Laboratory Evaluation of Newly Formulated Thermoplastic Resin Complete Denture Base Materials

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Abstract: Aim of the work: To investigate the physic-mechanical properties of newly formulated thermoplastic denture base material. Materials and Methods: Two parallel groups according to the denture base material used Group (A) conventional heat cure acrylic resin i.e. control group, Group (B) Vertex thermosens i.e. test group, 35 specimens each (14 impact, 14 flexural and 7 warpage) will be examined in this study. Each group will be divided into 2 subgroups: (I) No Thermocycling and (II) Thermocycling then subjected to 3 different tests, Impact strength, flexural strength and warpage. Results and Conclusions: 1. The vertex thermosens denture base material exhibited significantly higher impact and flexural strength as compared with conventional PMMA. 2. There was significant increase in impact strength of vertex thermosens and non statistically significant of flexural strength of both groups after thermocycling. 3. Warpage of vertex thermosens was statistically significant higher than conventional PMMA. 4. There was a significant increase of warpage after thermocycling for both groups.

Keywords: Denture base materials, polyamide, thermosens, impact, flexural, warpage, thermocycling.

1. Introduction

Poly methyl methacrylate (PMMA) resin has been widely used as a denture base material due to its desirable properties of excellent esthetics, ability to repair, low water sorption and solubility, adequate strength, low toxicity, and simple processing technique.[1, 2]

Poly methyl methacrylate denture base has dominated the market for more than 50 years.[3]

Conversely some disadvantages have also been described, such as polymerization shrinkage, weak flexural, lower impact strength, low fatigue resistance and in cases where patient is allergic to monomer.[4-6]

These often lead to denture failure during chewing or when fall out of the patient’s hand. Continuous efforts to increase material strength to decrease the risk of denture fractures can be listed as:

(1) reinforcement of denture materials by adding filling materials,[7] (2) changing the chemistry of the denture base polymer by copolymerization and cross-linking of resin materials, (3) incorporation of techniques new to the dental field; and (4) manufacturing new materials with increased resistance to fracture.[8, 9] One technique that improves the physical properties of dentures is injection molding technique to allow directional control of the polymerization process through the flask design, and involves a constant flow of new material from the sprue, thereby compensating for polymerization shrinkage.[10, 11]

Thermoplastic materials, such as polyamides (nylon plastics) were developed: acetal, polycarbonate, acrylic and nylon (resin). Nylon is a generic name used for certain types of thermoplastic polymers belonging to the class known as polyamides.[12] A new material called ThermoSens is superior to standard polyamide materials. The flexibility of this material can be controlled, and its shrinkage is extremely low. Owing to its composition, a homogenous color can be achieved, making this material suitable for the preparation of full dentures.[13]

In present study, the most important physico-mechanical properties were made to compare between the polyamide based denture base material (Vertex™ ThermoSens, Vertex-Dental B.V. The Netherlands) as and conventional poly methyl methacrylate resin PMMA (Acrostone, Egypt) and test the hypothesis that those properties could be affected by thermocycling.

2. Materials and Methods

The materials that used in this study were summarized in table 1.

Table 1: Materials used in the study

<table>
<thead>
<tr>
<th>Manufacture</th>
<th>Material</th>
<th>Manufacturing method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acrostone, Egypt.</td>
<td>PMMA</td>
<td>Compression method technique using conventional curing unit (long curing cycle) 70°C for 7-9 hours then 100°C for 30 minutes.</td>
</tr>
<tr>
<td>Vertex-Dental B.V. 3705 HJ Zeist The Netherlands.</td>
<td>Polyamide</td>
<td>Injection method technique using a pressure of 6.5 bar with preheating of the cylinder of the thermoject machine to 290°C for 18 minutes.</td>
</tr>
</tbody>
</table>

The following laboratory tests for evaluation of physico-mechanical properties were done: Impact strength test (IS), Flexural strength test (FS) and Warpage.
A total of 70 specimens that were prepared according to the international standards specifications for each test. The specimens were divided into two groups as following:

**Group (A) i.e (control group):** Specimens of conventional heat-cure acrylic denture base material (PMMA).

**Group (B) i.e (study group):** Specimens of thermoplastic resin material (Vertex™ Thermosens).

Each group was divided into 2 subgroups:

**Subgroup I:** Specimens were stored in the incubator at 37°C for 24 hours (called thermocycle zero or no thermocycle).

**Subgroup II:** Specimens for each test were thermocycled to examine the effects of temperature variations.

### Specimens Preparation

1. **For impact strength test:** Metallic mold having the dimensions of impact strength test specimens according to the method described in the ISO Standard (ISO 179-1; 2010) bar shaped of 75mm length, 10mm width, 10mm thickness with notch of 2mm depth at the mid-span was constructed.

