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The effect of different cooling procedures on mechanical properties

of denture base materials measured by instrumented indentation testing

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Abbreviated title: Cooling procedures and mechanical properties of PMMA denture base materials

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Original Article

Abstract

Purpose: To evaluate the different cooling procedures on the mechanical properties of five heat-cured polymethyl methacrylate (PMMA) denture materials.

Methods: 250 specimens were made equally from Meliodent (ME), Paladon 65 (PA), Probase Hot (PB), Stellon QC-20 (QC) and Vertex Rapid Simplified (VE) implementing five different cooling procedures (n=10/procedure): A) removal from water bath, bench-cooling (10 min) and cooling under water (15 min), B) remain in water bath till room temperature, C) removal from water bath and cooling in water for 15 min, D) removal from water bath and bench cooling till room temperature and E) removal from water bath, bench cooling for 30 min and cooling under water for 15 min. The specimens were immersed in distilled water (15 days/37°C) and then subjected to Instrumented Indentation Testing for Martens Hardness (HM), indentation modulus (E_{IT}) and elastic index (η_{IT}). Results were statistically analyzed by two- and one-way Analysis of variance (ANOVA) plus Tukey post hoc tests ($\alpha=0.05$).

Results: The highest values for HM were recorded for QC, PA, VE with B cooling procedure, PB with A and ME with E, for E_{IT} for QC, PB with A, for PA, VE with B and ME with E, and for η_{IT} for QC, PB with B, PB with E, ME with C and VE with D.

Conclusions: The cooling procedures recommended for PB resulted in the lowest mechanical properties. A and B may be considered as universal short- and long-cooling procedures respectively providing the highest mechanical properties for the materials tested.

1. Introduction

The most popular and reliable material used for fabrication of complete and partial dentures is still the conventional heat-cured poly methyl methacrylate (PMMA) [1], despite some recent developments in the field [2,3]. A great number of studies have been published concerning the physical [4,5] mechanical [6,7], chemical [8,9], biological [10,11] and aesthetic properties [12,13] of heat-cured PMMA. All these properties have established the effective clinical performance of this material in the wide spectrum of removable prosthodontic devices. Nevertheless, there are still fabrication steps, not standardized among the manufacturers of commercially available products, which raise questions on the rational of such instructions for a material being in service for almost a century.

One of these contradictory points is related to the cooling procedures advocated following heat curing. Currently, a variety of different, and sometimes controversial approaches are instructed including a combination of different temperature rates, incubation times at specific temperatures, use or not of water baths and different sequences between heating and cooling procedures, with very limited documentation on the advantages offered by each one. The results of studies investigating the effect of cooling procedure on denture base adaptation and linear dimensional changes have clearly documented that gradual cooling is the procedure of choice to avoid ill fitting dentures [14-16]. Surprisingly, the influence on the mechanical properties has been overlooked. This is important since currently fast-curing cycles have been advocated for many contemporary products, possibly leading to a lower conversion than traditional long-curing cycles [17] and therefore to inferior properties [18].

The aim of the present study was to evaluate the influence of different cooling procedures on mechanical properties of some commercial heat-cured PMMA denture base materials. The null hypothesis was that no statistically significant differences will be recorded in mechanical properties of the tested materials, independently of the applied cooling procedures.

