Optimizing the catalytic activity of flame-spray-pyrolyzed Pt/Fe₂O₃ catalyst toward CO oxidation: Effect of fluorination and reduction

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Funding information
National Natural Science Foundation of China, Grant/Award Numbers: 21978088, 91534202, 51673063; Shanghai Technology Research Leader, Grant/Award Number: 20XD1433600; Program for Professor of Special Appointment (Eastern Scholar) at Shanghai Institutes of High Learning; Basic Research Program of Shanghai, Grant/Award Number: 17JC1402300; Social Development Program of Shanghai, Grant/Award Number: 17DZ1200900; Fundamental Research Funds for the Central Universities, Grant/Award Number: 222201718002

1 | INTRODUCTION

Carbon monoxide (CO) is a highly noxious gas released from incomplete combustion processes and exhaust fumes in the industries and automobiles. Air polluted by CO can exert a pernicious impact on the nervous system and cardiac function of people.[1] As a result of the threatening effects, ensuring ecofriendly environment by the

Abstract
Comprehending the characteristics of nanocatalyst and the effect of pretreatment on its catalytic performance is of great importance in the field of catalysis. The present study probes the catalytic performance of fluorinated Pt/Fe₂O₃ prepared by one step flame spray pyrolysis and the effect of pretreatment in CO oxidation. The characterization reveals that in-situ fluorination of Pt/Fe₂O₃ in high temperature flame process contributed to low temperature reducibility, improved dispersion abundant surface oxygen vacancies and reactive hydroxyl groups. It is noteworthy that the optimal catalytic performance was attained after reduction of fluorinated Pt/Fe₂O₃ in 10% H₂ at 200 °C (F-Pt/Fe-200). The abundant surface hydroxyl group and oxygen vacancies facilitated faster reaction. The in-situ DRIFTS reveals the consumption of hydroxyl group in F-Pt/Fe-200 which contributed to the amelioration of CO oxidation. Aside the better stability of F-Pt/Fe-200, complete conversion of 2% and 4% CO was achieved at 150 °C and 200 °C, respectively, indicating the feasible utilization of flame-made fluorinated Pt/Fe₂O₃. Conclusively, the fluorine formed a strong bond with Pt, which ameliorated oxidative and thermal stability.

KEYWORDS
flame spray pyrolysis, fluorination, hydroxyl group, oxygen vacancies, Pt/Fe₂O₃
conversion of CO into CO₂ has received tremendous attention in the past decades. Catalytically converting CO by oxidation with single and heterogenous metal oxide catalysts such as Fe₂O₃, CeO₂, TiO₂, Pt/TiO₂, Au-FeOₓ/TiO₂, Pt/Fe₂O₃, Au/Fe₂O₃ etc. has been the most widely studied area. Among the metals, Pt nanoparticles on oxide supports is one of the important group of catalysts, which undertakes CO oxidation by Langmuir–Hinshelwood mechanism. However, Pt displays little activity in the room temperature range as a result of its strong adsorption of CO molecules at low temperature, which impedes the adsorption of sufficient O₂ required for CO oxidation. Transition metal oxide such as iron oxide (Fe₂O₃) has stimulated considerable scientific attention due to reasons such as cheap cost, environmental-friendly, and easy to prepare, but it is active at 200 °C and complete CO oxidation around 450 °C which requires high amount of energy to accelerate the spillover process for CO oxidation. Hence, Fe₂O₃ has been doped with precious and non-precious metals to improve upon its catalytic performance. This has been an effective mean of altering the electronic chemistry of Fe₂O₃ by incorporating surface defects like vacancies or the distortion of lattice to improve its catalytic reaction.

Aside metallic elements, doping with non-metallic heteroatoms such as F, S and N is of great interest since they hinder charge recombination, promote surface defects, improve metal-support interfacial bond and increases electron transition lifetime to improve catalytic activity. Fluorine doping endows distinctive electronic features and rich surface chemistry, significantly advancing their adsorption performance toward the removal of pollutant in ensuring eco-friendly environment. According to Chiarello et al., flame pyrolyzed F-doped Pt/TiO₂ emerges as a more efficient heterogeneous catalyst to catalytically produce H₂ from methanol under UV irradiation as compared to its fluorine-free catalyst. Due to the improvement realized in the properties of heteroatoms doped particulate material, Pelletier and Thiébaut propounds that the co-doping of heteroatom with other metals could be an interesting area of research.

Pyrolytic means of synthesizing nanomaterials have gained an edge over chemical routes as these methods deliver low cost, high yield, continuous and rapid preparation without post-treatment (such as drying) of nanoparticles like Fe₂O₃, TiO₂, and Pt/Ba/Al₂O₃, Bi₂O₃, Pt/TiO₂ on an industrial scale in the form of dry particles. In addition, it has been used to prepare several nanoparticles to reduce particle size and boost the dispersibility of dopant on the surface of support metals which play critical role in enhancing catalytic activity.

Pretreatment in reductive or oxidative atmosphere have frequently been employed to alter or enhance the structural defect of catalyst, to improve upon the synergistic structural and surface effect of metals. Also, highly promoted CO catalytic performance of catalysts was observed due to a cleaner surface, large amount of oxygen vacancies and a higher dispersion and smaller particles on the surface of the support after H₂ pretreatment. In addition, among numerous synthesis approaches, series of single and heterogenous oxide nanoparticles prepared by facile flame spray pyrolysis approach have been reported to facilitate the even dispersion of dopant on support. However, to the best of our knowledge, Pt/Fe₂O₃ and fluorine doped Pt/Fe₂O₃ metal oxide particles prepared by flame spray pyrolysis have not been reported.

