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Evaluation of microstructure and mechanical properties of PMMA Matrix composites reinforced with residual YSZ from CAD/CAM Milling process

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ABSTRACT

Background: Reinforcement of dental acrylics with fillers has yielded positive results. Different proportions of fillers have been added to strengthen the dental acrylics, but no consensus has been reached.
Aim: The purpose of this study was to investigate the effects of the addition of different concentrations of yttria-stabilized zirconium (YSZ) obtained from the residues generated from the CAD-CAM milling of YSZ on the microstructure and mechanical properties of poly-methyl-methacrylate (PMMA)-YSZ composites.
Materials and methods: Composite materials with different amounts (0.0 to 70.0% by weight) of recycled YSZ reinforced PMMA resin matrix were produced. Scanning electron microscope (SEM), energy dispersive electron spectrometer (EDS) and Fourier Transform Infrared Spectrometer (FTIR) were used for microstructural analysis. Among the mechanical properties, the Vickers micro-hardness test method for hardness, 2D profilometer for surface roughness and composite densities were evaluated by Archimedes method. Data were analyzed using a one-way analysis of variance (ANOVA) at a pre-set alpha of 0.05.
Results: Microhardness and density increased until 60% by weight YSZ addition, while surface roughness remained unchanged but increased after 60% by weight YSZ addition. The addition of more than 60% by weight of YSZ caused agglomeration in the microstructures. The mechanical properties of poly-methyl-methacrylate decreased with more than 60% YSZ by weight.
Conclusion: Reinforcement of PMMA with residue zirconia powder will increase the usage chance of residue YSZ powder and provide a safer use of PMMA.

1. Introduction

Polymethylmethacrylate (PMMA) is one of the most preferred prosthodontic material in dentistry owing to the reasons such as low-cost, simple application and easy polishing [1,2]. PMMA based provisional restoration is an important step that should not be neglected in the fixed prosthetic treatment process in order to protect the prepared tooth from heat and food to provide aesthetics, function, formation of the gum form and occlusion for a certain period [3,4].
Temporary restorations undergo repeated chewing forces and require specific mechanical properties to withstand long-span fixed prostheses, temporomandibular

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joint disorders, and parafunctional habits [5]. However, PMMA has insufficient mechanical strength and surface hardness [2], prolonged use can cause cracking or fracture of temporary restorations, especially in long-span fixed prostheses and areas with heavy occlusal load in the posterior region. When temporary restorations are broken, they can damage oral tissues and even be swallowed by the patient. The surface hardness of PMMA is low; hence the material is relatively softer and possibly become rough during the chewing process in the oral cavity. Consequent to this, the surface of PMMA are colonized easily by microorganisms [6-8].

In recent years, many different material additions such as metal wires [9], fibre [10-13], nanodiamond [14], metal oxide nanofillers such as silver [15,16], titanium [17-19], aluminum and zirconium oxide [20, 21] to resin matrix have been studied to improve the mechanical properties of PMMA. Some of these additions have the same disadvantages, such as low corrosion resistance [9], tissue irritation, etc. [7]. The hardness, density and roughness of the provisional restorative materials change according to the polymer and the reinforcement type [22]. Zirconia with superior biocompatibility, mechanical strength, high density, high surface hardness [23], good chemical resistance, good thermal stability [24] is a superior reinforcement material for PMMA.

Nowadays, the recycling and reuse of ceramic materials are even more important because of the environmental issue and the high cost of mining and transporting pure raw material [25]. The machining of crowns by CAD/CAM (Computer-assisted design/computer-assisted machining) can produce a waste of approximately 30% of the initial blank, generating a significant economic loss for the prosthetic laboratories. Zirconia residues have a high economic value and potential for recycling [26].

Therefore, this study aimed to evaluate the effect of incorporating residue YSZ on hardness, density, and roughness on PMMA. There are a few studies in the literature about YSZ reinforcement PMMA. However, they were mainly focused on low amount YSZ addition (10 wt%) to PMMA [27, 28]. However, there are no studies about the high percentage of YSZ reinforced to PMMA matrix in literature.

2. Materials and methods

In the present study, a commercially available cold cured provisional poly-methyl methacrylate (PMMA) powder and methyl methacrylate (MMA) (Integra, Ankara, Turkey) as base liquid were selected as matrix materials. Residue Yttria-stabilized zirconia-milled powder (Zirking, Huge Dental, China) were used as a reinforcing material to prepare the composite specimens.

