

# Ethylene permeation of silicalite-1 composite film

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## ABSTRACT

## INTRODUCTION

Thailand is an agriculture country and its major export income come from an agriculture product. The agriculture products have a short life, thus preserving freshness and keeping quality of the product are an important issue. Normally, the conservation of these products can be achieved by controlled oxygen, carbon dioxide and ethylene. In particular, the silicalite is regarded as hydrophobic molecular sieves with ethylene adsorption capacity and it has pore size bigger than kinetic diameter of ethylene 0.39 nm. The silicalite-1 was selected for ethylene adsorption. The ultimate objective of this research is to study ethylene permeation of silicalite-1 composite polymer film. The composite film is obtained from solution-casted process. The effect of polymer type and zeolite content on ethylene permeation in composite film was investigated.

## EXPERIMENTAL PROCEDURE

### 1. Synthesis and characterization of silicalite-1

The silicalite-1 was prepared from a clear hydro gel with the molar composition  $10\text{SiO}_2 : 1.4\text{TPABr} : 1\text{NaOH} : 110\text{H}_2\text{O}$ . The solution as mixed and aged for 24 hr. After aging, the initial mixture is heated at 180 °C for 24 hr. The silicalite was purified and dried at 110 °C for 24 hours in the oven. Finally, silicalite will be calcined under Air-zero at 550 °C with a heating rate of 2 °C/min for 5 hrs and cooled under stream of nitrogen. The silicalite-1 was characterized using XRD, SEM, BET and ethylene oxygen and carbon dioxide adsorption.

### 2. Preparation of the composite film

The composite film was prepared by solution-casted process. SEBS 1652(MFI 5 g/10min) and SEBS1657 (MFI 22 g/10min) was prepared 3%wt of the polymer in toluene. The mixture was heated to approximately 60°C with stirring with time 6.30 hr. The solution was sonicated for 15 min and then casted on a glass plate for 24 hr. After that, the film was evacuated in vacuum oven at 60°C for another 24 hr. The silicalite composite film of SEBS 1652 and SEBS1657 were prepared with silicalite-1 loading as 5 to 30% wt and 10% wt, respectively.

### 4 Gas Permeation of the composite film

The ethylene permeation of film was determined using Homemade Permeation cell equipment with FID Gas chromatograph. The ethylene and nitrogen used as a carrier gas were carried with 10ml/min by mass flow meter. The injection port, the column oven and FID detector is set at 185, 100 and 250 °C, respectively. After 5 hours, the permeate was analyzed every 15 minutes for at least 4 times in order to determine the permeate gas at steady state. The oxygen and carbon dioxide permeation of films were investigated by oxygen and carbon dioxide permeability analyzer, respectively.

## RESULTS AND DISCUSSION

### 1. Silicalite-1 characterization

The XRD pattern shown, the silicalite-1 crystal had the MFI structure. The crystal size was approximately 1 µm by SEM as shown in Figure1. From both results, it was shown that the silicalite-1 defined as a good crystalline material. The BET technique shown the specific surface area of silicalite-1 was 458 m<sup>2</sup>/g which has large specific surface area and thus high possibility of adsorption capacity would be obtained.

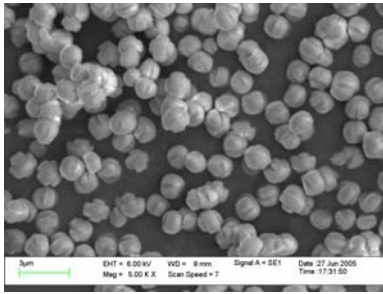


Figure 1. The morphology of silicalite

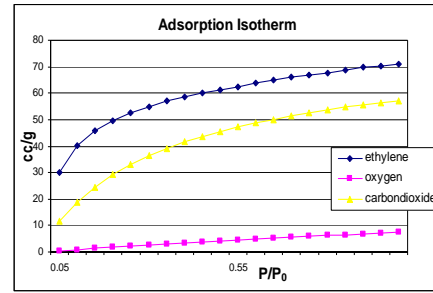


Figure 2. Gas adsorption isotherm of silicalite

For the adsorption isotherm in Figure 2, it was found that oxygen showed weaker adsorption compared to carbon dioxide and ethylene adsorption isotherm. The ethylene, oxygen, carbon dioxide adsorbed were 70, 7.3 and 57 cc/g respectively.

### 2. Gas permeation of the composite film

Consideration of the ethylene permeation of SEBS 1652 and 1657 film, it was found that the SEBS 1657 had a higher permeation than SEBS 1652 as shown in Figure 3.

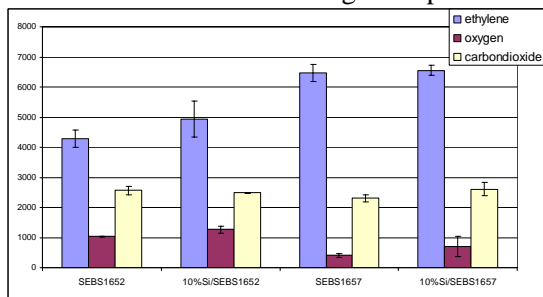


Figure 3 Ethylene, Oxygen and Carbon dioxide

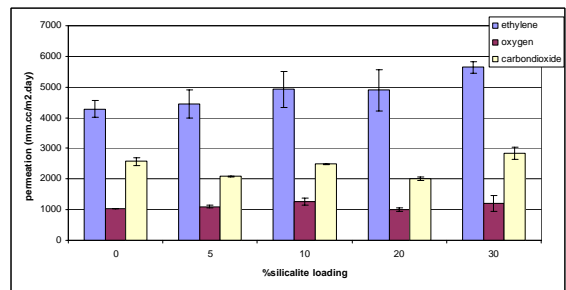


Figure 4 Ethylene, Oxygen and Carbon dioxide permeation of Silicalite-1/SEBS1652 composite film

Permeation of film SEBS1652 and SEBS1657 This is because SEBS 1657 had a higher Melt Flow Index which influence to a low viscosity. Also the SEBS 1657 had a lower styrene content that leads to a high flexibility. Both Melt Flow Index and styrene content affected on high ethylene permeation of SEBS 1657 more than SEBS 1652. Addition 10 %wt of silicalite-1 in a composite film resulted higher ethylene permeation compared to polymer film. However, the ethylene permeation of zeolite composite SEBS 1652 film was founded a significant increasing while SEBS 1657 exhibited insignificant increment. Consequently, the SEBS 1652 was chosen to be a prototype for a varied of silicalite contents from 5 to 30 %wt as shown in Figure 4. It was found that the ethylene permeation value increased from 4,284 to 5,648 mm<sup>3</sup>·cm<sup>2</sup>·day. The ethylene permeation was increased when silicalite was added. Moreover, oxygen and carbon dioxide permeation was founded the plateau trend. Therefore, the adding silicalite-1 was led to high ethylene permeation and high selectivity.

### CONCLUSION

From this research, the chosen SEBS was founded a high Melt Flow Index, which influenced to a low viscosity and had a lower styrene content that led to a high flexibility affected on high ethylene permeation. Furthermore, loading silicalite-1 into composite film enhanced high ethylene permeation. Consequently, the fact that silicalite-1 had a hydrophobic property, high surface area and prefer ethylene adsorption. Finally, the silicalite composite SEBS had higher ethylene permeation.

**KEY WORDS** – Ethylene permeation, Silicalite-1, composite film

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