Therefore, this study aims at flame synthesizing fluorine-free and fluorine doped Pt/Fe₂O₃ (F-Pt/Fe) nanoparticles, comparing the properties of the as-prepared catalysts, analyzing the effect of Pt and F on oxygen species and vacancies, and underlining the effect of pretreatment on their catalytic performance. A further study was undertaken to explore the catalytic performance of the best catalyst in 2% CO and 4% CO concentration under identical reaction conditions. In addition, understanding the intricate inter-relationship between dopant on the support metal is essential to comprehend the results of the catalytic performance. Hence, characterization of samples was conducted to define the rationale for an augmented catalytic performance of the catalysts. A schematic process of the catalyst composite which includes the precursor stage, flame spray pyrolysis of catalyst, collection of nanoparticles, mixing of catalyst with quartz sand, pretreatment of catalyst and CO oxidation test is illustrated in Figure 1.

2 | RESULTS AND DISCUSSION

2.1 | Characterization of the catalysts

The structural morphology of the catalysts was examined by TEM and HRTEM. In Figure 2A and B, the TEM images clearly reveal that among the catalysts, F-Pt/Fe presents improved dispersion of Pt species (20 %) on Fe₂O₃ as compared to Pt/Fe (9.4 %). The in-set of Figure 2 shows that F-Pt/Fe had smaller Pt size (Average = 4.5 nm) than Pt/Fe (Average = 9.6 nm). There exist Pt sintering in Pt/Fe₂O₃ (Pt/Fe) catalyst as compared to F-Pt/Fe catalyst. Agreeing with literature, fluorine doping enhances the dispersion of particulate materials by hindering sintering of Pt nanoparticles on the support metal.

As displayed in Figure 2C and D, the HRTEM images of Pt/Fe₂O₃ reduced in 10% H₂ at 200 °C (Pt/Fe-200) and F-Pt/Fe reduced in 10% H₂ at 200 °C (F-Pt/Fe-200) catalysts show two main interplanar lattice distance of 0.25...
and 0.28 nm corresponding to (311) and (220) phases of Fe₂O₃ (JCPDS Card No. 39–1346). On the other hand, the lattice interplanar spacing ranging from 0.22 nm corresponds to the (111) phase of Pt species. To give more insight, the above-mentioned phases confirm the successfully synthesized Pt-Fe₂O₃ catalyst. The XRD patterns illustrates that there was a shift in peak in F-Pt/Fe as compared to Pt/Fe and Fe catalysts (Figure S1c). The peak shift could be due to the partial substitution of oxygen atom by F⁻, since F⁻ (0.133 nm) has a slightly smaller radius than oxygen atom (0.140 nm).

Following the Pt and fluorine loading, XPS spectra analysis determining the electronic state and the elemental composition of the various catalysts (Fe, F, Pt, and O) in the various catalysts are profiled in Figure 3, while the actual load of the fluorine and Pt elements have been given in Table 1. It was realized that the actual load of Pt in the catalysts were slightly lower than the nominal value (1 wt%). The value of Pt in the catalysts determined by ICP was lower than that of XPS. This could be due to most Pt species located on the surface of the catalyst since XPS primarily detects the surface composition of a catalyst. Unlike Pt, the actual weight % of fluorine was generally higher than the nominal loading which could be attributed to the enrichment of fluorine on the surface of the catalyst. The Pt 4f XPS spectra of Pt/Fe and F-Pt/Fe catalysts are revealed in Figure 3A. The deconvolution of Pt 4f spectra into two pairs of doublets indicates that Pt species is in two states. The binding energies located at ~73.12 and ~76.07 eV correspond to the respective Pt 4f7/2 and Pt 4f5/2 levels of the Pt²⁺ state. The binding energies at ~74.87 and ~78.47 eV corresponding to Pt 4f7/2 and Pt 4f5/2 electrons suggest the existence of Pt⁴⁺ species. The existence of oxidized Pt species could have resulted from the oxidation of Pt at high temperature during synthesis of the catalyst. According to XPS peak fitting, the intensity of Pt⁴⁺ peak at 74.87 eV on F-Pt/Fe (51.11%) is slightly higher than the intensity on Pt/Fe catalyst at 74.86 eV (47.92 %). This implies that F-Pt/Fe possesses a higher ratio of oxidized Pt⁴⁺ state. As shown in the Figure 3B, the Fs core peak appearing at 685.13 eV on F-Pt/Fe corresponds to the surface chemisorbed fluoride ions (consistent with Fe-F bonding in FeF₃ or FeF₂). The minor peak at 686.31 eV is assigned to fluorine atoms in oxygenated environment, resulting from the substitution of F ions for O ions in the Fe₂O₃ lattice. There was no F 1s second peak on Pt/Fe spectrum due to it being a fluorine-free prepared catalyst.