2.1 Samples preparation

The residue yttria-stabilized ZrO2 powders and provisional PMMA powder were pre-weighted using an electronic balance (Radwag AS 60/220.R2 Dual Range Analytical Balance, Radwag USA L.L.C., FL, USA) to ensure 0.0 wt%, 12.5 wt%, 25 wt%, 30 wt%, 35 wt%, 40 wt%, 50 wt%, 60 wt%, 65 wt%, and 70 wt% concentrations (Table 1).

Zirconia blocks residue powders were added to the acrylic resin powder and mixed manually with a stainless-steel spatula until homogeneously dispersed. Subsequently, MMA liquid was added and thoroughly mixed. The dough was poured into a stainless steel mould of 10×2 mm cylindrical holes, and samples were allowed to cure in a pressure vessel (Vertex Poly Cure25, Vertex-Dental, Zeist, Netherlands) under 2.5 atmospheric pressure at 55ºC for 10 minutes. After polymerization, the specimens were wet-polished with 800-grit, 1000-grit and 1200-grit silicon carbide papers, respectively (n= 10).
The microstructure of the fractured surfaces of prepared samples was studied using a scanning electron microscope (SEM JSM-6060 LV, JEOL, Tokyo, Japan). Chemical composition was analyzed using an Electron Dispersive Spectrometer (EDS) (Hitachi SU 5000, Japan). Fourier transform infrared spectroscopy (version 660-IR FT-R Spectrometer, Agilent Technologies, Santa Clara, USA) was used to detect the functional groups. Vickers hardness of the samples was measured using a microhardness device (HMV, Shimadzu, Kyoto, Japan). Specimens of 10 mm diameter and 2 mm thickness were subjected to a load of 50 N for 10 seconds for hardness measurement. Densities of unreinforced PMMA and yttria-stabilized ZrO_2 reinforced PMMA matrix samples were measured using the Archimedes density measurement method. The surface roughness (Ra) test was performed with a profilometer (Surface Roughness Tester SJ-201, Mitutoyo, Kawasaki, Japan) to measure the arithmetic mean roughness of the surfaces. The cut off was set at 0.8 mm, and the total transverse length was 1.25 mm.

Table 1. Powder PMMA and liquid MMA weights with residue zirconia weight percentages of groups.

Groups	Zirconia (wt%)	Zirconia (g)	PMMA Powder (g)	Monomer (mL)
1	0.00	0.00	23.50	10.00
2	12.50	2.94	20.56	10.00
3	25.00	5.88	17.62	10.00
4	30.00	7.00	16.45	10.00
5	35.00	8.23	15.27	10.00
6	40.00	9.40	14.10	10.00
7	50.00	11.75	11.75	10.00
8	60.00	14.10	9.40	10.00
9	65.00	15.27	8.23	10.00
10	70.00	16.40	7.05	10.00

