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Influence of water sorption on mechanical properties of injection-molded thermoplastic denture base resins

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Abstract

Objective. This study investigated the influence of water sorption on certain mechanical properties of injection-molded thermoplastic denture base resins. ***Materials and methods.*** Six thermoplastic resins (two polyamides, two polyesters, one polycarbonate, one polymethylmethacrylate) and a polymethylmethacrylate (PMMA) conventional heat-polymerized denture-based polymer, selected as a control, were tested. Specimens of each denture base material were fabricated according to ISO 1567 specifications and were either dry or water-immersed for 30 days ($n = 10$). The ultimate flexural strength, the flexural strength at the proportional limit and the elastic modulus of the denture base materials were calculated. ***Results.*** Water sorption significantly decreased the ultimate flexural strength, the flexural strength at the proportional limit and the elastic modulus of one of the polyamides and the PMMAs. It also significantly increased the ultimate flexural strength of the polycarbonate. ***Conclusion.*** The mechanical properties of some injection-molded thermoplastic denture base resins changed after water sorption.

Key Words: *injection-molded thermoplastic denture base resin, polyamide, polyethylene terephthalate copolymer, polycarbonate, water sorption*

Introduction

Injection-molded thermoplastic resins are commonly used in dental practice [1–3]. There are many different types of injection-molded thermoplastic resins including polyamide, polyethylene terephthalate copolymer, polycarbonate, polycycloalkylene terephthalate copolymer and polymethylmethacrylate. Polyamide dentures are flexible, monomer-free and lightweight [4]. Patients frequently object to the placement of a metal clasp within a visible part of their removable partial dentures (RPDs) because the metal clasps are unesthetic [5,6] and frequently fracture due to fatigue [7–9]. Fortunately, RPDs without metal clasps that are made from injection-molded thermoplastic resins have been reported [1–3,5,6,10–14]. The use of RPD with retentive clasp arms without metal is obtained by extending a part of the denture base using an esthetic gingival shade. Therefore, RPDs without metal clasps are more appealing to the patient.

The flexibility of retentive clasp arms enables ease of insertion and removal of the RPD. Previous studies have investigated mechanical properties of injection-molded thermoplastic resins according to International Organization for Standardization (ISO) 1567 and ISO 1567:1999/Amd 1:2003 [11–13,15,16]. This test showed stress–strain curves for injection-molded thermoplastic resins that have been subjected to compressive stress. This curve was constructed from typical values of the ultimate flexural strength, flexural strength at the proportional limit and elastic modulus reported in the scientific literature. Mechanical properties that are important to dentistry include ultimate flexural strength, flexural strength at the proportional limit and the elastic modulus [17,18]. Ultimate flexural strength is distinct from compressive strength, which is calculated from the maximum

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recorded load and the original area. While the material is being plastically deformed the load may reduce, but this does not alter the total strain. Ultimate flexural strength is used to compare denture base materials in the basic research field. Flexural strength at the proportional limit is the magnitude of the elastic stress above which plastic deformation occurs. In other words, a resin with low flexural strength at the proportional limit will begin to deform permanently at a low stress. Denture material should have flexural strength at the proportional limit that is sufficiently high enough that permanent deformation does not result from the stress applied during mastication. Permanent deformation may result in loss of retention of RPD without metal clasps. Elastic modulus is the stiffness of a material that is calculated as the ratio of elastic stress to elastic strain. A RPD without metal clasps is retained at the undercuts of the abutment teeth by means of the denture base. Therefore, the elastic modulus of the injection-molded thermoplastic resin affects the ease of insertion and removal of the RPD and its retention. Previous studies have found that (1) all of the injection molded thermoplastic resins had a significantly lower flexural strength at the proportional limit (FS-PL) and a lower elastic modulus than the conventional heat-polymerized acrylic resin, (2) the polyamides and the polycycloalkylene terephthalate copolymer thermoplastic resins had low FS-PL and low elastic moduli, (3) the thermoplastic resin composed of polyethylene terephthalate copolymer had moderately high FS-PL and a moderate elastic modulus and (4) the thermoplastic resin composed of polycarbonate had moderately high FS-PL and elastic modulus. These injection-molded thermoplastic resin materials were stored in 37°C distilled water for 50 h before testing.

