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Synthesis of Nanocatalysts via Reverse Microemulsion Route for Fischer-Tropsch Reactions

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1. Introduction

The current hike on fuel prices, the discovery of more natural gas fields and dwindling of oil reserves have renewed interests in the production of synthetic crude via the gas-to-liquid (GTL) conversion process. The chemical conversion of natural gas into fuels is also an attractive option for monetizing stranded natural gas. The GTL process consists of the following steps (Bao et al., 2010):

i.   Production of synthesis gas (a mixture of carbon monoxide and hydrogen) from natural gas
ii. Synthesis of hydrocarbon via the Fischer-Tropsch synthesis (FT) reaction
iii. Product upgrading of the synthesized hydrocarbons to yield various products such as gasoline and diesel.

Fischer-Tropsch (FT) reaction involves conversion of syn-gas (a mixture of CO and H_2) into hydrocarbons such as olefins, alcohols, acids, oxygenates and paraffins. The Fischer-Tropsch (FT) reaction, shown in Equation (1), produces clean gasoline and diesel fuels. The fuels derived from GTL technology are environmental-friendly because they contain low particulates, sulfur and aromatics.

$$nCO + (2n+1)H_2 \rightarrow C_nH_{2n+2} + nH_2O \qquad (1)$$

Catalysts used for the Fischer-Tropsch reaction are generally based on iron and cobalt (Khodakov et al., 2007). Ruthenium is an active catalyst for FT but is not economically feasible due to its high cost and insufficient reserves worldwide. Iron has been the traditional catalyst of choice for FT reaction. It is reactive and the most economical catalyst for synthesis of clean fuel from the synthesis gas mixture. Compared to cobalt, iron tends to produce more olefins and also catalyzes the water-gas shift reaction (Equation 2). Iron catalyst is usually employed in the high-temperature (573-623 K) FT operating mode (Steynberg, et al., 1999).

$$CO + H_2O \rightarrow CO_2 + H_2 \qquad (2)$$

Cobalt has higher activity for Fischer-Tropsch reaction but more expensive compared to iron. The low-temperature (473-513 K) FT process usually employs cobalt-based catalysts

due to their stability and high hydrocarbon productivity. Catalyst supports that have been utilized include silica, alumina, titania, zirconia, magnesium, carbon and molecular sieves. The cost of catalyst support, metal and catalyst preparation contributes to the cost of FT catalyst, which represents a significant part of the cost for the FT technology.

Various types of reactors have been installed in the FT industry such as fixed-bed reactor, multi-tubular reactor, adiabatic fixed-bed reactor, slurry reactor, fluidized bed reactor and circulating fluid-bed reactor systems (Bao et al., 2004; Steynberg et al.,1999). Since FT reaction is highly exothermic, temperature control and heat removal constitute the two most important design factors for the FT reactors.

FT research ranged from preparation of catalysts to design of reactors. Fundamental understanding on the relationship between the catalyst performance and its physical properties, such as particle size, surface area and porosity is vital. The deactivation behavior of cobalt has been linked to its crystallite size, therefore, control of crystallite size is of importance (Saib et al.,2006). A very stable and active catalyst is required to ensure the catalytic system is economically attractive. A model catalyst consists of well-defined catalytically active metal deposited on non-porous support (Moodley, 2008). Spherical model catalysts can be used to bridge the gap between the poorly-defined porous industrial catalysts and the well-defined single crystal surfaces. Knowledge on the relation between the rate of the reaction to the composition and morphology of the catalyst is still lacking (Sarkar et al., 2007). Thus, characterization of model catalysts can relate the physical properties, such as size and shape of particles, to the catalytic behavior of the catalytic materials.

The term nanoparticle is used for particles having diameters ranging from 2 to 50 nm with variable crystallinity, whereas well-defined crystalline nanoparticles are classified as nanocrystals (Hyeon, 2003). The commercially applied iron catalyst is in the fused form comprising large iron particles and therefore difficult to investigate using microscopy. The loss of catalyst activity is associated with changes of iron into a mixture of iron oxide and iron carbide during the Fischer-Tropsch synthesis reaction. The relation between deactivation and changes in composition and morphology are not fully understood for iron and cobalt catalysts (Sarkar et al., 2007). The application of electron microscopy techniques on the supported nanoparticles are well suited to investigate the morphology of supported catalysts and morphological changes that occur during Fischer –Tropsch synthesis. The knowledge on the effect of particle size on the product selectivity and yields for Fischer-Tropsch reaction is still lacking. It has been reported (Trepanier et al., 2010) that particle size of the catalyst material has an effect on the pressure drop in the reactor, and can influence the product distribution. Iron and cobalt nanoparticles of sizes less than 10 nm are expected to improve the kinetics of the Fischer-Tropsch reaction, selectivity for gasoline and stability of the catalyst.

