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# Effects of Recycling-PEEK Waste from CAD-CAM on Surface Hardness and Roughness of PMMA

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**Abstract**
**Background** Being health care professionals, technicians should be aware of regarding safe disposal of biomedical waste and the recycling of dental materials to minimize biohazards to the environment. **Objectives** The aim of the present study was to assess the PMMA mechanical properties after recycling and reuse of waste Polyetheretherketone (PEEK) material from the CAD-CAM production method as effective filler.
**Materials and Methods** The PEEK fiber was collected from the CAM machine after the milling procedure. Then, standard sieves of ISO standardization no. 40, 60 and 100 were used respectively to achieve a fine particle grit size of 150µm. The PEEK of 1% wt. was added to the PMMA resin base of 99% wt., and PEEK of 2% wt. was added to the PMMA resin base of 98% wt. to achieve a PMMA/PEEK composite of two different filler percentage to compare with the PMMA with no additives. The conventional heat-curing method was applied using a water bath to polymerize the disc specimens for both surface roughness and hardness tests. Study data were analyzed via One-way ANOVA (post-hoc/Tukey test) performed at a significant P-value of ($p \leq 0.05$) and confidence level of 95%. **Results** After comparing the results, a significant difference in the surface hardness and roughness of PMMA/2%PEEK composites was noticed comparing to other tested groups ($p \leq 0.05$). **Conclusion** The recycling of PEEK waste from CAD-CAM production method and reuse it as dental filler at 1% and 2% wt. incorporating PMMA reduced the surface roughness and enhanced the surface hardness of PMMA denture base material.

**Keywords: CAD-CAM; recycle; surface hardness; surface Roughness; PEEK.**

## Introduction

Biomedical waste management has become a concern in the dental world as different materials start to increase in use. In dentistry, few studies have suggested to recycle and reuse some of the dental materials (Bhat, 1983; Baghele et al, 2013; Ranjan et al, 2016). The majority of such study population was completely clueless regarding knowledge pertaining to the recycling process. They were unaware of the proper disposal of some non-degradable dental materials (Elgayar and Aboushelib, 2014; Singh et al, 2014; Mattoo et al, 2014; Tippat and Pachkhade, 2015). Polyetheretherketone (PEEK) is a newly inova-

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tive 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 (Chen et al, 2019). The crystalline PEEK polymer shows a harder hardness compared to other polymers (Stuart and Briscoe, 1996). PEEK has been recently widely used for dental application as prosthodontics fixed restorations (Zoidis and Papathanasiou, 2016; Najeeb et al, 2016) and removable restorations (Muhsin et al, 2019; Peng et al, 2019). Computer-aided design and manufacturing technology (CAD-CAM) had become widely applicable in dental office and laboratory. It is possible to produce partial or complete restorations using PEEK material through use of CAD-CAM (Schwitalla et al, 2015; Chen et al, 2018; Harb et al, 2019). One of the most important challenges in using PEEK in the clinic is high cost due to the large unused volume of PEEK block after milling by CAD-CAM. Although newer devices have higher accuracy in cutting, but in the process of cutting the block, there are still large parts in the form of fibers and unused patches that have imposed a high cost on the patient. It also has problems in its decomposition and recycling in the environment, and there are no specific guidelines in some countries on recycling highly crystalline PEEK remnants. Dental appliances may be constructed using PMMA resins and subsequently be repaired or relined. Although Polymethlemethacrylate (PMMA) remains the most popular choice for polymeric prosthodontics restorations as it may relatively easy to be used in both clinical and laboratory fabrication process alongside its approperate cost (Alla et al, 2013; Alla et al, 2015), several studies reported different techniques for reinforcing PMMA through inclusion of other materials (John et al, 2001; Narva et al, 2005; Akinci et al, 2014; Gad et al, 2017). Also, many studies have shown that some modification in the inorganic fillers could remarkably improve the PMMA mechanical properties such as surface hardness (Vuorinen et al, 2008; Syngouna and Chrysikopoulos, 2011; Vojdani et al, 2012; Ahmed and Ebrahim, 2014). On the other hand, additional studies were examined the surface roughness of the non-polished PMMA denture base (Zissis et al, 2000, Berger et al, 2006, Abuzar et al, 2010, Mekkawy et al, 2015). However, few studies reported the effect of surface roughness on bacterial accumulation and plaque formation (Quirynen et al, 1990; Bollenl et al, 1997; Radford et al, 1999). They state that the bacterial colonization may occur with a higher incidence when the surface roughness value in some denture base materials was greater than 2.0μm. Since "composite" refers to reinforcing constituent either of long or continuous fibers (Kurtz, 2011). Hence, this study has been aimed to find out a simple scientific technique to recycle PEEK waste fibers from CAD-CAM and re-use it to assess its effectiveness as a filler agent on some PMMA mechanical properties.