The effect of thermal stress on the mechanical properties of injection-molded thermoplastic denture base resins have also been investigated [12,13], showing that (1) thermocycling significantly decreased the FS-PL of one of the polyamides tested and significantly increased the FS-PL of the other polyamide and polycycloalkylene terephthalate copolymer, (2) thermocycling significantly decreased the elastic modulus of one of the polyamides and significantly increased the elastic modulus of the other polyamide, the polyethylene terephthalate copolymer, the polycycloalkylene terephthalate copolymer and the polycarbonate. These injection-molded thermoplastic resin materials were thermocycled between 5°C water and 55°C water for 50 000 1-min cycles.

However, little attention has been given to the influence of water sorption on the mechanical properties of injection-molded thermoplastic denture base resins. It is known that water exerts significant effects on the mechanical and dimensional properties of the polymer [19]. Water molecules may also interfere with the entanglement of polymer chains, thereby changing the physical characteristics of the resultant polymer. The retentive force of RPDs without metal clasps is dependent on the elastic modulus of the thermoplastic resin. It is assumed that, along with the change of mechanical properties by water sorption, the retentive forces of RPDs without metal clasps may also be affected. To the authors' knowledge, there is a lack of information on the influence of water sorption on the properties of thermoplastic polymers other than PMMA.

The purpose of the present study was to investigate the influence of water sorption on mechanical properties of injection-molded thermoplastic denture base resins. The null hypothesis was that the mechanical properties of water-immersed injection-molded thermoplastic denture base resins were not different from the mechanical properties of non-water-immersed injection molded thermoplastic denture base resins.

Materials and methods

Six injection-molded thermoplastic resins were selected for this study and a conventional heat polymerized polymethylmethacrylate (PMMA) was used as a control (Table I).

The flexural properties of the denture base materials were measured according to ISO 1567 [15] and ISO 1567:1999/Amd 1:2003 [16].

Specimens

Each denture base material specimen that was tested for flexural properties was polymerized according to the manufacturers' instructions in gypsum molds with cavities (65 mm long, 10 mm wide, 3.3 mm high). All of the specimens were wet ground with 600-grit SiC paper. The accuracy of the dimensions was verified with a micrometer at three locations for each dimension to within a 0.05-mm tolerance for width and height.

The test specimens were conditioned in a desiccator at room temperature for 5 days or stored in water at 37 ± 1°C for 30 days in order to be saturated. The test specimens were divided into 14 groups and each group included 10 test specimens according to denture base material and storing condition.

Water sorption

Test specimens of the water sorption group were immersed in distilled water in a thermostatically controlled water bath at 37 ± 1°C. Water sorption was followed by a weighing procedure repeated on days 1, 2, 4, 5, 6, 9, 13, 19, 23, 27 and 30. Water sorption (wt%) was calculated according to the following formula:

Water sorption = $100(W_n - W_0)/W_0$

where W_n = the weight of the specimen at each measurement period (g) and W_0 = the weight of the

Table I. Denture base resins used in this study.

Constituent	Material	Manufacturer	Processing method	Lot number
Polyamide (PA12)	Valplast	Unival Co., Ltd., Tokyo, Japan	Injection molding technique; heat processed at 215°C for 20 min	091142
Polyamide (PACM12)	Lucitone FRS	DENTSPLY International Inc., PA, USA	Injection molding technique; heat processed at 300°C for 17 min	100323A
Polyethylene terephthalate copolymer	EstheShot	i-Cast Co. Ltd., Kyoto, Japan	Injection molding technique; heat processed at 230°C for 20 min	IKB
Polycycloalkylene terephthalate copolymer (PCAT)	EstheShot Bright	i-Cast Co. Ltd., Kyoto, Japan	Injection molding technique; heat processed at 280°C for 20 min	2A6277240
Polycarbonate	Reigning N	Toushinyoukou Co. Ltd., Niigata, Japan	Injection molding technique; heat processed at 320°C for 30 min	FMY31T
Polymethyl methacrylate (PMMA)	Acrytone	High Dental Japan Co. Ltd., Osaka, Japan	Injection molding technique; heat processed at 260°C for 25 min	1010087
Polymethyl methacrylate (PMMA)	Acron	GC Corp., Tokyo, Japan	Heat-polymerized, compression molding technique; heat-processed at 70°C for 90 min, then at 100°C for 30 min and bench cooled for 30 min	(P)1004123 (L)1003191

specimen just before storing in 37°C distilled water (g). The mean values and standard deviations of the water sorption were calculated before flexural-testing procedures.