This chapter provides an overview on the synthesis of FT catalysts using the microemulsion techniques as well as other common preparation techniques such as impregnation, precipitation and strong electrostatic adsorption method. It will focus on the results of catalyst characterization in terms of morphology, particle size distribution, and reducibility. FT reaction is considered as a surface-sensitive reaction in which the size of the active metal particles can influence the catalytic activity and product selectivity. The physicochemical properties of the FT catalyst prepared via microemulsion techniques shall be compared with those synthesized via other routes such as impregnation, precipitation and strong electrostatic adsorption methods. The performance of the cobalt-based and iron-based catalysts prepared via various formulations in the Fischer-Tropsch synthesis reaction will be discussed.

2. Methods of catalyst preparation

An extensive review on the development of cobalt Fischer-Tropsch catalysts was presented by Khodakov and co-workers (Khodakov et al., 2007). The performance of the cobalt catalyst in Fisher-Tropsch reaction is greatly influenced by the catalyst preparation method. The variables include suitable support, deposition method of the metal precursor, catalyst promoter, and the subsequent thermal treatments (Moodley, 2008). Cobalt has shown better resistant to deactivation and attrition, but it is also much more expensive compared to iron. Therefore, well-dispersed cobalt on the catalyst support is highly desired to gain economic attractiveness. The reactivity in FTS is correlated to the number of cobalt metallic particles exposed to the syn-gas molecules (Ling et al., 2011). This factor in turns depends on the cobalt loading, dispersion of cobalt species and its reducibility. Hence, an ideal supported catalyst would have uniformly distributed cobalt species that undergoes complete reduction, forming cobalt metallic nanoparticles at optimum size of 6~8nm, where high dispersion guarantees optimum use of cobalt without jeopardizing the FT performance. FT catalysts have been prepared by various methods such as impregnation, precipitation, colloidal method, strong electrostatic adsorption, and microemulsion method.

2.1 Impregnation

The incipient wetness impregnation method is a commonly used method for preparing cobalt and iron catalysts. Typically required amount of the precursor salt i.e. $Co(NO_3)_2.6H_2O$ is dissolved in deionized water and added dropwise to the support under constant stirring, followed by drying in an oven at 120 °C overnight and calcining at temperature of 500 °C (Saib et al., 2006). Variables which can affect the resultant catalyst are the rate of addition of precursor solution, rate of drying, temperature and duration of heating. Cobalt catalyst has also been prepared using the slurry (wet) impregnation method where the amount of impregnation liquid was in excess of the total pore volume of the support material (Khodakov et al., 2007). Impregnation method can produce small particles but difficult to obtain narrow particle size distribution, as depicted in Figure 1.
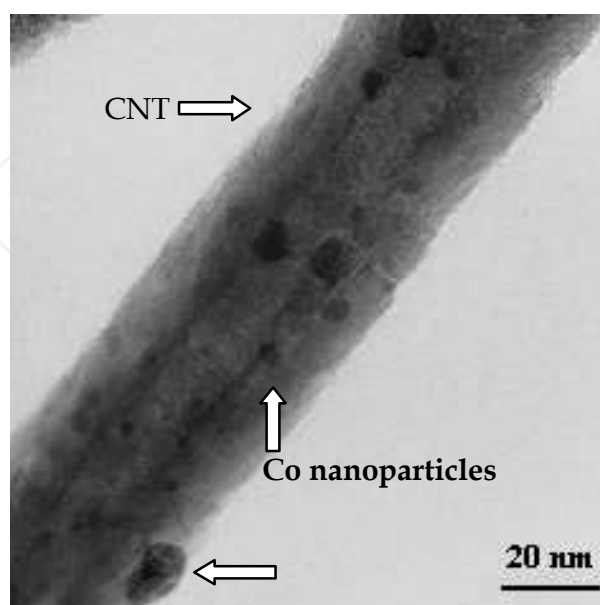

Fig. 1. TEM of 5 wt% Co impregnated on CNTs support. (Adapted from Ali et al., 2011)

2.2 Precipitation

The precipitation method is more common for iron catalyst than cobalt-based FT catalyst. The required amount of the precursor salt i.e. $Co(NO_3)_2.6H_2O$ is dissolved in deionized water and added to the support with constant stirring followed by the addition of precipitating agent i.e. ammonia (Tasfy, 2011) and the resultant slurry is stirred at slightly elevated temperature i.e. 90ºC for about 8 hours. The precipitate is dried and then calcined at 500°C.

