Original Article

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Modification of curing technique of a 'self-cure' injection molding denture base materials: Effect on flexural strength

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Objectives: This study compared the flexural strength of a self-cure injection-molding denture base material, which its curing process was modified from that recommended by the manufacturer, when stored in water at 7 days and 30 days.

Materials and methods: Twenty rectangular specimens (10mm x 64mm x 3.3mm) were prepared according to ISO 20795-1:2013 for each experiment group. SR Ivocap® High Impact were wet cured at 100 ^{O}C for 35 min (Ivocap wet curing). IvoBase® Hybrid were dry cured in an automated instrument which the curing temperature started from 40 ^{O}C up to 120 ^{O}C for 35 min (IvoBase dry curing). IvoBase® Hybrid were also wet cured at 100 ^{O}C for 35 min (IvoBase wet curing). Ten specimen in each group were immersed in 37^{o}C distilled water for 7 days and the other 10 specimens at 30 days. At the end of the storage periods, three-point bending flexural strength test with a 5 mm/min crosshead speed was carried out using a universal testing machine. Nonparametric test (Kruskal Wallis and Mann-Whitney) was used to compare the flexural strength data.

Results: The median, 25th percentiles and 75th percentiles values of flexural strength of SR Ivocap® High Impact wet curing, IvoBase® Hybrid dry curing and IvoBase® Hybrid wet curing and were 61.5(61.2,63.1), 78.9(75.6,81.7) and 68.0(61.4,70.2) MPa, respectively for 7 days immersion; and, 62.6(59.2,63.6), 68.3(66.4,72.5) and 64.4(50.7,67.2) MPa, respectively for 30 days immersion. The flexural strength of Ivocap High Impact was significantly lower than that of IvoBase Hybrid ($p<0.05$). The flexural strength of IvoBase® Hybrid dry curing was significantly higher than that of IvoBase Hybrid wet curing. ($p<0.05$). The flexural strength of IvoBase Hybrid decreased when immersion in water up to 30 days.

Conclusion: Flexural strength of IvoBase® Hybrid was higher than that of SR Ivocap® High Impact but decreased for water storage up to 30 days. Alternative curing of IvoBase Hybrid did not improve its flexural strength.

Keywords: curing temperature, flexural strength, injection molding denture base, water immersion

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Introduction

Dentures are believed to be a mode of treatment for replacing missing teeth. Denture base polymer was classified by ISO 20795-1: 2013 into 5 types: Heat-polymerizable materials, Autopolymerizable materials, Thermoplastic blank or powder, Light-activated materials and Microwave-cured materials. The most commonly used material for denture fabrication is heat-polymerizable denture base materials poly (methyl methacrylate). [1, 2] Processing methods for fabricating denture base are compression technique, injection technique and pouring technique. [3] Heat-polymerized poly (methyl methacrylate) resin system consists of power and liquid, which after being mixed, requires thermal energy for initiating polymerization reaction. Thermal energy source are from water bath or microwave oven. The polymerization reaction of denture base polymers is exothermic. The amount of heat from the processing method may affect properties of denture base polymers. [4]

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The frequent reason for failure of denture is fracture. Fracture occurs from initiation, propagation of crack and raise of stress. The causes of failure when using denture base are multifactor, for example, the denture deformation during function, stress within the denture, stress concentration at the weak point, denture with thin or under-extended flanges, poorly fitting dentures or a lack of adequate relief, dentures with a wedged or locked occlusion, poor clinical design and dentures which have been previously repaired. There are many methods to solve their problems such as using alternative materials to PMMA, addition of some strengthening agents to PMMA, changing the chemistry of the denture base polymer by co-polymerization and cross-linking of resin materials. Increasing the degree of polymerization of denture base polymers may be an alternative method of increasing the strength of denture base polymers. Heat-polymerizable acrylic resin, which has higher degree of polymerization than autopolymerizable acrylic resin, was reported to have lower residual monomer and more strength than autopolymerizable acrylic resin.

