

Preparation of α -Cordierite Ceramic Powder as a Filler and its Effect on the Thermal Properties of Poly(methyl methacrylate) Denture Base Resin

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ABSTRACT

Purpose: This study aimed to synthesize α -cordierite ceramic powder as a reinforcement filler and evaluate its incorporation into a poly(methyl methacrylate) (PMMA) denture base to enhance thermal performance.

Materials and Methods: α -Cordierite ceramic powder was prepared from Al_2O_3 , SiO_2 , and MgO powders, which were mixed for 3 hours, melted to form a frit, and subsequently crushed, milled, sieved, and calcined. The denture base formulation comprised PMMA, α -cordierite ceramic powder, benzoyl peroxide (BPO) as the initiator, methyl methacrylate (MMA) as the monomer, and ethylene glycol dimethacrylate (EGDMA) as the cross-linking agent. Following standard dental laboratory protocols, α -cordierite powder at loadings of 0, 5, 10, and 15 wt% was dispersed into the MMA monomer via hand-mixing; the resulting dough-stage blend was packed into molds and processed into specimens.

Results: The incorporation of α -cordierite filler into the PMMA matrix altered the thermal properties of the denture base. The filled specimens exhibited higher degradation temperatures compared with the unfilled PMMA matrix. Furthermore, increasing the filler content progressively elevated the degradation temperature and enhanced the thermal stability of the PMMA composites.

Conclusion: The addition of α -cordierite ceramic powder as a reinforcement filler effectively improves the thermal stability and degradation resistance of PMMA denture bases, with higher filler loadings yielding superior thermal performance. (*Open J Biomed Res* 2026;5:52-64)

Keywords: α -cordierite, reinforcement filler, denture base, thermal properties.

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AUDIOABSTRACT



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INTRODUCTION

Tooth loss remains a serious issue among the global population, adversely affecting their chewing function, speech, facial appearance, and overall quality of life. As we get older, we

can lose teeth for many reasons one of which may be dental caries or periodontal disease. Consequences of these also include trauma.¹ It is important to replace any missing teeth so

that they can function properly and to help ensure the proper psychological wellbeing of the patients. These days, removable dentures are less common, but they might still be an appropriate treatment option for older and/or disabled patients.² The proper clinical and laboratory procedures is fundamental for the success of restoration of the missing teeth with removable dentures.³

Poly methyl methacrylate (PMMA) is the most used material for denture bases because of its esthetic properties, low cost, and low density.⁴ However, conventional PMMA is vulnerable to fracture resulting from repetitive masticatory, flexural, and impact loading, hence the question of its resistance to long-term deformation under service conditions.⁵

One of the current approaches to improve the mechanical and thermal properties of PMMA is the addition of ceramic fillers, such as Al_2O_3 , ZrO_2 , TiO_2 , SiO_2 , hydroxyapatite, halloysite nanoclay, etc.^{4,6-10} The addition of ceramic fillers into poly(methyl methacrylate) is an innovative approach that improves the thermal properties of removable denture bases. Compared to conventional PMMA, modified samples exhibit higher thermal conductivity, which helps patients to better perceive hot and cold temperatures in the oral cavity.¹¹ Such an effect is explained by the fact that Al_2O_3 , ZrO_2 , and SiO_2 fillers have a much higher thermal conductivity than polymethyl methacrylate.¹² Several studies have shown that the addition of ceramics into PMMA facilitates reducing the coefficient of thermal expansion, which is critical for the denture's performance.¹³ Finally, improved thermal diffusivity allows the prosthesis to adapt to hot and cold environments faster, which minimizes discomfort and protects the patient's oral mucosa from burns.^{14,15}

The present study prepares and incorporates a novel ceramic filler, which has not previously been used for this purpose, into the polymer matrix. The effect of this ceramic filler on thermal properties of this polymer-

based moving power base was systematically studied in order to evaluate its potential in enhancing thermal performance and broadening the use of polymer composites in dental science.

MATERIALS AND METHODS

First Stage: Preparation of α -cordierite Ceramic Powder

The preparation of the basic α -cordierite ceramic powder was according to the experiment done by Banjuraizah et al.¹⁶ to make a filler material for the composite. The α -cordierite ceramic powder is composed of three raw materials. Raw materials are: Magnesium Oxide MgO 19.93 wt %, Silica SiO_2 27.01 wt % and Alumina Al_2O_3 53.06 wt %.

In this process, the basic raw materials was mixed for 3 hours. Then, transferred into an alumina crucible before subjected to the melting process at 1500°C for 4 hours using LENTON EHF 1700 glass melting furnace. The molten product directly quenched in distilled water to form frit. The frit was dried at 100°C for 3 hours prior subjected to two milling processes.