2.3 Colloidal method

Colloids are synthesized in the presence of surfactants which disperse and stabilize the nanoparticles in an organic solvent. Some of the approaches include polyol method, ethylene glycol method, modified coordination capture method and pseudo-colloidal method. The polyol process involves heating a mixture of catalyst precursor in surfactants, such as oleic acid and oleyl amine in a high-boiling solvent, such as diphenyl ether. The high temperature alcohol reduction of iron(III) acetylacetonate metal precursor resulted in monodispersed iron nanoparticles (Sun & Zeng, 2002). This synthesis process is also called "heating-up" process. The size of the nanoparticles is controlled by changing the concentration of the precursor, the amount and type of surfactant, the aging time and temperature of the reaction. Another synthetic method that produces uniform nanocrystals that is comparable to the "heating up" process is called the "hot injection" method. The "hot injection" method induces high supersaturation and leads to fast homogeneous nucleation reaction followed by diffusion-controlled growth process, which control the particle size distribution. The colloidal method has been shown to generate well-dispersed iron nanoparticles with average size of 5 nm, as depicted in Figure 2.
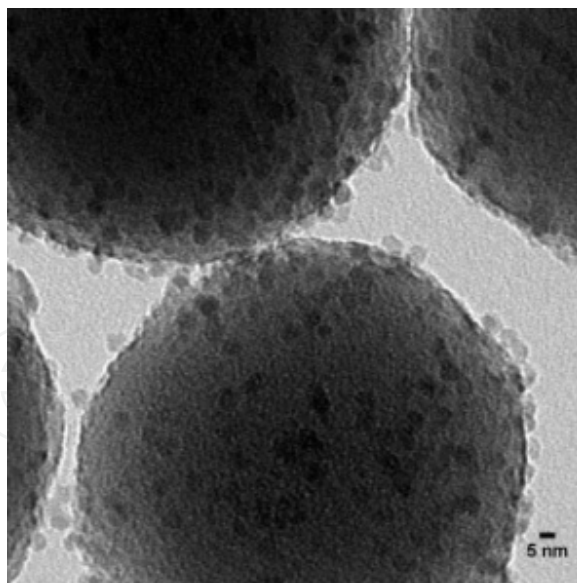

Fig. 2. TEM image of 6 wt% Fe on SiO_2 spheres prepared by the colloidal method (Adapted from Mohd Zabidi, 2010).

2.4 Strong electrostatic adsorption method

Synthesis of uniformly distributed Co nanoparticles remains a great challenge. Strong electrostatic adsorption (SEA) is a catalyst preparation method which is based on basic

concept of electrostatic attraction of oppositely charged particles (Jiao & Regalbuto, 2008). Silica and other metal oxides contain hydroxyl groups on its surface. Point of zero charge (PZC) is the pH value of a medium where the hydroxyl groups on the surface of the support remain neutral. In a pH<PZC medium, the hydroxyl groups will be protonated and become positively charged and thus attracting anions. When pH>PZC, the hydroxyl groups will deprotonate and became negatively charged and attracting cations. In other words, pH value plays an important role in the deposition of metal precursor. The SEA method was able to produce nanocatalyst of spherical-shaped Co nanoparticles on SiO_2 support, shown in Figure 3, with Co average size of 3 nm (Ling, 2011).
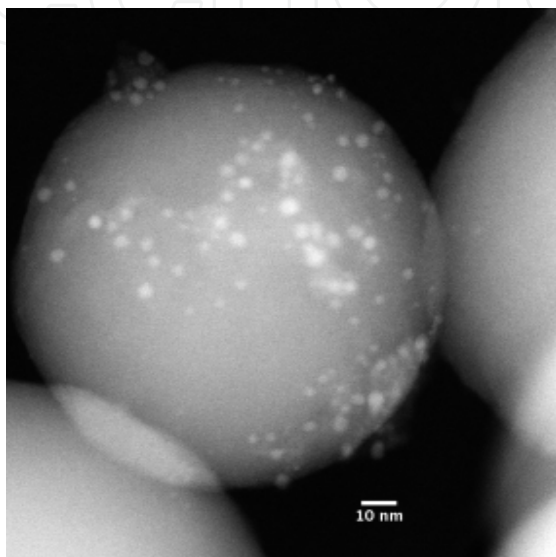

Fig. 3. STEM image of 10 wt% Co on SiO_2 spheres prepared via SEA method (Adapted from Ling, 2011).

3. Basic concepts: Microemulsion

A microemulsion is a liquid mixture of water, a hydrocarbon and a surfactant (Eriksson et al., 2004; Malik et al., 2010). A surfactant is a molecule that possesses both the polar (hydrophilic head) and the non-polar (hydrophobic tail) groups. When the concentration of the surfactant exceeds the critical micelle concentration, molecules aggregate to form micelles. When micelles are formed in an organic medium, the aggregate or tiny droplets is referred as a reversed micelle, in which the polar heads are in the core and the non-polar tails remain outside to maintain interaction with hydrocarbon. It is also referred as water-in-oil microemulsion (Figure 4). The core of the reverse (w/o) microemulsion is of interest as it can be viewed as an elegant nanoreactor which can accommodate chemical reaction. The core interior is hydrophilic thus certain water-soluble can be dissolved inside it. Microemulsion technique offers several advantageous for synthesizing nanoparticles as it enables controls of size, geometry and morphology (Malik et al., 2010).

