Pushing the limit of Raman detection of Melamine using SERS

Abstract

In this App Note we present results for a novel nanoparticle enhanced SERS substrate that overcomes many of the barriers to the adoption of SERS in Food Safety and demonstrate trace detection of melamine.

Introduction

The rise in popularity of plant-based eating brings with it a heightened risk of the unscrupulous use of protein substitutes, such as Melamine. Thus the need for fast, reliable and sensitive methods for detecting food adulterations has never been greater. Surface Enhanced Raman promises high sensitivity detection of food adulterations using Raman Spectroscopy [1] but has not been widely adopted due to issues with reliability, reproducibility, cost and ease of use. Liquid based SERS is most commonly used in laboratories in preference to expensive solid SERS substrates[2], but solution preparation and handling makes this technique unsuitable for in field use or for use by non-experts. Nikalyte's SERS substrates utilize gold nanoparticles, generated in vacuum by terminated gas condensation, which are free of ligands and hydrocarbons usually associated with chemical synthesis. Figure 1 shows a schematic of how the gold nanoparticles are generated from a solid gold target and deposited directly onto the substrates using the Nikalyte NL50 nanoparticle deposition system. The ultra-pure nanoparticles self-organise and trap the analyte at hot spots to provide high signal to noise Raman measurements required for reliable measurement of low analyte concentrations. In this App Note we investigate the limit of detection of Melamine using Nikalyte's nanoparticle SERS substrates.

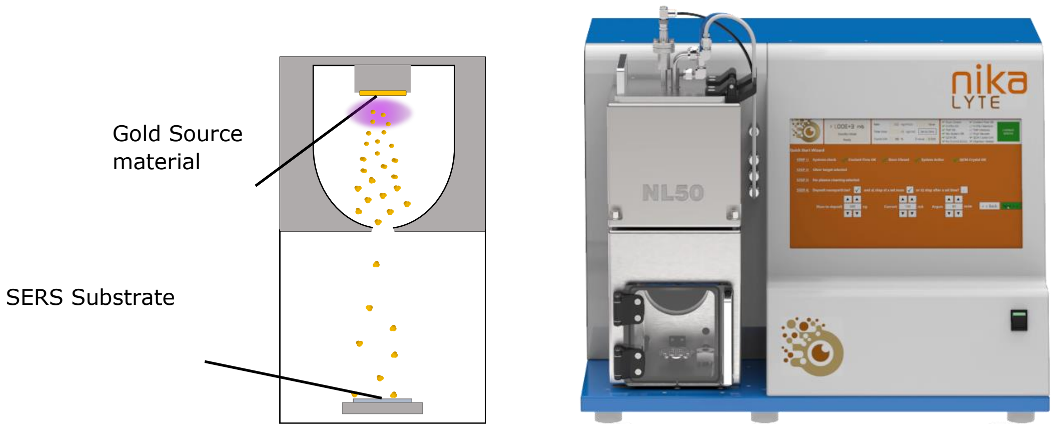

Figure 1 Schematic of the NL50, showing how the gold nanoparticles are generated from the solid gold source material (left) and the full NL50 benchtop nanoparticle deposition system (right).

[1] Z.Lin and L. He, *Current Opinion in Food Science* **28,** 82-87 (2019)
[2] L. Li and W. S. Chin, *Food Chemistry* **357** 129717 (2021)

NL50 Application Note

Experimental Conditions

Raman measurements were performed using a Wasatch Photonics-785-SR-25 spectrometer with integrated laser, a fibre coupled 785nm-nm Raman probe, inserted into a Raman sample holder with a microscope slide slot for the SERS substrates. A dilution series of melamine in water for 0, 1ppm, 10ppm, 100ppm and 1000ppm was prepared. A laser power density of 15mW/cm2 was focused into a 900um spot. For each Melamine concentration a Raman measurement was taken in vial and also using the Nikalyte SERS substrates, where a drop of the solution was simply applied to the active region of the substrate using a pipette and allowed to spread before measurement.

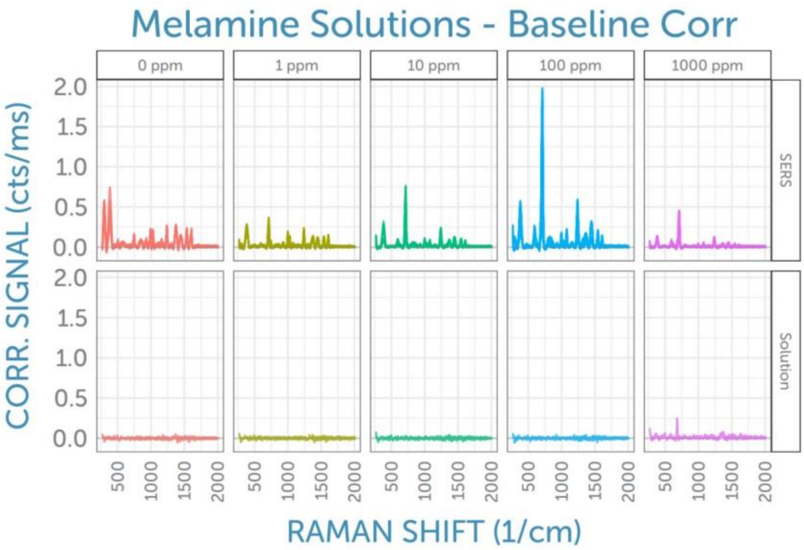

Figure 2 Baseline corrected Raman spectra for Melamine dilution series measured using Nikalyte SERS substrates (top) and in-vial (bottom)

Results: The baseline corrected Raman measurements for the in-vial and SERS substrates for the full range of Melamine concentrations are shown in Figure 2. The in vial measurements of the melamine solution shows no measurable Raman signal for concentrations less than 1000ppm, where a weak signal at 675cm-1 is observed. Conversely the 675cm-1 Melamine peak is clearly visible for all concentrations of the solution including the 1pm solution when measured using the SERS substrates.

Conclusions: The Limit of detection for Melamine was reduced by three orders of magnitude from 1000ppm down to 1ppm with the use of Nikalyte nanoparticle SERS substrates. The high sensitivity of the SERS substrates coupled with low cost and ease of use makes this technique ideal for the detection of food adulterations including Melamine, where fast detection of low concentrations is vital to consumer health.