

**Colorimetric Detection of Hg(II) Ion Using Silver Nanoparticles Capped with 3-Hydroxybenzoic Acid****Gusrizal Gusrizal<sup>1\*</sup>, Andi Hairil Alimuddin<sup>1</sup>, Ajuk Sapar<sup>1</sup>, Rizky Noviani Ridwan<sup>1</sup>,  
Sri Juari Santosa<sup>2</sup>**<sup>1</sup>Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Tanjungpura, Jl. Prof. Dr. H. Hadari Nawawi, Pontianak 78124, Indonesia<sup>2</sup>Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Gadjah Mada, Sekip Utara, PO BOX BLS 21 Yogyakarta 55281, Indonesia\*Corresponding author email: [gusrizal@chemistry.untan.ac.id](mailto:gusrizal@chemistry.untan.ac.id)**Received:** 23 Nov 2018; **Accepted:** 30 Jan 2019; **Available online** 5 Jun 2019

**ABSTRACT.** In this paper, we report for the first time the simple method for detection of Hg(II) ion in an aqueous medium using silver nanoparticles capped with 3-hydroxybenzoic acid. Silver nanoparticles capped with 3-hydroxybenzoic acid were prepared by reduction of the silver ion with 3-hydroxybenzoic acid and without surface functionalization. The formation of silver nanoparticles was characterized by a UV-Visible spectrophotometer. The yellow colloid of prepared silver nanoparticles capped with 3-hydroxybenzoic acid showed a surface plasmon resonance peak at 420 nm. The ability of silver nanoparticles capped with 3-hydroxybenzoic acid in detection was tested by doing a reaction with several metal ions individually (Na(I), K(I), Cu(II), Zn(II), Ca(II), Mg(II), Hg(II), Mn(II), Ni(II), Pb(II), and Co(II)). It was found that silver nanoparticles capped with 3-hydroxybenzoic acid highly selective to Hg(II) and then the prepared silver nanoparticles were developed for detection of Hg(II) ion. Addition of 1 mL Hg(II) ion into 4 mL the as-prepared silver nanoparticles reduced the peak of surface plasmon resonance spectra, and the reduction of peak intensity was proportional to the concentration of Hg(II) ion. A good linear relationship ( $R^2 = 0.998$ ) between absorbance at 420 nm and concentration of Hg(II) over the range  $1.0 \times 10^{-3}$  to  $5.5 \times 10^{-3}$  M was obtained. Silver nanoparticles capped with 3-hydroxybenzoic acid were highly sensitive to Hg(II) ion with the detection limit down to  $4.7 \times 10^{-5}$  M. Application the method to the real water sample showed the excellent result with recovery ranged from 98 to 101%. The proposed method was found to be useful for the colorimetric detection of Hg(II) ion in an aqueous medium.

**Keywords:** 3-hydroxybenzoic acid, colorimetry, Hg(II), nanoparticles, silver**INTRODUCTION**

Nanoparticles are generally defined as particles with one or more dimension at the nanoscale which size range typically between 1 to 100 nm. Particle in nanoscale size exhibit different properties compared to a bulk material with the same chemical composition (Lövestam et al., 2010). The properties include electronic, electrical, optical, magnetic, transport, photochemical, electrochemical, catalytic, and mechanical behavior (Ozin, 1992).

The optical properties of metal nanoparticles as gold and silver have been of interest in a variety of scientific field. Gold nanoparticles exhibit bright red and silver nanoparticles typically yellow (Kelly, Coronado, Zhao, & Schatz, 2003). The color is due to a phenomenon called surface plasmon resonance. Surface plasmon resonance results from the interaction of light with the mobile conduction electrons of the metal. The interaction causes the movement of mobile conduction electrons, and then the electron density in the particles is polarized that oscillates with the frequency of the exciting electric field. The resonance light absorption and scattering, named as surface plasmon resonance occur while the frequency of

light coincides with the frequency of electron (Krutyakov, Kudrinskiy, Olenin, & Lisichkin, 2008).

UV-Visible spectrophotometer has been used to measure surface plasmon resonance. A characteristic feature of absorption and scattering spectra of the metal nanoparticles is the presence of an intense and broad peak at the visible range. This band is called surface plasmon resonance band (Krutyakov et al., 2008). The surface plasmon resonance band of metal nanoparticles is dependent on the type of metal. Silver nanoparticles exhibit a peak at 420 nm and gold nanoparticles at 530 nm (Lee & Meisel, 1982). In addition to the type of metal, surface plasmon resonance band of metal nanoparticles also depends on the particle size and shape, interparticle distance, and dielectric properties of media (Eustis & El-sayed, 2006). The change of these factors as a result of the interaction of metal nanoparticles with analyte produces a different surface plasmon band. This phenomenon has been used as the basis to develop metal nanoparticles as a colorimetric sensor (Oliveira et al., 2015).