3.1 Synthesis of nanoparticles via microemulsion technique

The properties of nanoparticles synthesized using the w/o microemulsion are influenced by several factors such as size of water droplets, surfactant concentration and nature of precipitation agent. There are two approaches of synthesizing the nanoparticles via the

microemulsion route. The first method involves mixing two sets of microemulsions containing the metal precursor and the precipitating agent or reducing agent. In the second approach, the precipitating agent is added directly to the microemulsion containing the metal precursor.
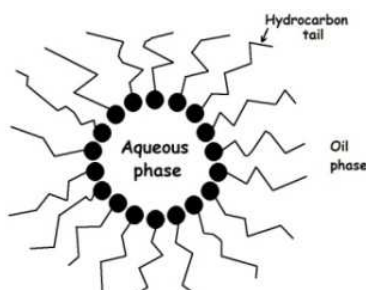

Fig. 4. Reversed micelle (Adapted from Eriksson et al., 2004**).**

3.1.1 Mixing of two microemulsion systems

The w/o microemulsion synthesis method consists of preparing two sets of microemulsions containing the metal salt and the reducing agent as shown in Figure 5 (Eriksson et al., 2004). The precursor metal salt and reducing agent are dissolved in the aqueous phase whereas the surfactant is prepared in an organic medium. For preparation of supported iron nanocatalyst, the reverse microemulsion method involved preparing two reverse microemulsions. The first reverse microemulsion consisted of $Fe(NO_3)_3 \cdot 9H_2O$ (aq) and sodium bis(2-ethylhexyl) sulfosuccinate (AOT, ionic surfactant) in hexanol and the second reverse microemulsion was prepared by mixing an aqueous hydrazine solution (reducing
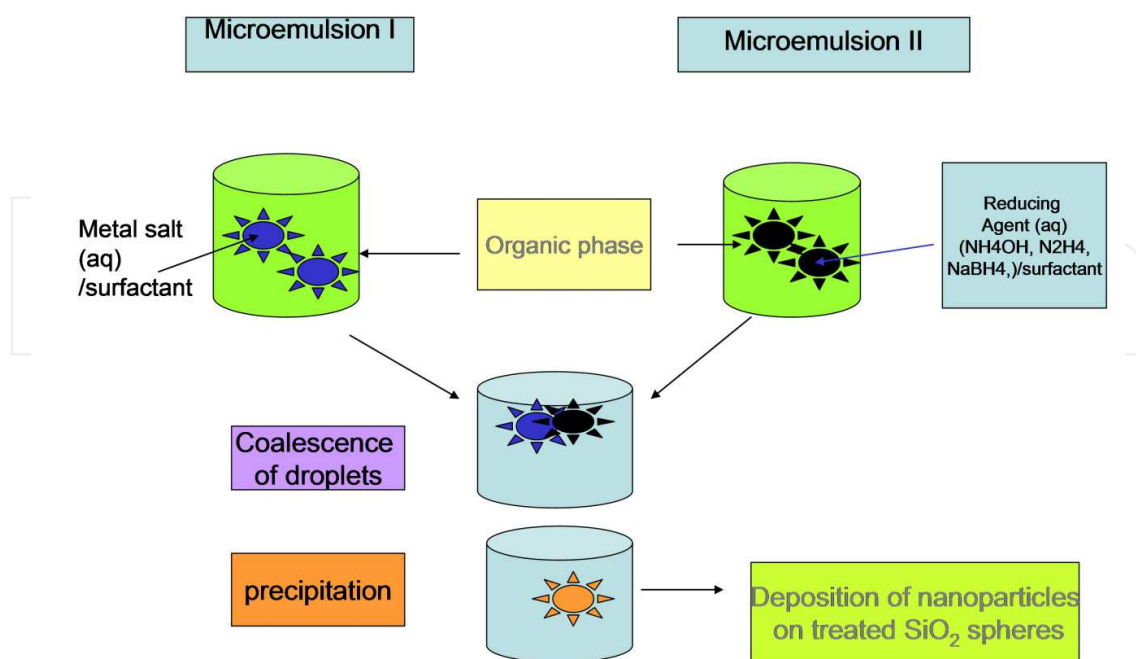

Fig. 5. Reverse microemulsion approach for synthesis of nanoparticles on catalyst support. (Adapted from Eriksson, et al., 2004).

