RESEARCH ARTICLE

Swift reduction of nitroaromatics by gold nanoparticles anchored on steam-activated carbon black via simple preparation

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Abstract
Gold (Au) nanoparticles supported on certain platforms display highly efficient activity on nitroaromatics reduction. In this study, steam-activated carbon black (SCB) was used as a platform to fabricate Au/SCB composites via a green and simple method for 4-nitrophenol (4-NP) reduction. The obtained Au/SCB composites exhibit efficient catalytic performance in reduction of 4-NP (rate constant \( k_{\text{app}} = 2.1925 \text{ min}^{-1} \)). The effects of SCB activated under different steam temperature, Au loading amount, pH, and reaction temperature and NaBH\(_4\) concentration were studied. The structural advantages of SCB as a platform were analyzed by various characterizations. Especially, the result of \( \text{N}_2 \) adsorption–desorption method showed that steam activating process could bring higher surface area (from 185.9689 to 249.0053 \( \text{m}^2/\text{g} \)), larger pore volume (from 0.073268 to 0.165246 \( \text{cm}^3/\text{g} \)), and more micropore for SCB when compared with initial CB, demonstrating the suitability of SCB for Au NP anchoring, thus promoting the catalytic activity. This work contributes to the fabrication of other supported metal nanoparticle catalysts for preparing different functional nanocomposites for different applications.

Keywords Carbon black · Steam activation · Au nanoparticles · Nitrophenols · Catalytic reduction

Introduction
As raw materials for pesticides, explosives, and other chemical products, nitroaromatics are widely used in the chemistry industry, which could certainly make the environmental pollution, especially water pollution, thus posing threat towards biology (Chen et al. 2022; Ye et al. 2022). For example, 4-nitrophenol (4-NP), a kind of nitroaromatics, is common toxic and in urgent need of removal (Cao et al. 2020; Fu et al. 2019a). Among the removal methods, catalytic reduction of 4-NP to 4-aminophenol (4-AP) is a suitable method, which can decrease the toxicity of 4-NP and simultaneously produce the added-value by-product 4-AP for many chemical synthesis, thus bringing economic benefits. With abundant explorations, the catalysts for 4-NP reduction are tremendously developed in recent years, especially supported gold nanoparticle (Au NP) catalysts (Neal et al. 2020; Zhang et al. 2020). Although the super high catalytic activity can be achieved by supported Au NP catalysts, the high cost and limited resource of Au hinder the practical application (Tan and Tan 2020). Hence, it is important to design a supported Au NP catalyst with excellent catalytic activity and desired cost.

As for catalysts platforms, carbon materials with many properties of well electrical conductivity, stable mechanical performance, easy modification, and low cost are widely used for Au NP anchoring (Ferry et al. 2015; Qin et al. 2019a). In our group, we have been working on ways to develop a sequence of carbon-supported Au NP catalysts, which are with suitable cost and exhibit efficient catalytic performance in reduction of 4-NP (Fu et al. 2019a, 2019b; Qin et al. 2019a, 2019b, 2019c). For instance, we employed...
hierarchical porous carbon black (CB) as supporting materials to prepare supported Au NP catalysts, on account of CB with low cost and hierarchical porous structure (Qin et al. 2019b; Wang et al. 2011). The results show that the rich pore structure of CB is conducive to restricting Au NPs in the pore structure, which improves the reuse ability and stability of Au NP catalysts. In addition, the graphite-like mixed layer structure of CB can produce π-π stacking interaction, thus adsorbing 4-NP for reaction. Compared with Au NP catalysts supported by other carbon materials (carbon nanotubes, activated carbon, and activated coke), the Au NPs supported by CB exhibits well catalytic activity (Qin et al. 2019b). In addition, to further promote the activity, stability, and dispersibility of Au NPs on CB surface, N-doped and oxygen-containing groups were introduced into CB by modifying with HNO₃. The catalytic performance of the prepared bimetallic HCB-Ni-Au catalysts was certainly enhanced compared with previous study (Qin et al. 2019c). Overall, it is obvious that CB is an ideal platform for Au NP anchoring.

The existing study has proved that Au NPs supported on CB catalysts exhibit potential for catalytic 4-NP reduction. But considering the secondary pollution to environment caused by HNO₃ modification and high toxicity of other introduced metal such as Ni, it is vital to develop an environmentally friendly method to functionalize and activate CB as a better platform for Au NP anchoring. Recently, it has been reported that steam can effectively activate carbon materials, which increases the specific area and pore volume, thus promoting the adsorption ability towards pollutants and thus accelerating catalytic reaction (Rajapaksha et al. 2015; Zhao et al. 2007). Lima and Huang et al. used steam to activate biomass by adjusting the steam temperature, thus effectively increasing the specific area and pore volume of biomass, and the steam activated-biomass exhibited efficient adsorption ability towards Cu²⁺ and tetracycline (Lima et al. 2010; Wang et al. 2020). In the research of Shcherban et al., they also used steam to activate carbon materials (Shcherban et al. 2014). The characterization results display that the steam-activated carbon materials possess increased specific surface area and higher adsorption capacity of hydrogen. Hence, it can be expected that steam activation is a green and simple method to modify CB to increase the specific surface area and provide more active sites for Au NP anchoring, thus promoting the catalytic activity. In addition, it has been reported by our group that ascorbic acid (AA) can serve as a mild reducing agent for Au³⁺ reduction (Fu et al. 2019b).