## Materials and Methods

### 1. PEEK-Waste Fiber Collection

In the present study, a decomposed PEEK blank was used. The PEEK fiber was collected after using the CAD-CAM production method (PEEK-JUVORA™, UK). The result fiber was brushed-off from the CAM machine after dry milling procedure (Roland-DWX-50, USA). Then a magnet is used during vibrating sieving procedure to remove any trace of metal particles may incorporated from the cutting bur. The standard stainless steel sieves of ISO standardization (Italy) no. 40 (420μm), 60 (250μm), and 100 (150μm) respectively was used to achieve fine particle grit sizes (Shin and Hryciw, 2004; Syngouna and Chrysikopoulos, 2011).

JODR
VOLUME 7, Issue 1, 2020



**2. Preparing the PMMA/PEEK Composite**

The proposed PMMA/PEEK study composite material was prepared according to the following measurements using sensitive balance as shown in table (1). To achieve an even PEEK fiber distribution within the PMMA powder, each prepared quantity was dispensed using a dispenser unit at 40 rpm/min for 5 h (12000 rpm).

**3. Study Sample Grouping**

A sample of 60 disc specimens was prepared for this study, and divided into two main groups. The subdivided group tested for surface hardness and roughness mechanical tests, (n=10). The X-ray diffraction (XRD) was used to analyses the effect of the PEEK filler on the PMMA crystallinity behavior. The XRD technique indicates the normal order of crystalline structure in the polymeric chains. XRD pattern was obtained in the 2θ range between 0 and 90 degree (LabX6000-Shimadzu, Japan).

**4. Sample Preparation**

Silicon mould for the wax disc specimens was prepared for the surface hardness and roughness tests. The dimensions were of 12(±0.1)mm in diameter and 2(±0.1) mm in thickness. PMMA and PMMA/PEEK composites cured followed the conventional compresion method using water-bath curing system. The PMMA powder/liquid mixing ratio was according to the manufacturer's instructions of (3/1) by volume (Veracril, Spain). Short-cycle heat polymerization processing method was timed for 3½ h. After curing, the flasks were kept on bench to cool overnight, deflasked, specimens' flashes removed, cleaned from the gypsum product using the ultrasonic unit for 15 minutes.

**5. Testing Procedure**

Figure (1) shows the surface hardness that measured using Shore D durometer hardness tester unit (China) and the surface roughness which measured using the profilometer surface roughness tester device (China).

**Statistical Methods**

The study data analyzed via One-way ANOVA (post-hoc, Tukey test) at a confidence level of 95% and a significant P-value of ($p \leq .05$).

**Results**

Table (2) and (3), Figure (2) and (3) show the result of surface hardness and roughness of PMMA/PEEK composites, while Figure (4) shows the XRD patterns of the experimental prepared composites. After comparing the results for surface hardness, a significant difference was noticed in the surface hardness between the PMMA as a control group and that of both the PMMA/1% PEEK and PMMA/2%PEEK composites ($p \leq 0.05$). For surface roughness, a statistically non-significant differences was reported in surface roughness between the PMMA and PMMA/1%PEEK composite, also between PMMA/1%PEEK and PMMA/2%PEEK composites ($p > 0.05$). However, there was a significant difference in the surface roughness between the PMMA and PMMA/2%PEEK composite ($p \leq 0.05$).

**Table (1): The composition of Polymethylmethacrylate/Polyetheretherketone (PMMA/PEEK) composite for the experimental group study.**

| Groups | PMMA Composite Resin | |
|---|---|---|
| | Resin Base (98–100 wt. %) | PEEK-Filler (0–2 wt. %) |
| | PMMA (g) | PEEK (g) |
| Control (I) | 100% | 0% |
| Group (II) | 99% | 1% |
| Group (III) | 98% | 2% |

**Table (2): ANOVA-test showing the surface hardness of the tested group specim.**

| Groups | | Mean Difference | p-value | Sig. |
|---|---|---|---|---|
| PMMA | PMMA + 1% PEEK | -5.3000* | .000 | S |
| | PMMA + 2% PEEK | -9.4000* | .000 | S |
| PMMA + 1% PEEK | PMMA + 2% PEEK | -4.1000* | .003 | S |

**Table (3): ANOVA-test showing the surface roughness of the tested group specimens.**

| Groups | | Mean Difference | p-value | Sig. |
|---|---|---|---|---|
| PMMA | PMMA + 1% PEEK | .4370 | .199 | NS |
| | PMMA + 2% PEEK | .8857* | .004 | S |
| PMMA + 1% PEEK | PMMA + 2% PEEK | .4487 | .183 | NS |

