Color changing in denture base polyamide 12 and polyamide microcrystalline after polishing in laboratory and dental clinic

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Abstract. This study’s purpose was to compare color changes from polyamide 12 and polyamide microcrystalline materials. Each material was divided into two groups, polished with laboratory and clinical polishing equipment, and then immersed in a 28 °C coffee solution for seven days. Surface roughness was measured with a profilometer before the immersion. Color (L*, a*, and b*) was measured using a spectrophotometer before and after immersion. There were no significant color changes in the polyamide 12 and polyamide microcrystalline materials after immersion in the coffee solution.

1. Introduction
A wide variety of dental materials have been developed to meet patients’ needs, including the base material for removable partial dentures. Polyamide, or thermoplastic nylon, is produced from the condensation between diamine and dibasic acid [1]. This material has the advantages of being more aesthetically pleasing and flexible, of possessing heat and chemical resistance, and of being easy to modify to increase rigidity and resistance to wear. A good balance between strength, customization, and ability to withstand temperature changes makes nylon a good candidate as a substitute for acrylic [2]. Polyamide has a variety of material modifications, such as polyamide 12 and polyamide microcrystalline, and is in great demand because it offers patients flexibility and a better aesthetic.

The denture base often experiences color changing after being used for a certain period of time. The color changing becomes an aesthetic problem and may occur due to intrinsic and extrinsic factors. The intrinsic factors relate to material changes in the matrix, conversion level, imperfect polymerization, and chemical reactivity, while the extrinsic factors are caused by adsorption, absorption, surface roughness, degradation due to chemicals and aging, oral hygiene, diet, cleaning fluid, smoking habit, saliva composition, denture cleaning, and the duration of using the denture [3-7].

The denture base’s surface roughness is important because it affects tissue health, comfort, and the aesthetic. A restoration with a high degree of roughness may lead to discoloration [8] According to Fueki et al., polyamide 12 materials have a greater surface roughness than polyamide microcrystalline [9]. However, this study did not examine the effect of clinical polishing, which is sometimes necessary during denture insertion. Adjustments are done to increase patient comfort, and several visits are sometimes required. However, the tools used in the clinic are limited compared to the polishing performed in a laboratory. The polishing done in a clinic can change the material’s surface roughness differently than the polishing performed by a technician [10]. The amount the roughness changes may affect the accumulation of dye that sticks to the material’s surface [5,7,9]. Therefore, this study
examines the differences of color changing in the polyamide 12 and polyamide microcrystalline denture bases after polishing in a laboratory and in a clinic.

2. Materials and Methods
The type of study conducted was a laboratory experimental research. The specimens were polyamide 12, trademark Valplast (Valplast®, Albany, NY, USA), and polyamide microcrystalline, trademark Lucitone FRS (DENTSPLY Trubyte, New York, PA, USA). The research specimens were 30 x 15 x 3 mm and were polished in the laboratory; the specimens were accurately sized, not porous, and not curved [11]. The number of specimens was determined with G Power, and at least 21 specimens were necessary in each group. Therefore, this study used 22 specimens for both groups of materials. Eleven plates of polyamide 12 were polished with a green stone at a speed of 5000 rpm for 90 seconds. Eleven plates of polyamide microcrystalline were polished with a tungsten carbide at a speed of 10,000 rpm and a silicon carbide (coarse, medium, and fine-light) at a speed of 5000 rpm for 90 seconds. The roughness measurement was conducted on the entire surface of the polyamide 12 and polyamide microcrystalline materials using a profilometer, trademark Mitutoyo (Mitutoyo Co., Kawasaki, Japan), and the color measurement was taken using a spectrophotometer, brand SpectroShade (MHT SpA, Verona, Italy). All specimens were then immersed in a coffee solution for seven days, which is equivalent to drinking coffee for one year (assuming drinking coffee for 15 minutes twice a day). Every 24 hours, the coffee solution was replaced with a new one. After seven days, each specimen was cleaned with flowing water and dried with an air spray. The color measurement was repeated on the polished side of each specimen using a spectrophotometer, brand SpectroShade (MHT SpA, Verona, Italy). The color changing was determined by analyzing the color differences that occurred on each specimen before and after immersion for seven days.