First process, the product frit was crushed in the planetary ball milling technique (Pulverisette 6 Fritsch, Germany) (300rpm, 10min, and 20 mm tungsten balls), the resultant powders were then sieved to pass $150\mu\text{m}$. Second process, the sieved powders were milled using (10mm diameter tungsten balls at 300 rpm for 3 hours. The obtained glass powders were calcined in air under isothermal conditions for 2 hours at 900°C with $5^\circ\text{C}/\text{min}$ heating rate.

Second Stage: Incorporating of the α -Cordierite Powders as Filler into PMMA Powder

The solid elements consisted of PMMA with high molecular weight (966,000 GPC–Aldrich U.S.A) plus 0.5% benzoyl peroxide (BPO) (Merck Chemical, Germany). The liquid

component comprised of methyl methacrylate (MMA) (Fluka UK), stabilized with 0.0025% hydroquinone plus the cross-linking agent (10%) ethylene glycol dimethacrylate (EGDMA) (Aldrich USA) and toluene. The α -cordierite powder had selected as a strengthening filler with an average particle size of 1.06 μm . The silane coupling agent 3-(trimethoxysilyl) propyl methacrylate was supplied by Sigma-Aldrich. Silane as coupling agent (γ -MPS) which has been used in this study has a boiling point of 190 $^{\circ}\text{C}$ and flashpoint at 92.22 $^{\circ}\text{C}$.

10g of α -cordierite powder and 10 wt% of silane were added into 200 ml toluene. The solution was refluxed for 15 h before filtering in order to collect the modified powder. The powder was washed with 200 ml of fresh toluene in a Soxhlet apparatus for 24 h. The final product dried at 110 $^{\circ}\text{C}$ for 3 h under a vacuum. Filler treatment was done according to the method as reported by.¹⁷ The treated α -cordierite powder was blended with matrix material (PMMA and 0.5% BPO) with different ratios (0, 5, 10, and 15 wt%, respectively) in a plastic ball mill for 20 min.

The blending of powder to liquid (P/L) was done according to standard dental laboratory usage. After reaching the dough stage, the mix was packed into a mould and was pressed under 14 MPa, at room temperature for 30 min. The final polymerization (curing process) was carried out in a water bath at 78 $^{\circ}\text{C}$ for 90 min before the mold was left to cool slowly at room temperature. After that, the samples were removed.

Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) was used to evaluate thermal stability and investigate the filler content of dumbbell-shaped specimens and examine the uniformity of filler distribution in the polymer, samples were collected from various regions. The substance was then analysed by a PerkinElmer Pyris 6 TGA analyzer. All the samples were heated from 50 $^{\circ}\text{C}$ to 550 $^{\circ}\text{C}$ at

a constant heating rate of 20 $^{\circ}\text{C}/\text{min}$ under N_2 atmosphere at a flow rate of 50 mL/min. During the test, a high-alumina crucible was used to hold the sample to be weighed on an analytical balance outside the furnace. As the temperature increased, the instrument was continuously recording the sample mass. Thus, TG curves were created. These curves represent the percentage of weight loss versus temperature. An analysis of the structural integrity of the polymer composites during heating was performed by using thermograms to evaluate the weight loss on agglutination and thermal stability of the samples.

RESULTS

Analysis on the Synthesized of α -Cordierite Ceramic Powder as Reinforcement Filler

As mentioned earlier, the basic raw materials for the preparation of α -cordierite was mixed before subjected to the melting process to form frit. The frit crushed to produce glass powder. Finally, the obtained glass powders were calcined to get α -cordierite ceramic powder. (Fig. 1- A, B).

Filler Particle Size Analysis

Reinforcing fillers enhance the properties by many mechanisms. In particular, the special properties of the fillers are of great significance. These would include the particle shape, its size and distribution of sizes, and the surface chemistry of the particle.^{18,19} In this study, the particle size analysis of α -cordierite ceramic powder is provided in (Fig. 2). It showed the statistics related to particle size of the α -cordierite ceramic powder tested in this study. The mean particle size of α -cordierite is 1.06 μm . In addition, there is no change in the size after treatment with silane.

Scanning Electron Microscopy and EDX

The sample of α -cordierite was observed under Scanning Electron Microscopy (SEM) to investigate the particle's size and shape. The morphology of the synthesized α -cordierite

ceramic powder is shown in (Fig. 3). at 1000 x magnification. It can be noted that α -cordierite ceramic powder has irregular shapes as well as small in particle size. Moreover, the filler was tested under EDX to confirm the essential

elements which formed α -cordierite. As illustrated, Figure 4 shows the EDX graph of α -cordierite ceramic powder. It was found the presence of the raw materials which formed α -cordierite filler.



