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Reconfigurable SERS platform based on magnetization-controlled self-assembly of magnetic nanoparticles on NdFeB composite substrate

Na Gao^{1†}, Liu Wang^{1†}, Xifeng Li¹, Mingxuan Wang¹, Sheng Li¹, Chenpeng Zhang², Stefanos Mourdikoudis³, Guangchao Zheng⁴ and Mingli Wang^{1*}

Abstract: Constructing surface-enhanced Raman scattering (SERS) substrates using magnetic control strategies is a common approach for the development of SERS-efficient tools. However, one persistent challenge lies in how to regulate the spatial coupling between the target analyte carrier and the plasmonic hot spots, in order to maximize the SERS enhancement activity. This study proposes a magnetic-field-controlled composite SERS substrate strategy, through regulating the spatial distribution of the target molecule carriers, to realize its coupling with the plasmon hot spot. This method allows to elaborate a NdFeB/PS/Ag composite structure as the plasmonic substrate, using Fe₃O₄@SiO₂@Ag (FSA) magnetic nanoparticles as the target molecule. By regulating the magnetic state of NdFeB magnetic sheets and optimizing the magnetization gradient, the spatial distribution state of FSA magnetic nanoparticles can be controlled. Meanwhile, based on the differences in magnetic field distribution at different positions on the NdFeB substrate, the regional control of SERS signals can be achieved. The finite element analysis method was also used to further verify the experimental results. Thanks to the advantages of differentiated signal distribution brought by magnetic field regulation, this system is expected to be applied to the actual detection of environmental pollutants, harmful substances in food, and biomarkers.

Keywords: SERS; NdFeB; magnetic drive; self-assembly; surface plasmonic hot spots

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1 Introduction

Surface-enhanced Raman scattering (SERS) is a promising technique for the detection of trace substances based on plasmonic nanostructures^{1–3}. With its ultra-high specificity in molecular fingerprint recognition⁴, it has garnered significant attention across numerous fields including environmental monitoring, food safety, and biomedical testing^{5–8}. Precious metals (such as gold and silver) in nanostructures serve as plasmonic materials. They can generate strong localized electromagnetic fields by exciting localized surface

plasmon resonance (LSPR), which is the core enhancement mechanism of SERS effect^{9,10}. To optimize the construction and regulation of plasmonic hot spots, various implementation schemes have been proposed in related research: for instance, multi-level nanoporous silver microstructures can be fabricated through electrochemical methods, and a clean and stable plasmonic hot spots can be obtained by relying on the regular microstructure morphology¹¹; a hydrogel composite SERS platform can also be constructed, and the particle spacing can be regulated by the reversible deformation of the gel, thereby dynamically changing the plasmonic

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coupling strength¹². Currently, magnetic-responsive SERS substrates mostly rely on soft magnetic materials¹³. Magnetic field-controlled SERS substrates have been extensively reported^{14–18}. In the existing related systems, the magnetic field is mainly used for passive enrichment and rapid separation of magnetic particles^{19–22}, or for driving particle movement and regulating the magnetic optical properties of one-dimensional nanostructures^{23–25}. Most of them adopt uniform external magnetic fields. In this study, a gradient magnetic field was constructed using an NdFeB hard magnetic substrate, and the magnetic carriers were coupled by the magnetic field gradient to establish a magnetic control dynamic assembly strategy.

NdFeB, as a high-performance permanent magnet material, exhibits characteristics such as high magnetic permeability and strong coercivity²⁶, of which the surface magnetic field strength can be flexibly controlled through magnetization and demagnetization processes; as a result, a stable magnetic field environment is achieved for regulating the distribution of magnetic nanoparticles²⁷. Polystyrene (PS) microspheres, characterized by their uniform size, regular morphology, and ease of functionalization, are frequently employed as templates for constructing ordered nanostructure substrates. They provide support for the loading of precious metals and the distribution of magnetic nanoparticles²⁸. Silver (Ag), being a typical precious metal material²⁹,

possesses outstanding plasmonic properties³⁰. It can be sputtered onto the surface of NdFeB magnetic sheet modified with PS microspheres, resulting in the formation of highly efficient plasmonic hot spots, thus ensuring SERS enhancement. On this basis, in this study, NdFeB magnetic sheet was used as the magnetic substrate³¹, and PS microspheres were loaded by the pulling method to construct an ordered template³². Subsequently, a plasmonic active layer was formed via silver sputtering; concurrently, $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{Ag}$ (FSA) magnetic nanoparticles were prepared to serve as carriers for target molecules and signal amplification auxiliary units. By magnetising and demagnetising the NdFeB magnetic sheet, the intensity and range of the surface magnetic field distribution are altered, thereby controlling the spatial distribution of magnetic FSA nanoparticles on the NdFeB/PS/Ag composite substrate surface. This leads to coupling between hot spots regions and target molecules loaded onto the FSA. In this work, magnetic fields are employed as a non-contact regulatory mechanism, eliminating the need for complex chemical modifications. This enables flexible control over the distribution of analytes, significantly enhancing their interaction efficiency with plasmonic hot spots, ultimately achieving a marked improvement in Raman detection performance for target molecules.

To elucidate the working principle of our SERS substrate, Fig. 1 presents a schematic illustration of the entire process,

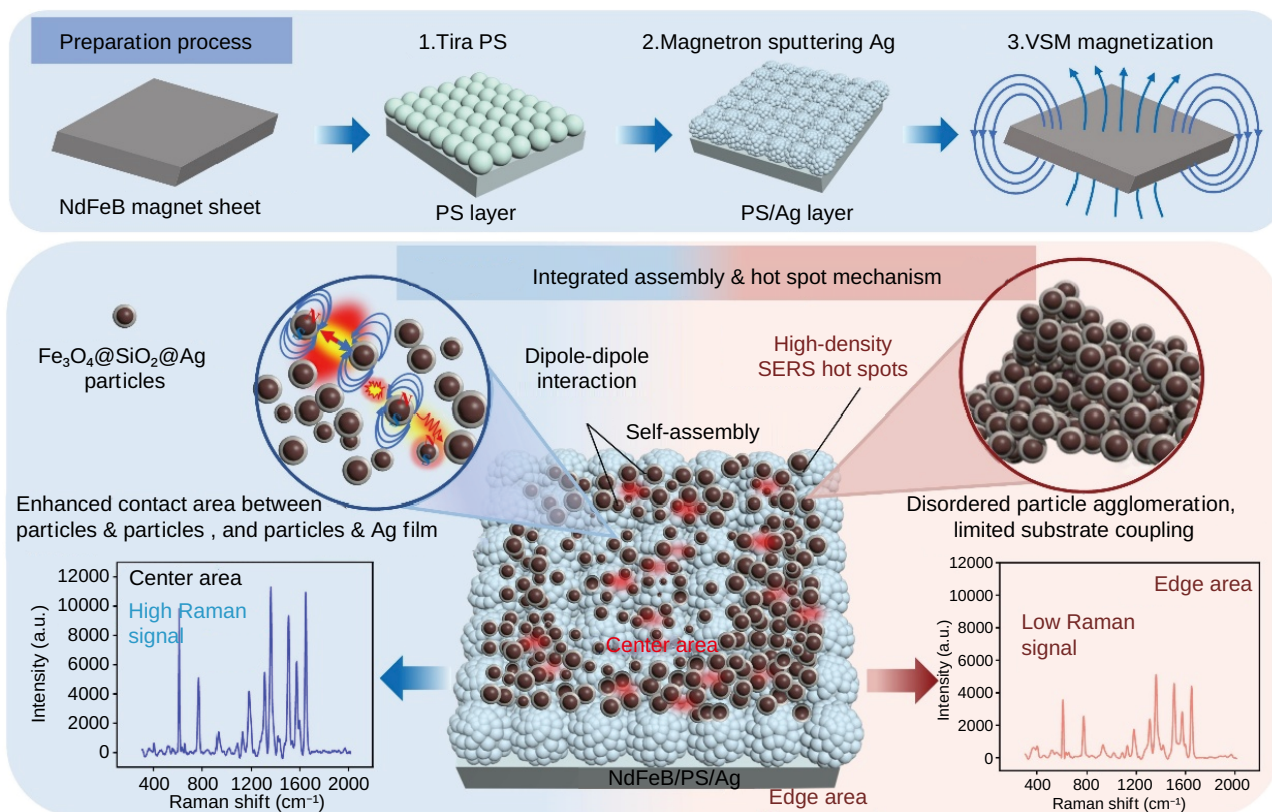


Fig. 1 | Schematic diagram of the preparation process of NdFeB/PS/Ag SERS substrate and the mechanism of magnetic field regulation.

encompassing the substrate fabrication, magnetic-field-driven particle assembly, and the resulting hot spot distribution.

