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# Competitive Transient Electrostatic Adsorption for In Situ Regeneration of Poisoned Catalyst

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Abstract: Catalyst deactivation by poisoning species is one important concern in catalysis processes and causes significant catalyst material and operation costs due to process shutdown and catalyst replacement. We report one new in situ catalyst regeneration method, which utilizes competitive electrostatic adsorption of charged ions which are generated with moderate DC voltage supply following the Townsend discharge mechanism to desorb poisoning species and the transient characteristic of electrostatically adsorbed ions to recover the active sites for catalysis. We demonstrate effectiveness of this new concept using HCOOH decomposition on Pt catalyst as model reaction and find the deactivated Pt regains the activity in presence of Ar<sup>+</sup> generation. DFT simulations and classical electrical discharge calculations show Ar<sup>+</sup> ions have significantly higher electrostatic adsorption energy than desorption energy of CO poisoning species that helps to desorb CO from Pt and generate more available active sites. This new in situ catalyst regeneration method, with convenient, noninvasive and low operation cost features, provides a promising strategy to overcome the challenges associated with current technologies that handle catalyst deactivation.

Poisoning is identified as one major cause of catalyst deactivation,<sup>[1]</sup> resulting in billions of dollars loss every year for catalyst replacement and process shutdown.<sup>[2]</sup> Due to strong chemisorption of poisoning species (impurities in reactant feeds or generated during reaction), active sites would be occupied and deactivated. For instance, H<sub>2</sub> purification is required prior to use for fuel cell and ammonia synthesis reactions since H<sub>2</sub> produced from hydrocarbon reforming often contains CO as byproduct that can readily poison transition metal catalysts in the two applications.<sup>[3]</sup> Formic acid is considered as hydrogen carrier for its allowance of catalytic decomposition into H<sub>2</sub> under ambient condition. Pt is discovered as one active catalyst for this reaction, which however, gets deactivated rapidly due to the generation and accumulation of CO species that occupy the active sites based on previous studies.<sup>[4]</sup> Thus regeneration of

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poisoned catalysts becomes essential to recover the catalytic activity to enable their reuses.

Till to date, off-site catalyst regeneration remains a common procedure in practice. However, off-site regeneration process requires periodic interruption of reaction for taking out deactivated catalyst materials for regeneration and replacing them with fresh ones, which often adds complexity over reaction control and additional operation cost.<sup>[5]</sup> To overcome this issue, in situ catalyst regeneration is demanded.

Although the past decades witnessed considerable research efforts in developing in situ catalyst regeneration techniques, few successes have been achieved. For instance, there were studies of in situ regeneration of coking catalyst by co-feeding hydrogen, air, hot steam and other reagents to reaction systems.<sup>[6]</sup> Despite of effectiveness in removing the coke, other poisoning species like CO and NO<sub>x</sub> were generated. Undesired reactions between the co-fed chemicals and reactants also occurred that led to altered product distribution.<sup>[2]</sup> Nonthermal plasma was proposed for in situ regeneration of Au/TiO2 catalyst in CO oxidation,<sup>[7]</sup> as it can generate oxygen radicals, electrons and excited molecules to help with desorption of poisoning species.<sup>[8]</sup> However, plasma generation requires high energy input that raises an efficiency issue, and the generated radicals are highly reactive that would cause undesired reactions. To realize in situ catalyst regeneration without side effects, new mechanisms that allow efficient removal of poisoning species from surface and in the meantime do not affect the reactions are desired.

Herein we report one new Competitive Transient Electrostatic Adsorption (CTEA) mechanism for energy-efficient and noninvasive in situ regeneration of poisoned catalyst. We utilize the Townsend discharge phenomena for Ar<sup>+</sup> generation at a moderate DC voltage, and utilize CTEA of Ar<sup>+</sup> for competition against chemisorbed poisoning species to regenerate the active sites. Effectiveness of this interesting mechanism is demonstrated with Pt nanoparticle catalysis in HCOOH decomposition, which exhibits dramatically recovered activity with unaltered reaction pathway.

