



Industrial Consultancy & Sponsored Research (IC&SR)

A PROCESS FOR MANUFACTURE OF HYDROGENATED HYDROXYL TERMINATED POLYBUTADIENE (HHTPB) USING Pd-Zr-ACTIVATED CHARCOAL AS CATALYST

IITM Technology Available for Licensing

Problem Statement

- The problem statement discussed in the present invention is **how to provide an improved process for preparing hydrogenated hydroxyl terminated polybutadiene** that selectively hydrogenate the unsaturated double bond of the HTPB by addressing various factors influence the catalytic hydrogenation, i.e. **type of catalyst** that **affect the selectivity of hydrogenation** that influence the solubility of reactants including other issues.
- Hence, there is a need to provides the solution efficiently.

Technology Category/ Market

Technology: Hydrogenated Hydroxyl Terminated Polybutadiene (HHTPB) using Pd-Zr-activated Charcoal as catalyst

Industry: Advanced Material Industry;

Application: Binder for soft Explosives, missiles, portable rockets, fireworks, incendiaries;

Market: The global Hydroxyl-Terminated Polybutadiene (HTPB) market is projected to grow at a **CAGR of 12.6%** during the forecast period (2024-29).

Technology

- Present patent describes a **process for hydrogenation** of a **hydroxyl terminated polybutadiene (HTPB)** in presence of **Pd-Zr/ charcoal catalyst** and an **organic solvent** system to prepare various levels of degree of **hydrogenated hydroxyl terminated polybutadiene (5%-90%)**.
- This hydrogenated **HTPB** can be used as an **advanced binder** for **composite solid propellant system**.

The process comprises the step of:

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•selective hydrogenation to achieve various levels of degree of hydrogenation (5%-90%) of unsaturated double bond of the HTPB;

• characterized in that hydrogenating the HTPB in presence of hydrogen gas/nitrogen gas, Pd-Zr/ charcoal catalyst, and an organic solvent system at a temperature of 180°C to 270°C to obtain various levels of degree of hydrogenated hydroxyl terminated polybutadiene (5%-90%).

- Hydrogenating the HTPB in presence of **hydrogen gas, Pd-Zr/activated charcoal** catalyst, & an organic solvent system at a temperature (180°C to 270°C) to **obtain 40%-60%** degree of **hydrogenated hydroxyl terminated polybutadiene**.

Intellectual Property

IITM IDF Ref. 2690;
IN Patent No. 537079 (Granted)

TRL (Technology Readiness Level)

TRL-4, Proof of Concept ready, tested and validated in Laboratory

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Images

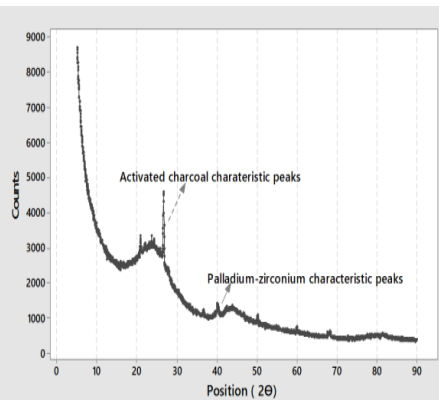


Fig.1 illustrates X-ray diffraction (XRD) of Pd-Zr/ activated charcoal catalyst;

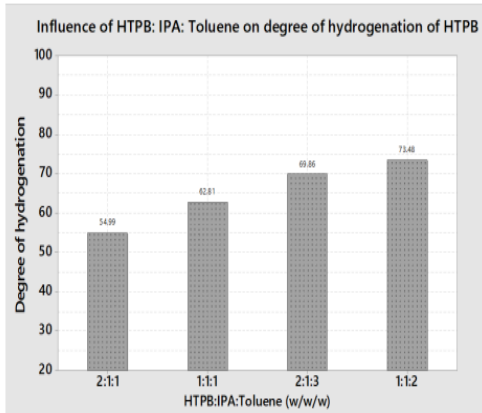


Fig.2 shows Influence of solvents ratio of IPA: toluene on degree of hydrogenation of HTPB;

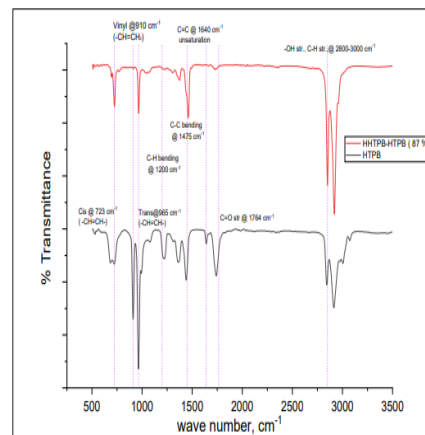


Fig.3 depicts FTIR spectrum of HTPB and HHTPB-HTPB (87%) mixture;

Key Features / Value Proposition

❖ Technical & Industrial Perspective:

❖ Experimental Setup for Hydrogenation of HTPB:

❑ The process was carried out in **reactor with magnetically driven variable speed impeller** (Fig.4). The process is carried out at room temperature for a period of **6h to 12h**.

❑ Catalyst details:

- **Pd, Zr and charcoal** in the catalyst is in the weight ratio of **2% to 5 % palladium, 5% to 8 % zirconium and 87% to 93 % charcoal** based on total weight of catalyst. (Fig. 3)
- The amount of **catalyst** is in the range of **1.5 to 2.5 %** of the **hydroxyl terminated polybutadiene**.
- **Charcoal** is **activated charcoal**.
- **Organic solvent system** is selected from **isopropyl alcohol, toluene, cyclohexene, methanol, or combination thereof**.

❑ Utility:

- The present invention provides the use of the selectively partially hydrogenated hydroxyl terminated polybutadiene for the **binder of a composite solid propellant system, polymer modification, synthesis of polyurethanes (thermoplastic and thermo-setting), encapsulants, adhesives and sealants, high temperature grease, and coatings** etc.
- The **composition and methods** are effective in the synthesis of **HHTPB in large scale** for making **solid propellant industries** like ISRO, DRDO, Ordnance factories and others.

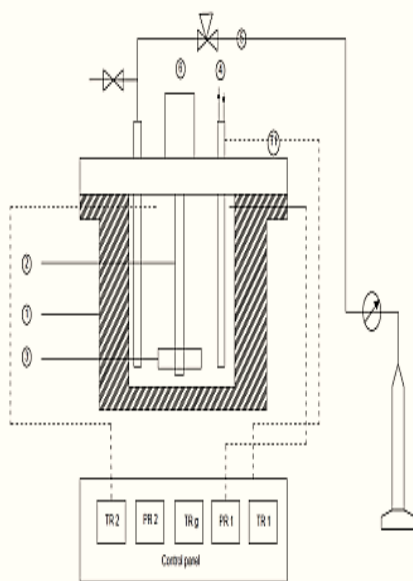


Fig.4 depicts Schematic representation of experimental set-up for hydrogenation of HTPB (1) Reactor made up of Inconel, (2) magnetic stirrer, (3) agitator blades, (4) cooling water, (5) sample valves for hydrogen/nitrogen supply, (6) magnetically coupled drive for the stirrer and reaction mechanism for hydrogenation of HTPB.

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