



Depositing a flexible substrate of triangular silver nanoplates onto cotton fabrics for sensitive SERS detection

Deshan Cheng^{a,b}, Mantang He^{a,b}, Jianhua Ran^{a,b,*,1}, Guangming Cai^{a,b}, Jihong Wu^{a,b},
Xin Wang^{c,**,2}

^a School of Textile Science and Engineering, Wuhan Textile University, Wuhan 430200, China

^b Key Laboratory of Textile Fiber & Product (Wuhan Textile University), Ministry of Education, Wuhan 430200, China

^c School of Fashion and Textiles, RMIT University, Melbourne 3056, Australia

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ABSTRACT

A flexible and effective substrate for surface-enhanced Raman spectroscopy (SERS) detection has been developed by dip coating of triangular silver nanoplates (TSNPs) onto cotton fabrics. The morphology, chemical composition and crystalline structure of the coated cotton fabrics were characterized by scanning electron microscope (SEM), ultraviolet-visible spectroscopy (UV-vis), X-ray photoelectron spectroscopic (XPS) and X-ray diffraction (XRD), respectively. These results confirmed the presence and well dispersion of the TSNPs on the surface of cotton fabrics. *p*-aminothiophenol (PATP) has been used as the probe molecule to evaluate the performance of the coated cotton fabrics as a flexible SERS substrate, and the PATP in diluted solutions can be detected with the concentration as low as 10^{-8} M. The results indicate that the TSNPs-coated fabrics exhibit sensitive SERS signals with excellent reproducibility and stability. The flexible SERS substrate has been demonstrated to rapidly detect carbaryl pesticides on fruits.

1. Introduction

As a powerful analytical technique for detecting molecular species on surfaces, SERS is sensitive, accurate, trustable and repeatable, with application in materials science, surface science, biological and medical testing and diagnosing [1–5]. The determining factor for SERS is the active surface that decides the intensity and repeatability of SERS signals. Metal nanoparticles exhibit strong enhanced Raman scattering effect, and the preparation of nanostructural surface from them has been an important challenge for SERS [6–8]. TSNPs have been extensively studied as the substrate for SERS due to their strong SERS performance [9–12].

Currently, TSNPs for SERS substrates are mainly in the form of self-assembled nanoparticles. In order to enhance the SERS performance with stability, nanoparticles are usually piled up to form a membrane on the substrate [13]. A SERS active surface was usually formed from metal nanoparticles on a solid substrate, such as silicon [14], copper [15], quartz [16] or glass [17]. The flexible SERS substrates are more favorable in application as they have good mechanical properties with

well-covering effect and large surface areas. TSNPs have been applied as flexible SERS substrates with flexible matrices, such as filter papers [18], egg shell [19], graphene oxide [20] and textile fabrics [21].

Textiles are promising flexible SERS substrates for adsorption and diffusion of probe molecules with strong Raman signals due to their mechanical flexibility, porous structure, large specific surfaces and permeability. Liu et al. prepared SERS substrates through in-situ synthesis of gold nanoparticles on silk fabrics, and the SERS performance was discussed [22]. Qu et al. fabricated SERS cotton swabs by assembly of Ag-NPs with a detection limit of Rhodamine 6G (R6G) at 8.1×10^{-13} M [23]. Zaffino et al. presented a silver colloid on wool fibers to obtain Fourier-transform surface-enhanced Raman spectra (FT-SERS) of natural historical dyes [24]. However, there are no reports on the application of TSNPs as a flexible SERS substrate.

In this study, a flexible SERS substrate was prepared by self-assembly of TSNPs on cotton fabrics. The morphology and structure together with thermal stability of the TSNPs-coated cotton fabrics were characterized by SEM, UV-vis, XPS, XRD and TG. PATP was used as the probe molecule to evaluate the sensitivity, reproducibility and stability

* Corresponding author at: School of Textile Science and Engineering, Wuhan Textile University, Wuhan 430200, China.

** Corresponding author at: School of Fashion and Textiles, RMIT University, Melbourne 3056, Australia.

E-mail addresses: jhran@wtu.edu.cn (J. Ran), xin.wang@rmit.edu.au (X. Wang).

¹ Textile Road, Hongshan District, Wuhan, Hubei 430073, China.

² 25 Dawson Street, Brunswick, Melbourne 3056, Australia.

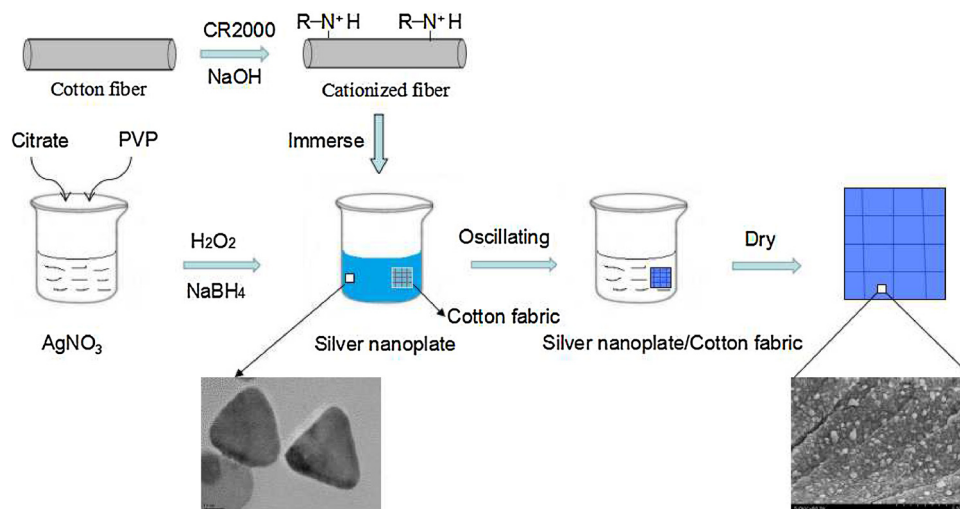


Fig. 1. Schematics illustration of the preparation of TSNPs and dip coating of cotton fabrics.