Mechanistic studies have identified two reaction routes in HCOOH decomposition, namely dehydrogenation and dehydration pathways:  $^{\left[9\right]}$ 

 $HCOOH \leftrightarrow CO_2 + H_2 \quad \Delta G = -48.4 \ kJ \ mol^{-1} \qquad (1)$ 

 $HCOOH \leftrightarrow CO + H_2O \quad \Delta G = -28.5 \ kJ \ mol^{-1}$  (2)

Pt was found as one most active catalyst, but can readily deactivate with reaction time.<sup>[10]</sup> This is because Pt can catalyze both reactions, which would generate CO that exhibits strong chemisorption (Figure 1a). Consequently, the generated CO would accumulate on Pt surface and block the active sites, resulting in a rapid decay in the catalytic activity.<sup>[11]</sup>

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**Figure 1.** Schematic illustration of (a) CO poisoning species generation and accumulation on Pt surface in HCOOH decomposition and (b) the concept of in-situ regeneration of poisoned catalyst by removing CO poisoning species utilizing competitive and transient electrostatic adsorption of Ar<sup>+</sup> species.

Figure 1b illustrates the concept of CTEA for in situ regeneration of CO-poisoned Pt catalyst. An applied moderate DC voltage would generate an electric field in the reaction atmosphere, which accelerates free electrons to collide with Ar molecules for charge avalanche multiplication, following the Townsend discharge mechanism.<sup>[12]</sup> Driven by the electric field, the generated Ar<sup>+</sup> would migrate and electrostatically adsorb to Pt catalyst being grounded. The electrostatic adsorption competes with chemisorbed CO that forces CO desorption. Meanwhile, charge transfer between electrostatically adsorbed Ar<sup>+</sup> and grounded Pt leads to charge neutralization, followed by rapid Ar desorption. Thus the Ar<sup>+</sup> electrostatic adsorption is a transient process that regenerates active sites to allow continuous HCOOH decomposition catalysis without process interruption.

This CTEA mechanism was tested by examining HCOOH decomposition at room temperature in one flow reactor of design (Figure S1), which consisted of two parallel silicon wafers positioned in distance of 3 mm and connected with a DC voltage supply. One thin layer of commercial Pt/C catalyst was deposited on the grounded wafer (see Supporting Information for more experimental details). Transmission electron microscopy (TEM) of the catalyst showed a fine dispersion of Pt nanoparticles on carbon support, with an average particle size of around 3 nm (Figure S2a). Clear lattice fringes were observed with high-resolution TEM (HRTEM), indicating a good crystallinity of Pt. The lattice fringe spacing was measured as 0.224 nm, which was indexed to the (111) plane of face-centered-cubic (*fcc*) Pt.<sup>[13]</sup>

The catalytic properties of the Pt catalyst in HCOOH decomposition was firstly investigated in absence of DC voltage

supply by flowing Ar gas-carried HCOOH into the reactor and analyzing the product effluents with gas chromatography (GC). Original GC counts were directly used to evaluate the products because of the usage of tiny catalyst amount that led to minimal conversion. Both H<sub>2</sub> and CO<sub>2</sub> were detected at the beginning of reaction (Figure 2a), suggesting an initial good activity of Pt. It exhibited a rapid decay in the activity, with barely any gas products being detectable after 120 min on stream. The observed Pt deactivation was consistent with previous studies and was attributed to the generation of poisoning CO. <sup>[11]</sup> No CO product detected throughout the experiment, which could be caused by the fact that any generated CO would not desorb owning to the strong chemisorption.

