Fabrication and characterization of composite dental material using X-ray radiography

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Abstract. Acrylic resin is a material that often used in the manufacture of dentures. Composite dental base materials consist of acrylic and glass powder. The addition of glass powder aims to improve the physical and mechanical properties. The increases in the mass fractions of glass powder in composite dental were optimized between 12.5% - 75% and added 2 ml of liquid for each sample. The composite dental surface is characterized using x-ray radiography, CCD microscopy, and the hardness is characterized using Vickers hardness tester, and the density is also measured. The result shows a mass fraction of 75% was the optimum condition with a radiographic gray level image of 196.814 at a voltage of 50 kV and a current of 16 mA, hardness of 26.7 g/mm$^2$, density of 2.28 g/mm$^3$ and greater mass fraction of glass powder, the surface of the composite dental look tighter and lighter in color.

1. Introduction
The losing teeth not only reduces aesthetics, but also decreases the function of chewing and reduces nutritional intake, so this will affect the general health condition and quality of life of a person. Therefore, it is necessary to make artificial teeth as a substitute for natural human teeth [1]. Dental resin composites are popular materials to repair caries [2]. At present, dental procedures and engineering of artificial teeth are preceded by a simulation stage [3,4,5].

In modern era, the world of dentistry has developed ways to maintain the health of teeth [6]. Due to many patients demands for better aesthetics and the development of dental adhesive techniques, composite resin has become the choice in various dental treatments [2,7].

The acrylic resin is the most widely used material in dentistry as a base material for dentures, because it has good strength, physical and aesthetic properties, low water absorption, low dimensional change, and it is easy to repair. This composite resin is also one of the restorations that can fulfill a conservative cavity design because there is no need to take healthy tooth tissue for additional mechanical retention as is the case with metal restorations [8]. The indications and contraindications for using composites are as sealants in preventive resin restorations to prevent caries in deep and narrow basins. Therefore, in this study, denture materials were made using heat polymerization acrylic resin with the addition of glass powder as a composite material. Bioactive glass is one of the filler materials for dental resin composites [9]. The purpose of this study was to determine the optimum composition of glass powders, observe surface properties, determine the material hardness [10], and measure the density of composite teeth. By adjusting the optimum composition of glass powder, the mechanical and physical properties of the composite teeth can be obtained. This is a preliminary research to engineer artificial composite teeth.
2. Method

The research was conducted at the Physics Laboratory of the Universitas Negeri Semarang following flowchart as in Figure 1. There are two types of materials used, acrylic with glass powder, and acrylic without glass powder. Other ingredients are Liquid ISO 1567 Type 1 Class 1, and water. This research uses X-ray equipment, Mednif SF-100BY, MS-804 CCD microscope, Micro Vickers Hardness Tester FM-800, heater, hydraulic press, and Kris Chef Type EK9350H digital balance. Composite teeth were made by means of a molding process using a hydraulic press. The X-ray machine was used to generate X-rays which will be exposed to the composite teeth and then the image was processed using integrated software. The microscope was used to observe the surfaces of the composite teeth that had been made and its hardness were tested using a microhardness vickers test apparatus.

The first stage is making sample A (without glass powder) which is done by mixing acrylic powder and liquid without glass powder at a concentration of 4 g/ml. The mixture is then stirred using a spatula slowly in a glass for 15 minutes until thick and let stand for 15 minutes. Then it was inserted into the cuvette and the cuvette mold was placed under hydraulic press of 1000 psi by heating at 100 °C for 30 minutes. After the polymerization process, the cuvette was allowed to cool slowly to room temperature. The addition of glass powder mass fraction was optimized between 12.5% -75% and added 2 ml of liquid in each sample.

The surface morphology was observed using a CCD Microscope MS-804 in 100x magnification. Observation of the surface morphology of the material was also carried out using X-ray radiography at a setting of 16 mA electric current, 0.125 s exposure time, and the X-ray tube voltages were varied at 40-50 kV with voltage interval 5 kV. This voltage variation is applied to determine the gray level of each of these voltages [11,12]. Composite tooth hardness numbers were tested using the Micro Vickers Hardness Tester FM-800 with a minimum load of 25 gf and calculated using the Equation (1):
In Equation (1), \( VHN \) is sample hardness \( (kg/mm^2) \), \( P \) is load weight (kg), and \( D \) is diagonal length (mm) [10].

The steps to determine the density of composite teeth are to compare the volume of water before the object is immersed with the volume of water after the object is inserted. After an object is immersed in water, the water will experience an upward force where the volume of water is equal to the volume of the object. The density of an object is the ratio between the mass and volume of object, calculated using Equation (2).

\[
\rho = \frac{m}{V} \tag{2}
\]

In Equation (2), \( \rho \), \( m \), and \( V \) shows density (kg/mm\(^3\)), mass (kg), and volume of object (mm\(^3\)), respectively.

3. Results and Discussion

3.1. The measurement of gray level of radiography image using Matlab Software

This measurement is to determine the level of light falling on each pixel as an intensity value. The images of exposure to composite dental material using digital radiography are shown in Figure 2. The three images are generated from X-ray exposure at different acceleration voltages, namely 40 kV, 45, and 50 kV. At the higher acceleration voltage, the resulting image appears brighter. Qualitatively, the image resolution looks good which can be seen from the outside of the visible sample circle. The areas that are exposed to more X-rays will appear lighter. If the mass fraction of the glass is higher, the composite will become denser, the intensity of the X-rays transmitted will be smaller, and produce a darker image.