Silver nanoparticles have been developed as a colorimetric sensor. Silver nanoparticles have been used for the detection of metal ions (Ravindran et al., 2012;

Shrivasa, Sahu, Patra, Jaiswal, & Shankar, 2016) and nonmetals (Ma, Niu, & Cai, 2011; Sachdev et al., 2016). Aggregation of silver nanoparticles is used as the principal mechanism in the development of silver nanoparticles as a sensor. The presence of an analyte in colloid silver nanoparticles initiates the aggregation of particles and changes the surface plasmon band. Aggregation of nanoparticles is driven by electrostatic, hydrogen bond, van der Waals interaction and donor-acceptor reaction (Vilela, González, & Escarpa, 2012) and facilitated by capping agent (Ravindran et al., 2012; Shrivasa et al., 2016) or ligand used to modify the surface of nanoparticles (Ratnarathorn, Chailapakul, Henry, & Dungchai, 2012). Dissolution of nanoparticles results from oxidation of silver nanoparticles with an analyte which reduces the intensity of surface plasmon resonance band is also used as the mechanism in the detection of the analyte (Annadhasan, Muthukumarasamyvel, Babu, & Rajendiran, 2014).

Chemical reduction is the common method used by the chemist in the synthesis of silver nanoparticles. Reducing agent reduces the silver ion and resulted silver nanoparticles are stabilized by capping agent (Rycenga et al., 2011). Various reducing and capping agent have been used in synthesis silver nanoparticles. In the previous study, we have successfully prepared stable silver nanoparticles capped with m-hydroxybenzoic acid (Gusrizal, Santosa, Kunarti, & Rusdiarso, 2017, 2018). To the best of our knowledge, these nanoparticles have not been explored for metal detection. In this paper, we report the selectivity of silver nanoparticles capped with m-hydroxybenzoic acid toward metal ions and proposed a simple method for detection of Hg(II) ion using silver nanoparticle capped with m-hydroxybenzoic acid.

## EXPERIMENTAL SECTION

### Material and Instrumentation

In all the experiment, we used analytical grade chemicals as received without further purification. 3-hydroxybenzoic acid was obtained from Sigma-Aldrich, and other compounds such as NaOH, NaCl, KCl, CuCl<sub>2</sub>, Zn(NO<sub>3</sub>)<sub>2</sub>, CaCl<sub>2</sub>, FeCl<sub>3</sub>, MgCl<sub>2</sub>, HgCl<sub>2</sub>, MnSO<sub>2</sub>, NiCl<sub>2</sub>, PbCl<sub>2</sub>, CoCl<sub>2</sub>, KMnO<sub>4</sub>, and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> was purchased from Merck. Prepared silver nanoparticles were characterized with a UV-Visible spectrophotometer (Shimadzu UV-1280) using 1 cm cell.

### Synthesis of Silver Nanoparticles

Synthesis of 3-hydroxybenzoic acid was performed by the adoption of our previously published method (Gusrizal et al., 2017). The solution of silver nitrate (2.0x10<sup>-4</sup> M) was added to the pH 11 3-hydroxybenzoic acid (2.0x10<sup>-4</sup> M) with the mol ratio of 1:10. The mixture was then heated in a boiling water bath for 15 minutes and cooled in tap water after synthesis finished. The formation of silver nanoparticles was indicated by the appearance of yellow color in the solution and monitored using a UV-Visible spectrophotometer. The spectra were recorded in the range of 300-800 nm

### Selectivity Silver Nanoparticles toward Metal Ion

The ability of silver nanoparticles capped with 3-hydroxybenzoic acid as a colorimetric sensor for the

detection of metal ions was tested for each metal ion involving Na(I), K(I), Cu(II), Zn(II), Ca(II), Mg(II), Hg(II), Mn(II), Ni(II), Pb(II), and Co(II). A 1 mL of fixed concentration (1x10<sup>-2</sup> M) of each metal ion was added to 4 mL as-prepared silver nanoparticles. The spectra were then recorded using a UV-Visible spectrophotometer.