2 Experiments and methods

2.1 Materials

Polystyrene microspheres (diameter 0.6–1.0 μm , 2.5% w/v), Sodium dodecyl sulphate (SDS), R6G, MB, thiram, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, tetraethoxysilane (TEOS), sodium citrate, ammonia solution, citric acid, ascorbic acid, ethylene glycol (EG), and sodium acetate were procured from Aladdin Reagent Co. Ltd. Polyvinylpyrrolidone (PVP) was obtained from China Pharmaceutical Co. Ltd. Silver nitrate (AgNO_3) was bought from Kemiou Chemical Reagent Co. Ltd. and ethanol was purchased from Kaithong Chemical Reagent Co. Ltd. Deionised water was prepared in-house, the grapes were all supplied from the local market. Silver target material (99.99%). The NdFeB magnet plate (5×5×1.5 mm, N30, the magnet is magnetized along the thickness direction, forming a bipolar structure with the upper and lower surfaces each having an N pole and an S pole) was obtained from Shanghai Yanyi Magnet Co. Ltd.

2.2 Preparation of the substrate

NdFeB magnetic sheets were placed in anhydrous ethanol and subjected to ultrasonic agitation for 3–10 minutes. After rinsing, the cleaned NdFeB sheets were dried at 80 °C for 15–30 minutes. The treated NdFeB magnet sheets were submerged in a 10% SDS solution for 24 h to impart hydrophilicity³³. Subsequently, a 2.5% mass fraction suspension of 600 nm polystyrene microspheres was mixed with anhydrous ethanol in a 1:1 ratio and sonicated for 10 minutes. The mixture was slowly dripped onto the water surface until the polystyrene microspheres covered the entire liquid surface. A 10% SDS solution was then added to induce the tight arrangement of the microspheres. The hydrophilic-treated NdFeB sheet was inserted at an angle into the water, slowly moved beneath the PS film, then lifted and dried to obtain a neatly arranged and dense PS microsphere film.

The dried sheets were fixed onto microscope slides. An RF magnetron sputtering apparatus (DHRM-3) was utilized within the magnetron sputtering system to modify PS microspheres with Ag nanoislands via sputtering at varying durations. Operating parameters were: working voltage 100 V, current 0.3 A and the vacuum level was 5×10^{-3} Pa.

2.3 Preparation of $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{Ag}$ microspheres and base self-assembly

The preparation of magnetic particles $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{Ag}$ was carried out using the three-step method described in the

literature with some modifications³⁴; in the third step, a seed-mediated growth mode was employed: 0.025 g of $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{Ag}$ seeds were dispersed in 50 mL of a solution containing 0.2 g AgNO_3 and 0.3 g citric acid. Under mechanical stirring, 20 mL of a 0.3 g ascorbic acid solution was added dropwise over 15 minutes. The mixture was then centrifuged (5000 r/s, 8 min), washed, and dried. Self-assembly stage: 0.001 g of FSA magnetic particles were dispersed in 10 mL of ultrapure water, the reasons for choosing the dosage are detailed in Supplementary information Section 1 and Fig. S1. The structural side of the NdFeB/PS/Ag magnetic base was immersed in the water, and through magnetic adsorption, the FSA magnetic particles were dispersed on the surface of NdFeB/PS/Ag.

2.4 Characterization of the substrate and SERS measurements

Surface-enhanced Raman spectroscopy data were acquired using a Raman spectrometer (Renishaw inVia) Raman spectra were collected from 200–2000 cm^{-1} using a 0.05 mW laser, 1 s integration time, and one accumulation per point, under a 532 nm laser. Magnetic properties were measured using a Tesla Meter (DLX-STW1044). Crystal structure and morphology were analysed via X-ray diffraction (XRD). The morphology of the PS-Ag array, microstructure of magnetic nanoparticles, and elemental analysis of the substrate were investigated using scanning electron microscopy (Hitachi S4800 II) and transmission electron microscopy (JEM-2010). Magnetisation, demagnetisation, and physical property measurements of neodymium-iron-boron magnet sheets were conducted under varying fields using a Vibration Sample Magnetometer (VSM, Lakeshore 7407).

2.5 Finite element analysis

Using finite element simulation (COMSOL Multiphysics 6.2), the electromagnetic field distribution under illumination was simulated to elucidate the fundamental mechanism of the underlying Raman signal enhancement. The background refractive index was set to 1, and the nanostructure dimensions were determined based on statistical measurements of average sizes derived from experimental results. The model adopts periodic boundary conditions in the x and y directions, and sets a perfectly matched layer boundary condition in the z direction. The electric field amplitude of the incident plane wave is 1 V/m³⁵. Simultaneously, finite element analysis was employed to create a three-dimensional model of the neodymium-iron-boron magnet plate, with the residual magnetic flux density oriented along the z -axis. Air was utilised as the background medium with a refractive index of 1. The system simulated and analysed the three-dimensional spatial distribution characteristics of magnetic field lines and the distribution patterns of magnetic flux density, in that way providing a theoretical

basis for the structural design of composite substrates and the regulation of magnetic response performance.

3 Results and discussion

3.1 Optimization of NdFeB/PS/Ag and NdFeB/PS/Ag-FSA magnetic substrates

By controlling different sputtering durations, Ag films of varying sizes were prepared, aiming to obtain the most suitable substrate. Five distinct durations were set: 0, 5, 10, 15, and 20 minutes. Fig. S2 shows its characterization under an electron microscope. SERS performance was tested with R6G at a concentration of 10^{-6} M³⁶. The SERS intensity exhibited a trend of first increasing and then decreasing. Specifically, the strongest signal intensity was clearly observed when the sputtering time was 10 minutes. However, as the sputtering time increased, the Raman intensity conversely decreased, as demonstrated in Fig. S3. This was attributed to excessive Ag layer growth, which substantially filled the gaps between PS microspheres. Consequently, the number of surface's "hot spots" diminished (as shown in Fig. S2, a1–a5 represent the morphologies of the Ag layers at different sputtering times), and the original "hot spot" structure was disrupted. Therefore, for subsequent experiments, the NdFeB/PS/Ag magnetic substrate with a 10-minute sputtering time was selected. At the same time, to evaluate the influence of the PS intermediate layer on SERS activity, three substrate configurations were compared at a 10^{-6} M R6G concentration Fig. S4. The NdFeB/PS/Ag-FSA tri-layer structure exhibited optimal performance, while NdFeB/Ag-FSA (without PS layer) showed a significant reduction in peak intensity at 612 cm^{-1} to approximately 4000 a.u. Conversely, the NdFeB/PS-FSA (without Ag layer) displayed a peak intensity reduction to approximately 2000 a.u., confirming that Ag nanoparticles are an essential component for SERS activity. This comparative experiment illustrates that the PS interlayer enhances SERS intensity by regulating the structure and stabilising the nanopag.