Thus, we expect to fabricate Au NPs deposited on steam-activated CB (Au/SCB) via a green, simple, and low-cost method, using AA as reducing agent. The obtained composites are applied in the model reaction of 4-NP reduction. The main experimental contents of this study include (i) characteristic analysis of SCB compared with initial CB and discussion of the effect of steam temperature; (ii) study on the catalytic activity of Au/SCB composites and some experiment influencing factors; (iii) investigation on the catalytic principle for 4-NP reduction over the Au/SCB composites; and (iv) comparison with previous studies.

**Experimental**

**Materials**

Commercial spherical Vulcan XC-72 carbon black was purchased from Nanjing XFNANO Materials Technology Corporation. Hydrogen tetrachloroaurate hydrate (HAuCl₄·4H₂O); ascorbic acid (AA); nitrophenols including 2-nitrophenol (2-NP), 3-nitrophenol (3-NP), 4-nitrophenol (4-NP), and 2,4-dinitrophenol (2,4-DNP); and azo dyes including Methyl orange (MO), Congo red (CR), and Eriochrome Black T (EBT) were both proposed from Sinopharm Chemical Reagent Corporation Limited.

**Synthesis of steam-activated carbon black depositing Au NPs (Au/SCB)**

Steam-activated carbon black (SCB): Commercial spherical Vulcan XC-72 carbon black was firstly purified by ultrapure water. The above samples were activated by steam for 45 min in tube furnace the peak temperature of 300°C, 400°C and 500°C with 5°C min⁻¹ heating rate, and the obtained composites were denoted as T-SCB (T = 300°C, 400°C, and 500°C).

Steam-activated carbon black supported Au NPs (Au/SCB): 0.2 g SCB was added to 100 mL of ultrapure water with ultrasound of 0.5 h. Then, 0.5 ~ 2.5 mL of HAuCl₄·4H₂O (24.28 mM) was injected into SCB suspension with stirring for 0.5 h. After that, 10 mL of AA (0.1 M) was dropwise appended into the above solution with stirring for 24 h under ambient environment. The resulting samples were collected after washing and drying overnight for further use, which were marked as xAu/T-SCB (x = 0.5, 1, 1.5, 2, and 2.5; T = 300°C, 400°C, and 500°C). In addition, Au/initial CB was prepared through the same method.

**Characterization**

The X-ray diffraction (XRD) was conducted on a 6100 powder diffractometer (Shimadzu Corporation, Japan). Transmission electron microscopy (TEM) was conducted on Tecnai G² F20. The Au dispersion was calculated according to the formula as shown in supporting information reported by Ye et al. (Wang et al. 2018). Raman spectroscopy was performed on HORIBA JY LabRAM HR Evolution. Fourier transform-infrared spectroscopy (FT-IR) was collected on Perkin Elmer spectrum 100. Nitrogen adsorption isotherms were obtained by using BET surface area apparatus on
Micromeritics ASAP 2460 at 423 K. X-ray photoelectron spectroscopy (XPS) was performed on apparatus of Thermo Scientific. Zeta potential was tested using a Zeta-sizer Nano-ZS (Malvern). The UV–Vis absorption spectra were monitored using UV-2700 spectrophotometer purchased from Shimadzu Corporation. The leaching amount of Au was tested by inductively coupled plasma optical emission spectrometer (ICP-OES).

**Catalytic experiments**

Firstly, 10 mg xAu/T-SCB composites were decentralized in 50 mL 4-NP (0.2 mM) and then mixed for 30 min to realize the equilibrium of adsorption-desorption. Next, 0.0757 g NaBH₄ \( (C_{4-NP}/C_{NaBH_4} = 1/200) \) was introduced to the above solution. The solution was sampled at specific intervals time and filtrated by 0.45-μm syringe filter and then monitored on a UV–vis spectrometer. The catalytic reduction of various nitrophenols and azo dyes (0.3 mM) was also performed at same reaction conditions.

In the recycling experiment, the syringe filter containing the used composites was unpicked and washed and then with ultrasound for 1 h in ultrapure water. Afterwards, the used composites were gathered by suction filtration and washed with ultrapure water for three times and then dried in vacuum at 60°C for 24 h for the next recycling reused.

**Results and discussion**

**Characterizations**

Firstly, XRD was measured to determine the crystal structure. As shown in Fig. 1a, the typical diffraction peaks of SCB at 20 = 25° and 44° attribute to the plane (002) and (101) of carbon, which is identical with the previous results (Qin et al. 2019b, 2019c). It can be initial inferred that the crystal structure of CB would not be affected by the steam. The diffraction peaks of 2Au/500°C-SCB centered at 38.1°, 44.3°, 64.5°, and 77.5° can be ascribed to the plane (111), (200), (220), and (311) of face-centered cubic (fcc) Au (JCPDS No.04–0784), respectively. It can be noticed that the peak intensity attributed to plane (002) of SCB decreases in 2Au/500°C-SCB sample, because the Au NPs may weaken
the internal structure order of SCB (Qin et al. 2019b; Xia et al. 2016).

