Comparative study of sorption and solubility of heat-cure and self-cure acrylic resins in different solutions

Rajesh Saini, Ravindra Kotian, Prashanthi Madhyastha, Srikant N

ABSTRACT

Objective: The objective of this study was to compare the sorption and solubility of heat-cure and self-cure acrylic resins in different solutions.

Materials and Methods: One heat-cure acrylic resin (Trevalon) and one self-cure acrylic resin (Rapid Repair) were studied. Five groups of square-shaped specimens (20 mm × 20 mm × 2 mm) were prepared for each acrylic resin and then immersed in five solutions: distilled water, artificial saliva, denture cleansing solution, distilled water, and denture cleaning solution for 12 h alternatively, artificial saliva and denture cleaning solution for 12 h alternatively at 37 ± 2°C, and tested sorption and solubility by weight gain/loss method, respectively, after 1, 6, and 11 weeks. The data were analyzed by one-way analysis of variance followed by post hoc Tukey’s test.

Results: Water sorption mean values varied from 17.5 ± 0.88 to 27.25 ± 1.04 µg/mm³ for heat cure and from 12.75 ± 0.55 to 19.75 ± 1.04 µg/mm³ for self-cure in the different solutions after different interval periods of 1, 6, and 11 weeks. These values were statistically significant (P < 0.001). Water solubility mean values varied from 0.25 ± 0.55 to 1.5 ± 0.55 µg/mm³ for heat cure and from 1.5 ± 0.55 to 6.5 ± 0.55 µg/mm³ for self-cure in the different solutions after different interval periods of 1, 6, and 11 weeks. These values were statistically not significant (P > 0.05). There was no linear correlation between sorption and solubility values. Overall, analysis of results showed the maximum sorption value in denture cleansing solution followed by alternative soaking in distilled water and artificial saliva. Least sorption was observed with artificial saliva followed by distilled water.

Conclusion: Both heat-cure and self-cure acrylic resins showed varying water sorption and solubility. The results of both water sorption and solubility showed compliance with the International Standards Organization specification. No correlation was found between water sorption and solubility. Artificial saliva solution is a better storage medium than distilled water and denture cleansing solution for both heat-cure and self-cure acrylic resins.

Key words: Acrylic resin, heat-cure acrylic, self-cure acrylic, solubility, water sorption

Acrylic resin is the most utilized material in partial removable and complete denture bases and facings for fixed bridges to improve the esthetic value of the restoration. Prediction of the service life of acrylic resin material is difficult since many environmental factors affect the durability. One of the properties of acrylic resins is water sorption and release, which causes dimensional instability, thereby subjecting the material to internal stresses that may result in crack formation and eventually fractures of the denture.¹²

Acrylic resins absorb water slowly over a period of time. This imbibition is due primarily to the polar properties of the resin molecules.³⁴ Sorption of material represents the
amounts of water adsorbed on the surface and absorbed into the body of the material during fabrication or while the restoration is in service. Since both adsorption and absorption are involved, the term sorption is generally used to include the total phenomena. Usually, a serious warpage and dimensional change in the material are associated with a high percentage of water sorption. 

Dimensional change that occurs on prostheses as a result of water absorption of acrylic is an alternate event. If the prosthesis is left in open air and a dry place, it allows water to leave its structure and undergoes contraction. Therefore, for patients using removable prostheses, it is recommended to store the prosthesis in water when they are not in use. The water absorbed into the material acts as a plasticizer and decreases the mechanical properties such as hardness, transverse strength, and fatigue limit.  

According to the rules of diffusion, water sorption can be explained in the diffusion forces of the resin molecules to separate from each other. This, in turn, creates an expansion in size with water helping to relax the stresses with the acrylic resin.  

Solubility represents the mass of the soluble materials from polymers. The only soluble materials present in denture base resins are initiators, plasticizers, and free monomer. Any observed loss of weight of the resin is the measure of the specimen’s solubility. Therefore, the water sorption and solubility are the critical problems that affect the durability.  

Takahashi et al. found that water molecules spread between the macromolecules of the material, forcing them apart. This behavior affects the dimensional behavior and denture stability; therefore, water sorption and solubility of these materials should be as low as possible. Ideally, polymer networks should be insoluble materials with relatively high chemical and thermal stability. However, most of the monomers used in dental resin materials can absorb water and chemicals from the environment, and also release components into the surrounding environment.  

