Original Article

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Curing process modification of a 'self-cured' injection molding material: Effect on water sorption and solubility

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Objectives: to determine water sorption and solubility of 'self-cured' IvoBase® Hybrid material in 2 curing techniques and immersion times in water, and to compare them to 'heat-cured' SR Ivocap® High Impact material.
Materials and Methods: disc shaped (50 mm diameter and 0.5 mm thickness) of SR Ivocap® High Impact was cured in water at 100°C for 35 minutes, then at 25°C for 30 minutes (Ivocap wet curing). IvoBase® system was either processed via the IvoBase injection machine programme at 120°C (IvoBase dry curing) or cured by the same method as SR Ivocap® (IvoBase wet curing). The specimens were kept in the desiccator containing freshly dried silica gel, weighed daily until their mass (m_1) was constant to 0.2 mg and, the volume (V) were recorded. The conditioned specimens were immersed in distilled water for 7 days and 30 days. The specimen weight (m_2) were recorded after removal from the water. The specimens were reconditioned to constant mass (m_3) in the desiccator. Then water sorption (w_{sp}) and water solubility (w_{sl}) at 7 days and 30 days were calculated. Two-way ANOVA was used to analyze the data.
Results: water sorption of Ivocap wet curing, IvoBase dry curing and IvoBase wet curing were 20.8±0.5, 22.4±0.9 and 23.2±0.3 μg/mm^3 respectively for 7 days immersion and 21.8±0.9, 22.6±1.1 and 23.9±0.1 μg/mm^3 respectively for 30 days immersion. The water solubility were 0.83±0.01, 0.33±0.04 and 0.69±0.03 μg/mm^3 respectively for 7 days immersion and 0.89±0.05, 0.62±0.08 and 0.65±0.02 μg/mm^3 respectively for 30 days immersion. Water sorption of IvoBase material was significantly higher than that of Ivocap at 7 days and 30 days ($p<0.05$). On the contrary, water solubility of Ivocap material was significantly higher than that of IvoBase ($p<0.05$). While the 2 methods of curing IvoBase did not affect the material water sorption but water solubility at 7 days water storage of IvoBase cured at 100°C was significantly higher than the material cured via the automated instrument. All the specimens passed the ISO 20795-1 requirement for water sorption not exceeding 32 μg/mm^3 and water solubility not exceeding 1.6 μg/mm^3.
Conclusion: Water sorption of IvoBase material was significantly higher than that of Ivocap but water solubility of Ivocap material was significantly higher than that of IvoBase. The methods of curing IvoBase did not affect the material water sorption but these had an influence on water solubility significantly.

Keywords: acrylic resin, curing technique, water sorption, water solubility

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Introduction

Acrylic resins are hard, brittle and glassy polymer. [1] Their use is mainly for removable prostheses to support artificial teeth [2], for fabrication custom trays, baseplate for denture construction, and uses as denture repair materials, soft liners and denture teeth [1, 3] There are many types of processing technique of acrylic resin, including compression molding and injection molding technique. [4, 5] When comparing two system of processing acrylic resin, the injection technique has been shown to be more accurate than the compression molding technique. Current preferred technique in Thailand is the compression molding technique because there is not necessary to use special equipment as well as the lower cost. [2, 6]

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The injection molding technique requires a specially designed flask. After the lost wax process, resin is injected into the mold cavity in the flask by pressure at room temperature. [7] The curing process is different depending on the manufacturer of the equipment. IvoBase® Hybrid material uses the IvoBase® Injector which initial curing temperature at 40°C,total curing time for 35 minutes, then cool it in cold water for 15 minutes. [4] As stated in the manufacturer scientific documentation the IvoBase® materials belongs to the category of self-cured polymers. [4] Another injection molding material is the heat-cured Ivocap® High Impact. This material is heated up to 100°C and boiled for 35 minutes, then is cooled down in cold water for 30 minutes.