TEM and HRTEM characterizations were conducted to study the morphology and microstructure of 500°C-SCB and 2Au/500°C-SCB composites. As shown in Fig. 1b, it can be observed that SCB has a typically spherical morphology with the size about 30 nm, but it tends to aggregate, thus constituting the stacking and aggregated platforms because of the high surface energy and van der Waals forces (Qin et al. 2019b). As for the morphology of 2Au/500°C-SCB composites shown in Fig. 1c–e, it can be preliminary estimated that Au NPs with size of ~65 nm are deposited on the surface of stacking and aggregated SCB, thus forming Au NP/SCB composites, indicating that CB activated by steam can provide some active sites on the surface for Au anchoring (Wang et al. 2011). HRTEM image (Fig. 1e) displays the distinct lattice fringes of 0.235 nm, ascribed to the plane (111) in fcc Au NPs, further demonstrating that the Au NPs were resoundingly immobilized on the 500°C-SCB surface (Fu et al. 2019a). Figure 1f displays the STEM image of 2Au/500°C-SCB composites. Interestingly, it can be found that except large-sized Au NPs (~65 nm) deposited on the surface of stacking and aggregated SCB, the small-sized Au NPs (~5 nm) are also observed in the composites. As shown in Fig. 1e, Au NPs with size about 5 nm can be clearly observed in HRTEM. The formation of small-sized Au NPs may be because the plentiful hierarchical porous texture and high specific area of SCB favor AuCl₄⁻ entering to the hierarchical porous structure during stirring process of preparation, thus nucleating small-sized Au NPs when the reductant AA is added into solution (Qin et al. 2019c). Based on the formula reported by Ye et al. (Wang et al. 2018), it can be calculated that the dispersion of Au is 1.8%, which is unimpressive when compared with other Au NP-based catalysts.

Next, Raman and FT-IR spectroscopy were used to provide more information on the crystallinity and surface groups of samples. As shown in Fig. 2a, two characterization peaks at 1345 cm⁻¹ attributed to D band and 1560 cm⁻¹ attributed to G band appear in the Raman spectrum of initial CB (Reddy et al. 2020; Revathy et al. 2018). The D band...
indicates the disorder or defect in this lattice, and G band indicates the “in-plane” vibrations. Usually, the ratio of $I_D/I_G$ is used to represent the degree of disorder in the graphitic material (Revathy et al. 2018). The $I_D/I_G$ ratio of initial CB and SCB is calculated to be 1.06 and 1.16, respectively. Obviously, the $I_D/I_G$ ratio of SCB increases when compared with initial CB, representing more defects may be brought into SCB after steam activating (Song et al. 2014). After loading Au NPs, as for 2Au/500°C–SCB, the $I_D/I_G$ ratio decreases to 1.07 from 1.16. The value of $I_D/I_G$ ratio for CB, SCB, and 2Au/500°C–SCB increases firstly after declining, indicating the depositing Au NPs makes a difference to SCB. Combing with TEM results (Fig. 1e and f), it can be inferred that the nucleation of Au NPs with size about 5 nm at SCB surfaces fills up a part of defect sites on SCB surface (Ballesteros et al. 2008), thus causing the decreased $I_D/I_G$ of 2Au/500°C–SCB. In addition, the $I_D/I_G$ of Au NPs deposited on initial CB (Au/initial CB) is calculated to be 1.38, which is higher than that of initial CB. Different from the $I_D/I_G$ change of initial CB and Au/initial CB, the $I_D/I_G$ of 2Au/500°C–SCB is below that of 500°C–SCB, further indicating the occupation defects of Au NPs with about 5 nm on SCB surface. Furthermore, FT-IR spectra provide the information about surface chemistry as shown in Fig. 2b. All spectra of samples display a peak at 3680–3080 cm$^{-1}$, ascribed to C–OH (Fu et al. 2019a, 2019b). The peak at 1626 cm$^{-1}$ is associated to C=C bonds or characteristic of condensed aromatic structures (Shcherban et al. 2014). In addition, the peaks around 1170–1000 cm$^{-1}$ and 695–554 cm$^{-1}$ are responsible for stretching and plane bending vibrations of C–H bonds (Fu et al. 2019a; Qin et al. 2019b). Compared with initial CB that we have test the characterization in other study, there is no characteristic band in the defects were brought by steam activation (Wang et al. 2010). Remarkably enough, the larger specific surface area may be owing to more pores from stacking together, and the decreased average pore size in 500°C–SCB could be ascribed to the large amount of emerged micropores (Li et al. 2021). After loading Au NPs, the specific area and pore volume of 2Au/500°C–SCB display a little decrease, which may be because the Au NPs occupy a portion of surface and pore channel of 500°C–SCB, in accord with the results of TEM analysis.

