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Optimization of supported Bimetallic (Fe-Co/CaCO₃) catalyst synthesis parameters for Carbon Nanotubes Growth using Factorial Experimental Design

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Abstract

The Fe-Co bimetallic catalyst supported on CaCO₃ was prepared by a wet impregnation method. The interactive effects among the key synthesis parameters such as drying time, calcination temperature, stirring speed, mass of CaCO₃ support on the yield and quality properties of the catalyst were investigated using 2⁴ Factorial design of experiment. Additionally, the catalyst obtained under the optimal conditions was used to prepare Multi-walled carbon nanotubes (MWCNT) via chemical vapour deposition (CVD) of acetylene gas in a tubular horizontal reactor. The quality of the prepared materials was examined for their morphology, microstructure, elemental composition, surface chemical properties, thermal stability, surface area, and mineralogical phase by HRSEM, HRTEM, EDS, TGA, FTIR, BET and XRD. The results revealed that maximum catalyst yield of 99.6% obtained at the drying time of 12 h, calcination temperature of 100 °C, stirring speed of 1000 rpm and mass of CaCO₃ support of 10 g favored the formation of MWCNT at 700°C and 60 min reaction time. The results of statistical analysis demonstrated a direct relationship and synergetic effect between stirring speed and mass of support and the two factors exerted highest impact on the yield of catalyst than other parameters. Microscopy analysis revealed successful dispersion of Fe and Co particles onto CaCO₃ support while XRD patterns confirmed the presence of highly active crystalline mixed oxide (CoFe₂O₄) as the dominant phase. The HRSEM/XRD analysis displayed the formation of tubular networks of graphitic carbon materials with few traces of by-products. The study established the absorption and not diffusion mechanism of Fe and

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