Both water sorption and solubility would lead to a variety of chemical and physical processes that may result in deleterious effects on the structure and function of dental polymers. Denture base acrylic resins have low solubility, and the little that occurs is a result of the leaching out of the traces of unreacted monomer and water-soluble additives into the oral fluids. However, these monomers sometimes produce a soft tissue reaction. It is important to determine the residual monomer content and solubility of the tested materials as these properties influence the allergy susceptibility of these materials.  

The water sorption and solubility were determined according to the International Standards Organization (ISO) standards 1567:1999. According to this specification, water sorption should not exceed 32 µg/mm³ for heat-cured or self-cured materials. The loss in mass per unit volume (soluble material) should not exceed 1.6 µg/mm³ for heat-cured and 8.0 µg/mm³ for self-cured materials.  

Because water sorption and solubility play a very important role in the clinical and mechanical performance of acrylic materials, it was, therefore, decided to study these properties of heat-cure and self-cure acrylic resins in different solutions. The aim of this study was to compare the sorption/solubility of heat-cure acrylic resin and self-cure acrylic resin in the different solutions after 1 week, 6 weeks, and 11 weeks.

**MATERIALS AND METHODS**

In this study, one heat-cure and one self-cure acrylic denture base materials were selected for comparative evaluation of sorption and solubility in different solutions at different time intervals. The materials and solutions selected are given in Table 1.  

For the study, 25 samples each with dimensions of 20 mm × 20 mm × 2 mm were prepared using heat-cure and self-cure acrylic resins separately for the different time intervals. For each solution, five samples were used. Gypsum molds were used to prepare the samples by using a preformed wax pattern of standard dimension.  

**Gypsum mold preparation**

Wax patterns measuring 20 mm in length, 20 mm in width, and 2 mm in thickness were used to prepare a gypsum mold. Wax patterns were invested in the dental stone in the base of the flask. Two coatings of alginate separating media (cold mold seal) were then applied onto the set dental stone. The counterpart of the flask was positioned over the base and filled with dental stone. The flask was placed on top of the vibrator, taking care not to cause air entrapment.

The flask was clamped immediately to ensure metal-to-metal contact between the base and the counterpart of the flask. After setting, the wax was removed by immersing in a hot water bath without damaging the molds. The molds formed were then flushed with hot water to remove any traces of residue followed by the application of separating media. The mold cavities so obtained were used for the preparation of acrylic resin specimen.

**Preparation of Trevalon - heat-cure acrylic resin specimen**

The appropriate amount of Trevalon (Dentsply India Pvt., Ltd., Bangalore, India) heat-cure acrylic resin required was
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Table 1: List of materials, batch number, and manufacturers

| Material type                           | Material brand name | Batch number | Manufacturer                        |
|-----------------------------------------|---------------------|--------------|-------------------------------------|
| Heat cure acrylic denture base resin    | Trevalon            | T121037      | Dentsply India Pvt. Ltd.             |
| Self-cure acrylic denture base resin    | RR                  | R130102      | Dentsply India Pvt. Ltd.             |
| AS                                       | Wet mouth           | C12002       | ICPA Health Products Ltd.           |
| Denture cleansing tablets                | Fitty dent          | FDT13024     | Group Pharmaceuticals Ltd.          |
| DW                                       | Demineralized water | MD112        | MediLife Chemicals                  |

RR=Rapid Repair, DW=Distilled water, AS=Artificial saliva

Prepared from a mixture of polymer and monomer in the ratio of 3:1 by volume. The monomer was poured into a mixing jar and the polymer was slowly added to allow for wetting of the powder. Excess powder was removed. Then, it was thoroughly mixed for 1 min. After attaining the dough forming time, the dough was thoroughly kneaded between the fingers and the mold cavities were filled. The flask was closed and trial closure was carried out using flask clamp. The flask was then clamped, and the pressure was maintained for 30 min to allow proper penetration of monomer into polymer.

The flask was immersed in an acrylizer (Confident Dental Equipment Ltd., Bengaluru, Karnataka, India) at room temperature. The temperature was raised to 74°C for 1.5 h, and then the temperature of the water bath was increased to boiling (100°C) for an additional hour.

