



## Evaluating the Effect of Polyetheretherketone Particles Adding on Some Mechanical Properties of Polymethacrylate Denture Base Material.

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### Abstract

**Aims:** The aim of the present study was to assess some mechanical properties 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. The PEEK of 1%, 2% and 3% wt. respectively was added to the PMMA resin base to achieve a PMMA/PEEK composite of three different particles percentage to compare with the PMMA with no additives (control). The conventional heat-curing method was applied using a water bath to polymerize the specimens for both Vickers hardness, tensile and transvers strength tests. Study data were analysed via One-way ANOVA (post-hoc/Tukey test) performed at significant P-value of ( $p \leq 0.05$ ) and confidence. **Results:** After comparing the results, a significant alteration in the hardness, tensile and transvers strength of PMMA/2%PEEK composites was noticed comparing to other tested groups ( $p \leq 0.05$ ). **Conclusions:** the polyetheretherketone particles used as dental filler at 1%, 2% and 3% wt. added to PMMA reduced the tensile strength and enhanced the hardness and transvers strength of PMMA denture base material.

### الخلاصة

**الأهداف:** الهدف من هذه الدراسة هو تقييم بعض الخواص الميكانيكية لـ بولي ميثال ميثاكريلك بعد دمجها مع مادة بولي إيثر إيثر كيتون بنسب مختلفة. **المواد وطرائق العمل:** حجم جسيم بولي إيثر إيثر كيتون 130 ميكرومتر. تمت إضافات نسبه 1% و 2% و 3% من بولي إيثر كيتون إلى قاعدة راتنج بولي ميثال ميثاكريلك لتحقيق مركب بولي ميثال ميثاكريلك / بولي إيثر إيثر كيتون من ثلاثة نسب مئوية مختلفة من الجزينات للمقارنة مع بولي ميثال ميثاكريلك بدون إضافات. تم تطبيق طريقة المعالجة الحرارية التقليدية باستخدام حمام مائي للبرمة عينات لكل من اختبارات الصلابة والشد والمستعرضات. تم تحليل بيانات الدراسة عبر ANOVA أحادي الاتجاه (اختبار ما بعد المخصص / Tukey) الذي تم إجراؤه بقيمة P كبيرة ( $p > 0.05$ ). **النتائج:** بعد مقارنة النتائج، لوحظ وجود فرق كبير في الصلابة والشد والمستعرضات لمركب بولي ميثال ميثاكريلك / بولي إيثر إيثر كيتون بنسبه 2% مقارنة بالمجموعات المختبرة الأخرى ( $p \leq 0.05$ ). **الاستنتاجات:** تستخدم حبيبات بولي إيثر إيثر كيتون كمادة مألنة عند دمجها مع بولي ميثال ميثاكريلك بنسبه 1% و 2% و 3%. مما يؤدي إلى تقليل قوة الشد ويعزز الصلابة، وقوة الضغط لمادة بولي ميثال ميثاكريلك الأساسية لأطقم الأسنان.

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## INTRODUCTION

The acceptance of PPMA resin was belonged to easy processing, low cost, perfect aesthetic properties hardness, lightweight, low solubility and water sorption, and ability to easy repair. <sup>(1, 2)</sup>

Numerous studies showed with goal of improving the properties of PMMA by adding fillers inside the composition. Adding fillers and particles to PMMA is used to develop enhancement in physical and mechanical properties. <sup>(2, 3, 4)</sup>

The addition of inorganic Filler in acrylic resin alter properties of end product depend on the kind, sizes, shape, concentration and interaction among polymer matrix itself. <sup>(3)</sup>

Khalaf, (2013), said that the mechanical properties of denture base resin PMMA (tensile strength and transvers strength) is reinforcement by addition of Siwak particle with size (75 $\mu$ m) in different concentration of (3%, 5% and 7%) by weight, the results indicated that the addition of Siwak particles in low concentration (3% and 5%) to heat polymerized denture base acrylic resin did not disturb greatly the mechanical properties, while the addition of (7 %) Siwak powder showed a significant decline in tensile strength in contrast to the control group. <sup>(5)</sup>

## MATERIALS AND METHODS:

A sample of 120 specimens was prepared for this study, and divided into three main groups. The subdivided group

tested for surface hardness, tensile and transvers mechanical tests, (n=40).

