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Wear resistance of injection-molded thermoplastic denture base resins

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ABSTRACT

Objective This study investigated the wear resistance of injection-molded thermoplastic denture base resins using nanoindentation instrument.

Materials and methods Six injection-molded thermoplastic denture base resins (two polyamides, two polyesters, one polycarbonate, one polymethylmethacrylate [PMMA]) and a PMMA conventional heat-polymerized denture-based polymer control were tested. Elastic modulus, hardness, wear depth, and roughness were calculated using a nanoindentation instrument.

Results Elastic modulus and hardness of the injection-molded thermoplastic denture base resins were significantly lower than those of the PMMA conventional heat-polymerized denture-based polymer. Wear depth of polycarbonate and PMMA conventional heat-polymerized denture-based polymer were significantly higher than that of other injection-molded thermoplastic denture base resins. The roughness of injection-molded thermoplastic denture base resins was significantly more than that of PMMA conventional heat-polymerized denture-based polymer after testing.

Conclusions Wear resistance of injection-molded thermoplastic denture base was low compared to PMMA conventional heat-polymerized denture-based polymers.

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Nanoindentation; polyamide; polyester

Introduction

Injection-molded thermoplastic denture base resins, such as polyamide, polyester, polycarbonate, and polymethylmethacrylate (PMMA), have been used as material in removable partial dentures (RPDs) without metal clasps.[1–8] RPDs without metal clasps, but with denture base resin parts which are located to the undercuts of retaining teeth have developed for overcoming problems of poor esthetics, metal allergies, and fatigue failures of metal clasps.[9–11] Retention and bracing of RPDs without metal clasps could be achieved by extending a part of the denture base to the undercut area of the retaining tooth. Denture base resins of RPDs without metal clasps are required for high elasticity and durability.[6–9] Injection-molded thermoplastic resins are adequate for materials of RPDs without metal clasps, because injection-molded thermoplastic resins have advantageous characteristics, such as higher elasticity and impact strength compared to conventional heat-polymerized acrylic resin.[6–8]

In our previous studies,[6–8] the mechanical properties of injection-molded thermoplastic denture base resins (polyamides, polyesters, and polycarbonate) were examined. These studies revealed that (1) all of the injection-molded thermoplastic resins had significantly lower flexural strength at the proportional limit (FS-PL) and lower elastic modulus than conventional heat-polymerized acrylic resin, (2) the polyamides and the polycycloalkylene terephthalate copolymer thermoplastic resins had low FS-PL and low elastic modulus, (3) the thermoplastic resin composed of polyethylene terephthalate copolymer had moderately high FS-PL and moderate elastic modulus, and (4) the thermoplastic resin composed of polycarbonate had moderately high FS-PL and elastic modulus.

Despite these earlier studies, little known about how the hardness and wear resistance of injection-molded thermoplastic denture base resins. An evaluation of the hardness and wear resistance of denture base resins is beneficial for clinical purposes, because surface roughness has an effect on microbial adhesion to the surface. High surface roughness is caused by adhesive dental plaque and dental calculus among others.[12–14] Furthermore, resins are known to adsorb more proteins and microbes on their surface than metals and ceramics.[15–18] RPDs without metal clasps where there is contact of polymer part of the denture to the surface of retaining teeth and gingiva can potentially cause denture-induced caries, periodontal disease, and halitosis.

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Hardness is the resistance of a material to plastic deformation typically measured under an indentation load.[19,20] Hardness values have been used as a measurement of the mechanical property of materials and to predict wear resistance of many restorative materials. Several studies have found a correlation between hardness and wear resistance.[20–23] The other interesting property is elastic modulus which represents the tendency of a material to deform elastically when a force is applied. Elastic modulus is relative to the stiffness of a material [19] and may affect its wear resistance.[20]

A nanoindentation instrument was used for this study. Indentation is a versatile measurement procedure because of its relative ease of use. Advances in nanoindentation technology have made it a useful research tool for many different systems across size scales (macro to nano) and in numerous scientific disciplines.[24–26] The reasons for its popularity are the relative simplicity of specimen preparation and that a single specimen can be indented several times. Elastic modulus and hardness are the two mechanical properties most commonly measured using load and depth-sensing indentation techniques.[27] Moreover, some nanoindentation instruments can perform roughness measurements, wear testing, and scratch testing.

