Synthesis of colloidal zinc nanoparticles by pulse laser ablation technique using fundamental Nd:YAG laser 1064 nm

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Abstract. The synthesis of high-purity Zinc nanoparticle (ZnNPs) using the pulse laser ablation method has been successfully performed. In this study, Low-power neodymium-doped yttrium aluminum garnet (Nd:YAG) laser was used as an energy source. Pulse laser beam (1064 nm, 7ns, 10 Hz) was focused on a high-purity (99.95%) zinc plate surface, which was immersed in 10 ml of aquades. Light brownish color of colloidal ZnNPs was successfully produced. The SEM image of ZnNPs shows that the produced nanoparticle had a spherical shape. The average diameter of ZnNPs was 35 nm with a standard deviation of 8.7 nm, and the surface plasmon resonance is located approximately at 300 nm.

1. Introduction

Zinc (Zn, Z=30) is a metallic element that very useful in human life. With the development of science and technology, a lot of metals has been synthesized as nanoparticle to optimize the use of the particle. The nanoparticle is a microscopic particle with a size ranging from 1-100 nm and can be classified by their nature, shape, and size [1]. Zinc nanoparticles (ZnNPs) also show a good foresight. In agriculture, ZnNPs can be used as a fertilizer [2]. Besides, it can also be used as materials for making solar cells [3]. In a medical application, ZnNPs can be applied as an anti-bacterial as well as molecular diagnostics [4,5]. Thus, the synthesis of ZnNPs is very important in various fields.

There are several methods used to synthesize ZnNPs, such as wet chemical method [6], precipitation methods [7], sol-gel method [8], spray pyrolysis method [9], and one-step levitational gas condensation method [10]. However, those methods have some disadvantage, such as complex preparation, complex equipment, and some methods need the addition of chemical solution in the production process, and therefore, the nanoparticle produced has low-purity.

In the medical application, the high purity of ZnNPs is very crucial. Some chemical and reactant used in chemical methods can be hazardous and not very suitable for the medical application. Because of that, we need a method that can produce ZnNPs with high purity and simple preparation. The other method to synthesize ZnNPs is pulse laser ablation (PLA).

In PLA method, the laser was focused on a sample surface that is located in pure aquades. The laser then ablated the surface causing the evaporation of small amount of sample that will merge with the surrounding liquid and forming a colloidal nanoparticle. The advantage of this method compared with other methods is the method needs very simple sample preparation, simple equipment, low cost and high-purity of the nanoparticle can be achieved.

In this study, a pulsed Nd:YAG laser was used as an energy source for ZnNPs production. The nano-colloid was then characterized by using scanning electron microscopy (SEM) to show the
morpohology of produced nanoparticle. Nanoparticle size distributions and surface plasma resonances were also studied.

2. Material and Methods
Experimental setup used in this study was shown in Figure 1(a). In this research, we used a pulsed Nd:YAG laser (New Wave Research, Polaris II, 20 Hz) as the radiation source. The laser wavelength, energy, repetition rate, and pulse width were 1064 nm, 40 mJ, 10 Hz and 7 ns, respectively. The laser beam was reflected the silver mirror and then focused by a convex lens with 30 mm focal length. The focused beam was directed to the high-purity (99.95%) zinc plate that located at the bottom of the petri dish containing 10 ml of aquades. The ablation process happened for 80 minutes.

![Experimental setup](image1)

**Figure 1.** (a) Experimental set-up, (b) Zinc nanoparticle colloid

The colloidal nanoparticle was then characterized by using Scanning Electron Microscopy (SEM-EDX, JEOL, JSM-6510 LA) to study the morphology of nanoparticles produced. The size of the nanoparticle is calculated from the photograph obtained by SEM using imageJ software. Surface plasma resonance of produced nanoparticle is obtained by using Ultraviolet-Visible light spectroscopy (UV-Vis, Spectroquant Pharo 300).

3. Results and Discussion
The colloidal ZnNPs produced in aquades is shown in figure 1(b). The colloidal solution shows the brownish color. The change of color from clear white (aquades) to light brown color indicates that the nanoparticle is successfully produced. The morphological photograph of produced ZnNPs is shown in figure 2(a). To obtain this photograph, 1 ml of produced ZnNPs was dripped on SiC sheet and then heated in the oven at 100°C for about 7 minutes.

![Zinc plate](image2)

It can be seen that ZnNPs was synthesized with various size distribution and mostly in spherical shape. The part marked with a red circle is an example of some produced spherical nanoparticle. Furthermore, nanoparticles size and size distribution are calculated by using SEM image utilizing imageJ software. The average diameter of ZnNPs is 35 nm with a standard deviation of 8.7 nm. The size distribution is shown in figure 2(b).

It should be noted that nanoparticle diameter is decreased at lower laser energy. The average diameter was 28 nm when the laser energy was reduced to 36 mJ. This trend is following the result from another researcher. Increasing the laser energy will increase the energy that bombarded the
sample, the ablated material becomes denser in the liquid environment. Because of that, the bigger nanoparticle can be formed either because of longer growth time or cluster aggregation [11].

![Figure 2. (a) Morphological photograph of produced ZnNPs, (b) Average diameter of ZnNPs](image)

To examine the purity of produced nanoparticle, EDX analysis was conducted at the surface of dried nanoparticle on SiC sheet. The EDX result is shown in figure 3. The spectrum shows that only Zn and O elements are detected on the prepared sample, while Si and C can be ignored because Si and C are basic materials of SiC sheet. It is also shown that the resulting colloidal nanoparticle forms zinc oxide molecules.

Because of the high reactivity of zinc particles, they tend to reacted and got oxidized by water and lead to the production of ZnO nanoparticle. The possibility of occurring reaction is shown in the scheme below [12]:

\[
\begin{align*}
\text{Zn (atoms/clusters)} + \text{H}_2\text{O} & = \text{Zn(OH)}_2 + \text{H}_2 \\
2\text{Zn(OH)}_2 & = 2\text{ZnO} + 2\text{H}_2\text{O}
\end{align*}
\]

![Figure 3. EDX spectrum of prepared ZnNPs on SiC sheet](image)

Figure 4 shows the absorption spectra of produced ZnNPs. The spectrum was obtained by using UV-Vis spectroscopy. The spectrum shows that the surface plasmon resonance (SPR) of the produced nanoparticle is located approximately at 300 nm. The single SPR peak indicates that the nanoparticle
is produced in a spherical shape. This result is following the result obtained by Khumaeni et al. [13], which shows spherical gold nanoparticle has single SPR peak.

![Figure 4. UV-Vis Spectrum of produced ZnNPs in aquadest](image)

### 4. Conclusion
The synthesis of high-purity ZnNPs by using pulse laser ablation method has been successfully conducted. The low energy Nd:YAG laser (1054 nm) was employed to produce colloid ZnNPs in aquadest. The morphological characteristic of produced ZnNPs was obtained by using SEM-EDX. From the photograph, spherical shape with the various size of ZnNPs was successfully synthesized. The average diameter of ZnNPs is 35 nm with standard deviation at 8.7 nm. The UV-Vis absorption spectrum shows that the surface plasmon resonance is centered approximately at 300 nm.

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