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## Silver Nanoparticle-based Colorimetric Sensors for Metal Ions Detection: A Narrative Review

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#### ABSTRACT

Silver nanoparticle (AgNP) has a distinctive optical characteristic, that is strong surface plasmon resonance (SPR) absorption at visible wavelength. Aggregation or morphological changes of the nanoparticles will shift the SPR band to a longer wavelength. Then, this shifting can be applied as the basis of AgNPs-based colorimetric sensors. AgNPs can be applied as sensor probes in a colloidal form or immobilized on paper as a paper-based analytical device (PAD). PAD is a simplified analytical instrument for an easy, inexpensive, and portable sensing application. This article provides an up-to-date overview of the application of AgNPs as metal ion sensors. We review the properties and potential of AgNPs as a colorimetric sensor probe of metal ions. In addition, we present the sensor probe in the form of colloidal nanoparticles as well as a paper-based analytical device. We also discuss the analytical performance of AgNPs in detecting various metal ions.

#### I. INTRODUCTION

In many applications today, sensors play an important role, spanning from biomedical to safety devices, where they detect and alert us about environmental alterations[1]–[6]. The advancement in technology makes the sensor devices become smaller, quicker, smarter, portable, more sensitive, and easier to use. One of the major types of sensors is chemical sensors, which detect a particular analyte in a liquid, gas, or solid phase[7].

Nanoparticles, characterized by a large specific surface area and versatility to be functionalized are the ideal material for sensitive, selective, fast response time, and portable design of sensors. With these features, sensors based on nanoparticles cannot only be applied in high-end detection of analytes, but also for private and everyday usage.

Methods for metal ions measurement are generally carried out through tedious processes, and complex equipment, such as Atomic Absorption Spectroscopy (AAS), Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) dan UV-Visible spectrometry[8]–[12]. Therefore, alternative measurement methods that are easier, and more practical need to be developed. Such an alternative method is a nanoparticles-based colorimetric sensor. The colorimetric method is based on observing color or change in color, so that the measurement result can be observed directly by naked eye.

The principle of colorimetry based on metal nanoparticles is a change in the optical properties of metal nanoparticles due to changes in morphology or changes in disperse-aggregation form. The optical property referred to is surface plasmon resonance (SPR). When the nanoparticles aggregate, it will induce the integration of interparticle plasmons thus inducing a shift of SPR absorption band. This absorption shift causes color changes that can be observed visually or with the aid of a visible spectrophotometer. One of the things that causes aggregation is its interaction with metal ions. Thus, metal nanoparticles can be applied to detect the presence of metal ions by observing color change or shifting the SPR absorption band.

Paper is a versatile, porous, and environmentally friendly medium for the fabrication of competitive analytical instruments for fast detection of relevant analytes. Paper-based analytical devices (PADs) offer inexpensive devices to deteg analytes reliably, accurately, and rapidly. Paper-based devices provide an affordable technology [3] create easy and compact analytical instruments that can be extremely applicable in resource-limited environment, such as in remote areas or augere environments, where there is a shortage of fully equipped facilities and highly trained persorel. In PADs, the identification of analytes from a sample is enabled by a chemical reaction that causes a change in color, electrochemical properties and absorption or emission of light as an analytical signal. The most frequently used detection method among these techniques is based on colorimetric transition. Results can be measured simply by producing a color product generated by chemical or physical processes that can be quantified using low-priced scanners, cameras, or mobile phones[2].

Silver nanoparticles (AgNPs) is one of the most studied nanoparticles. AgNPs has a distinctive SPR absorption in the visible wavelength region [13]. In addition, AgNPs also have a high extinction coefficient, even higher than gold nanoparticles (AuNPs) of the same size [14]. When silver nanoparticles aggregate, the aggregates will induce the incorporation of interparticle plasmons and the SPR band will widen and shift to the longer wavelength. This effect can be used as an easy, sensitive, and inexpensive sensor probe.

This review article discusses the properties and potential of AgNPs as colorimetric sensor probe of metal ions. We present the sensor probe in a paper-based device that is versatile, inexpensive,

portable, and easy to dispose. In addition, we introduce the evaluation of the analytes concentration by digitalized the color intensity to quantify the analytes.

#### II. SILVER NANOPARTICLES

Nanoparticles has a tiny size, ranging from 1 to 100 nm, so that they have different optical and electronic properties compare to their bulk material. This difference in properties is due to their quantum and surface effects. This allows control over the properties of the nano-sized materials as needed by varying their size and geometric shape [15].

Compared to AuNPs, AgNPs are much cheaper and demonstrate a greater absorption of surface plasmon resonance with a high extinction coefficient [1], [16], [17]. It is helpful for the extremely sensitive of sensor arrays, resulting in naked eyes reading easily.