Evaluating the wear resistance, hardness, and elastic modulus of injection-molded thermoplastic denture base resins is important, but presently, there is insufficient information about it. The purpose of the present study was to evaluate the wear resistance of injection-molded thermoplastic denture base using a nanoindentation instrument. The hypothesis was that the wear resistance of injection-molded thermoplastic denture base resins did differ from each other.

Materials and methods

Test specimen preparation

Six injection-molded thermoplastic denture base resins were selected for this study, and a conventional heat-polymerized PMMA was used as a control (Table 1). Each denture base material was fabricated according to the manufacturers' instructions in gypsum molds with cavities (10 mm long, 10 mm wide, 3 mm high). The surfaces of each denture base materials were abraded under running water with up to 4000-grit silicon carbide paper (SiC-Paper, Struers A/S, Ballerup, Denmark) and were further polished with felt cloth (MD-Cloths and MD-Fuga, Struers A/S, Ballerup, Denmark) and 0.1 µm Al_2O_3 paste (AP-A Suspension, Struers A/S, Ballerup, Denmark) using a grinding machine (Rotopol-11, Struers A/S, Ballerup, Denmark).

Nanoindentation

Nanoindentation measurements were performed at 23 °C using a nanoindentation instrument (UBI 750, Hysitron, Minneapolis, MN, USA) equipped with a Berkovich (three-sided pyramidal) diamond tip. The load and displacement resolution of the instrument were 1.0 µN and 0.1 nm, respectively. Calibration indents were made on fused polycarbonate with a reduced modulus of 3.10 GPa and Poisson's ratio of 0.37.

Sixteen indentations were performed on randomly selected areas on each specimen ($n = 16$). The load of the indentater was up to a maximum force of 1000 µN with a constant speed of 200 µN/s. The load–displacement curve was generated after indentations. The software (TriboScan, version 9.1.1.0, Hysitron, Minneapolis, MN, USA) was used to calculate elastic modulus (E) and hardness (H).

Table 1. 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)	LucitoneFRS	DENTSPLY International Inc., PA, USA	Injection molding technique; heat processed at 300 °C for 17 min	100323A
Polyester (polyethylene terephthalate copolymer) (PET)	EstheShot	i-Cast Co. Ltd., Kyoto, Japan	Injection molding technique; heat processed at 230 °C for 20 min	IKB
Polyester (polycycloalkylene terephthalate copolymer) (PCTA)	EstheShot Bright	i-Cast Co. Ltd., Kyoto, Japan	Injection molding technique; heat processed at 280 °C for 20 min	2A6277240
Polycarbonate (PC)	ReigningN	Toushinyoukou Co. Ltd., Niigata, Japan	Injection molding technique; heat processed at 320 °C for 30 min	FMY31T
Polymethylmethacrylate (injection-PMMA)	Acrytone	High Dental Japan Co. Ltd., Osaka, Japan	Injection molding technique; heat processed at 260 °C for 25 min	1010087
Polymethylmethacrylate (conventional-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

The elastic modulus was calculated based on the slope of the upper portion of the unloading curve from the load–displacement data. The modulus obtained from analysis of the raw data (load–displacement curve) incorporated stiffness of the specimen and the indenter. This is usually referred to as the reduced modulus.[28,29] The relationship of the reduced modulus to the elastic modulus of the material is given by the following formula:

$$1/E^* = \left(1 - v^2\right)/E + \left(1 - v_{ind}^2\right)/E_{ind}$$

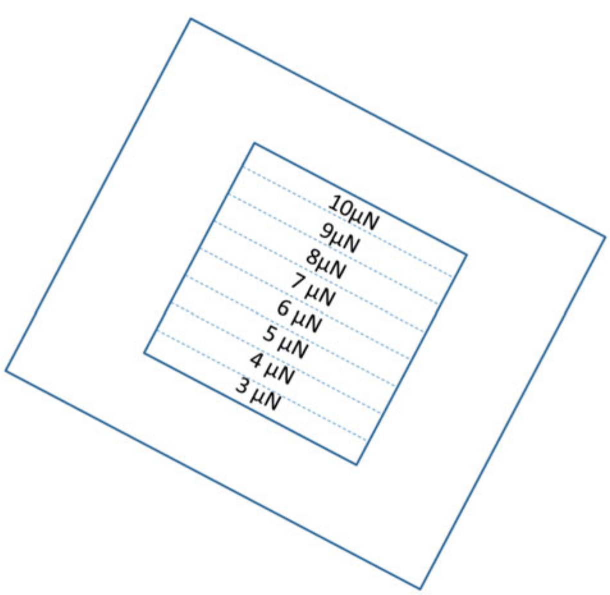
where E* is the reduced modulus, v and E are the Poisson's ratio and elastic modulus of the material, respectively and v_{ind} and E_{ind} are the Poisson's ratio (0.07) and elastic modulus (1141 GPa) of the indenter, respectively. The elastic modulus data were presented as the mean of 16 indentations. Hardness was obtained from depth-sensing indentation instruments according to the following formula:

$$H = P/A_P$$

where P is the applied load and A_P is the projected area of indentation. The hardness data were presented as the mean of 16 indentations.

Wear test

A nanoindentation instrument (UBI 750) using a three-sided pyramid diamond Berkovich tip at a constant wearing rate of 30 µm/s and a wear force of 3–10 µN, was used for polished specimens. A wearing area of 10 × 10 µm^2 of resin for 25 cycles was created (Figure 1). The morphology and depth of wear scratch were recorded and analyzed using the software available on the instrument (TriboScan). The wear depth was measured after wear testing. Roughness (Ra) was measured before and after wear testing using the instrument software ($n = 10$). The temperature of the testing chamber was the same as room temperature (23.0 ± 1 °C). All testing was performed under uniform atmospheric conditions of 23.0 ± 1 °C and 50 ± 1% relative humidity.



Statistical analyses

The roughness data were analyzed statistically using 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 wear testing. A one-way ANOVA (STATISTICA) was applied if there was a significant difference resulting from an interaction between these two variables ($p = 0.05$). The Newman–Keuls *post-hoc* comparison ($p = 0.05$) (STATISTICA) was applied when appropriate.

The other data were analyzed statistically using a one-way ANOVA (STATISTICA), and Newman–Keuls post-hoc comparison test (STATISTICA) was applied when appropriate (95% confidence level).

Results

All of the injection-molded thermoplastic resins had a significantly lower elastic modulus than the denture base control, Acron (PMMA) ($p < 0.05$). The elastic modulus of EstheShot, ReigningN, and Acrytone arranged in descending order were: ReigningN (3.46 ± 0.05 GPa) > EstheShot (3.14 ± 0.03 GPa) > Acrytone (2.98 ± 0.16 GPa) ($p < 0.05$). As a group, LucitoneFRS and EstheShotBright had a lower elastic modulus than EstheShot, ReigningN, and Acrytone and a higher elastic modulus than Valplast ($p < 0.05$) (Table 2).

All of the injection-molded thermoplastic resins had a significantly lower hardness than the denture base control, Acron (PMMA) ($p < 0.05$) and were significantly different from each other ($p < 0.05$). The hardness of all the thermoplastic resins arranged in descending order were: ReigningN (0.231 ± 0.004 GPa) > Acrytone (0.210 ± 0.008 GPa) > LucitoneFRS (0.193 ± 0.002 GPa) > EstheShot (0.184 ± 0.004 GPa) > EstheShotBright (0.179 ± 0.003 GPa) > Valplast (0.152 ± 0.006 GPa) (Table 2).

Acron and ReigningN had a significantly lower wear depth than other injection-molded thermoplastic denture base resins ($p < 0.05$). As a group, Valplast, EstheShot, EstheShotBright, and Acrytone had the

Table 2. Mean and standard deviation (SD) of elastic modulus (GPa), hardness (GPa), wear depth (nm), and roughness Ra (nm).