Flexural properties

The ultimate flexural strength, the flexural strength at the proportional limit [20–24] and elastic modulus of the specimens were tested. Each specimen was placed on a 50 mm long support for three-point flexural testing. A vertical load was applied at the mid-point of the specimen at a crosshead speed of 5 mm/min on a load testing machine (LR30K Plus, Lloyd Instruments Ltd., Hampshire, UK).

The ultimate flexural strength (MPa) was calculated according to the following formula:

Ultimate flexural strength = $3Fl/2bh^2$

where F = the maximum load (N), l = the span distance (50 mm), b = the width (mm) of the specimen and h = the height (mm) of the specimen.

The flexural strength at the proportional limit (MPa) was calculated according to the following formula:

Flexural strength at the proportional limit = $3F_1l/2bh^2$

where F_1 = the load (N) at the proportional limit.

The load at the proportional limit was determined from each load/deflection graph. The elastic modulus (GPa) was calculated using the following formula:

Elastic modulus = $F_2l^3/4bh^3d$

where F_2 = the load (N) at a point in the straight line portion of the load/deflection graph and d = the deflection (mm) at load F_2.

All the tests were performed under uniform atmospheric conditions of 23 ± 2°C and 50 ± 1% relative humidity.

A two-way analysis of variance (ANOVA) (STATISTICA, StatSoft Inc., Tulsa, OK) was applied to study the differences among the denture base materials and the effect of the water sorption. A one-way ANOVA (STATISTICA) was applied to determine if there was a significant difference resulting from the interaction between the two variables ($p = 0.05$). The Newman-Keuls post-hoc comparison ($p = 0.05$) (STATISTICA) was applied when appropriate.

Results

The water sorption of injection-molded thermoplastic denture base resins is depicted in Figure 1 and Table II. The one-way ANOVA and the Newman-Keuls post-hoc comparison were applied to the 30-day water-immersed denture base material specimens. There were significant differences among the denture base materials ($p < 0.05$). The 30-day water-immersed specimens of Acron possessed significantly higher

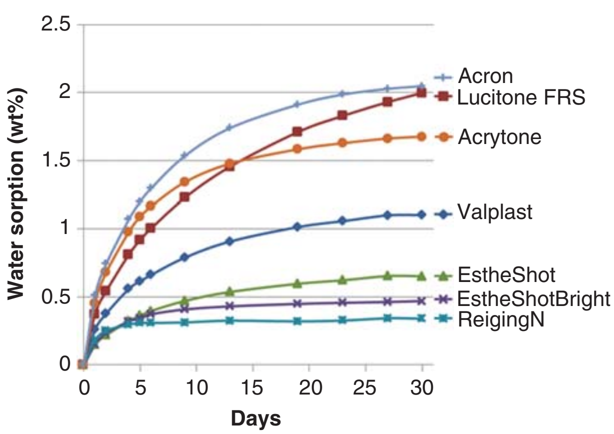

Figure 1. Water sorption of test specimens plotted to the storage time (n = 10).

Table II. Mean and standard deviation (SD) of the water sorption of the denture base resins for 30 days (n = 10).

Denture base resin	Water sorption (wt%); M (SD)
Valplast	1.1029 (0.0027)
Lucitone FRS	1.9968 (0.0153)
EstheShot	0.6532 (0.0046)
EstheShot Bright	0.4704 (0.0074)
Reigning N	0.3409 (0.0388)
Acrytone	1.6750 (0.0089)
Acron	2.0471 (0.0225)

water sorption compared with the other specimens. The 30-day water-immersed specimens of ReigningN possessed significantly lower water sorption compared with other specimens.

The two-way ANOVA revealed that there were significant differences in the ultimate flexural strength due to the denture base material, the effect of water sorption and the interaction between the denture base material and the water sorption period ($p < 0.05$). The one-way ANOVA and the Newman-Keuls post-hoc comparison were applied to the denture base material/ water sorption interaction. The results are depicted in Table III. The 30-day water-immersed specimens of Valplast, Acrytone and Acron possessed significantly lower ultimate flexural strengths compared with the dry specimens. The 30-day water-immersed specimens of ReigningN possessed significantly higher ultimate flexural strengths compared with the dry specimens.

The two-way ANOVA revealed that there were significant differences in the flexural strength at the proportional limit due to the denture base material, the effect of water sorption and the interaction between the denture base material and the water sorption ($p < 0.05$). The one-way ANOVA and the Newman-Keuls post-hoc comparison were applied to the denture base material/water sorption interaction. The results are depicted in Table III. The 30-day water-immersed specimens of Valplast, Acrytone and Acron possessed significantly lower flexural strength at the proportional limit compared with the dry specimens.