As well-verified in numerous studies relating to CO oxidation, oxygen species in a catalyst plays a crucial role in the process of CO oxidation. Increasing the ratio of
oxygen vacancies on the catalyst enhances catalytic oxidation. The O 1s second regions of Pt/Fe, F-Fe, and F-Pt/Fe catalysts were deconvoluted into three peaks, assigned to hydrated/carbonated oxygen, hydroxyl group, and lattice oxygen species of the heterogeneous metal oxide at ~533, ~532 and ~530 eV, respectively, as shown in Figure 3C. Abundant surface hydroxyl radical is observed in the fluorinated catalysts ([F-Fe (33.2 %) and F-Pt/Fe (31.9 %)] as compared to Pt/Fe (25.4 %). Also, the shift in the OH peak from 531.4 eV (Pt/Fe) to a higher binding energy of 531.7 eV (F-Fe) and 531.9 eV (F-Pt/Fe) could possibly be related to the presence of more electronegative transfer triggered by fluorine doping. In other words, fluorination causes exceptional surface OH coverage and the creation of abundant oxygen vacancy defects, in complete agreement with the observation established in literature. The intensity shoulder of F-Pt/Fe lattice oxygen atom reduced with respect to Pt/Fe as a result of the F for O substitution, confirming the creation of oxygen vacancy for easy occupation by the gaseous oxygen to accelerate CO oxidation. This indicates that fluorine could create a charge imbalance in the bulk of Fe$_2$O$_3$ (shift in binding energy of Fe2p $^{3/2}$ on F-Pt/Fe as a result of the interaction between F$^-$ ions and Fe ions) that forms considerable amount of surface OH group. The plentiful OH groups housed on the surface of the catalyst structure and oxygen vacancy could induce fast catalytic reaction. To solidly confirm the existence of vacancies, as shown in Figure 4, Pristine Fe$_2$O$_3$ (Fe), Pt/Fe, F-Fe and F-Pt/Fe display similar resonance signal ($g = 2.002$), indicating the electrons trapped in the vacancy defects. The F-Fe catalyst presented similar magnitude of defect like Pt/Fe. This implies that the substitutional effect of fluorine ion on the oxygen atoms in the support created vacancy defect on the surface of the Fe$_2$O$_3$. Comparatively, F-Pt/Fe catalyst shows a greatly enhanced intensity in EPR signal, followed by Pt/Fe, F-Fe and then Fe, indicating that the good interaction of Pt and fluorine on the support enhanced the creation of high magnitude of oxygen vacancies. This result indicates that numerous vacancies were successfully generated on Pt/Fe$_2$O$_3$ catalysts after the incorporated fluorine.

In view of the Fe 2p element, two characteristic peaks assigned to Fe 2p$_{3/2}$ and Fe 2p$_{1/2}$ orbits as observed in Figure 3D. The main peak positions of Fe 2p $^{3/2}$ in Pt/Fe are located at 711.07 and 713.23 eV, respectively, F-Fe peaks at 710.95 and 713.08 eV, while in F-Pt/Fe, the peaks at 710.83
and 713.16 eV are respectively assigned to Fe 2p$^{3/2}$ ($\text{Fe}^{3+}$ octahedral and tetrahedral species, respectively). The other peaks at 724.31 and 724.31 eV in Pt/Fe, 724.42 and 726.58 eV in F-Fe, and 724.35 and 726.68 eV in F-Pt/Fe correspond to Fe 2p$^{1/2}$.[27] The change in positions to a relatively low region in Fe 2P$^{3/2}$ after fluorination indicates the alteration of the chemical state of Fe atom by the fluorine element. Notable satellites of Fe 2p$^{3/2}$ and Fe 2p$^{1/2}$ are located at 718.82–718.88 eV and 732.59–732.78 eV. Aside the blueshift in the Fe 2p$^{3/2}$ peak of F-Pt/Fe, fluorination of Pt/Fe led to the reduction in the intensity of Fe$^{3+}$, from 54.6 % to 51.4 %. This is buttressed by the 47.3 % $\text{Fe}^{3+}$ and 52.7 % $\text{Fe}^{2+}$ species

TABLE 1  The chemical composition and textural properties of catalysts

| Catalysts  | Pt loading (wt%)$^a$ | Pt loading (wt%)$^b$ | F loading (wt%) | S$^{\text{BET}}$ (m$^2$ g$^{-1}$) | Pore volume (cm$^3$ g$^{-1}$) | Pore size (nm) |
|------------|---------------------|---------------------|----------------|----------------------|----------------------|----------------|
| Fe         | –                   | –                   | –             | 83.7                 | 0.17                 | 8.53           |
| Pt/Fe      | 0.67                | 0.84                | –             | 111.2                | 0.30                 | 11.01          |
| F-Pt/Fe    | 0.79                | 0.98                | 4.39          | 151.6                | 0.32                 | 8.05           |
| Fe-200     | –                   | –                   | –             | 87.0                 | 0.21                 | 9.5            |
| Pt/Fe-200  | 0.72                | 0.79                | –             | 118.0                | 0.26                 | 9.00           |
| F-Pt/Fe-200| 0.61                | 0.54                | 3.21          | 188.4                | 0.47                 | 10.12          |

NB: $^a$ Elemental composition determined by ICP-AES, $^b$ Determined by XPS, $S^{\text{BET}}$ is the BET surface area of the catalysts.
in F-Fe catalyst. Meanwhile, no other peak associated with Fe$^{2+}$ ion or Fe metal were observed, which implies that the Fe species in the as-prepared catalysts reveal only Fe$^{3+}$ ion. The crystal phases, existence and the dispersibility of the Pt metal on support, were investigated by X-ray diffraction analysis and presented in Figure S1a. Diffraction peaks observed at 2\(\theta\) = 30.29°, 35.70°, 43.47°, 57.43°, and 62.90° were attributed to the presence of Fe$_2$O$_3$ phase in the catalysts.\[7a\] In view of Pt on the support, its detectability by XRD strongly depends on the concentration and crystal size. The most intense peak of Pt species which appears at 39.8°.\[28\] were rarely identified in all the as-prepared catalysts. It is also worth mentioning that Pt species were well dispersed on the support. The fluorine could replace oxygen to contribute extra free electrons\[29\] to enhance catalytic performance. Fluorine also interact with Pt/Fe$_2$O$_3$ crystal and refrains crystal growth. The effect of reduction in H$_2$ on the crystal structure of the Pt/Fe and F-Pt/Fe was also examined and presented in Figure S1b. The XRD spectra of Pt/Fe -200 and F-Pt/Fe-200 exhibit utterly unrecognizable peak of Pt species, diminished and broadened Fe$_2$O$_3$ phases as compared to as-prepared catalysts. Evidently, the fluorine and pretreatment in H$_2$ did not have any other evidential impact on the crystal structure of catalyst as compared to untreated catalysts, aside broadening of Fe$_2$O$_3$ phases.