3.2 Evaluation of NdFeB/PS/Ag-FSA magnetic substrates

Figure 2(a) shows the ultraviolet-visible absorption spectrum of the R6G aqueous solution. A pronounced absorption peak is observed at 527 nm ³⁷, nearly coinciding with the excitation wavelength of 532 nm , satisfying the conditions for resonant Raman scattering. Figure 2(b) presents the ultraviolet-visible absorption spectrum of the NdFeB/PS/Ag composite magnetic substrate and its intermediates (NdFeB/

PS, NdFeB). The NdFeB magnetic sheet and the PS-coated substrate exhibit a broad, flat absorption curve in the visible region without distinct characteristic peaks. Upon further loading with Ag nanoparticles, a pronounced absorption peak emerges at 318 nm , indicating the successful formation of an Ag shell layer with a concentrated particle size distribution³⁸. Therefore, a 532 nm laser was chosen as the excitation source. The material crystal structure was investigated by XRD Fig. 2(c), revealing characteristic peaks attributable to Fe_3O_4 ³⁹, while typical XRD diffraction peaks were observed at 38.1° , 44.3° , and 64.4° , corresponding to the (111), (200), (220) and (311) lattice planes of face-centered cubic (fcc) Ag⁴⁰. As shown in Fig. 2(d–h), EDS analysis of the NdFeB/PS/Ag-FSA structure successfully detected Fe, Si, Ag, and O elements. The high Ag content observed in Fig. 2(g) stems from the Ag shell surrounding the FSA particles and the Ag film deposited onto the magnetic substrate. These results confirm the successful preparation of the NdFeB/PS/Ag-FSA composite.

TEM imaging was employed to investigate the core-shell structure of FSA. Figure 2(i) displays a TEM image of FSA nanoparticles, revealing a nearly spherical morphology. The elemental distribution mapping in Fig. 2(j) further clarifies the spatial distribution of nanoparticle components: Si elements predominantly concentrate in the outer layer of the particles, corresponding to the structural characteristics of the silica shell. Fe elements are distributed at the particle core, confirming the successful formation of the Fe_3O_4 magnetic core. The element Ag exhibits a shell-like distribution pattern, mainly concentrated in the outer region corresponding to Si elements, with high overlap in distribution areas: this indicates the successful loading of Ag components onto the mesoporous silica shell surface. The superimposed mapping image of the three elements visually demonstrates the spatial separation of Si, Fe, and Ag components, illustrating the successful preparation of a core-shell structure comprising an Fe_3O_4 , SiO_2 shell, and active Ag outer layer.

3.3 Magnetically driven mechanism of Raman response in NdFeB/PS/Ag-FSA magnetic substrates

Figure 3(b–c) depicts the hysteresis loop of the magnetic nanoparticles and the substrate^{41,42}. The saturation magnetisation of NdFeB is approximately 8032.27 G . For a rectangular NdFeB magnet with dimensions $a \times b \times c$ (length $\times$ width $\times$ height), the resulting magnetic field distribution is as follows^{43,44}:

$$B(z) = \frac{Br}{\pi} \left(\arctan \frac{ab}{2z\sqrt{4z^2 + a^2 + b^2}} \arctan \frac{ab}{2(z+c)\sqrt{4(z+c)^2 + a^2 + b^2}} \right), \quad (1)$$

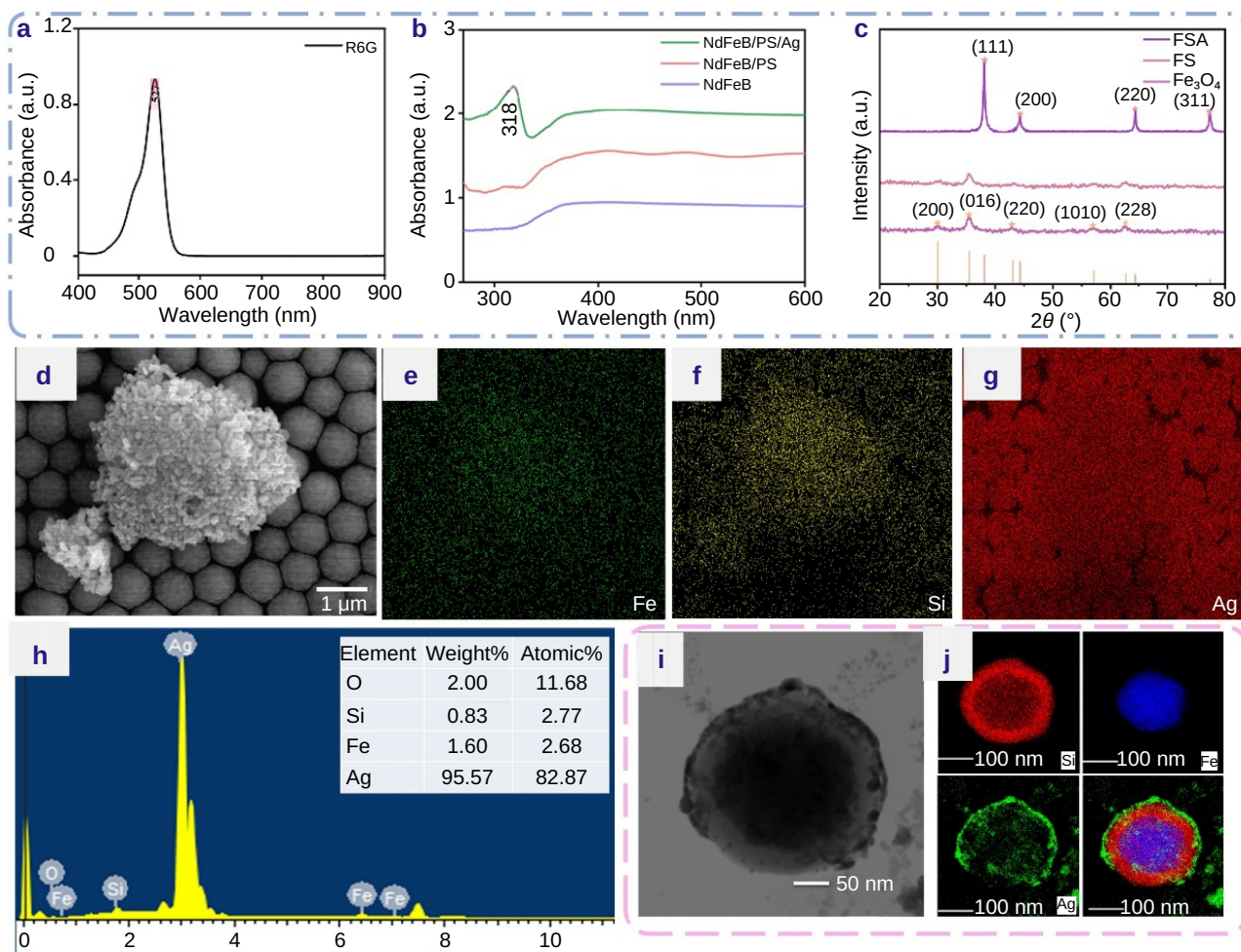


Fig. 2 | (a) Ultraviolet-visible absorption spectrum of the R6G solution. (b) Ultraviolet-visible absorption spectra of NdFeB, NdFeB/PS, and NdFeB/PS/Ag. (c) XRD diffractograms of FSA, FS, and Fe_3O_4 . (d) SEM morphology image of the substrate. (e–g) Elemental mapping of Si, Ag, and Fe elements in (d). (h) EDS spectrum and elemental composition table of the substrate. (i) HAADF-STEM microstructural features of FSA. (j) Elemental mapping of Si, Fe, and Ag in (i).

where z denotes the distance measured along the magnet's central axis from its surface ($z \geq 0$). When z equals 0, the formula for the surface magnetic flux density of a rectangular magnet can be derived as shown below:

$$B_{\text{surface}} \approx Br \times \left[\frac{1}{2} - \frac{1}{\pi} \arctan \frac{ab}{2c\sqrt{4c^2 + b^2 + a^2}} \right]. \quad (2)$$

The surface magnetisation of the neodymium iron boron magnets used in this experiment was approximately 180 mT. The saturation magnetisation of the magnetic nanoparticles was about 2.3 emu/g, with these materials exhibiting superparamagnetic behaviour. Through the series of operations shown in Fig. 3(a), the neodymium iron boron sheet undergoes magnetization and demagnetization treatment. First, the neodymium iron boron magnet was subjected to high-temperature demagnetisation, yielding a surface magnetisation of approximately 20 mT. For magnetised plates with surface magnetisation ranging from 0 to 20 mT, an alternating magnetic field method was subsequently employed via a

VSM machine to demagnetise the high-temperature demagnetised plates further. This process resulted in ferromagnetic plates with a surface magnetisation of approximately 2 mT. We magnetised the NdFeB magnet sheet by applying an external magnetic field. By stacking NdFeB magnet sheet magnetised under different magnetic field conditions, seven ranges were set up in the experiment. In particular, the following ranges were established: 0–20 mT, 20–40 mT, 40–60 mT, 60–80 mT, 80–100 mT, 100–120 mT, and 120–140 mT. Using R6G at a concentration of 10^{-6} M as the probe molecule, Raman spectra of the base were collected under different gradients. The results are shown in Fig. 3(d–f). R6G correlates with C-C-C ring bending, C-H out-of-plane bending, and C-C stretching vibrations at 612 cm^{-1} , 775 cm^{-1} , and 1648 cm^{-1} ^{45,46}. As the surface magnetic field of NdFeB magnetic sheets increases, the corresponding Raman signal intensity exhibits a progressive enhancement trend.