Denture base material	Elastic modulus (GPa)	Hardness (GPa)	Wear depth (nm)	Roughness Ra (nm)	
				Polished surface	After testing
Valplast	1.39 (0.02)	0.152 (0.006)	159.167 (16.866)a	17.08 (4.04)b	36.38 (3.34)
LucitoneFRS	2.45 (0.02)a	0.193 (0.002)	118.792 (18.446)	9.45 (1.24)c,d	33.37 (3.76)
EstheShot	3.14 (0.03)	0.184 (0.004)	167.041 (14.212)a	16.93 (1.11)a,b	61.78 (6.14)
EstheShotBright	2.46 (0.02)a	0.179 (0.003)	153.389 (20.245)a	12.43 (1.87)c	48.17 (6.72)
ReigningN	3.46 (0.05)	0.231 (0.004)	46.060 (13.024)b	8.21 (1.09)c,d	20.07 (3.21)a
Acrytone	2.98 (0.16)	0.210 (0.008)	152.408 (16.393)a	11.84 (0.78)c,d	28.65 (2.74)
Acron	5.41 (0.06)	0.368 (0.008)	51.835 (21.101)b	8.28 (0.55)d	11.78 (3.21)c,d

The same letter denotes groups that were not significantly different from each other in group of elastic modulus, hardness, wear depth, and roughness Ra ($p > 0.05$).

highest wear depth among the thermoplastic resins ($p < 0.05$).

The two-way ANOVA revealed that there were significant differences in roughness because of the denture base material variable and the interaction between the denture base material and the effect of wear testing ($p < 0.05$). A one-way ANOVA and the Newman–Keuls post-hoc comparison were applied to the denture base material/wear testing combination. The results are depicted in Table 2 and Figure 1. The roughness of all denture base resins after wear testing arranged in descending order were: EstheShot (61.78 ± 6.14 nm) > EstheShotBright (48.17 ± 6.72 nm) > Valplast (36.38 ± 3.34 nm) > LucitoneFRS (33.37 ± 3.76 nm) > Acrytone (28.65 ± 2.74 nm) > ReigningN (20.07 ± 3.21 nm) > Acron (11.78 ± 3.21 nm) ($p < 0.05$) (Table 2). As a group, Valplast and EstheShot had the highest roughness among LucitoneFRS, EstheShotBright, ReigningN, Acrytone, and Acron before wear testing ($p < 0.05$) (Table 2). The roughness of Acron before and after wear testing was not significantly different (Table 2) ($p < 0.05$).

Discussion

In this study, nanoindentation was successfully used for investigating the elastic modulus, hardness, wear depth, and roughness of the injection-molded thermoplastic denture base resins. Nanoindentation allows the investigation of selected material properties on small amounts of materials. Measurement of mechanical properties by nanoindentation has been suggested as advantageous over the conventional methods for its high resolution of force and accurate indent positioning.[27,30–32] Reasons for its popularity include the relative simplicity of specimen preparation and that a single specimen can be indented several times. Different volumes of materials can be examined by a suitable choice of tip geometry and load. However, the three-point bending test requires a large number of specimens for accurate measurement. Generally, nanoindentation can measure the elastic modulus and hardness of materials. Moreover, nanoindentation used in this study can evaluate wear testing and roughness of specimens.

In previous studies,[6,8] the mechanical properties of injection-molded thermoplastic denture base resins (polyamides, polyethylene terephthalate copolymer, polycycloalkylene terephthalate copolymer, and polycarbonate) were examined. It was found that (1) all of the injection-molded thermoplastic resins had significantly lower elastic moduli than conventional heat-polymerized PMMA, (2) the polyamides and polycycloalkylene terephthalate copolymer had low elastic moduli, (3) the polyethylene terephthalate copolymer had a moderate elastic modulus, and (4) the polycarbonate had a moderately high elastic modulus. In this study, all of the injection-molded thermoplastic resins had significantly lower elastic moduli than conventional PMMA ($p < 0.05$). The elastic modulus of EstheShot, ReigningN, and Acrytone arranged in descending order were: ReigningN > EstheShot > Acrytone ($p < 0.05$). As a group, LucitoneFRS and EstheShotBright had a lower elastic modulus than EstheShot, ReigningN, and Acrytone and a higher elastic modulus compared to Valplast. These results were similar to those in previous studies.[6,8] However, the elastic moduli values from nanoindentation were always higher than those from conventional methods as well in previous studies.[20,33] This discrepancy was attributed to three factors. First, denture base resins were associated with viscoelasticity. This time-dependent property was not accounted for in the current calculation which might have led to approximation errors. Second, the loading time of the three-point bending test is longer than that of nanoindentation test. Third, the higher indentation modulus value obtained could due to the different stress fields developed within the materials when subjected to different loading modes. In the three-point bending test, the measured flexural modulus was primarily due to both bending moment and shear deformation. While in the nanoindentation test, the deformation around the indenter was far more complex with the resulting deformation stress field being comprised of compressive, tensile, and shear. However, the nanoindentation test cannot supersede the three-point bending test, because nanoindentation cannot measure the ultimate FS and the FS-PL. Moreover, there may be no correlation in hardness values when different indenter shapes are compared.[34,35]