After the completion of the curing cycle, the flask was removed from the water bath, bench-cooled for 30 min, and immersed in cold water for 15 min prior to deflasking. The acrylic specimens were then retrieved, finished, and polished. The dimensions and quality of the specimens were verified. Twenty-five specimens of 20 cm × 20 cm × 2 mm dimension were obtained by this procedure.

Method to obtain sorption and solubility

The specimens were stored in different solutions [Table 1] at 37 ± 2°C and sorption and solubility were tested by the weight gain/loss method, respectively, after 1, 6, and 11 weeks.

Sorption and solubility were determined by the method described in the American Dental Association (ADA) specification 12 for denture base polymers.[16]

Each sample was dried in desiccators containing anhydrous calcium chloride until a constant weight was obtained. This was considered to be the initial weight of the specimen (W₁).

The samples were then immersed in:
- Solution 1: Distilled water
- Solution 2: Artificial saliva
- Solution 3: Denture cleansing solution
- Solution 4: Distilled water and denture cleaning solution for 12 h alternatively
- Solution 5: Artificial saliva and denture cleaning solution for 12 h alternatively.

Of the processed samples, five each for both heat-cure and self-cure were placed in sealed polyethylene containers each containing different solutions for 1, 6, and 11 weeks at 37 ± 2°C.

After each time period, respectively, the samples were removed from their container, excess water was removed by blotting with filter paper, and the samples were weighed by an electronic precision balance (Essae Teraoka Limited, Gurgaon, India) capable of measuring to 0.001 g. This was the weight of the specimen after absorption (W₂).

The amount of soluble material lost was measured by placing the specimens back in the desiccators after each sorption cycle and then weighing them at regular intervals until a constant weight after desiccation (W₃).

The water sorption and water solubility per unit volume are determined according to the ISO standards 1567:1999.[14]
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({\text{\mu}g/mm^3}) sorption: \( W_2 - W_3/V_{\text{Sample}} \)

({\text{\mu}g/mm^3}) solubility: \( W_1 - W_3/V_{\text{Sample}} \)

\( V_{\text{Sample}} = 20 \text{ mm} \times 20 \text{ mm} \times 2 \text{ mm} = 800 \text{ mm}^3 \)

\( W_1 = \) The initial weight

\( W_2 = \) The weight after absorption

\( W_3 = \) The final weight after desiccation.

The above procedure was repeated, and sorption and solubility data were collected for 1, 6, and 11 weeks. Mean values and standard deviation of both sorption and solubility were calculated. Mean values were compared statistically with one-way analysis of variance (ANOVA) followed by post hoc Tukey’s test to determine the significant differences among the groups at \( P < 0.05 \) level of significance.

RESULTS

SPSS 20.0 (Version 20, Dentsply India Pvt., Ltd, Bangalore, India) was used to obtain the statistical results of our study. One-way ANOVA test followed by post hoc Tukey’s test was used to evaluate the sorption and the solubility values of the heat-cure and self-cure resins at a time period of 1, 6, and 11 weeks.

The results of mean and standard deviation are presented in Tables 2-4 that show the consolidated results of ANOVA/post hoc test for sorption of heat-cured and self-cured resins, respectively. Analysis of the results showed a significant difference in the sorption values at all time periods as assessed by one-way ANOVA. No significant difference was observed in the solubility values in heat-cure and self-cure resins separately. There was no linear correlation between sorption and solubility values.

According to ANOVA, the type of material, time, and solution of storage were statistically significant on water sorption and water solubility \( (P < 0.001) \).

According to post hoc Tukey’s test, the water solubility between the time and solution of storage was not statistically significant \( (P > 0.05) \).

When the values of water sorption and solubility were observed according to time, there was a significant increase in the values of all samples during the 1st week of the study.

For heat cure, no linear increase in water sorption was found during the immersion period of 1 to 11 weeks. Most of the increase occurred during the first 6 weeks compared to weeks 6–11 (19.6 - 25.05 - 25.8 ANOVA). Whereas a linear increase in water sorption was observed in self-cure from 1 to 11 weeks (14.3 - 15.75 - 17.85 ANOVA). Water sorption values for heat cure were significantly higher than self-cure in the different interval periods in different solutions \( (P < 0.001) \).