Magneto-mechanical phenomena occur when a static

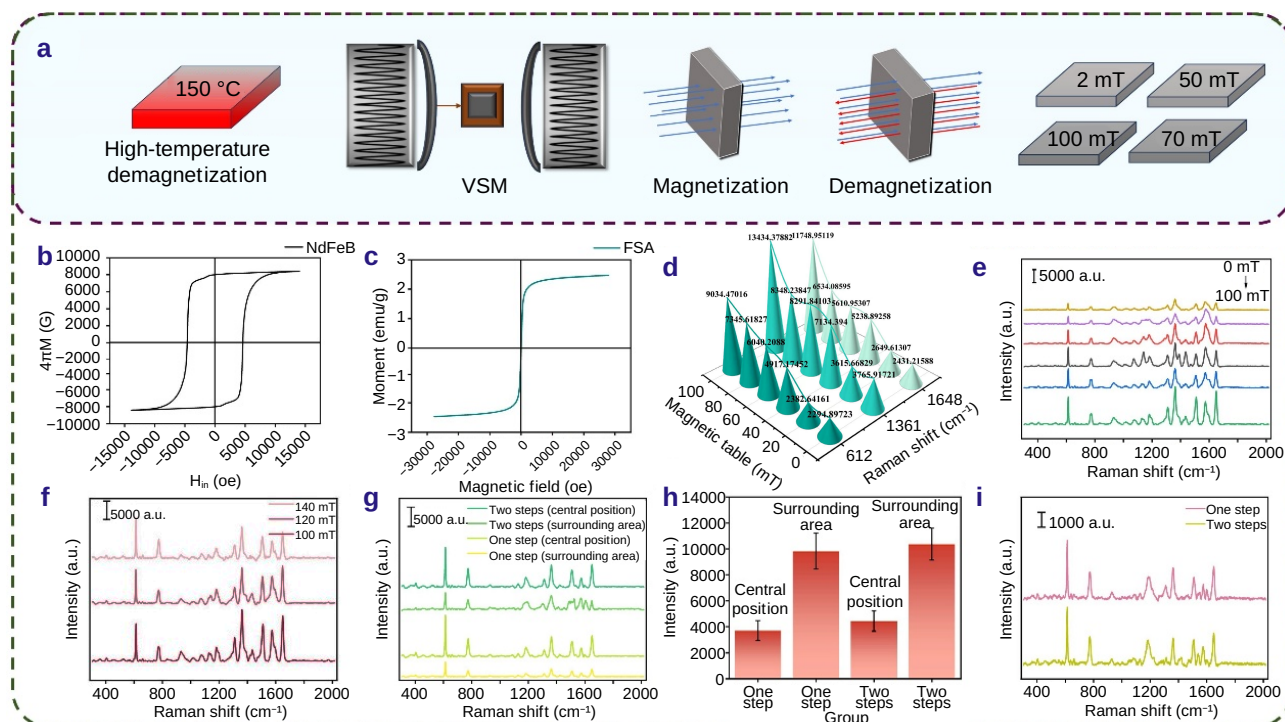


Fig. 3 | (a) Schematic diagram of NdFeB magnetisation and demagnetisation. (b) Hysteresis loop of NdFeB. (c) M-H plot for FSA. (d) Spatial distribution map of Raman signal intensity on the substrate surface. (e–f) Raman spectra comparison of the substrate at different applied magnetic fields (0–100 mT) and (100–140 mT) for 10⁻⁶ M R6G. Comparison of SERS signals in the central and peripheral regions of the substrate under one-step and two-step assembly systems. (g) Raman spectra corresponding to the center and edge positions of the NdFeB/PS/Ag substrate. (h) The statistical column chart of Raman intensities at the center and edge of the 612 cm⁻¹ characteristic peak in different assembly systems. (i) Raman spectra comparison of FSA magnetic particles under different assembly systems on glass slides.

magnetic field exerts mechanical force on a magnetic field, resulting in direct drive⁴⁷. Under low magnetic fields, the surface magnetism regulates the spatial distribution of FSA magnetic particles through magnetic induction. Meanwhile, dipole-dipole interactions occur between the magnetic particles. Induce the particles to self-assemble along the magnetic field direction⁴⁸. The contact area between adjacent magnetic particles and the particles themselves, as well as between the magnetic particles and the silver film, has been increased¹⁸; as a result, the number of SERS hot spots is enhanced. When the surface magnetic field exceeds 80 mT, the Raman signal intensity essentially stabilises and no longer undergoes significant changes. This value corresponds to the saturation magnetic field strength of the FSA magnetic particles, where their distribution achieves the optimal state under magnetic induction. Consequently, NdFeB magnetic sheets with surface magnetic fields within the 80–100 mT range were selected as substrates for magnetic particle enrichment.

We intend to explore how the addition sequence of analyte molecules affects magnetic-field-induced particle aggregation and SERS enhancement performance in this work. Two different sample preparation methods are designed for comparative experiments. One is the one-step

mixing-magnetization strategy, and the other is the two-step strategy of magnetic aggregation followed by analyte loading. Simultaneous Raman signals from the center and edge regions of the magnetic substrate were collected. The relevant test results are shown in Fig. 3(g–h). The results indicate that, regardless of whether the one-step or two-step method is used for treatment, the Raman intensity of the characteristic peaks in the edge area of the substrate is significantly higher than that in the central area; and under both magnetic attraction methods, there is no significant difference in the signal strength values at the same detection position. Figure 3(i) shows that without applying a magnetic field, the magnetic nanoparticles are randomly and discretely distributed on the glass sheet. There is no significant difference in the Raman spectra of FSA magnetic particles in the two different assembly systems. The results of the two control experiments confirm that the introduction sequence of the analyte molecules does not interfere with the particle aggregation behavior dominated by the magnetic field. The differential hot spot enhancement effect in different regions of the substrate is solely determined by the varying magnetic field intensities at different locations on the magnetic sheet.

Within this study, we have also conducted a three-dimensional finite element simulation of the magnetic field

distribution of NdFeB magnet sheets, where the magnetic flux density is a key parameter that characterizes the magnetic field intensity and distribution characteristics around the NdFeB rectangular magnet. Simulation results are shown in Fig. S5, and it can be observed that the magnetic flux density of the rectangular magnet is highly concentrated at the edges: the magnetic field distribution in its x - y plane exhibits the characteristic of "strong at the periphery and weak in the center" actually. The high magnetic flux density in the surrounding area plays a dominant role in the distribution of FSA magnetic particles. This results in most of the magnetic particles being concentrated around the periphery of the NdFeB magnetic substrate. The interaction between the magnetic particles in the middle area and those adjacent to them also contributed to the better dispersion of these particles on the NdFeB magnetic substrate⁴⁸. Observed the particle distribution state of the actual substrate with a magnetic field of approximately 100 mT after magnetic aggregation by magnetic particles. The respective result is shown in the inset of Fig. S5. The magnetic particles exhibit a distribution characteristic on the magnetic substrate where the outer area is densely packed

while the middle area is sparsely populated, and this is in excellent agreement with the simulation results of the magnetic field distribution.