The BET Surface area (S$_{BET}$) of the as-prepared and reduced catalysts are recorded in Table 1. The N$_2$ adsorption–desorption isotherms of the as-prepared and reduced catalysts have been illustrated in Figure 5. Undoubtedly, the N$_2$ adsorption–desorption isotherms represent type III with an H3-type hysteresis loop in the IUPAC classification, clearly indicating the existence of mesoporosity within the catalysts (Pore size of 9–10 nm).

The isotherm reveals specific surface area of 83.74, 111.17, and 151.64 m$^2$ g$^{-1}$, corresponding to Fe, Pt/Fe and F-Pt/Fe while pretreated catalysts reports surface area of 86.96, 117.89, and 188.41 m$^2$ g$^{-1}$, corresponding to Fe-200, Pt/Fe-200, and F-Pt/Fe-200, respectively. There was an increase in surface area of the catalyst after doping, especially in F-Pt/Fe-200. The presence of mesopores improves the diffusivity of the reactant molecules during the catalytic reaction. Even though the specific surface area of Pt/Fe-200 increases relative to as-prepared Pt/Fe and Fe-200, F-Pt/Fe-200 clearly displays the highest specific surface area among all the flame-pyrolyzed catalysts. The advance enhancement of the surface area is assigned to the pretreatment of catalysts in H$_2$ atmosphere which triggers abundant and cleaner active sites on the surface of the catalyst. F-Pt/Fe catalyst exhibits good textural properties among all the as-prepared catalysts, but F-Pt/Fe-200 renders the best textural properties which could be related to the enhance dispersion resulting from fluorination and reduction.

In the catalytic oxidation of CO, it has been agreeably established that Fe$_2$O$_3$ catalysts principally follow a redox mechanism. Accordingly, the ability of Fe$_2$O$_3$ catalyst to lose oxygen atoms in reduction atmosphere has meritable leverage on its catalytic performance. As profiled in Figure 6A, the catalysts exhibit two main peaks. The peak at ~ 400 °C corresponds to the reduction of Fe$_2$O$_3$ to Fe$_3$O$_4$ while the extensive peaks at ~ 600 °C is attributed to the reduction of Fe$_3$O$_4$ to FeO species.\[30\] The initial reduction peaks of Fe, Pt/Fe and F-Pt/Fe are located at ~396 °C, 274 °C, and 171 °C, respectively. The lower temperature reduction on F-Pt/Fe catalyst is assigned to the reduction of well dispersed oxidized Pt into metallic Pt, with some contribution from partial reduction of Fe$_2$O$_3$ linked with the Pt. This linkage is assigned to the enhanced metal-support interaction between Pt species and Fe$_2$O$_3$ support interface caused by fluorination. The F-Fe demonstrates easy reducibility than Fe catalyst. This implies that the fluorine doping enhances the metal-support interaction (Pt $\rightarrow$ Fe$_2$O$_3$ interaction) and the reducibility of the catalyst at lower temperature. This is a prospective measure of the catalytic performance of the catalysts.

CO-TPD analysis is used to investigate the properties of surface oxygen of catalysts\[31\] or the thermodynamics and kinetics of desorption from the surface of heterogenous catalysts. Desorption peaks at low temperature (below 200 °C) and at high temperature have been ascribed to linear CO molecules and bridge CO molecules, respectively.\[32\] The CO-TPD spectra of Pt/Fe-200 and F-Pt/Fe-200 have been illustrated in Figure 6B. The two catalysts show CO desorption at low temperature but a much recognizable desorption peak from 94.5 - 185.1 °C on F-Pt/Fe-200 spectrum indicates the presence of bonded CO.}

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**FIGURE 4** EPR spectra of Pristine Fe, Pt/Fe, F-Fe and F-Pt/Fe catalysts
molecules on the surface of the catalyst which are release in the form of CO$_2$. This demonstrates that surface oxygen of F-Pt/Fe-200 catalyst was active at low temperature, confirming the numerous surface oxygen. Additionally, the temperature of the main CO desorption peak from the surface of F-Pt/Fe-200 catalyst which began from 289.7 °C (desorption peak at 345 °C) herein is lower than that of Pt/Fe-200 catalyst (desorption peak at 360 °C). The difference in the desorption peaks could be related to the abundant surface oxygen species and enhanced specific surface area of F-Pt/Fe-200 created by the incorporated fluorine relative to Pt/Fe-200 catalyst. This points out that F-Pt/Fe-200 catalyst had numerous active sites for the oxidation of CO molecules on its surface at low temperature, thereby probably enhancing its catalytic activity. The higher temperature broad desorption peak at about 540 °C corresponds to the desorption of CO dissociatively adsorbed and coordinatively unsaturated O$^{2-}$ ions or coordinatively unsaturated F$^{-}$ ions that desorb on the surface.[33,34]

2.2 | Catalytic performance of as-prepared and pretreated Pt/Fe2O3 in varying CO concentration

CO catalytic oxidation carried out in 1% CO, 10% O$_2$, balanced with Argon as a function of temperature for the evaluation of the catalytic performance of the F-Pt/Fe and Pt/Fe catalysts are plotted in Figure 7A. As displayed in Figure 7A, the gradual increase in the percentage of conversion with respect to increasing temperature demonstrates a progressive CO oxidation. The complete
CO oxidation of Pt/Fe, F-Fe, and F-Pt/Fe catalysts was recorded in the range of 300 °C and 225 °C, respectively, which is more than 100 °C lower than that of the Fe catalyst. The incorporation of Pt on Fe₂O₃ renders a sharp rise in the catalytic performance, obviously revealing an improvement in the catalytic activity after Pt doping. The better catalytic performance of Pt/Fe catalyst could be assigned to the enhanced reducibility and metal-support interaction relative to Fe catalyst.