In situ regeneration of the poisoned Pt catalyst was studied by applying -800 V DC voltage to the reactor (a negative value stands for the Pt electrode being grounded). This voltage generated an electric field between the two plate electrodes, with 2.67 kV/cm field strength and the field direction towards the Pt electrode. Such a field is below the breakdown voltage limit for Ar and falls in the Townsend discharge regime for Ar<sup>+</sup> generation.<sup>[14]</sup> An immediate activity recovery of the deactivated Pt was observed, evidenced by instant increases in the H<sub>2</sub> and CO<sub>2</sub> gas products (Figure 2b). Interestingly, a low content of CO was also detected, which would result from desorption of this poisoning species from the deactivated Pt surface. This finding confirmed effectiveness of DC voltage supply on catalyst regeneration.

The effectiveness of this new method was further validated by switching the voltage off and on and observing corresponding loss and recovery of the catalytic activity for multiple cycles. In the meantime, the recovered activity of Pt exhibited an exponential dependency on the DC voltage, with a higher voltage leading to a higher reaction rate (Figure 2c). Control experiments with DC voltage supply but without Pt catalyst showed little reaction, which excluded occurrence of HCOOH decomposition in the gas bulk phase, suggesting the regained HCOOH decomposition activity was attributed to the catalyst regeneration. Considering an exponential voltage-current correlation induced by the Townsend avalanche discharge mechanism, we also examined the correlation between the recovered catalyst activity and the Townsend discharge current (Figure 2d). Both the H<sub>2</sub> and CO<sub>2</sub> generation rates showed linear proportion with the measured DC current, implying a critical role of the current in regenerating poisoned catalyst. Considering the generated Ar<sup>+</sup> accounts for the current and the associated electric field direction, a plausible explanation would be that Ar\* ions migrate and electrostatically adsorb to Pt catalyst that regenerate the surface by competing against chemisorbed CO molecules and forcing them to desorb. This mechanism would also be able to explain the observation that the H<sub>2</sub> and CO<sub>2</sub> production rates gradually decreased from the initially recovered values and became eventually stabilized when the DC voltage was switched on and maintained at designated value (Figure 2b), which could be attributed to changes in Ar<sup>+</sup> generation accompanied with a capacitive charging process induced by a sudden jump in voltage. A positive DC voltage was also capable of regenerating the poisoned Pt, but to a lower extent comparing with the negative voltage of same value. Figure 3a shows the generation rates of H<sub>2</sub>, CO<sub>2</sub> and CO when an 800 V voltage was

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switched on and off, exhibiting a same trend as using -800 V but less effective activity recovering. This was likely caused by the fact that electrons rather than Ar<sup>+</sup> would electrostatically adsorb

to the catalyst surface to facilitate CO desorption at a positive voltage.



Figure 2. (a) HCOOH decomposition on the Pt catalyst as function of reaction time, (b) the catalyst activity regeneration and deactivation profile corresponding to an -800 V DC voltage being switched on (with red topping on the black column) and off (black column), (c) the influence of the DC voltage on the catalyst activity regeneration, and (d) the established correlations among the reaction rates, the applied DC voltage and the generated DC current.



Figure 3. (a) The catalyst activity regeneration and deactivation profile corresponding to an 800 V DC voltage being switched on and off, (b) the established correlations among the reaction rates, the applied DC voltage and the generated DC current in  $N_2$  atmosphere, and (c) control experiments for examination of the Joule effect.

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Figure 4. DFT-simulated reaction pathways and energy diagram for HCOOH decomposition on Pt (111).

