UNIVERSITI TEKNOLOGI MARA

THE EFFECT OF PREPARATION PARAMETERS ON THE SIZE OF Ag-Co NANOPARTICLES SUPPORTED ON SILICA RICE HUSK FOR THE LIQUID-PHASE OXIDATION OF CYCLOHEXENE

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ABSTRACT

Nanosized Ag-Co particles were successfully prepared via sol-gel method using silica extracted rice husk (RH) as the support. The synthesizing parameters such as pH, temperature, type of reducing agents and concentration of Ag-Co precursors were varied. The smallest particle size of 3.28 nm was registered when bimetallic Ag-Co nanoparticles was synthesized at pH 3, using glucose as reducing agent, with 0.1 wt.% Ag and 20 wt.% Co at room temperature. Along with the optimized Ag-Co nanoparticles (AgCoGlucose-NP), the parent silica (SiO_2-RH), monometallic Ag (AgGlucose-NP) and Co nanoparticle (CoGlucose-NP), and bimetallic Ag-Co nanoparticles without glucose (AgCo-NP) were also prepared under the optimized condition. Several characterization analyses such as FT-IR, TEM, XRD, DR/UV-Vis, N_2 adsorption-desorption, AAS, ^{29}Si MAS NMR, SEM/EDX/Mapping and Analysis Images Processing Cell Sense for particle size measurement were used to characterize the prepared materials. The successful inclusion of Ag-Co onto silica framework were evidenced by AAS analysis. About 13.1 wt.% Co and 0.1 wt.% Ag were successfully analyzed by AAS analysis for AgCoGlucose-NP. TEM and SEM analyses evidenced the formation of spherical shape particles on the silica surface. The metal-silica interaction of Si-O-M^+ and M-O bond were observed by FT-IR and XRD analyses. However, the peaks for Co-O and Co_3O_4 were not observed on XRD in AgCoGlucose-NP and AgCo-NP spectra. This phenomenon occurs due to well-dispersion of Co cations onto silica. While, the ^{29}Si MAS NMR analysis showed the presence of sidebands for cobalt metal loaded material. The DR/UV-Vis analysis proved the presence of Co^{2+} and Co^{3+} in all materials however, the peak of Ag^+ at 200 nm and 420 nm were not detected for AgCo-NP which proved the requirement of glucose to reduce the metal. The prepared materials were evaluated in the oxidation of cyclohexene and the reaction was run at 55°C for 6 h using 0.05 g catalysts, 25 mmol of cyclohexene, 75 mmol aqueous H_2O_2 and 10 mL acetonitrile, yielding 2-cyclohexene-1-ol and 2-cyclohexene-1-one as the product. AgCoGlucose-NP recorded the highest conversion of 98.17% while AgGlucose-NP showed the lowest conversion of 89.13%. The synergistic effect of bimetallic Ag-Co metal was found to aid the catalysis. The metals itself can act as active sites thus having two or more metals species is believed to improve the conversion. Furthermore, the conversion was increased upon increasing the pore diameter, following the trend; AgGlucose-NP (2.63 nm) < CoGlucose-NP (8.15 nm) < AgCo-NP (10.84 nm) < AgCoGlucose-NP (17.95 nm). The reactant can easily attach with inner active sites of catalyst thus increasing the conversion.

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