The two-way ANOVA revealed significant differences in the elastic modulus because of the denture base material, the water sorption and their interaction ($p < 0.05$). The one-way ANOVA and Newman-Keuls post-hoc comparison were applied to the denture base material/water sorption interaction. The results are depicted in Table III. The 30-day water-immersed specimens of Valplast, Acrytone and Acron possessed a significantly lower elastic modulus compared with the dry specimens.

Discussion

The null hypothesis of this study was rejected based on the fact that the mechanical properties of the water-immersed injection-molded thermoplastic denture base resins were different from those of the non-water-immersed injection-molded thermoplastic denture base resins.

In the present study, the ultimate flexural strengths, flexural strength at the proportional limit and elastic modulus of a conventional heat polymerized PMMA (Acron) significantly decreased after 30 days water sorption; these results were similar to those in other

Table III. Mean and standard deviation (SD) of the flexural properties of the denture base resins after dry and 30 days water sorption (n = 10).

Denture base resin	Water sorption	Ultimate flexural strength (MPa); M (SD)	Flexural strength at proportional limit (MPa); M (SD)	Elastic modulus (GPa); M (SD)
Valplast	Dry	59.55 (1.84)	23.61 (5.30)^{c}	1.42 (0.04)
Valplast	30 days	38.17 (1.64)	11.69 (3.85)	0.88 (0.04)
Lucitone FRS	Dry	80.92 (2.76)^{d}	31.30 (3.84)^{b}	1.80 (0.07)^{d}
Lucitone FRS	30 days	78.70 (1.96)^{d}	31.54 (6.18)^{b}	1.77 (0.05)^{d}
EstheShot	Dry	88.83 (3.42)^{b,c}	39.96 (4.79)^{a}	2.43 (0.12)^{a,b}
EstheShot	30 days	86.96 (3.49)^{c}	37.97 (7.98)^{a}	2.38 (0.13)^{b}
EstheShot Bright	Dry	71.69 (1.07)^{e}	29.33 (3.59)^{b}	1.86 (0.04)^{c,d}
EstheShot Bright	30 days	70.09 (1.18)^{e}	30.59 (3.81)^{b}	1.80 (0.09)^{d}
Reigning N	Dry	89.51 (1.30)^{b,c}	31.94 (4.86)^{b}	2.50 (0.05)^{a}
Reigning N	30 days	93.04 (3.60)^{a}	35.28 (3.60)^{a,b}	2.51 (0.15)^{a}
Acrytone	Dry	64.92 (1.37)	23.85 (3.75)^{c}	1.93 (0.08)^{c}
Acrytone	30 days	53.91 (2.24)	17.63 (5.04)	1.59 (0.06)
Acron	Dry	90.41 (10.38)^{a,b}	45.31 (6.97)	3.31 (0.12)
Acron	30 days	82.45 (3.71)^{d}	35.12 (4.39)^{a,b}	2.77 (0.18)

a–e denotes groups that were not significantly different from each other ($p > 0.05$).

studies with 30-day [21], 4-month [23], 6-month [25], 48-week [26] and 10-year [27] water immersion. Thus, the ultimate flexural strengths, flexural strength at the proportional limit and elastic modulus of denture base materials all seem to decrease after water sorption.

Compared with previous studies, the results (the flexural strength at the proportional limit and the elastic modulus) for the injection-molded thermoplastic denture base resins of the dry specimens in the present study were similar to those found in the previous study [11–13]. Moreover, the relative ultimate flexural strengths of denture base materials were similar to that of the flexural strength at the proportional limit. However, water sorption significantly increased the ultimate flexural strengths of ReigningN. In previous studies [12,28], the effect of water and temperature on polycarbonate were estimated; water and temperature decreased the molecular weight and changed the polymer from ductile to brittle and the elastic modulus of the polycarbonate significantly increased and the impact strength of the polycarbonate significantly decreased. In the present study, it is hypothesized that ReigningN was becoming brittle and this increased the ultimate flexural strength. However, since the present study only included short-term water immersion, the elastic modulus of ReigningN was not significantly increased.