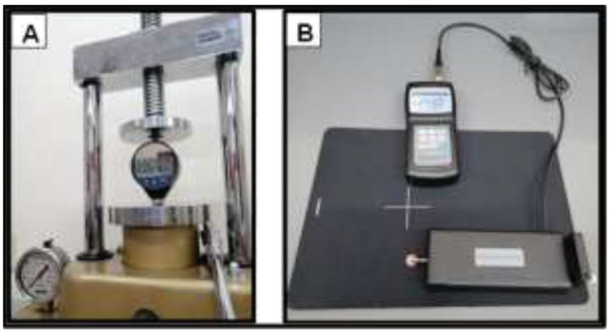


**Figure 1: A, Shore D durometer surface hardness unit; and B, Surface roughness profilometer tester device.**

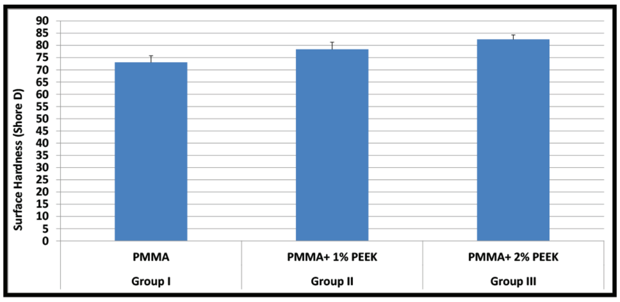


**Figure 2: Mean distribution of the surface hardness of the tested groups.**

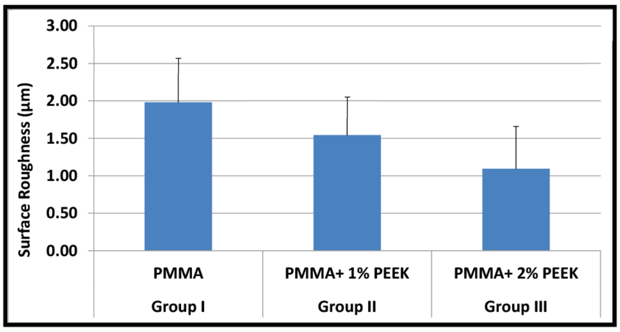


**Figure 3: Mean distribution of the surface roughness of the tested groups.**

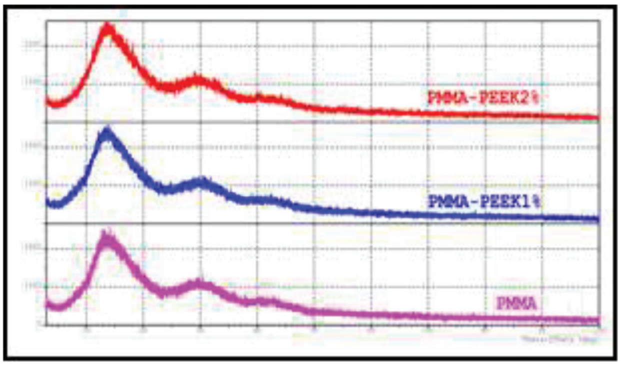


**Figure 4: Diagram shows the XRD pattern of PMMA base resin, PMMA/1%PEEK, and PMMA/2%PEEK prepared study material.**

## Discussion

The initial mechanical properties of any dental material may predict the primary mode of clinical failure and provide a determination for a specific application. Recently, the development of new materials for load-bearing areas were suggested to improve the mechanical properties. PMMA was one of the most commonly used denture bases with inferior mechanical properties. In the present study, the surface hardness and roughness of PMMA base resin are evaluated after the addition of 1% and 2% of PEEK fiber. The present data results showed that the value of surface harness may affect greatly by the measurements of PEEK fiber that added to the PMMA. The additions of 1% or 2% of PEEK fibers to the PMMA base resin have shown higher surface hardness than that of non-additives. According to (Kurtz, 2011), the annealing process for Juvora™ material provided a high crystallinity behaviour for CAM purposes. The increase in the PEEK fibers filler percentages could affect positively the surface hardness. This result may agree with (Stuart and Briscoe, 1996) who state that the material hardness may increase by the presence of polymer of high crystallinity polymer. On the other hand, the surface roughness of the non-polished PMMA/PEEK composite was evaluated as a mean to predict the colonization of diferent microorganisms. The additions of 1% of PEEK fibers to the PMMA base resin have shown statistically the same surface roughness of that of PMMA and that of 2% PEEK filler. The lowest mean value in the surface roughness was of 1.09(±0.6)μm, which was recorded within the PMMA/2%PEEK composite. The addition of 2% of PEEK fibers statistically reduced the surface roughness of the PMMA composite. Yet, the present results could agree with (Quirynen et al, 1990; Bollenl et al, 1997; Radford et al, 1999) as the surface roughness less than 2μm. Hence, PMMA/2%PEEK composite may offer promises as a new modified denture material with less opportunities for future microbial accumulation. XRD indicates similar non-sharp diffraction peaks of crystalline nature for all the tested composites. The behavior of the PMMA/PEEK composite shows no phase differences in the chemical composition due to the addition of the PEEK fibers.

**Conclusion**

PMMA-based composites prepared with 1% and 2% wt. of PEEK fibers were submitted to evaluate the surface hardness and roughness mechanical properties. These results indicated that the addition of PEEK filler to PMMA

**Conflict of interest**

We are the author's (Aseel Hayder Salim, and Assist. Prof. Dr. Saja Ali Muhsin) state that the submitted manuscript for this paper is original. It has not been published previously, and it's part of MSc. dissertation and is not under consideration for publication elsewhere, and that the final version has been seen and approved by all authors.

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