3. Results and Discussion
3.1 Results
An univariate analysis was performed on the roughness data. The highest mean value of roughness was found in polyamide microcrystalline with clinical polishing, while the smallest mean value was found in polyamide 12 and polyamide microcrystalline with laboratory polishing (Table 1).

| Table 1. Magnitude of roughness on the polished surface of polyamide 12 and polyamide microcrystalline denture base materials after polishing; Ra Mean±SD (μm) |
|-------------------------------------------------|-----------------|-----------------|-----------------|
| Laboratory polishing | Clinical polishing | p-value |
| Polyamide 12 | 0.168 ± 0.0275 | 0.876 ± 0.195 | 0.000 |
| Polyamide Microcrystalline | 0.169 ± 0.0157 | 1.080 ± 0.089 | 0.000 |
| p-value | 0.925 | 0.007 |

Table 2. Magnitude of discoloration on the polished surface of polyamide 12 and polyamide microcrystalline denture base materials after polishing and then being immersed for seven days in a coffee solution

| Table 2. Magnitude of discoloration on the polished surface of polyamide 12 and polyamide microcrystalline denture base materials after polishing and then being immersed for seven days in a coffee solution |
|-------------------------------------------------|-----------------|-----------------|-----------------|
| Laboratory polishing | Clinical polishing | p-value |
| Polyamide 12 | 2.132 ± 0.837 | 2.340 ± 0.575 | 0.506 |
| Polyamide Microcrystalline | 2.007 ± 0.642 | 2.295 ± 0.578 | 0.282 |
| p-value | 0.697 | 0.858 |
### Table 3.
The results of an unpaired t-test regarding the magnitude of discoloration on the polished surface of polyamide 12 compared with polyamide microcrystalline denture base materials after being immersed in a coffee solution for seven days

| Material type          | N  | Mean ± SD  | Mean difference (CI95%)                      | p-value |
|------------------------|----|------------|---------------------------------------------|---------|
| Polyamide 12           | 22 | 2.236 ± 0.708 | 0.085 ([-0.318]-0.488)                      | 0.673   |
| Polyamide Microcrystalline | 22 | 2.151 ± 0.614 |                                            |         |

The univariate analysis regarding the magnitude of discoloration on the polished surface of polyamide 12 and polyamide microcrystalline denture base materials after polishing in the laboratory and in the clinic and then being immersed for seven days in a coffee solution showed that polyamide 12 material experienced the greatest color changing with clinical polishing, while the polyamide microcrystalline material experienced the smallest color changing with laboratory polishing (Table 2). A bivariate analysis was performed to see the differences in color changing on the polished surfaces of polyamide 12 compared to polyamide microcrystalline denture base materials after immersing in a coffee solution for seven days; an unpaired t-test was performed and concluded there was no significant difference in color changing (Table 3).

To analyze the relationship between the roughness and the color changing on the polished surface of a polyamide 12 denture base material after polishing and immersing in a coffee solution for seven days, a Pearson correlation test was performed; the correlation was not significant (p = 0.374), with a correlation of 0.199. To analyze the relationship between the roughness with the color changing on the polished surface of the polyamide microcrystalline denture base material after polishing and immersing in a coffee solution for seven days, a Pearson correlation test was performed and determined that the correlation between the roughness with the color changing on the polished surface polyamide 12 denture base material was not significant (p = 0.326), with a correlation of 0.220.