agent) with the AOT solution (Mohd Zabidi, 2010). SiO_2 was chosen as the catalyst support and SiO_2 spheres were then added to the mixture followed by stirring for 3 hours under nitrogen environment. Figure 6 shows the TEM micrograph of a spherical model catalyst prepared using the reverse microemulsion method comprising 6 wt% Fe on SiO_2 support (Mohd Zabidi, 2010). The reverse microemulsion method produced spherical-shaped iron oxide nanoparticles with average diameters of 6.3 ± 1.7 nm, however, the coverage of the SiO_2 surfaces was found to be non-uniform.
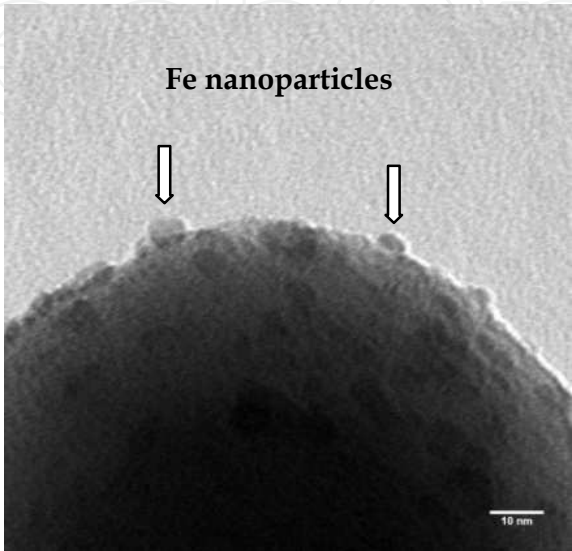

Fig. 6. TEM of 6 wt% Fe on SiO_2 support (Adapted from Mohd Zabidi, 2010)

3.1.2 Direct addition of precipitating reagent to the microemulsion

Another approach of synthesizing nanoparticles from microemulsion is via direct addition of the reducing agent or precipitating agent to the microemulsion containing the metal precursor, as depicted in Figure 7 (Eriksson et al, 2004). It was reported that 5 wt% Fe/SiO_2
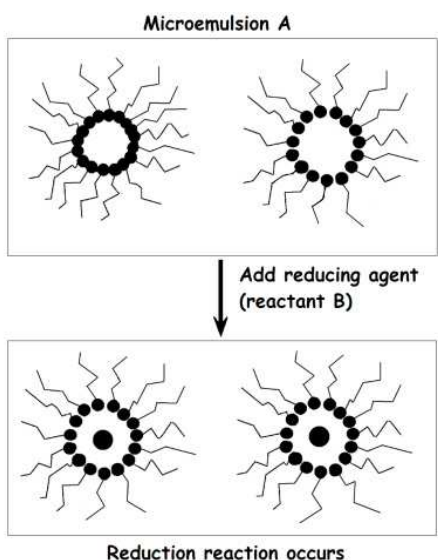

Fig. 7. Direct addition approach for preparation of nanoparticles using the reverse microemulsion method (Adapted from Eriksson et al., 2004).

prepared using the water-in-oil microemulsion resulted in Fe particles with average size of 5.2 nm (Hayashi et al, 2002). At the same Fe loading, the impregnation method produced much larger Fe particles (14.2 nm) which resulted in approximately 30% lower CO conversion in the Fischer-Tropsch reaction.

3.2 Influence of synthesis parameters on the size of nanoparticles

The polydispersity of the system and the size of nanoparticles are influenced by the nature of solvent, surfactant or co-surfactant, presence of electrolyte, concentration of reducing / or precipitating reagents and molar ratio of water to surfactant (Eastoe et al., 2006). The size of water droplets in the microemulsion, the concentration of surfactant and the nature of the reducing/precipitating agent will influence the particle size of metal prepared via microemulsion technique (Eriksson et al., 2004). Increase in water-to-surfactant ratio will increase the size of the water droplets in the microemulsion and hence the size of the resultant metal nanoparticles. Varying the water-to-surfactant ratio (R) from 5 to 50 increased the particle size of cobalt from 5.1 nm to 9.4 nm which resulted in a decrease in turnover frequency (TOF) from 8.6×10^{-3} s^{-1} to 1.2×10^{-3} s^{-1} for FT reaction conducted at 493 K, 2.0 MPa over 10%Co/ITQ catalyst (Prieto et al., 2009).

The number of droplets will increase with increasing presence of surfactant in the system (Eriksson et al., 2004). The surfactant molecules could result in steric hindrance which retard the fast rate of growth of the nuclei. Subsequently, the particles grow at the same rate and led to formation of nanoparticles with narrow particle size distribution.