Strength of denture base polymers can be determined from many mechanical properties. Flexural strength is one of the important mechanical properties to evaluate strength of the denture base polymers. [4, 5-10] Flexural strength (bending strength or transverse strength or modulus of rupture) is the force per unit area at the instant of fracture in a test specimen subjected to flexural loading. [3, 4] Ali et al. compared properties between two PMMA denture base polymers. They showed that heat-polymerized denture base polymer had statistically higher values of surface hardness, flexural strength, and modulus than autopolymerized denture base polymer. [11] Gharechahi, et.al, [12] Ganzarolli et al, [13] Uzun, et.al. [14] compared flexural strength of acrylic resin denture base (PMMA) processed by compression molding and injection molding technique by three-point flexural strength test. Injection-polymerized acrylic resin had higher flexural strength than the material polymerized by conventional method. [12, 13, 14]

An aqueous environment, such as oral cavity, can induce changes in physical properties of denture base polymers. Water sorption decreases the mechanical properties of denture base polymers, such as flexural strength and surface hardness. Different monomer compositions and polymerization activator systems of denture base polymers have different resistance to the influence of water. [15, 16, 17, 18] Valittu et al. reported that heat-cured and auto-polymerized denture base polymer stored in water up to 4 weeks significantly decreased their ultimate flexural strength and flexural modulus. [15, 16]

According to many studies, flexural strength is one of the important material properties to evaluate long-term clinical application of denture base. The immersion period of the denture base resin in water also affected the flexural strength. Until now, no research studies which compared the effect of water storage on the flexural strength of autopolymerizable and heat-polymerizable injection molding denture base polymers have been found. Therefore, it was decided to investigate the effect of water storage on the flexural strength of auto-polymerizable and heat-polymerizable injection molding denture base polymers. The materials used in this work were Ivocap® High Impact which is a heat-cure 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-cure material. It was expected that heat-treatment would increase its flexural strength.

Materials and methods

Two commercial injection molded acrylic resins products, one was a self-cure and the other was heat-cure, were used in this investigation. The manipulation and processing procedures are presented 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. [3, 20] 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. [19] In this study, the curing of IvoBase Hybrid® was modified to resemble that of SR Ivocap High Impact® (Group 3)

The specimen strips for flexural strength test were prepared to size 64 mm x 10 mm x 3.3 mm. The surface of the specimens were polished with standard metallographic grinding papers no. P500, P1000 and P1200. All specimens were stored in water at temperature 37±1 °C for 7 and 30 days. During the test, the specimens were laid immediately to the flat surface symmetrically on the supports of 3-point flexural test. Then a constant increasing rate of force was applied to the specimen at 5±1 mm/min until the specimen was broken by a universal testing machine. [3, 4] The load and the flexural displacement data of all specimens were recorded.

Three-point flexural strength was calculated from $\sigma = \frac{3Fl}{2bh^2}$, σ is the flexural strength (in MegaPascals), *F* is the failure load of the specimen (Newtons), *l* is the distance between the supports (±0.01mm) (millimetres), *b* is the width of specimen measured immediately prior to water storage (millimetres), *h* is the height of specimen measured immediately prior to water storage (millimetres). [3, 4] For the specimens that failure occurred beyond the flexural strain of 5.0%, the failure load at 5.0% strain determined from the load-deflection curve was used to calculate the flexural strength (Figure 1).

The statistical analysis done by SPSS for Windows (IBM Corp. Released 2015. IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY: IBM Corp.) showed that data distribution of each experiment group was not normal (Shapiro-Wilk test $p>0.05$) and Nonparametric test (Kruskal Wallis test) was used to compare flexural strength among the 3 groups of materials within the same immersion time. Mann-Whitney U test was used to compare the flexural strength of each material with different immersion time (7 days and 30 days). Bonferroni test was used to adjust error occurred from multiple comparisons.