Despite previous studies,[1–9] little is known about how hardness compares among denture base resins of RPDs without metal clasps. In this study, all of the injection-molded thermoplastic resins had a significantly lower hardness than Acron ($p < 0.05$) and were significantly different from each other ($p < 0.05$). The hardness of all the thermoplastic resins arranged in descending order were: ReigningN > Acrytone > LucitoneFRS > EstheShot > EstheShotBright > Valplast (Table 2). Several authors have reported that a positive correlation exists between hardness and elastic modulus of material.[20] In this study, a positive correlation existed between hardness and elastic moduli of all denture base resins except for EstheShot. However, the hardness value of EstheShot was relatively low, in spite of its elastic modulus being high. Hardness may be influenced by several factors such as compressive strength, PL, and ductility.[36] Therefore, elastic modulus only cannot fully describe hardness.

Nanoindentation has been successfully used for investigating the hardness and elastic modulus of dental materials. However, the nano-scale wear of dental materials by nanoindentation has not been widely evaluated to the authors' knowledge.[37] Thus, the wear depth was evaluated by using forces ranging from 3 μN to 10 μN for the denture base resins (Figure 1). The wear depth of all tested specimens was derived from measuring the distance from a flat surface to the deepest wear. Acron and ReigningN had a significantly lower wear depth than other injection-molded thermoplastic denture base resins ($p < 0.05$). As a group, Valplast, EstheShot, EstheShotBright, and Acrytone had the highest wear depth among the thermoplastic resins ($p < 0.05$).

The result of this study does not support the suggestion that the hardness value of the material can provide a forecast of its wear resistance, because the results here have not shown any correlation between material properties and wear resistance. Furthermore, it was surmised that the wear depth was not sufficient to evaluate the wear resistance. Because the wear generated by the nanoindentation machine is ideal, it should resemble the wear pattern shown in Figure 4; however, only Acrytone and Acron indicated clear patterns of wear (Figure 4). Other denture base resins had irregular wear patterns (Figures 2 and 3). Even if the wear depths were the same, surface roughness differed. Despite there being no significant differences in the wear depths of

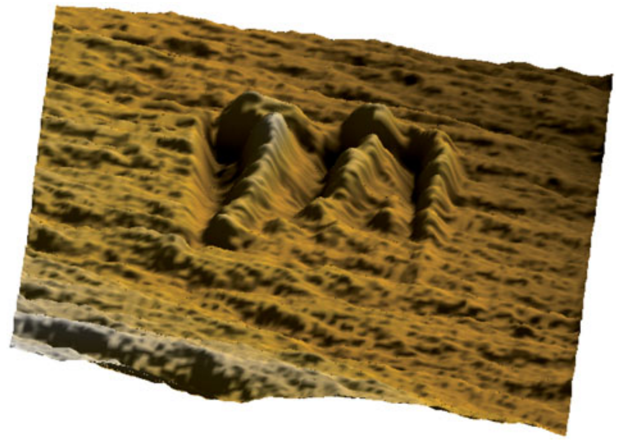


Figure 2. Wear image of Valplast, LucitoneFRS, EstheShot, and EstheShotBright.

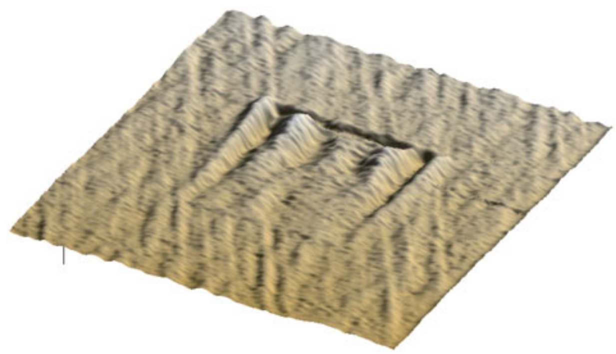


Figure 3. Wear image of ReigningN.