Water sorption properties of denture base affects mechanical and dimensional properties of the polymer. [7] Water molecules penetrate the polymethyl methacrylate mass, and occupies space between polymer chains. The affected polymer chains are then forced apart. [7] These cause two important phenomena. Firstly, a slight expansion of the polymerized mass that cause dimensional changes. [2, 5, 7, 8] Secondly, the interfering of water molecules with the entanglement of polymer chains thereby act as plasticizers. [7] Moreover, the water sorption also affects the color stability. [9]

It has been estimated that for each 1% increase in weight produced by water absorption, acrylic resin would expand 0.23% linearly which partially compensates the polymerization shrinkage (0.5% linear shrinkage). [7] The diffusion coefficient (D) of water in heat-polymerizable denture acrylic resin is 0.011×10^{-6} cm^2/s at 37°C and 0.023×10^{-6} cm^2/s for a chemically activated resin. [7] Since the diffusion coefficients of acrylic resins are relatively low, the time required for a denture base to reach saturation is important. This depends on the thickness of the resin, as well as the storage conditions. Mostly, acrylic resins may require a period of 17 days to become fully saturated with water. [7] It is indicated that the expansion of the acrylic resin would be proportional to the time of the exposure in water until equilibrium is reached. [10] Drying or wetting of acrylic denture at room temperatures can cause small dimensional changes, but higher temperatures (such as water at 100°C) may lead to increased absorption of water and produce more significant change. [11]

Although denture base resins are soluble in a variety of solvents, they are virtually insoluble in the fluids commonly encountered in the oral cavity. [7] Although PMMA shows low water solubility, residual monomer may diffuse into the oral environment. The small quantity that is dissolved results from nonpolymerized monomer and water-soluble additives (colorant constituents etc.). [2, 12-14]

From previous studies, the materials were characterized with respect to degree of cure, considering the difference of residual monomer, indentation resistance, tensile strength and water sorption. Residual monomer concentration is important parameter in the determination of the properties of the materials. [15] When using temperature under 100°C for curing acrylic resins, higher residual MMA were found compare to those produced with a prolonged curing period at 100°C. [16]

This study investigated water sorption and solubility of an auto-polymerizable and a heat-polymerizable injection molding denture base polymers from two immersion times. At 7 days was the standard immersion time according to ISO specification 20795-1:2013 [17], at 30 days was represented for longer immersion time than standard. The materials used in this work were Ivocap® High Impact which is a heat-cured material and IvoBase® Hybrid which is classified as an auto-polymerizable material by the manufacturer. The curing process of IvoBase® Hybrid was modified from that recommended by the manufacturer to resemble that of the heat-cured material. It was expected that the alternative method of curing the 'self-cured' material would decrease the material water sorption and solubility.

Materials and methods

Two injection molding denture base materials were chosen in this study: SR Ivocap® High Impact and IvoBase® Hybrid. Their processing methods and measurements are shown in Table 1.

The curing of SR Ivocap® High Impact (Group 1) was set in such a way that the water boils during the entire polymerization period. The polymerization temperature was about 100°C. IvoBase® Hybrid curing process (Group 2) was developed by combined the advantages of heat-curing polymer and those of self-curing polymer (dual cured polymerization). This system uses a low initial polymerization temperature approximately 40°C, then heating up to 120ºC by automatic machine. In this study, the curing of IvoBase® Hybrid was modified to resemble that of SR Ivocap® High Impact (Group 3). In this study, water sorption and solubility were measured after 7days immersion in water followed the method in ISO 20795-1:2013. [17] The water immersion conditions were 7 days and 30 days.

Ten disc-shaped specimens were made for each experiment group, using a metallic mold with diameter of 50±1 mm and depth 1±0.1 mm. This specimen size was 0.5 mm thicker than the size specified in the ISO Specification No. 20795-1:2013. From our pilot study when using the specimen with thickness 0.5 mm, voids and porosities appeared throughout the specimens. When the thickness was increased to 1 mm, porosities disappeared. After the injection process were done, the specimens were cured as mentioned in Table 1. The specimens were then removed from the metallic mold and checked for the completion of the injection process. The top and bottom surfaces of the specimens were flat and parallel. All specimens were polished with silicon carbide abrasive paper from no.80, 200, 400, 600, 800 to 1000 (TOA, Samutprakarn, Thailand), respectively. The specimen thickness (0.5 ± 0.1 mm) were measured with a micrometer at 5 different positions. Their diameters (50 ± 1 mm) were measured with a dial caliper.