2. Materials and methods

For the purpose of the study five commercial PMMA denture base materials were used (Table 1). Disk-shaped specimens (diameter=30 mm, thickness=3 mm) were made of each material (n=50), employing the heat-curing methodology proposed. The specimens were distributed in five subgroups (n=10) according to the cooling procedures instructed as summarized in Table 2. An additional cooling procedure (C: removal of flasks from water bath and cooling under running water for 15 min), considered as the fastest one, was also included in the study. After deflasking the specimen surfaces were wet-ground with SiC papers up to 1000 grit-size in a metallographic grinding/polishing machine (Dap-V, Struers, Ballerup, Denmark), ultrasonicated in water for 10 min and immersed in distilled water for 15 days at 37 °C. The specimens were then subjected to Instrumented Indentation Testing (IIT) employing a universal hardness testing machine (ZHU0.2/Z2.5, Zwick Roell, Ulm, Germany), equipped with a Vickers indenter. For each specimen, force-indentation depth curves were monitored under 9.8 N load with a 15 s dwell time. Three measurements were performed on each specimen, as specified and averaged. From the force-indentation depth curves the Martens Hardness (HM), the indentation modulus (E_{IT}) and the percentage of the elastic part of the indentation work (elastic index- η_{IT}) were determined, according to the ISO 14577-1 specification [19].

Each of the above mentioned properties were measured by the following equations:

$$HM = F / 26.43 \times h^2 \quad (1), \quad E_{IT} = 1 - (v_s)^2 / 1/E_r - 1 - (v_i)^2 / E_i \quad (2), \quad \text{and} \quad \eta_{IT} = W_{\text{elast}} / W_{\text{total}} \times 100 \quad (3)$$

For equations (1) and (2), F is the test force, h the indentation depth, v_s the Poisson's ratio of the specimen (acrylic resin=0.35) [1], v_i the Poisson's ratio of the indenter (0.07) and E_i the elastic modulus of the indenter (1140 GPa). The reduced modulus (E_r) was calculated using the equation $E_r = \sqrt{\pi / 2C} \sqrt{A_p}$ (4) where, C is the compliance of the contact determined by the slope of dh/dF of the unloading curve between 95 and 60 % of F_{max} and A_p the projected contact area under load in mm^2 . For equation (3), W_{elast} represents the work required for unloading, as determined by the area under the unloading curve and W_{total} the sum of energy

needed for the elastic and plastic deformation, as defined by the total area below the loading curve.

The results of HM, E_{IT} and η_{IT} passed the normality (Shapiro-Wilk, p-values 0.539-HM, 0.074- E_{IT} , 0.22- η_{IT}) and equal-variance (Levene's, p-values 0.44-HM, 0.063- E_{IT} and 0.114- η_{IT}) tests and were statistically analyzed by two-way Analysis of variance (ANOVA) employing the material type and cooling procedure as independent variables. To evaluate the effect of the heat-curing and cooling procedure as instructed by the manufacturers, an one-way ANOVA was additionally performed. Significant differences among individual groups were allocated by Tukey's post hoc multiple comparison analysis. In all cases a 95% confidence level was used ($\alpha=0.05$). Statistical analysis was performed by Sigma Plot 12.5 software (Systat Software Inc, San Jose, CA, USA).

3. Results

Representative force-displacement depth graphs are illustrated in Fig. 1. A peak force shifting towards left indicates an increase in hardness; the steeper the unloading curve the higher the E_{IT} and the smaller the unloading curve angle near zero load, the higher the elastic recovery.

The results of HM are presented in Table 3. The 2-way ANOVA revealed a statistically significant effect for the factors material type, cooling procedure and their interaction ($p<0.001$ for all). Due to the interaction, the effect of material type on HM within each treatment category was examined. Meliodent (ME) and Vertex Rapid Simplified (VE) were insensitive in the cooling procedures tested, demonstrating no statistically significant differences between them. Treatment E resulted in the lowest HM values from all other treatments in Stellan QC-20 (QC), Paladon 65 (PA) and ProBase Hot (PB). All other treatments were ranked in a statistically homogeneous group, whereas for QC the ranking of the statistical significance was A,B>D,C. Under the same cooling procedures statistically significant differences were found only in treatment E with a statistical ranking ME>QC,PA>PB, whereas VE showed insignificant differences from ME, QC and PA. Only

in PB the advised cooling procedure resulted in significantly lower values from the other procedures.

