

# **Development of Polymer Composite Filament of PLA-ZrO<sub>2</sub> for Dental Applications**

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## **Abstract**

The polymer ceramic composite has various dental applications due to its superior mechanical properties. The processing of polymer ceramic material through fused filament fabrication (FFF) is still a challenge due to specific requirements related to feedstock. Therefore, the current study attempts to develop a feedstock filament of Polylactic Acid (PLA) and Zirconia (ZrO<sub>2</sub>) for FFF. The solvent casting and screw extrusion methods have been used for polymer composite and filament preparation respectively. The quality of the developed filament has been examined using Scanning Electron Microscope. The results show good homogeneity and particle dispersion and the diameter of the developed filament is in the range of 1.65 mm to 1.75mm.

## **Keywords**

Additive manufacturing, fused filament fabrication, tooth crown, and dental applications.

## **1. Introduction**

The tooth is one of the most important parts of the human body. For feeding, it is very necessary to have a healthy tooth but with time teeth get damaged. It can happen due to many reasons like tooth decay, accident, injuries, or just use over time, because of this tooth can lose their original shape. It is one of the most common teeth tissue diseases in oral clinical practice (Dai et al. 2016). To overcome this problem, tooth-shaped crowns are placed over the teeth (Francis et al. 1995). Dental crowns are made from many materials, but the selection of material is based on many factors such as good strength, cost-beneficial analysis, and good biocompatibility (Rathi and Verma 2018). Materials for crown restoration fall into three basic types metal (Wassell, Walls, and Steele 2002), ceramic, and metal-ceramic (Iridio-platinum and Palladium 1956). Presently, CAD/CAM method is used for the fabrication of a dental crown and other dental applications. But CAD/CAM method (Mantri and Bhasin 2010) used for crown fabrication is capital extensive. To reduce capital extensive, an Additive Manufacturing method like Fused filament fabrication (Mohd Pu'ad et al. 2019) is used because it has the capabilities to fabricate complex three-dimensional structures. For fabrication of dental crown using FFF, it is necessary to have filament under a certain range of diameter. A composite of Polylactic Acid (PLA) and Zirconia (ZrO<sub>2</sub>) ceramic powder is used for the development of customizable filament. PLA and ZrO<sub>2</sub>. Zirconia is an amphoteric oxide with a crystalline structure that can be monoclinic, tetragonal, or cubic and can react as either acid or a base substance. It is widely employed because of its superior physicochemical characteristics, which include high chemical stability, high mechanical strength, high melting point, high resistance to fracture, high corrosion resistance, low thermal conductivity, ionic conduction, and bio inertness (Albanés-Ojeda et al. 2020). Polylactic acid (PLA) is a thermoplastic polymer developed by molecules of lactic acid, which are linked by ester bonds and it has good biocompatibility and biodegradability (Tümer 2021).

### **1.1 Objectives**

- Synthesis of polymer ceramic composite.
- Development of polymer composite filament.

### **2. Literature Review**

Albanés-Ojeda et al. (2020) fabricated polymer ceramic composite using PLA and zirconia. In this experiment different ratios of PLA and zirconia have been used for the preparation of composite and the tests were also done on composite. Polymer and ceramic mixed well for 24 hours for getting a homogeneous mixture. After testing it has been found that the mechanical properties of polymer increased by mixing ceramic into it. The most significant finding was that composites are mesoporous materials and that mesoporousness is caused by ZrO<sub>2</sub> nanoparticles, which suggests that PLA/ZrO<sub>2</sub> composite may be employed as a biomaterial.

(Sida and Sida 2019) fabricated a tooth using FDM. The goal of our effort is divided into two tasks. The first involved developing the flexible ceramic filament using extrusion technology, and the second involved modifying a normal rep-rap printer to use this filament so that it could extrude the material through a heated nozzle. The author used ceramic as a material because of its mechanical properties.

### 3. Materials and Methods

#### 3.1 Materials

Major material properties required for dental applications are chemical resistance, density, hardness, ability to be colored, and shades. The main material used for the development of the polymer ceramic composite is Polylactic Acid and Zirconia of a size of 500 nanometres. But apart from that other chemicals like Chloroform ( $\text{CHCl}_3$ ) and Ethanol ( $\text{C}_2\text{H}_5\text{OH}$ ) were also used. These chemicals are solvents in the preparation of the composite (Figure 1).

