A METHOD OF PREPARING PALLADIUM DENDRITES

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Problem Statement

- **Palladium** is important **catalyst** in reactions involving **electro-oxidation** and **reduction of oxygen**.
- There are **various** palladium nanostructures **preparation method**, wherein the **SPE reactors** are used for the **hydrogenation** of organic compounds.
- However, **untreated carbons** are often **hydrophobic** in nature that allows **poor adsorption** of catalyst precursors and catalysts.
- The **deposition of metals** on the electrochemically activated carbon black substrates favored a good deposition and well dispersion, but with **spherical morphology**.

Hence there is a need to develop an improved method to overcome above-mentioned issues.

Technology Category/ Market

Chemical Engineering: Material Science
Industry: Manufacturing of Catalyst & Chemical synthesis, Food & Beverage Industries
Applications: Field Of Fuel Cells, Organic Synthesis, Production of Benzene, Allyl Alcohol, Chloroalkali Process, Hydrogen Storage and Sensing.
Market: The global Palladium Catalyst market size was valued at USD **547.99** M in 2022, expected with **CAGR** of **4.79%**, reaching USD **725.68 million** by **2028**.

Technology

A method of preparing **palladium dendrites** without using a **template, surfactant and additive** comprising:

Coating carbon on a graphite substrate by dispersing carbon powder in a mixture of an ionomer and a solvent followed by blending

Activating the carbon surface electrochemically by potential cycling in an acidic electrolyte

Electrodepositing palladium on the electrochemically activated carbon coated graphite substrate by potential cycling for 10 to 25 cycles using palladium chloride as a precursor at a conc of 1.5 mM to 3 mM

Wherein, the graphite substrate is **graphite electrode**, and the carbon is **Vulcan XC-72R**, functionalized **Vulcan XC-72R**, carbon nanotubes (**CNT**), functionalized CNT, and made from **wood apple fruit or graphene**. Refer to **FIG 1 & 2.**

- The **morphology** of the palladium particles is tailored from **spherical to dendritic structure:**

a) by increasing electrochemical activation cycles from **25 to 100 cycles**
b) by increasing the metal deposition cycles from **10 to 25 cycles**
c) by increasing loading of the carbon substrate from **100 µg cm-2 to 400 µg cm-2**
d) by increasing the precursor concentrations from **1.5 mM to 3 mM**

- wherein controlling the activation cycles, deposition cycles, carbon loading & precursor concentrations increases dendrites growth and initiates growth on already grown palladium dendrites. The method has following Properties:
- The ionomer is **Nafion** & the solvent is **isopropanol**, the acidic medium is **perchloric acid** with **0.01 M** strength. The blending is performed **ultrasonically** followed by air drying.
- The acidic electrolyte is **sulfuric acid** with a strength of about **0.5 M**. The electrochemical activation increases hydrophilicity and generates surface defects on the carbon substrate.

Key Features / Value Proposition

❖ ***Technical Perspective***

The **potential ranges** and number of cycles for optimizing **electrochemical activation** and **electrodeposition** in specific is disclosed in the present patent.

The potential cycling in an acidic medium has potential range of **-0.2 to 1.1 V Ag/AgCl electrode** at a scan rate of **20 mVs-1**

Palladium-based catalysts in **nanostructure forms** are ideal **electro-catalysts** due to their increased surface area and activity. They are comparatively **economic**.

❖ ***Industrial Perspective***

Enhancing the **hydrogenation reaction rates** with **better selectivity & activity** by employing palladium nanostructures as the catalyst.

Intellectual Property

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TRL (Technology Readiness Level)

TRL- **3/4** Proof of concept ready Stage

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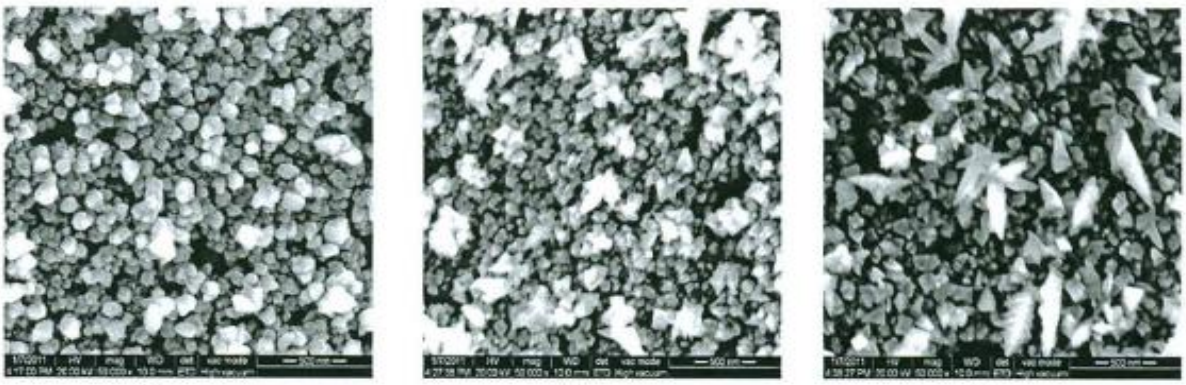
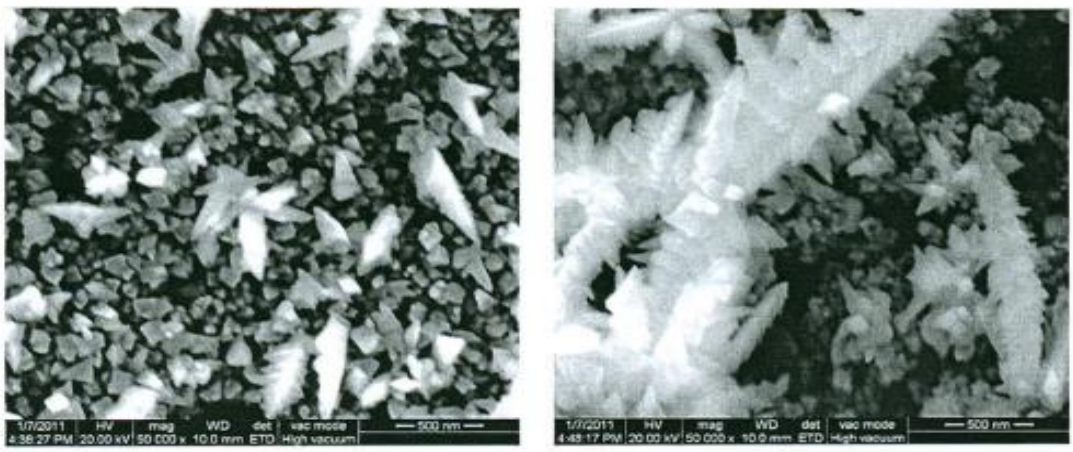


(a) (b) (c)

Figure 1 Scanning electron micrographs of Pd deposited on Vulcan XC-72R coated graphite substrate subjected to different cycles of electrochemical activation: (a) 25 cycles, (b) 50 cycles and (c) 100 cycles. Pd electrodeposited from 2 mM $PdCl_2$ in 0.01 M $HClO_4$ with 10 potential cycling between -0.2 to 1.1 V.



a) b)

Figure 2 Scanning electron micrographs of Pd deposited on Vulcan XC-72R coated graphite substrate subjected to different cycles of deposition: (a) 10 cycles and (b) 25 cycles. Vulcan coated substrate was subjected to 100 cycles of electrochemical activation, and Pd electrodeposited from 2 mM $PdCl_2$ in 0.01 M $HClO_4$.

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