Based on this simulation prediction, as well as the actual experimental phenomena, in this experiment, 10^{-6} M R6G was used for point detection of Raman spectra in a typical area, as shown in Fig. 4(a). For each of the five positions, a $5\ \mu\text{m} \times 5\ \mu\text{m}$ area was selected for SERS testing. Figure 4(a) on the right presents the characterization of NdFeB/PS/Ag-FSA under an electron microscope. Considering that the strong magnetic NdFeB substrate would possibly interfere with the electron beam of the electron microscope, and this might affect the imaging quality and observation accuracy, we adopted a substrate with a lower magnetic intensity. In Fig. 4(b–e), the magnetic substrate has a RSD (relative standard deviation) higher than 15% at positions I, II, V, and IV³⁴. Compared with the central V area, as shown in Fig. 4(f), the RSD is 9.27%, demonstrating excellent uniformity. Figure 4(g) presents a more intuitive comparison of the Raman intensities of R6G at $612\ \text{cm}^{-1}$ at different positions on the magnetic substrate. Detected the V area at the center of the base, and its Raman intensity is the highest. In sharp

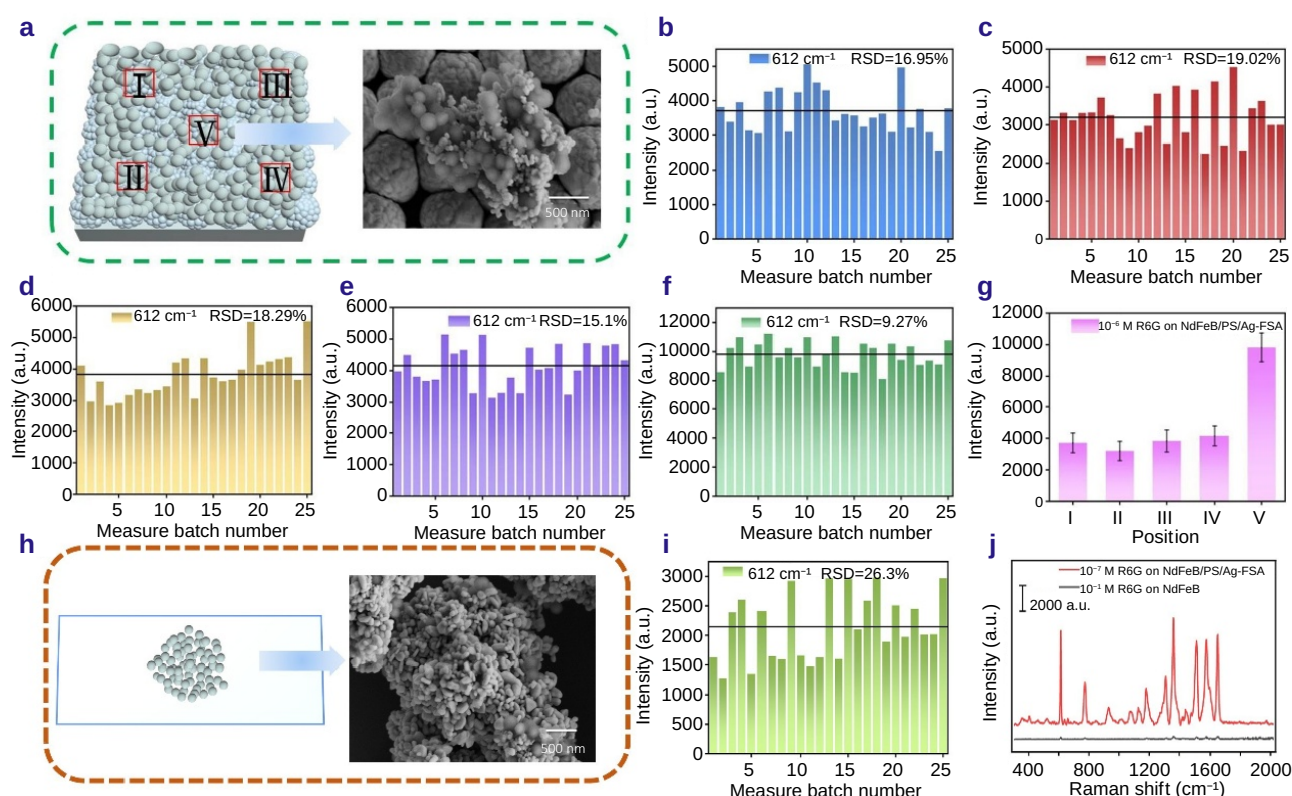


Fig. 4 | (a) Schematic diagrams of five different positions on the magnetic substrate, with their top-view SEM images of NdFeB/PS/Ag-FSA. (b–f) Raman intensities and corresponding RSD values of the characteristic peaks at $612\ \text{cm}^{-1}$ in the left upper, left lower, right upper, right lower and middle positions of the sample. (g) Raman intensities and corresponding RSD values of the characteristic peak at $612\ \text{cm}^{-1}$ of R6G tested on the glass sheet using FSA. (h) Schematic diagram and top-view SEM image of FSA on the glass sheet. (i) The Raman intensity test results of FSA magnetic particles on the surface of the glass plate. (j) SERS spectra of 10^{-7} M R6G in NdFeB/PS/Ag-FSA and 10^{-1} M R6G in NdFeB.

contrast to this, as shown in Fig. 4(h), when the FSA magnetic particles are loaded onto the glass slide, their SEM characterization shows that they are highly aggregated, forming a cluster-like distribution, stacking to form irregular aggregates without obvious dispersion. In what concerns their Raman SERS detection, in combination with Fig. 4(i), the Raman peaks with R6G characteristics exhibited low intensity and uneven signal distribution. The RSD was 26%, which further demonstrated that the magnetic field is the key factor regulating the spatial distribution of FSA particles and the necessity of using NdFeB as the base.

To more intuitively illustrate the sensitivity of this SERS magnetic substrate, we have also evaluated the EF of the magnetic substrate. EF is calculated using the following formula^{49,50}:

$$EF = \frac{I_{\text{SERS}}}{I_{\text{Bulk}}} \times \frac{N_{\text{Bulk}}}{N_{\text{SERS}}}, \quad (3)$$

where I_{SERS} and I_{Bulk} denote the Raman intensity on a complete magnetic substrate and on a NdFeB magnet sheet respectively, while N_{SERS} and N_{Bulk} represent the average number of molecules adsorbed on a complete magnetic substrate and on a NdFeB magnet sheet substrate within the scattering volume respectively. We can calculate N by the following formula:

$$N = N_A \times C \times V \frac{S_{\text{laser}}}{S}. \quad (4)$$

Equivalent volumes of high-concentration (10^{-1} M) C_{Bulk} and low-concentration (10^{-7} M) C_{SERS} solutions were separately spotted onto NdFeB magnet substrate and fully functional substrate surfaces. Assuming R6G molecules distribute uniformly across the SERS substrate surface, S_{SERS} and S_{Bulk} may be considered equivalent. Consequently, Eq. (4) may be simplified to

$$EF = \frac{I_{\text{SERS}}}{I_{\text{Bulk}}} \times \frac{C_{\text{Bulk}}}{C_{\text{SERS}}}. \quad (5)$$

As shown in Fig. 4(j), based on the above data, the EF value of the NdFeB/PS/Ag-FSA magnetic substrate was calculated to be 4.71×10^7 .