Table 4 summarizes the results of E_{IT} . The 2-way ANOVA showed no statistically significant differences for material type ($p=0.167$), but statistically significant differences for cooling procedure and their interaction ($p<0.001$ for both). Because of the interaction, the effect of material type per treatment on E_{IT} was examined separately. VE and PA, ME were not affected by the treatments used. Treatment E showed the lowest values in QC and PB. There were no statistically significant difference between groups A-D in PB. For QC insignificant differences existed between A-D and C-E treatment groups.

Comparison of the same treatment among the denture materials showed statistically significant differences only in treatment E between PB and ME, whereas QC, PA, VE had insignificant differences from PB, ME and among them. Again, the advised treatment for PB resulted in the lowest E_{IT} values.

For the results of η_{IT} (Table 5), a statistically significant difference ($p=0.002$) was recorded for cooling procedure and the interaction between material type-cooling procedure ($p=0.017$), whereas there was no statistically significant difference for material type ($p=0.059$). Since an interaction was identified, the influence of material type within each treatment mode on η_{IT} was evaluated by multiple comparisons, as before. The various cooling treatments had no effect on the η_{IT} of QC, PA, ME and VE. The only statistically significant difference was found in PB, where E treatment was significantly lower from all others, the latter demonstrating a statistically homogeneous group. The effect of the same treatment among the materials was significant only in treatments A and E. For treatment A significant differences were found between QC and PA, whereas for treatment E between PA and PB. In both cases the remaining materials demonstrated insignificant differences from each of the aforementioned products. In accordance with the previous findings, the proposed treatment for PB provided the lowest η_{IT} values.

Table 6 summarizes the results of the IIT properties per denture system, employing the heat-curing and cooling procedures instructed by the manufacturers. PB exhibited the

lowest IIT values from all the material tested. For the remaining materials statistically significant differences were found only in E_{IT} between QC and VE.

4. Discussion

According to the results of the present study, the null hypothesis should be partially accepted for PA and VE regarding HM and E_{IT} . For η_{IT} the null hypothesis was accepted for all materials except for PB.

The instrumented indentation testing (IIT) is a relatively new method for hardness measurements without relying on the optical assessment of the indentation [20]. The real time monitoring of the indentation process can provide simultaneously information for important material properties like the modulus, yield strength, viscoelastic response, creep, etc, through a simple HM measurement [21], without the need of setting up the array of traditional mechanical tests otherwise required. Moreover, this technique is free of the disadvantages associated with the microscopic measurement of the indentation in materials with elastic recovery [22].

The HM values recorded in the present study for heat-cured PMMA (116-189 MPa) are in agreement with previous IIT studies. More specifically, the HM of acrylic denture teeth was estimated as to 142 MPa [20], whereas for PMMA-based CAD temporary block materials ranged from 119-185 MPa, depending on the material microstructure and storage conditions [23,24]. Moreover, the results of conventional optical Vickers hardness measurements of heat-cured PMMA (10.2-16.5 VHN) [22,25,26] correspond to 100-162 MPa, values registered within the range measured in the present study.

The rigidity of a denture is clinically important for the even distribution of the masticatory forces to the underlying tissues [27]. Since flexural modulus is related to denture rigidity [28], an international standard has been issued (ISO 20795-1) [29], specifying 2 GPa as the lower limit of an acceptable flexural modulus, with higher values presenting better clinical behavior of the denture. The E_{IT} values recorded in the present study range between 3.2 and 4.6 GPa, which are in agreement with previous IIT study [23,24]. These values are higher from those calculated by three-point flexural strength tests (3PFS), where the flexural

modulus ranged from 1.7-2.77 GPa [2,26,30-32] including the materials tested in the present study 2.05 and 2.77 GPa for PA [30,32], 1.7-2.12 GPa for ME [2,31] and 2.51 GPa for VE [26].

The η_{IT} property reveals the elastic fraction of the total work spent for the entire deformation (when the indenter is in the deepest point). In the present study the tested materials exhibited η_{IT} values between 34-40%. For PMMA, a recovery of approximately 65% has been registered with conventional Vickers measurements mainly attributed to recovery of the depth of the indent and not of the diagonals [33]. Although direct comparison of these values is invalid, the elastic fraction given from the IIT measurements seems more reliable, since changes of the entire indentation are monitored.