Aside the pristine Fe catalyst, Pt/Fe presents a low catalytic efficiency, which shows 100% CO conversion as the temperature reaches as high as 300 °C. The non-metallic
**TABLE 2**  A summary of the test conditions, pretreatment conditions and catalytic performance of catalysts (half and full conversion temperature)

| Test conditions | Catalysts | Pretreatment conditions | T<sub>50</sub> | T<sub>100</sub> |
|-----------------|-----------|-------------------------|---------------|---------------|
| 1% CO, 10% O<sub>2</sub>, balanced with Argon | Fe        | 10% H<sub>2</sub> at 200 °C | 293           | 425           |
|                 | F-Fe      | As-prepared             | 302           | 400           |
|                 | Pt/Fe     | As-prepared             | 234           | 275           |
|                 | F-Pt/Fe   | As-prepared             | 187           | 225           |
| 1% CO, 10% O<sub>2</sub>, balanced with Argon | F-Pt/Fe   | 10% H<sub>2</sub> at 200 °C (1hr) | 205           | 225           |
|                 | Pt/Fe     | 10% H<sub>2</sub> at 200 °C | 183           | 200           |
|                 | F-Pt/Fe   | 5% H<sub>2</sub> at 200 °C | 131           | 140           |
| 2% CO, 10% O<sub>2</sub>, balanced with Argon | F-Pt/Fe   | 10% H<sub>2</sub> at 200 °C | 130           | 150           |
|                 | Pt/Fe     | 10% H<sub>2</sub> at 200 °C | 178           | 200           |
|                 | F-Pt/Fe   | 10% H<sub>2</sub> at 200 °C | 145           | 200           |
|                 | Pt/Fe     | 20% H<sub>2</sub> at 200 °C | 229           | 250           |

* T<sub>50</sub> and T<sub>100</sub>, corresponding to 50% and 100% CO conversion, respectively. All the pretreatment conditions were kept at 30 minutes dwell time, except specified.

Fluorinated Fe<sub>2</sub>O<sub>3</sub> showed full conversion at 400 °C. The incorporation of fluorine relatively shows better catalytic performance than the pristine Fe<sub>2</sub>O<sub>3</sub>. Most importantly, among the as-prepared catalysts, F-Pt/Fe grants optimal catalytic performance at 225 °C, which is 75 °C and 200 °C lower than Pt/Fe and pristine Fe catalysts, respectively. The significant improvement in catalytic performance of F-Pt/Fe could be related to the well-dispersed Pt nanoparticles on support, lower temperature reducibility, improved specific surface area and pore volume, and abundant oxygen vacancy defects resulting from unique electronic features and rich surface chemistry with fluorine ion. Additionally, a straightforward comparison of the test conditions, pretreatment conditions and catalytic performance for pristine Fe, Pt/Fe, and F-Pt/Fe catalysts is synopsisized in Table 2.

The effect of pretreatment by in situ pretreatment of the F-Pt/Fe in O<sub>2</sub> and H<sub>2</sub> at varying percentage and temperature were done to ascertain the influence of pretreatment on the surface of the catalysts toward CO oxidation (Figure 7B). The catalytic activity of F-Pt/Fe pretreated in 5%–10% H<sub>2</sub> at 200 °C (F-Pt/Fe-200) increase notably the active sites of the catalyst, giving rise to improvement in CO conversion catalytic activity. However, further increase in the percentage of H<sub>2</sub> to 20% at 200 °C offers complete conversion at 150 °C which indicates no significant effect as compared to the performance of F-Pt/Fe-200 (as seen in Figure S3). Calcination in 20% O<sub>2</sub> at 200 °C did not have any huge influential effect on the performance after or without H<sub>2</sub> pretreatment (as shown in Figure 7C). This implies that cleaner surface, large amount of oxygen vacancies were formed due to surface reconstruction of the catalyst after reduction in H<sub>2</sub> gas, which enriched oxygen supply for the catalytic oxidation of CO<sup>[17,18,35]</sup> In comparison with Pt/Fe, F-Fe, and F-Pt/Fe, the temperature of complete CO oxidation over Pt/Fe -200 and F-Pt/Fe-200 significantly decreases from 300 °C to about 250 °C and 225 °C to 150 °C, respectively, while F-Pt/Fe-200 (T<sub>100</sub>: 375 °C) attained a similar performance as compared to F-Fe catalyst (T<sub>100</sub>: 400 °C) (Figure 7B). The H<sub>2</sub>-pretreated catalysts were more active in the lower temperature range below 100 °C than that of as-prepared catalysts. What is noteworthy, Figure S2 reveals that the enhanced dispersion of Pt particles of F-Pt/Fe-200 catalyst were maintained after reduction in H<sub>2</sub>, similar to the TEM image of as-prepared F-Pt/Fe (Figure 2B) but Pt phase (111) of Pt/Fe-200 and F-Pt/Fe-200 was barely identified in the XRD patterns as compared to that of as-prepared catalysts (Figure S1). This demonstrates that in the process of surface reconstruction by hydrogenation, the interaction of Pt particles on the support was further improved to boost the catalytic performance.