The proposed PMMA/PEEK study composite material was prepared according to the following measurements (the PEEK of 1% wt. was added to the PMMA resin base of 99% wt., PEEK of 2% wt. was added to the PMMA resin base of 98% wt. and PEEK of 3% wt. was added to the PMMA resin base of 97% wt. to achieve a PMMA/PEEK composite using sensitive balance. To achieve an even PEEK distribution within the PMMA powder, each prepared quantity was dispensed using a dispenser unit at 40 rpm/min for 1/2 h (12000 rpm). <sup>(7)</sup>

Silicon mould for the wax disc specimens was prepared for the hardness, tensile and transvers mechanical tests. PMMA and PMMA/PEEK composites cured followed the conventional compression method using water-bath curing system. The polymethylmethacrylate powder/liquid mixing relation was according to the manufacturer's instructions of (3/1) by volume (Veracril, Spain). Short-cycle heat polymerization processing method was timed for (1.5 hr. at 74°C then 1/2 hr. at 100°C), according to the manufacturers instruction. After the completion of curing, flasks were permitted to bench cool for 30minuts at room temperature, deflasked, specimens' flashes removed, cleaned from the gypsum product using the ultrasonic unit for 15 minutes and finishing the specimens. <sup>(8)</sup>

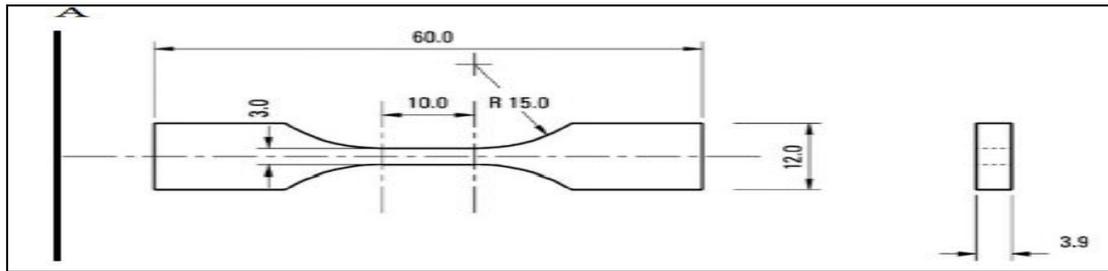
**Testing Procedure:**

**Hardness test (Vickers hardness):**

Hardness tested (Micro hardness tester- Shimadzu, Japan), to determine Vickers value, the dimensions were of 12(±0.1) mm in diameter and 2(±0.1) mm in thickness a specimen was measured by applying a load under 25g load at 30s

penetration. Three readings were recorded for each specimen (one on the middle and two on the boundary) the mean value was calculated for each specimen. <sup>(7)</sup>

**Tensile Strength Test:** For the measurement of tensile strength, forty dumbbell-shaped samples were prepared according to ISO 5271. <sup>(9)</sup> Figure (1)



**Figure 1:** Sample for measurement of tensile strength

The testers were clasped by two arm of the machine and the quantity of force applied was 0.1 Kilo Newton per second continuous increase tension force until fracture of sample occurred in universal testing machine. According to the ASTM specification. <sup>(10)</sup>

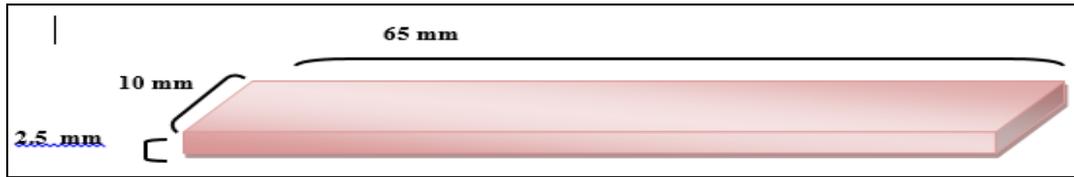
The results were recorded from a program on a CPU of tensile machine for each sample. The force at failure was recorded in Newton (N) and the true tensile value was calculated by the following formula:

Tensile strength =  $F (N)/A (mm^2)$ , F= Tension force at Failure (N), A= Cross Section of specimens. <sup>(12)</sup>

**Transverse Strength Test:**

fluxual strength test samples (40 samples) were organized with sizes of 65x10x2.5±0.03mm (length, width and thickness) separately were prepared, according to ADA specification no.12, Figure (2). The samples stored in a distilled water at 37°C for 48 hours. <sup>(11)</sup>

The test was applied by using Digital Electronic Force Gauge at three points bending on universal testing machine. The device was provided with a central loading plunger and two supports, with polished cylindrical surfaces of 3.2 mm in diameter and 50 mm between supports. The supports should be parallel to each other and perpendicular to the central line.



