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Functional Group Analysis of Metal Surface-Modified Gold Nanoparticles by Pulsed Laser Ablation

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Abstract–In this work, the functional group of metal surface-modified gold nanoparticles by the Pulsed Laser Ablation in Liquid (PLAL) approach employing Nd:YAG laser (1064 nm) is described. The synthesis was carried out in distilled water and a 1 mM bismuth nitrate solution as the modifier source. A color change of the fluid indicating the creation of nanoparticles was seen during the ablation process. Characterization using Fourier Transform Infrared (FTIR) indicated the presence of Au–O vibrations at wavenumber 493 cm^{-1} in the pure gold sample, and the appearance of additional absorption bands such as Bi–O and Bi–O–Bi at wavenumber 557 cm^{-1} and 825 cm^{-1} in the Au+Bi_2O_3 sample. These variations in the FTIR spectrum reflect chemical interactions and success of surface-modified process. These results illustrate the efficiency of the PLAL approach for the manufacture of high-purity surface-modified gold nanoparticles without the usage of other chemicals.

Keywords – Pulsed laser ablation; gold nanoparticles; bismuth; Fourier Transform Infrared Spectroscopy.

I. INTRODUCTION

Metal nanoparticles are materials of nanoscale size and they exhibit unique physical and chemical properties as the effect of their particle size, shape and composition [1]. Among different various types of metal nanoparticles, gold nanoparticles (Au NPs) are one of the most extensively studied materials because to their high atomic number (Z=79), high chemical stability, excellent biocompatibility and unique optical properties in the form of surface plasmon resonance (SPR), which is sensitive to changes in the particle size and environment [2]. These properties make Au NPs promising for applications in biology, sensing, catalysis, imaging and photonics [3]. The performance of Au NPs can be improved by structural alteration by modified with other elements with high atomic numbers [4]. Bismuth (Bi) is one element that could be used as a modifier because of its high atomic number (Z = 83) and high electron density and comparatively moderate toxicity compared to other heavy metals [5]. It is known that the addition of bismuth to nanoparticles changes the electronic structure, parameters of the crystal lattice and optical properties of the material, which improves the physicochemical features of the resulting nanoparticles [6].

The preparation of nanoparticles might be by chemical or physical processes. Chemical approaches typically necessitate the use of further reducing agents and surfactants, which may result in residues on the nanoparticle surface [7]. Furthermore, the physical method called Pulsed Laser Ablation in Liquid (PLAL) has been studied extensively as it can generate high-purity nanoparticles without adding any other chemicals [8]. In the PLAL approach, a high energy laser pulse is focused on a metal target immersed in a liquid media to generate plasma and cavitation bubbles, which then surface-modified nanoparticles before rapidly cool to create nanoparticles [9]. The fast condensation process of the ablation when the cavitation-bubble/quenching dynamics — bubble lifetime

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~100–500 µs, peak plasma temperatures of several thousand kelvin, cooling rates of ~10^{10} K/s — strongly favour kinetic trapping of modifier, but the actual yield and uniformity of incorporation depend on the diffusivity of the modifier precursor in the cavitation bubble and on the timing of nucleation relative to bubble collapse. The liquid medium is modified with a modifier substance such as bismuth allows the inclusion of the modifier atoms in the crystal structure of the resultant gold nanoparticles [10]. Chemical interaction and functional groups can be proved by material characterisation, one of them is Fourier transform infrared (FTIR).

Fourier Transform Infrared (FTIR) is a characterisation technique that is utilized to detect functional groups and analyze chemical interactions in the nanoparticle materials [11]. FTIR is useful to identify chemical groups from oxide compounds and molecules adsorbed on the surface of nanoparticles in the metal surface-modified gold nanoparticle system [12]. Although in general gold is less active in FTIR as a pure metal, since it does not show any changes in dipole moment [13]. However, the presence of bismuth might give rise to distinctive absorption bands especially in the low wavenumber area associated with Bi–O bond vibrations [14]. Shifts in the position or strength of FTIR peaks may also suggest the chemical interactions or structural changes due to the modified process [15]. Therefore, FTIR is a significant supporting analytical technique to confirm chemical interactions and success of modified procedure in gold–bismuth nanoparticle systems.

In this study, the function of metal surface-modified gold nanoparticles utilizing PLAL technique with Nd:YAG laser is studied. The synthesized products were analyzed by Fourier Transform Infrared (FTIR) for the determination of functional groups and chemical interactions.