The specimens were separately placed in the rack and put into the desiccator which contained silica gel, freshly dried for 300 ± 10 min at 130 ± 5°C. The desiccator was stored in the oven at 37 ± 1°C for 23 ± 1 hours. Then the rack were moved into the second desiccator, which was supplied with freshly dried silica gel. The second desiccator was kept at 23 ± 2°C. After 60 ± 10 min in the second desiccator, the specimens were ready for weighing. An analytical balance, which has an accuracy of 0.1 mg, was used to weigh the specimen to an accuracy of 0.2 mg. The desiccator was kept sealed except for the shortest possible period required for removing and replacing specimens. The specimens were weighed repeatedly until a constant mass, m_1, (the "conditioned mass"), was reached, i.e. until the loss in mass of each specimen was not more than 0.2 mg between successive weightings. At this point the volume of each specimen (V) was calculated, using the mean of three diameter measurements and the mean of five thickness measurements. The thickness measurements were made in the center and at four equally spaced locations around the circumference.

The conditioned specimens were immersed in distilled water at 37 ± 1°C for 7 days ± 2 hours (n=5) and 30 days ± 2 hours (n=5). After these time, the discs were gentle removed from the water with polymer coated tweezers, wiped with a clean, dry towel until free from visible moisture, waved in the air for 15 ± 1 seconds and weighed 60 ± 10 seconds after removal from the water (to an accuracy of 0.2 mg). The mass was recorded as m_2. The specimens were reconditioned to constant mass (m_3) in the desiccator as the first step before immersion in the water.

Table 1 Injection molding acrylic resins used in this study (n=5)

Material	Processing	Measurement Powder:liquid
SR Ivocap® High Impact (Group 1-Ivocap wet curing)	Place mold in water, heat up to 100°C and boil it for 35 minutes. Then cool in cold water for 30 minutes.	20 g : 30 ml
IvoBase® Hybrid (Group 2-IvoBase dry curing)	Dry curing following the program in the automated injection unit: initial cure at 40°C then at 120ºC, total curing time 35 minutes. Then cool in cold water for 15 minutes.	34 g: 20 ml
IvoBase® Hybrid (Group 3-IvoBase wet curing)	Place mold in water, heat up to 100°C and boil it for 35 minutes. Then cool in cold water for 30 minutes.	34 g: 20 ml

The value for the water sorption, w_{sp} , was calculated for each specimen, expressed in micrograms per cubic millimeter from the equation: $W_{sp} = \frac{m_2 - m_2}{V}$ m_2 is the mass of the specimen, in micrograms, after immersion in water; m_3 is the reconditioned mass of the specimen, in micrograms; V is the volume of the specimen, in cubic millimeters. The values of water sorption were round off to the nearest microgram per cubic millimeter.

Water solubility, w_{sl} , was calculated for each specimen, expressed in micrograms per cubic millimeter from the following equation: $W_{sl} = \frac{m_1 - m_3}{V}$: m_1 is the "conditioned" mass of the specimen, in micrograms; m_3 and V are as described above. The values of water solubility were round off to the nearest 0.1 microgram per cubic millimeter.

Water sorption and solubility data were normally distributed according to Shapiro-Wilk test. The water sorption and solubility data were analyzed using two-way analysis of variance at α=0.05. Multiple comparison was used to reveal which groups differed.

Results

Means and standard deviations of water sorption and solubility are shown in Table 2, 3.

Water sorption of Ivocap wet curing, IvoBase dry curing and IvoBase wet curing were 20.8±0.5, 22.4±0.9 and 23.2±0.3 μg/mm^3 respectively for 7 days immersion and 21.8±0.9, 22.6±1.1 and 23.9±0.1 μg/mm^3 respectively for 30 days immersion. The water solubility were 0.83±0.01, 0.33±0.04 and 0.69±0.03 μg/mm^3 respectively for 7 days immersion and 0.89±0.05, 0.62±0.08 and 0.65±0.02 μg/mm^3 respectively for 30 days immersion.

Table 2 Mean and standard deviation values of water sorption (μg/mm^3) of 3 experiment groups at 7 days and 30 days immersion (n=5)

w_{sp}	7 days	30 days
Group 1 SR Ivocap® High Impact (wet curing)	20.8(0.5)$^{A, a}$	21.8(0.9)$^{B, c}$
Group 2 IvoBase® Hybrid (dry curing)	22.4(0.9)$^{C, b}$	22.6(1.1)$^{C, cd}$
Group 3 IvoBase® Hybrid (wet curing)	23.2(0.3)$^{D, b}$	23.9(0.1)$^{D, d}$

Note : within the same group (horizontal row), means with different superscripts written in uppercase letters were significantly different ($p < 0.05$).

