Evaluating the Effect of Polyetheretherketone Particles Addition on Water Sorption and Solubility of Polymethylmethacrylate Denture Base Material.

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Abstract
Aims: To assess water sorption and solubility of PMMA after incorporating with polyetheretherketone particles (vectrex peek polymer PEEK) material with a different percentage (1%, 2%, and 3%). Materials and Methods: The PEEK particle size of 130μm. Peek with (1%, 2%, 3% by wt.) concentrations were added to the PMMA resin base to compare with the PMMA with no additives (control). The conventional heat-curing method was applied using a water bath to polymerize the specimens and study water sorption and water solubility of prepared samples. Study data was analysed via One-way ANOVA performed at significant P-value of (p≤0.05) and confidence. Fourier transform infrared spectroscopy used to detect any chemical modification occurs. Results: After comparing the results show significant difference in Water sorption of PMMA/PEEK. Water Solubility show no significant difference comparing to other tested groups (p≤0.05). Conclusions: the PEEK particles use it as dental filler at 1%, 2% and 3% wt. incorporating PMMA material with 3% PEEK material have lowest mean value for water sorption and solubility.

الخلاصة
الأهداف: الهدف من هذه الدراسة هو تقييم خواص الامتصاص والذوبان في الماء لـ بولي ميثيل ميالاكريدك بعد دمجه مع مادة بولي إتر إكريلك بنسب مختلفة. المواد وطرق العمل: حجم جسيم بولي إتر إكريلك = 130 ميكرومتر. تم إضافة نسبة (1% و 2% و 3%) من بولي إتر إكريلك إلى قاعدة راتينغ بولي ميثال ميالاكريدك. تم تحقيق مركب بولي ميثال ميالاكريدك/بولي إتر إكريلك بني وبينة من تسع باصدقاء مع ثلاث نسب مختلفة من المكونات الحرارية المختلطة باستعمال جهاز مائة لدراسة مقدار اختلاف الماء والذوبان بالبلاك. تم تحليل بيانات الدراسة عبر اختبار ANOVA أحادي الاتجاه (اختيار ما بعد المختصر / Tukey) (p<0.05). العناصر الفنلية في الأمطار تحت الحمراء وهو أسلوب طيفي شائع يستخدم كيميائي عضوي وغير عضوي، ويستخدم لكشف أي تغيير كيميائي يحدث. النتائج: بعد مقاومة النتيجة، نلاحظ وجود فرق في اختلاف الماء والذوبان. الاستنتاجات: ملاحظات على وجود اختلاف في اختلاف الماء والذوبان بالبلاك. النتائج في اختلاف الماء والذوبان بالبلاك كمعدلة عند دمجها مع بولي ميثال ميالاكريدك بنسبة (1% و 2% و 3%). يناسب 1% الأكثر تقليل اختلاف الماء والذوبان بالبلاك مادة بولي ميثال ميالاكريدك الأساسية لذوبان الأنسان.

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INTRODUCTION

The most popular denture base material is heat–cured acrylic resin poly methyl methacrylate (PMMA). It is including good aesthetics, accurate fit, stability in the oral environment, easy laboratory and clinical manipulation and inexpensive equipment (1, 2). PMMA possesses dimensional change during processing and service. PMMA absorb water slowly over period of time. This imbibition is due to the polarity properties of the resin molecules (3, 4).

Polyetheretherketone (PEEK) is newly innovative polymer with outstanding thermal and mechanical properties. It is light in weight, non-toxic, highly resistant to corrosion with a low moduli close to that of natural bone. PEEK has been recently widely used for dental application as prosthodontics fixed restorations and removable restorations. The crystalline PEEK polymer shows a harder hardness compared to other polymers (18). PEEK is available containing fibres or powder in differing combinations, e.g., carbon or glass fibres in short or endless chain structures reducing wear and improving flexural strength or hardness (17).

The absorbance water exerts a significant effect on the mechanical and dimensional properties of polymer (5). The incorporation of recycled polymethylmethacrylate to heat acrylic resin with different percentages decrease the water sorption and solubility (6). Water sorption and solubility have been measured by means of mass change in the materials after water saturation and dehydration (7).

The purpose of adding PEEK particles to increase mechanical and physical properties of PMMA.

MATERIALS AND METHODS

Fourty specimens of PMMA (vergeril, Spain) and classified into four groups 10 for control group without addition, 10 for 1% PEEK addition to PMMA, 10 for 2% PEEK addition to PMMA, 10 for 3% PEEK addition to PMMA. The PEEK material was added to PMMA powder in to 1%, 2% and 3% by weight respectively. Fourier transform infrared spectroscopy used for an organic and inorganic chemist, used to detect any chemical modification occurs.