To further understand the effect of steam activation on samples, XPS was performed to analyze the chemical composition and chemical bonds over the initial CB, 500°C–SCB, and 2Au/500°C–SCB. The full XPS spectra presented in Fig. 3a display the compositional elements of them, illustrating C and O elements are the dominant species in each sample. As shown in Fig. 3b, the high-resolution C 1 s spectra are deconvolved into four peaks with binding energy (BE) at 284.6, 285.8, 286.9, and 289.8 eV, ascribed to sp$^2$ carbon (graphitic C=C), sp$^3$ carbon (hydrocarbon C–C), C–O, and π-π transition loss (Wang et al. 2011; Xia et al. 2016). The relative ratios (%) of four species for initial CB, 500°C–SCB, and 2Au/500°C–SCB are listed in Table S2. It is obvious that the relative percentage of C=C decreases to 55.59% from 66.1%, and C–C increases to 28.35% from 9.83% after steam activation. The results are consistent with the discussion in the section of Raman analysis that more defects are brought into SCB after steam activation, owing to the etch of crystal structures. In addition, the relative percentage of C–O increases to 7.91% from 6.43% after steam activation. As shown in Fig. 3c, the percentage of O also increases for SCB, corresponding to the results of increased percentage of C–O. After loading Au NPs, the high-resolution spectrum of C 1 s displays that the percentage of C–O still increases to 16.81%. According to the previous reports by Xie et al., it could be associated with the improved uniformity of Au NPs on SCB because the C–O could serve as active sites.
with high charge density on the oxygen sites (Wang et al. 2011). The Au 4f spectrum shown in Fig. 3d displays the typical characteristic peaks at 83.2 and 86.9 eV, displaying Au 4f7/2 and Au 4f5/2 of Au0 (the difference value between the two peaks is 3.7 eV). The additional peaks at the high BE side are also found, attributed to oxidized Au+ species (Qin et al. 2021, 2019c).

Catalytic performance of Au/SCB composites for nitrophenol reduction

Catalytic performance for 4-NP reduction

The reaction of 4-NP reduced to 4-AP is thermodynamically feasible (E° = −0.76 V) at normal conditions with NaBH4 serving as reductant (E° = −1.33 V) (Zhang et al. 2021). However, the reaction progress is hard to proceed without catalysts, owing to the kinetic barrier brought from large potential difference between 4-NP and NaBH4. But the barrier could be got over through Au NP-based composites, and the reaction progress is readily detected by UV–vis spectrometer (Jiang et al. 2021). Hence, Au/SCB composites were used for catalytic 4-NP reduction. Firstly, adsorption experiments of 4-NP by SCB and Au/SCB composites were conducted. Figure S1a–b shows that the peak intensity at 316 nm of 4-NP decreases, but there is no new peak, demonstrating the adsorption ability of SCB and Au/SCB composites. Figure S1c displays that the absorbance peak of 4-NP shifts from 316 to 400 nm after adding NaBH4, illustrating 4-nitrophenolate ions are formed (Fu et al. 2019a; Qin et al. 2019c). When adding 2Au/500°C-SCB composites, the peak intensity at 400 nm slowly weakens with the reaction proceeding and could be detected no longer after 3 min, indicating the 4-NP reduction. Meanwhile, a new peak occurs in the XPS spectra.
at 300 nm, indicating the formation of 4-AP (Fig. 4a). In addition, the color of reactants gradually fades. The NaBH₄ concentration is 200 times as 4-NP concentration, so the catalytic 4-NP reduction can be regarded as pseudo-first-order kinetic reaction. After simulating, the rate constant $k_{app}$ was obtained (2.1925 min⁻¹), displaying excellent catalytic activity of 2Au/500°C-SCB composites (Fig. 4b). In addition, Au/initial CB prepared in the same method was also used to catalytic 4-NP reduction. As shown in Fig. S1d, 4-NP reduction catalyzed by Au/initial CB finished within 6 min and the rate constant $k_{app}$ was 0.7284 min⁻¹, illustrating the catalytic activity of Au/initial CB is not as good as Au/SCB (3 min), which highlights the advantages of steam treatment.

Effect of steam activating temperature and Au loading amount

According to the previous study, it is known that the temperature of steam activation may impact on the structure of SCB, thereby influencing the catalytic activity of Au/SCB composites. Besides, the loading amount of Au is also an important parameter for the Au-based composites. Hence, the effects of steam activation temperature, and Au loading amount were investigated. As shown in Fig. S2a-c, all the xAu/T-SCB composites ($x = 0.5, 1, 1.5, 2, 2.5; T = 300°C, 400°C, and 500°C$) can successfully catalytic 4-NP reduction 4-NP (Fig. S2d–f). As evidenced in the kinetic rates, the catalytic performance of different xAu/T-SCB composites is closely related to the steam activating temperature and Au loading amount. Obviously, the catalytic performance is promoted as the steam activation temperature increasing from 300 to 500°C. Among the Au/SCB composites prepared in different temperature, the Au/500°C-SCB displays the highest catalytic activity for 4-NP reduction. It might be because that the higher temperature is beneficial for superheated steam diffusing, thus resulting in high specific area and abundant pore structure, especially micropore, which is demonstrated by the BET characterization (Table S1). The abundant pore structure is conducive to concentrating 4-NP, thus facilitating the catalytic activity (He et al. 2020). It should be noticed that the water vapor activation temperature would affect the specific surface area, graphitization degree, pore structure, and surface functional groups of activated carbon materials (Kan et al. 2015). Hence, not just 300–500°C, higher treatment temperature for carbon materials as metal platforms should be a matter of concern in the future laboratory work.

