Continuous Argon Plasma Synthesis of Palladium Nanoparticles

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Abstract: In this contribution, we reported a novel continuous flow method for the synthesis of Palladium (Pd) nanoparticles in a water-ethylene glycol mixture under the non-thermal Argon (Ar) plasma (NTP). The synthesis of the Pd nanoparticles is confirmed using ultraviolet-visible (UV-Vis) spectroscopy. Current findings suggest further optimization of the size- and facet-controlled nanoparticle synthesis process.

1. Introduction

Palladium (Pd) is incredible due to its extraordinary H2 storage capacity of up to 900 times its volume at standard temperature pressure (STP) conditions. Pd is an excellent catalyst for the (de)hydrogenation reactions and petroleum cracking in its finely divided form. Metallic Pd and its compounds also catalyze some prominent carbon-carbon bond-forming reactions in organic chemistry, such as the Suzuki or Heck coupling. The catalytic converters employed Pd as an efficient catalyst for hydrocarbons, carbon monoxide (CO), and nitric oxide (NO) conversion up to 90% [1-2].

In this work, we reported the rapid synthesis of the Pd nanoparticles using continuous liquid flow under Ar-plasma under non-thermal conditions, followed by synthesized Pd nanoparticle characterizations using UV-vis spectroscopy.

2. Methods

An initial solution of 1 mM in a water-ethylene mixture (70:30) was prepared by mixing a known amount of Sodium tetrachloropalladate (II) (Na_2PdCl_4) (1 mM) (Thermo Fischer Scientific) and placed in a four-neck flask. The details of the experimental setup and the plasma reactor operational conditions are reported in our previous work [3]. The reaction was run for 1 hour, and the samples were taken using the known amount for the UV-vis spectroscopy before and after the reaction.

3. Results and Discussion

The formation of the Pd nanoparticles has been studied using UV-vis spectroscopy within the 300-700 nm region range. **Figure 1** revealed the UV-vis spectra of the fresh sample before the plasma operation and after one hour of the plasma treatment. The results showed no significant observable peak at 300 nm, indicating the complete reduction of the Pd(II) in the metallic Pd nanoparticles. Researchers such as Yonezawa et al. [4] and Ho et al. [5] have reported similar results. These results also indicate that the synthesized Pd nanoparticles might have particle sizes of less than 10 nm, which needs further characterization, such as transmission electron microscopy (TEM), for the particle size analysis and distribution [6].

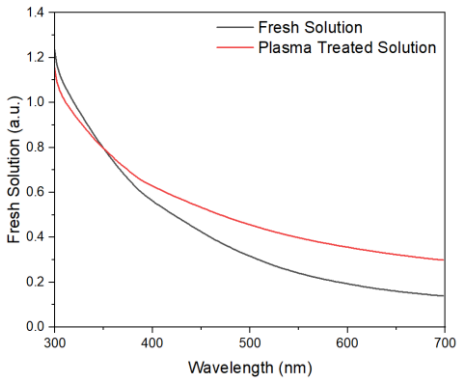

Fig. 1. UV-vis spectra of the fresh and plasma treated solution for the Pd nanoparticle synthesis under Ar-based NTP.

4. Conclusion

This work successfully demonstrated a rapid synthesis method for synthesizing the Pd nanoparticle using the Ar-based NTP in a water-ethylene glycol mixture. The ethylene glycol in water behaves as a stabilizing and mild reducing agent, preventing the oxidation of the synthesized Pd nanoparticles and avoiding the agglomeration to maintain the nanoparticle size. We recommend x-ray photoelectron spectroscopy (XPS) and TEM to investigate the oxidation state, particle shape, facet, and size, respectively.

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References

[1] Younan Xia, et al., Angewandte Chemie International Edition 48, no. 1 (2009): 60-103.
[2] Rupprechter G, et al., Nano Today. 2007; 2:27.
[3] Dinithi Mohotti, et al., Separation and Purification Technology 354 (2025): 128940.
[4] Yonezawa, et al., Langmuir 2001, 17, 4701.
[5] Ho, et al., Nanotechnology 2004, 15, 1059.
[6] Abderrafik Nemamcha, et al., The Journal of Physical Chemistry B 110, no. 1 (2006): 383-387.