Since F-Pt/Fe-200 turns out as the best after employing varying percentage of H<sub>2</sub> at 200 °C, further CO oxidation test with emphasis on conversion at 120 °C, 130 °C, 140 °C, and 150 °C was conducted to identify the narrow temperature at which a full CO conversion is achieved. The complete conversion was attained at 140 °C as observed.
in the inset of Figure 7B. Also, F-Pt/Fe was reduced in 10% H₂ for 1 hour (F-Pt/Fe-200/1) and also at 300 °C for 30 minutes (F-Pt/Fe-300) to assess the effect of time and high temperature on their performance, respectively. As shown in Figure 7B, complete CO oxidation was attained at 150 °C and 200 °C for the respective F-Pt/Fe-200/1 and F-Pt/Fe-300 catalysts. Obviously, the F-Pt/Fe-200/1 grants similar result as F-Pt/Fe-200; hence, the length of time did not significantly affect the catalytic performance. As tabulated in Table S1, the reaction rate of F-Pt/Fe-200 (129.62 mmolCO h⁻¹ gPt⁻¹) was highly enhanced more than Pt/α-Fe₂O₃–C (0.6 wt% Pt), Pt/Fe₂O₃{001}(5.11 wt% Pt) and also Pt[100]/Fe₂O₃{104}(5.49 wt% Pt) with consideration to the Pt loading reported in literature [36] (Table S1). No catalytic activity enhancement was realized by further increasing the reduction temperature to 300 °C. The increase in the reduction temperature decreased the complete CO conversion temperature from 150 °C for F-Pt/Fe-200 catalyst to 200 °C for F-Pt/Fe-300. This could be assigned to the sintering of Pt nanoparticles on the surface of the support metal which could have led to reduction in metal-support interaction. Impressively, among pretreatment in H₂ and O₂, it could be established that F-Pt/Fe-200 catalyst had enhanced interfacial active sites with improved metal-support interaction, surface area and spillover process of frequent oxygen supply for CO oxidation.

A further investigation on the catalytic activity of F-Pt/Fe-200 and F-Pt/Fe-300 by exposing the catalysts to 2% CO and 4% CO was conducted and illustrated in Figure 7D. The experimental result of the 2% CO and 4% CO over the two catalysts is quite dramatic. A full 2% CO conversion over F-Pt/Fe-200 and F-Pt/Fe-300 was achieved at 150 °C and 200 °C, respectively, while in 4% CO, the respective catalysts attained complete CO conversion at 200 °C and 250 °C. Comparatively, F-Pt/Fe-200 presents a highly promoted complete 2% and 4% CO conversion at lower temperature. The F-Pt/Fe-200 in 2% CO oxidation shows slight difference to 1% CO oxidation and enhance reaction rate relative to catalysts reported in studies [36] (Table S1). This bidly props that fluorination and hydrogenation of catalyst plays a crucial role in the promotion of the catalytic performance of fluorinated Pt/Fe₂O₃.

Moreover, the knowledge about the stability of catalysts also improves the understanding of the performance and deactivation of catalyst during time-on-stream. Hence, the experiment of time-on-stream was done to probe the stability of the F-Pt/Fe-200 and F-Pt/Fe-300 catalysts under 2% CO, 10% O₂, balanced with Argon at 150 °C as shown in Figure 7E. For comparison, the stability of Pt/Fe -200 and F-Pt/Fe-300 catalysts at 200 °C was also studied and presented in Figure S4. There was no decline in the CO conversion throughout the time-on-stream for F-Pt/Fe-200 catalyst at 150 °C. Furthermore, the F-Pt/Fe-200 catalyst shows no significant decline after three CO oxidation runs as seen in Figure 7F. Amelioration in thermal stability could be assigned to adequate active sites and sinter-resistance related to the formation of Pt-F bond with strong bond energy on the surface of the catalyst. The Pt-F bond helps reduce the sintering ability of Pt particles on the surface of the support throughout the stability test. Unlike F-Pt/Fe-200, F-Pt/Fe-300 catalyst experienced deactivation from 20.10% to 15.12% CO conversion but at 200 °C. This could be assigned to state of inadequate active sites to ensure the stability of F-Pt/Fe-300 at 150 °C. Evidently, the F-Pt/Fe-200 catalyst displays excellent stability for CO oxidation due to the facile oxygen transfer mechanism caused by the interaction between Pt and fluorine. The summary of the study with consideration to test conditions, catalysts, pretreatment conditions, and the temperature at which 50% and 100% CO conversion was attained over pristine Fe₂O₃, Pt/Fe, and F-Pt/Fe catalysts have been tabulated in Table 2.