Table 1 Processing of SR Ivocap® High Impact and IvoBase® Hybrid.

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 for 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

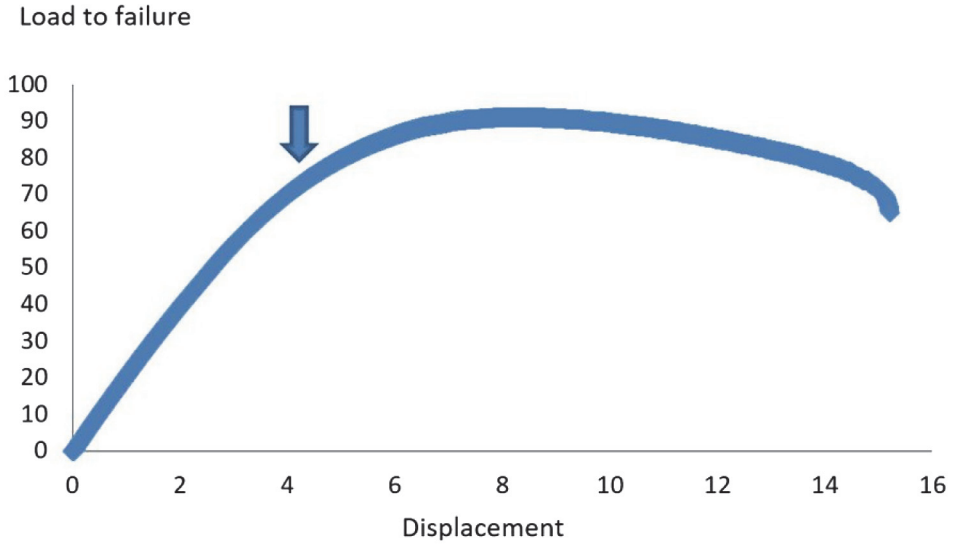

Figure 1 Load-deflection plots of a specimen that failure occurred beyond 5.0% flexural strain (arrow)

Results

The medians and percentiles of the flexural strength are shown in Tables 2 and Figure 2.

The median (25^{th},75^{th} percentiles) values of flexural strength of SR Ivocap® High Impact wet curing (Group 1), IvoBase® Hybrid dry curing (Group 2) and IvoBase® Hybrid wet curing (Group 3) were 61.5(61.2,63.1), 78.9(75.6,81.7), and 68.0(61.4,70.2) MPa, respectively for 7 days immersion; and 62.6(59.2,63.6), 68.3(66.4,72.5) and 64.4(50.7,67.2) MPa, respectively for 30 days immersion.

Table 2 Median of Flexural strength in MPa (numbers in parentheses are P25 and P75) of 3 experiment groups at 7 days and 30 days immersion (n=10)

	Flexural strength	
	7 days	30 days
Group 1 SR Ivocap High Impact (wet curing)	$61.5^{a,A}$ (61.2,63.1)	$62.6^{a,A}$ (59.2,63.6)
Group 2 IvoBase Hybrid (dry curing)	$78.9^{b,A}$ (75.6,81.7)	$68.3^{b,B}$ (66.4,72.5)
Group 3 IvoBase Hybrid (wet curing)	$68.0^{a,A}$ (61.4,70.2)	$64.4^{ab,A}$ (50.7,67.2)

Note: Group 1 SR Ivocap® High Impact; polymerization in boiled water at 100°C (Ivocap wet curing). Group 2 IvoBase® Hybrid; polymerization via injection machine at 40°C up to120°C (IvoBase dry curing). Group 3 IvoBase® Hybrid; polymerization in boiled water at 100°C (IvoBase wet curing)

: within the same material (horizontal), same capital letters indicate no significantly different. (α =0.05).

: within the same water storage period (vertical), same lowercase letters indicate no significantly diffirent. (α =0.05).