To explain this phenomenon, we conducted further magnetic field simulations using finite element analysis method for a rectangular NdFeB magnet sheet that was magnetized in the thickness direction. The results are shown in Fig. 5(a–d). The simulation results show that the magnetic field in the central area of the rectangular magnet sheet is mainly composed of the z -direction component. However, in the edge and corner areas, due to the influence of the limited size boundaries, the magnetic field lines exhibit significant leakage and bending. The magnetic field gradually changes from being dominated by the z -direction to a distribution state with significant x and y direction components. Therefore, the edge region not only has a strong leakage magnetic field, but also the direction and intensity of the magnetic field change rapidly in space,

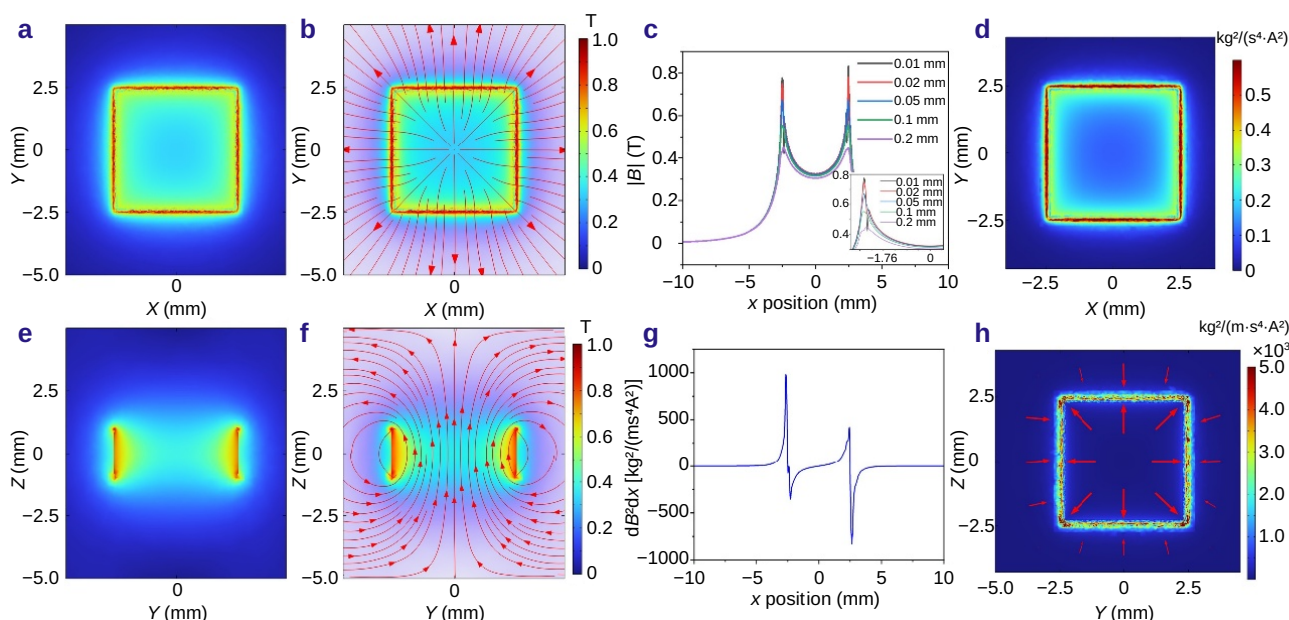


Fig. 5 | The COMSOL simulation results of NdFeB magnets, with the xy plane section located at a distance of 0.01 mm from the upper surface of the magnet, and the yz plane section located at the center of the magnet where $x = 0$. (a) The magnetic field distribution on the xy plane of the magnet surface. (b) Magnetic field line distribution in the xy plane. (c) Along the x -axis direction, the magnetic field intensities at different heights. (d) $\nabla(B^2)$ distribution in the xy plane. (e) The distribution of magnetic field lines in the yz plane. (f) The magnetic field distribution in the yz plane. (g) $\nabla(B^2)$ along the x -axis direction. (h) Schematic diagram of the force and migration direction of magnetic nanoparticles on the surface of a magnet.

exhibiting a more pronounced magnetic gradient.

For the explanation of the migration behavior of magnetic nanoparticles in this magnetic field, the nearly spherical magnetic particles in the low field and in a non-saturated state can be approximately regarded as linear and isotropic magnetic response particles, and their magnetization intensity satisfies the relationship $M = \chi H$. The particles are induced to generate magnetic moments in an external magnetic field, and their magnetic potential energy can be expressed as

$$U_m = -\frac{1}{2}\mu_0 V\chi_{\text{eff}} H^2. \quad (6)$$

Among them, represents the particle volume, and represents the effective magnetization rate after considering the demagnetization effect of the particles. Therefore, the magnetic swimming force acting on the particles is

$$\mathbf{F}_m = -\nabla U_m = \frac{1}{2}\mu_0 V\chi_{\text{eff}} \nabla (H^2). \quad (7)$$

In media such as air, water, or ethanol, which are approximately non-magnetic, there is $B \approx \mu_0 H$. Therefore, this equation can also be written as

$$\mathbf{F}_m = \frac{V\chi_{\text{eff}}}{2\mu_0} \nabla (B^2). \quad (8)$$

From this, it can be seen that the migration direction of the magnetic particles is mainly determined by $\nabla(B^2)$. In three-dimensional space:

$$\nabla(B^2) = \frac{\partial B^2}{\partial x} \mathbf{i} + \frac{\partial B^2}{\partial y} \mathbf{j} + \frac{\partial B^2}{\partial z} \mathbf{k}. \quad (9)$$

Therefore, the components of the magnetic drag force in the three directions can be expressed as

$$\begin{aligned} F_x &= \frac{V\chi_{\text{eff}}}{2\mu_0} \frac{\partial B^2}{\partial x}, \\ F_y &= \frac{V\chi_{\text{eff}}}{2\mu_0} \frac{\partial B^2}{\partial y}, \\ F_z &= \frac{V\chi_{\text{eff}}}{2\mu_0} \frac{\partial B^2}{\partial z}, \end{aligned} \quad (10)$$

among them, F_z mainly affects the adsorption and deposition of particles onto the magnetic surface, while F_x and F_y determine the lateral migration direction of the particles above the magnetic surface. By combining the magnetic field intensity shown in Fig. 5(e–g) and $\nabla(B^2)$, it can be observed that although the central area of the rectangular magnet sheet may have a relatively high normal magnetic induction intensity, the lateral gradient is weak. Therefore, it is difficult to effectively drive the particles to undergo directional migration along the surface. In contrast, the edge and corner areas of the magnet experience significant increases in $\partial B^2/\partial x$ and $\partial B^2/\partial y$ due to the leakage of magnetic field lines, their bending, and the local enhancement of the retreating magnetic field, thereby generating a stronger

lateral magnetic swimming force.

Therefore, on the surface of ordinary rectangular NdFeB magnets, magnetic nanoparticles tend to accumulate towards the edge area. This is mainly due to the finite size boundary, which causes rapid changes in the direction and intensity of the magnetic field at the edge, and results in a stronger lateral magnetic field gradient. This result is consistent with the schematic diagram of particle force and migration direction shown in Fig. 5(h), that is, the larger lateral magnetic propulsion force in the edge area drives the particles to migrate towards the edge of the magnetic plate and undergo enrichment.

The preliminary local detection has already revealed significant positional differences in the SERS response of this magnetic substrate. To systematically characterize this spatial heterogeneity, a 10×10 grid scan was conducted over the entire magnetic substrate (with a step size of $500 \mu\text{m}$), and a heat map of the intensity distribution of the 612 cm^{-1} characteristic peak of 10^{-6} M R6G was plotted, as presented in Fig. S6. The heat map clearly shows that the signal intensity is non-uniformly distributed: the figure demonstrates that the central area of the base presents a significant SERS "hot spot" effect, whereas the edge areas have relatively weaker signals. The overall distribution indicates that the FSA is non-uniformly assembled on the surface of the NdFeB/PS/Ag base, and the distribution driven by the magnetic field gradient is one of the major reasons for the central enhancement.