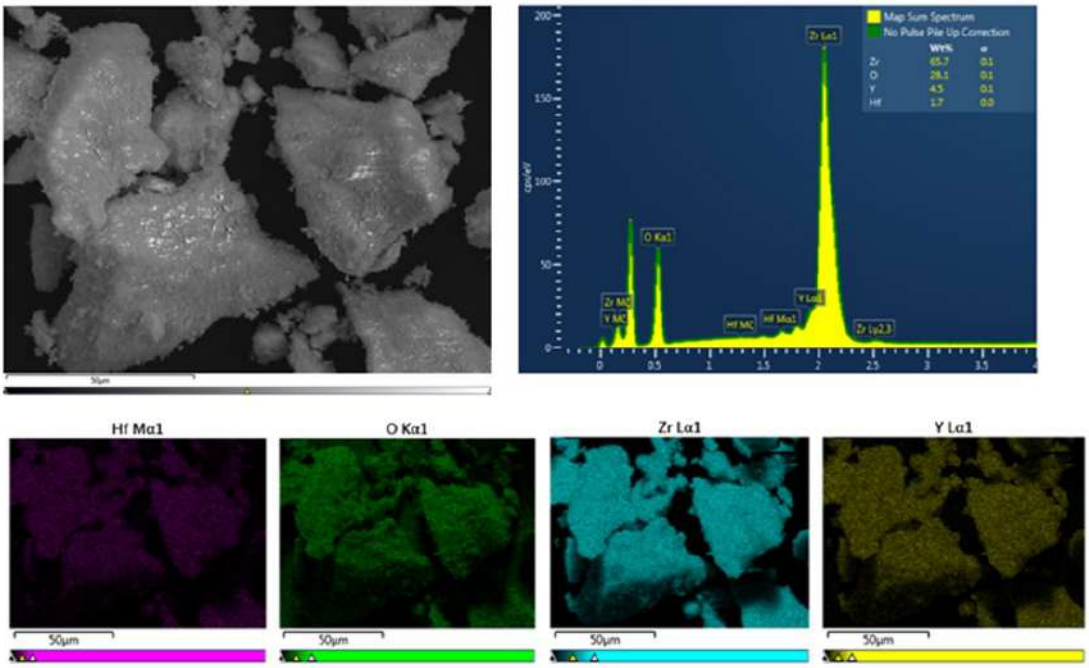

Figure 1. YSZ particles SEM image and EDS analysis.

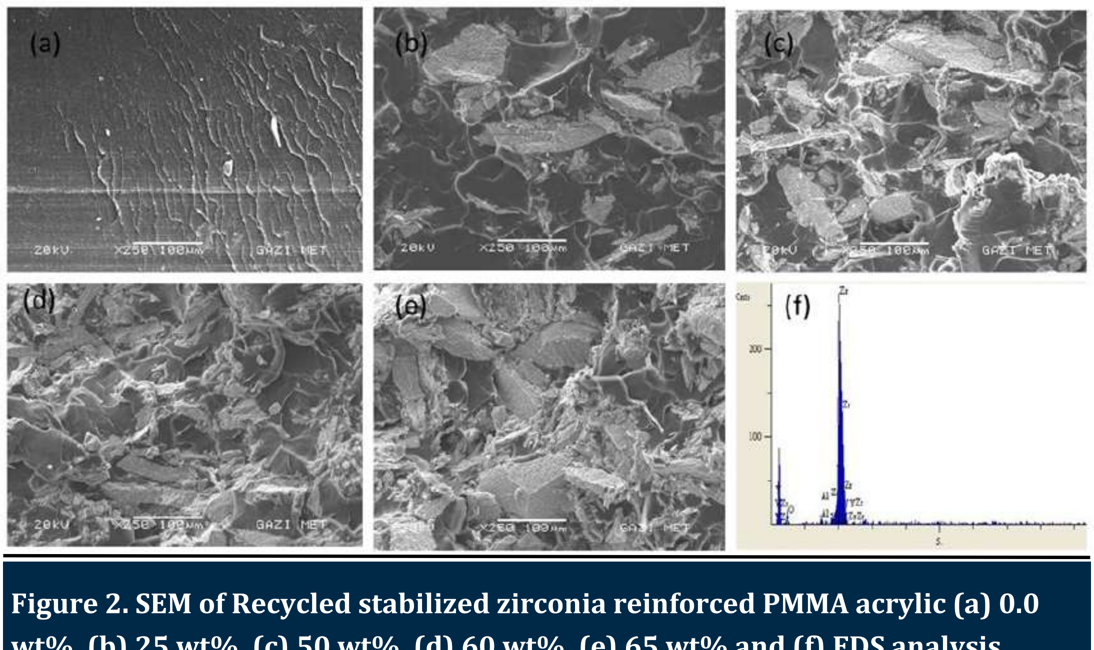

Figure 2. SEM of Recycled stabilized zirconia reinforced PMMA acrylic (a) 0.0 wt%, (b) 25 wt%, (c) 50 wt%, (d) 60 wt%, (e) 65 wt% and (f) EDS analysis

2.2 Statistical analysis

Data quality control and statistical analyses were done using IMB SPSS Statistics Version 25.0 (IBM SPSS, Chicago, IL, USA). Normal distribution and homogeneity of variance showed with Shapiro–Wilk and Levene's tests. Data were analyzed using a one-way analysis of variance (ANOVA) with the Tukey post-hoc test at a pre-set alpha of 0.05.

3. Results

3.1 SEM analysis and microstructural characteristics

According to SEM analysis (Figure 1), residue YSZ particles size was between 500 nm and 70 µm. According to EDS results, YSZ particles chemical compositions consist of 65.7 Zr wt%, 28.1 O wt%, 4.5 Y wt% and 1.7 Hf wt%. It can be seen from Figure 1 that EDS map analysis indicates homogeneous distributions of all phases in the particles. Figure 2 shows SEM microstructure and EDS analysis of unreinforced PMMA and ZrO_2 reinforced composites fracture structures.

