

CONCLUSION AND RECOMMENDATION

Conclusion

From the experiment results and discussion of this study to achieve the objectives of this research, the conclusion can be drawn into four parts; 1) SUZ-4 zeolite synthesis and characterization 2) Nanostructured composite synthesis and characterization 3) Evaluation of nanostructured sorbents in differential bed reactors for elemental mercury capture and 4) Photocatalytic degradation of methyl orange dye.

1. Part I: SUZ-4 Zeolite Synthesis and Characterization

Hydrothermal sol-gel process with TEAOH (Tetraethylammonium hydroxide) as structure directing agent has been used to successfully synthesize SUZ-4 zeolite. The effect of parameters; stirring and rotation, chemical compositions, time and temperature, for crystallization has been carried out. Na^+ cation inhibited the formation of SUZ-4 and it make the promoting the crystallization of the impurity competing phase. The overall results obtained here show that stirring plays one of the key roles for reproducible synthesis. SUZ-4 zeolite structure with well-defined phase was only obtained under stirring of 300 rpm or rotating of 30 rpm for different autoclave reactors model. The molar ratio of $\text{SiO}_2:\text{Al}_2\text{O}_3$ was varied at 33.3, 21.2 and 16.2 and it related to $\text{TEA}_2\text{O}:\text{Al}_2\text{O}_3$, $\text{H}_2\text{O}:\text{Al}_2\text{O}_3$ and $\text{K}_2\text{O}:\text{Al}_2\text{O}_3$, crystallization temperature and time. The results reveal that gel chemical composition of mixture was also very sensitive for SUZ-4 synthesis since unidentified and phillipsite were common competing phases. Specific surface area, pore volume and pore size distribution has been reported and confirmed for microporous crystals. Under the conditions employed in the experiments here, the suitable SUZ-4 formation takes place for $\text{SiO}_2:\text{Al}_2\text{O}_3$ ratio of 21.2 for 4 days at 155 °C crystallization under autogenous pressure. It is clearly possessed a high micropore volume at 0.1089 cm^3/g , 440.4 m^2/g BET surface area, narrow pore size distribution and 0.07 μm diameter and 0.64 μm long of needle crystal shape. Novel SUZ-4 zeolite membrane has been produced under this condition in rotating autoclave reactor. The results also show the

negative zeta potential values both SUZ-4 zeolite powder and mullite. The experiments were needed to control and adjust the conditions to get good characteristic of SUZ-4 zeolite membrane. Moreover, it was found that with seeding has more thickness of zeolite layer than without one. However, these experiments for SUZ-4 membrane were preliminary results and should be developed in systematic to further study.

2. Part II: Nanostructured composite Synthesis and Characterization

One step flame aerosol synthesis has been successfully carried out to produce $\text{SiO}_2\text{-TiO}_2$ and $\text{SiO}_2\text{-Al}_2\text{O}_3$ nanocomposite particles. Depending on experimental conditions, particles with varied precursor molar ratio of Si:Ti and Si:Al and quench ring position could be readily produced. The addition of secondary material into an oxide can control phase transition. The different precursor concentrations for nanocomposite synthesis make different crystal phases for both systems. However, at a given precursor concentration and at lower quench ring position, a binary mixture of $\text{SiO}_2\text{-TiO}_2$ was obtained in a core-shell structure, but $\text{SiO}_2\text{-Al}_2\text{O}_3$ system was found only as solid-solid solution structure. Different types of precursors have different decomposition kinetics leads to different particle morphologies. Different residence time for sintering and coalescing from changing quench ring position also produces different morphologies for $\text{SiO}_2\text{-TiO}_2$ nanocomposite. Quenching can stop the growth kinetics at different positions in the particle formation process. SiO_2 can inhibit TiO_2 transformation of anatase to rutile phase and can suppress γ -phase of Al_2O_3 for $\text{SiO}_2\text{-TiO}_2$ and $\text{SiO}_2\text{-Al}_2\text{O}_3$, respectively. It was found that the specific surface area of nanocomposites slightly increased with increasing SiO_2 content for both systems. All synthesized nanocomposites showed Si-O-Ti bonding and Si-O-Al bonding for $\text{SiO}_2\text{-TiO}_2$ and $\text{SiO}_2\text{-Al}_2\text{O}_3$ system, respectively. Moreover, nanostructure composites of $\text{SiO}_2\text{-TiO}_2$ were found to be better dispersion than pure TiO_2 . $\text{SiO}_2\text{-Al}_2\text{O}_3$ nanocomposite is also better dispersion than single component of SiO_2 or Al_2O_3 which is a result of more surface charge by zeta potential.