3.4 Analysis of the SERS enhancement mechanism based on the synergistic effect of magnetic field-particles-hot spots

To further investigate the influence of magnetic substrate morphology on the electrical enhancement properties of Raman signals, we measured the dimensions of the corresponding magnetic substrates based on SEM images. Using finite element analysis, we modelled the SERS substrates for NdFeB/PS/Ag and NdFeB/PS/Ag-FSA, as shown in Fig. 6(a–c), and simulated their electromagnetic field distributions. Ports were set at the top and bottom of the z -axis, with the z -direction serving as the excitation source and an excitation wavelength of 532 nm . As depicted in Fig. 6(d) the hot spots within this model exhibit three-dimensional distribution throughout the entire volume, thereby generating enhanced SERS signals. As one can see at the Fig. 6(e–g), the hot spots mainly originate from the silver components of adjacent magnetic particles on the substrate surface, and between the magnetic particles and the silver layer^{51,52}. This further confirms that the phenomenon of highest Raman intensity in the central region of the substrate is not primarily governed by magnetic particle enrichment, but may be related to the distribution of SERS "hot spots" formed by the PS microsphere array/silver film and the localised electromagnetic field enhancement effect¹⁸.

3.5 Evaluation of fundamental Raman properties

For the quantitative detection performance of the target molecule, the central area configuration of the NdFeB/PS/Ag-FSA magnetic substrate was used to test a series of R6G solutions with different concentrations. Figure 7(a) and 7(b) present the SERS responses of this substrate to a series of analytes at different concentrations: as the content of R6G

in the solution decreases, the SERS signal intensity also drops. However, within a wide concentration range of 10^{-11} to 10^{-6} M, clear Raman characteristic signals can be obtained. The intensity of the 612 cm^{-1} feature peak shows a good linear correlation with the logarithm ($\log C$) of the R6G concentration. The result of linear equation fitting is $I = 1761.33659 \log C_{\text{R6G}} + 19886.85868$ and the program used for calculating LOD is $3\sigma/\text{slop}^{53,54}$. Here, σ represents

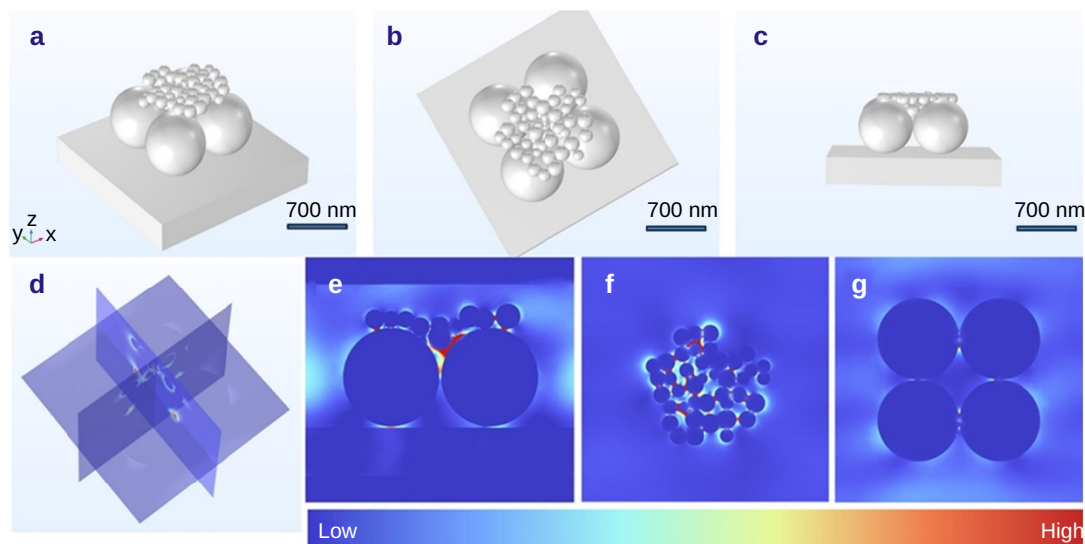


Fig. 6 | (a–c) Three-dimensional simulation model, top view, and side view of a multi-particle assembly structure. (d) Simulated contour map of electric field distribution in a three-dimensional spatial model. (e–f) Localised electric field simulation contour plots for the x-z and x-y planes of NdFeB/PS/Ag-FSA multi-particle assemblies. (g) Simulated contour plot of electric field distribution in the x-y plane for the NdFeB/PS/Ag structure.

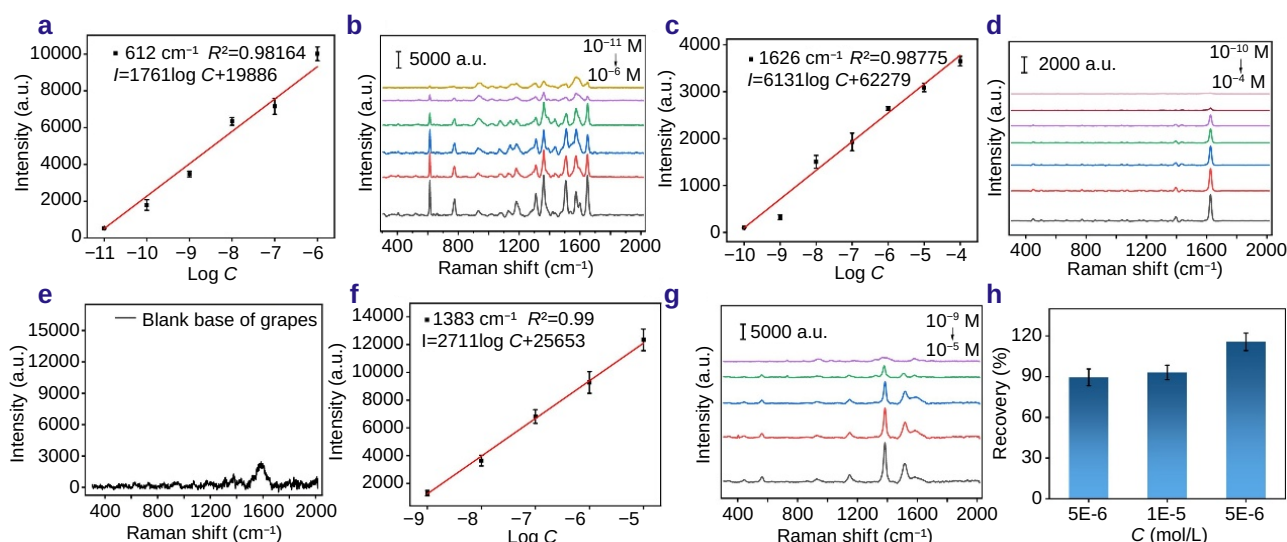


Fig. 7 | (a) Linear relationship between the intensity of the 612 cm^{-1} characteristic peak and the logarithm of R6G concentration ($\log C$). (b) SERS spectra corresponding to different concentrations of R6G solutions (10^{-11} to 10^{-6} M). (c) Linear relationship between the intensity of the 1626 cm^{-1} characteristic peak and the logarithm of MB concentration ($\log C$). (d) SERS spectra corresponding to different concentrations of MB solutions (10^{-10} to 10^{-4} M). (e) SERS spectrum of a blank grape sample. (f) The intensity of the characteristic peak at 1383 cm^{-1} shows a linear relationship with different concentrations of thiram in grapes. (g) SERS spectra corresponding to different concentrations of thiram in grapes (10^{-9} to 10^{-5} M). (h) Recovery rates of three different concentrations of thiram in grapes.

the standard deviation of the blank sample signal, while $\text{slop a.u.}/(\text{mol/L})$ is the slope of the standard curve. Its detection limit is 10^{-10} M, the LOD is calculated to be 10^{-11} M. This indicates this base has a certain detection capability for low-concentration target molecules. At the same time, within this experiment we also carried out a series of concentration SERS responses for MB: as shown in Fig. 7(c), a linear relationship was established between the intensity of the MB characteristic peak and the logarithm of the corresponding concentration. Its concentration is 10^{-11} mol/L, as shown in Fig. 7(d).