SPR is a characteristic of noble metal nanoparticles. It is related to conduction of electrons at the surface of the nanoparticles when visible light interacts with them. The interaction of analyte and the nanopaticles, however, induces particles aggregation and results in the color change and shift of SPR band to longer wavelength. Thus, low concentration of analytes which exist in various sample types can be identified by SPR-based probe [18], [19].

AgNPs can be synthesized by various methods. It includes chemical reduction, sonochemistry, photochemistry, electrochemistry, microwave, and coprecipitation [20], [21]. In the chemical reduction process, Ag<sup>+</sup> from precursor materials such as silver nitrate, silver acetate and silver citrate is reduced to Ag<sup>0</sup> by a reducing agent such as sodium borohydride (NaBH<sub>4</sub>), sodium citrate (Na<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>), hydrazine (N<sub>2</sub>H<sub>4</sub>), ascorbic acid (C<sub>6</sub>H<sub>8</sub>O<sub>6</sub>) and others [22]–[24]. In addition, more eco-friendly processes are also being researched and developed, which is by applying biomass-based natural reducing agents. Biomass extracts containing phenolic compounds can play a role in the reduction process [13], [25].

Identification and characterization of AgNPs can be carried out by several methods, including UV-Visible spectroscopy to identify the SPR absorption band, Dynamic Light Scattering (DLS)

to identify the size distribution and stability of colloidal AgNPs, and Fourier Transform Infra-red (FTIR) spectroscopy to determine functional groups or organic compounds accompanying AgNPs. In addition, the X-ray diffraction (XRD) method can be used to identify the shape, size, and crystal structure of AgNPs, while Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) are used to determine the AgNPs size and shape [21].

The characteristic SPR absorption band of AgNPs appears at 400-460 nm (Fig. 1) [1]–[4], [6], [26]. This can be used to easily mark the presence of AgNPs. The SPR absorption also provides a distinctive color for the colloidal AgNPs as pale yellow. Thus, the presence of AgNPs can be easily identified visually with the naked eye.

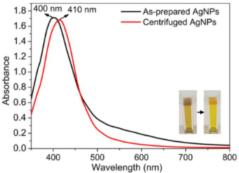
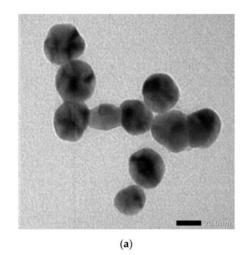


Fig 1. UV-vis spectrum and color of colloidal AgNPs [27].

Most of the studies show that AgNPs are spherical, but some have reported other morphologies including cubical, plate, hexagonal, triangular, flower, and rod structures [16], [21]. Meanwhile, the crystalline form of AgNPs is mostly face centered cubic (fcc) as widely reported based on XRD studies (Fig. 2) [28], [29]. Only a few shows cubic and hexagonal structures.



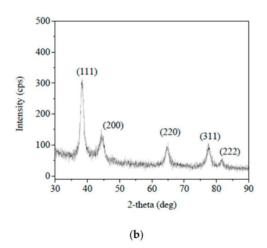


Fig 2. a) TEM image of colloidal AgNPs showing a spherical shape, b) XRD pattern of AgNPs showing a face-centered cubic crystal system [30].

#### III. AGNPS AS COLORIMETRIC SENSOR PROBE

AgNPs in aqueous phase (colloid) can be directly used as sensor probes [27], [28], [30]. Detection is performed by examining the color change or SPR absorption shift of the colloid system. This has been applied to various analytes, such as metal ions [13], [31], [32], antibodies[33], amino acids [34], and pesticides [35], [36]. The detection is carried out by adding sample solution into the aqueous AgNPs, then the mixture was stirred gently before observing the color change or recording its SPR spectrum.

Another method of colorimetric detection that has been widely developed is by applying paperbased analytical devices (PADs). PADs have recently been utilized as a miniaturized analytical instrument for an inexpensive and compact application. Due to flexibility, disposability, low sample-reagent use and suitability for routine measurements, paper-based sensors can be beneficial for many applications in medical, industrial, and environmental monitoring [5], [37]–[39]. With PADs, color change generated from the chemical reaction between a particular analyte and colorimetric reagent can easily be observed by a digital camera, a smartphone, or even by naked eye.

The key benefits of the paper comprise high surface-to-volume ratio, capillary action, adsorption properties, biological sample compatibility, and easy disposal by incineration [37], [40]. Moreover, paper is lightweight and globally available [41].

The basic of fabricating PADs is by building a pattern on paper that constituting hydrophobic walls of mili- or micrometer size to guide the flow. To achieve this purpose, several approaches have been proposed, such as wax printing, photolithography, inkjet printing, hot embossing, and others [42], [43]. On the patterned paper, AgNPs was then deposited onto the non-hydrophobic pattern followed by the sample solution. The color developed was examined visually or recorded using camera or smartphone for more quantitative analysis.