**Figure 2:** Transverse Strength testing specimen dimensions.

The tests were approved with cross head speed of 5mm/min. The test samples held at each end of the two supports, and the loading plunger placed mid-way between

the supports, the samples were deflected until fracture occurred. The transverse strength was calculated by using this equation: <sup>(13)</sup>

$$S = 3PL / 2bd^2 \quad \text{N/mm}^2$$

S = transverse strength, P = maximum force exerted on specimen (N),

L = distance between supports (mm),

b = width of specimen (mm)

d = depth of specimen (mm).

## RESULTS

### Tensile test:

Descriptive statistical analysis of tensile strength for all tested groups were presented in Table (1).

**Table (1):** Descriptive analysis of Tensile Strength (n/mm<sup>3</sup>) of all tested group

Tensile strength	N	mean	Std.deviation
Control HCAR	10	69.2	6.61785
1% PEEK	10	56	5.39547
2% PEEK	10	51	4.59468
3% PEEK	10	40.2	3.83342

One way ANOVA of tensile strength of control group (HCAR) (0%), groups (1% PEEK mixed with 99% HCAR, 2% PEEK mixed with 98% HCAR

and 3% PEEK mixed with 97% HCAR) showed significant differences at  $P \leq 0.05$  between tested groups as show in Table (2).

**Table (2):** One-way ANOVA of Tensile Strength (n/mm<sup>3</sup>) of all tested groups

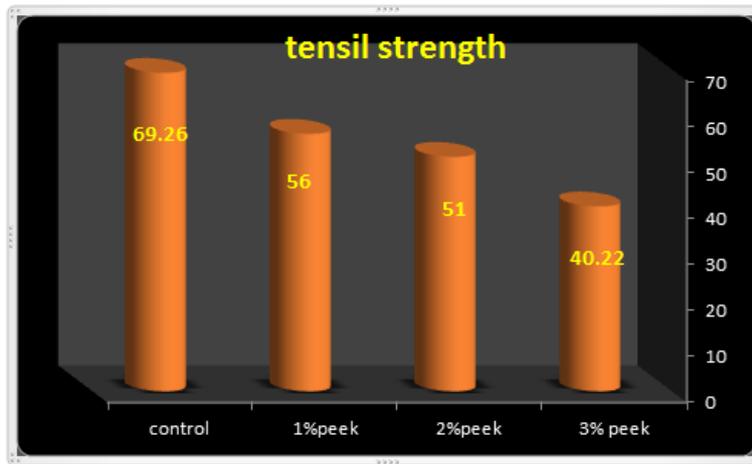
Tensile Strength	Sum of Squares	Df	Mean Square	F	p-value
Between Groups	4356.984	3	1452.328	53.437	000
Within Groups	978.420	36	257.892		
Total	5335.404	39			

Duncan's multiple range of the Tensile strength (N/ mm<sup>2</sup>) for the tested groups (PMMA resin mixed with PEEK at concentration (1 %, 2% and 3%)) was

significantly decreased tensile strength at P≤0.05 in comparison with the control group PMMA as shown in Table (3), Figure (1).

**Table (3):** Duncan's multiple range of the Tensile strength for the tested groups.

<b>Tensile strength</b>	<b>N</b>	<b>mean</b>	<b>DMRT</b>
Control HCAR	10	69.2	A
1% PEEK	10	56	B
2% PEEK	10	51	C
3% PEEK	10	40.2	D



**Figure 3:** Means of Tensile strength for the tested groups

**Transverse strength:** Descriptive statistical analysis of Transverse strength

for all tested groups were presented as show in Table (4).

**Table (4):** Descriptive analysis of Transverse strength (N/mm<sup>2</sup>) of all tested groups.

<b>Transverse strength</b>	<b>N</b>	<b>mean</b>	<b>Std.deviation</b>
Control HCAR	10	74.3	4.69993
1% PEEK	10	78.9	12.70449
2% PEEK	10	85.1	4.43241
3% PEEK	10	99.5	11.39700

One way Analysis of variance ANOVA of Transverse strength presented

significant differences between the tested groups at P< 0.05 as show in Table (5)