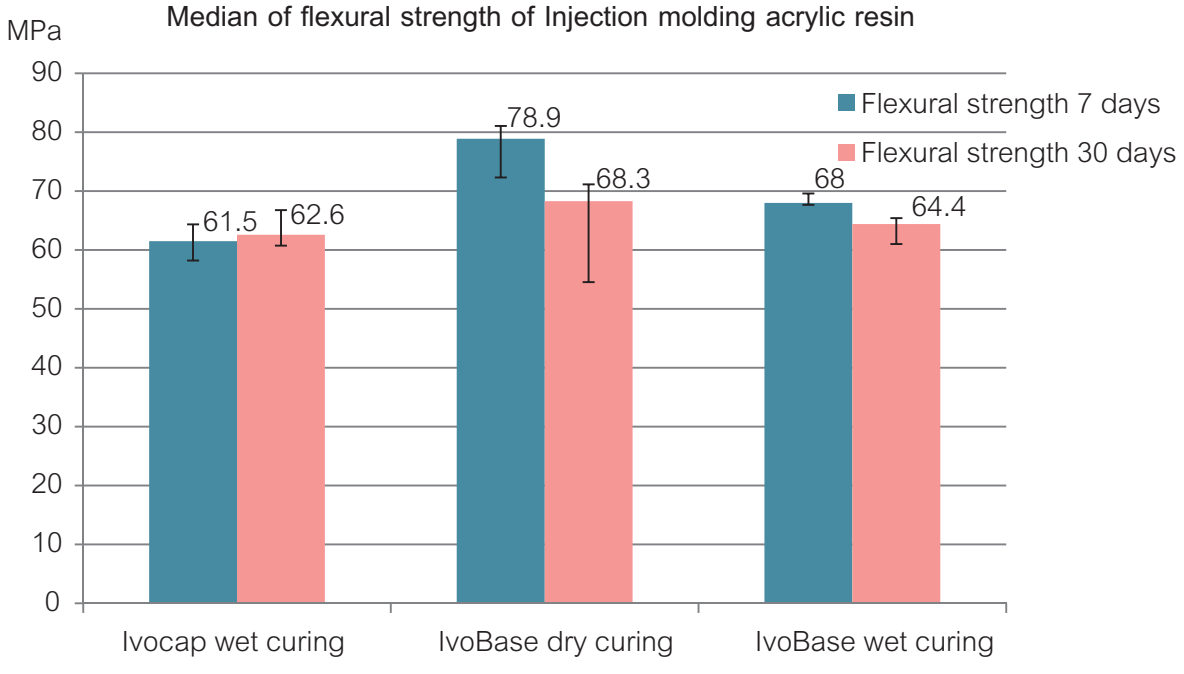

Figure 2 Median, P25 and P75 of flexural strengths of injection molding denture base materials at 7 days and 30 days immersion

The flexural strengths of SR Ivocap High Impact and IvoBase Hybrid when cured using the same curing method as SR Ivocap High Impact were not significantly different between 7 days and 30 days water immersion period ($p>0.05$). However, the flexural strengths of IvoBase Hybrid when cured following the manufacturer recommended method were significantly higher at 7 days than at 30 days ($p<0.05$). The immersion time affected the flexural strength of IvoBase Hybrid when cured following the manufacturer recommended method only, longer immersion time up to 30 days decreased its flexural strength.

At 7 days water storage, the flexural strength of IvoBase Hybrid when cured following the manufacturer recommended method were significantly higher than the other two groups ($p<0.05$). At 30 days water storage, the flexural strengths of the 3 experiment groups were not significantly different ($p>0.05$).