3.3 Mechanism of formation of nanoparticles

The mechanisms by which nanoparticles are formed have been described by Malik and co-workers using two models (Malik et al., 2010). The first model is based on the Lamer diagram where nucleation occurs when the concentration of the precursor reactant reaches a critical supersaturation value, resulting in a constant number of nuclei thus size of particles increase with concentration (Malik et al, 2010). In the second model, variation in the concentration of precursor does not change the size of particles as the particles are thermodynamically stabilized by the surfactant.

4. Comparison of physicochemical properties between catalysts prepared via different routes

The preparation methods influenced the morphologies of the nanocatalyst particles as depicted by the TEM micrographs in Figure 8. The reverse microemulsion method resulted in relatively larger Co nanoparticles and more agglomeration compared to those synthesized via impregnation and strong electrostatic adsorption (SEA) methods. The size of Co nanoparticles synthesized using reverse microemulsion method ranged from about 5 nm to 15 nm. The coverage of SiO$_2$ surface was less than that obtained using the other two methods.

Figure 9 shows the H$_2$-TPR profile of SiO$_2$-supported cobalt nanocatalyst prepared via the impregnation and reverse microemulsion methods. The first reduction stage (around 350 °C)

corresponds to reduction of Co_3O_4 to CoO while the peak around 400 $^{\circ}$C was for the reduction of CoO to Co^0 (Fishcer et al., 2011). The Co/SiO_2 sample prepared via the reverse microemulsion method exhibited a second reduction stage at 700 $^{\circ}$C, which was much higher compared to that obtained for the sample prepared via the impregnation method indicating poorer reducibility, possibly due to strong metal-support interactions.
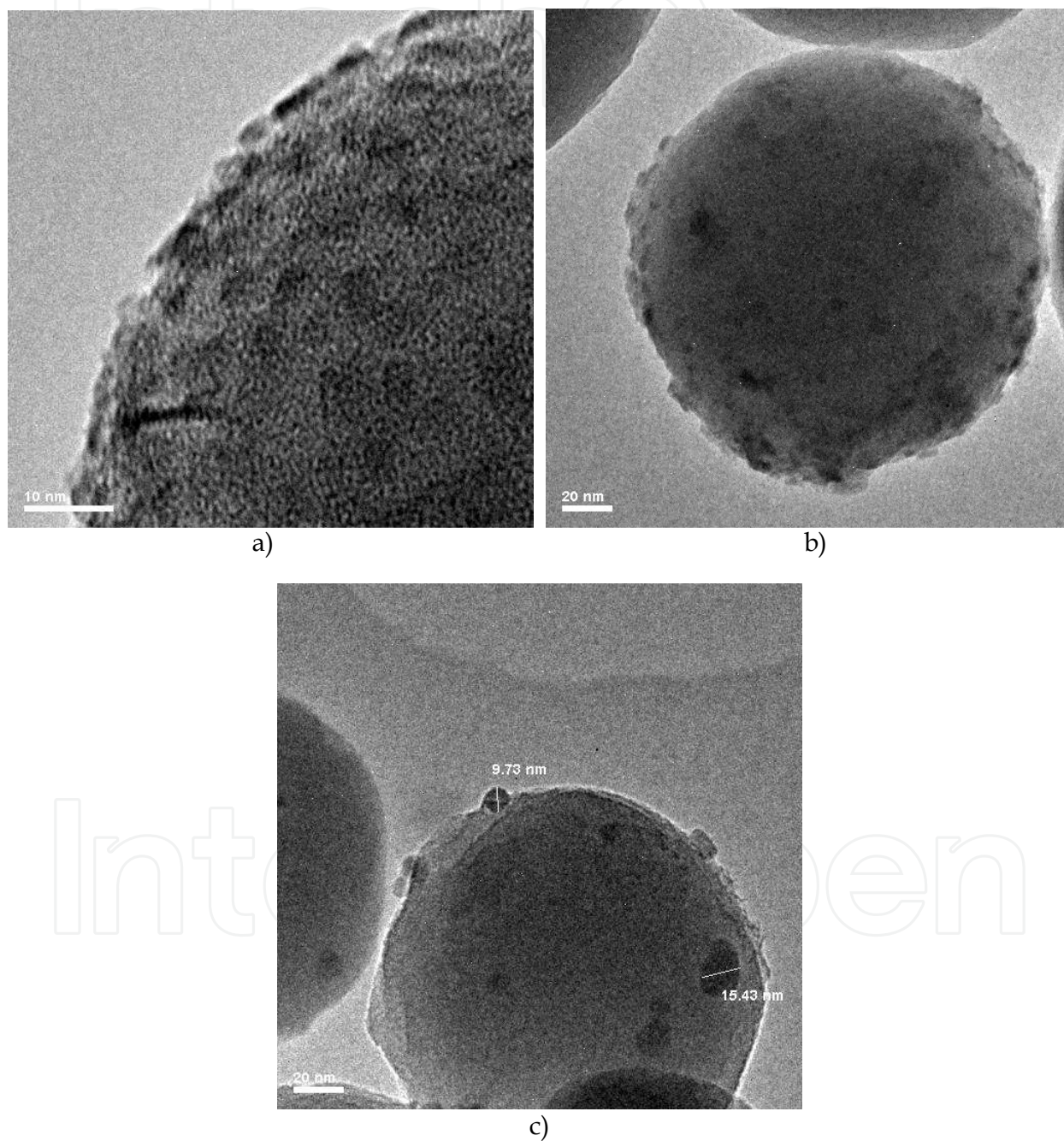