To understand the change in oxidation states of Pt and Fe species in the catalysts after reduction prior to CO oxidation, XPS spectra of the reduced catalysts were also presented (Figure 8). The Pt 4f spectra result of F-Pt/Fe-200 and Pt/Fe-200 were quite different from the spectra of untreated catalysts, in which metallic Pt species were not observed. It showed that the Pt phase was transformed progressively from the oxidized state to metallic Pt upon exposure to hydrogen. A possible elucidation could be the sublimation of oxidized Pt species into vapor during reduction which resulted in condensation into metallic Pt particles. Compared to the untreated catalysts, the magnitude of oxidized Pt was decreased after reduction in hydrogen. Pt/Fe-200 and F-Pt/Fe-200 contain 25% and 29% metallic Pt, respectively. Nevertheless, the presence of high content of Pt⁴⁺ in F-Pt/Fe-200 (39%) as compared to Pt/Fe-200 (34.7%) indicates incomplete reduction of the oxidized Pt into Pt⁰. This indicates good sintering-resistibility of F-Pt/Fe-200 catalyst. The Fe 2P of the reduced catalysts record a shift in binding energy to lower values to form Fe²⁺ ions, confirming that Fe species have an essential influence on the Pt toward fast CO oxidation. This electron transformation between Fe and Pt results in electron-rich Pt atoms and the shift of the Fermi level to a higher energy, which weakens CO poisoning of Pt. The surface Pt content of the reduced catalysts was lower than that of the untreated catalysts (Table 1) even though the bulk Pt content (recorded by ICP) of the fresh and reduced catalysts were similar. This could imply that the surface Pt species were reduced after hydrogen treatment of catalyst, as reflected in XPS spectra. Also, the improved dispersion in F-Pt/Fe-200 could have enabled easy reduction of surface Pt as compared to Pt/Fe-200.

Figure 9 shows in situ DRIFTS of CO adsorption and Ar purging on the Pt/Fe-200 and F-PtFe-200 catalysts. The peaks between 1700 and 1200 cm⁻¹ are assigned to
FIGURE 8  XPS spectra of a) Pt and b) Fe 2p for Pt/Fe-200 and F-Pt/Fe-200 catalysts

FIGURE 9  In-situ DRIFTS of CO adsorption and Ar purging on Pt/Fe-200 and F-Pt/Fe-200 catalysts at (A) 30 °C, (B) 40 °C, (C) 50 °C, (D) 60 °C, (E) 70 °C, (F) 80 °C, (G) 90 °C and (H) 100 °C
vibration and stretching of carbonate, bicarbonate and formate species, which are the intermediates in CO catalytic oxidation.\[^{8,27,37}\] Peaks belonging to carbonate and formate species can also be detected at low temperatures on both catalysts. It is observed that the intensity of carbonate and formate species downshifted rapidly on F-Pt/Fe-200 as the temperature increases but remains constant on Pt/Fe-200. The bands detected at 2169 and 2112 cm\(^{-1}\) are assigned to gaseous CO and CO adsorption on metallic Pt, respectively. The two main peaks that appears simultaneously at 2335 and 2357 cm\(^{-1}\) originate from gaseous CO\(_2\).\[^{36}\] The bands ascribed to gaseous CO\(_2\) on F-Pt/Fe-200 blue-shifted to form another peak at 2318 cm\(^{-1}\) as the temperature increases; indicating the easy release of CO\(_2\) from the surface of the catalyst. Pt/Fe-200 recorded CO\(_2\) peaks at all temperature at 2112 and 2169 cm\(^{-1}\) with no shift but negative CO\(_2\) peaks. The negative CO\(_2\) peaks observed on Pt/Fe-200 are due to the subtraction of residual CO\(_2\) in the background spectrum from the sample spectrum under reaction flow, at which the CO\(_2\) concentration was lower than the background CO\(_2\) concentration. In other words, absolutely low or no CO\(_2\) was produced as the temperature increases which is quite the opposite in F-Pt/Fe-200. Also, the consumption of OH is confirmed by the downshifting at the OH region (broad band between 3600 and 3200 cm\(^{-1}\))\[^{39}\] as seen in Figure S5. This indicates that the CO adsorption process is associated with the consumption of OH groups evidenced by the downshifting feature occurring around 3666 cm\(^{-1}\) which favors CO oxidation.\[^{37}\] The CO reacted with the OH groups of F-Pt/Fe-200 and desorbing some carbonate species from the surface in the formation CO\(_2\), resulting in a negative peak at the carbonate regions. In situ DRIFTS of CO adsorption on the catalysts were followed by Ar flushing. Notably, when only Ar was introduced into the gas stream, the doublet bands attributed to gaseous CO and Pt-CO were removed on the catalysts. The CO\(_2\) peak was almost constant on Pt/Fe-200, while F-Pt/Fe-200 recorded an increase in CO\(_2\) peaks as the temperature increases. Lastly, when O\(_2\) was introduced (as shown in Figure S6), F-Pt/Fe-200 recorded an increase in the CO\(_2\) peaks as temperature increases, which is similar to the result attained in Ar flow. The intensity of CO\(_2\) peaks on Pt/Fe-200 was almost constant, depicting a hindrance in the formation of CO\(_2\) due to poor interaction of CO and O\(_2\) molecules. Generally, the low temperature reducibility and strong metal-support interaction promotes the spillover process of CO oxidation. In other words, CO gas undergoes initial adsorption on the surface of Pt nanoparticles and then oxidize by the surrounding less coordinated surface adsorbed oxygen species already activated by charge exchange with the support to release CO\(_2\), as buttressed by H\(_2\)-TPR analysis. The gaseous oxygen in the gas stream reacts with the surface oxygen vacancies to regenerate surface oxygen atoms on or around Pt sites to effectively proceed with the oxidation process (as shown in Figure 10).