Water solubility values for both heat-cure and self-cure resins increased significantly from 1 to 11 weeks in different solutions (HC 0.25 - 0.85 - 1.4 and SC 1.5 - 4.35 - 6.35 ANOVA). Water solubility values for heat-cure resins were markedly less when compared to self-cure resins in the different interval periods in different solutions and was statistically significant \( (P < 0.001) \).

Table 2: Mean and standard deviation of the samples

| Brands        | Groups | 1 week          | Mean (SD)          | 6 week          | Mean (SD)          | 11 week         | Mean (SD)          |
|---------------|--------|-----------------|--------------------|-----------------|--------------------|-----------------|--------------------|
| Water sorption (\( \mu g/mm^2 \)) | Heat cure | DW | 20.5 (0.68)*   | 26.75 (0.68)*   | 27.0 (0.68)*   | AS | 17.5 (0.88)*   | 21.25 (0.00)*   | 23.5 (0.55)*   | DC | 22.5 (0.00)*   | 27.25 (1.04)*   | 26.25 (0.88)*   |
|               |        | A ow+DC | 19.0 (0.55)*   | 26.5 (0.55)*   | 27.25 (1.04)*   | AS+DC | 18.5 (1.04)*   | 23.5 (0.55)*   | 25.0 (0.88)*   | DW | 13.75 (0.88)*   | 16.25 (0.00)*   | 19.75 (0.55)*   |
|               |        | AS+DC | 14.75 (1.04)*   | 18.0 (0.55)*   | 19.75 (1.04)*   | DC | 14.25 (0.88)*   | 17.0 (0.68)*   | 18.25 (0.68)*   | DW+DC | 14.0 (1.04)* | 15.0 (0.88)* | 16.0 (1.04)* |
| Water solubility (\( \mu g/mm^2 \)) | Heat cure | DW | 0.25 (0.55)*   | 0.75 (0.68)*   | 1.25 (0.00)*   | AS | 0.25 (0.55)*   | 0.75 (0.68)*   | 1.25 (0.00)*   | DC | 0.25 (0.55)*   | 0.75 (0.68)*   | 1.5 (0.55)*   |
|               |        | DW+DC | 0.25 (0.55)*   | 1.0 (0.55)*   | 1.5 (0.55)*   | AS+DC | 0.25 (0.55)*   | 1.0 (0.55)*   | 1.5 (0.55)*   | AS+DC | 1.25 (0.00)* | 4.0 (1.04)* | 6.0 (0.55)* |
|               |        | AS+DC | 1.5 (0.55)*   | 4.25 (1.11)*   | 6.25 (0.00)*   | DC | 1.5 (0.55)*   | 4.25 (0.68)*   | 6.5 (0.55)*   | DC | 1.5 (0.55)*   | 4.75 (0.55)*   | 6.5 (0.55)*   |

*Values are not statistically significant \( (P>0.05) \). Solution 1=DW, Solution 2=AS, Solution 3=DC, Solution 4=DW+DC, Solution 5=AS+DC, SD=Standard deviation, DW=Distilled water, AS=Artificial saliva, DC=Denture cleanser.

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For heat-cure resins, water sorption mean values varied from 17.5 ± 0.88 to 27.25 ± 1.04 µg/mm³ in the different solutions after different interval periods (1, 6, and 11 weeks). These values were statistically significant (P < 0.001). There was minimum water sorption in artificial saliva (solution 2) at week 1, i.e., 17.5 ± 0.88 µg/mm³ while maximum water sorption was observed in distilled water + denture cleanser (solution 4) at 11 weeks, i.e., 19.75 ± 1.04 µg/mm³.

For self-cure resins, water solubility mean values varied from 1.5 ± 0.55 to 6.5 ± 0.55 µg/mm³ in the different solutions after 1, 6, and 11 weeks. These values were statistically not significant (P > 0.05). There was minimum water solubility in distilled water (solution 1) at week 1, i.e., 0.25 ± 0.55 µg/mm³, while maximum water solubility was observed in distilled water + denture cleanser (solution 4) at 11 weeks, i.e., 1.5 ± 0.55 µg/mm³.

Overall analysis of results showed the maximum sorption value in the denture cleansing solution followed by immersion in distilled water and artificial saliva alternatively. Least sorption value was seen with artificial saliva followed by distilled water.