### Performance of Silver Nanoparticles as Colorimetric Sensor

The performance of silver nanoparticles capped with 3-hydroxybenzoic as a colorimetric sensor for Hg(II) ion detection was determined by measuring linear range, the limit of detection, the limit of quantitation, precision, and accuracy (Harris, 2010).

## RESULTS AND DISCUSSION

### Synthesis of Silver Nanoparticles

Silver nanoparticles capped with 3-hydroxybenzoic acid were prepared by reduction of 2.0x10<sup>-4</sup> M silver nitrate with 2.0x10<sup>-3</sup> M 3-hydroxybenzoic acid. The mole ratio of silver nitrate and 3-hydroxybenzoic acid use in the reaction was 1:10. After the reaction, the color of the solution changed from colorless to yellow. UV-Visible spectrophotometer measurement resulted in the spectra exhibited the maximum peak at 420 nm, as shown in **Figure 1**. The yellow color of the solution and a band of UV-Visible spectra results from a surface plasmon resonance of silver nanoparticles (Eustis & El-sayed, 2006; Krutyakov et al., 2008). The result indicated that silver nanoparticles successfully synthesized (Lee & Meisel, 1982; Tejamaya, Römer, Merrifield, & Lead, 2012). The ability of 3-hydroxybenzoic acid as a reducing agent in the formation of silver nanoparticles has been proved by transmission electron microscope and published in our previous paper (Gusrizal et al., 2017). Several published data show that the position of maximum peak varies with the reducing and capping agent used in the synthesis of silver nanoparticles. The reducing and capping agent determine the size and shape of silver nanoparticles (Rycenga et al., 2011) and the position of the maximum peak is dependent on the size and shape of nanoparticles (Mock, Barbic, Smith, Schultz, & Schultz, 2002). The bathochromic effect occurs for the bigger particles. Lee and Meisel (1982) synthesized silver nanoparticles with two types of reducing agent. Synthesis using sodium borohydride and polyvinyl alcohol resulted in silver nanoparticles with the position of the maximum peak at 400 nm and silver nanoparticles synthesized using sodium citrate showed a peak at 420 nm. Although synthesized with the same reducing agent, silver nanoparticles synthesized with different capping agent also have different size. Silver nanoparticles capped with sodium citrate, polyethylene glycol, and polyvinylpyrrolidone show the maximum peak at 390, 393, and 400 nm, respectively (Tejamaya et al., 2012).

Our previous report shows that silver nanoparticles capped with 3-hydroxybenzoic acid show high stability for long time storage (Gusrizal et al., 2018). In addition to acting as a reducing agent, 3-hydroxybenzoic acid also plays a role as a capping agent on the surface of silver nanoparticles.

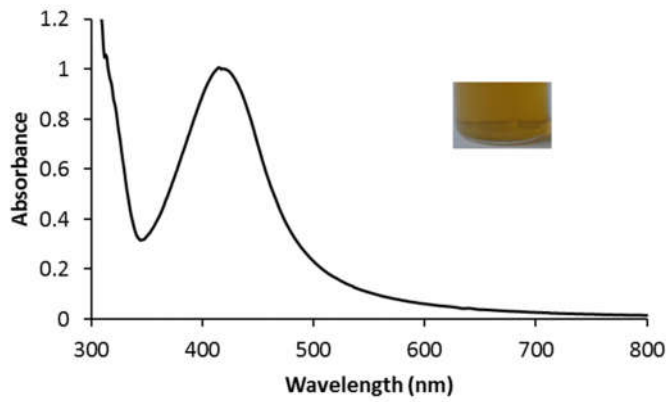


Figure 1. UV-Visible spectra of silver nanoparticles capped with 3-hydroxybenzoic acid