Unreinforced PMMA fractured surfaces were very dense, smooth and homogeneous without porosity (Figure 2a). With the addition of YSZ to PMMA, the surface roughness of fractured surfaces significantly increased. Additionally, fracture surfaces roughness proportionally increased with increasing YSZ amount in structure Figures 2b-e. According to Figure 2e, over 60 wt% YSZ amounts caused YSZ agglomeration in the matrix. There are some voids among the YSZ agglomerated particles because of the unwet YSZ by PMMA matrix. These vacancies reduce the mechanical properties. FTIR test was carried out on unreinforced PMMA, and YSZ reinforced PMMA to evaluate the different active groups.

Figure 3 shows FTIR analysis results of unreinforced PMMA, 25.0 wt %, 40.0 wt%, 60.0 wt%, 70.0 wt% YSZ reinforced PMMA. Five new strong absorption picks were seen at wavenumbers of 2919.3, 1616.1, 1535.6, 598.4 and 567.7 cm^{-1} in the FTIR spectrum of YSZ reinforced PMMA composites compared to unreinforced PMMA. 1616.1 cm^{-1} resulted from the tension vibration of the hydroxyl groups for the YSZ surface. The stress vibration of the C-H bond at 2940 and 3000 cm^{-1} and that YSZ bond in the range of 1530-1620 cm^{-1} showed that PMMA successfully bond to YSZ (Figure 3). However, 70 wt% YSZ FTIR peaks are not strong as other FTIR peaks because of YSZ agglomerations and insufficient wetting by PMMA. Therefore, only weak chemical bonding between the PMMA and YSZ occurs over the 60 wt.% YSZ addition. These results were exactly matching with SEM images.

Figure 4 represents Vickers hardness and density variation with YSZ amount. Microhardness increased with increasing YSZ amount until 60 wt% YSZ addition from 23.45 to 31.18 (Table 2), indicating that surface hardness increased by over 24%. However, above 60 wt% YSZ addition, microhardness decreased because of ZrO_2 agglomeration in the composite structure. This issue proved successfully load transfer mechanisms that occur in the composite structures. Densities increased with the increasing second phase (YSZ) amount in the composite due to the higher densities of YSZ, which is 5.97 compared to the density of PMMA which is 1.17. This shows that YSZ density is 5.1 times higher than PMMA; hence density increased with the increasing YSZ amount.

As shown in Figure 5, unreinforced PMMA and YSZ reinforced PMMA matrix composites, low amount YSZ addition does not significantly affect the surface roughness of composites. However, with the increasing YSZ addition, surface roughness increased proportionally. Over 60 wt% surface roughness sharply increased due to the high amount of YSZ agglomerations.

The mean values of Vickers hardness in Table 2 showed statistically significant differences ($p<0.05$). There was no statistically significant difference between groups up to 25% YSZ addition ($p<0.05$). A maximum increase in microhardness was observed at 60 wt% zirconia ($p<0.05$) (Table 2). A sudden decrease was observed in the group containing 65 wt% zirconia, whereas 70 wt% zirconia powder addition was found to have the least microhardness ($p<0.05$).

The mean values of surface roughness in Table 2 showed statistically significant differences ($p<0.05$). The addition of 50 wt% zirconia did not make a significant difference in the roughness, while 60 wt%, 65 wt% and 70 wt% YSZ powder produced a significant increase in the roughness ($p<0.05$).

4. Discussion

This study showed that the residual powders obtained

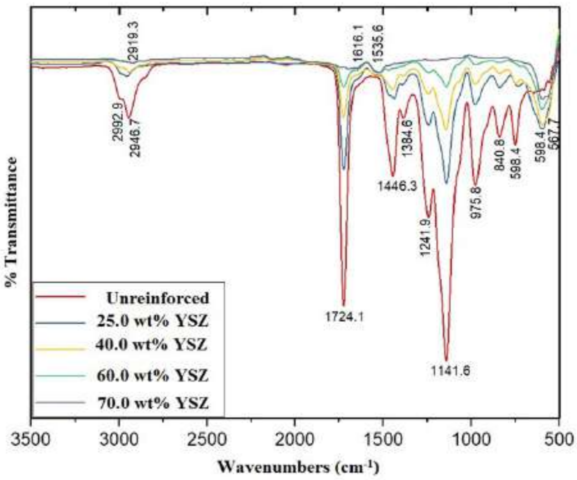

Figure 3. FTIR analysis of unreinforced, and reinforced PMMA with 25.0 wt %, 40.0 wt%, 60.0 wt%, 70.0 wt% of YSZ