In addition, this experiment helped to verify the reliability of the NdFeB/PS/Ag-FSA magnetic substrate that was established for detecting complex samples. The detection of thiram in grape samples was carried out⁵⁵. First, carried out preliminary processing on the commonly available fresh grapes on the market: extract its juice, perform centrifugation, and collect the supernatant. Use this grape juice as the solvent. Take the known blank grape samples for Raman detection, as shown in Fig. 7(e). Then, a series of thiram standard solutions were prepared, and a linear fitting was performed on the characteristic peak at 1383 cm^{-1} of thiram (C-N stretching vibration) as shown in Fig. 7(f–g). To verify the actual application performance of this base in real samples, the method was evaluated through the addition recovery rate experiment, added $5\text{ }\mu\text{L}$, $10\text{ }\mu\text{L}$, and $50\text{ }\mu\text{L}$ of 10^{-2} M of thiram to 10 mL of grape juice for SERS detection and calculate the recovery rate. The respective results are

shown in Fig. 7(h). The application potential of this substrate in the detection of actual samples was verified through three concentration systems.

A random area was selected on the NdFeB/PS/Ag-FSA substrate as shown in Fig. 8(a), and a $5\text{ }\mu\text{m} \times 5\text{ }\mu\text{m}$ (step size of $1\text{ }\mu\text{m}$) R6G concentration of 10^{-6} M SERS detection was implemented. Among the 25 test points, the SERS spectral intensity did not change significantly. We quantitatively evaluated their uniformity using the relative standard deviation (RSD) value: as shown in Fig. 8(b), the RSD of the SERS peak at 612 cm^{-1} was 8.79%, and in Fig. 8(c), the RSD of the SERS peak at 775 cm^{-1} was 16.05%, indicating that the NdFeB/PS/Ag-FSA magnetic substrate was relatively uniform.

Figure 8(d) displays the time-dependent intensity curve of the 612 cm^{-1} characteristic peak for 10^{-6} M R6G over 27 days on the self-fabricated NdFeB/PS/Ag-FSA magnetic SERS substrate. Test conditions: room temperature (25 ± 2) °C, stored in the dark and sealed contain, with sampling every 3 days. Measurements were repeated three times in the same microarea and averaged. As shown, the substrate maintained stable signal intensity during the first 3 days of storage, with an average intensity of (9600 ± 400) a.u., implying excellent short-term repeatability. At day 9, the intensity began a gradual decline, reaching 7700 a.u. by day 27, corresponding to a retention rate of 80.4%. This demonstrates good temporal stability, meeting the core requirement for "long-term stable operation" in practical detection scenarios.

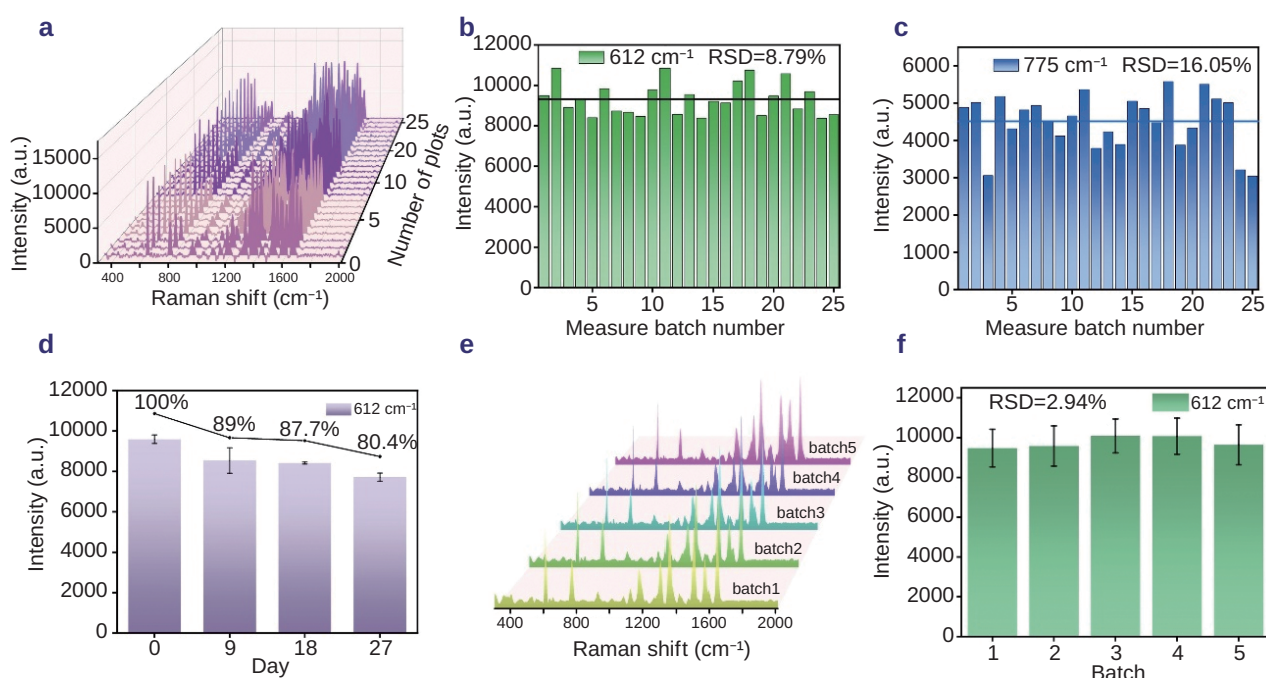


Fig. 8 | (a) SERS spectra of 10^{-6} M R6G at 25 points on the magnetic substrate NdFeB/PS/Ag-FSA. (b–c) SERS intensity distribution and corresponding RSD values at 612 cm^{-1} , 775 cm^{-1} in the measurement of (a). (d) SERS spectra of 10^{-6} M R6G after storage for different times (0–27 days) on the NdFeB/PS/Ag-FSA magnetic substrate. (e–f) The SERS spectra after R6G molecules were adsorbed on five batches of NdFeB/PS/Ag-FSA magnetic SERS substrates intensity values at 612 cm^{-1} .

To evaluate the reproducibility of this preparation method, five different batches of NdFeB/PS/Ag composite SERS magnetic substrates were prepared and their SERS spectra were measured, as shown in Fig. 8(e). The RSD of the intensity of the characteristic peak at 612 cm^{-1} was 2.94% (Fig. 8(f)), indicating that this preparation method has good batch reproducibility.

4 Conclusions

Overall, in this paper we demonstrate the construction of a NdFeB/PS/Ag composite SERS magnetic substrate, the NdFeB magnet sheets are magnetized. Due to the fact that the magnetic flux density of the NdFeB sheets increases radially from the center outward, this radial increase in magnetic field couples with the magnetic response of the FSA magnetic nanoparticles, achieving magnetic control enrichment and ordered assembly of the particles in the detection area. This was enabled by leveraging the synergistic plasmonic effect between the PS/Ag structure and the outer Ag layer of FSA, thus showing the successful development of a SERS detection system with magnetic control capabilities. The experiment shows that the intensity of the R6G characteristic Raman peak in the central region is approximately 2.6 times higher than that in the edge region. With the optimization of the magnetization gradient, the plasma coupling effect of FSA magnetic particles at the center region of the NdFeB/PS/Ag magnetic substrate is maximized, and the Raman signal enhancement efficiency reaches its highest value. This strategy can reconfigure the spatial distribution of the magnetic field through the flexible design of the magnetization mode. Meanwhile, the particle-substrate magnetic attraction induced by the magnetic gradient and the magnetic dipole-dipole interaction between particles show a beneficial synergistic action, enhancing the dispersion of FSA magnetic particles in the central area of the substrate: in this manner, we achieved a significant boosting of the "hot spot" density and the electromagnetic coupling efficiency, thus offering a new idea for the elaboration of portable and self-contained active SERS detection platforms.

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Author contributions

Na Gao: conceptualization, data curation, writing-original draft, writing-review & editing. Li Xifeng: data curation, software. Mingxuan Wang: data curation, software. Sheng Li: methodology. Liu Wang: methodology. Chenpeng Zhang: methodology. Mingli Wang: formal analysis, funding acquisition, resources, supervision.

Competing interests

The authors declare no conflict of interest.

Supplementary information

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