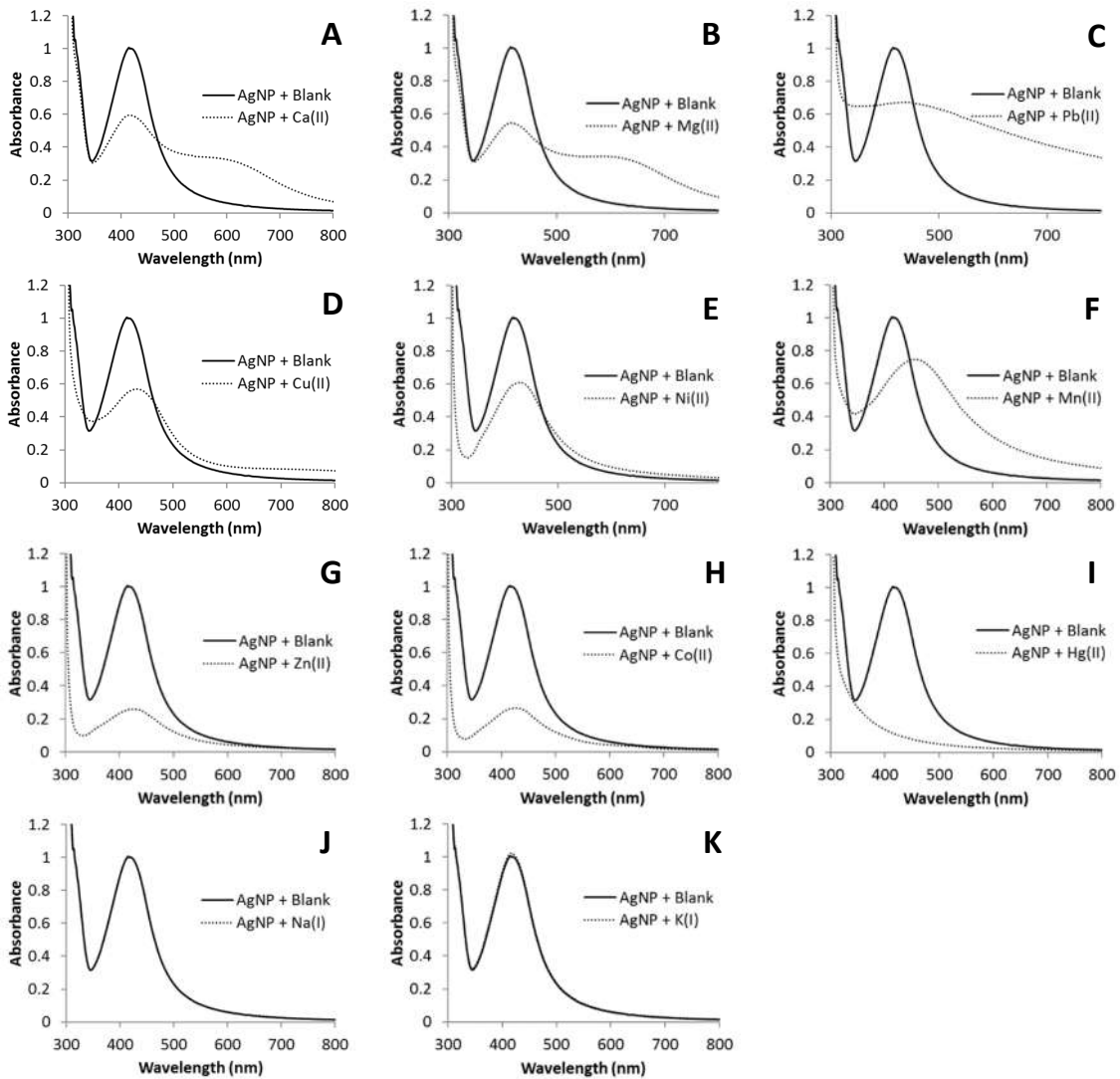


Figure 2. UV-Visible spectra of silver nanoparticles capped with 3-hydroxybenzoic acid after addition of different metal ions

The functional groups of 3-hydroxybenzoic acid can facilitate the interaction of silver nanoparticles capped with 3-hydroxybenzoic acid with metal ions.

### The selectivity of Silver Nanoparticles towards Metal Ion

The selectivity of silver nanoparticles capped with 3-hydroxybenzoic acid towards metal ion was determined by addition of 1 mL  $1.0 \times 10^{-2}$  M different metal ions into 4 mL as prepared silver nanoparticles. The interaction of silver nanoparticles with various metal ions was monitored by UV-Visible spectrophotometry measurement, and the change of surface plasmon resonance spectra is shown in **Figure 2**.

Addition of Ca(II), Mg(II), and Pb(II) ion to silver nanoparticles caused the decrease of peak intensity at 420 nm and followed simultaneously by the appearance of a secondary peak around 600 nm (**Figure 2: A, B, C**). The bathochromic shift and reduction of peak intensity also happened to the silver nanoparticles mixed with Cu(II), Ni(II), Mn(II), Zn(II), and Co(II) ion (**Figure 2: D,E,F,G,H**). The change of surface plasmon resonance spectra resulted from the aggregation of silver nanoparticles after the addition of metal ions (Wei *et al.*, 2015; Jin *et al.*, 2015). However, the addition of Na(I) and K(I) resulted in no significant change in the surface plasmon resonance spectra (**Figure 2: J, K**). The other remarkable change was observed when Hg(II) ion was added to silver nanoparticles (**Figure 2 I**). The peak was disappeared, and the color of the solution changed from yellow to colorless.