   Base plate wax softened and poured into the mold. After hardening, the wax was removed. A total of 28 wax specimens were prepared according to the type of denture base materials. For construction of specimens the 28 test specimens were divided equally into two groups (A&B) of 14 specimens each. **Subgroup I:** 7 specimens were stored in distilled water at 37°C in the incubator. Time (time taken by the specimens to be transferred from one bath to another) of 5 seconds. **Subgroup II:** 7 specimens were thermocycling using a thermocycling machine for 500 cycles between 55°C and 5°C with a dwell time (time taken by the specimens in each bath) of 60 seconds and transfer.

   The specimens were accurately mounted in the vice of the pendulum testing machine (charpy type impact tester) (Mfd for Dayton Electric Co. Chicago 60648 USA (CEDM-4)).

   The specimens were struck at the mid-span by the pendulum as it releasing out of position (The force was applied to the specimen from the unnotched side).

   The charpy impact strength of the specimen was calculated using the following equation [14, 15]:

   \[
   \text{Impact strength} = \frac{E}{bd} \times 10 \text{J}
   \]

   **Where:**

   - E: is the impact absorbed energy in joules.
   - b: is the width in millimeters of the test specimens.
   - d: is the thickness in millimeters of the test specimens.

2. **For flexural strength test:** Metallic mold having the dimensions of flexural strength test specimens according to the American Dental Association Specification No.12 for denture base polymer (65 mm length, 10mm width and 2.5 mm thickness) was constructed. Base plate wax softened and poured into the mold. After hardening, the wax was removed. A total of 28 wax specimens were prepared Fig. (2).

   According to the type of denture base material used for construction of specimens the 28 test specimens were divided equally into two groups (A&B) of 14 specimens each, processing was done according to the manufacturer instructions. In order to test the effect of thermocycling on the flexural strength of the specimens under testing, the specimens of each group (A&B) were divided into equal subgroups of seven specimens each. **Subgroup I:** 7 specimens were stored in distilled water at 37°C in the incubator. **Subgroup II:** 7 specimens were thermocycling using a thermocycling machine. Individual specimens were placed on supports of the universal testing machine (Comten Industries, Inc. St. Petersburg, Florida, USA, Model No. 942D10-20). While placing the specimen on the testing device, care was taken that the central loading plunger was touching the midline of the sample. The force in Newton was applied perpendicular to the center of specimen strips and the specimen was gradually loaded at a crosshead speed of 0.5 mm min⁻¹. The maximum fracture load was measured for Acroston (group A). Thermosens specimens (group B) did not fracture, so the load was applied till maximum capacity of the three-point testing device will be recorded. The flexural strength will be calculated according to the following equation [6, 16]:

   \[
   \text{Flexural strength} = \frac{3PL}{2bd^2} \text{ (Mpa)}
   \]

   **Where:**

   - P = load at fracture (or, load at maximum deflection).
   - L = distance between the two supporting point (50 mm).
   - b = width of the test specimens (10 mm).
   - d = thickness of specimen (2.5 mm).

   ![Figure 2: Flexural test specimen.](image)

3. **For warpage test:** A sectional maxillary metal cast which nearly represents the dimensions of the edentulous ridge was constructed. Five fine parallel lines were inscribed on the posterior aspect of the metal master cast to be used as a reference measuring locations. A rubber base mold was prepared for this metallic cast.

   1) A mix of pure stone with a constant water/powder ratio 3:1 was maintained for all casts. Then the mix poured in the rubber base mold which was placed on a vibrator to remove any air bubbles present in the mix. After hardening, the stone casts were removed.

   2) Following the previous steps identical standardized sectional stone casts were prepared for all the experiment.

   3) A uniform baseplate wax was adapted upon each a sectional stone cast with a 2.0mm thickness and sealed at the peripheries.
4) Flasking, wax elimination (dewaxing), packing, curing and deflasking was performed carefully to avoid damage of casts. Polishing was usually carried out under running water to avoid rise in temperature and subsequent warpage by using motor lathe.

The posterior peripheral seal area displays the greatest discrepancy of adaptation after processing due to shrinkage of denture base material and strain release induced by thermal change[17].