A



B

Fig. 1: A) Frit with pieces of alumina crucible after quenching in distilled water. B) Frit (prior subjected to two milling processes to produce powder).

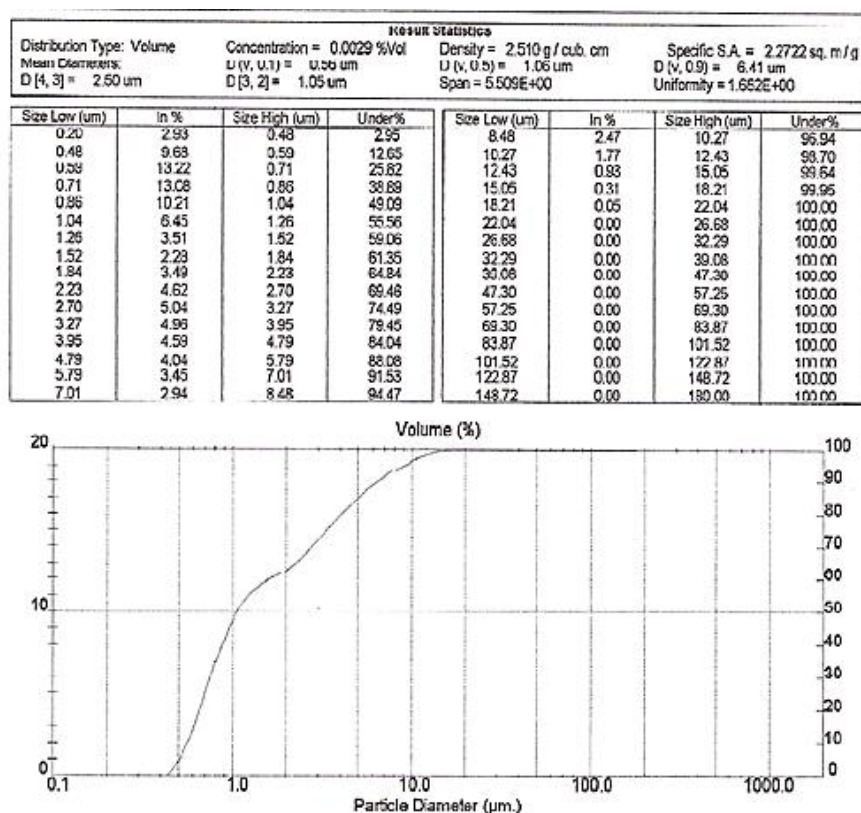


Fig. 2: The result analysis report of α -cordierite particle size.