3 | CONCLUSION

A rational design approach by reduction of fluorinated Pt/Fe\(_2\)O\(_3\) flame spray pyrolyzed catalysts in H\(_2\) for efficient catalysts toward CO catalytic oxidation was raised. The study demonstrates that the overall fluorine doped noble Pt metal on Fe\(_2\)O\(_3\) support extensively enhanced CO catalytic activity especially after pretreatment in H\(_2\). Catalytic evaluation studies employing varying CO percentage and/or gas conditions discloses F-Pt/Fe-200 as efficient in CO oxidation. Aside the improved catalytic performance of Pt/Fe compared to pristine Fe\(_2\)O\(_3\), the incorporation of fluorine played a great role by enhancing the spillover process of CO oxidation due to the formation of numerous active surface hydroxyl radicals and oxygen vacancies. An ideal relationship between the catalytic performance of F-Pt/Fe-200 and the following parameters which include lower temperature reducibility depicting strong metal-support interaction, well dispersed Pt nanoparticles, enhanced CO desorption, high surface area and oxygen vacancies, was established. In-situ DRIFTs study reveals that OH groups played a role in the formation of CO\(_2\) to enhance catalytic performance on F-Pt/Fe-200. Also, the enhanced sinter-resistant Pt-F bond led to improved stability of the catalyst. On the basis of this study, the three main catalysts presenting the best activity toward CO oxidation follows this order: F-Pt/Fe-200 > F-Pt/Fe-300 for pretreated catalysts and F-Pt/Fe for as-prepared catalysts. Principally, this study indicates that the incorporation of fluorine with Pt and Fe prepared by flame spray pyrolysis and the exposure of catalysts to reduction atmosphere suitably engineered the interfacial interaction for improving the CO oxidation activity of catalyst.
**EXPERIMENTAL SECTION**

**4.1 Synthesis of the catalysts**

Flame spray pyrolysis method of nanoparticle preparation was used to synthesize all the catalysts as described in our previous work.[2] Regarding the synthesis of fluorine-free Pt/Fe$_2$O$_3$ (Pt/Fe), ferrocene and chloroplatinic acid hexahydrate were dissolved in 15 vol% propanoic acid in xylene and ultrasonicated to obtain a homogeneous liquid precursor. Oxygen gas, the oxidant/dispersion gas was used to atomize liquid feed supplied by a syringe pump maintained at a constant pressure (flow rate: 5 mL min$^{-1}$). The well-dissolved liquid precursor was sprayed into a turbulent high-temperature H$_2$ (flow rate: 100 L min$^{-1}$) and O$_2$ (flow rate: 0.15 MPa) flame, where the spray droplets ignite and undergo chemical reaction, condensation and coagulation, leading to nucleation and particle growth. For fluorinated Pt/Fe$_2$O$_3$, the procedure was exactly the same as that of Pt/Fe. Hexafluorobenzene was added into the precursor solution. The nanoparticles were sucked and received on fiberglass filters. The designed concentration of Pt was fixed at 1 wt% while that of fluorine was 4 wt% (weight ratio with respect to the support metal). In the further study, pretreatment of catalysts by reduction in H$_2$ and/or calcination in oxygen were done at varying temperatures prior to CO oxidation test; unless specified (Pt/Fe-200, F-Pt/Fe-200, F-Pt/Fe-300 correspond to Pt/Fe and F-Pt/Fe treated in 10% H$_2$ at 200 °C, and 300 °C for 30 minutes, respectively).

**4.2 Characterization of the catalysts**

The catalysts were characterized by TEM, HRTEM, XRD, XPS, ICP-AES, EPR, N$_2$ adsorption–desorption, H$_2$-TPR, CO-TPD, and In-situ DRIFTS. The chemicals used and characterization procedures are described in the Supporting Information.

**4.3 Evaluation of catalytic performance**

The catalytic performance over the catalysts were conducted in a fixed-bed reactor. Approximately, 50 mg of the catalyst was diluted with quartz sand (200 mg) and then loaded in the middle of a quartz tube reactor with 6 mm inner diameter. Afterward, it was exposed to a gas stream containing CO (1%, 2%, and 4%), 10% O$_2$, balanced with Argon in 50 mL min$^{-1}$ flow rate, corresponding to gas hourly space velocity of 60,000 mL g$^{-1}$ h$^{-1}$. For an extensive probing, the catalytic performance of as-prepared catalysts and in-situ pretreated catalysts was assessed. An Online gas chromatograph (Agilent) equipped with a TCD detector was utilized to measure the influent and effluent gas. The O$_2$, CO, and CO$_2$ were separated by TDX-01 column (2 m × 4 mm). The CO conversion (CO$_2$) was defined according to equation (1), depicting the change in CO concentration: where [CO]$_i$ and [CO]$_o$ is the inlet and outlet CO concentration, respectively. The interval between two temperature assessment points was 25 °C, unless specified in the study.

\[
CO_2 = \frac{[CO]_i - [CO]_o}{[CO]_i} \times 100\%
\]  

**ACKNOWLEDGMENTS**

This work was supported by the National Natural Science Foundation of China (21978088, 91534202, 51673063), Shanghai Technology Research Leader (20XD1433600), the Program for Professor of Special Appointment (Eastern Scholar) at Shanghai Institutes of High Learning, the Basic Research Program of Shanghai (17JC1402300), the Social Development Program of Shanghai (17DZ1200900), the Shanghai City Board of education research and innovation project, and the Fundamental Research Funds for the Central Universities (222201718002).

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SUPPORTING INFORMATION
Additional supporting information may be found online in the Supporting Information section at the end of the article.

How to cite this article: Sarkodie B, Hu Y, Bi W, Jiang J, Li C. Optimizing the catalytic activity of flame-spray-pyrolyzed Pt/Fe2O3 catalyst toward CO oxidation: Effect of fluorination and reduction. Nano Select. 2021:2:744–757. https://doi.org/10.1002/nano.202000211