As for different Au loading amount, all the composites with 2 mL of Au loading amount prepared under different temperature exhibits higher catalytic activity. Figure S2d–f displays the -ln ($C_t/C_0$) vs. time of 4-NP reduction over xAu/T-SCB composites ($x = 0.5, 1, 1.5, 2, 2.5; T = 300°C, 400°C, and 500°C$). With Au loading amount increasing, the catalytic rate firstly increases and then decreases. It might be because the active sites increase with Au content increasing, while the active sites would decrease caused by Au NP agglomeration with Au content further adding (Jiang et al. 2021). The rate constant $k_{app}$ is 1.4869, 2.0382, and 2.1925 min⁻¹ for 2Au/300°C-SCB, 2Au/400°C-SCB, and 2Au/500°C-SCB, respectively. Overall, considering the catalytic efficiency and economic cost, 2Au/500°C-SCB exhibits better performance, thus employed in the following study. Actually, on account of the high cost and limited abundance of Au, the principle of “less is more” should also be considered. Hence, in the future work, we would keep

![Fig. 4](image-url)

**Fig. 4** a Time-dependent UV–Vis absorption spectra of 4-NP reduction over 2Au/500°C-SCB, and b plots of $C_t/C_0$ and -ln ($C_t/C_0$) vs. time. Reaction conditions: $C(4$-NP$) = 0.2$ mM, $C(NaBH_4) = 40$ mM, $m(2Au/500°C-SCB) = 10$ mg, initial pH = 5.62, and room temperature.
our eyes open minimizing Au usage and systematically study and balance the relationship between the Au usage and catalytic activity.

Effect of initial pH

The effect of pH in this catalytic system was investigated at initial pH 2–9. As shown in Fig. 5a, it is obvious that initial pH can significantly affect the catalytic activity of 2Au/500°C-SCB, and the acidic pH condition is beneficial for promoting the catalytic efficiency. With the initial pH increasing from 2 to 9, the catalytic activity decreases to 0.4022 min⁻¹ from 3.8387 min⁻¹ (Fig. 5b). It has been reported that the effect of pH in this catalytic system is closely associated with the pHIEP of 2Au/500°C-SCB and pKₐ of 4-NP (Nellaiappan et al. 2017, Nguyen et al. 2019). Zeta potential of the 2Au/500°C-SCB was measured to better understand the influence. Figure S3 shows the Zeta potential of 2Au/500°C-SCB with a decreasing trend from positive potential to negative potential. The measured pHIEP of 2Au/500°C-SCB is at about pH 4, indicating that the composites are positively charged at pH < 4. It can been known that the catalytic 4-NP reduction by 2Au/500°C-SCB in the presence of excessive NaBH₄ conforms to Langmuir–Hinshelwood kinetic model, in which the primarily step procedure is adsorption for catalytic reduction reaction (Qin et al. 2019c). Hence, the BH₄⁻ with negative charge can be readily gathered round the 2Au/500°C-SCB surface with positive charge at acidic condition, thus leading to an improved rate constant. In addition, protons in solution can easily bond with BH₄⁻ to produce H₂ at acidic condition, promoting the formation of activated H and thus enhancing the catalytic activity (Lin and Doong 2014). The results are in well accord with the previous study of Lin and Doong et al. that low pH condition is conducive to accelerating the catalytic efficiency (Lin and Doong 2014, Nguyen et al. 2019). When pH increases over 5, the form of 4-NP with pKₐ of 7.2 is anionic. It means that 4-NP anions can be not easily adsorbed onto the

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**Fig. 5** a Plots of C/C₀ and b -ln (C/C₀) vs. reaction time of 4-NP reduction under different pH conditions. c Plots of C/C₀ and d -ln (C/C₀) vs. reaction time of 4-NP reduction under different reaction temperature (T = 20°C, 40°C, and 60°C) over 2Au/500°C-SCB composites. Reaction conditions: C(4-NP) = 0.2 mM, C(NaBH₄) = 40 mM, m(2Au/500°C-SCB) = 10 mg
negatively charged 2Au/500°C-SCB surface on account of the electrostatic repulsion, thus decreasing the catalytic efficiency (He et al. 2020). The results reflect that the adsorption process is vital for the catalytic reaction, which would be focused on following discussion about the catalytic mechanism.

**Effect of reaction temperature**

Reaction temperature on 4-NP reduction was also studied from 20 to 60°C. Figure 5c–d displays that the catalytic rate of 4-NP reduction increases with temperature increasing, with the rate constant increasing from 0.8952 min⁻¹ at 20°C to 2.7531 min⁻¹ at 60°C. According to the previous study, increasing temperature can accelerate decomposition of NaBH₄ and generation of activated H on catalyst surface, thus enhancing the catalytic activity (Ozerova et al. 2020). Furthermore, activation energy (Eₐ) can be used to reveal the relation and dependency between the temperature and rate constant in catalytic reactions (Shin et al. 2012; Wang et al. 2021), which is calculated based on the Arrhenius equation (Eq. (1)).

\[
\ln k = \ln A - \frac{E_a}{RT}
\]

in which \(A\) and \(T\) display pre-exponential factor and temperature respectively, and \(R\) indicates gas constant (8.314 JK⁻¹mol⁻¹). As shown in Fig. S4, the linear fitting of \(\ln k\) vs. \(1/(T \times 10^3)\) is obtained, in which \(E_a\) can be figured from the slope (-\(E_a/R\)) (Cui et al. 2020). Hence, the \(E_a\) of 2Au/500°C-SCB for reducing 4-NP is calculated as 23.235 kJ/mol. It is approximate with the \(E_a\) of other reported Au-based catalysts, indicating the well activity of 2Au/500°C-SCB for 4-NP reduction. Generally, the \(E_a\) in the range of 8 – 42 kJ/mol could be ascribed to surface catalyzed reactions. Hence, it can be concluded that the catalytic 4-NP reduction over 2Au/500°C-SCB fits surface catalytic mechanism (Bogireddy et al. 2020; Cao et al. 2020; Cui et al. 2020; Wang et al. 2021).

