Optimization of supported bimetallic (Fe-Co/$CaCO_3$) catalyst synthesis parameters for carbon nanotubes growth using factorial experimental design

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Highlights

- Development of bimetallic Fe-Co catalyst supported on $CaCO_3$.
- Effects of drying time, drying temperature, stirring speed and mass of $CaCO_3$ support on the catalyst yields.
- Application of statistical approach to establish the most significant parameters influencing the yields.
- Carbon nanotubes development using catalytic chemical vapour deposition method.

Abstract

The Fe–Co bimetallic catalyst supported on $CaCO_3$ 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 $CaCO_3$ support on the yield and quality properties of the catalyst were investigated using 2^4 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 $CaCO_3$ support of 10 g favoured 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 $CaCO_3$ support while XRD patterns confirmed the presence of highly active crystalline mixed oxide ($CoFe_2O_4$) 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 Co onto $CaCO_3$ in the reaction sequence produced $CoFe_2O_4$ during the wet impregnation process employed for catalyst preparation.

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Keywords

Optimization, Factorial design, Bimetallic (Fe-Co) catalyst, Calcium carbonate, Carbon nanotubes

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