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Development of a simple method for sensing melamine by SERS effect of Ag particles

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ABSTRACT

A simple and inexpensive procedure for melamine sensing based on surface-enhanced Raman scattering (SERS) of Ag particles was developed. The controllable shape of Ag particles was performed using co-precipitate at room temperature without surfactant. SERS signals were obtained due to the melamine were directly adsorbed on Ag particles without substrate. The enhancement factor of SERS effect depended on the architectural form of Ag particles. Among the three types of Ag nanostructures, the raspberry-like Ag sample displayed the strongest signal. The proposed probe exhibits great potential for melamine detecting and good reproducibility (the relative standard deviation (RSD) = 3.7%).

1. Introduction

Recently, among the vibration spectroscopy, surface-enhanced Raman scattering (SERS) offers extremely low limited of detection and is capable of qualitative and quantitative analysis. SERS has progressed rapidly during the past decade as a potential technique for trace detection due to cost effective and non-destructive detection. There are the two primary mechanisms for the enhancement effect of SERS involving chemical and electromagnetic theories were proposed. For the chemical theory, it is an enhancement of charge transfer between a target and a surface of metal [1,2]. By contrast, the electromagnetic theory proposes the enhancement of local electromagnetic that originates from the excitation of the collective oscillation of free electron [3,4]. Based on the electromagnetic and chemical enhancement effects of the noble metal (e.g. gold, silver or copper), the scattering cross section of target molecules locating on or near the surface of the metal can be enhanced. Therefore, the target molecules can be detected via the enhanced signals. As a powerful tool, the SERS technique has been widely applied in various fields, such as analytical chemistry, environmental science, and life science [5–10].

There are several main classes of SERS substrates, such as rough surfaces, nanoparticles colloids, and periodic nanostructures [11–14]. Many methods have been used to synthesize the silver particles, such as salt reduction [15], template method [16,17], and deposition technique [18]. Among of them, the salt reduction approach is the most extensively method for the SERS application. By this method, Ag particles can be easily, inexpensively fabricated with high reproducibility. Ag particles are often produced with special forms, such as either rough surface or stranger shape due to using a surfactant, such as poly (vinyl pyrrolidone) [11,19], or pyramids form [20]. In order to obtain high enhance factor (EF) of SERS, some new techniques were reported. For example, Au- integrated core-shell composite molecularly imprinting polymer (MIP), Ag@MIPcore@shell hybrid [21,22], silver on gold coated microarray with MIP solid phase extraction [23,24]. Gao et al. [25] used MIP as a stationary phase for thin layer chromatography (TLC) plate, which was deposited Au. For increase selectivity of SERS sensor, a combination of two-dimensional correlation spectroscopy and TLC-SERS was proposed [26]. By using the experiments and simulations, Hao et al. [27] reported that SERS signal of a metal nanoparticle-nanowire structure was improved due to existence of the adjacent part of the nanoparticle-nanowire. However, these approaches require complicated procedures, such as MIP process, etching step.

Melamine became a toxicity issue in 2008 when baby milk products were found an amount of melamine. This resulted in the death of six infants and the hospitalization of several ten thousands persons for kidney problems. In addition, melamine is widely used in fire-resistance plastics, cleansing agent, food contamination. Therefore, the detection of melamine has been emerged. The most methods reported are based on gas chromatography/mass spectrometry [28], capillary electrophoresis [29], molecularly imprinting polymer [30], colorimetric [31,32]. Li et al. [33] proposed to use a poly(styrence-*co*-acrylic acid)@Ag@ polyvinylpyrrolidone core@shell nanospheres as a SERS substrate.

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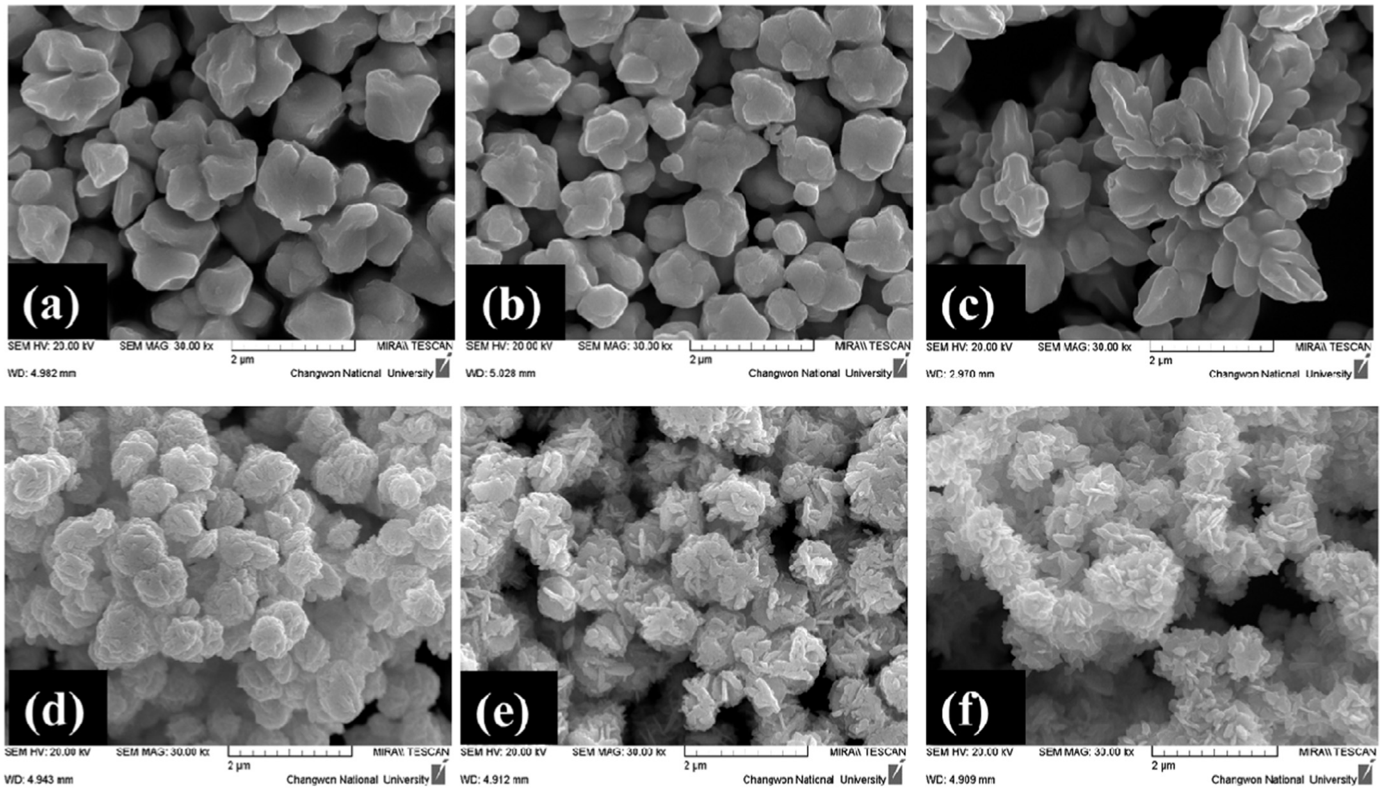