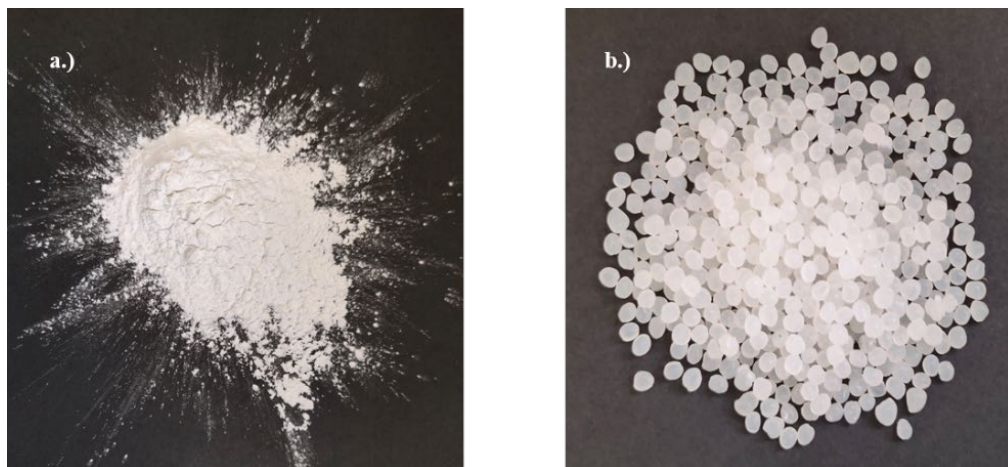


Figure 1. (a) Zirconia powder, (b) PLA pellets.

#### 3.2 Synthesis of Polymer Ceramic composite

PLA-ZrO<sub>2</sub> composite is synthesized with solvent casting in which a ratio of 6:1 of PLA and Zirconia has been used. 6 gm of PLA pellets and 1 gm of Zirconia powder has been mixed in 100 ml of Chloroform for 12 hours at room temperature using a magnetic stirrer as shown in Figure 2 below. After that, 33 ml of ethanol was used to form a homogeneous mixture. Material is again stirred for 20 minutes after adding ethanol to it. After all the process a polymer ceramic composite is developed in wet form, and that is further dried at room temperature for 8 hours. After that polymer ceramic composite is heated at 60 degrees Celsius using a hot oven air machine to remove chloroform from the composite (Table 1).

Table 1. Ratio of PLA and ZrO<sub>2</sub>

Sample	PLA ( $\pm 0.05$ ), gm	ZrO <sub>2</sub> ( $\pm 0.05$ ), gm	PLA/ZrO <sub>2</sub>
PLA-ZrO <sub>2</sub>	6	1	6



Figure 2. (a) Polymer ceramic composite preparation using Magnetic stirrer, (b) The mixing process of polymer composite

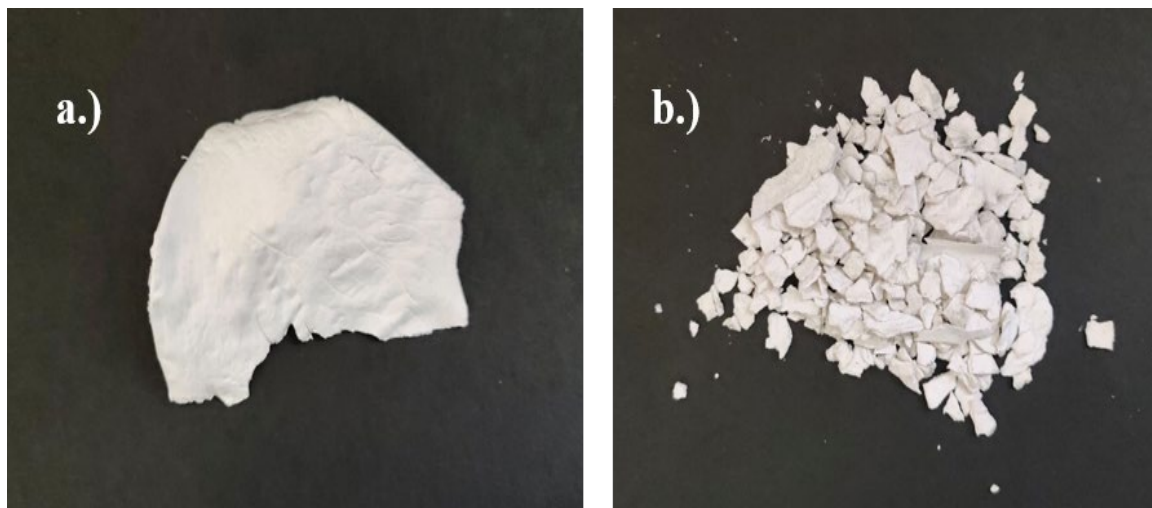


Figure 3. Prepared PLA-ZrO<sub>2</sub> composite, (b) Crushed PLA-ZrO<sub>2</sub> composite.

#### 4. Development of Filament

Development of filament has been done using a customized filament extruder. Two major factors can affect the filament diameter during the extrusion process. The first factor is screw rotation which determines the feed rate of the pellets in the heater and screw rotation is set at the optimum value using a DC motor. The second factor is the temperature of the extruder which is controlled using a PID controller. The temperature of the heater is set at 190 degrees Celcius using a PID controller, as PLA is the binder in the polymer ceramic composite, so the extrusion temperature should be the same as PLA. But there is some air gap between the thermocouple and heater, and because of that the set temperature is much higher than the required temperature, so the temperature is set at 135 degrees Celcius for optimum result. After that, the Composite has been crashed down into smaller pieces and filled inside the hopper, and the DC motor is started for the rotation of the screw. Because of the rotation of the screw, the feeding of composite pieces is started inside the barrel. A filament of composite is come out from the nozzle of the extruder and air is used for the cooling of filament.