The color change of AgNPs in colorimetric detection can be rapidly visualized through the naked eye. But, for more quantitative purpose, the color change can be evaluated by using a color meter or by digitalized the color product. With color meter, the color intensity can be measured in L\*a\*b\* unit, adopted from CIE (Commission Internationale d'Eclairage) [44]. The total color change can be expressed by  $\Delta E^*$  which is a single value as the difference in L\*, a\*, and b\* values between the sample and a standard. Color can also be digitalized using a camera or cellphone, then the color intensity of resulting image is read by a color analysis software [39]. Furthermore, the results can be quantitated with a standard series (Figure 3).

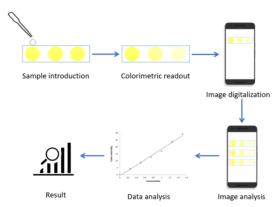


Fig 3. Colorimetric detection process using paper-based analytical device

A paper-based for selective detection of Pb(II) been developed using a plasmonic colorimetric sensing strategy [45]. The probe was based on polyvinyl alcohol (PVA) podified AgNPs. The linear range of the detection was 50-1000 μg.L<sup>-1</sup> with 2 limit of detection (LoD) of 20 μg.L<sup>-1</sup>. A paper impregnated with AgNPs/CTAB has also been fabricated for handy measuremennt of Fe(III) from water and blood plasma samples Another PAD was developed [45]. Ratnarathorn et al. using homocysteine and dithiothreitol modified AgNPs for quantitative detection of Cu(II). The analysis was based on the formation of coordination complex between Cu(II) and capping agent that induced the color change of NPs on the paper substratum surface [46]. Apilux et al. developed the use of paper-based sensor for determination of Hg(II) in real water samples. The determination was based on the color shift of silver nanoplate after the injection of sample on the paper substrate [17].

#### IV. AGNPS-BASED METAL IONS SENSOR

In AgNPs-based sensors, shape and size of the particles can affect the sensor's performance. Spherical particles and smaller size will have a larger surface area. As a sensor, the surface of the nanoparticles will interact with the analyte and cause changes on their properties in response to the presence of the interest analyte [47]. Thus, a sensor made of small spherical nanoparticles will provide better sensitivity and selectivity.

In its application as a sensor, gott sensor characteristics, such as response time, selectivity, sensitivity, robustness, accuracy, precision, and linearity must be achieved. The linearity of a method can be determined by constructing a graph showing a linear relationship of concentration and an analytical signal. The selectivity of a sensor indicates its ability to detect the presence of a particular analyte among the sample matrices. Meanwhile, the property of sensors to detect analytes at the lowest concentration to called sensitivity, usually expressed as Limit of

Detection (LoD) and Limit of Quantitation (LoQ) [48].

The analytical characteristics of AgNPs-based colorimetric detection for metallic ions are varied (Table 1). So far, the best limit detection was achieved on the analysis of Pb(II) using PVA-stabilized AgNPs, with a LoD of about 0.64 μg.L<sup>-1</sup>. In general, the AgNPs-based method can detect metal ions up to μg.L<sup>-1</sup> level. This is comparable to the established methods, such as AAS and ICP-MS, even better than UV-Vis spectrometry which offers a LoD in the range of a few mg.L<sup>-1</sup>.

 $\label{table 1} Table~1$  AgNPs-based metal ion sensors and their analytical characteristics

Probe	Metal ion	Linear range	LoD	LoQ	Ref.
		(μg.L <sup>-1</sup> )	(μg.L <sup>-1</sup> )	(μg.L <sup>-1</sup> )	
Casein peptide-functionalized	Cu(II)	5.08-91.50	10.17	-	[27]
AgNPs					
AgNPs nanocolloid	Fe(III)	335-558	116	-	[28]
Epicatechin-capped AgNPs	Pb(II)	207 – 20700	314.6	1558.7	[49]
PVA-modified AgNPs	Pb(II)	200-1000	8	-	[31]
nanocolloid					
PVA-modified AgNPs PAD	Pb(II)	50-1000	20	-	[31]
PVA-stabilized AgNPs	Pb(II)-	0.5-10	0.64	2.1	[30]
nanocolloid	dithizone				
	complex				
Reduced graphene oxide-	Cr(IV)	5.2-1300	1.6		[35]
wrapped AgNPs					
AgNPs/CTAB Paper-based	Fe(III)	50-900	20	-	[32]
Carbon dots-modified AgNPs	Cu(II)	19-508	24	-	[50]
2-aminopyrimidine-4,6-diol	Hg(II)	0-13038	70.2	-	[51]
functionalized AgNPs					

#### V. CONCLUSIONS

AgNPs-based metal ion sensors have been developed both in the form of nanocolloids and PADs. The detection of the AgNPs-based sensors is based on the color change or shift of the SPR absorption band. The sensors exhibit good analytical performance. In general, their sensitivity is comparable to the established methods, which is in the  $\mu$ g.L<sup>-1</sup> level. Therefore, this can be applied as an alternative measurement method that is simple, rapid, cost-effective, and portable.

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