Summarizing the results of the properties measured by IIT, it seems that hardness is within the same range with conventional optical measuring methods and the indentation modulus is higher in comparison with 3PFS tests.

The denture base materials tested were all based on MMA/PMMA liquid/powder systems. Differences in the crosslinker quantity and thermal history during curing may affect the crosslinking density and curing capacity of the PMMA polymer, anticipating variations in the mechanical properties. It has been documented that the crosslinking agent tetramethylene dimethacrylate (BDMA) is more reactive than ethyleneglycol dimethacrylate (EDMA) [34] with more pronounced effects in auto-cured than in heat-cured PMMA resins and that an increased amount of residual monomer has a detrimental effect in the mechanical properties of PMMA denture based materials, acting as a plasticizer [34,35]. Since the exact composition of these materials is proprietary, a reliable structure-property relationship cannot be made. Nevertheless, comparison of the mechanical properties between the product and processing procedures advised, assigned as a system, demonstrated statistically significant lower HM values in PB. For E_{IT} significant differences were registered only between QC, VE and PB, whereas for η_{IT} no significant differences were found. It is not known if these differences are associated with the curing cycles, the cooling-procedures or both.

The cooling rate of heat-cured polymers has been recognized as a means of controlling their crystallinity and shrinkage [36]. PMMA is amorphous, because the pendent groups do not allow the molecules to get close to form crystalline bonds; therefore the predominant effect of the cooling rate is on shrinkage, which is associated with ill fitting dentures. It has long been recognized that the least deformation of the prosthesis is attained when gradual post processing cooling is used (for 12 h or more) [14]. This is mainly related to the conformation of the polymer chains. When fast cooling rates are used, the polymer chains are highly constrained, with reduced segmental mobility and the resultant residual stresses are high, affecting the dimensional stability and fitting of the prosthesis. Worst results have been experience by sudden temperature reduction (quenching), that creates differential shrinkage vectors and contraction at various denture areas [14-16,37]. Despite these findings, two of the methods proposed by the manufacturers involve a quenching stage after a short period (15-30 min) of bench-cooling.

Residual stresses in PMMA are known to lower the effective softening temperature and impair the mechanical properties due to implications with water adsorption/desorption stresses [38,39]. Nevertheless, there is no information on the mechanical properties of PMMA subjected to the cooling procedures proposed. From the cooling treatments tested in the present study a discrepancy was noticed in the values of PB; the proposed procedure (E) resulted in significantly lower values in all the IIT parameters tested. The same procedure manifested the lowest values in HM for PA, QC and E_{IT} for QC. The reason for this performance may be related to the quenching after a prolonged bench-cooling period (30 min) in comparison with treatment A (10 min). In the former, the temperature after 30 min bench-cooling is expected to be lower than the glass transition temperature of PMMA (95-105°C), where the original soft, rubbery state becomes a hard, glassy state. Quenching in water at the glassy state may induce more internal stresses and affect the properties tested. This hypothesis is validated also by the results of treatment C, where immediate bench-cooling provided higher values than treatment E for PB.

The results of the present study show that the cooling rates have an impact on the mechanical properties of the PMMA denture base materials, besides the already documented effect on denture fitting. From the treatments tested, A and B can be proposed as universal short and long cooling procedures respectively for all the materials tested, since they provide the highest values of the IIT properties tested. Further studies are required to investigate whether these cooling procedures provide acceptable dimensional changes related to denture fitting and MMA conversion with minimal residual monomer.