#### 5. Results and Discussion

A filament wire of a diameter range of 1.65 mm to 1.75 mm is successfully developed with the help of a customized filament extruder the developed filament is shown in the figure, and a part is printed with this filament using FFF technique. The temperature during the printing is 220 degrees Celsius. Developed filament and 3d printed parts are shown in Figure 4(b) below.

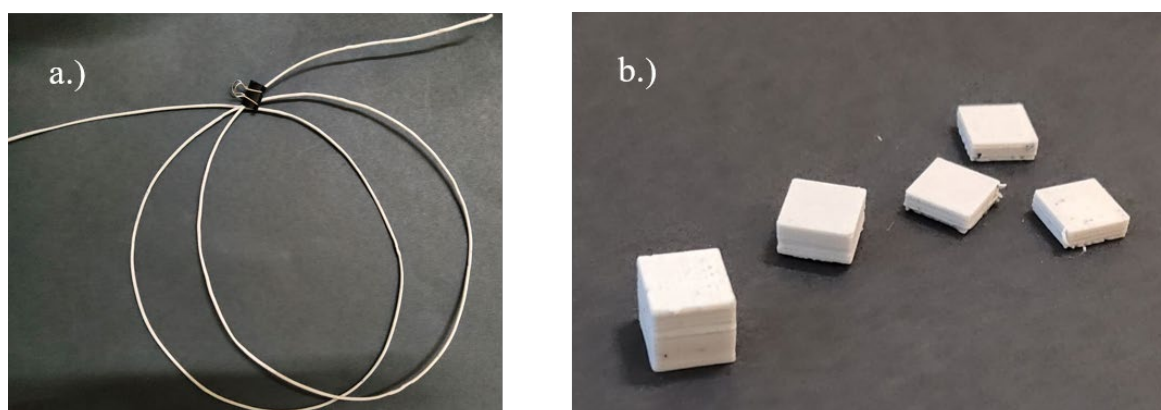


Figure 4. (a) Developed filament (b) 3d printed parts

In SEM analysis, a signal can produce a two-dimensional image and reveal information about the sample, including external morphology. High-definition image of PLA-ZrO<sub>2</sub> composite produced using SEM analysis is shown in Figure 5.

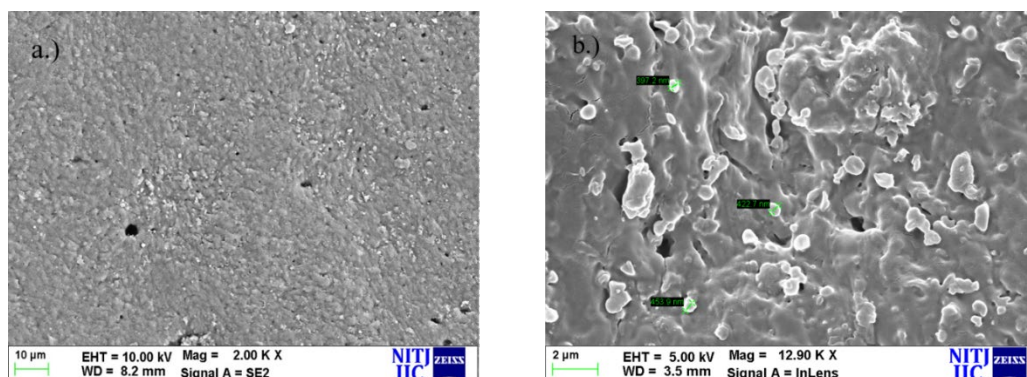


Figure 5. (a) SEM of the surface of composite material, (b) The size of ceramic material in the composite.

From the SEM analysis, it is concluded that the polymer and ceramic are mixed homogeneously, and a proper particle dispersion takes place. The average size of ceramic inside PLA-ZrO<sub>2</sub> composite is 400 nm as shown in Figure 5(b).

## 6. Conclusion and Future scope

Using the solvent casting method, it is possible to synthesize PLA-ZrO<sub>2</sub> Composite with homogeneity and good particle dispersion. In this study, it is examined that ceramic powder should have a sub-micron size for homogeneity of the polymer composite. For the development of filament temperature and screw rotation are important factors. After examining different temperatures, it is found that extrusion of filament takes place at 135 degrees Celsius and the diameter ranges from 1.65 mm to 1.75 mm. By changing PLA-ZrO<sub>2</sub> percentage ratio in the composite, there may be a difference in homogeneity and mechanical strength. But there are many ceramics and polymers available which can be used for the synthesis of composite according to their applications. Apart from composite, a DC motor regulator can be used for controlling screw rotation which can improve filament extrusion and another cooling system can be used for filament cooling.

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