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The Study Redox Catalytic of KARES@PdNPs Using Formic Acid

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Abstract: This study aims to synthesize Calixresorcine material and KARES@PdNPs nanoparticles as catalysts to support the synthesis of PdNP nanoparticles. The process of interaction of the hydroxy groups with palladium (II) cations led to a significant decrease and increase in the quality of the nanoparticles and could increase the specific quality of KARES@PdNPs nanoparticles and increase the catalytic activity of KARES@PdNPs with a very high number of PdNPs. Thus, the KARES@PdNPs is very suitable and shows a very efficient catalytic ability in the redox reaction process.

Keywords: Calixresorcine, KARES@PdNPs, catalytic, reduxtion toxiccity.

INTRODUCTION

The palladium nanoparticles were found to be very sensitive and reactive to many carbon-carbon coupling reactions. Considered to be able to catalyze the Heck reaction of carbon-carbon coupling according to [1]. The preparation of metal nanoparticles by the reduction method involves reducing metal ions to zero oxidation state. To prevent aggregation, after reduction, the nanoparticles are stabilized by incorporating a stabilizer, such as PANI. So the preparation of metal nanoparticles in which the reactants can act as reducing agents and stabilizers for the process of forming metal nanoparticles. Various types of stabilizers and reducing agents have been used in the preparation of PdNP nanoparticles [18]. The Palladium cycle is an important model complex because of its very high catalytic activity in various carbon-carbon formation reactions. It has been demonstrated that the palladium cycle is a catalyst that acts as a source of nanoparticles and acts as a catalyst for free palladium as a homogeneous ligand. This cycle is very unstable under the conditions of the Mizoroki reaction and undergoes decomposition, with the insertion of alkenes into the carbon-palladium bonds and subsequent reduction. By using the Palladium cycle it has been found that the catalytic activity can be maintained in water systems and can be used repeatedly [5]. The reduction process works slowly and only works well if the amount of acid is increased. Formic acid, very different from other types of carboxylates, has a strong reducing ability and can undergo decarboxylation in the presence of a transition metal catalyst and without the presence of intermediate compounds, making it interesting to use as a changing agent as a reducing agent for Cr(VI) [6]. Catalysts combining polyaniline with metal ions may pose practical difficulties due to the low solubility of some solvents, which does not make them completely heterogeneous. The consumption of PANI for product synthesis is also quite high. in some solid products, materials, and then treating them with metal ions to prepare stronger heterogeneous catalysts. Despite the presence of PANI support with silica gel, the less acidic celite was chosen as the solid inorganic support for its heterogenization [16]. Model of the method based on the palladium nanoparticles on filter paper as the catalyst and very high efficiency the PdNP nanoparticles are coated with oleyl-amine, dispersed in organic solvents, and strongly adsorbed on cellulose, which shows a strong ability to adsorb liquids due to its microfiber structure. The prepared PdNP paper substrates were tested for several reaction models such as aryl-boronic acid as an oxidator. Suzuki coupling reaction, and reduction of nitro to amines. Displays highly efficient catalytic activity [20].

METHODS

Materials

Synthesis Material of KARES@PdNPs; According to [20], $K_2Cr_2O_7$, ethanol, aquadest.

Synthesis of KARES@PdNPs nanospheres

All chemicals of Calixresorcine were prepared based on the procedure according to [12], weighed 1 gram of Calixresorcine dissolved in distilled water, stirred for 60 minutes at 50^0C, and added 20 mg of $PdCl_2$ and refluxed for 6 hours. The success of the product indicated that there had been a change in solids, namely the initial pale-yellow solid became a white-darkened solid. After stirring, the KARES@PdNPs solution turned into black powder and then dried at 50^0C for 24 hours. Yields of KARES@PdNPs were in the form of black powder as a catalyst and were analyzed by IR and UV-Visible.

Qualitative of Catalytic Redox Test

Potassium bichromate solution is used to study the catalytic process because there is a transfer of electrons from the ligand to the metal. By using KARES@PdNPs as a catalyst in the catalytic process for 30 minutes, this process is complete which is marked by a change in color which indicates a process of catalytic activity. The reduction reaction is complete when the color of the initial solution has changed from yellow to colorless.