These discrepancy or space observed between the denture bases and their casts. The vertical distance between the end of the finished plate and the original standardized casts were measured at “five locations” at the posterior palatal border. Location (C) represent nearly the center of the palate. Location (R) and (L) represent nearly the crest of the ridge. location (A) and (B) represent nearly the intermediate points between the center of the palate (C) and the crest of the ridge (R, L)[18] Fig. (3).

The measurement were carried out by using Stereomicroscope with camera attached was used to Stereomicroscope(SZII, Olympus optical co.ltd.2-43, 2Hatagaya, Shibuy-Ku, Tokyo, Japan.)

The measurement of warpage (for each specimen in different groups) were carried out at the following stages:
1. Immediately after processing, deflasking and finishing of plates.
2. After stored in distilled water at 37°C in the incubator for 24 hours.
3. After thermocycling the specimens for 500 cycles between 55°C and 5°C[17].

The captured photographs were loaded on an image-J software that was used to measure the distance in micron between the predetermined location on the cast and the base plate in vertical direction.

3. Results

Data were fed to the computer and analyzed using IBM SPSS software package version 20.0. Quantitative data were described using range (minimum and maximum), mean, standard deviation and median. Significance of the obtained results was judged at the 5% level.

Impact Strength Test

During testing, all the specimens of two groups fractured immediately after the first strike producing a clean fracture surface, the fracture line start from the base of the v-shape notch and extended downward. All the fractured specimens could be reassembled. Table (2) & figure (4) compare the mean values of impact strength of specimen for both groups at different conditions, and represent the effect of thermocycle on them.

- Man whitney test was used for comparing between the two studied group at different condition.
- In general the impact strength of thermostens was higher than conventional PMMA.
- Wilcoxon signed ranks test was used for comparing between Zero and after thermocycle for each group where (Z2=1.572) (p=0.116) for group A & (Z2=2.028) (p=0.043) for group B.
- At zero thermocycle the results were analyzed and compared between the two studied groups which revealed a significant increase in impact strength for group II (Z1=2.310)(P=0.021).
- After thermocycling, there was significant difference in the impact strength with (Z1=2.719)(P=0.007) of vertex thermostens.

### Table 2: Comparison between the mean values of impact strength for the two studied groups and subgroups in (KJ/m²)

<table>
<thead>
<tr>
<th>Impact Strength values</th>
<th>Group A Conventional PMMA (n = 14)</th>
<th>Group B Vertex thermostens (n = 14)</th>
<th>Z1</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Zero thermocycle</strong></td>
<td>Min. – Max.</td>
<td>12.50 – 16.25</td>
<td>12.50 – 25.50</td>
<td>2.310*</td>
</tr>
<tr>
<td></td>
<td>Mean ± SD.</td>
<td>15.02 ± 1.38</td>
<td>20.80 ± 4.04</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Median</td>
<td>15.60</td>
<td>21.30</td>
<td></td>
</tr>
<tr>
<td><strong>After thermocycle</strong></td>
<td>Min. – Max.</td>
<td>15.0 – 35.0</td>
<td>16.50 – 173.7</td>
<td>2.719*</td>
</tr>
<tr>
<td></td>
<td>Mean ± SD.</td>
<td>18.73 ± 7.28</td>
<td>87.77 ± 70.46</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Median</td>
<td>15.60</td>
<td>52.50</td>
<td></td>
</tr>
<tr>
<td>% of change</td>
<td>1.572 (0.116)</td>
<td>2.028 (0.043)</td>
<td>119.09</td>
<td></td>
</tr>
</tbody>
</table>

Z1: Z value for Mann Whitney test for comparing between the two studied groups
Z2: Z value for Wilcoxon signed ranks test for comparing between Zero and after thermocycle for each group.
*: Statistically significant at p ≤ 0.05.
Flexural Strength Test:
During testing, the acrylic specimens for group A showed gradual bending then finally broken in clean cut. In all the specimens the fracture line was sharp and located nearly at the center of the specimens where the load applied. All fractured specimens could be reassembling and the line of fracture was not clearly seen. In group B the specimens showed gradually bending till the maximum deflection capacity of the material without fracture.

- Table (3) and figure (5) show the mean flexural strength values for both studied groups (i.e. group A and group B) at both condition (i.e. zero thermocycling and after thermocycling).
- When compare the two groups we found that the thermosens denture base material has a higher flexural strength than conventional denture base material in both (zero and after thermocycling) with significantly different ($t_1$=7.396) ($p<0.001$) at zero thermocycle and ($t_1$=3.476) ($P=0.005$) after thermocycling.

Table 3: Comparison between the two studied groups according to Flexural Strength in Mpa at zero thermocycle and after thermocycle.

