reported.[48] Meanwhile, manipulation and constant handling of samples causes a little abrasion on the sample surface and decreases the sample weight.[49] Hence, studies aiming to determine the water sorption and solubility of resin-based materials are important for their relative values, and they are sometimes impossible to be quantitatively compared.[50] Particularly because there was no similar study on the effect of FSA on direct reline materials, the findings of the current study in not comparable to those of any other study.

In the current study, the specimens were not exposed to artificial aging; therefore, future studies are suggested to simulate the aggressive oral environment through thermomechanical cycling. In addition, the determination of the effects of food agents on other mechanical and physical properties is also necessary for the clinical success of direct reline materials. Furthermore, the immersion period should be longer to mimic long-term application. Compatibility between liners with beverages and food should be studied to protect adverse effects. The findings of these studies could help the clinicians and consequently the patients if the prostheses are expected to function over an extended period of time. Therefore, further *in vivo* and *in vitro* studies are recommended.

## Conclusion

According to the results of this study, significant differences of water sorption and solubility exist not only among different direct reline acrylic resins in the same solution but also among the same resin in different solutions. Although water sorption and solubility values of hard reline materials were within the clinically acceptable range, clinicians should warn the patients about the possible effect of certain foods on prostheses relined by direct hardliners.

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### Conflicts of interest

There are no conflicts of interest.

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