**Effect of NaBH₄ concentration**

Then, the effect of NaBH₄ concentration was investigated on 4-NP reduction at the different ratio of \(n(4-NP)\) to \(n(NaBH₄)\), such as 1/50, 1/100, 1/150, 1/200, 1/250, and 1/300. As shown in Fig. 6, the catalytic activity of 2Au/500°C-SCB gradually increases with NaBH₄ concentration increasing. The enlarged figure from Fig. 6b shows the rate constant \(k_{\text{app}}\) for 4-NP reduction increases from 0.6054 to 2.8674 min⁻¹ with \(n(4-NP):n(NaBH₄)\) ratio increasing. It may be because higher concentration of NaBH₄ make that more BH₄⁻ take up on the surface of catalysts and consequently promote the catalytic process (Ren et al. 2021). It is obvious that the catalytic activity of 2Au/500°C-SCB increases significantly under the \(n(4-NP):n(NaBH₄)\) ratio of 1/200 (the NaBH₄ concentration = 40 mM), possibly because this dosage of NaBH₄ is enough to trigger the reaction with fixed 4-NP concentration (Qin et al. 2019a). Hence, the NaBH₄ concentration of 40 mM can be considered as the optimum dosage and selected during the whole experiment.

**Catalytic activity for isomers and homologues of 4-NP**

To manifest the generality of 2Au/500°C-SCB composites and figure out the impacts of substituent groups, catalytic reduction of isomers of 4-NP such as 2-NP

![Fig. 6](image)

**Fig. 6** a Plots of \(C/C_0\) and b \(-\ln(C/C_0)\) vs. reaction time of 4-NP reduction catalyzed by 2Au/500°C-SCB composites under different NaBH₄ concentrations. Reaction conditions: \(C(4-NP) = 0.2\) mM, \(m(2Au/500°C-SCB) = 10\) mg, and room temperature.
and 3-NP and homologues including 2, 4-DNP was investigated under the same reaction conditions. As shown in Fig. S5a–c, the 2Au/500°C-SCB composites exhibit satisfactory catalytic activity for various nitrophenols. The absorbance peaks attributed to 2-NP (414 nm), 3-NP (390 nm), and 2, 4-DNP (443 nm) gradually weakens after adding 2Au/500°C-SCB composites (Fu et al. 2019b), and all the color of reactants fades from natural color. Figure S5d displays that 2-NP and 3-NP can be totally reduced within 3 min, and the catalytic reduction of 2, 4-DNP is accomplished within 4 min. The different catalytic efficiency for isomers and homologues of 4-NP may be attributed to the molecular orientation and number of nitro-substituent, which has been reported in our previous study (Fu et al. 2019b).

Catalytic activity for azo dyes

In addition to nitrophenols, azo dyes involving azo bond (–N=N–) in wastewater could also bring serious threat to environment and human health. The catalytic reduction of –N=N– over metal NPs catalyst has proved an efficient method to cleavage –N=N–, thus reducing its hazard (Zhang et al. 2021). Hence, some typical azo dyes such as MO, CR, and EBT were selected as target pollutants to further study the catalytic universality of 2Au/500°C-SCB composites. Figure S6 shows that the peak intensity of MO (463 nm), CR (497 nm), and EBT (530 nm) decreases as the reaction proceeding with the decolorization of reaction solution, until it can be detected no longer. Simultaneously, a new peak occurs at ~250 nm, illustrating the formation of new colorless products. The new peak may be ascribed to derivatives of azo dyes after –N=N– splitting (Fu et al. 2019a). Obviously, the catalytic efficiency of MO and CR reduction was higher than that of EBT, possibly because 2Au/500°C-SCB exhibits different adsorption capacities towards various azo dyes, and different azo dyes possess different structures (Qin et al. 2019c). Even so, the 2Au/500°C-SCB composites exhibit well catalytic activity in various azo dye reductions.

Recycling stability of catalyst

As shown in Fig. 7a, the 2Au/500°C-SCB composites exhibit well stability with no obvious deactivation after 5 recycle experiments. But the rate constant $k_{app}$ for 4-NP reduction decreases when the composites are reused. It may be caused by the –NH$_2$ on the 4-AP products bonding with Au NPs and occupying the active sites. It has been known that –NH$_2$ could strongly bind with Au NPs, therefore blocking the catalytic sites on Au NPs. To verify the assumption of surface blocking by –NH$_2$, 4-AP was used to pretreat the 2Au/500°C-SCB composites prior the reduction of 4-NP. Figure 7b displays that the catalytic 4-NP reduction over the 4-AP pretreated 2Au/500°C-SCB composites is complete within 11 min, and the rate constant $k_{app}$ decreases to 0.3765 min$^{-1}$ from 2.1925 min$^{-1}$, verifying the assumption of surface blocking by –NH$_2$. In addition, the weak deactivation of catalysts could be ascribed to the leach of Au. Through ICP-OES test, the leaching amount of Au was 5.23% after 5 cycles, thus reducing the active sites and decreasing the catalytic activity. As shown in Fig. S7, TEM images...
of the used Au NP/SCB composites display that some Au NPs still deposit on stacking and aggregated SCB after reaction, guaranteeing the catalytic activity for next recycling reaction.