II. EXPERIMENTAL PROCEDURE

2.1 Materials and Characterization Methods

High purity (99.99%) Au plate internationally certified by the London Bullion Market Association (LBMA) with dimensions of

1.5x0.8 cm was employed as the starting material for synthesis. The plate came from PT Aneka Tambang Tbk (Antam), Jakarta, Indonesia. Bismuth nitrate (394.89 g/mol) was employed as modifier and supplied by Merck, Darmstadt, Germany. Additional supplies such as alcohol and distilled water were bought from commercial providers. Material characterisation was performed using glass slides as substrates to get functional groups from Fourier Transform Infrared Spectroscopy (FTIR) spectra with a Shimadzu IRSpirit-TX.

2.2 Preparation and Synthesis of Nanoparticle

The synthetic liquid medium was generated by preparing a stock solution. 0.08 g of bismuth nitrate was dissolved in 100 mL of distilled water and homogenized by a magnetic stirrer for about 6 hours till a solution with concentration of 2 mM was achieved. A solution with a concentration of 2 mM was initially prepared to reduce the error in weighing small amounts of bismuth nitrate. Then, the solution was diluted 1:1 (50 mL of 2 mM solution + 50 mL distilled water) to get a 1 mM solution with a volume of 100 mL. In the synthesis process distilled water was employed as a control synthesis medium. A Gold plate cut to approximately 1.5 x 0.8 cm was cleaned using 96% alcohol to remove contaminants, then rinsed with distilled water and dried with gauze. Next, the plate is placed in a beaker, and 10 mL of liquid medium is added, as shown in Figure 1.

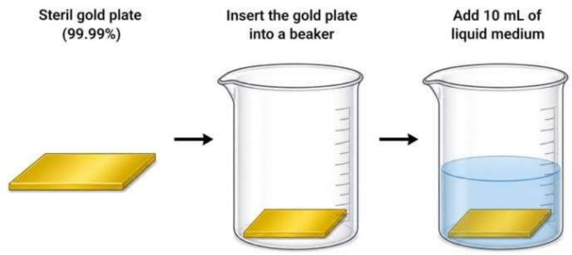

Fig. 1. Sample preparation.

Gold nanoparticles were prepared by pulsed laser ablation technique employing 1064 nm Nd:YAG laser with a pulse width of 7 ns, a Q-switch delay of 145 µs, a drive level of 80%, and a repetition rate of 10 Hz, producing an output energy of 83 mJ per pulse. The emitted beam was directed toward the target by a silver mirror placed at a 45° angle and specifically optimized for reflection at 1064 nm. The reflected beam was subsequently focused through a convex quartz lens with a focal length of 10 cm, resulting in a spot size of 50 µm in diameter. This configuration provided an estimated laser fluence of approximately 8 GW/cm² at the target.. The laser directed onto the metal surface from above to generate plasma and nanoparticles which are disseminated in the liquid medium as illustrated in Figure 2.

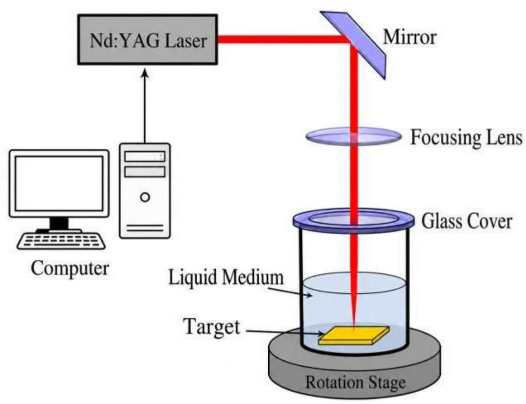

Fig. 2. Experimental set-up for the metal-gold nanoparticle production.

III. RESULTS AND DISCUSSION

3.1 Synthesis of Gold Nanoparticles in a Liquid Medium

Metal surface-modified gold nanoparticles were effectively produced utilizing Nd:YAG pulsed laser ablation technique. The production of the nanoparticles was detected by the change of color in the liquid medium during the synthesis process. Figure 3 presents two types of colloidal nanoparticle samples. Figure 3a presents the gold nanoparticle and distilled water as the reference (Au) and Figure 3b presents the gold nanoparticles modified with 1 mM of bismuth (Au+Bi). Visually, the hue of dark red of Au sample is brighter and more homogenous, whereas the sample modified with bismuth nitrate solution has a darker color [16]. This color change shows an interaction between the bismuth and the gold nanoparticles.

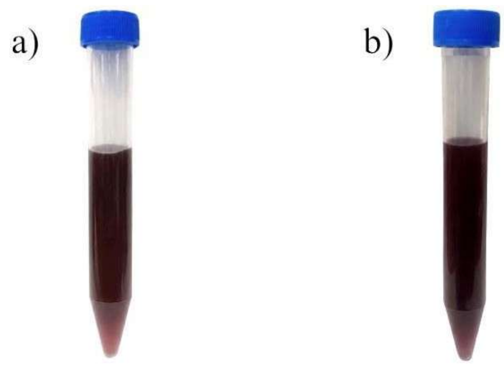

Fig. 3. Gold nanoparticles in medium liquid: (a) distilled water (b) 1 mM bismuth.