The poisoned catalyst could also be in situ regenerated in N<sub>2</sub> atmosphere (Figure 3b), revealing that CTEA mechanism is generic and applicable to catalyst regeneration in various reaction environment. The product generation on the applied DC voltage exhibited a same trend as Ar, but with significantly higher GC counts that suggested more effective activity recovery. This could be attributed to generation of more  $N_2^+$  species and more effective interaction of  $N_2^+$  species with Pt surface than Ar<sup>+</sup>. N<sub>2</sub> was reported to have lower ionization energy than Ar that makes it easier to generate  $N_2^+$  species with a same DC voltage,<sup>[15]</sup> as evidenced by the measurement of a larger current, and have an elastic scattering cross-section that is nearly two orders of magnitude larger than Ar,<sup>[16]</sup> which would make  $N_2^+$ CTEA competes more effectively against CO chemisorption. To exclude the possible Joule heat effect on catalyst regeneration,<sup>[17]</sup> control experiments were conducted by applying a current directly through the Pt catalyst electrode and keeping all other condition parameters unchanged. No measurable influences on the catalyst regeneration were observed with the current up to 25 mA (Figure 3c), suggesting negligible Joule heat effect in this study. These results further validated that the catalyst regeneration process induced by the DC voltage supply should be mainly attributed to the generation of an ionized species (Ar<sup>+</sup>, e<sup>-</sup>) that facilitate desorption of CO poisoning species owing to CTEA. It needs mentioned that this mechanism could be easily introduced to the industry by running the Dielectric Barrier Discharge (DBD) reactor of industrial scale [18] in Townsend Discharge region with appropriate DC voltage.

To achieve more insights of the in situ regeneration process, we conducted DFT simulations of HCOOH decomposition pathways on the Pt (111) and classical electrical discharge calculation for electrostatic adsorption of Ar<sup>+</sup> ions (see Supporting Information for more details). Figure 4 shows the DFT-calculated energy diagrams, with the data being summarized in Table S1 and in good agreement with previous

studies.<sup>[19]</sup> HCOOH decomposition on Pt was found to follow the formate route, <sup>[20]</sup> in which free HCOOH molecule approaches and adsorbs to Pt with its carbonyl oxygen and hydroxyl group (HCOOH\*). HCOOH\* dissociates into one H\* and one bidentate bridging intermediate (HCOO<sub>boob</sub>\*), which undergoes isomeric transformation to another bidentate bridging intermediate that has one oxygen interacting with two surface Pt sites (HCOO<sub>bob</sub>\*). HCOO<sub>bob</sub>\* would decompose via either dehydration or dehydrogenation mechanisms. In the dehydrogenation pathway, HCOO<sub>bob</sub>\* decomposes into CO<sub>2</sub>\* and a second H\*, followed by release of  $CO_2$  via  $CO_2^*$  desorption and generation of  $H_2$  via recombination of two H\*. In the dehydration pathway, HCOObob\* reacts with H\* to generate CO\* and H<sub>2</sub>O\*, which then desorb to form final CO and H<sub>2</sub>O products. The dehydrogenation route exhibits a significantly lower activation energy barrier compared with the dehydration route (1.14 eV versus 3.35 eV), which agrees well with the experimentally observed good selectivity of Pt catalyst towards H<sub>2</sub> production. Meanwhile, CO\* desorption route requires a big energy barrier of 2.10 eV, which is significantly higher than that for other products. It needs noted that this 2.10 eV barrier includes desorption of both CO\* and H<sub>2</sub>O\*. Considering previous studies have reported an energy barrier of around 0.6 eV for the desorption of  $H_2O^*$ , <sup>[21]</sup> the energy barrier for CO\* desorption would be around 1.5 eV in this study. This value is consistent with literature reported data that vary between 1.2 eV and 1.74 eV with different functionals and model structures of use.<sup>[22]</sup> It implies a slow desorption kinetics and consequently gradual accumulation of CO\* species on Pt surface, which would block the active sites from HCOOH decomposition catalysis. This DFT data is in good consistence with our experimental results and the identified poisoning role of CO\*. It needs mentioned that the calculated 3.35 eV energy barrier for the dehydration route in this work is close to that reported by Hu et al (2.92 eV) but larger than that in the work of Scaranto et al (1.82 eV),<sup>[19, 23]</sup> which is due to the different

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reaction pathways being followed (See Supporting Information for details).