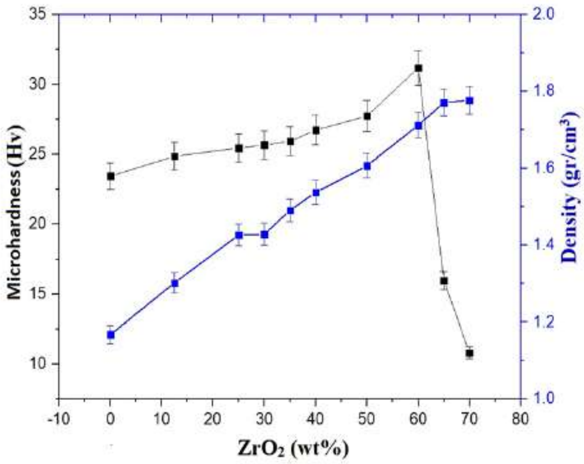

Figure 4. Microhardness and density of PMMA with various concentrations of ZrO_2 amount.

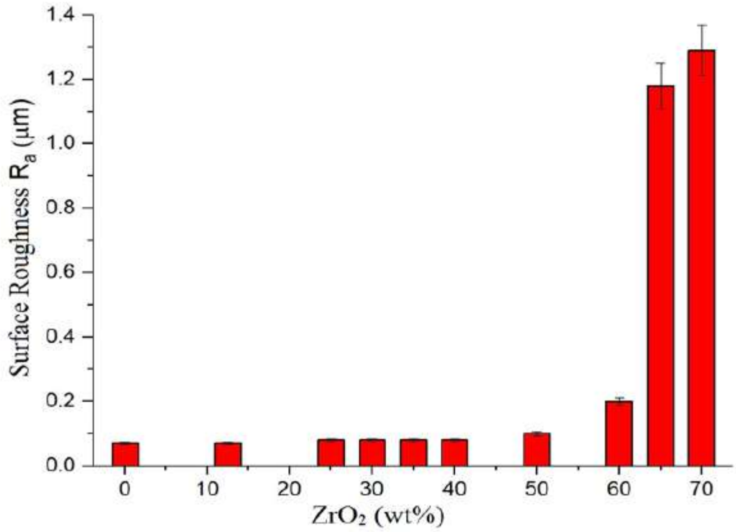

Figure 5. Surface roughness (Ra) variation with ZrO_2 concentration

Table 2. Mean and Standard deviation (SD) of Vickers hardness (kg/mm²), surface roughness and density of reinforced and unreinforced PMMA resin.

	Weight (%)	Vickers Hardness	Surface Roughness	Density
1	0.00	23.45±1.08 A	0.07±0.01 A	1.17±0.03 A
2	12.50	24.87±1.01 AC	0.07±0.01 A	1.30±0.07 B
3	25.00	25.44±3.59 AC	0.08±0.03 A	1.43±0.02 C
4	30.00	25.66±2.34 AC	0.08±0.02 A	1.43±0.05 C
5	35.00	25.03±3.12 AC	0.08±0.01 A	1.49±0.04 CD
6	40.00	26.73±1.03 BC	0.08±0.00 A	1.54±0.10 DE
7	50.00	27.73±2.49 BC	0.10±0.03 A	1.61±0.02 E
8	60.00	31.18±1.08 D	0.18±0.06 B	1.71±0.01 F
9	65.00	15.98±0.80 E	0.20±0.00 C	1.77±0.06 F
10	70.00	10.81±0.45 F	0.29±0.00 D	1.78±0.10 F

Within a column, values identified using similar upper-case letters are not significantly different.

after CAD/CAM machining had improved the mechanical properties of PMMA.

In the present study, Unreinforced and various amount residues YSZ reinforced PMMA matrix composites were successfully produced. The addition of residue zirconia powders addition at higher amounts increased Vickers hardness and density but did not affect the surface roughness of the PMMA composites.

PMMA fully covered all YSZ particles until 60 wt% YSZ addition, and good bonding between the PMMA and YSZ was observed with the formation of a very dense microstructure. Similarly, composites microhardness and densities increased with increasing YSZ amount in the composite structure.

Surface roughness was not significantly changed until 60 wt% YSZ additions. However, over the 60 wt% YSZ addition sharply increased surface roughness.

However, the microhardness and roughness values of PMMA acrylic resin groups with less than 50 wt% zirconia powder addition did not differ significantly from the control group ($p>0.05$).