Discussion

Poly (methyl methacrylate) or PMMA is commonly used denture base material. [1, 2] Heat-polymerized PMMA requires thermal energy for polymerization. The polymerization of denture base polymers is exothermic. The amount of heat released can affect the properties of denture base polymers. [4] The frequent reason of denture failure is fracture of dentures. Fracture occurs from initiation and propagation of crack, and raise of stress. Causes of failure of using denture base are multifactor. One reason for the improvement of materials and processing techniques is to reduce fracture of denture base. The strength of denture base polymers can be measured from many mechanical properties. Flexural strength is one of the important mechanical properties to evaluate strength of the denture base polymers. The three-point bending test is useful for evaluate flexural strength of denture base. [4]

The minimum value of flexural strength for Type 2 auto-polymerizable denture base polymers is 60 MPa (ISO 20795-1: 2013). From this study the flexural strength of IvoBase Hybrid when cured according to the manufacturer recommendation (Group 2) and when cured using the process of Ivocap High Impact (Group 3) at 7 days water storage were 78.9 and 68.0 MPa, respectively. These values exceeded the 60 MPa requirement. On the other hand, the flexural strength of heat-cure SR Ivocap High Impact (61.5 MPa) was lower than the minimum value specified in the ISO 20795-1: 2013 for Type 1 heat-polymerizable denture base polymers (65 MPa). [3]

Increasing the degree of polymerization of denture base polymers is expected to increase the strength of denture base polymers. Heat-polymerizable acrylic resin was reported to have more degree of polymerization, has lower residual monomer and more strength than autopolymerizable acrylic resin. [4, 5-10] However, this statement was not true concerning the flexural strength of heat-cure Ivocap and self-cure IvoBase materials. The Ivocap material were cured at 100ºC for 35 min. while the IvoBase material were initial cured at 40°C then at 120ºC for 35 min. The final curing temperature of the IvoBase was higher than the final curing temperature of Ivocap. From ISO 20795-1: 2013, the curing temperature of auto-polymerizable acrylic resin was less than 65 ºC. Therefore, the IvoBase Hybrid material should be classified as heat-cure material, not self-cure material as mentioned by the manufacturer.

From this study, The flexural strength of IvoBase Hybrid when cured according to the manufacturer recommendation (Group 2) was significantly higher than that when cured using the process of Ivocap High Impact (Group 3), both at 7 days and 30 days. This was due to the improvement in the curing process via the automated instrument provided by the manufacturer. However, when this expensive instrument is not available for use, curing this material by using the common method of boiling it in 100°C water for 35 min. still yield flexural strength exceeding the minimum ISO given value. Nisar et al. [21] and Bartoloni et al. [22] said that conversion of monomer to polymer is time dependent and rate of conversion is increased by increasing curing temperature. A greater rate of conversion is affected lower porosity and higher flexural strength. Jorge et al. [23] said that when curing temperature of denture base material was increased, the residual monomer decreased and the flexural strength increased.

Powder to liquid mixing ratio also makes the flexural strength of IvoBase material higher than that of Ivocap material, apart from the higher curing temperature. The power/liquid ratio of Ivocap material is 20g/30ml which is lower than that of IvoBase material (34g/20ml). Arora et al. [25] and Okuyama et.al. [26] suggested that higher powder to liquid ratio of acrylic resin may be associated with a greater enlargement of polymers, produces a closer three-dimensional network structure and decreases quantities of the unreacted monomers. Thus, the values of hardness and flexural strength are increased. [25, 26] Dogan etal. [27] suggested that lower powder to liquid ratio of acrylic resin may be associated with excessive leaching of residual monomer. The higher levels of residual monomer lead to higher number of void formation in the resin. The higher levels of residual monomers are increased percentage of water sorption, and the flexural strength is decreased. [27]

Conclusion

Within the limit of this study, the following conclusion may be drawn:

The flexural strength of the ‘self-cure; IvoBase Hybrid was higher than the ‘heat-cure’ Ivocap High Impact due to 2 reasons. 1. The powder to liquid ratio of the IvoBase material is higher and 2. The curing temperature of the IvoBase material is higher than that of the Ivocap material.

The water storage period had an effect on the flexural strength of Ivocap and IvoBase materials differently. The flexural strength of Ivocap did not decrease up to 30 days water storage, but the flexural strength of IvoBase deceased significantly.

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