Denture base materials typically exhibit considerable resin deformation before fracturing. Resin deformation that goes beyond its proportional limit permanently alters the dimensions of a denture and is not clinically acceptable. In this study, the flexural strength at the proportional limit was evaluated, but the ultimate flexural strength was not. Changes in flexural strength at the proportional limit and elastic modulus affect retention and stabilization of RPD because the retentive forces of RPDs without metal clasps depend on the elastic modulus of each injection molded thermoplastic resin and the resistance of denture polymers was impacted by the flexural strength at the proportional limit of each injection molded thermoplastic resin. Therefore, some studies [12,13] evaluated the resistance of denture polymers to resin deformation and thermocycling.

PMMA absorbs relatively small amounts of water when placed in an aqueous environment [19]. It is known that water exerts significant effects on the mechanical properties of the polymer [19]. It is possible that in previous studies [21,23] the water molecules penetrated the denture base material when the dry specimens were immersed in water and then acted as plasticizers to facilitate the movement of polymeric chains in the denture base material; therefore, the elastic modulus of the denture base material decreased compared with that of the dry specimen. The microvoids that are then formed are filled with water molecules through inward diffusion. Both the outward leakage of the soluble constituents and the inward diffusion of water are time-dependent processes. Therefore, the relative amount of these molecules within the denture polymer changes over time until equilibrium is reached [27]. Water and plasticizers adversely affect the strength of the polymer to different extents because the molecules facilitate the movement of the polymer chains to varying degrees. The water sorption specimens of Valplast and Acrytone resembled Acron; they possessed a significantly lower elastic modulus and flexural strength at the proportional limit and were easier to deform than the dry specimens. However, LucitoneFRS, EstheShot, EstheShotBright and ReigningN were different from Acron. The flexural strengths at the proportional limit and elastic modulus of the 30-days water sorption specimens of LucitoneFRS, EstheShot, EstheShotBright and ReigningN were not significantly different from the dry specimens.

In the present study all of the specimens absorbed water (Figure 1) and water sorption of the polyamides (Valplast and LucitoneFRS) and PMMAs (Acrytone and Acron) were higher than that of other denture base resins; these results were similar to a previous study [10]. However, changes in mechanical properties were not associated with high or low water sorption. It is possible that the molecule structure is related to water sorption because water molecules interfere with the entanglement of polymer chains and thereby act as plasticizers. When this occurs, polymer chains generally become more mobile [19]. However, when the polymer chains are complex structures, polymer chains are hard to move. It is well known that polymers have complex ring structural formulae; however Valplast, Acrytone and Acron do not have the ring structural formula (e.g. benzene ring), in contrast to other materials. This study indicated that the water exerts significant effects on the mechanical properties of part of the denture base materials, especially Valplast and Acrytone. The elastic modulus and the flexural strengths at the proportional limit of Valplast and Acrytone were decreased by water sorption.

As mentioned earlier, the elastic modulus of the injection-molded thermoplastic resin for RPDs without metal clasps affects the retentive forces (e. g. the ease of insertion and removal of the RPD); hence, changes in the elastic modulus after water sorption is clinically very important. The flexural strengths at the proportional limit of the injection-molded thermoplastic resin for RPDs without metal clasps are attributed to permanent deformation to receive an external force (e.g. force of insertion and removal and occlusion force). The findings of this study suggest that the flexibility of the part of thermoplastic resin at the retention area in an RPD without metal clasps will change as the patient uses the denture over time, especially in the early stages. This makes it easier to deform an RPD without metal clasps. All of the injection-molded thermoplastic resins absorbed water. Therefore, the RPDs without metal clasps that are fabricated from injection-molded thermoplastic denture base resins must absorb water as long as possible before adjusting the retentive forces. This study was unable to demonstrate the minimum clinically relevant water exposure time. Because this study's specimens were 3.3 mm high, but retentive clasp arms of RPD without metal clasps has a thickness thinner than it [6]. It is presumed that the water sorption time indicated in this study is longer than that of a clinically-placed RPD without metal clasps. Clinically an aspect of concern when using polymer clasps in contact with the tooth surface relates to the adsorption of proteins and microbes by the polymer in higher quantities than metals [29–32], which may lead to an increased risk of caries in the abutment teeth. This question requires further investigation before large scale use of polymer clasps.

Conclusions

Under the present experimental conditions, the following conclusions can be drawn:

(1) Water sorption significantly decreased the flexural strength at the proportional limit of one of the polyamides and polymethylmethacrylate.
(2) Water sorption significantly decreased the elastic modulus of one of the polyamides and polymethylmethacrylate.

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