From the perspective of Townsend discharge theory, Ar would be ionized by acceleration of free electrons upon applying an electric field. The generated Ar<sup>+</sup> would be accelerated by the field and collide to deionize more Ar molecules, leading to charge avalanche multiplication. The mean kinetic energy ( $\varepsilon$ ) of Ar<sup>+</sup> ions striking the negative electrode was estimated to be 11.7 eV with 800 V DC voltage under the experimental condition in this study using the Townsend discharge theory<sup>[24]</sup> and symmetric charge-transfer cross section data of Ar from previous studies.<sup>[25]</sup> This  $\varepsilon$  value would lead to strong CTEA of Ar<sup>+</sup> to Pt that competes with other chemically adsorbed species. The impact of CTEA of Ar<sup>+</sup> on the Pt catalyst was evaluated by calculating the concentration of vacant active sites  $(C_{\nu})$  with the Pseudo Steady-State Hypothesis (PSSH) method (See Supporting Information for details).<sup>[26]</sup> The calculations show strong CO chemisorption caused a low  $C_v$ . In comparison, the presence of Ar<sup>+</sup> ions and their CTEA to the negative electrode (i.e., the Pt catalyst surface) would compete with CO chemisorption. Because the  $\varepsilon$  was significantly bigger than the CO desorption energy (11.7 eV vs. 1.5 eV), the concentration of vacant active sites  $(C_{\nu})$  in presence of Ar<sup>+</sup> was found to be dramatically higher than  $C_{v}$ . In other words, the catalyst would be regenerated by creating more vacant active sites with Townsend discharged Ar<sup>+</sup> by applying an appropriate electric field. It needs noted strong electrostatic adsorption of Ar<sup>+</sup> ions resultant of a high  $\varepsilon$  would possibly cause catalyst surface rearrangement, which could induce changes in the activity property as well. Nevertheless, the fact that the catalyst became deactivated rapidly after turning off DC voltage indicated that the observed regaining of catalyst activity with Ar<sup>+</sup> ions was mainly caused by active site regeneration. Our rough estimation of the power energy input needed per HCOOH molecule decomposition gave a small value of 0.0023 eV/per HCOOH, which was minimal compared to energy barriers required for the reaction and indicated cost effectiveness of this new competitive electrostatic adsorption mechanism (See Supporting Information for details).

In summary, we demonstrated the concept of utilizing CTEA to compete with poisoning species chemisorption for in situ regeneration of poisoned catalyst, especially for the catalyst poisoned by reversibly chemisorbed species. We verified effectiveness of this new concept by studying HCOOH decomposition as one model reaction and examining the effects of CTEA of Ar<sup>+</sup> on the reaction properties. By applying a moderate DC voltage to create an electric field that generates Ar+ ions following the Townsend discharge mechanism, the deactivated Pt catalyst exhibited an immediate recovery of the activity. The extent of the catalyst activity recovery was discovered to increase proportionally to the measured Ar<sup>+</sup> current, which was attributed to a larger number of Ar<sup>+</sup> ions that facilitate the desorption of chemisorbed CO poisoning species to regenerate the active sites via the CTEA mechanism. DFT simulations together with Townsend discharge theory suggested that the electrostatic adsorption energy of Ar<sup>+</sup> ions was dramatically bigger than the desorption energy of poisoning CO (11.7 eV @ 800 V vs. 1.5 eV), which would force CO desorption to recover availability of the Pt active sites. The findings suggest CTEA mechanism offers a new, convenient and effective method to in situ regenerate poisoned catalyst materials. With the catalyst itself being significantly recovered at cost of a minimal power input (at 0.1 W magnitude), this new method shows a good potential to help reduce the operation cost associated with poisoned catalyst replacement in many reaction processes.

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**Keywords:** catalyst regeneration • electrostatic adsorption • Townsend discharge • catalyst poisoning • formic acid decomposition

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