Catalytic mechanism

The catalytic principle for 4-NP reduction over Au-based catalysts has been widely proposed and verified in previous studies. Based on the Langmuir–Hinshelwood model reported by Bal-lauff et al., a widely possible electron transfer principle is proposed, i.e., the 2Au/500°C-SCB composites serve as adsorption sites for reactants and electron transfer bridge between BH₄⁻ as electron donors and –NO₂ on 4-NP as electron acceptor (Ren et al. 2021). In the 2Au/500°C-SCB composites, Au NP anchoring on SCB can guarantee Au active sites to effectively promote BH₄⁻ with H₂O to form BO₂⁻ and active Au-H species. Then, the electron transfer between active sites Au NPs and platform SCB can easily donate the electron to the electron-withdrawing –NO₂ group, thus producing –NH₂ as the product of six electron reduction of 4-NP. Overall, the catalytic reduction is originated from the electron transfer between the NaBH₄ and the –NO₂ when Au NPs directly contact with –NO₂. In brief, the catalytic process can be divided into three main stages (Bian et al. 2021; Liu et al. 2021). Firstly, BH₄⁻ and 4-NP are both adsorbed on Au/500°C-SCB surface. Meanwhile, the adsorbed BH₄⁻ is dissociated and activated by Au NP active sites to form the surface bound hydride. Meanwhile, the surface bound hydride transfers to Au NP surface, thus producing active Au-H species. Then, the active Au-H species attacks the 4-NP with electron transferring, leading to –NO₂ reducing to –NH₂. Finally, 4-AP desorbs naturally from 2Au/500°C-SCB surface (Bogireddy et al. 2020; Liu et al. 2021).

Comparison with previous studies

As for a catalyst used in catalytic reaction, the critical factors in practical application should possess super high activity and effective cost. Our group has been working on ways to design supported Au NP-based catalysts with super high activity and low cost. As discussed above, the 2Au/500°C-SCB composites exhibit expected catalytic performance towards 4-NP reduction. Table 1 compares the obtained catalysts in this study with Au NP catalyst reported by our previous studies and other recent publications. The catalytic performance is emphasized.

It should be noticed that although the 2Au/500°C-SCB composites are similar to the Au/CB catalysts in our previous study, in which the initial CB was used for
Table 1: Comparison of the catalytic performance of Au/500°C-SCB with our previous studies and recent published studies