**Table (5):** One way ANOVA of Transverse strength (mg/cm<sup>3</sup>) of all groups.

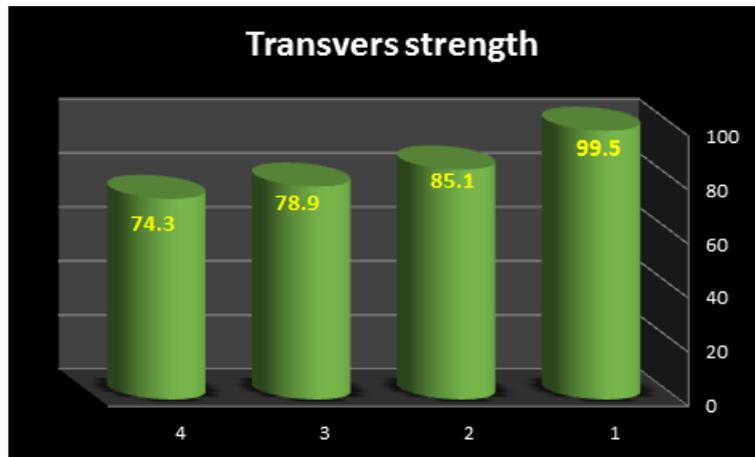
Transverse Strength	Sum of Squares	df	Mean Square	F	p-value
Between Groups	8212.900	3	2737.633	7.129	.001
Within Groups	13823.980	36	383.999		
Total	22036.880	39			

Transverse strength mean and SD and Duncan's multiple range for the groups are presented in table (6), Figure (2) confirmed that there were significant

differences at P< 0.05 in measurement of transverse strength of PMMA for all tested groups among different concentrations of PEEK additions.

**Table (6):** Mean and Duncan's multiple range for the group of The transverse strength.

Transversers test	N	mean	DMRT
Control HCAR	10	74.3	D
1% PEEK	10	78.9	C
2% PEEK	10	85.1	B
3% PEEK	10	99.5	A



**Figure (4):** Mean of Transverse Strength test of all tested groups

**Hardness test:** Descriptive statistical analysis of Hardness test for all tested groups were presented in Table (7).

One way ANOVA of Hardness test presented significant differences among the groups at P< 0.05as in Table (8)

**Table (7):** Descriptive analysis of Hardness test of all tested groups.

Hardness Test	N	mean	Std.deviation
Control HCAR	10	85.2000	3.68239
1% PEEK	10	93.0182	1.51909
2% PEEK	10	93.6909	2.48856
3% PEEK	10	98.9545	1.97452

**Table (8):** One way ANOVA of Hardness of all tested groups.

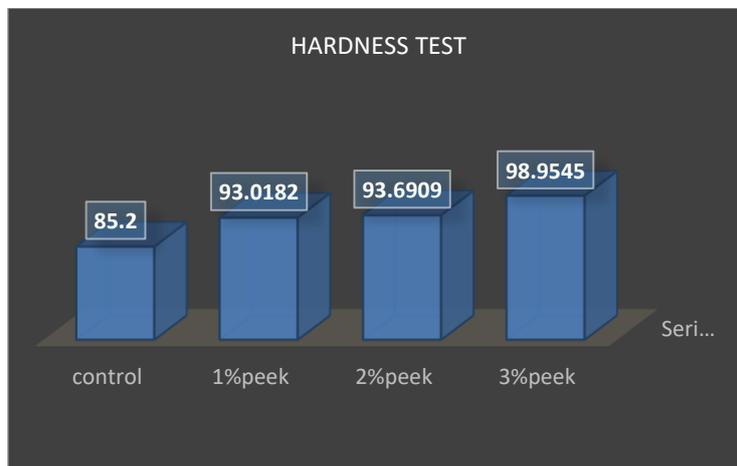
Hardness test	Sum of Squares	df	Mean Square	F	p-value
Among Groups	1106.027	3	368.676	28.946	.000
Within Groups	458.524	36	12.737		
Total	1564.551	39			

Means and Duncan's multiple rang test for Hardness of all tested groups proven that there were significant differences at P< 0.05 in measurement of hardness for all

tested groups among different concentrations of PEEK (1 %,2% and 3%) in comparison with the control groups as shown in Figure (3), Table (9).

**Table (9):** Duncan's multiple test for Hardness for all tested groups

Hardness test	N	Mean	DMRT
Control HCAR	10	85.20000	D
1% PEEK	10	93.0182	C
2% PEEK	10	93.6909	B
3% PEEK	10	98.9545	A



**Figure (5):** Mean of the Hardness test of all tested groups

## DISCUSSION

### Tensile Strength Test:

Tensile strength is the resistance of the material to a tensile or stretching force. The tensile strength results and Duncan's multiple range tests are presented in tables (1, 2 and 3), figure (3) presented that addition of PEEK (1%, 2% and 3%) to PMMA significantly decreased tensile strength in comparison with the control PMMA due to the entering of the particles between the resin chains, which is playing the role of restricting the chains motion so, the slipping of these chains will decrease and leading to decreasing tensile strength. <sup>(15)</sup>

