



CHAPTER 5

CONCLUSIONS

From the experiment results, the following conclusion can be drawn:

Preparation of amorphous silica from rice husk

Rice husk byproduct could be converted into high valuable silica source at optimum conditions. The best quality of silica, in terms of purity, surface area and phase formed were obtained by acid leaching and heat treatment. Precleaning by acid leaching method can improve chemical and physical properties of RHA. A high purity of silica (~99.6%) and amorphous phase with a large surface area ($240 \text{ m}^2/\text{g}$) can be obtained by burning acid-leached RH at $700 \text{ }^\circ\text{C}$ for 3 h in air.

Synthesis of ZSM-5 zeolite and silicalite from RHA

1. ZSM-5 zeolite with a high crystallinity could be synthesized from a precursor gel containing a silica source from RHA. The crystallinity and crystallization rate depended on the properties of RHA used. Acid-leached RHA yielded higher crystallinity of ZSM-5 and provided faster crystallization rate than that of unleached RHA, due to its high purity and high specific surface area enhancing the dissolution of silica. In addition to acid-leached RHA, the use of ground RHA also enhances the dissolution of silica in NaOH alkali solution, leading to a dramatic decrease in the organic template content and the synthesis time.

2. The fine grain size with a large surface area, high degree of purity and the amorphous state of RHA silica combined with the constrained composition ($\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio) of the precursor gel, which enhances the dissolution of silicate and aluminate species, serve to thermodynamically speed up the nucleation and crystallization rate, and stability of ZSM-5. As a result, well crystalline ZSM-5 zeolite with a large surface area and well crystalline silicalite are successfully synthesized from RHA silica by

hydrothermal hydrolysis at 150 °C under autogenous pressure in a short reaction time, 4-24 h. The maximum yield of zeolite, at 84% (w/w), was obtained at SiO₂/Al₂O₃ molar ratio of 80, and the transition of ZSM-5 zeolite to silicalite occurs at SiO₂/Al₂O₃ molar ratio of >200. This also demonstrates that without additional Al⁺³ ions, the original SiO₂/Al₂O₃ molar ratios (2075) of the RHA silica alone can produce silicalite.

Photocatalytic activity of TiO₂/ZSM-5 composite

The SO₄²⁻ ion did enhance photocatalytic reaction but there was an optimal content limit. Filtering removed most of the SO₄²⁻ salt which would adhere to the TiO₂ surface, and the remaining salt after calcination was the ionizable Na₂SO₄ which acted as a bridging agent for the TiO₂ sol, promoting flocs which enhanced the physical adsorption through a charge effect of the dye and the subsequent photocatalytic reaction. The combined effect of SO₄²⁻ ion and the composite TiO₂/ZSM-5 was to initiate the large flocs with fast settling and ease of removal leaving a clear supernatant liquid. In summary, the presence of SO₄²⁻ ion, the effect of the composite TiO₂/ZSM-5 and high temperature calcinations could improve the photocatalytic reaction of the obtained TiO₂ close to those of P-25 and ST-01. The instantaneous dye adsorption in the dark by sample with high TiO₂ loading 1:1 was very interesting since its mechanism was independent of light and the ease of removal of the settled spent flocs could be of good benefit in wastewater treatment. This finding is very interesting in that it can greatly reduce the working time in waste management. The effectiveness of catalysts owes much to the principles underlying the process of textile dyeing.