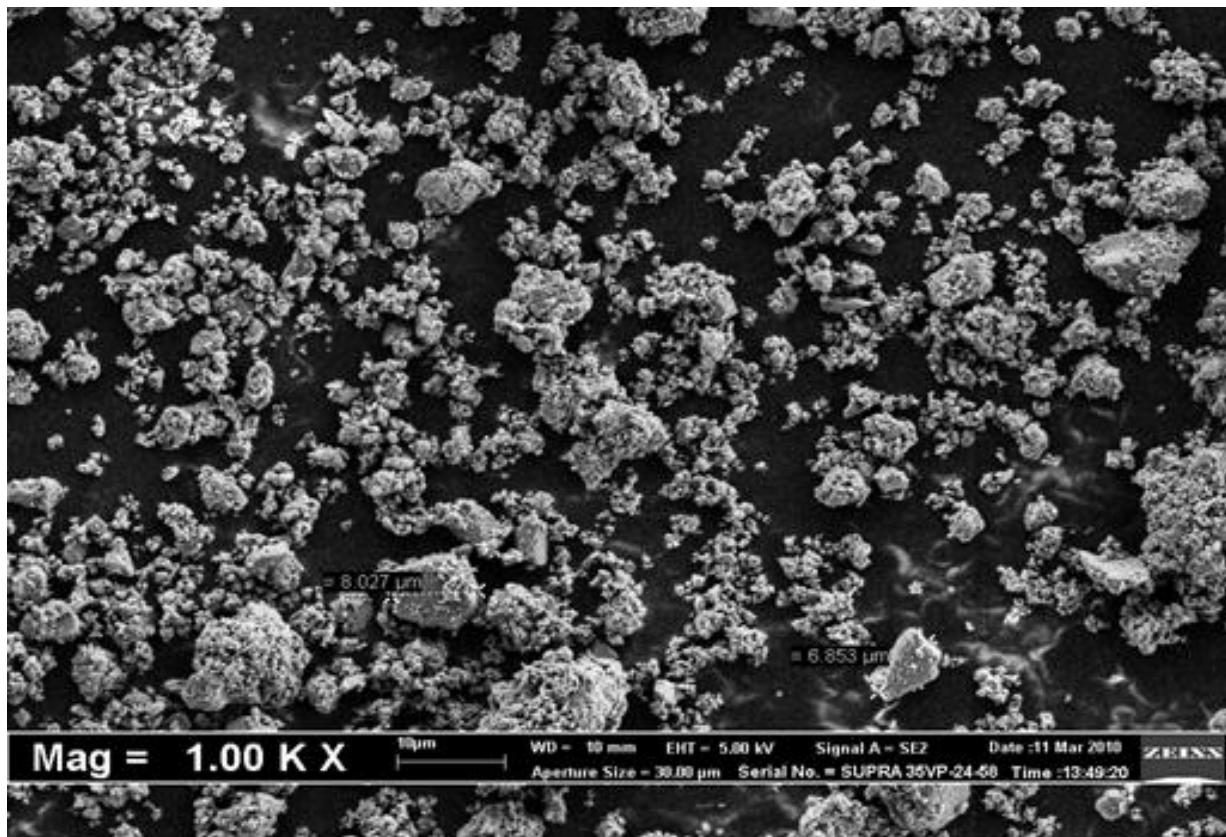


Fig. 3: The micrograph of α -cordierite ceramic powder at 1000x shows the particle size.

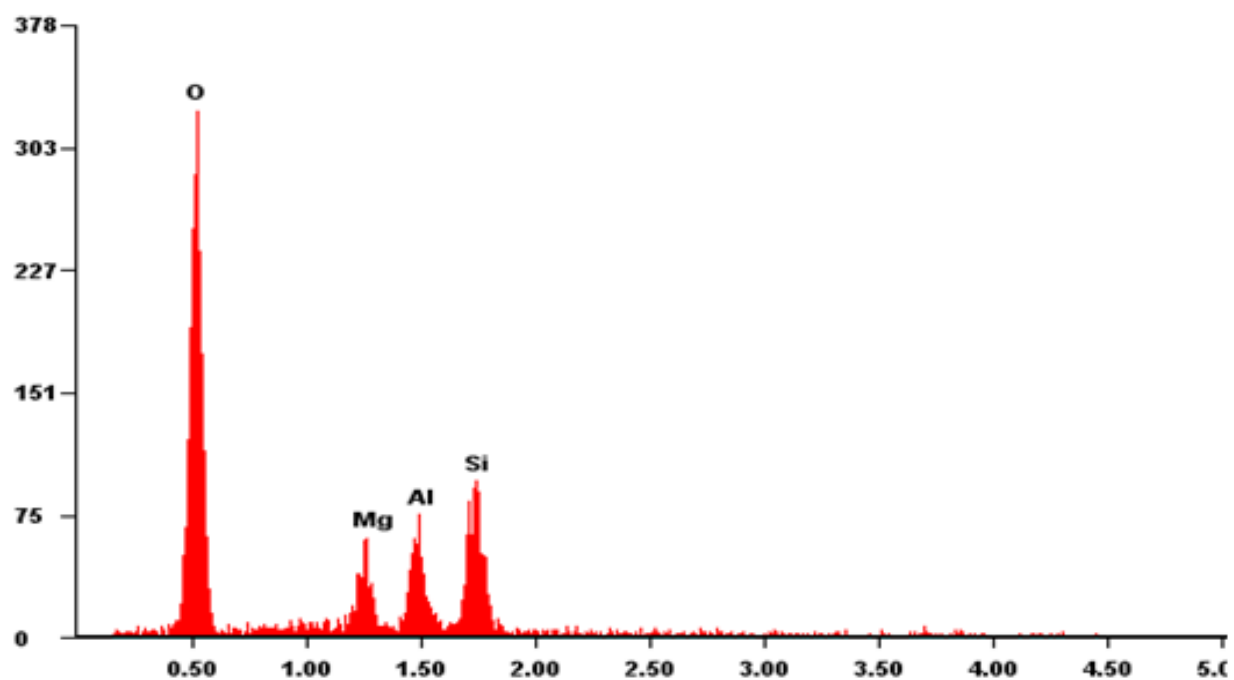


Fig. 4: The EDX graph of α -cordierite ceramic powder.