Nada et al (2020) who concluded that the tensile strength was decreased with increasing at different average particle size of (53 $\mu$ m, 106 $\mu$ m, 150 $\mu$ m and 212 $\mu$ m) and weight percentage of (12%wt.) of Pistachio Shell powder in PMMA resin. The lowest values of them were obtained at (12wt. %) and at average particle size of (212 $\mu$ m). <sup>(18)</sup>

Mohammed et al (2020) concluded that incorporated the eggshell particle (before burning 15.  $\mu$ m) and after burning 10.  $\mu$ m) and (1%,3%,5%,7%) into the acrylic resin developed the tensile strength, that the matrix resin and filler to the filler PMMA. This is lead to making interfaces among the matrix resin and filler and increase in the capacity of stress passage from the resin polymethyl methacrylate to the filler composite. The specimen pure polymethylmethacrylate is weaker than other specimens mixed with eggshell fillers because the matrix resin alone is unable to resist the tensile force applied to it <sup>(19)</sup>

### Transverse test:

The transverse of material is a degree of stiffness and resistance to fracture. Transverse strength tests were under taken as these were considered relevant to the loading characteristic of a denture base. <sup>(20)</sup>

The transverse strength results and Duncan's multiple range tests presented in tables (4, 5 and 6), Figure (4) showed that adding PEEK (1%, 2% and 3%) to PMMA was significantly increased in comparison with the control PMMA. This is lead to the adhesion between PEEK particles and acrylic resin polymer matrix so that the load can be distributed equally. <sup>(5)</sup>

Gokul et al., (2018) and Sri (2019) concluded that an improved in transverse strength in acrylic resin group additional with 1% and 1.5% E-glass fiber lead to the main composition of glass fiber involves of silica dioxide (SiO<sub>2</sub>) that was stiff and high rate of strength, so that the glass fiber structure becomes denser and strong and able to absorbs all loads received by heat-polymerized acrylic resin. When the load is transferred from the heat-polymerized acrylic resin to the glass fiber, it will increase the transverse strength of the heat-polymerized acrylic resin denture base <sup>(21)</sup>

Khalaf (2013) Concluded that the adding of low concentrations (3%, 5%) Siwak to the heat PMMA acrylic resin did not affect significantly the tested mechanical properties. this may be explained that some kind of physical adhesion result from downgrading of the energy between two surfaces (Siwak additive and PMMA acrylic resin) increased the adding of additives greater than 5% by

weight the increase in transverse strength mean values for studied groups this could be attributed to good distribution of the Siwak powder. The Siwak particles enter between the chains of the polymer, motions of these chains are restricted result in improvement of transverse strength. <sup>(5)</sup>

Jaikumar et al (2015) who concluded that (PMMA) reinforced with glass fibers showed the highest transvers strength values this was followed by PMMA reinforced with butadiene styrene, and the least strength was detected in the conventional denture base resins. The reason behind this would be the presence of glass fibers, which may prevent the propagation of a crack when the stresses are applied, glass fibers are strongly effect in patients with dense occlusal load or when fracture of denture base resin is occur. <sup>(22)</sup>

### **Hardness:**

Vickers micro hardness test is very effective method to evaluate the stiff polymers and capability of material that resist the diffusion of load. <sup>(14)</sup>

According to the result in the current study Table (7, 8 and 9), Figure (5) the hardness value was increased with increase PEEK particles incorporated material. This due the insertion and linkage of particles to heat cure resin, create polymerization of acrylic resin and appeared more stiff and lower deformation. Increase of hardness is a result of cross-linking agent occurred to neutralize by result of residual monomer substance. <sup>(23)</sup>

Amrah et al (2020) The incorporation of recycled polymethylmethacrylate to heat

acrylic resin with different percentages increase hardness means value, this leads to improving some properties of heat acrylic resins. <sup>(15)</sup>

Ana et al., 2012 showed that the acrylic resin displayed higher hardness values with glass fiber reinforcement <sup>(16)</sup>.

Asopa et al., 2015 said that a greater filler content reducing the strength and the resin cannot adding more filler particles. This leads to a disturbance in the resin matrix that leads to reduce the strength of the reinforced specimens. Addition fillers with different percentages may decrease the hardness value compared to the control group; a decrease of hardness was found when the filler added more than eleven per cent of filler. <sup>(17)</sup>

## **CONCLUSIONS**

The PEEK particles use it as dental filler at 1%, 2% and 3% wt. incorporating PMMA reduced the tensile strength but enhanced the hardness and transvers strength of PMMA denture base material.

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