Fig. 8. TEM images of 5 wt% Co on SiO_2 support prepared via (a) impregnation (Adapted from Ali et al., 2011) (b) SEA method (Adapted from Ling et al., 2011) (c) reverse microemulsion (Adapted from AbdulRahman, 2010).
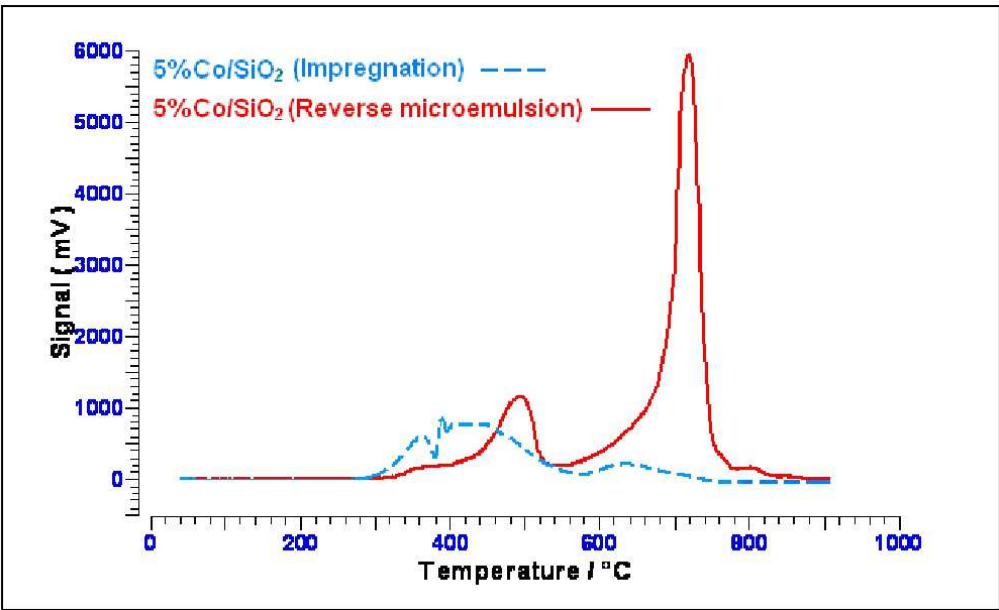
Fig. 9. H$_2$-TPR profile of 5 wt% Co on SiO$_2$ spheres prepared via impregnation and reverse microemulsion methods. (Adapted from Abdul Rahman, 2010).

4.1 Catalytic activity

The performance of the nanocatalyst in Fischer-Tropsch synthesis reaction is influenced by the catalyst's preparation route. Table 1 and 2 show the performance of supported cobalt-based and iron-based nanocatalysts, respectively, in the FT reaction. The cobalt

Catalyst	Preparation route	Particle size (nm)	X_{CO} (%)	S_{CH4} (%)	S_{C5+} (%)	Reaction conditions
10%Co/CNTs	Reverse micromulsion	7.8	62	6	92	220°C, 2MPa (Trepanier et al., 2010)
10%Co/CNTs	Impregnation	7.9	54	10	86	220°C, 2MPa (Trepanier et al., 2010)
10%Co/SiO$_2$	Strong electrostatic adsorption (SEA)	5	2	0.1	80	220°C, 0.1MPa (Ling, 2011)
10%Co/SiO$_2$	Impregnation	-	10	70	10	220°C, 0.1MPa (Ling, 2011)
5%Co/SiO$_2$	Impregnation	5	4.7	17.7	1.3	270°C, 0.1MPa (Ali et al., 2011)
5%Co/CNTs	Impregnation	3	15.7	16.4	14	270°C, 0.1MPa (Ali et al., 2011)

Table 1. Comparison of performance of Co-based nanocatalysts in FT reaction

nanoparticles synthesized using the reversed microemulsion technique was found to be more uniform and exhibited narrow size range compared to those synthesized via the impregnation method (Trepanier et al., 2010). The more uniform cobalt clusters also resulted in a decrease in the CH$_4$ selectivity and increase in C$_{5+}$ selectivity due to effective

participation of olefins in the propagation of carbon chain. The CNTs support also served as a better support for Co nanoparticles compared to those of SiO_2 as indicated by the higher value of C_{5+} selectivity likely due to confinement of the Co nanoparticles inside the channels of the CNTs (Trepanier et al, 2010).