X-Ray Fluorescence (XRF)

Table 1 and figure 5 show the results of the X-Ray Fluorescence (XRF) analysis. XRF test was performed on the α -cordierite ceramic powder. It can be noted that XRF analysis has indicated that all the raw materials used for the preparation of α -cordierite ceramic powder and the total percentage is 100%.

Table 1: The results of the XRF analysis of α -cordierite.

Components	Results (wt %)
MgO	18
Al ₂ O ₃	29
SiO ₂	53
K ₂ O	0.027
CaO	0.19
Fe ₂ O ₃	0.050
CO ₂ O ₃	0.011
NiO	0.014
CuO	Trace
WO ₃	0.32

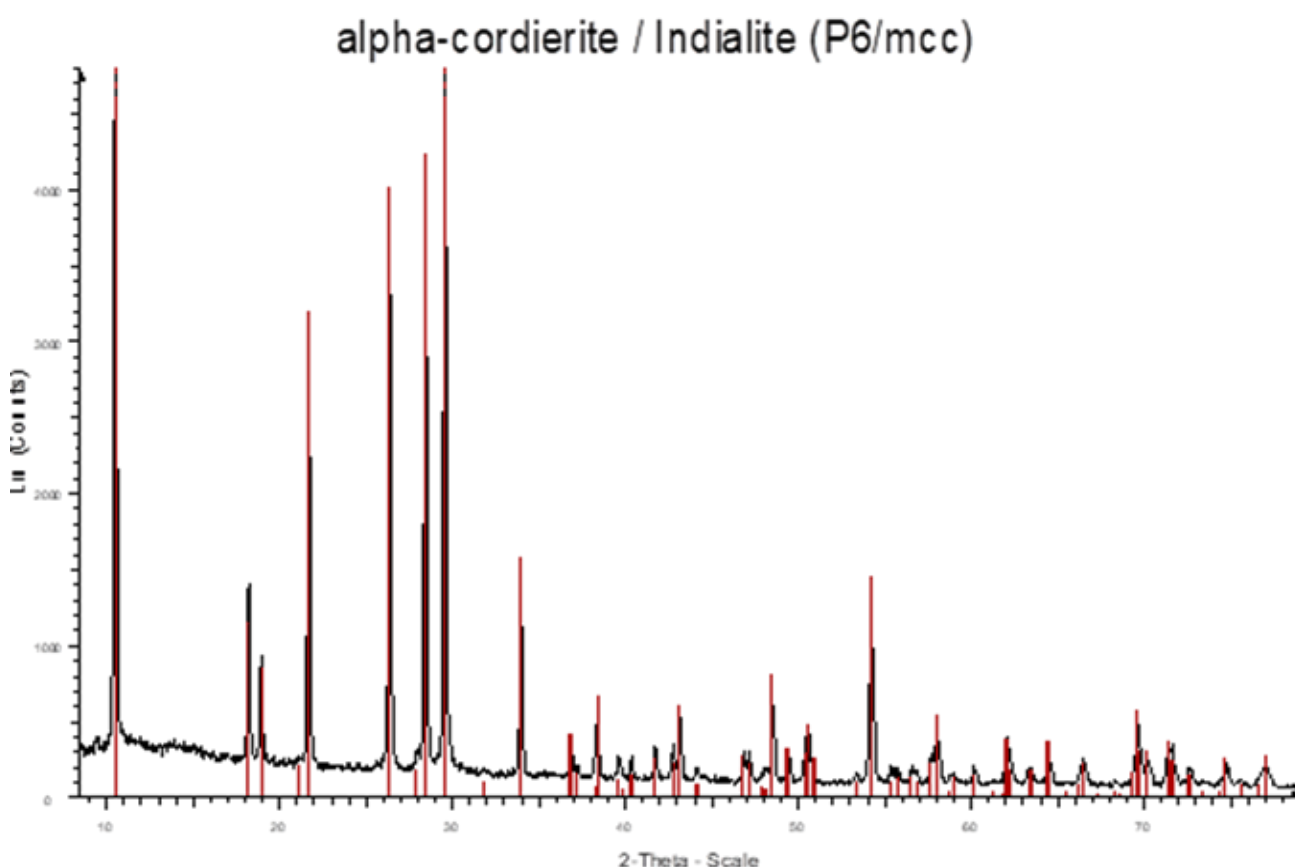


Fig. 5: The XRD patterns of α -cordierite ceramic powder.