Fig. 1. SEM images of the prepared samples with different ratios between AA and $AgNO_3$: Shurikent-like with AA:Ag^+ = 1.1 (a), 3:1 (b), (c) Flower-like with AA: Ag^+ = 4:1 in case of using nitric acid; Raspberry-like with AA: Ag^+ = 1:1 (d), 3:1 (e), and 4:1 (f) in case of using citric acid.

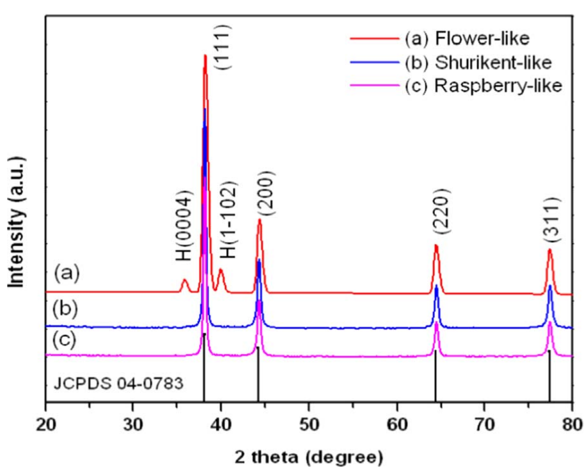

Fig. 2. XRD patterns of Ag samples with different forms.

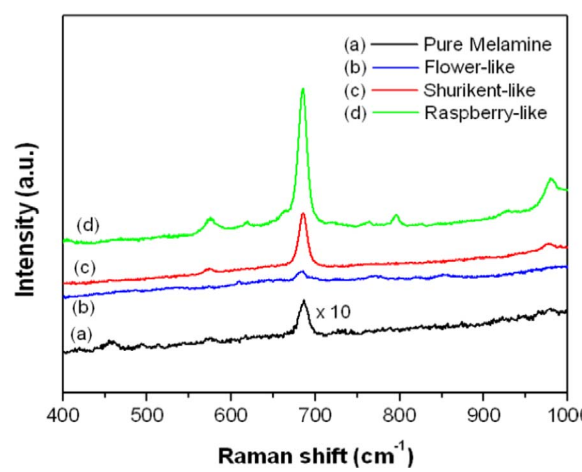

Fig. 3. Effect of morphology of Ag particles on SERS from melamine.

The three dimensional hierarchical nanostructure of Ag-decorated ZnO rods on Au array for detecting melamine was reported [34]. Due to a super-hydrophobic-oleophobic 3D Ag nanowire platform, the ultra-trace detection of melamine was reported [35]. Wang's group designed a microfluidic SERS chip based on an ITO modified with Ag-Au nanocomposites for the determination of melamine [36]. However, most of existing methods are noted for labor intensive, time-consuming procedures, and costly instrumentation requirements. Betz's group used dendritic, polygonal Ag structure, which were obtained by using galvanic displacement technique, for detecting melamine without additional sample preparation or purification [37]. However, the obtained Ag amount was small quantity. Therefore, a development of an efficient analytical and method for detecting melamine is highly desirable.

In this report, a simple procedure for the sensing melamine based on the SERS effect using direct Ag particles, which were prepared from aqueous solution using the salt reduction without surfactant at room temperature, was developed. The effect of different architectures of Ag particles on SERS enhancement factor for detecting melamine was investigated. This proposed method enables to precise control of the form of silver, large scale and easily to make a melamine probe using SERS technique.

2. Materials and experiments

2.1. Apparatus and reagents

The X-ray powder diffraction (XRD) patterns of the samples were carried out on an X'pert pw3040/00 (PANalytical) diffractometer using Cu Kα radiation ($\lambda = 0.15405$ nm). Field emission scanning electron