Temporary crown material may wear out due to functional forces and foods; thus, it must have adequate abrasion resistance. Adding inorganic filler into PMMA can increase microhardness, especially the addition of hard fillers such as ZrO_2 [27]. By examining the acrylic resin hardness, residual monomer amount [24], resistance to abrasion [29], and ease of finishing of the material [9] are determined.

The results of the present study were in agreement with a study presented by Ahmed *et al. (2014)* that evaluated the hardness of PMMA/ZrO_2 nanocomposites with different ZrO_2 concentrations (1%, 3%, 5%, 7%) using the Vickers hardness test [27]. They found that all specimens showed hardness mean values higher than that control group, and PMMA specimen with 7% zirconium oxide nanofillers showed the highest mean hardness significantly.

Over 60 wt% YSZ additions to PMMA caused YSZ agglomeration in the structure and decreased mechanical properties because of vacancies among the agglomerated YSZ. This agglomeration can be attributed to insufficient wetting by PMMA.

With the addition of residual zirconia powder, Vickers hardness was increased up to 60wt% of YSZ, while it decreased significantly after adding 65wt% and 70wt% powder. The decrease in surface hardness with higher filler loading was caused by poor adhesion of the particles to the resin matrix and filler clustering within the matrix [28]. Zhang et al. investigated the effect of zirconia nanoparticles and aluminum borate whiskers (ABW) in PMMA denture bases on the surface hardness at concentrations of 1 wt%, 2 wt%, 3 wt% and 4 wt% [28]. The results showed an increase in surface hardness with an increase in ZrO_2/ABW content, and the optimum hardness was achieved at 3 wt% ZrO_2 nanoparticles.

In another study, Ergun *et al*. (2018) [30] investigated the physical and mechanical properties of PMMA reinforced with various ratios of zirconium oxide nanoparticles and observed a significant increase in hardness and surface roughness.

Hardness is the resistance of a material to plastic deformation. Surface hardness can be used as an indicator of density, and it can be hypothesized that a denser material would be more resistant to wear and surface deterioration [31].

Vojdani *et al*. (2012) [7] reinforced PMMA mixtures with Al_2O_3 at loadings of 0.5, 1, 2.5 and 5 wt%. They stated that Vickers hardness values increased linearly but could not find any difference in surface roughness. The Vickers hardness significantly increased after the incorporation of 2.5 and 5 wt% Al_2O_3 addition. No significant difference was detected in surface roughness between the reinforced and control groups.

The addition of recycled zirconium powder has been shown to increase microhardness, as in previous studies. This microhardness increase is due to the hard zirconium oxide (ZrO_2). An increase in microhardness was observed due to the crushing tip of the microhardness device hitting zirconium metal oxides.

One of the problems that surface roughness can cause in temporary dentures is colour change, adhesion of food and plaque formation, depending on the amount of roughness [32].

In the present study, with the addition of 60% or more recycled zirconia powder by weight, the roughness increase was found to be below 0.2micrometres, which can be accepted as clinically [33]. The roughness was directly proportional to the amount of recycled zirconia powder incorporated into the resin matrix. This increase is because of the zirconium particles in the matrix approaching the surface.

In the study groups, a statistically significant difference in density changes up to 50% zirconia, but no difference was found with the addition of 60% or more zirconia. As the recycled zirconia powder content increased, their values increased due to decreased pores [34]. The effect of nanoparticles on the mechanical properties of PMMA depends on several factors, including polymer particle interface, particle size, fabrication method, and particle dispersion in the PMMA matrix [35].

5. Conclusion

Different amount of residual YSZ reinforced PMMA was produced within a pressure vessel system. FTIR results showed good bonding between the residue YSZ and the PMMA matrix. According to SEM results, residue YSZ was homogenously distributed in the PMMA matrix until 60 wt.%. Over 60 wt.% additions caused YZS agglomerations in the matrix. Also, a high amount of residue YSZ addition (60 wt.%) decreased mechanical properties because of unwetted residue YSZ and space between the agglomerated YSZ powders. Another effect of high amount second phase additions sharply increased surface roughness. Composite structure density was increased with increasing residue YSZ additions due to the higher density of residue YSZ.

Provisional PMMA with superior mechanical properties was created using recycled zirconia without the need for special additional processing steps. Best mechanical and structural properties were achieved with 60 wt.% residue YSZ additions.

Conflicts of interest: Authors declared no conflicts of interest.
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