Thermal Characterization

The TGA results of different ratio of α -cordierite filled PMMA are shown in (Fig. 6). It is clear to note that the PMMA matrix sample and PMMA/ α -cordierite composites sample all loss the weight up to a temperature 250-300 °C. After this temperature, a sharp decrease in weight is observed at 430-450 °C. PMMA matrix sample it is assumed to be completely burnt out at temperature 430-450 °C. In addition, the residual can only be the added α -cordierite. It can be noted that there are several differences in the thermal degradation patterns for different formulation.

The degradation temperature of the α -cordierite ceramic-filled samples is slightly higher than that of the PMMA unfilled samples. The PMMA matrix samples showed no residual material at temperature of 435 °C. Moreover, figure 6 shows that all PMMA/ α -cordierite composites samples illustrate similar thermal pattern. Also, it is found that some slight differences are seen in the thermal degradation temperatures of 15 and 5 wt% formulations. As such, a 15 wt% is slightly higher degradation temperature than that of 5 wt% sample. This indicates that the 15 wt% filled content is more thermally stable than that of 5 wt% sample. Finally, TGA result confirmed α -cordierite filler content exactly.

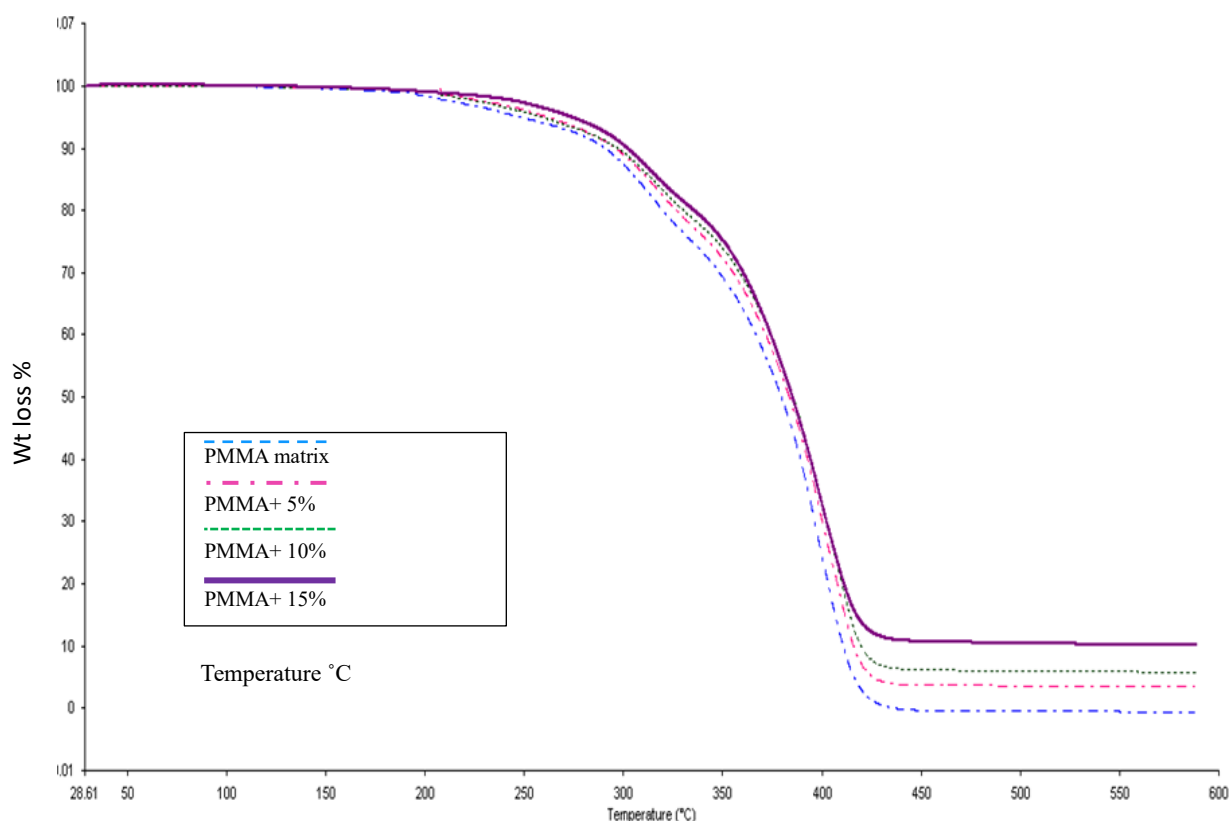


Fig. 6: TGA Curves showing the thermal degradation temperature of the PMMA composites compared to PMMA matrix.

DISCUSSION

The clinical performance and long life of denture base polymers are highly influenced by the thermal properties of the polymers used.

