SIZE-CONTROLLED SYNTHESIS OF NANO-NI AND ITS CATALYTIC HYDROGENATION CHARACTERIZATION

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Highlights

- nano-Ni was prepared by liquid reduction method without surfactant
- the size-controlled nanoparticles were prepared as the catalyst in rotating packed bed
- the catalytic hydrogenation characterization for the cinnamaldehyde hydrogenation showed high activity

1. Introduction

Nano-catalysis has been widely investigated by many researchers for its high activity and selectivity. In the nano-catalysis process, especially for the mono metallic catalysis, the size of mono metallic catalyst can be greatly affect the catalytic efficiency. In this work, the size-controlled nano-Ni was prepared in a rotating packed bed without adding surfactant, and the catalytic hydrogenation characterization of the prepared nano-Ni was carried out by the cinnamaldehyde (CAL) hydrogenation.

2. Methods

The synthesis process of nano-Ni without adding surfactant was carried out in the rotating packed bed (RPB) by liquid reduction method. The 50 ml reducing mixed liquid contained 4.8 M N_2H_4, 1 M NaOH and 0.06 M $NaBH_4$ was pumped into the RPB. Also, 50 ml of 0.8 M $Ni(SO_4)_2$ was pumped into the RPB. The reaction was carried out in the RPB to intensify the micromixing of the reaction system. And then the product was filtered, washed and dried.

The catalytic hydrogenation of CAL by nano-Ni was performed in the STR (with a four-blade impeller and a volume of 1 L). For each experiment, 30 mL of CAL, 270 mL of ethanol (CAL concentration of 795 mol/m^3) and 3 g nano-catalyst (nano-Ni) were added into the reactor for the catalytic hydrogenation characterization. The process was carried out under stirring speed of 1500 rpm, hydrogen gas pressure of 2 Mpa, temperature of 393 K. The reaction product was analyzed by gas chromatography with a gas capillary column of 30m×0.32mm×1.00m.

3. Results and discussion

Through the optimization of the operating conditions in the nano-Ni synthesis process, the nano-Ni with a cubic morphology, a uniform size particle, an average particle size of 40 nm and particle size distribution of 30-60 nm was prepared under the optimal operating conditions (as shown in Figure 1). In addition, the figure 1(b) showed that the nano-Ni has a well-defined lattice fringes and the lattice spacing 200 and 111 values are 0.199 nm and 0.179 nm, respectively.

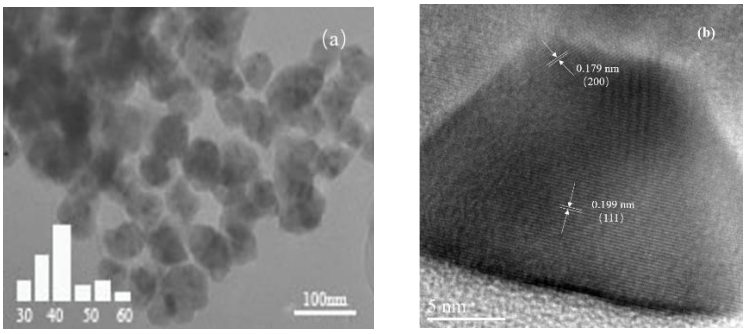

Figure 1. (a) TEM and (b) HRTEM image of nano-Ni catalyst

Figure 2 shows the concentration vs time plots of CAL hydrogenation. It can be seen that the main products were HCAL and HCOL, and the hydrogenation selectivity of C=C bond was higher than that of C=O bond. In addition, the nano-Ni initial activity of 1.5 mmol·min^{-1}·g^{-1} can be calculated through the decrease in concentration of CAL with reaction time.

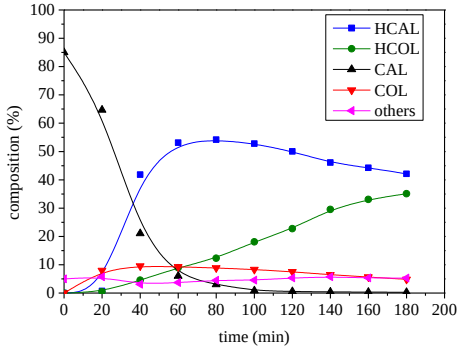

Figure 2. Production distribution of CAL hydrogenation at different reaction time

(stirring speed of 1500 rpm, hydrogen gas pressure of 2 Mpa, temperature of 393 K)

4. Conclusion

In this work, a novel synthesis process of nano-Ni without adding surfactant was investigated in the RPB by liquid reduction method. The nano-Ni with a face centered cubic structure, an average particle size of 42 nm and a particle size distribution of 30-60 nm was obtained in the RPB. The catalytic hydrogenation characterization showed that the nano-Ni has a high activity and selectivity for hydrogenation of C=C bond in the cinnamaldehyde hydrogenation process.

References

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Keywords

Nano-Ni, Rotating packed bed, Cinnamaldehyde hydrogenation