### DISCUSSION

In the present study, the method recommended by the ISO for measuring water sorption and solubility was used. The water sorption was determined according to an increase in mass per unit volume. In addition, water solubility was determined according to loss of mass from polymers. The soluble materials present in acrylic resins are initiators, plasticizers, and free monomer. It has been suggested that there might be a correlation between residual monomer and the weight loss determined by the solubility test.

Acrylic resins absorb water slowly over a period of time, primarily because of the polar properties of the resin molecules. High equilibrium uptake of water can soften an acrylic resin because the absorbed water can act as a plasticizer of acrylate and reduce the strength of the material. The extent and rate of water uptake into polymer

### Table 3: Consolidated results of analysis of variance/post hoc test for sorption at 1, 6, and 11 weeks for heat-cure resins

| Sorption 1 (%) | DW | AS | DC | DW with DC |
|---------------|----|----|----|------------|
| Sorption at 1 week | AS | Yes (DW) | No | No |
| | DC | Yes (DC) | Yes (DC) | No |
| | DW with DC | Yes (DW) | Yes (DW+DC) | Yes (DC) |
| | AS with DC | Yes (DW) | No | Yes (DC) |
| Sorption at 6 weeks | AS | Yes (DW) | No | No |
| | DC | No | Yes (DC) | No |
| | DW with DC | No | Yes (DW+DC) | No |
| | AS with DC | Yes (DW) | Yes (AS+DC) | Yes (DC) |
| Sorption at 11 weeks | AS | Yes (DW) | No | No |
| | DC | No | Yes (DC) | No |
| | DW with DC | No | Yes (DW+DC) | No |
| | AS with DC | Yes (DW) | Yes (AS+DC) | Yes (DW+DC) |

DC=Denture cleanser, DW=Distilled water, AS=Artificial saliva

### Table 4: Consolidated results of analysis of variance/ post hoc test for sorption at 1, 6, and 11 weeks for self-cure resins

| Sorption 1 (%) | DW | AS | DC | DW with DC |
|---------------|----|----|----|------------|
| Sorption at 1 week | AS | No | No | No |
| | DC | No | Yes (DC) | No |
| | DW with DC | Yes (DW+DC) | Yes (DW+DC) | No |
| | AS with DC | No | No | Yes (DW+DC) |
| Sorption at 6 weeks | AS | Yes (DW) | No | No |
| | DC | No | No | No |
| | DW with DC | No | Yes (DW+DC) | No |
| | AS with DC | No | No | Yes (DW+DC) |
| Sorption at 11 weeks | AS | Yes (DW) | No | No |
| | DC | No | Yes (DC) | No |
| | DW with DC | Yes (DW+DC) | Yes (DW+DC) | No |
| | AS with DC | Yes (DW) | Yes (DC) | Yes (DW+DC) |

DC=Denture cleanser, DW=Distilled water, AS=Artificial saliva
networks are predominantly controlled by resin polarity, dictated by the concentration of polar sites available to form hydrogen bonds with water and network topology.\textsuperscript{[12]}

Arima \textit{et al.}\textsuperscript{[18]} suggested that the chemical nature of the polymer versus that of the water molecule directly affected the water sorption of resin. Water is absorbed into polymers by the polarity of the molecules in the polymers by unsaturated bonds of the molecules or unbalanced intermolecular forces in the polymers. The present study showed the least sorption value (12.75 \(\mu\)g/mm\(^3\)) for self-cure resin in artificial saliva and highest sorption value (27.25 \(\mu\)g/mm\(^3\)) for heat-cure resin in distilled water + denture cleanser.

The solubility value of the heat-cure resin showed the highest value (1.5 \(\mu\)g/mm\(^3\)) in distilled water + denture cleanser and for self-cure resin, the highest solubility value (6.5 \(\mu\)g/mm\(^3\)) was seen in denture cleanser. The principal difference between self-cure and heat-cure resins is that more residual monomer is present in the self-cure resins. In addition, with a high monomer to polymer ratio, residual monomer content in the polymerized acrylic resin would be large.\textsuperscript{[13]}

Fletcher \textit{et al.}\textsuperscript{[19]} found that self-cure resins exhibited higher residual monomer levels than did heat-cure resins. These higher residual monomer contents could be related to the higher solubility levels of self-cure resins reported. It is obvious that the greater amount of residual monomer in self-cure polymethyl methacrylate (PMMA) also affects the solubility values more than heat-cure PMMA does, of which the residual monomer content is considerably lower.\textsuperscript{[15]}

In the present study, heat-cure acrylic resin had lower solubility values than the self-cure acrylic resin. The results of the water sorption and solubility either self-cure or cold-cure acrylic resins were in accordance with the ISO specification.\textsuperscript{[16]}

According to earlier studies, the water sorption of different types of acrylates is 10–25 \(\mu\)g/mm\(^3\). In the present study, the water sorption values were in accordance with these studies.