In the normal oral condition, polymer material shows good thermal stability. But it is possible for this material to show dimensional change, thermal degradation, and deterioration of

mechanical property when exposed to heat.²⁰⁻²² The improvement of the thermal resistance of the polymer-based denture materials is necessary to preserve its physical and service life. The use of appropriate fillers especially ceramic fillers has been reported to improve thermal stability by restricting mobility of polymer chains and increasing resistance against deformation due to heat.^{23,24} Polymer composites often undergo standardized thermal tests like thermogravimetric analysis (TGA) for thermal degradation. Test results show that denture base materials are suitable for use in clinical use for a long time at different temperature.^{25,26}

The PMMA matrix samples showed no residual material at temperature of 435 °C. This attributed to the fact that all polymers start the decomposition at high temperature thus, the temperature between 300-600°C enough to decompose even very stable polymers.²⁷⁻²⁹

Many researches stated that the addition of ceramic fillers leads to a significant increase in thermal conductivity. The improvement observed in the α -cordierite ceramic filler-reinforced PMMA denture base materials can be attributed to several factors, and these findings are consistent with those reported in several previous studies. Several previous studies have reported similar findings, including the following-:

The enhanced thermal properties of the dentures made from ceramic-reinforced polymers are attributed to the high thermal conductivity of fillers, such as alumina and zirconia, compared to PMMA.^{30,31,32} Increasing the filler content facilitates the formation of connected domains of ceramic particles, thus creating avenues for easier heat conduction and dissipation.^{30,31} Besides, the silanization process also plays an essential role by improving the interfacial bonding, thereby reducing thermal resistance.¹¹ More importantly, the use of nanoparticles as fillers enhances the surface area, which improves thermal diffusivity and stability.^{13,31} Altogether, the described mechanisms explain why

ceramic-reinforced PMMA prostheses offer a more natural sensation of heat and cold to the patient.^{30,33} The increase in the thermal properties observed in the present study can be attributed to the small particle size of the α -cordierite filler used. It is observed that the increasing in thermal properties can be related to reduce in particle size of fillers. According to previous literature,³⁶⁻³⁹ the smaller particle size facilitates the homogeneous distribution of particles within the polymer matrix compared with larger particle sizes. Since the α -cordierite filler used in the present study possessed a very small particle size, a more uniform distribution within the PMMA matrix was achieved, which may have contributed to the improved thermal properties. Additionally, The particle shape of the filler has the influence on the most properties of the composites. For example, the spherical shape provides a uniform stress distribution on its surface.⁴⁰ Polymer composites with flake or lamellar reinforcements can give higher modulus than composites with irregular shaped filler. However, irregular shaped fillers have the sharp edges or points create a high stress concentration which can lead to initiation of premature failure of a polymer matrix composite.^{41,42}

Moreover, figure 6 shows that all PMMA/ α -cordierite composites samples illustrates similar thermal pattern. Also, it is found that some slight differences are seen in the thermal degradation temperatures of 15 and 5 wt% formulations. As such, a 15 wt% is slightly higher degradation temperature than that of 5 wt% sample. This indicates that the 15 wt% filled content is more thermally stable than that of 5 wt% sample. Finally, TGA result confirmed α -cordierite filler content exactly. This finding is in agreement with one made by Abudalazez who reported that the formulation 15 wt% of the opaque dental porcelain-filled samples was more thermally stable than that of 5 wt% sample.⁴³

Studies mentioned that the best filler concentration of 15% was achieved due to filler

particles arrangement and interaction that took place within the material structure, where ceramic fillers distributed into the polymeric matrix formed connected regions or structures allowing an easier energy transfer pathway compared to the insulating polymer chains.^{15,44}

Figure 7 shows SEM micrograph of the fractured surface of the 15 w% (treated α -cordierite filled) PMMA matrix obtained at 1000x magnification. It can be seen that filler particles were embedded and bonded to the matrix. The interpretation was aligned to what other studies concluded that the interfacial bond between the filler and matrix can hinder

the energy transfer route, the uniform dispersion of these powders the reason why distribution at this concentration reduced the occurrence of isolated gaps with low thermal resistance; thus, increasing the overall thermal diffusivity of the fabricated composite.¹⁵ By achieving this level, the benefit of maximizing the high thermal conductivity of the ceramic phase was attained without inducing a negative impact on the polymer such as agglomeration at higher levels that could jeopardize the stability of the denture base.¹² Further research is needed to evaluate the mechanical and physical properties of the tested material and its biocompatibility.

