

**SYNTHESIS, CHARACTERIZATION, OPTIMIZATION
AND CATALYTIC ACTIVITY OF PALLADIUM(II)-4-
NITROMETHOXY-*N'*-(3-NITROBENZYLIDENE)
BENZOHYDRAZONE COMPLEX
IN HECK REACTION**

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ABSTRACT

SYNTHESIS, CHARACTERIZATION, OPTIMIZATION AND CATALYTIC ACTIVITY OF PALLADIUM(II)-4- NITROMETHOXY-*N'*-(3-NITROBENZYLIDENE) BENZOHYDRAZONE COMPLEX IN HECK REACTION

Palladium metal offers various applications in the field of fine chemicals. As one of the most widely used catalytic metal due to its high activity and selectivity for carbon-carbon formation in the organic synthesis. This study focused on catalytic performances of palladium(II) – hydrazone complexes namely Palladium(II)-4-nitromethoxy-*N'*-(3-nitrobenzylidene) benzohydrazone as catalyst in Heck reaction. The hydrazone ligand has been derived from refluxing the mixture of 4-methoxybenzohydrazide and 3-nitrobenzaldehyde in methanol while the palladium(II) – hydrazone complex was prepared by reacting palladium chloride with the synthesised hydrazone ligand. The synthesised of palladium(II) – hydrazone complex were characterized by using Fourier transform infrared (FTIR), ¹H and ¹³C nuclear magnetic resonance (NMR) and UV-Vis spectroscopies. The melting point of hydrazone ligand in the range 188 °C – 189 °C meanwhile, the melting point of palladium(II) – hydrazone complex in the range 220 °C – 221 °C. The presence of important functional group in the palladium catalyst were confirmed by FTIR spectroscopy meanwhile, ¹H and ¹³C NMR results were confirmed the number of hydrogen and carbon in the hydrazone ligand and palladium(II) – hydrazone complex. The absorption maxima of Pd²⁺ cation and hydrazone ligand were showed through the results of UV-Vis spectroscopy. The preliminary complexation between Pd²⁺ cation and hydrazone ligand shows stoichiometric of 1 Pd²⁺ cation and 1 hydrazone ligand. Catalytic activity of palladium(II) – hydrazone complex was tested in the Heck reaction which is carbon-carbon coupling reaction between 1-bromo-4-nitrobenzene and styrene. The product of the reaction was characterized by using FTIR to confirm the successful of reaction by using the palladium(II) – hydrazone complex as catalyst. Two parameters have been optimized which are effect of different catalyst loading and reaction time. The catalytic activity of palladium(II) – hydrazone complex was determine by using Gas chromatography spectroscopy. The excellent catalytic activity is achieved using catalyst loading 0.5 mmol % and 60 minutes of reaction time in DMA solvent with the presence of Na₂CO₃ as a base.