Catalyst	Preparation route	Particle size (nm)	X_{CO} (%)	S_{CH4} (%)	S_{C5+} (%)	Reaction conditions
5%Fe/SiO$_2$	Reverse microemulsion	-	1.5	27.5	10.1	250°C,1.01MPa (Heranz et al., 2006)
5%Fe/SiO$_2$	Reverse microemulsion	5.2	11.1	18.2	-	260°C,4.0 MPa, (Hayashi et al., 2002)
5%Fe/SiO$_2$	Impregnation	14.2	3.5	35.5	-	260°C,4.0 MPa, (Hayashi et al., 2002)
6% Fe/SiO$_2$	Impregnation	8.6	54.0	57.0	20.7	250°C,0.1MPa (Tasfy, 2011)
6% Fe/SiO$_2$	Precipitation	17	45.7	63.4	9.6	250°C,0.1MPa (Tasfy, 2011)

Table 2. Comparison of performance of Fe-based nanocatalysts in FT reaction

These results also show that the optimum size of Co nanoparticles is around 7.8 – 7.9 nm. Smaller Co nanoparticles which were obtained via other preparation routes were found to result in lower C_{5+} selectivity. SiO_2-supported Co nanocatalysts prepared via the strong electrostatic adsorption method resulted in higher C_{5+} selectivity than those obtained using the impregnation method when FT reaction was conducted at atmospheric pressure (Ling, 2011, Ali et al., 2011).

It has been reported that the iron nanocatalyst prepared via microemuslion exhibited high selectivity towards C_{2+} oxygenates (Hayashi et al, 2002). At the same Fe loading, the size of Fe nanoparticles prepared via impregnation was about three times larger than that of microemulsion which could have led to higher CH_4 production even when the reaction was carried out at 4 MPa pressure. As expected, the selectivity to CH_4 increased by decreasing the reaction pressure. The impregnated iron nanocatalyst exhibited better performance towards C_{5+} than that obtained from the microemulsion nanocatalyst. However, the microemulsion catalyst resulted in much lower CH_4 selectivity compared to that of the impregnated and precipitated Fe nanocatalysts. At atmospheric pressure, the Fe nanocatalyst prepared via impregnation resulted in lower CH_4 selectivity and higher C_{5+} selectivity compared to those obtained over the precipitated Fe nanocatalyst.

5. Conclusion and outlook

The average size and size distribution of the active metal nanoparticles namely, iron and cobalt, are influenced by the catalysts preparation route. Each preparation method has its own advantageous and disadvantageous. The impregnation method seems to be the simplest preparation route, unfortunately, it does not always produce small nanoparticles at narrow particle size distribution. The reverse microemulsion method has several advantageous

compared to the conventional impregnation method, as theoretically the size of the metal nanoparticles could be easily controlled through manipulation of the size of the microemulsion droplets by varying the water-to-surfactant ratio. These elegant nanoreactors, however do not always generate nanoparticles at the desired size range with good dispersion due to interaction with the catalysts support.

For the Co-based nanocatalyst, the reverse microemulsion technique was able to produce cobalt nanoparticles at 7.8 nm which showed good performance in terms of achieving high selectivity to C_{5+} and low CH_{4+} selectivity. More improvements are still required before the reverse microemulsion method can substitute the simpler, less elegant impregnation method.

6. Acknowledgment

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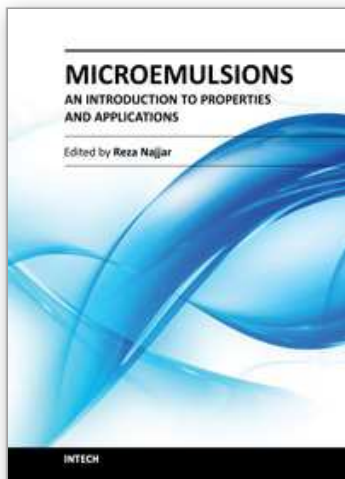
Microemulsions - An Introduction to Properties and Applications

Edited by Dr. Reza Najjar

The rapidly increasing number of applications for microemulsions has kept this relatively old topic still at the top point of research themes. This book provides an assessment of some issues influencing the characteristics and performance of the microemulsions, as well as their main types of applications. In chapter 1 a short introduction about the background, various aspects and applications of microemulsions is given. In Part 2 some experimental and modeling investigations on microstructure and phase behavior of these systems have been discussed. The last two parts of book is devoted to discussion on different types of microemulsion's applications, namely, use in drug delivery, vaccines, oil industry, preparation of nanostructured polymeric, metallic and metal oxides materials for different applications.

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