To achieve an even distribution of PEEK particles within the PMMA powder, each prepared quantity was dispensed using a dispenser unit at 40 rpm/min for 1/2 h (12000 rpm). (7)

Gypsum moulds for the wax patterns were prepared for water sorption and solubility with dimensions of 12x10x4mm (length, width and thickness) respectively, figure (1) (8).

PMMA and PMMA/PEEK composites were cured according the conventional compression method using water-bath curing system. The PMMA powder/liquid mixing ratio was according to the manufacturer’s instructions of (3/1) by volume. Short-cycle heat polymerization method was timed for (1.5
hr. at 74°C then 1/2 hr. at 100°C), according to the manufacture instructions. After the completion of curing, flasks were allowed to bench cool at room temperature and deflasked and the specimens’ flashes were removed, the specimens were cleaned from the gypsum product using the ultrasonic unit for 15 minutes and finished.

The specimens were dried by silica gel in desiccators place in an incubator at 37±1 °C, Figure (2) they were daily weighing by using electronic balance until their masses became constant (M1).

**Figure (2)** Acrylic specimens were dried by silica gel

The volumes of the specimens were measured. The specimens were immersed in distilled water and maintained in an incubator at 37±1 °C for a week. After that the samples were removed, blotted to remove surface water, dried in air for 15 seconds, and start to weight. The results were recorded as (m2). The specimens were placed in the desiccators and dried in an incubator at 37±1 °C, daily weighed until a final constant mass was obtained (m3). To calculate the water sorption and solubility the following equations were used: (8)

\[
\text{Water sorption} = \frac{m_2 - m_3}{v}
\]

\[
\text{Water solubility} = \frac{m_1 - m_3}{v} \text{ g/mm}^3
\]

**Fourier Transform Infrared Spectroscopy Test (FTIR):**

To detect any chemical modification materials were tested by (FTIR) carried by CL Alpha-P FTIR spectrophotometer. FTIR was carried out for 5 materials: [1st control PMMA, the 2nd PMMA mixed with 1% PEEK, the 3rd PMMA mixed with 2% PEEK, the 4th PMMA mixed with 3% PEEK and the 5th pure].

**RESULTS**

**Water Sorption Test:**

A descriptive statistical analysis of Water sorption for all tested groups was presented in Table (1).
Table (1): Descriptive analysis of Water sorption (mg/mm$^3$) of all tested groups.

<table>
<thead>
<tr>
<th>Water sorption</th>
<th>N</th>
<th>mean</th>
<th>Std.deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control HCAR</td>
<td>10</td>
<td>.000016</td>
<td>.000006541118</td>
</tr>
<tr>
<td>1% PEEK</td>
<td>10</td>
<td>.000012</td>
<td>.000006848714</td>
</tr>
<tr>
<td>2% PEEK</td>
<td>10</td>
<td>.000008</td>
<td>.000004551141</td>
</tr>
<tr>
<td>3% PEEK</td>
<td>10</td>
<td>.000006</td>
<td>.000002936665</td>
</tr>
</tbody>
</table>

One-way ANOVA of Water sorption showed significant differences between the tested groups at P< 0.05 as shown in Table (2).

Table (2): One-way ANOVA of Water Sorption (mg/mm$^3$) of all tested groups

<table>
<thead>
<tr>
<th>Water sorption</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between Groups</td>
<td>.000</td>
<td>3</td>
<td>.000</td>
<td>6.267</td>
<td>.002</td>
</tr>
<tr>
<td>Within Groups</td>
<td>.000</td>
<td>36</td>
<td>.000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>.000</td>
<td>39</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Duncan's multiple range test of water sorption for the tested groups were shown there were significant differences at P< 0.05 in water sorption for group (1% PEEK) from other tested groups in Table (3), Figure (3) demonstrated that Table (3): Duncan's multiple range of Water sorption for the tested groups.

<table>
<thead>
<tr>
<th>Water sorption</th>
<th>N</th>
<th>mean</th>
<th>DMRT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control HCAR</td>
<td>10</td>
<td>.000016</td>
<td>D</td>
</tr>
<tr>
<td>1% PEEK</td>
<td>10</td>
<td>.000012</td>
<td>CD</td>
</tr>
<tr>
<td>2% PEEK</td>
<td>10</td>
<td>.000008</td>
<td>AB</td>
</tr>
<tr>
<td>3% PEEK</td>
<td>10</td>
<td>.000006</td>
<td>A</td>
</tr>
</tbody>
</table>

Figure (3) Means values for Water sorption test for tested groups
Water Solubility Test: Descriptive statistical analysis of Water Solubility for the tested collections were presented in Table (4).

Table (4): Descriptive analysis of Water Solubility (mg/mm$^3$) of all tested groups.