#### 3.2 Discussion
This study aims to analyze the magnitude of color changing of polyamide 12 and polyamide microcrystalline materials after polishing in the laboratory with a conventional polisher and in the clinic with a dentist kit. Polyamide 12 and polyamide microcrystalline materials were selected in this study because many patients choose to avoid metal dentures due to increasing aesthetical expectations. The polyamide 12 material has been developed for more than 50 years and is most widely used for a flexible denture base; however, this material has advantages and disadvantages. The polyamide microcrystalline material represents the next generation that can overcome some polyamide 12 deficiencies. Each has a manufacturing provision for production and adjustments in the dental unit. Coffee is the most widely consumed drink in Indonesia after tea. Adimarza conducted a research comparing the color changing on heat-cured acrylic resin and thermoplastic nylon materials immersed in a solution of coffee and tea, and the results showed a coffee solution produces a greater color change than the tea [12]. Buyuyilkamaz and Ruyter performed research on heat polymerized and three brands of auto-polymerized denture base materials immersed for 1000 hours in a solution of coffee and tea, and the coffee produced a greater color change than the tea [1]. The color changing in the polymer material due to the adsorption and absorption of coffee and this solution penetrated deeper than the tea, which only happened due to adsorption on the material surface [1,13]. Some other color-changing researches have used colored food or drinks, such as cola, red wine, soy sauce, and curry [1,4,13]. However, this study used coffee, as it is the beverage most consumed in Indonesia on a daily basis.

The results of the univariate analysis determined that the roughness with the highest mean value was found in polyamide microcrystalline with clinical polishing, while the smallest mean value was found in polyamide 12 and polyamide microcrystalline with laboratory polishing. In the roughness analysis of polyamide 12 and polyamide microcrystalline materials, the results showed no significant difference in the roughness mean value between polyamide 12 and polyamide microcrystalline materials.
materials after polishing in a laboratory. This result was different from Fueki et al., who had conducted a literature review and reported that the polyamide 12 material had greater roughness than the polyamide microcrystalline [9]. These different results may occur due to the differences in the polisher types. In this study, the tools and materials used for laboratory polishing were the same for both materials. The polishing tools used in the laboratory were carbide burs, green stone, and black rubber point, and the polishing was performed with a polishing machine using a brush with pumice. The brush was then replaced with a cotton buff, and the pumice was still used as a polishing material. The base was then washed and dried with an air gun. Next, Tripoli (green compound) with a different cotton buff was used before a shiny compound with wool polish was used to shine the base surface. Analysis of the roughness differences on the polished surface of polyamide 12 compared with the polyamide microcrystalline denture base materials showed a significant difference of roughness mean value in the clinical polishing. This result could be because different types of polishers were used. The manufacturer recommended a green stone polisher for the polyamide 12 material, and the same polishers used for polishing the acrylic could be used for the polyamide microcrystalline, which in this study were a carbide bar, a gray coarse silicon carbide bar, a chocolate medium silicon carbide bar, and a gray silicon carbide bur fine-light. The roughness on the polyamide microcrystalline material was found to be greater because this material used acrylic polishers, which were the polishers recommended by the manufacturer. This showed that the use of polyamide microcrystalline material after polishing with acrylic polishers required additional polishing with a polishing machine to produce a smoother surface.

Analysis showed a significant difference in the roughness on the polished surface of polyamide 12 and polyamide microcrystalline denture base materials after polishing in the laboratory and in the clinic, likely due to the different polishing materials used. Kuhar and Berger stated that polishing with conventional equipment in the laboratory produced a smoother surface than the polishing tools in the clinic [10,14]. Berger estimated that this happened because of the differences of grit pumice and polishing compound in the polishing kit composition used [14]. Therefore, polishing in the clinic with a polishing kit may require additional polishing with a polishing machine using pumice. The use of a cotton buff/wet cloth wheel and rough pumice followed by a high-shine buff can produce a polyamide surface roughness below the threshold that can be received by the oral cavity and that can reduce the possibility of bacterial colonization on the surface of the denture base (Ra = 0.2 μm) [15]. There are roughness variations in both the clinical and the laboratory polishing because an operator performs the polishing. The base material polishing tool is not a flat surface, and the pressure on the polisher is difficult to control, particularly during clinical polishing [10]. It is important to always follow every stage of polishing based on the roughness level of the polishing materials: start with a rough polisher, then use a medium roughness polisher, and finally a smooth polisher.