<table>
<thead>
<tr>
<th>Flexural Strength test</th>
<th>Group A Conventional PMMA (n = 14)</th>
<th>Group B Vertex thermosens (n = 14)</th>
<th>$t_1$</th>
<th>$P$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zero thermocycle</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Min. – Max.</td>
<td>85.01 – 148.9</td>
<td>181.9 – 232.1</td>
<td>7.396</td>
<td>$&lt;0.001^*$</td>
</tr>
<tr>
<td>Mean ± SD.</td>
<td>119.1 ± 29.12</td>
<td>212.6 ± 16.53</td>
<td>7.396</td>
<td>$&lt;0.001^*$</td>
</tr>
<tr>
<td>Median</td>
<td>120.9</td>
<td>216.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>After thermocycle</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Min. – Max.</td>
<td>118.7 – 178.4</td>
<td>175.6 – 290.4</td>
<td>3.476</td>
<td>0.005*</td>
</tr>
<tr>
<td>Mean ± SD.</td>
<td>141.4 ± 26.06</td>
<td>201.2 ± 39.19</td>
<td>3.476</td>
<td>0.005*</td>
</tr>
<tr>
<td>Median</td>
<td>127.9</td>
<td>191.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$t_1$ (p)</td>
<td>1.369 (0.220)</td>
<td>0.630 (0.552)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>% of change</td>
<td>118.72</td>
<td>44.42</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$t_1$: Student t-test for comparing between the two studied groups.

Warpage Test:
Warpage appears in the form of separation of the plate from the stone cast. The mean value of space between the plates and their stone casts at different location measurement for two groups in all observational stages are presented in Table (4).

The analysis of data showed that the amount of space between the plates and their stone casts in both groups was greatest at the center of plates and gradually decreased toward the crest of the ridge (fig. 6a, b). When compared the two study groups (A and B) by using Student t-test & ANOVA with repeated measures in Table (5) figure (7) at each stages the P value was statistically different at each different level ($p<0.001$).

Data was expressed by using mean ± SD.

Table 4: The mean values ± SD of spaces in micron at each location for both groups.

<table>
<thead>
<tr>
<th>Measurements points</th>
<th>Group A Conventional PMMA</th>
<th>Group B Vertex thermosens</th>
</tr>
</thead>
<tbody>
<tr>
<td>R (Mean ± SD)</td>
<td>153.8 ± 75.7</td>
<td>194.8 ± 50.1</td>
</tr>
<tr>
<td>A (Mean ± SD)</td>
<td>193.5 ± 62.2</td>
<td>258.7 ± 77.7</td>
</tr>
<tr>
<td>C (Mean ± SD)</td>
<td>247.4 ± 100.3</td>
<td>275.9 ± 69.1</td>
</tr>
<tr>
<td>B (Mean ± SD)</td>
<td>217.8 ± 86.2</td>
<td>233.8 ± 62.1</td>
</tr>
<tr>
<td>L (Mean ± SD)</td>
<td>192.2 ± 53.6</td>
<td>228.1 ± 83.6</td>
</tr>
</tbody>
</table>

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**Figure 6a:** Represent the mean values of spaces at each measurement location in three different stages for group A.

**Figure 6b:** Represent the mean values of spaces at each measurement location in three different stages for group B.

**Table 5:** Comparison between the mean values of warpage in micron for the two studied groups before and after thermocycle

<table>
<thead>
<tr>
<th>Warpage</th>
<th>Immediately</th>
<th>Zero thermocycle</th>
<th>After thermocycle</th>
<th>F</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A Conventional PMMA (n = 35)</td>
<td>Min. – Max.</td>
<td>75.30 – 409.6</td>
<td>108.6 – 385.9</td>
<td>115.0 – 483.5</td>
<td>8.725*</td>
</tr>
<tr>
<td></td>
<td>Mean ± SD.</td>
<td>201.0 ± 79.23</td>
<td>238.3 ± 71.11</td>
<td>264.4 ± 83.7</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Median</td>
<td>185.7</td>
<td>235.0</td>
<td>257.4</td>
<td></td>
</tr>
<tr>
<td>Sig. bet. periods</td>
<td>p&lt;0.009*, p2=0.001*, p3=0.068</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>% of change</td>
<td>↑18.56</td>
<td>↑31.54</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

| Group B Vertex thermosens (n = 35) | Min. – Max.  | 97.92 – 691.93 | 84.20 – 875.0 | 215.4 – 884.0 | 19.158* | <0.001* |
|            | Mean ± SD.  | 352.3 ± 142.2 | 406.6 ± 158.3 | 502.2 ± 156.0 |         |         |
|            | Median      | 313.6           | 378.6           | 481.9     |         |         |
| Sig. bet. periods | p1=0.065, p2<0.001*, p3<0.001* | | | | |
| % of change | ↑15.41      | ↑42.55         |       |         |         |         |
| t          | 5.501*      | 5.740          | 7.948* |         |         |         |
| p          | <0.001*     | <0.001         | <0.001 |         |         |         |