<table>
<thead>
<tr>
<th>Water Solubility</th>
<th>N</th>
<th>mean</th>
<th>Std deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control HCAR</td>
<td>10</td>
<td>0.000012</td>
<td>0.000006410894</td>
</tr>
<tr>
<td>1% PEEK</td>
<td>10</td>
<td>0.000009</td>
<td>0.000004312050</td>
</tr>
<tr>
<td>2% PEEK</td>
<td>10</td>
<td>0.000008</td>
<td>0.000004762399</td>
</tr>
<tr>
<td>3% PEEK</td>
<td>10</td>
<td>0.000008</td>
<td>0.000003861333</td>
</tr>
</tbody>
</table>

One-way ANOVA of Water Solubility showed significant differences between the tested groups at $P \leq 0.05$ as shown in (Table 2).

Table (5): One-way ANOVA of Water Solubility (mg/mm$^3$) of all tested groups.

<table>
<thead>
<tr>
<th>Water Solubility</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between Groups</td>
<td>.000</td>
<td>3</td>
<td>.000</td>
<td>1.623</td>
<td>.201</td>
</tr>
<tr>
<td>Within Groups</td>
<td>.000</td>
<td>36</td>
<td>.000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>.000</td>
<td>39</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Duncan's multiple range for Water Solubility of all tested groups demonstrated that there is no significant difference at $P \leq 0.05$ in water solubility between all groups.

Figure (4) Mean values of Water Solubility test for tested groups
**FTIR Test:**

In the IR charts, important absorbance bands appeared (the absorbance of the C=O band from the polymethacrylate group at 1140 cm\(^{-1}\) and the absorbance peak of the C=O from the ester group at 1719 cm\(^{-1}\)). Important absorbance bands appeared at CH\(_3\) at 2992, absorbance bands O-H appeared at 1432, absorbance bands C=O appeared at 1237, absorbance bands O-H appeared at 1383, absorbance bands C=C appeared at 963 as show in Figure (5). Control HCAR, Absorbance bands of PMMA. In the IR charts for polyetheretherketone, the absorbance of the C=C band (1506-1593) for benzene ring, the absorbance band C=C at 1657, the absorbance band O-H at 1417. As show in Figure (9) Control PEEK Absorbance bands of PEEK. The compound is identical for IR. Another FTIR for polyetheretherketone at percentage (1%, 2%, 3%) mixed with PMMA absorbance bands is similar to absorbance bands control PMMA and control peek without mixed as show in Figure (6). Peek 99% +1%peek, Figure (7) PMMA 98% +2% peek, Figure (8) PMMA 97% +3% peek.

![Figure (5) FTIR for Control PMMA](image1)

![Figure (6) FTIR for PMMA 99% +1%PEEK](image2)

![Figure (7) FTIR for PMMA 97% Peek](image3)

![Figure (8) FTIR for PMMA 98%+2% +3%Peek](image4)
Figure (9): Pure peek
DISCUSSION

PMMA will remain the favourite material selected for the construction of dentures. Try to improve the strength features of the material leads in lengthening the durability of acrylic dentures. Reinforcement of acrylic resin with any type of fillers this is not an absolute have revealed a significant improvement in mechanical properties.\(^{(10,11)}\)

Our result shows that water (sorption, solubility) decrease greatly as compared to control group, incorporated material with1% PEEK material have lowest mean value for water (sorption, solubility, this is due to proper impregnation of PEEK particles prevents polymerization shrinkage of PMMA. this lead to decrease water that destroyed polymer matrix by water molecules and lead to plasticization which cause interaction between the PEEK particles and PMMA resin \(^{(12)}\)

(Al-Nori et al., 2007) demonstrated that (PMMA) absorbs water little by little over a period of time when located inside an aqueous environment. That led to mechanical characteristics of the resin become lower.\(^{(5)}\)

Abdul karim et al (2018) who concluded that the Water sorption was significantly reduced with organic nanofibers due to produce a strong crosslinked network organic molecules with decreased interstitial spaces between PMMA polymer chains that consequently might decrease water sorption and water solubility of nanofibers reinforced PMMA resin.\(^{(16)}\)

The procedure employed for the incorporation of a higher percentage of filler to PMMA resin affected the water sorption and water solubility, with the increase ratio of incorporated material, water sorption and water solubility decrease, this is in line with the current study.\(^{(12)}\)

(Ozlem et al., 2006) studied the reinforcement of denture PMMA with powdered glass fibres, which leads to reduce water sorption and generally unaltered solubility.\(^{(13)}\)

The addition of silver fillers and glass fibres cause decreasing in of water sorption therefore alteration of heat cured acrylic resin with adequate amounts of silver particles and glass fibres can be useful into avoiding an unwanted physical change of dentures resultant from oral fluids.\(^{(14,15)}\)

CONCLUSIONS

The incorporation of PEEK particles to heat acrylic resin with 3% significantly decrease the water sorption and solubility.

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