3.2 Characterization technique: Fourier Transform Infrared Spectroscopy (FTIR)

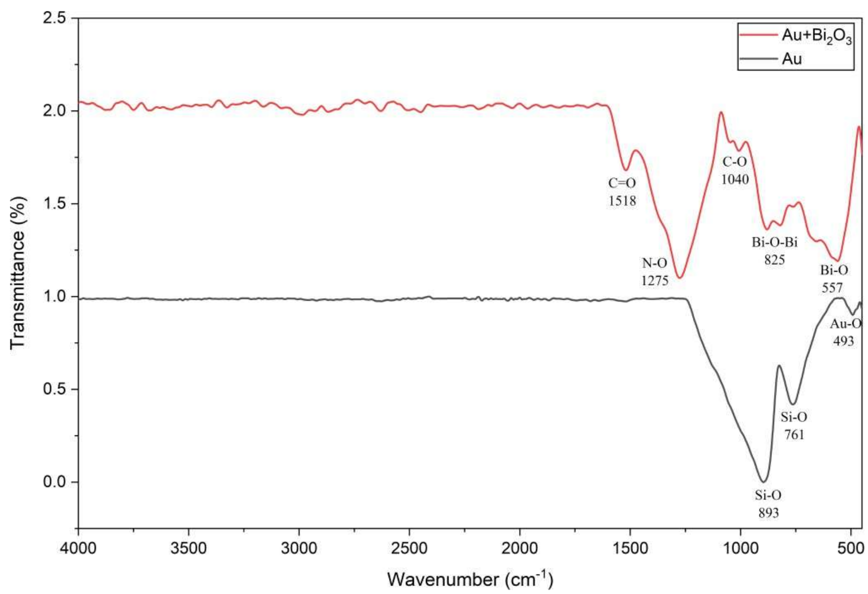

Fig. 4. FTIR spectra of gold produced in the distilled water and 1 mM bismuth (Au+Bi).

Based on Figure 4, the FTIR spectrum for the pure Au sample shows an absorption peak at a wavenumber of 760 cm^{-1}, indicating a correlation with Au-O bond vibrations, thus confirming the presence of gold nanoparticles. Theoretically, an absorption peak at a wavenumber of 493 cm^{-1} was also detected, indicating the presence of Au-O bond vibrations. Generally, this bond is found at a wavenumber of 500 cm^{-1} [17]. The peak absorption at a wavenumber of 761 cm^{-1} and 881 cm^{-1} corresponds to the Si-O bond vibration originating from the glass slide used as the sample's thin-film substrate [18]. Meanwhile, for the Au+Bi sample, an absorption peak appears at a wavenumber of 1275 cm^{-1} as an N-O bond vibration. This peak is associated with residual nitrate (NO_3^-) originating from the bismuth nitrate precursor used as the raw material for bismuth nitrate solution [19].

Next, an absorption peak appears at a wavenumber of 1040 cm^{-1} as a C-O bond vibration from the adsorption of atmospheric CO_2 during the thin-film drying process for FTIR testing in open air [20]. Subsequently, an absorption peak appears at a wavenumber of 825 cm^{-1} with a Bi-O-Bi bond vibration, indicating the presence of Bi from bismuth nitrate solution [21]. Then, another bismuth absorption peak appeared at a wavenumber of 557 cm^{-1} as a Bi-O bond vibration [19]. The appearance of this Bi absorption peak was around or overlap the wavenumber position in the pure Au nanoparticle sample, indicating the presence of chemical interaction between the Au nanoparticle surface and Bi ions as modifier [22]. Previous studies have also shown that Au can interact with metals such as Pt and Cu, allowing these metals to integrate into the gold structure and alter its electronic and catalytic properties [23].

IV. CONCLUSIONS

Based on the research results, functional group of metal surface-modified gold nanoparticles were successfully synthesized using the Pulsed Laser Ablation in Liquid (PLAL) method with an Nd:YAG laser. The color change of the solution during the synthesis process indicated the formation of nanoparticles and the presence of interactions between gold and bismuth. FTIR characterization results confirmed the presence of Au–O groups in pure gold nanoparticles as well as the appearance of additional absorption bands such as Bi–O and Bi–O–Bi in the modified samples, indicating the success of the modified process. Overall, bismuth modified affects the chemical structure and properties of the nanoparticles, and the PLAL method has proven to be an effective and environmentally friendly technique for the synthesis of modified metal nanoparticles.

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