RESULT AND DISCUSSION

In Figure 2 FT-IR Investigation of KARES and KARES@PdNPs. There are absorption areas in the area of 3350 - 3400 cm^{-1} between KARES where absorption is very high compared to KARES@PdNPs which is very low, this is due to the interaction between KARES and KARES@PdNPs.

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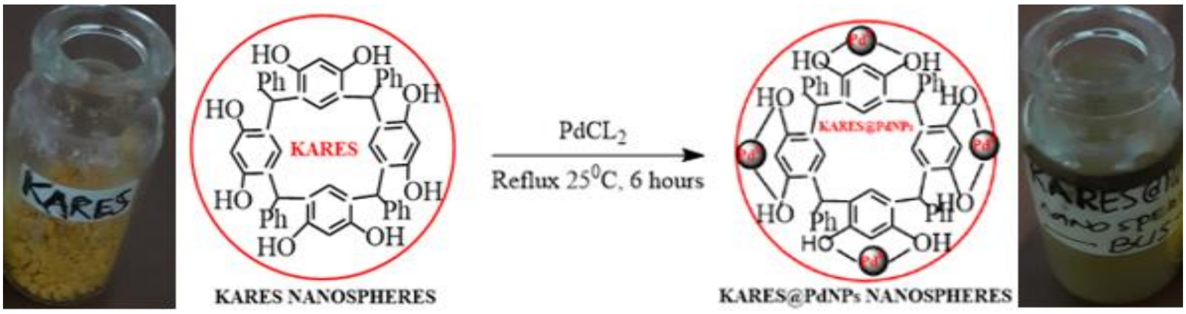

Figure 1. Reaction of Calixresorcine (KARES) using $PdCl_2$

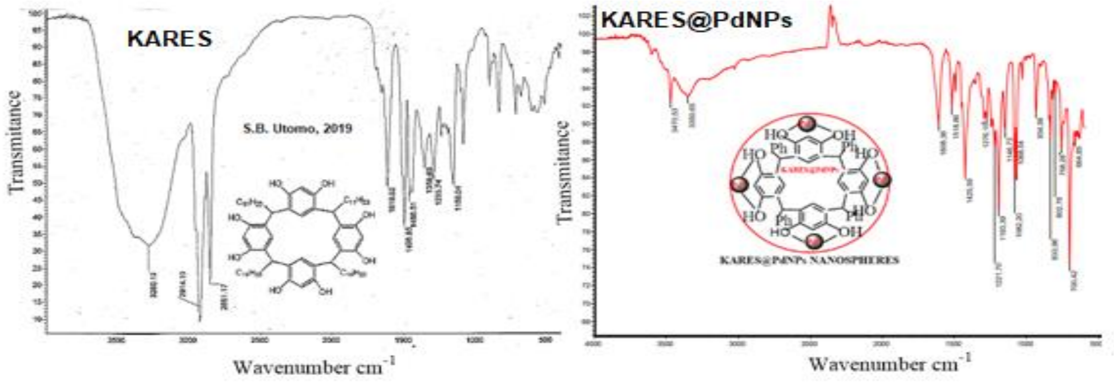

Figure 2. Spectra FTIR of KARES [12], and KARES@PdNPs

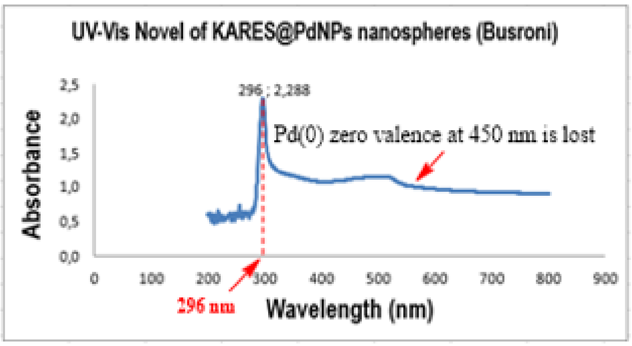

Figure 3. UV-Vis Analysis of KARES@PdNPs nanoparticle

This can be seen in Figure 2 which shows different colors in KARES to KARES@PdNPs indicating the formation of PdNPs. Literature studies according to [2, and 7] that black is the color KARES@PdNPs.