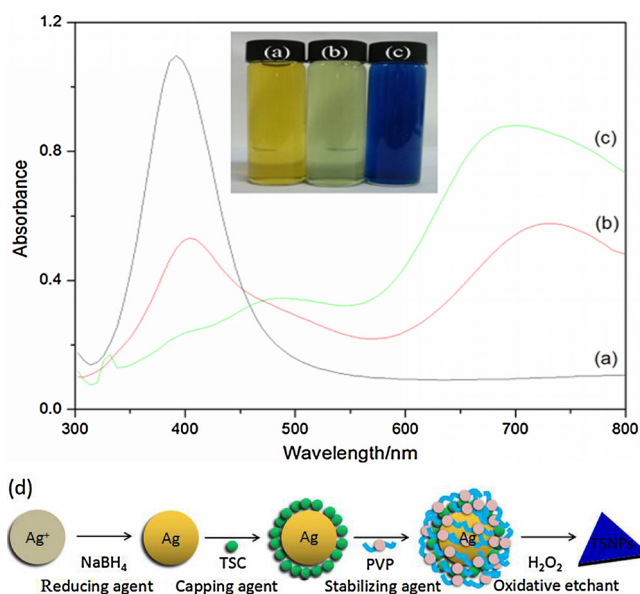


Fig. 2. UV-vis spectra of the reaction solution at different stages of the synthesis process (Inset: photos of the solution at different stages and schematic illustration of the formation process of TSNPs).

of the SERS substrates. Moreover, the application of the flexible SERS substrates in rapid detection of carbaryl pesticides on fruits was demonstrated.

2. Experimental

2.1. Materials

Cotton fabrics (plain, 135 g/m²) were supplied from Yudahua Textile Co., Ltd (Wuhan, China). 3-Chloro-2-hydroxypropyl trimethyl ammonium chloride (CHTAC, 65%, w/w) was purchased from TCI Chemical Industry Co., (Tokyo, Japan). Silver nitrate (AgNO₃), trisodium citrate (TSC), poly(vinylpyrrolidone) (PVP), hydrogen peroxide (30%, H₂O₂) and sodium borohydride (NaBH₄) were obtained from Aladdin Chemical Agent Co., (Shanghai, China). *p*-aminothiophenol (PATP) and carbaryl were purchased from Macklin Biochemical Co., Ltd (Shanghai, China). All the chemicals were in analytic grade and used without further purification.

2.2. Preparation of TSNPs

Colloidal TSNPs were prepared via a chemical reduction method with a few modifications [25]. A 6 mL TSC (0.3 mM), PVP (0.5 mg/mL) and 0.2 mL H₂O₂ (30%) were sequentially added into a 100 mL of AgNO₃ (0.1 mM) solution followed by vigorously stirring for 10 min. Then, a 0.6 mL NaBH₄ solution (50 mM) was rapidly injected into the mixture solution, and the color of the mixture solution changed from yellow to red, and then purple, followed by green, and finally to blue. The blue colloids were stored in the dark for 2 h prior to use.

2.3. Fabrication of SERS substrate

The deposition of TSNPs onto cotton fabrics are schematically shown in Fig. 1. Cotton fabrics were firstly cationized by CHTAC to introduce activity sites on the surface of fibers. CHTAC was first activated by reacting with sodium hydroxide at a 1:2 mol ratio with pH below 11. Fabrics were treated with the mixture solution at 75 °C for 90 min, followed by a thorough rinsing with water and neutralizing with 1% acetic acid. Finally, the fabrics were rinsed again with water and dried.

The cationized cotton fabrics were dipped into the prepared TSNPs colloids in an oscillating water bath at room temperature. After the color of colloids changed from blue to transparent, the cotton fabrics were removed and washed with deionized water followed by a blow dry with nitrogen.

2.4. Measurements and characterization

Structure of TSNPs was obtained from a transmission electron microscopy (2100F, JEOL Inc, Japan) at an accelerating voltage of 15 kV and 200 kV. The zeta potential of the TSNPs was assessed by a ZetaSizer (3000HSA, Malvern Instruments Ltd, US). Ultraviolet-visible spectroscopy (UV-vis) was obtained from a Hitachi U-4100 instrument (Hitachi, Japan). Fourier transform infrared (FTIR) spectra were obtained on a Nicolet 5700 instrument (Nicolet Co., US) accompanied with a wavenumber range of 4000–400 cm⁻¹ and a resolution of 2 cm⁻¹. The surface morphology and elemental composition were observed from a scanning electron microscopy (SEM, JSM-5600LV, JEOL, Japan). XPS measurement was performed on a PHI 5000C ESCA system with an Mg K α source at 14.0 kV and 25 mA (Perkin-Elmer, America). The XRD pattern was collected at 40 kV and 200 mA on an X-ray diffractometer (D/max 2500, Rigaku, Japan) using Cu K α radiation with the diffraction angle 2 θ range of 20–80°. Thermogravimetry analysis (TGA) was conducted on a Netzsch TG209 F1 thermal analyzer at a

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