The change of surface plasmon resonance spectra is the base for the development of the silver nanoparticles-based colorimetric sensor. The change of spectra results from the interaction of silver nanoparticles with the analyte and proportional to the concentration of the analyte (Vilela, 2012). From **Figure 2**, it can be observed the selectivity of silver nanoparticles capped with 3-hydroxybenzoic acid towards Hg(II) ion. Furthermore, we explored the performance of silver nanoparticles capped

with 3-hydroxybenzoic acid as a colorimetric sensor for Hg(II) ion in an aqueous medium.

### Performance of Silver Nanoparticles for Detection of Hg(II) ion

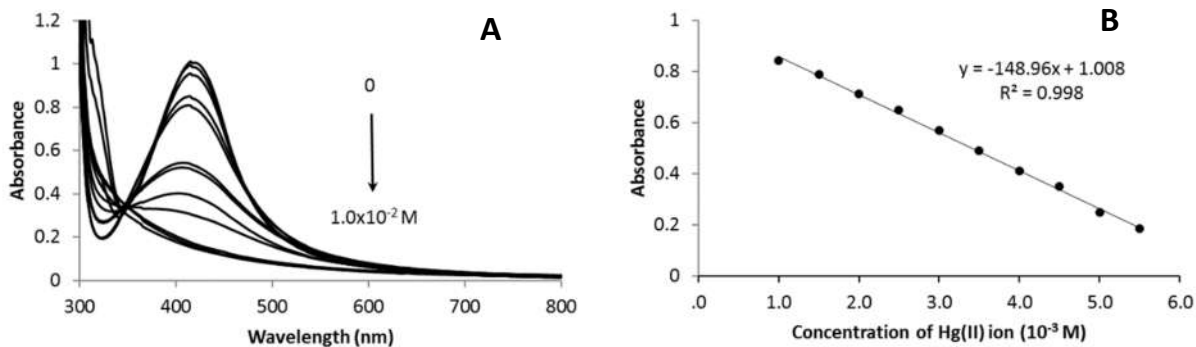
The performance of silver nanoparticles capped with 3-hydroxybenzoic acid for the detection of Hg(II) ion was evaluated using the parameter of linearity, limit detection (LOD), limit of quantitation (LOQ), precision, and accuracy or recovery when the method was applied to the real sample (Harris, 2010).

#### Linearity

The quantitative assessment of silver nanoparticles in Hg(II) ion detection was studied by observing the relationship between the peak intensity of silver nanoparticles and concentration of Hg(II) ion. A 1 mL of Hg(II) ion with different concentration ( $5.0 \times 10^{-4}$  to  $1.0 \times 10^{-2}$  M) was added to 4 mL as prepared silver nanoparticles. The change of surface plasmon resonance spectra was monitored by UV-Visible spectrophotometer and the results are shown in **Figure 3A**. The addition of Hg(II) ion to silver nanoparticles gradually decreased the peak intensity of silver nanoparticles spectra with the concentration of Hg(II) ion. The change of peak intensity was linear with the concentration of Hg(II) ion ranging from  $1.0$ - $5.5 \times 10^{-3}$  M, and the linear regression coefficient ( $R^2$ ) was 0.998, as shown in **Figure 3B**.

#### LOD and LOQ

Limit of detection (LOD) and limit of quantitation (LOQ) were determined by measurement of blank in seven replications. The standard deviation of blank sample measurement was used to determine the LOD and LOQ. LOD and LOQ are defined as  $3m/s$ , and  $10m/s$ , respectively, where  $s$  is the standard deviation of blank sample measurement and  $m$  is the slope of the calibration curve. It was found that LOD and LOQ were  $4.7 \times 10^{-5}$  and  $1. \times 10^{-4}$  M, respectively.



**Figure 3.** UV-Visible spectra of silver nanoparticles capped with 3-hydroxybenzoic acid after addition of different concentration of Hg(II) ion (**A**) and the relationship between the absorbance of silver nanoparticles and the concentration of Hg(II) ion (**B**)

Table 1. The performance of silver nanoparticles in the detection of Hg(II) ion.