| Samples              | Support            | Modification method for support | Synthesis method                          | $k_{app}$ (min$^{-1}$) | $k_{nor}^b$ (min$^{-1}$mg$^{-1}$) | m (catalyst, mg)/V (solution, mL) | Ref                          |
|----------------------|--------------------|---------------------------------|-------------------------------------------|------------------------|----------------------------------|-----------------------------------|------------------------------|
| 2Au/500°C-SCB        | Carbon black       | Steam activating CB             | AA as reducing agent                      | 2.1925                 | 0.2192                           | 1/5                               | This study                   |
| Au/CB                | Carbon black       | -                               | Ethylene glycol as reducing agent         | 0.8302                 | 0.0830                           | 1/5                               | (Qin et al. 2019b)           |
| HCB-Ni-Au            | Carbon black       | HNO$_3$ modifying CB            | N$_2$H$_2$H$_2$O as reducing agent        | 1.9617                 | 0.3923                           | 1/10                              | (Qin et al. 2019c)           |
| Au/AC                | Activated coke     | -                               | AA as reducing agent                      | 1.1496                 | 0.1277                           | 3/10                              | (Fu et al. 2019b)            |
| Au NPs/CTS/AC        | Activated coke     | Chitosan functionalized AC      | Chitosan as reducing agent                | 0.6994                 | 0.1399                           | 1/10                              | (Fu et al. 2019a)            |
| PDA-g-C$_3$N$_4$/Au  | g-C$_3$N$_4$       | Polydopamine decorated g-C$_3$N$_4$ | Polydopamine as reducing agent          | 3.0840                 | 0.6168                           | 16/6                              | (Qin et al. 2019a)           |
| Au/M$_{2.5}$-ZSM-5(2d) | Silica ZSM-5 zeolite | -                              | NaBH$_4$ as reducing agent                | 0.3450                 | 0.1725                           | 2/3                               | (He et al. 2020)             |
| Au@graphitic carbon nitride | graphitic carbon nitride | -                              | Photodeposition method                    | 0.3198                 | 0.1599                           | 1/1                               | (Nguyen et al. 2019)         |
| Fe$_3$C/Au@NG       | Fe$_3$C@NG         | -                               | Sodium citrate reduction method           | 1.8600                 | 3.7200                           | 1/6                               | (Wang et al. 2021)           |
| Au/NCB               | Carbon black       | Nitrogen doping                 | Adsorption-in situ reduction strategy     | 0.2979                 | 3.5047                           | -                                 | (Han et al. 2021)            |
| Au$_{NC}@ZIF-8(Cu)$ NRAs | ZIF-8 nanorod arrays | Cu doping                      | A bottom-up strategy                      | 0.4890                 | -                                | -                                 | (Gao et al. 2019)            |
| CeO$_2$/Pd nanocomposites | CeO$_2$ nanorods | -                               | Adsorption-in situ reduction strategy     | 0.3310                 | -                                | -                                 | (Wang et al. 2022)           |
| Ag@RF@Fe$_3$O$_4$ nanocatalysts | Resorcinol formaldehyde@Fe$_3$O$_4$ | -                              | Photocatalytic reduction                 | 2.2700                 | 5.4900                           | 1/10                              | (Cao et al. 2020)           |
| Pt/biogenic SiO$_2$ hybrid | Biogenic porous silica particles | -                              | Impregnation method                      | 1.7400                 | 7.2500                           | 1/250                             | (Bogireddy et al. 2020)      |
| Ni/CB                | Carbon black       | Acidification by nitric acid    | Soft chemistry method                     | 0.5970                 | 0.5970                           | 1/50                              | (Xia et al. 2016)            |
| SSBC-800             | Sewage sludge derived biochar | -                              | Pyrolysis method                         | 0.4800                 | 0.0480                           | 1/5                               | (Ren et al. 2021)            |
supporting Au NPs, 2Au/500°C-SCB composites display different structure. The results of nitrogen adsorption isotherms and TEM show that the pore structure of initial CB mainly consists of micropores, mesopores, and macropores, possibly caused by the aggregation of dispersed CB. After depositing Au NPs on the initial CB, the specific area, pore volume, and average pore size decrease, indicating that Au NPs are limited in the porous structure of CB. But in this study, the results of nitrogen adsorption isotherms illustrate that the specific surface area of SCB increases to 249.0053 m²/g, and more micropores structure appears when compared with initial CB. From TEM results, it can be found that Au NPs are located inside and on the surface of SCB. In addition, the Raman structure displays that more defects are brought into SCB, providing more active sites for Au NP anchoring. It can be reasonable speculated that steam activation brings the structure advantages to SCB for supporting Au NPs. Hence, the performance of 2Au/500°C-SCB composites in this study is improved greatly. Under the same reaction condition, the catalytic reduction of 4-NP over 2Au/500°C-SCB composites could be finished within 3 min, while the reaction over Au/CB catalysts was complete within 5 min. The rate constant \( k_{\text{app}} \) of Au/500°C-SCB for 4-NP reduction increased to 2.1925 min⁻¹, but the rate constant \( k_{\text{app}} \) of Au/CB catalysts was 0.8302 min⁻¹ (Qin et al. 2019b). The enhanced catalytic activity of 2Au/500°C-SCB composites could be ascribed to the steam activation, bringing more active sites for Au NP anchoring and more hierarchical pore structure. Compared with other previous study by our group, the catalytic activity of 2Au/500°C-SCB composites is not as good as the HCB-Ni-Au and PDA-g-C_3N_4/Au catalysts, but the modification method for CB platforms is eco-friendly and easy accessibility. And the reductant AA is gentler than \( \text{N}_2\text{H}_4\cdot\text{H}_2\text{O} \), which would not bring about secondary pollution during synthesis.

Except previous studies reported by our group, other related literatures are also compared with this work. As shown in Table 1, some other Au NP-based catalysts also exhibit comparable catalytic activity. Besides, other noble metal-based catalysts (Ag, Pt) exhibit higher catalytic activity than 2Au/500°C-SCB catalysts. But the highlight of this work is that the CB platforms are cheaper and easily available. In addition, the steam activation for platform and AA as reduction as synthesis method in this work is easy to repeat, thus guiding to prepare other supported metal-based catalysts for different applications. Moreover, other metal or metal-free catalysts are also listed and compared in Table 1 and display general catalytic activity \( k_{\text{app}} \) of Ni/CB = 0.5970 min⁻¹; \( k_{\text{app}} \) of SSBC-800 = 0.4800 min⁻¹, further displaying the satisfying activity of Au NP active sites. Nevertheless, the activity of the obtained catalysts in this study should be further promoted, which is doing under our laboratory.

**Conclusions**

In this paper, we have designed a green and economic way to fabricate xAu/T-SCB composites using steam activated CB as platforms and AA as reducing agent. By comparing the characteristics of initial CB and SCB activated by steam at different temperatures, the 500°C-SCB is more apt to serve as benign platforms to anchor Au NPs, which has applicable defects and high BET specific surface area, thus providing quite active sites for Au NP anchoring both inside and on the surface of SCB. The obtained 2Au/500°C-SCB composites exhibit high catalytic efficiency in 4-NP reduction with \( k_{\text{app}} \) of 2.1925 min⁻¹. The superiority of 2Au/500°C-SCB composites can be explained by the adequate catalytic active sites of Au NPs and competent adsorption ability for pollutants ascribed to the above properties of SCB. Furthermore, the enhanced hydrogen adsorption may also be conducive to producing active hydrogen species on Au NPs, thus promoting the catalytic activity. In addition, the 2Au/500°C-SCB composites exhibit well universality and recyclability, which is important for the industrial application. The steam activated SCB for Au NP anchoring in this study offers a guidance for anchoring different metal nanoparticles to prepare functional nanocomposites, which could be used in different applications.

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**Data availability** The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.
Declarations

Ethics approval Not applicable.

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