- t: Student t-test
- F: F test (ANOVA) with repeated measures
- Sig. bet. periods was done using Post Hoc Test (LSD) for ANOVA with repeated measures
- p1: p value for comparing between Immediately and Zero thermocycle
- p2: p value for comparing between Immediately and After thermocycle
- p3: p value for comparing between Zero thermocycle and After thermocycle
- *: Statistically significant at p ≤ 0.05

**4. Discussion**

**Impact Strength Test**

Impact strength is an important property for denture base materials which have tendency to fracture if accidentally dropped on to a hard surface.[14] Therefore the impact strength test was conducted in this study as one of the tests for evaluation of mechanical properties of denture base.

As regards the this study, when compare the mean values of impact strength of specimen for both groups at different conditions, and represent the effect of thermocycle on them, we founded that Vertex thermosens showed significantly higher impact strength than conventional PMMA in both situation. This is may be due to, polyamide structure is based primarily on aliphatic chains.[19] The backbone of Polyamide is regular and symmetrical, so forms very good resistance to shock and repeating stress.[7]
In addition, Porosities of PMMA more than that of Polyamide denture base material, this comes from presence of residual monomer and its evaporation leads to formation of these porosities, also the compression packing technique of conventional PMMA leads to more porosities than injection molding processing of polyamide.[20] Polyamide contains flexible agents in its composition, so it absorbs more energy to fracture.[21] Generally, our finding are supported by Ameer (2012)[3] and Takahashi et al (2012).[22]

Flexural Strength Test

Flexural strength is an important parameters that can reflect the ability of a denture base material to withstand functional masticatory forces. The three-point flexural strength test is useful in comparing denture base materials because it simulates the type of stress that is applied to the denture during mastication.[23, 24]

Moreover, the flexural strength test is a combination of tensile and compressive strength tests and includes some of the elements of proportional limit and elastic modulus measurements.[25] When comparing (group A& group B), the vertex thermosens flexural strength was significantly higher than that conventional PMMA in both condition (Zero thermocycle& after thermocycling).

This may be due to different in the composition of denture base material and polymerization technique, Our finding are supported by Takahashi et al (2012)[22] and disagree with Yunus et al (2005)[23] and Ameer (2012)[3] they reported that flexural strength of polyamide was lower than conventional PMMA.

Warpage Test

Warpage appears in the form of separation of the plate from the stone cast and was selected for testing the specimens since an accurate fit is relatively important because the distance between the base and supporting tissue is one of the principal factors in retention and control of the amount of forces necessary to dislodge the dentures.[26]

The amount of warpage for both groups was measured at the posterior palatal area of sectional plates placed on their stone casts at five predetermined references locations in different stages; because it can be easily measured and greater degree of dimensional changes were observed in this region in previous study.[27]

Since it is more susceptible to strain release because it is flatter and less restrictive than anterior region due to its anatomy. The results of this study showed that the greatest dimensional change between the plates and stone cast occurred in the median region of the palate (location c) and gradually decrease toward crest of the ridge. The explanation is that the processing shrinkage which occurred in the upper denture tends to draw the flanges inward on the lateral aspects of the tuberosities and as a result the denture was raised from the palate.

When comparing between two groups and sub groups, there were significant difference between conventional PMMA & vertex thermosens at all different stages, the amount of warpage in group B was higher than group A. The dimensional characteristics of processed denture bases are affected by many factors, such as the type of acrylic, type of investment material, method of resin introduction and temperature used to activate the polymerization process, separating medium used and number of sprues used.[28]

Our finding is supported by Parvizi et al (2004)[28] and Ameer(2012).[17]

5. Conclusions

Within limitations of this in vitro study, the following conclusions were drawn:

1) The vertex thermosens denture base material exhibited significantly higher impact and flexural strength as compared with conventional PMMA.

2) There was significant increase in impact strength of vertex thermosens and non statistically significant of flexural strength of both groups after thermocycling.

3) Warpage of vertex thermosens was statistically significat higher than conventional PMMA.

4) There was a significant incease of warpage after thermocycling for both groups.

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