Univariate analysis of the magnitude of color changing after immersion for seven days in a coffee solution showed that the polyamide 12 material polished in the clinic experienced the most significant color change, while the polyamide microcrystalline material polished in the laboratory experienced the least color change. This result was because the crystallinity level influenced the color changing. Polymers with higher crystalline levels are more resistant to color changing compared to polymers with higher amorphous levels because color molecules penetrate more easily through the amorphous portions [16]. Materials with a high degree of crystalline produce an atomic arrangement that is regular, strong, and stiff [17]. Polyamide microcrystalline material is more rigid than polyamide 12, as judged by the greater modulus of elasticity [9]. Polyamide microcrystalline has more crystalline percentage than polyamide 12; thus, the color changing in polyamide microcrystalline is smaller than in polyamide 12. The denture base material has chromophores (>C = O), which are known to be polarized. Chromophores are part of the molecules that are responsible for the coloring. On the polyamide denture base, there is also auxochromes (>N=), which are atomic functional groups attached to chromophores that modify the ability of chromophores to absorb light and to change wavelength or absorption intensity. Auxochromes when combined with chromophores and free radicals in a solution can cause staining [18]. The color measurement with a CIE Lab system obtained color changing
The values of +L*, which represents the base material becoming darker; +a*, which indicates a decrease in red color; and +b*, which indicates an increase in yellow color. This is in line with Koksal’s study, which examined the color changing of materials immersed in coffee and tea [19]. The color changing after immersion in a coffee solution is due to yellow coloring with various polarities inside. Tannic acid is a compound in coffee that causes a color changing [1,13].

In this study, a bivariate analysis was performed to look at the differences of color changing on a polished surface of polyamide 12 compared to polyamide microcrystalline denture base materials after immersion in coffee solution for seven days. There was no significant difference in color changing on the polished surface of polyamide 12 compared to polyamide microcrystalline denture base materials after immersion in a coffee solution for seven days. This was different from Takabayashi’s research, which used a polyamide 12 material that experienced a greater color change than the polyamide microcrystalline immersed in a coffee solution [18]. This difference may have occurred because Takabayashi used an immersion material in the form of a coffee solution with a temperature of 70 °C for 60 hours, while this present study performed immersion at room temperature (± 28 °C) for seven days (168 hours). According to Hersek, the temperature factor affected the diffusion coefficient, which then affected the amount of liquid absorption [12]. The amount of liquid absorption can affect color changing [5]. Therefore, this study used room temperature, and the color-changing results did not differ significantly. Furthermore, the kind of coffee also affects color changing.

The different result of mean value measurement in color changing of both materials after polishing in the laboratory and in the clinic showed no significant difference of color changing mean value between polyamide 12 and polyamide microcrystalline immersed in a coffee solution. This research also analyzed the color-changing differences on a polished surface of polyamide 12 denture base material after polishing in the laboratory and in the clinic and the analysis of color-changing differences on the polished surface of polyamide microcrystalline denture base material after polishing in the laboratory and the clinic after immersion in a coffee solution for seven days. The four analyses showed no significant difference in mean values of color changing between the two polished materials in the laboratory or in the clinic. This finding was probably due to the immersion time of only seven days, which is equal to drinking coffee for approximately one year (assuming drinking coffee for 15 minutes twice a day). A seven-day immersion at room temperature (±28 °C) may not have been enough to initiate color changing in the polyamide material. The color changing that occurs in polyamide 12 and polyamide microcrystalline materials increases according to the roughness increase in both materials. Rough surfaces provide spreading reflections because the surface affects the amount and the type of light reflections, while a smooth surface provides the reflections in the same direction [3]. However, the statistical correlation between roughness and color changing on polyamide microcrystalline and polyamide 12 materials was not significant. There was a variation in magnitude of color changing in this study. The magnitude of the variation in the color-changing value on this thermoplastic nylon material is also seen in the study of Adimarza [12]. Further research is needed with different solution concentrations, immersion materials, temperature, and immersion times. Other research tasks should include determining the magnitude of differences in roughness levels on thermoplastic nylon material with a wide range of commonly used trademark polishing kits.

4. Conclusion
There were significant roughness differences on the polished surface of polyamide 12 and polyamide microcrystalline denture base materials after polishing in the clinic; however, after polishing in the laboratory, there was no difference. There was no significant difference in color changing between the different materials (polyamide 12 and polyamide microcrystalline) and between the same material (between polyamide 12 or between polyamide microcrystalline) after polishing in the laboratory and in the clinic and then immersing the materials in a coffee solution for seven days.
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