3. Part III: Evaluation of Nanostructured Sorbents in Differential Bed Reactors for Elemental Mercury Capture

A differential bed reactor (DBR) system was used to evaluate the Hg^0 capture by various nanostructured sorbents. The total of eight sorbents was chosen: four types of commercial iron oxide, titanium dioxide, titania PILC, synthesized magnetite, and synthesized SUZ-4 zeolite. Experimental results indicated that the capture rates were higher in presence of UV irradiation. Thus, the effective pathway of mercury capture in tested sorbents: TiO_2 , TiO_2 PILC, Trans Oxide Brown, and SUZ-4 zeolite involved a Hg^0 oxidation step on the surface of the sorbent. The maximum initial rate of Hg^0 capture per unit mass of sorbent was $13.5 \mu\text{g}/(\text{m}^3\text{min})$ for the nanostructured TiO_2 . The parameter α in the rate equation for each sorbent was determined having the value ranging from 1.03 to 1.51. Moreover, the rate constant (k) was in the range of 3.41×10^{-20} to $2.43 \times 10^{-17} \mu\text{g}^{1-\alpha} \text{m}^{3\alpha-2} \text{s}^{-1}$. Binding mechanisms and the mobility of mercury for four types of sorbent were investigated by using a three step sequential extraction. The mercury associated with Trans Oxide Brown was found to be more labile than the mercury associated with the other sorbents. TiO_2 shows the greatest potential to be used as a sorbent to capture elemental mercury with UV irradiation based on the high capture efficiency rate and firm binding characteristics. However, sorbents regeneration and reuse should be considered and studied for potential applications of mercury capture in the reactors in practical conditions (packed bed geometries would have some problems from a view point of pressure drop issues). Moreover, further studies are needed to gain a better understanding of adsorption of mercury on zeolite and Trans Oxide Brown adsorbents.

4. Part IV: Photocatalytic Degradation of Methyl Orange Dye

The photocatalytic degradation of methyl orange dye in pure TiO_2 , TiO_2 -PILC, SUZ-4 zeolite and SiO_2 - TiO_2 nanocomposite materials suspension was tested at different degradation times by UV-vis spectra. The decrease in the photocatalytic activity and UV shielding effect of silica-coated TiO_2 nanoparticles was revealed. The photocatalytic of methyl orange was not degraded by the TiO_2 -PILC and SUZ-4

zeolite. This finding shows that the photocatalytic degradation of TiO_2 is suppressed well by the amorphous SiO_2 layer. Moreover, dispersion of the silica-coated TiO_2 nanoparticles was confirmed by zeta potential analysis. Thus the silica-coated particles can be dispersed more rapidly when compared with the non-coated particles.

Recommendation

Even though the results from this study indicate new findings for novel technique of nanostructured materials synthesis both sol-gel method and flame aerosol technique and their applications, the other issues related to this study should be further deeply studied. From the result of the study, recommendations are given as follow.

1. Synthesized SUZ-4 zeolite membranes should be reproduced and tested for gas permeation as catalytic membrane reactor. The preparation of in-situ crystallization with/without seeding on supporter is important. Due to synthesis conditions of new SUZ-4 zeolite membrane are sensitive and complicated, high-quality membrane preparation can prevent cracking film and achieve for testing as catalytic membrane reactor. The systematic experiments of synthesis in crystal orientation, plane deposition and surface charge should be studied.

2. Synthesized SUZ-4 zeolite powder should be studied for other application from their beneficial physical property. Moreover, local raw materials (both china clay and rice husk ash) should be studied for SUZ-4 zeolite powder and membrane to raise a value added.

3. Type of precursors and configuration of flame reactor should be considered to synthesize $\text{SiO}_2\text{-TiO}_2$ and $\text{SiO}_2\text{-Al}_2\text{O}_3$ nanocomposite materials in different morphology and crystal phase according to directly applications.

4. From this research, evaluation of nanostructured sorbents for elemental mercury capture only focused on initial rate capture. Saturation adsorption of

sorbents in different running time and reaction temperature for mercury capture should be studied to fully access the real application.