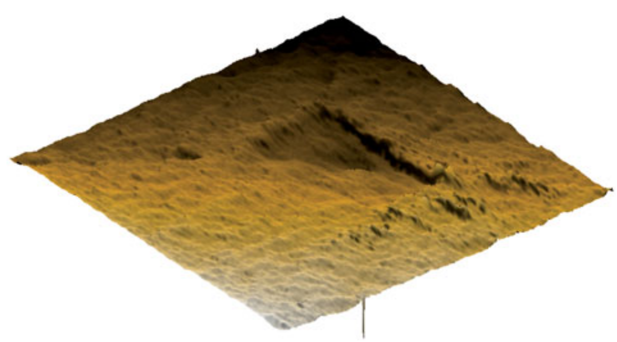


Figure 4. Wear image of Acrytone and Acron.

Valplast, EstheShot, EstheShotBright, and Acrytone, there were significant differences in wear after wear testing ($p < 0.05$). It was found that (1) the thermoplastic resin composed of EstheShot and EstheShotBright as polyester had high roughness, (2) the thermoplastic resin composed of Valplast and LucitoneFRS as polyamide and Acrytone as PMMA had moderately high roughness, (3) the thermoplastic resin composed of ReigningN as polycarbonate and Acron as PMMA had low roughness. These differences of clear and indistinct wear may be related to the viscoelasticity of denture base resins. PMMAs that demonstrated clear wear were less viscoelastic and brittle compare to other denture base resins. As a result of the aforementioned reasons, PMMA were worn by the nanoindentation's tip and most shavings were removed from the specimens. In contrast, polyamide, polyester, and polycarbonate were properties of viscoelasticity. The viscoelasticity of polymers refers to the ease and extent to which deformity including liquid-like flow may occur.

Polyamide, polyester, and polycarbonate were deformed as shown in Figures 2 and 3 at the tip and both sides of wear that be worn by the tip were raised upward from the flat surface. This finding was also reported in a previous study.[12] Rather than specimens were shaved by the tip, it is instead that specimens were deformed. It was anticipated that the viscoelasticity of denture base resins would be significantly affected. Acrytone had the highest wear depth in spite of PMMA, because Acrytone includes additives such as rubber in order to achieve a lower elastic modulus. Therefore, Acrytone had a high wear depth and ideal form of wear (Figure 4). Valplast and EstheShot had the highest roughness among denture base resins before wear testing. It was hypothesized that Valplast and EstheShot were hard to polish. The present result suggested that (1) a conventional heat-polymerized PMMA is easy to polish and hard to wear, (2) polycarbonate is easy to polish and moderately hard to wear, (3) polyamide is moderately easy to wear, and (4) polyester is easy to wear.

The surface roughness of dentures may have consequences for patient oral health. This is because excess surface roughness can promote the development of adhesive dental plaque and dental calculus. Furthermore, resins are known to adsorb more proteins and microbes on their surface than metals and ceramics.[15–18] RPDs without metal clasps where there is contact with the polymer part of the denture to the surface of retaining teeth and gingiva can potentially cause denture-induced caries, periodontal disease, and halitosis. Clinicians should be well aware of the properties of injection-molded thermoplastic denture base resins in order to choose one for an RPD without metal clasps that is suitable for each patient. Moreover, RPDs without metal clasps fabricated from injection-molded thermoplastic denture base resins require a level of maintenance far exceeding that of conventional RPDs. If an RPD without metal clasps is roughened, polishing may be applied.

Conclusions

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

(1) All of the injection-molded thermoplastic resins were worn more easily than conventional heat-polymerized acrylic resin.
(2) The two tested polyesters were exceedingly easy to wear.
(3) The two types of tested polyamides and one PMMA were moderately easy to wear.
(4) Polycarbonate was shown to be hard wearing.

Declaration of interest

The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper. This study is part of the BioCity Turku Biomaterials Research Program (www.biomaterials.utu.fi).

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