Figure 2. Radiographic X-ray images are generated at applied voltages (a) 40 kV, (b) 45 kV, (c) 50 kV

Figure 3 shows that the level of gray is higher when the mass fraction of glass powder on the composite teeth is increased. Based on the results of image analysis in this study, it can be seen that 50 kV is the highest voltage. The higher the tube voltage is applied, the image contrast decreases. Changes in tube voltage will affect the quantity and quality of X-rays. Increasing the working voltage of the tube affects the intensity and size of the X-ray penetrability. Therefore, the resulting image at a voltage of 50 kV produces a large gray level and looks brighter. Meanwhile, at the 40 kV and 45 kV voltages, the resulting radiographic image looks darker. Therefore, the lower the density of an object, the greater its penetrating power, so that the image of composite with mass fraction 25% looks darker and that of mass fraction 75% looks brighter.
Figure 3. The Gray level values of composites with different mass fractions at accelerating voltages of 40 kV, 45 kV, and 50 kV.

3.2. Observation of composite tooth surface morphology using a CCD microscope (Charge Couple Device)

The results of observing the surface of the composite teeth using a CCD microscope with a magnification of 100x are shown in Figure 4. In Figure 4, the surfaces of the composite teeth generally show bright contours which are marked with white spots, these show that the homogeneity of the composites surfaces is different. In the sample with the smallest glass powder mass fraction, which is 12.5%, there are many piling up gray-white patterns. Whereas in samples with a mass fraction of glass powder 75%, the surface of the composite teeth was more dominated by glass powder, the tighter and brighter it looked.

Figure 4. Composite tooth surface images using a CCD microscope for samples: (a) without glass powders, with glass powders ((b) 12.5%, (c) 25%, (d) 37.5%, (e) 50%, (f) 62.5%, & (g) 75%).

3.3. Composite Tooth Hardness Testing

Figure 5 shows the hardness of composite teeth with different glass powder levels (12.5%, 37.5%, 50%, 50%, 62.5%, and 75%) and the hardness values compared to teeth made of acrylic without glass powder. In Figure 5, it can be seen that the largest Vickers hardness value of 26.7 kg/mm\(^2\) is owned by composite with a mass fraction of 75%. While the results of the hardness test for composites with mass fractions of 12.5%, 25%, 37.5%, 50%, and 75% were 14.7 kg/mm\(^2\), 16.5 kg/mm\(^2\), 16.8 kg/mm\(^2\), 17.1 kg/mm\(^2\), 19.7 kg/mm\(^2\), and 21.1 kg/mm\(^2\), respectively. These results indicate that the composite dental material
using acrylic resin undergoes heat polymerization, and when the glass mass fraction is increased, the hardness value increases. These values indicate that the composite dental material using acrylic resin undergoes heat polymerization with the addition of glass powder to the sample. Glass has a higher hardness than acrylic, so the addition of glass powder to composite teeth increases its hardness properties.

Figure 5. The hardness of composite teeth for mass fractions of 0-75%.

3.4. Composite tooth density analysis

Material density measurements using Equation (2) were carried out using a beaker filled with water and the results are presented in Table 1. The hardness of the fluctuating composite teeth is thought to be due to the polymerization process which is influenced by environmental and technical factors, resulting in defects such as voids.

| Mass fraction (%) | Density (g/cm³) |
|-------------------|-----------------|
| 12.5              | 1.63            |
| 25.0              | 1.81            |
| 37.5              | 2.00            |
| 50.0              | 1.81            |
| 62.5              | 1.86            |
| 75.0              | 2.10            |

Table 1 shows the densities of the composite teeth with varying levels of glass powders were compared the density of acrylic teeth. The highest density of samples was produced by composite with mass fraction 75% (2.1 g/cm³). The average density of other other composites (with mass fractions 12.5%, 25%, 37.5%, and 62.5%) were 1.63 g/cm³, 1.81 g/cm³, 2 g/cm³, 1.81 g/cm³, and 1.86 g/cm³, respectively. While the control composite density was 2.28 g/cm³. This shows that the decrease of the composite tooth density is due to the porosity of the sample.

4. Conclusion

Composite tooth composition was successfully optimized with relatively good hardness, surface conditions and density. The greatest hardness of specimens of 26.7 kg/mm² was found in composite with 75% mass and the lowest one (16.5 kg/mm²) was found in acrylic mass fraction of 12.5%. As the
mass fraction of glass powder increases, the hardness of the resulting composite teeth improves as well. Next, the greater the applied electrical voltage, the greater the gray level measured in the digital radiographic image. We also observed that the greater the mass of the glass powder given, the tighter the surface of the composite teeth looks lighter. An optimum density of composite dental materials, 2.1 g/cm³, was found in composite with a mass variation of 2 g of acrylic and 6 g of glass powder, and the lowest density, 1.63 g/cm³, was found in composite with a mass variation of 7 g of acrylic and 1 g of glass powder. As the mass of given glass powder increases, the density enlarges as well.

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