5. Conclusion

Under the limitations of the present in-vitro study the following conclusions can be drawn:

- The cooling procedure employing bench-cooling for 30 min and then cooling under running water for 15 min demonstrated the lowest HM (QC, PA, PB), E_{IT} (QC, PB) and η_{IT} (PB) values.
- Two of the treatments tested, treatment A (bench-cooling for 10 min and then cooling under running water for 15 min) and treatment B (cooling in water-bath till room temperature) provided the highest HM, E_{IT} and η_{IT} values. Therefore, these treatments may be considered as short- (A) and long- (B) cooling procedures respectively for all the denture base polymers tested.

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CAPTION TO FIGURE

Fig. 1. Force-indentation depth curves representing high, medium and low hardness values for QC material employing B cooling procedure and PA, PB materials employing E cooling procedure. Shifting of the peak force value towards left indicates an increase in HM hardness, a steep inclination of the unloading curve implies a high E_{IT} and a small angle of the unloading curve at zero load, denotes a material with high elastic recovery.

Table 1 - The acrylic denture base products tested

Material	Lot Number	Code	Composition/curing method*	Manufacturer
Meliodent	Powder: 20141107-2 Liquid: R010059	ME	Powder: Product based on methacrylate monomers Liquid: MMA (>90%), BDMA (0-5%). Curing: Boiling water, switch heater off and leave for 15 min. Then boil for 20 min	Heraeus Kulzer GmbH, Hanau, Germany
Paladon 65	Powder: 010164 Liquid: R010048	PA	Powder: Methacrylate copolymonomers (0-5%), BPO <1% Liquid: MMA (>90%), BDMA(0-5%) Curing: Water, 80°C/15 min & 100°C/20 min	Heraeus Kulzer
ProBase Hot	Powder: UN0447 Liquid: V33082	PB	Powder: PMMA (>95%), BPO (1-1.5%) Liquid: MMA (50-100%), EGDMA (3-10%) Curing: Boiling water for 45 min	Ivoclar-Vivadent, Schaan, Liechtenstein
Stellon QC-20	Powder: 130CT053 Liquid: 13JUL117	QC	Powder: PMMA, BPO Liquid: MMA, HQ (0.01%) Curing: Water, 100°C/20 min	DeguDent GmbH, Hanau-Wolfgang, Germany
Vertex Rapid Simplified	Powder: XT382P02 Liquid: XT381L02	VE	Powder: PMMA (>99%), accelerator (<1%), colour agents (<1%) Liquid: MMA (>95%), crosslinker (<5%), accelerator (<1%) Curing: Water, 100°C/20 min	Vertex Dental B.V, Zeist, The Netherlands

* According to the manufacturers' information. MMA: Methyl methacrylate, PMMA: Polymethyl methacrylate, BPO: benzoyl peroxide, HQ: Hydroquinone, EGDMA: Ethyleneglycol dimethacrylate, BDMA: Tetramethylene dimethacrylate.

Table 2 - The cooling procedures used in the study

Procedure code	Procedure description	Instructed for products
A	Remove flask from water bath, bench-cool for 10 min and then cool under running water for 15 min	QC
B	Flask remains in water bath till room temperature	PA, ME
C	Experimental: Remove flask from water bath and cool under running water for 15 min	—
D	Remove flask from water bath and bench-cool till room temperature	VE
E	Remove flask from water bath, bench-cool for 30 min and then cool under running water for 15 min	PB

Table 3 - Mean values and standard deviations of Martens Hardness (HM)

Cooling procedures	HM (MPa)				
	QC	PA	PB	ME	VE
A	183±7 ^{a,c,A}	167±12 ^{a,A}	178±6 ^{a,A}	178±9 ^{a,A}	171±8 ^{a,A}
B	189±6 ^{a,c,A}	177±9 ^{a,A}	162±4 ^{a,A}	176±10 ^{a,A}	175±9 ^{a,A}
C	164±7 ^{b,A}	175±10 ^{a,A}	168±13 ^{a,A}	156±11 ^{a,A}	171±7 ^{a,A}
D	168±9 ^{b,A}	176±13 ^{a,A}	168±10 ^{a,A}	175±7 ^{a,A}	167±7 ^{a,A}
E	157±4 ^{d,A}	157±4 ^{b,A}	116±14 ^{b,B}	178±7 ^{a,C}	171±10 ^{a,A,C}

Same superscript letters show statistically insignificant differences ($p > 0.05$) among materials treated with the same cooling procedure (upper case) and among treatments for the same material (lower case). Bold numbers correspond to the values of the procedure advised by the corresponding manufacturer.