The rate at which the materials absorbed water or lost soluble components varied considerably with the type of material, the amount of plasticizer or filler content, and the solution in which they were immersed.\textsuperscript{[20]}

This study has demonstrated that the solubility of heat-cure and self-cure resins in artificial saliva with denture cleanser was significantly higher than that in distilled water.

The observed weight loss probably occurred because the plasticizers are more soluble in ionic solutions than in water. Conversely, the percentage sorption of artificial saliva by heat-cure and self-cure resins was significantly lower than that of distilled water (\(P < 0.001\)).

The lower uptake in artificial saliva is explicable in terms of the ionic impurities in the polymer. This leads to an enhanced uptake in distilled water since water droplets will form at the impurity sites until elastic and osmotic forces balance.

The osmotic pressure will be proportional to the difference in ionic concentrations between the polymer and external liquid, this difference being greater for water than for artificial saliva.\textsuperscript{[20]} Acrylic resins undergo two processes when immersed in water. Plasticizers and other soluble materials have leached into the water, and water is absorbed by the polymer. The balance between these two processes affects both the compliance and dimensional stability of the materials.

The absorption of water by the material results in a weight and volume increase. The compliance of the materials tested is dependent either on the presence of a plasticizer as in the case of the acrylic materials. An ideal material should, therefore, have no component which is soluble in saliva or water and should have a low level of absorption.\textsuperscript{[20]}

The water sorption and water solubility were determined according to the ISO standards 1567:1999.\textsuperscript{[16]} According to this specification, water sorption should not exceed 32 \(\mu\)g/mm\(^3\) for heat-cured or self-cured materials. The loss in mass per unit volume (soluble material) should not exceed 1.6 \(\mu\)g/mm\(^3\) for heat-cured and 8.0 \(\mu\)g/mm\(^3\) for self-cured materials.\textsuperscript{[14,15]}

The values of water sorption that were obtained as a result of our study are consistent with ADA standards and the values of water sorption that were mentioned by researchers above. In general, acrylic resins with incomplete polymerization cannot interact with the media during polymerization because of using all reactive substances. Furthermore, polymer molecules have very little capability of passing through the layers of epidermal tissue and being diffused because of their sizes.

The reaction products and residual monomers that are present in the polymer structure move toward the surface and interact with the soft tissues. Thus, only the substances being on the surface are biologically important.\textsuperscript{[21]}

To reduce the amount of these substances in acrylic resin, this material needs to be kept in water for a long time. In this way, the substances with low molecular weight, in other words, more reactive substances pass into the water.\textsuperscript{[22]}

Consequently, these substances cannot solute into a patient’s mouth and, in this way, the amount of potential allergen...
is reduced. The concentration of residual monomer can be reduced following an appropriate method of polymerization and for that reason, sufficient temperature and time are required. \[^{[21,23]}\]

In our study, highly polymerized resin samples absorb more water than other samples. To reduce the amount of residual monomer in acrylic resins and pass it into water, prolonged boiling during processing and keeping in water for long period is essential. The water molecules fill the space created in the structure of acrylic resin by diffusion. Therefore, more water sorption in heat-cured acrylic resins was observed compared to self-cured resins in different time intervals and solutions.

**CONCLUSION**

Following conclusions can be drawn from the present investigation:

1. Both heat-cure and self-cure acrylic resins showed varying water sorption and solubility
2. The results of both the water sorption and solubility showed compliance with the ISO specification
3. No correlation was found between water sorption and solubility
4. The highest sorption value was in denture cleansing solution followed by alternate immersion in distilled water and artificial saliva
5. The least sorption value was seen with artificial saliva followed by distilled water
6. Both heat-cure and self-cure acrylic resins showed higher solubility in artificial saliva + denture cleaner than in distilled water alone; the value was not statistically significant.

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**Conflicts of interest**

There are no conflicts of interest.

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