:within the same water storage period (vertical column), means with different superscripts written in lowercase letters were significantly diffirent ($p < 0.05$).

Table 3 Mean and standard deviation values of water solubility (μg/mm^3) of 3 experiment groups at 7 days and 30 days immersion (n=5)

w_{sl}	7 days	30 days
Group 1 SR Ivocap® High Impact (wet curing)	0.83 (0.01)$^{A, a}$	0.89 (0.05)$^{A, d}$
Group 2 IvoBase® Hybrid (dry curing)	0.33 (0.04)$^{B, b}$	0.62 (0.08)$^{C, e}$
Group 3 IvoBase® Hybrid (wet curing)	0.69 (0.03)$^{D, c}$	0.65 (0.02)$^{D, e}$

Note : within the same group (horizontal row), means with different superscripts written in uppercase letters were significantly different ($p < 0.05$). :within the same water storage period (vertical column), means with different superscripts written in lowercase letters were significantly diffirent ($p < 0.05$).

Water sorption of IvoBase material was significantly higher than that of Ivocap ($p<0.05$) and immersion times effect only Ivocap material. On the contrary, water solubility of Ivocap material was significantly higher than that of IvoBase ($p<0.05$). While the 2 methods of curing IvoBase did not affect the material water sorption but water solubility at 7 days water storage of IvoBase cured at 100°C was significantly higher than the material cured via the automated instrument. All the specimens passed the ISO 20795-1 requirement for water sorption not exceeding 32 μg/mm^3 and water solubility not exceeding 1.6 μg/mm^3.

Discussion

When acrylic resin absorbs water, the molecules of water are trapped in the PMMA chain polymer. [7] This situation leads to dimensional expansion, [2, 5, 7, 8] plasticity [7, 18] and color instability of the acrylic resin. [9] Although dimensional expansion of acrylic resin may compensate for the decreased volume or shrinkage during polymerization process, a decrease of flexural strength and hardness and color change of the material are the disadvantages that may occur. Because of these, water sorption of acrylic resin as indicated in ISO 20795-1:2013 should not exceed 32 μg/mm^3. [17]

Water soluble parts in acrylic resin come from non-polymerized monomer and water-soluble additives (colorant constituents etc.). [2, 12-14] These results in material's water solubility and residual monomer. [12, 19] Auto-polymerized acrylic resin was reported to release more residual monomer than heat-polymerized acrylic resin. The water solubility of auto-polymerized acrylic resin should not exceed 8.0 μg/mm^3 while other dental polymer types not exceeding 1.6 μg/mm^3. [17]

According to the manufacturer technical documentation, the IvoBase materials belong to the category of self-cured polymers. [4] However, the IvoBase injection machine was programmed to raise the curing temperature from 40°C to 120°C and maintain the temperature until the polymerization process completed. From the ISO 20795-1:2013 the polymerization temperature of a self-cure material must use the curing temperature less than 65°C.

The water sorption and solubility values of all experiment groups in this study comply with the values given in the ISO20795-1:2013. The results in this study were also in agreement with the previous studies. [6, 9, 18, 20, 21] The water sorption difference between 7 days and 30 days was significant only for Ivocap materials. However, the water solubility of IvoBase increased from

7 days to 30 days storage, while water solubility of the Ivocap did not increase. This may indicate that the IvoBase material contains some of the auto-polymerized material compositions, and may had more residual monomer leaching out.

This study is preliminary research which investigated water sorption and solubility only at two immersion times, at 7 days as following ISO specification 20795-1:2013 and at 30 days. For further study of water sorption and solubility, it should evaluate at least three proper immersion times.

The major problem in this study is the processing method of materials, especially the IvoBase. Incomplete polymerization, porosities and incomplete injection were found in some of the specimens. These may result from the thickness of the specimen which was only 1 mm. and the pressure used in wet curing technique that may be lower than the pressure used in the injection machine.

In conclusion, within the limitation of this study, it can be concluded that water sorption of IvoBase material was significantly higher than that of Ivocap but water solubility of Ivocap material was significantly higher than that of IvoBase. The methods of curing IvoBase did not affect the water sorption of material but these had an influence on water solubility significantly. The water sorption and solubility of all the experiment group passed the ISO 20795-1:2013 requirement with water sorption not exceed 32 μg/mm^3 and water solubility not exceed 1.6 μg/mm^3.

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