Table 4 - Mean values and standard deviations of indentation modulus (E_{IT})

Cooling procedures	E_{IT} (GPa)				
	QC	PA	PB	ME	VE
A	4.6±0.2 ^{a,A}	4.0±0.3 ^{a,B}	4.2±0.1 ^{a,A,B}	4.2±0.1 ^{a,A,B}	4.0±0.2 ^{a,A,B}
B	4.5±0.2 ^{a,A}	4.2±0.2 ^{a,A}	3.7±0.1 ^{a,A}	4.2±0.2 ^{a,A}	4.1±0.3 ^{a,A}
C	4.0±0.1 ^{a,b,A}	4.2±0.2 ^{a,A}	4.1±0.3 ^{a,A}	3.6±0.4 ^{a,A}	4.0±0.1 ^{a,A}
D	4.0±0.2 ^{a,b,A}	4.2±0.4 ^{a,A}	4.0±0.2 ^{a,A}	4.1±0.2 ^{a,A}	3.9±0.1 ^{a,A}
E	3.4±0.3 ^{b,A,B}	3.8±0.2 ^{a,A,B}	3.2±0.3 ^{b,A}	4.3±0.1 ^{a,B}	4.1±0.5 ^{a,A,B}

Same superscript letters show statistically insignificant differences ($p > 0.05$) among materials treated with the same cooling procedure (upper case) and among treatments for the same material (lower case). Bold numbers correspond to the values of the procedure by the corresponding manufacturer.

Table 5 - Mean values and standard deviations of elastic index (η_{IT})

Cooling procedures	η_{IT} (%)				
	QC	PA	PB	ME	VE
A	37±0.5 ^{a,A}	39±0.8 ^{a,B}	39±1 ^{a,A,B}	39±0.5 ^{a,A,B}	38±0.4 ^{a,A,B}
B	39±0.9 ^{a,A}	39±0.7 ^{a,A}	40±0.3 ^{a,A}	38±1.4 ^{a,A}	39±2 ^{a,A}
C	37±1.4 ^{a,A}	38±0.3 ^{a,A}	39±0.8 ^{a,A}	40.0±4 ^{a,A}	39±1 ^{a,A}
D	38±0.6 ^{a,A}	39±0.8 ^{a,A}	39±1 ^{a,A}	39±0.7 ^{a,A}	39±0.3 ^{a,A}
E	37±3 ^{a,A,B}	40±1.2 ^{a,A}	34±3 ^{b,B}	37±0.9 ^{a,A,B}	37±1 ^{a,A,B}

Same superscript letters show statistically insignificant differences ($p > 0.05$) among materials treated with the same cooling procedure (upper case) and among treatments for the same material (lower case). Bold numbers correspond to the values of the procedure advised by the corresponding manufacturer.

Table 6 - Mean values and standard deviations of the IIT properties for the materials prepared according to the manufacturers' instructions

Property	QC	PA	PB	ME	VE
HM (MPa)	183±7 ^a	177±9 ^a	116±14 ^b	176±10 ^a	167±7 ^a
E_{IT} (GPa)	4.6±0.2 ^a	4.2±0.2 ^{a,b}	3.2±0.3 ^c	4.2±0.2 ^{a,b}	3.9±0.1 ^b
η_{IT} (%)	37±0.5 ^a	39±0.7 ^a	34±3 ^b	38±1.4 ^a	39±0.3 ^a

Same superscript letters show statistically insignificant differences ($p > 0.05$) between the materials tested per property.

Fig. 1