Type of silver nanoparticles	Analytical performance	Reference
Silver nanoparticles were synthesized using L-tyrosin as reducing and capping agent	Limit of detection: 16 nm Linear range concentration: 16-660 nM	Annadhasan et al., 2014
Silver nanoparticles were synthesized using N-(2-hydroxybenzyl)-valine as reducing and capping agent	Detection of Hg(II) at the ppm level	Kumar & Anthony, 2014
Silver nanoparticles were synthesized using glucose as a reducing agent and gelatine as a capping agent	Limit of detection: 0.45 mg/L %RSD 1.97 %recovery: 88.00-92.86%	(Sulistiawaty et al., 2015)
Silver nanoparticles were synthesized using glucose as a reducing agent and Tween-20 as a capping agent	Limit of detection: 0.45 mg/L %RSD 0.23 %recovery: 95.95-97.67%	Sulistiawaty et al., 2015
Silver nanoparticles were synthesized using the extract of <i>Syzygium aqueum</i> as reducing agent	Limit of detection: $8.5 \times 10^{-7}$ M Precision 5%	(Firdaus et al., 2017)

#### Precision

Precision shows the closeness of the result obtained from the replicate measurement and usually expressed as the standard of deviation and coefficient of variation or percent relative standard deviation (%RSD). The precision was determined by a replicate measurement of  $3.5 \times 10^{-3}$  M Hg(II) ion and found the standard of deviation and %RSD was  $8.8 \times 10^{-5}$  M and 2.0%, respectively.

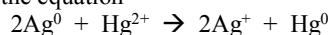
#### Experiment with the real sample

The applicability of silver nanoparticles capped with 3-hydroxybenzoic acid in the detection of Hg(II) was tested with a real water sample. The real sample was taken from the Kapuas River. The sample was firstly filtered using Whatman 40 filter paper. Recovery test was performed by spiking 100 mL of the water sample with 1 mL of 0.2 M standard solution of Hg(II). Using seven replications experiment, the recovery value obtained varied from 98 to 101% indicating that silver nanoparticles capped with 3-hydroxybenzoic acid could be used in the detection of Hg(II) ion in the real sample.

Several papers have reported the application of silver nanoparticles in colorimetric detection of Hg(II) ion with little variation in analytical performance, as shown in **Table 1**. The limit of detection produced by the measurement using silver nanoparticles capped with 3-hydroxybenzoic acid is comparable with the previously proposed method using silver nanoparticles capped with gelatin, Tween-20 (Sulistiawaty, Sugiarti, & Darmawan, 2015), and N-(2-hydroxybenzyl)-valine (Kumar & Anthony, 2014). It is predicted that the capping agent used to stabilize the silver nanoparticles affects the performance of silver nanoparticles in the detection of Hg(II) ion.

#### Mechanism of Hg(II) Ion Detection Using Silver Nanoparticles

Addition of Hg(II) ion into silver nanoparticles capped with 3-hydroxybenzoic acid resulted in the reduction of the peak intensity of surface plasmon resonance spectra and followed by reduction color intensity of silver nanoparticles to colorless. It is supposed that the dissolution of silver nanoparticles occurs. The dissolution could be due to the redox reaction between silver nanoparticles with Hg(II) ion and supported by the standard reduction potential of Ag(I) and Hg(II). Standard potential of Ag(I) is 0,7996 V whereas that of Hg(II) is 0,851 V (Lide, 2010). Silver nanoparticles with zero-valent Ag were oxidized to form Ag(I), and Hg(II) ion was reduced to Hg atom as shown in the equation



The formation of Hg atom was confirmed by the appearance of absorption peak measured with atomic absorption spectrophotometer using mercury lamp. This mechanism is similar to the previously proposed mechanism (Annadhasan et al., 2014).

#### CONCLUSIONS

The method for detection of Hg(II) ion using silver nanoparticles capped with 3-hydroxybenzoic acid was developed. Addition of Hg(II) ion into as prepared silver nanoparticles changed the yellow color of silver nanoparticles to colorless linearly with the increase of Hg(II) concentration over the range of  $1.0 \times 10^{-3}$  to  $5.5 \times 10^{-3}$  M. The color change of silver nanoparticles was due to the redox reaction between silver nanoparticles with Hg(II) resulted in the dissolution of silver nanoparticles. Performance test of the method showed that limit detection of the method was found to be  $4.7 \times 10^{-5}$  M with %RSD 2.0%. The method has been

successfully applied for the determination of Hg(II) in a river water sample.

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