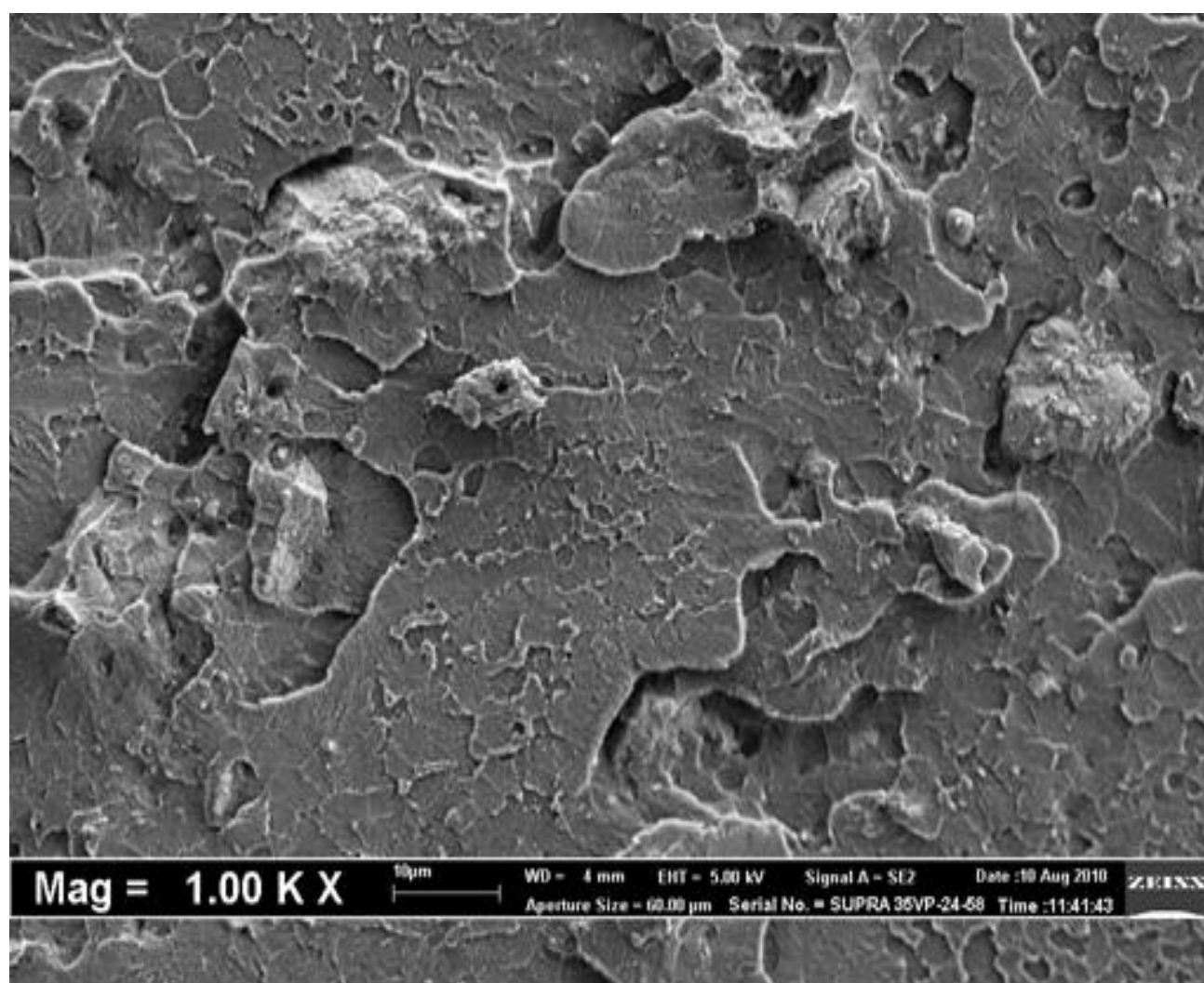


Fig. 7: SEM micrograph of the fracture surfaces of the 15 w% (treated α -cordierite-filled) PMMA matrix at 1000x magnification.⁴⁵

CONCLUSION

The use of α -cordierite ceramic powder as a reinforcing filler in the composition of PMMA denture base resin was found to be effective. The α -cordierite filler was successfully prepared and incorporated into the polymer matrix using standard dental laboratory techniques. The results of thermogravimetric analysis confirmed that the addition of α -cordierite as a filler improved the thermal stability of the material. Moreover, the increase in thermal stability was found to be related to the amount of filler added. Thermal analysis showed that the higher the concentration of α -cordierite filler, the higher the degradation temperature of the composite material. These findings indicate the potential of α -cordierite for use in PMMA denture base resin to improve thermal resistance and create more thermally stable dentures.

Conflicts of Interest

The authors declare no conflicts of interest.

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