Investigation of UV-Vis.of KARES@PdNPs nanoparticle

Figure 3 Investigations using UV-vis spectroscopy. Evidenced by a change in the yield color is black powder, absorption in Figure 3 where KARES@PdNPs nanoparticles had been formed. Thus the results of KARES@PdNPs nanoparticles as catalysts and used for catalytic tests. UV-Visible spectroscopy is mainly used to evaluate that KARES@PdNPs nanoparticles have occurred, and there has been a color change from yellow to black as seen in Figure 3, according to research results according to [2, 7].

In Figure 3 KARES@PdNPs nanoparticles are the result of investigations using UV-visible for maximum absorption of palladium nanoparticles. To the knowledge of the researchers, no one has reported the use of changing Calixresorcine (KARES) to KARES@PdNPs nanoparticles, in Figure 3, have prepared PdNPs using palladium chloride and new Calixresorcine (KARES) in water without using external reducing agents. Thus, KARES@PdNPs can form colloids in water.

Catalytic Redox Test

The material of KARES@PdNPs nanoparticles as a catalyst process for the reduction of Cr(VI) to Cr(0) where the color changed from yellow to black. KARES@PdNPs were added HCOOH to an aqueous mixed solution. Potassium bichromate was followed by magnetic stirring, for 30 minutes in equilibrium. Then, formic acid is added to start the catalytic reaction as in Figure 4 below.

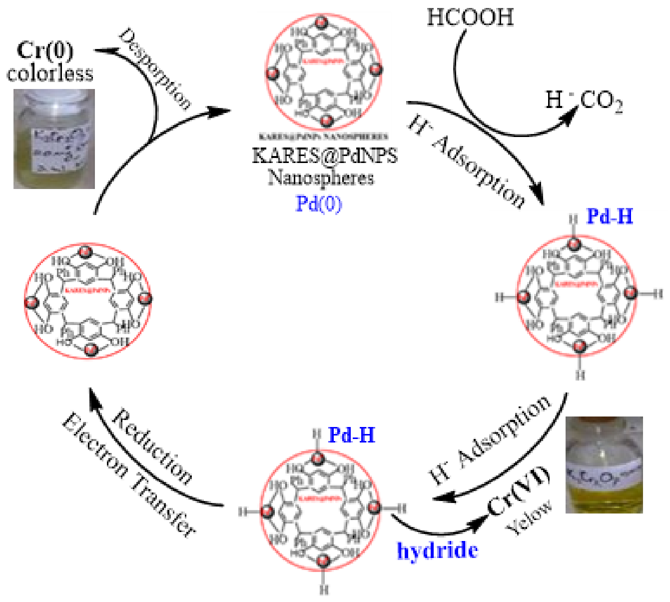

Figure 4: The Heck-type of Catalytic Redox Test of KARES@PdNPs onto K2Cr2O7 Using HCOOH

In Figure 4 according to the research results, it is suspected that transition metal hydrides and many metal hydrides are formed with hydrogen gas on the surface of transition metals. Most likely formed by transition group elements for which interaction models are not well understood, only palladium has been studied by researchers [13, 16] and some of its derivatives appear to form certain hydrides. Studies of alternative reducing agents using promising alternative methods. The slow decomposition of formic acid in principle offers the potential for temporally controlled hydride equivalent generation i.e. the generation rate is demand-driven [6].

CONCLUSION

The synthesis of KARES@PdNPs nanoparticles and PdNPs dispersed on the surface of KARES@PdNPs nanoparticles as a catalyst. Thus, the catalyst showed very efficient catalytic activity towards Cr(VI) reduction and showed excellent potency.

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